

WORLD CERAMICS ABSTRACTS

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World Ceramics Abstracts provides comprehensive coverage of the world's literature on ceramic materials, processing and applications.

Coverage includes advanced materials, engineering ceramics and electroceramics, refractories, traditional ceramics (tableware, tiles and sanitaryware), bricks and roofing tiles, glass and vitreous enamels.



COMMERCIAL AND ECONOMIC

See also Abstract(s): 213303 213380

Production organisation and planning

See also Abstract(s): 213685 213686

ENVIRONMENT AND POLLUTION

See also Abstract(s): 213311 213312 213313 213332 213334 213357 213390 213391 213395 213396 213397 213406 213408 213411 213438
213470 213510 213538 213556 213627

213297

VITRIFICATION OF MUNICIPAL SOLID WASTE INCINERATION FLY ASH: AN APPROACH TO FIND THE SUCCESSFUL BATCH COMPOSITIONS

Sharifikolouei E; Bairo F; Salvo M; Tommasi T; Pirone R; Fino D; Ferraris M - *Turin, Polytechnic*

Ceram.Int. 47, No.6, 2021, p.7738-7744

Two different sources of municipal solid waste incinerator fly ash were vitrified to inhibit the release of potentially toxic heavy metals. Two different sources of silica, i.e. silica sand and glass cullet, were added to each type of fly ash in an attempt to obtain vitrifiable batches. The standard leaching test on the vitrified products was performed, according to EN12457-2, and confirmed no heavy metal leaching and, therefore, they passed the waste acceptance criteria to be classified as an inert material. Furthermore, the previously reported data for the vitrification of fly ash was combined with the present work and their compositions, presented in the SiO₂-Al₂O₃-CaO, and SiO₂-Sigma(M₂O₃)-Sigma(MO + M₂O) ternary phase diagrams, identified the region in which successful compositions were concentrated. This analysis could help to find the right composition for the vitrification of fly ash.

213298

HOW TO PAVE THE WAY WITH STRATEGIES FROM REFRACTORY SUPPLIERS FOR A LOW-CARBON FUTURE OF THEIR USER INDUSTRIES?

Parr C; Edwards P - *Imerys Aluminates*

Refract.Worldforum. 13, No.3, 2021, p.8-10

Steps taken by Imerys to move towards a low-carbon future for themselves as refractory mineral suppliers and for refractory user industries, such as steel and cement, by reducing their CO₂ footprint, reducing pollutant emissions and reducing water consumption are outlined. These include use of alternative energy burners, using CFD to model combustion and furnace dynamics, implementing more recycling and remapping refractory binders to zero-emission processes. A range of products is also available to provide sustainable solutions.

213299

ON THE FUTURE OF THE BRICK INDUSTRY IN EUROPE

Modinger F

Interceram. 70, No.3, 2021, p.14-15

Ways in which the European brick industry can meet the challenge of climate neutrality by 2050 are discussed. The main challenges are the costs of electrical energy from alternative energy sources and the CO₂ balance of their generation processes. Estimates for increased costs of bricks are given and whether such increases will lead to different construction methods and living habits is discussed.

INDUSTRIAL HEALTH AND SAFETY

See also Abstract(s): 213310 213628

213300

IMMOBILISATION OF HEAVY METALS IN HAZARDOUS WASTE INCINERATION RESIDUE USING SiO₂-Al₂O₃-Fe₂O₃-CaO GLASS-CERAMIC

Chen H; Lin H; Zhang P; Yu L; Chen L; Huang X; Jiao B; Li D - *Chongqing, University; Chongqing, Environment Monitoring Centre; Anhui, University of Technology*

Ceram.Int. 47, No.6, 2021, p.8468-8477

Hazardous waste incineration residue (HWIR) is categorised as hazardous waste due to the presence of heavy metals such as Zn, Cu, and Cr. Based on the abundant components of silica, alumina, iron oxide and calcium oxide in a HWIR from an environmental protection corporation in China, a 100% HWIR was used to prepare SiO₂-Al₂O₃-Fe₂O₃-CaO glass-ceramic samples. Results showed that Zn, Cu, and Cr present in the glass-ceramic samples were effectively immobilised during the melting-sintering process. Among the four parameters of heat treatment, the crystallisation temperature played the most significant role. The glass-ceramic, formed under optimal conditions, contained haematite, pseudobrookite and anorthite, with superior compressive strength, volume density and water absorption of 204.84 MPa, 2.80 g/cm³, and 1.20%, respectively. Compared with the raw HWIR sample, the leaching concentrations of Zn, Cu, and Cr from the prepared glass-ceramic samples decreased and their immobilisation efficiencies exceeded 99%. Physical encapsulation in the glass-ceramic samples together with ion exchange in the amorphous glassy matrix and iron-rich crystalline phases were the main immobilisation mechanisms for Zn, Cu, and Cr.

RAW MATERIALS

See also Abstract(s): 213312 213640

213301

APPLICATION OF QUALITATIVELY IMPROVED EXPANDED PERLITE (REVIEW)

Gurgenyan N V; Khachanova I B; Kostandyan M F; Grigoryan A E; Vardanyan N K - *Yerevan, Institute of General and Inorganic Chemistry; Yerevan, National University of Architecture and Construction of Armenia*

Glass Ceram. 78, No. 3/4, 2021, p. 161-167

A comparison of the physicochemical characteristics of current and planned perlite heat-insulation materials, agricultural components, and acoustic fillings is provided, indicating considerable increase in the latter's quality. In this regard, a selective method for developing perlite deposits in thin extraction layers is proposed in order to obtain a homogeneous perlite rock in terms of average density with classification by size fractions, allowing for the production of a homogeneous raw material and expanded perlite with a stable set of properties.

Minerals

See also Abstract(s): 213298 213315

213302

ON THE POSSIBILITY OF ENRICHING QUARTZ RAW MATERIALS OF UZBEKISTAN FOR THE GLASS INDUSTRY

Aripova M Kh; Mkrtchyan R V; Erkinov F B - *Tashkent, Chemical Technology Institute*

Glass Ceram. 78, No. 3/4, 2021, p. 120-124

The lack of enrichment facilities for quartz raw materials in Uzbekistan makes it difficult to improve product quality and competitiveness. The goal of this research was to see if it was possible to enhance quartz raw materials physically. In the laboratory, sieving, washing, sorting, scrubbing, and magnetic influence were employed. The most viable techniques for enriching quartz sands, as well as the potential for enriching vein quartz and quartzite, are highlighted for the glass industry.

213303

VIRTUAL REFRACTORY MINERALS FORUM 2021

O'Driscoll M - *IMFORMED Industrial Mineral Forums & Research Ltd.*

Refract. Worldforum. 13, No. 3, 2021, p. 28-32

Papers presented at the online 2021 IMFORMED Forum on refractory raw materials are summarised. The 13 papers were on: global refractory materials overview; the effect of the circular economy and climate change on refractory mineral markets; supply trends for Chinese refractory minerals; India's response to refractory raw material supply and demand; dolomite; global refractories outlook; review of refractory magnesia supply outside China; raw material selection for refractory applications; refractory bauxite trends; First Bauxite's bauxite supply from Guyana; andalusite supply; the graphite supply chain; and speciality grade chromite.

213304

PURIFICATION AND DENSIFICATION OF HIGH PURITY SINTERED MAGNESIA FROM NATURAL MAGNESITE IN CHINA

Guo Z; Bi D; Sun S; Yu Z - *Yingkou Jindai Technologies; Shenyang Dongda Dongke*

Refract. Worldforum. 13, No. 3, 2021, p. 67-71

China is the largest magnesia producer in the world, but the high-grade resources are diminishing due to the amount of mining in the last three decades. There are still tremendous reserves of low grades and abandoned small lump magnesite. A flotation process has been developed and is in operation to upgrade magnesite to the purity of > 98% MgO (LOI-free), removing impurities such as SiO₂, CaO, Fe₂O₃ and Al₂O₃. The floated magnesite is calcined in a newly designed suspension calciner, instantaneously in flash decomposition, to produce caustic calcined magnesia (CCM) with much higher activity and ground fineness compared to that made in conventional reverberatory and multiple hearth furnaces. Successful densification of CCM briquettes was achieved in the industrial production (capacity > 100000 t/a) of sintering high purity magnesia (> 98% MgO), obtaining a high bulk density of 3.42 g/cm³ from natural macrocrystalline magnesite.

213305

KAOLINITE-BASED CLAY CERAMICS BLENDED WITH RESIDUAL FIQUE FIBRES FOR POTENTIAL PLASTIC SOIL APPLICATIONS

Garcia E F; Perez A C; Colorado H A - *Medellin, Universidad de Antioquia*

Int. J. Appl. Ceram. Technol. 18, No. 4, 2021, p. 1086-1096

The high content of clay minerals in soil has a significant effect on its engineering behaviour; water affects the interaction of the minerals which, in turn, influences their plasticity and deformability. Structures made with, or on, plastic materials can experience problems due to the high deformation in the soil. Decreasing the deformability of plastic soils contributes to the stability and serviceability of the structures supported by this kind of soil. The effect of fique fibre (a natural fibre from Colombia) on the mechanical behaviour of a plastic soil based on a kaolinitic clay was studied. Soil with additions of 0.0, 0.2, 0.7 and 1.0 wt% fique fibres was fabricated with the aim of enhancing the compressive strength of the soil and adding an eco-friendly reinforcement to the soil, thereby targeting a circular economy solution. The density, optimal water content and unconfined compressive strength were determined. The microstructure was characterised by SEM and XRD. The results showed that fique fibres improved not only the compressive strength by almost three times for the sample containing 1.0 wt% fibres with respect to the fibre-free reference sample but also enhanced the ductility.

Processed raw materials, synthesised powders

See also Abstract(s): 213304 213356 213384 213401 213403 213452 213469 213470 213473 213478 213491 213496 213501 213620 213646

213655 213672 213693

213306
PREPARATION OF HIGH-TEMPERATURE ACTIVE ZIRCONIUM BORIDE POWDERS VIA PRECURSOR ROUTE AND MICROWAVE SINTERING
Ding Z; Huang X; Liu W; Kim I J; Han Y-H - *Guangzhou, University; Ningbo Tuopu Group Co., Ltd.; Seosansi, Institute for Processing and Application of Inorganic Materials; Wuhan, University of Technology*
Adv. Appl. Ceram. 120, No. 4, 2021, p. 222-226
Microwave heating utilising the sol-gel technique for a precursor with eight C₆H₁₄O₆ (ZrOCl₂·8H₂O), boric acid (H₃BO₃), citric acid (I), and mannitol resulted in ZrB₂ powders (as raw materials). According to the data, the temperature at which the ZrB₂ powder was entirely synthesised by microwave heating was 1400 C, which is 300 C lower than the traditional method. The powders' phase composition and shape were studied using XRD and SEM. Furthermore, the impacts of raw material ratios and synthesis temperature on powder synthesis were studied. Temperature had a significant impact on the purity of the synthesised zirconium boride. Microwave synthesis of almost spherical ZrB₂ ultra-fine particles of high purity was accomplished at 1400 C, with C:Zr ratios of 7.44:1 and B:Zr ratios of 2.77:1.

213307
ENZYME IMMOBILISATION USING TWO PROCESSING METHODS ONTO SILICA CORE-SHELL PARTICLES
Nikolic M P; Pavlovic V B; Stanojevic-Nikolic S; Srdic V V - *Kragujevac, University; Belgrade, University; Novi Sad, University*
Bol. Soc. Esp. Ceram. Vidrio. 60, No. 4, 2021, p. 243-254
Two methods of enzyme immobilisation onto silica core-shell particles were developed. The first involved the immobilisation of *Candida rugosa* lipase inside a previously synthesised mesoporous silica layer (deposited at 80 C) surrounding a dense silica core. An outer mesoporous silica layer was deposited at 40 C around the first silica layer containing the immobilised lipase at a relatively lower temperature, to prevent thermal inactivation of the immobilised enzyme. The internal silica layer was obtained by assembling primary silica nanoparticles generated from highly basic sodium silicate solution at 80 C on the surface of PDDA functionalised silica core particles. The average shell thickness and pore size of the internal silica layer was about 60 nm and 24 nm, respectively. The effect of processing parameters on the generation and aggregation of silica nanoparticles prepared from highly basic sodium silicate solution was also studied. The aggregation of silica particles generated at 40 C and 80 C took place after 840 s and 570 s of the reactions, respectively. The immobilisation efficiency of the lipase on the mesoporous silica monolayer was 80%. Immobilised lipase activity declined by about 6 times after 10 reaction cycles due to lipase leakage from the monolayered shell. An outer mesoporous silica layer was deposited at 40 C onto the surface of previously PDDA-functionalised monolayered silica core-shell particles containing the immobilised lipase. The layer had an average thickness and pore size of about 60 nm and 17 nm, respectively. The activity of lipase immobilised inside the bilayered shell was further reduced due to diffusion resistance within the outer silica layer and PDDA layer but it was retained for the next reaction cycles. The pore size of the mesoporous silica layer obtained at 80 C was insufficient to allow invertase immobilisation. The second method for the immobilisation of invertase involved the preparation of the mesoporous silica layer simultaneously with invertase immobilisation at 40 C. The immobilised invertase showed decreased activity, but no substrate inhibition, as in the case of the free enzyme, due to the location of the enzyme inside the mesoporous silica layer, where the mass transfer resistance for the substrate to the enzyme active site was present.

213308
LOW TEMPERATURE-RAPID PREPARATION OF HfB₂-SiC POWDERS BY MICROWAVE/MOLTEN SALT ASSISTED BORO/CARBOTHERMAL REDUCTION
He J; Zeng Y; Huang Z; Liu J; Cao Y; Yuan G; Li K; Jia Q; Zhang H; Zhang S - *Wuhan, University of Science & Technology; Zhengzhou, University; Exeter, University*
J. Ceram. Soc. Jap. 129, No. 8, 2021, p. 528-534
Mixtures of hafnium dioxide, silica, boron carbide and activated carbon were used to synthesise HfB₂-SiC powder mixtures by borothermal/carbothermal reduction in molten salt with microwave heating. The molten salt was a mixture of NaCl and KCl. Temperatures were in the range 1100-1250 C, maintained for 0-40 min. The prepared powders were evaluated by microstructure and morphology studies, and determinations of phase composition. Rod-shaped single crystals of HfB₂ (length 0.5 micron, diameter 300 nm) and SiC particles of irregular shape, with a diameter of about 300 nm, were obtained. The optimum HfO₂:SiO₂:B₄C:C raw material proportions were 3.0:1.0:3.2:4.0, and the optimum processing conditions were 1250 C for 20 min. The synthesis at a relatively low temperature for a short time is attributed to the use of molten salt media combined with microwave heating.

213309
PREPARATION OF Al₂O₃@CaCO₃ AGGREGATES AND ITS EFFECTS ON THE THERMAL SHOCK RESISTANCE OF Al₂O₃-MgO CASTABLES
Qu J; Ding D; Xiao G; Lei C; Luo J; Chen J; Zang Y - *Xian, University of Architecture & Technology*
Int. J. Appl. Ceram. Technol. 18, No. 4, 2021, p. 1379-1391
Al₂O₃@CaCO₃ aggregates were prepared by impregnating corundum aggregates (particle sizes of 1-3 and 3-5 mm) in precursor solutions (calcium hydrogen citrate, CaHC₆H₅O₇) followed by heat treatment at 430 C. The phase composition and microstructure of the coatings were characterised by XRD and SEM. The novel aggregates were used in Al₂O₃-MgO castables, and their effect on the physical properties and thermal shock resistance (TSR) of the castables was evaluated. The results show that a uniform CaCO₃ coating of aggregates (C15) with a thickness of about 10 micron can be attained when the Ca²⁺ concentration in solution was 0.15 mol/L. There was strong bonding between the aggregates and coating; the particle size of the latter was about 0.2 micron. Improvements in both the physical properties and TSR of the castables are related to the unique layer structure, a calcium hexaluminate (CA₆) layer formed in-situ at the aggregate-matrix interface, of the added Al₂O₃@CaCO₃ aggregates. Multi-deflection of cracks along with the CA₆ layer consumes more fracture surface energy. The castables with C15 exhibit optimal TSR, and the residual strength ratio after thermal shock testing is 29.5%, 12.8% higher than castables with conventional corundum aggregates.

CERAMICS FOR BUILDING

See also Abstract(s): 213431 213432

Building materials: clay-based

See also Abstract(s): 213299

213310

CERAMIC TILES AS SUSTAINABLE, FUNCTIONAL AND INSULATING MATERIALS TO MITIGATE FIRE DAMAGE

Naser M Z; Thavarajah P - *Clemson, University; Tile Council of North America*

Adv. Appl. Ceram. 120, No. 4, 2021, p. 227-239

Their reduced prices and functional qualities ensure that synthetic organic polymer (SOP) materials are increasingly being used in the building sector as linings, interiors, and non-load bearing structural components. While SOPs have favourable qualities and attributes at ambient temperatures, the same materials frequently function poorly at moderate-to-high temperatures, such as those caused by a structure fire. In fact, unlike typical construction materials like as concrete and ceramic tiles, most SOPs tend to burn and disintegrate at high temperatures, which has been shown to not only add to the fire but also negatively impact evacuation and firefighting efforts. From the standpoint of fire engineering, this study investigates the premise that ceramic tiles (CTs) will outperform SOPs and frequently used insulations as finishing and lining materials under fire circumstances. As a result, this study provides a comprehensive evaluation of the behaviour of frequently available CTs, SOPs, and insulations in temperatures ranging from 25 to 1000 C. Then, using machine learning, this study analyses published CT models to create temperature-dependent material models (ML). In contrast to SOPs, composites, or insulations, the findings of this study support the use of CTs as favourable finishing and interior lining materials to achieve enhanced structural fire performance and fire response management. This work's findings are likely to be of interest to architects, first responders, building officials, fire and structural engineers.

Building materials: calcium silicates, cements etc

See also Abstract(s): 213384 213658 213681

213311

MECHANICAL PROPERTIES OF MORTAR CONTAINING RECYCLED ACANTHOCARDIA TUBERCULATA SEASHELLS AS AGGREGATE PARTIAL REPLACEMENT

Suarez-Riera D; Merlo A; Lavagna L; Nistico R; Pavese M - *Turin, Polytechnic*

Bol. Soc. Esp. Ceram. Vidrio. 60, No. 4, 2021, p. 206-210

A common solution to the problem of waste management is to use waste materials as a partial substituent of the inert fraction in concretes and mortars. The possibility of using *Acanthocardia tuberculata* seashells, a food waste otherwise destined for landfill, as a partial substituent in mortar was investigated. The results showed that the reduction in mechanical properties (in terms of toughness and flexural stress) was mainly due to the water absorption properties of the seashell aggregates, which affect the hydration of the cement. However, such a decrease in mechanical properties does not affect the performance of the material when used for civil applications.

213312

METAKAOLIN AND DEMOLITION WASTES IN ECO-BASED SAND CONSOLIDATED CONCRETE

Lazaar K; Hajjaji W; Moussi B; Rocha F; Labrincha J; Jamoussi F - *Tunisia, Georesources Laboratory; Gabes, University; CERTE; Aveiro, University*

Bol. Soc. Esp. Ceram. Vidrio. 60, No. 4, 2021, p. 229-242

An attempt was made to valorise naturally occurring silica sand in the synthesis of new consolidated concrete materials. Mixtures of silica sand, calcium sulphate, commercial metakaolin and construction demolition materials were designed to make sulphate and sodium silicate/NaOH activated concretes and geopolymers, respectively. Three raw silica sand samples from various locations in Tunisia were collected. The new materials obtained were characterised by SEM and the mechanical properties were studied. The calcium sulphate-based concretes had good technological properties with a compressive strength close to 15 MPa and 40-56% water absorption. When metakaolin and demolition waste were added, the mechanical resistance decreased due to the lower pozzolanic properties of the materials. Lower compressive strengths were obtained for the geopolymer-based sand concrete. By incorporating demolition materials, the mechanical resistance decreased in all consolidated products. The effect of the metakaolin reactivity was more significant when it was activated with an alkaline solution. However, the sodium silicate/NaOH activation of metakaolin governed the reaction when it was highly reactive. Raw silica sand from Tunisia was shown to give good consolidated concrete materials in the presence of calcium sulphate, and the silica sand provided good geopolymers in the presence of metakaolin and alkaline solution.

213313

LITERATURE MINING FOR ALTERNATIVE CEMENTITIOUS PRECURSORS AND DISSOLUTION RATE MODELLING OF GLASSY PHASES

Uvegi H; Jensen Z; Hoang T N; Traynor B; Aytas T; Goodwin R T; Olivetti E A - *Massachusetts Institute of Technology; IBM Watson; IBM*

T. J. Watson Research Center

J. Am. Ceram. Soc. 104, No. 7, 2021, p. 3042-3057

Lower-emission alternatives are needed to reduce the carbon footprint associated with cement and concrete production and a small subset of potentially useful precursor materials has been found. In order to expand the precursor pool, results of parallel literature mining and rate modelling activities are presented. As a result of literature mining, materials with appropriate SiO₂, Al₂O₃ and CaO concentrations were assembled into a comprehensive, representative ternary diagram. 23000+ materials were extracted from 7000 journal articles, and 7500 materials from 6000 articles with SiO₂ + Al₂O₃ + CaO = 80-105 wt% automatically classified. Both supervised and semi-supervised models were used to predict the dissolution rate of glassy materials with all models from a single data set (802 reported dissolution rates from 105 different glasses). Supervised modelling used linear and decision tree regressions to determine the features most predictive of dissolution rate, resulting in log-linear relationships between rate and pH, inverse temperature (1/K) and non-bridging oxygen per tetrahedron (NBO/T). Semi-supervised modelling was more robust to broader feature inclusion, providing similar predictive ability with a relatively larger set of descriptive features. Results indicated that models trained on data from disparate scientific communities were adequately predictive (root mean square error about 1), particularly under pH at least 7 conditions relevant to the cement and alkali activation communities.

213314

INTERNAL CURING OF BLENDED CEMENT PASTES WITH ULTRA-LOW WATER-TO-CEMENT RATIO: ABSORPTION/DESORPTION KINETICS OF SUPERABSORBENT POLYMERLiu J; Ma X; Shi C; Drissi S - *Hunan,University; Henan,University of Urban Construction; British Columbia,University***J.Am.Ceram.Soc.** 104,No.7,2021,p.3603-3618

Internal curing by a superabsorbent polymer (SAP) is an effective way to mitigate the autogenous shrinkage of cement-based materials with low water-to-cement ratio (w/c). The water absorption/desorption kinetics of SAP were studied quantitatively in blended cement pastes with ultra-low w/c. An absorption process at a rate of 0 to 6 g/g.h was calculated at early ages, after which SAPs showed mainly two distinct water desorption behaviours with a rate of 0 to 1.1 g/g.h, mainly governed by the osmotic pressure and capillary pressure triggered by the drop of internal relative humidity. The size and amount of SAP was important in controlling its absorption and desorption kinetics in the cement paste. Compared with ordinary Portland cement, a different desorption process with a higher release rate was seen in binary and ternary cement pastes, primarily due to the changes in osmotic pressure resulting from the acceleration of cement hydration by silica fume at early ages. Overall, the mitigation of autogenous shrinkage was found to be highly dependent on the absorption and desorption kinetics of SAP.

213315

HYDRATION OF CUBIC TRICALCIUM ALUMINATE IN THE PRESENCE OF CALCINED CLAYSMaier M; Scherb S; Neisser-Deiters A; Beuntner N; Thienel K-C - *Munich,Bundeswehr University***J.Am.Ceram.Soc.** 104,No.7,2021,p.3619-3631

Calcined clay blended cements are important in the strategy of the cement industry to reduce CO₂ emissions. During their hydration, an accelerated aluminate reaction often affects the sulphate balance. An attempt was made to understand the effect of different calcined clays on the hydration of cubic tricalcium aluminate (C3A). A cementitious model system consisting of cubic C3A, quartz powder, calcium sulphate and a model pore solution was studied. The effect of three different calcined clays and one nanolimestone was examined by successive replacement of the quartz powder and variation of the calcium sulphate. Heat flow and hydrate phase development were followed by isothermal calorimetry and quantitative in-situ XRD. Accelerated ettringite formation and sulphate depletion were seen for all calcined clays, while the nanolimestone showed the opposite effect. It was found that adsorption of SO₄ ions and/or Ca-SO₄-complexes at the surface of the calcined clay particles was the main factor inhibiting retardation of the C3A hydration in the absence of a silicate reaction. In the Al-rich systems, retardation through sulphate adjustment seemed to be impeded by additional Al ions, which reacted with Ca adsorbed onto and leached from the C3A surface.

213316

IMPACT OF Fe DOSAGE ON THE ETTRINGITE FORMATION DURING HIGH FERRITE CEMENT HYDRATIONWan D; Zhang W; Tao Y; Wan Z; Wang F; Hu S; He Y - *Wuhan,University of Technology***J.Am.Ceram.Soc.** 104,No.7,2021,p.3652-3664

The influence of Fe dosage and temperature on the formation of sulphotoaluminate hydrates can help to understand the early hydration process of high ferrite cement under steam curing. The impact of Fe dosage on ettringite formation at different temperatures was shown through the combination of experiments and computational molecular dynamics simulations. A Fe dosage no more than 20% clearly accelerated the crystal growth of ettringite by increasing the surface energy in the (001) direction, whereas a higher dosage suppressed the formation of ettringite as the high incorporation of Fe ions into the ettringite crystal was energetically unstable. The chemical environment analysis of Fe in products showed that Fe(OH)₆(3-), compared with Al(OH)₆(3-), preferred to participate in the formation of Al₂O₃-Fe₂O₃-mono rather than ettringite, which was also confirmed by the calculated thermodynamic properties. Understanding the impact and mechanism of Fe on the formation of ettringite under steam curing conditions plays an important role in the use of high ferrite cement.

213317

SELF-SETTING BETA-TRICALCIUM PHOSPHATE GRANULAR CEMENT AT PHYSIOLOGICAL BODY CONDITION: EFFECT OF CITRIC ACID CONCENTRATION AS AN INHIBITORWei L J; Shariff K A; Momin S A; Bakar M H A; Cahyanto A - *Penang,Universiti Sains Malaysia; Jatinangor,Padjadjaran University; Bandung,Padjadjaran University***J.Aust.Ceram.Soc.** 57,No.3,2021,p.687-696

The effect of citric acid (CA) on inhibiting the properties of beta-tricalcium phosphate (beta-TCP) granular bone cement was studied. Beta-TCP granules were exposed to an acidic calcium phosphate solution containing 0.01, 0.10 and 1.00 mol/L of CA under physiological conditions for 10 min. It was shown that increasing the CA concentration up to 1.00 mol/L reduced the self-setting ability of the granules. Compositional and morphological analyses showed that the amount of dicalcium phosphate dihydrate (DCPD) crystals that precipitated to form an interlocking structure between the granules decreased when the CA concentration increased. The decreasing trend of DCPD formation in set specimens increased their porosity and reduced the compressive strength by up to 27.4%. Thus, it was shown that, by altering the CA concentration in an acidic calcium phosphate solution, the setting reaction of beta-TCP granules under physiological conditions was inhibited.

213318

COMPARATIVE STUDY: NANOSILICA, NANOALUMINA, AND NANOZINC OXIDE ADDITION ON THE PROPERTIES OF LOCALISED GEOPOLYMERZidi Z; Lifi M; Zafar I - *Gabes,University; Riyadh,Imam Muhammad Ibn Saud Islamic University***J.Aust.Ceram.Soc.** 57,No.3,2021,p.783-792

Interest in improving the performance of geopolymers by introducing nanoparticles has grown rapidly in recent years. A detailed comparative analysis of nano-added geopolymers was conducted using XRD, XRF, FTIR, SEM, mercury intrusion porosimetry and TG, along with mechanical and physical characterisation. For a given compressive strength, the optimised values of nanoSiO₂, nanoAl₂O₃ and nanoZnO were 5%, 2% and 0.5%, respectively. Test results clearly showed an improvement in the microstructure when adding all three types of nanoparticle. NanoAl₂O₃ showed superior behaviour as regards porosity, while nanoSiO₂ gave the lowest values with regard to water absorption. Si/Al trends for each nanoparticle differed with respect to their effect on compressive strength. An analytical model to predict the compressive strength of the nano-added geopolymers in terms of density was defined. NanoZnO geopolymer showed the highest cost reduction, making it the most economical option for achieving the required strength.

- 213319
EVALUATION OF THE HYDRATION HEAT AND STRENGTH PROGRESS OF CEMENT-FLY ASH BINARY COMPOSITE
 Wang X-Y - Kangwon, National University
J.Ceram.Process.Res. 21, No.6, 2020, p.622-631
 A mathematical model, which considered the initial dormant, boundary reaction and diffusion stages, was developed for the hydration of the cement and the reaction of fly ash with calcium hydroxide in binary cement-fly ash mixtures. The model were verified by measuring the isothermal hydration heat of mixtures containing 0-40% fly ash, at temperatures of 20-50 C. A further linear model was developed for the compressive strength evolution of the mixtures, based on the hydration of the cement and the pozzolanic properties of the fly ash. The coefficients for the strength model were established experimentally. It was concluded that the model is applicable to ordinary strength, high strength and ultrahigh strength concretes with different fly ash contents and water:binder ratios.
- 213320
OPTIMISATION OF RICE HUSK ASH (RHA) AS PARTIAL REPLACEMENT OF CEMENTING MATERIAL IN STRUCTURAL CERAMIC COMPOSITE CONCRETE USING RESPONSE SURFACE METHODOLOGY (RSM) STATISTICAL EXPERIMENTAL DESIGN
 Fazli A A M; Zakaria S K; Abd Rahman N I N; Salleh S Z; Yusoff A H; Salleh N A; Taib M A A; Budiman F; Ali A; Teo P T - Kelantan, Universiti Malaysia; Sintok, Universiti Utara Malaysia; Taiping, Advanced Technology Training Center; Bandung, Telkom University
J.Ceram.Process.Res. 21, No.6, 2020, p.667-682
 A series of concrete mixes was prepared with a cement:sand:aggregate ratio of 1:1.5:3, in which 10-30% of the Portland cement was replaced by black or grey rice husk ash. Cast specimens were cured in water and evaluated after 3-7 d by determinations of phase composition, density, porosity, water absorption, and compressive strength. Analysis of variance was used to assess the results. The replacement of 20% of cement with black rice husk ash followed by 7 d curing gave the highest compressive strength. The grey ash contained a higher amorphous silica content than the black, making it more reactive and leading to higher compressive strength concretes.
- 213321
STUDY ON THE PROPERTIES OF MAGNESIUM OXYCHLORIDE CEMENT PREPARED BY THE MAGNESIUM-RICH BYPRODUCTS FROM THE PRODUCTION OF LITHIUM CARBONATE FROM SALT LAKES
 Miao M; Wu C; Zhang H - Qinghai, University; Chinese Academy of Sciences
J.Ceram.Soc.Jap. 129, No.8, 2021, p.516-527
 The residue remaining following the extraction of lithium carbonate from salt lake brine consisted mainly of magnesium hydroxide, magnesium borate and a small amount of magnesium oxide. Active MgO was prepared by calcining the residue at temperatures in the range 400-900 C. Magnesium oxychloride cement pastes were prepared from mixtures of active MgO, magnesium chloride, potassium dihydrogen phosphate, fly ash, fumed silica and magnesium aluminosilicate slag. The MgO:MgCl₂:H₂O molar ratio was 5:1:13 or 8:1:13. The KH₂PO₄ addition was 1% of the mass of the MgO. Cement paste compositions were prepared in which 0-30% of the active MgO was replaced by fly ash or magnesium aluminosilicate slag, and also in which 0-15% was replaced by fumed silica. Cast specimens were cured at 20 C, 50% relative humidity and characterised by microstructural studies, and by measurements of setting and hardening times, heat of hydration, pore size distribution, water resistance, and compressive strength after subsequent curing for 1-28 d. The fastest hydration rate was achieved using residue calcined at 500 C. The highest strength was achieved using residue calcined at 600 C and a MgO:MgCl₂:H₂O molar ratio of 8:1:13. It is proposed that the additions of fly ash, fumed silica and magnesium aluminosilicate slag can reduce the cement cost, but only provide short-term improvement in water resistance.
- 213322
STUDY ON PROPERTIES OF MAGNESIUM PHOSPHOSILICATE CEMENT USING DIFFERENT REACTIVE MAGNESIA
 Dai Y; Wu C - Qinghai, University; Qinghai, Key Lab. Of Energy-Saving Building Materials & Engineering Safety
J.Ceram.Soc.Jap. 129, No.8, 2021, p.540-550
 Mixtures of light-burned magnesia and borax were calcined to temperatures in the range 1000-1200 C. The calcined material was mixed with potassium phosphate, fumed silica and water to obtain magnesium phosphosilicate cement pastes with a magnesia:potassium phosphate ratio of 2, a fumed silica content of 40% of the MgO content, and containing 0% or 5% borax. Cast specimens were cured at 20 C, 50% relative humidity and evaluated by microstructural studies, and by determinations of setting time, hydration heat release rate, chemical composition, crystalline phase composition, porosity, pore size distribution, and compressive strength after curing for 1-28 d. The reactivity of the magnesia decreased with increasing calcination temperature, attributed to a reduction in surface area and an increase in particle size. The addition of 5% borax gave a further reduction in magnesia reactivity. The use of magnesia of high reactivity gave good early strength, but the curing reaction was too fast, such that the strength decreased with time. It is proposed that the preparation of magnesium phosphosilicate cement with a suitable setting time and with high early strength requires the use of magnesia with an appropriate reactivity. The cured porosity decreased with increasing magnesia reactivity, attributed to a smaller crystal size, and to reaction with silica, so forming void-filling gels.
- 213323
DECONSTRUCTING WATER SORPTION ISOTHERMS IN CEMENT PASTES BY LATTICE DENSITY FUNCTIONAL THEORY SIMULATIONS
 Zhang Y; Liu H; Zhao C; Ju J W; Bauchy M - Shijiazhuang, Tiedao University; California, University at Los Angeles; Wuhan, University
J.Am.Ceram.Soc. 104, No.8, 2021, p.4226-4238
 The moisture content in cement pastes affects their mechanical properties and durability, but their complex, multiscale nature makes it difficult to isolate the contributions of each scale to their macroscopic water sorption isotherms. In particular, the contribution of the calcium-silicate-hydrate gel (the binding phase of cement pastes) is still only partially understood. A density functional theory lattice model describing water sorption in calcium-silicate-hydrate, which reproduces experimental water sorption isotherms in cement pastes, was developed. Based on this model, the contribution of each pore scale (interlayer spacing, gel pores and capillary pores) to the total sorption isotherm was deconstructed. It was shown that, when the relative humidity was < 80%, the calcium-silicate-hydrate gel accounted for > 90% of the moisture content adsorbed in the cement pastes. It was also found that the contribution of the interlayer space within the calcium-silicate-hydrate grains is governed by the competition between the rate of interlayer space opening and the increasing propensity for water to fill larger pores upon increasing relative humidity. The results show the key role played by the calcium-silicate-hydrate in governing the sorption isotherms of cement pastes.

GLASS

Glass

See also Abstract(s): 213313 213372 213385 213406 213410 213411 213413 213443 213459 213460 213641 213655 213685 213686 213688 213324

ARTIFICIAL INTELLIGENCE DENSITY MODEL FOR OXIDE GLASSES

Kareem-Ahmmad S; Jabeen N; Ahmed S T U; Ahmed S A; Rahman S - *Hyderabad, M.J. College of Engineering & Technology; Hyderabad, Osmania University*

Ceram.Int. 47, No. 6, 2021, p. 7946-7956

A comprehensive study to predict glass density using artificial intelligence and a dataset of 6630 oxide glass samples is reported. The prediction was carried out based on the ionic packing ratio as the independent variable and experimental densities from the dataset as the dependent variable. Random forest regression and artificial neural networks were observed as the best models training the density datasets. The random forest regression had the least average R2 score for the large datasets. Artificial neural networks, employing sigmoid and ReLU activation functions, dominated in predicting the glass density as compared to tan-h and identity activation functions. Based on this study the density of any oxide glass could be theoretically predicted to a maximum accuracy for a known glass composition.

213325

GADOLINIUM-BASED HYBRID ULTRA-LOW-BACKGROUND MATERIAL FOR PROTECTING THE DARKSIDE20K DARK MATTER DETECTOR FROM BACKGROUND NEUTRONS

Zykova M P; Khomyakov A V; Grishechkin M B; Nikulin I S; Chepurinov A S; Avetisov I Kh - *Moscow, Mendeleev University of Chemical Technology; Belgorod, State University; Marafon LLC.*

Glass Ceram. 78, No. 3/4, 2021, p. 91-96

A laboratory approach for producing an ultra-low-background hybrid material based on organic glass- polymethyl methacrylate- was devised (PMMA). In the hybrid material, 157Gd nuclei are employed as an effective thermal neutron absorber. By adding gadolinium in the form of a coordination molecule- gadolinium acetylacetonate- into the hybrid material, a uniform distribution of gadolinium in the PMMA matrix was obtained. In a 5 cm thick matrix of hybrid material with a gadolinium mass concentration ranging from 1.0 to 1.5%, a homogeneous gadolinium distribution was obtained. It was demonstrated that the mechanical properties of the hybrid material samples correspond with those of nominally pure PMMA at 298 K, but that at 77 K the hybrid material is inferior to pure PMMA within permissible limits.

213326

EFFECT OF THE ADDITIVES B2O3 AND P2O5 ON SILICATE AND GLASS-FORMATION PROCESSES IN MAKING STRONTIUM ALUMINOSILICATE GLASSES

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Glass Ceram. 78, No. 3/4, 2021, p. 97-103

The findings of experiments on decreasing the melting temperature of strontium aluminosilicate glass by including boron and phosphorus oxides into the batch have been published. The order and temperature intervals of the key physicochemical events happening in the batches on and heating were determined.

213327

DIAGNOSTICS OF THE WORKING ZONE OF GRADIENT STABILITY IN A MAGNETOMETER WITH PARTICLE SUSCEPTIBILITY CONTROL (FOR MAGNETIC SEPARATION PROBLEMS)

Sandulyak A A; Polismakova M N; Sandulyak D A; Sandulyak A V; Ershova V A - *Moscow, Technological University; Moscow, Polytechnic University*

Glass Ceram. 78, No. 3/4, 2021, p. 104-110

The ponderomotive technique of getting information on the magnetic susceptibility of ferroparticles in various media, including glass and ceramic manufacturing, is investigated. The corresponding magnetometer with spherical pole pieces is characterised, allowing for the creation and identification of the required (for locating the studied samples) stability zone of the induction gradient in the vicinity of the extremum (characteristic for such poles) in the gradient's coordinate dependence. There are examples of identifying the locations and sizes of such zones, especially with up to 3% deviation from the average value of the gradient.

213328

HEAT-RESISTANCE ENHANCEMENT OF FIBREGLASS-REINFORCED PLASTICS IN MANUFACTURING ENVIRONMENTS

Blaznov A N; Samoilenko V V; Zimin D E; Komarova M V; Ananieva E S; Firsov V V; Sakoshev Z G - *Russian Academy of Sciences; Barnaul, Altai State Technical University*

Glass Ceram. 78, No. 3/4, 2021, p. 111-114

On a production line, three batches of fibreglass rods were wound experimentally. The optimum EDI binder composition was chosen, with component weight ratios of ED-22/IMTGFA/UP-606/2 = 100/85/(1.5 - 3), which raises the glass transition temperature of the products by 10 - 15 C when compared to the control samples. TMA and DSC verified the rise in the glass transition temperature in all three pilot batches made at various dates. Heat-treatment temperatures of 35 and 40 C are recommended for the new EDI composition in an impregnating bath to enhance viscosity, resulting in a 15% weight content of the binder in fibreglass that fulfils regulatory code criteria.

213329

STRESS STATE OF A GLASS COATING IN PIPES

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Glass Ceram. 78, No. 3/4, 2021, p. 125-128

The stress condition of a glass covering in a petrochemical industrial pipe is investigated. The longitudinal, radial, and tangential stresses occurring in the cross sections of a coating are calculated using formulas. The stress condition of a glass covering on a pipe was experimentally examined. The coating's tensile strength at high temperatures is given.

213330
SYNTHESIS AND PROPERTIES OF Nd-DOPED CHLOROFLUOROSILICATE LEAD GLASSES
 Butenkov D A; Runina K I; Petrova O B - *Moscow, Mendeleev Russian Chemical Technology University*
Glass Ceram. 78, No. 3/4, 2021, p. 135-139
 Glasses, including those activated by neodymium, were produced by studying the glass transition zone in the system $\text{PbCl}_2\text{-PbF}_2\text{-PbO-SiO}_2$. The glasses' mechanical, optical, and spectral-luminescence characteristics were studied. It was demonstrated that substituting chlorine for fluorine moves the peaks of the luminescence and absorption lines to longer wavelengths.

213331
SYNTHESIS AND SPECTRAL PROPERTIES OF GLASSES IN THE SYSTEM BISMUTH OXIDE-GERMANIUM OXIDE-CERIUM OXIDE
 Serkina K S; Savenko L M; Stepanova I V; Petrova O B - *Moscow, Mendeleev Russian Chemical Technology University*
Glass Ceram. 78, No. 3/4, 2021, p. 145-147
 Glasses of bismuth germanate doped with cerium oxide were created. The effect of cerium oxide addition on the spectrum characteristics of bismuth-germanate glasses was investigated. The concentration ranges of Bi_2O_3 and CeO_2 in which the spectral properties of these glasses may be changed were determined.

213332
ENERGY-SAVING TECHNOLOGY FOR IRISATION OF GLASS ARTICLES
 Bessmertnyi V S; Zdorenko N M; Puchka O V; Makarov A V; Bragina V S; Bondarenko M A; Muravitskaya A I - *Belgorod, State Technological University; Belgorod, University of Cooperation, Economics & Law; Staryi Oskol, Technological Institute*
Glass Ceram. 78, No. 3/4, 2021, p. 153-156
 A high-temperature plasma torch was used to produce an energy-saving technique for irisation of glass products. The influence of the plasma-forming gas's outflow on the intensity of heating of the glass was examined. A glassware irisation installation was created. The irisation of glass objects' optimum process parameters were found.

213333
EXPERIMENTAL AND NUMERICAL STUDY ON ABRASIVE WATER JET MACHINING OF LAMINATED GLASS
 Vedrtnam A; Gunwant D; Chaturvedi S; Sagar D; Pawar S J - *Bareilly, Invertis University; Allahabad, Motilal Nehru National Institute of Technology*
Glass Technol. 62, No. 2, 2021, p. 65-74
 The use of laminated glass (LG) in structural applications is growing. The traditional method for cutting LG involves cutting the glass and the interlayer separately, so the productivity is low. The current work reported the use of abrasive water jet machining (AWJM) for LG cutting for the first time in public literature. Statistical methods have determined the best process parameters of AWJM for LG cutting. Atomic Force Microscope (AFM) was used to check the boundaries of LG samples cut using conventional methods and AWJM. The three-point bending test highlighted the superiority of the LG samples cut by AWJM in terms of load-bearing capacity and fracture mode. The experimental results were explored using the finite element (FE) model formed by the explicit dynamic module of ANSYS. The finite element model predicted the stress and strain of the glass and polyvinyl butyral (PVB) interlayer due to the impact of a single SiC abrasive particle.

213334
THERMAL, OPTICAL AND MECHANICAL PROPERTIES OF NEW GLASS COMPOSITIONS CONTAINING FLY ASH
 de Lima C J; Veer F; Zhang H; Fiho F F d M; Copuroglu O; Nijse R - *American Glass Research Inc.; Delft, Technical University*
Glass Technol. 62, No. 3, 2021, p. 96-108
 The research of new components is essential to expand the possible applications of glass, from typical usages in architectural engineering (in the form of ingots or float glass), to more advanced technologies such as 3D printing glass or glass-to-metal connections. Since high melting temperature and brittleness are two important shortcomings of glass, this work aims to improve these two characteristics. Characterisation techniques, such as thermal analysis, nanoindentation, and ultraviolet/visible spectroscopy, were used to evaluate the characteristics of the sample. Compounds such as phosphorous pentoxide, aluminum oxide, and boron oxide were used to achieve performance changes by changing the glass composition. Then, the selection of different glass formers and modifiers helped to develop a composition with a lower melting temperature and glass transition temperature. The lowering of the melting temperature allowed energy savings in the manufacturing process. The structure of the glass was different from standard soda lime silicate glass and borosilicate glass, resulting in different mechanical properties. In addition, these new compositions included up to 35% fly ash in their formulations. The added value of these by-products reduces costs and gas emissions.

213335
INTENSE EMISSION AT 2.9 MICRON FROM $\text{Yb}^{3+}/\text{Ho}^{3+}$ CO-DOPED $\text{TeO}_2\text{-Ga}_2\text{O}_3\text{-ZnO}$ TELLURITE GLASSES
 Zhang Y; Li X; Wang W; Zhang X; Xu Y - *Wuhan, University of Technology; Guangzhou, South China University of Technology; Rennes I, University*
J. Am. Ceram. Soc. 104, No. 7, 2021, p. 2924-2931
 Mid-IR lasers have applications in IR countermeasures, sensing, environmental monitoring, biomedicine and military and civilian fields. An intense emission at 2.9 micron from $\text{Yb}^{3+}/\text{Ho}^{3+}$ co-doped $\text{TeO}_2\text{-Ga}_2\text{O}_3\text{-ZnO}$ (TGZ) glass was reported. 2 micron, 1.2 micron and visible emissions were also performed to understand the competitive luminescent mechanism. With increasing Yb^{3+} concentration, all the emissions of Ho^{3+} increased, whereas the emission of Yb^{3+} decreased due to the phonon-assisted energy transfer from Yb^{3+} to Ho^{3+} . The lifetimes of optimised 3 mol% Yb_2O_3 and 1 mol% Ho_2O_3 co-doped TGZ glass, which have the maximum emission intensity, were 548 micros and 1.7 ms at 2.9 and 2 micron, respectively. The Judd-Ofelt intensity parameters, absorption and emission cross sections were calculated to evaluate the mid-IR fluorescence properties of this new glass matrix material. The gain coefficients showed that the 2 and 2.9 micron laser gain could be realised by small pump energy, indicating that the glass is a promising medium for mid-IR optical fibre lasers.

213336

PRESSURE EFFECTS ON SHEAR DEFORMATION OF BOROSILICATE GLASSESLee K-H; Yang Y; Ding L; Ziebarth B; Davis M J; Mauro J C - *Pennsylvania, State University; Schott AG; Schott North America Inc.***J.Am.Ceram.Soc.** 104, No.7, 2021, p.3073-3086

The shear behaviour of two multicomponent borosilicate glasses, Borofloat33 (Boro33) and N-BK7, under different pressures was studied by molecular dynamics simulations. The addition of alkali ions lowered the yield stress and changed the pressure dependence of the shear modulus. Shear-induced densification was seen in both glasses. The decreases of the oxygen-centred bond angle and the coordination number change of B were found to be responsible for the density changes at low pressures, and the increase of 5-coordinated Si was the dominant mechanism for densification at high pressures. The average shear stresses experienced by Si and B decreased with pressure except the flow stress of Si at the end of shear deformation in N-BK7. The average shear stress of B was more sensitive to the applied pressures compared to Si, suggesting that B could relax mechanical stress more easily under pressurised-shear. Analysing the nonaffine displacement of atoms showed that N-BK7 had more localised plastic deformation compared to Boro33 at low pressures and the local rearrangements in both glasses became more homogeneous with increasing pressure. The mean squared nonaffine displacement curves showed that alkali ions had the highest mobility induced by shear compared to the network formers and B was more mobile than Si for both glasses. It was found that plastic deformation tended to occur around boron atoms for Boro33, whereas it occurred in the alkali-rich regions for N-BK7, indicating that these two glasses have different atomic-scale deformation mechanisms.

213337

PHOTOELASTIC CONFIRMATION OF SURFACE STRESS RELAXATION IN SILICA GLASSES: FIBRE BENDING AND ROD TORSIONHausmann B D; Aaldenberg E M; Tomozawa M - *Rensselaer Polytechnic Institute***J.Am.Ceram.Soc.** 104, No.7, 2021, p.3087-3096

Silica glass samples were heat treated under stress at low temperatures and their residual stress distributions in terms of retardance were observed by polarised light microscopy, confirming previously reported fast surface stress relaxation and giving more detailed characterisation. Retardance profiles of silica glass fibres heat-treated under a constant bending strain in the presence of atmospheric water vapour were measured and fitted to a previously developed diffusion-based relaxation model. The retardance of a cross-section of a silica glass rod heat-treated at 650 C in air under applied torsional shear strain was also measured to confirm the presence of residual surface shear stress, predicted by the decrease of torque with time for the rod. The results confirm the low-temperature fast surface stress relaxation which occurs when water vapour is present for both bending and shear stresses.

213338

EFFECT OF ANIONIC SUBSTITUTION ON THE HIGH TEMPERATURE STABILITY OF POLYMER-DERIVED SiOC GLASSESSoraru G D; Girardini K; Narisawa M; Biesuz M - *Trento, University; Osaka, Prefecture University***J.Am.Ceram.Soc.** 104, No.7, 2021, p.3097-3104

The high-temperature stability (crystallisation and decomposition) of two silicon oxycarbide glasses with a similar amount of free carbon (8.3 and 9.6 wt%) but different content of Si-C bonds ($\text{SiC}_{0.22}\text{O}_{1.57}$ and $\text{SiC}_{0.07}\text{O}_{1.86}$) was studied. The two SiOC glasses were obtained from the same precursor (2 micron methyl-silsesquioxane spheres) via pyrolysis at 1100 C in inert (Ar) or reactive (CO_2) atmospheres. Further annealing in Ar flow at > 1100 C and up to 1500 C was carried out and the samples were characterised by FTIR and XRD. For comparison, the same precursor was annealed in air flow to obtain SiO_2 and its high-temperature evolution was also studied. Results suggested that the onset for carbothermal reduction did not depend on the amount of Si-C bonds. Contrary to usual reports in the scientific literature, silica phase present in the SiOC glasses did not show, under the same experimental conditions, superior crystallisation resistance compared to pure silica glass.

213339

EFFECTS OF THERMAL ENVIRONMENT ON DEHYDROXYLATION OF POROUS SILICA PREFORM IN TWO-STEP CVD SYNTHESISMa Q; Li C; Shang C; Zheng L; Fang H - *Huazhong, University of Science & Technology; Hubei Feilihua Quartz Glass Co.Ltd.; Beijing, Tsinghua University***J.Am.Ceram.Soc.** 104, No.7, 2021, p.3105-3118

Two-step CVD is a promising method for the industrial fabrication of low-hydroxyl silica glass ingots, in which a porous silica preform is synthesised by flame hydrolysis deposition, then sintered and vitrified to form silica glass. During sintering, hydroxyls can be easily removed through the small pores in the porous silica preform (dehydroxylation). In order to control dehydroxylation, understanding of the heat and mass transport characteristics involved is needed. Previously, a numerical model was developed to simulate the sintering and dehydroxylation of porous silica preform considering heat and mass transfer in porous media at high temperatures, chemical reaction of dehydroxylation and volume shrinkage of preform, and it was shown that dehydroxylation largely depends on the temperature fields in the furnace. Here, the effects of thermal environments, including heating curve, heater position and crucible structure, on the dehydroxylation process of porous silica preforms were studied by numerical methods. Based on the results, increasing temperature at either stage was found to be helpful for hydroxyl removal as well as increasing the hydroxyl distribution uniformity. However, long-time exposure to 1500 C will worsen the dehydroxylation effect. Compared to top or bottom heating, side heating is more beneficial to hydroxyl removal. To improve the dehydroxylation condition at the bottom of porous silica preforms, a novel crucible structure was designed which showed better performance than the ordinary crucible. An optimised heating scheme is proposed, which obtains much better dehydroxylation effect compared to other cases.

213340

EVAPORATION BEHAVIOUR OF SiO₂ GLASS DOPED WITH VARIOUS TRANSITION METAL OXIDESCheng C; Xie W; Li H; Fu Q - *Xian, Northwestern Polytechnical University***J.Am.Ceram.Soc.** 104, No.7, 2021, p.3130-3138

Nine transition metal oxides (TiO_2 , ZrO_2 , HfO_2 , V_2O_5 , MoO_3 , WO_3 , Nb_2O_5 , Ta_2O_5 and Cr_2O_3) were added to SiO_2 glass to determine their effect on evaporation at 1700 C. It was shown that V_2O_5 , MoO_3 and WO_3 had little effect on the high-temperature stability of SiO_2 glass due to their evaporation at 1500-1700 C. Nb_2O_5 and Ta_2O_5 reduced the evaporation of SiO_2 by covering the surface of the sample. By improving the cristobalite content, the evaporation of the SiO_2 - Cr_2O_3 sample decreased. TiO_2 , ZrO_2 and HfO_2 were beneficial to decreasing the evaporation of SiO_2 glass because Ti, Zr and Hf diffuse into c- SiO_2 . The SiO_2 - HfO_2 sample had the least evaporation.

213341

LUMINESCENT PROPERTIES OF DOPED AMORPHOUS AND POLYCRYSTALLINE Y₃Al₅O₁₂-Al₂O₃Zheng G; Wu J; Xu Z; Fang Z; Liu X; Qiu J - *Zhejiang, University; Taiyuan, University of Technology; Zhejiang, University of Technology; Jinan, University; Shanghai, Center for Excellence in Ultra-intense Laser Science***J. Am. Ceram. Soc.** 104, No. 7, 2021, p. 3139-3148

Lab-scale transparent polycrystalline ceramics (TPCs) based on the typical refractory Y₂O₃-Al₂O₃ system were produced through full congruent crystallisation of the parent glass prepared by aerodynamic levitation melting. Doping of the glass and TPCs by rare earth ions (Ce³⁺, Tb³⁺, Nd³⁺ and Yb³⁺) and transition metal ions (Cr³⁺) resulted in strong visible and near-IR photoluminescence with high quantum yield. The dominance of Stark splitting of the emission band for rare earth and transition metal ions in the TPCs compared with that of the glass confirmed crystallisation of the parent glasses. The TPCs have potential applications in solid state lighting, laser and photonics.

213342

MOSSBAUER SPECTROSCOPY, XRPD, AND SEM STUDY OF IRON-CONTAINING Na₂O-B₂O₃-SiO₂ GLASSESKonon M; Polyakova I; Stolyar S; Simonenko N; Simonenko T; Zolotov N; Semenova E; Antropova T - *St. Petersburg, Grebenshchikov Institute of Silicate Chemistry; Moscow, Inst. of General & Inorganic Chemistry; St. Petersburg, Institute of Precambrian Geology & Geochronology***J. Am. Ceram. Soc.** 104, No. 7, 2021, p. 3149-3157

Glasses in the Na₂O-B₂O₃-SiO₂-Fe₂O₃ system are suitable for producing magnetic porous glasses which are host matrices for multiferroic composite materials for spintronics applications. Successful synthesis of such materials depends on knowledge of crystallisation and immiscibility areas in the system. New findings are reported for such glasses with a constant SiO₂ concentration of 70 mol%, containing 7-12 mol% Fe₂O₃, with compositions in the low (2 mol% Na₂O) and higher (8-12 mol% Na₂O) alkali regions, heat-treated at 550 C. Glasses were studied using analytical chemistry and investigated by Mossbauer spectroscopy, XRPD and SEM. The immiscibility area boundary in the chosen silica cross-section for 550 C was outlined, indicating the region of interconnected morphology and the crystallisation fields of magnetite and FeSiO₃, and correlations made with data on the valence and coordination state of iron in glasses. It was found that both Fe³⁺ and Fe²⁺ ions in the low-alkali region were octahedrally coordinated, and in higher alkali area were tetrahedrally and octahedrally coordinated. A significant amount of Fe²⁺ in the low-alkali region could be the cause of the FeSiO₃ formation. The usual crystalline phase in non-X-ray amorphous glasses is magnetite, precipitating at the annealing stage.

213343

SURFACE DAMAGE RESISTANCE AND YIELDING OF CHEMICALLY STRENGTHENED SILICATE GLASSES: FROM NORMAL INDENTATION TO SCRATCH LOADINGSani G; Limbach R; Dellith J; Sokmen I; Wondraczek L - *Jena, Friedrich-Schiller-Universität; Jena, Institute of Photonic Technology; Sisecam Science & Technology Center***J. Am. Ceram. Soc.** 104, No. 7, 2021, p. 3167-3186

The surface elasticity, plastic deformation and crack initiation of chemically strengthened soda-lime silicate and sodium aluminosilicate glasses during lateral indentation and scratch testing were studied. Instrumented indentation using a normal indenter set-up corroborated previous findings on the effects of chemical strengthening on surface Young's modulus, hardness and indentation cracking. Using lateral indentation in the elastic-plastic regime, the scratch hardness was found to increase considerably as a result of chemical strengthening, as seen in the higher work of deformation required to create the scratch groove. The glass composition was therefore more important than the absolute magnitude of surface compressive stress. Using a blunt conical stylus for instrumented scratch testing revealed three distinct modes of scratch-induced surface fracture, which occurred during scratching or after unloading. Occasional micro-cracking caused by pre-existing surface flaws at low scratching load could be completely suppressed through chemical strengthening. The intrinsic defect resistance to microcracking was reduced as a result of ion stuffing, depending on the initial glass composition, whereas the resistance to abrasive yielding was enhanced by several hundred MPa.

213344

THERMAL STRENGTHENING OF LOW-EXPANSION GLASSES AND THIN-WALLED GLASS PRODUCTS BY ULTRA-FAST HEAT EXTRACTIONSajzew R; Wondraczek L - *Jena, Friedrich-Schiller-Universität***J. Am. Ceram. Soc.** 104, No. 7, 2021, p. 3187-3197

Thermal strengthening is the primary method to enhance the practical strength of commodity glass products, but the process is limited in terms of applicable glass thickness and coefficient of thermal expansion. The main reasons for the limitation are the achievable heat transfer coefficient when using conventional gas cooling and the occurrence of transient surface tension in the early stages of rapid quenching. Thermal strengthening of thin borosilicate glass sheet was studied. Using liquid gallium as the cooling medium, ultra-fast heat extraction was achieved, with a heat transfer coefficient > 5000 W/m.K. The low vapour pressure of gallium, even at high temperatures, enabled preheating to a wide range of sheet entrant temperatures. Thermal strengthening of low-expansion borosilicate glass with persistent surface compression of up to 85 MPa and quenching to a fictive temperature of about 190 K above the glass transition temperature was demonstrated. Glass sheet thus obtained had clearly enhanced surface defect resistance to sharp indentation. In addition to thermal strengthening, the extraordinarily high heat extraction rates achieved by liquid metal immersion enabled exploitation of high-T_f glass properties beyond small and thin sample geometry.

213345

AB INITIO MOLECULAR DYNAMICS SIMULATION OF THE STRUCTURAL AND ELECTRONIC PROPERTIES OF ALUMINOBOROSILICATE GLASSGong H; Song B; Yang Y; Wang P; Cao Z; Chen X; Zhao G; Peng S; Liu Y; Han G - *Zhejiang, University; Bengbu, Glass Design & Research Institute***J. Am. Ceram. Soc.** 104, No. 7, 2021, p. 3198-3211

The structural and electronic properties of aluminoborosilicate glass were examined by density functional theory-based ab initio molecular dynamic simulations. Computing models containing 220 atoms correctly described the local structure of the glass. The consistency between the experimental results, obtained using high-energy XRD and solid-state NMR, and the simulation results pertaining to structural factors, pair distribution functions, Qn distribution and elastic properties verified the reliability of the models. The presence of B and Al increased the flexibility and asymmetry of the system, as shown by the bond angle and ring size distributions. Based on the electronic properties, it was seen that the introduction of Al and B atoms into the network could also cause covalent interactions with the O atoms, similar to that with Si atoms. However, the Na and Mg atoms still interacted with all types of atoms in the network via charge transfer and showed highly non-localised effects on the charge of the network formers. The results help to understand the structure of aluminoborosilicate glass and can help to improve and design new types of this glass.

213346

EFFECT OF STRUCTURAL RELAXATION ON CRYSTAL NUCLEATION IN A SODA-LIME-SILICA GLASSRodrigues L R; Abyzov A S; Fokin V M; Zanotto E D - *Sao Carlos, Federal University; Kharkov Institute of Physics & Technology***J.Am.Ceram.Soc.** 104, No.7, 2021, p.3212-3223

The effect of structural relaxation on crystal nucleation is little researched. Its possible effect on nucleation was studied using a stoichiometric soda-lime-silica ($2\text{Na}_2\text{O}\cdot\text{CaO}\cdot 3\text{SiO}_2$) glass as a model system. It was shown that the relaxation effect was powerful at low temperatures, close and below the glass transition, T_g , and led to a continuous increase in the nucleation rate. At any given temperature, the nucleation rate eventually reached its ultimate steady-state corresponding to the fully relaxed supercooled liquid. However, the time to reach the steady-state was two to three orders of magnitude longer than the average relaxation time estimated by the Maxwell relation (shear viscosity/shear modulus). The proposed nucleation mechanism and model, which took relaxation into account, and related experimental results also explain the alleged breakdown of classical nucleation theory at low temperatures reported for various glasses. It confirmed some recent papers that this apparent flaw is due to the nucleation treatment not being prolonged long enough to complete the relaxation process to achieve a steady state. The actual maximum nucleation temperature, T_{max} , was also significantly lower than previously reported values. A comparative analysis of the kinetic coefficient using viscosity versus growth velocity favoured the latter. The results for this soda-lime-silica glass extend and validate recent findings for lithium disilicate on the significant (but often neglected) effect of relaxation on crystal nucleation.

213347

INVESTIGATION OF THE ACOUSTO-OPTICAL PROPERTIES OF Ge-As-Te-(Se) CHALCOGENIDE GLASSES AT 10.6 MICRON WAVELENGTHCao Z; Dai S; Liu Z; Liu C; Ding S; Lin C - *Ningbo, University; Shanghai, Fudan University; Ningbo, Institute of Oceanography***J.Am.Ceram.Soc.** 104, No.7, 2021, p.3224-3234

The acousto-optic parameters of $\text{Ge}_{10}\text{As}_{90-x}\text{Te}_x$ ($x = 30, 40, 50, 60, 70 \text{ mol}\%$) and $\text{Ge}_{10}\text{As}_{20}\text{Te}_{70-y}\text{Se}_y$ ($y = 20, 30, 40, 50 \text{ mol}\%$) glasses, were studied to compare Te-based and Se-based chalcogenide glasses for acousto-optic performance and thermomechanical properties. In the $\text{Ge}_{10}\text{As}_{90-x}\text{Te}_x$ system, the acousto-optic figure of merit (M_2) increased with increasing Te content, and the maximum M_2 of $2279 \times 10 \exp(-18) \text{ s}^3/\text{g}$, 13 times that of commercial single-crystal Ge, was obtained in $\text{Ge}_{10}\text{As}_{20}\text{Te}_{70}$ at 10.6 micron. However, its thermal properties and elastic modulus decreased and the acoustic attenuation (α) at different ultrasonic frequencies increased accordingly. In the $\text{Ge}_{10}\text{As}_{20}\text{Te}_{70-y}\text{Se}_y$ system, the thermomechanical performance of the glasses improved with the introduction of Se, the overall α was lower than that of Te-based chalcogenide glasses and the minimum α was 5.29 dB/cm at 30 MHz ultrasonic frequency, although its M_2 was inferior to that of Te-based chalcogenide glasses. The difference in the α of these glasses was also smaller, at low compared to high ultrasonic frequencies. The practical application of chalcogenide glasses as promising materials with outstanding acousto-optic properties in low ultrasonic frequency acousto-optic devices was demonstrated.

213348

MODELLING THE NANOINDENTATION RESPONSE OF SILICATE GLASSES BY PERIDYNAMIC SIMULATIONSCao Y; Kazembeyki M; Tang L; Krishnan N M A; Smedskjaer M M; Hoover C G; Bauchy M - *California, University at Los Angeles; Arizona, State University; Indian Institute of Technology; Aalborg, University***J.Am.Ceram.Soc.** 104, No.7, 2021, p.3531-3544

Nanoindentation is widely used to determine the mechanical properties of glasses, but interpreting the response of glasses to nanoindentation can be challenging due to the complex nature of the stress field under the indenter tip and the lack of in-situ characterisation techniques. A numerical model was developed describing the nanoindentation of an archetypical soda-lime silicate window glass by peridynamic simulations. It was shown that, although it did not capture shear flow and permanent densification, peridynamics agreed well with experimental nanoindentation data and offered direct access to the stress field forming under the indenter tip.

213349

CHARACTERISATION OF IMMISCIBILITY IN CALCIUM BOROSILICATES USED FOR THE IMMOBILISATION OF Mo^{6+} UNDER Au-IRRADIATIONPatel K B; Schuller S; Facq S P; Farnan I - *Cambridge, University; Montpellier, University***J.Am.Ceram.Soc.** 104, No.7, 2021, p.3632-3651

Factors affecting primary and secondary phase separation in simplified calcium borosilicate glasses for nuclear waste applications were determined. Several glasses with varying $[\text{MoO}_3]$ and $[\text{B}_2\text{O}_3]$ were synthesised and exposed to Au-irradiation to examine compositional effects on the glass structure and domain size of separated phases induced by accumulated radiation damage resulting from alpha-decay over a timeframe of about 1000 years. The produced glasses fell within the immiscibility dome of $\text{CaO-SiO}_2\text{-B}_2\text{O}_3$ and showed a unique microstructure of embedded immiscibility with three identifiable amorphous phases according to electron microscopy, Raman spectroscopy and diffraction. The glasses were then bombarded with 7 MeV Au^{3+} ions to a dose of $3 \times 10 \exp(14) \text{ ions}/\text{cm}^2$ creating an estimated 1 dpa of damage. Several changes to the morphology, spatial distribution and size of secondary phases were seen, indicative of significant structural reorganisation and changes to the chemical composition of each phase. A general mechanism of coalescence to form larger particles was seen for $[\text{MoO}_3] < 2.5 \text{ mol}\%$, whereas segregation to form smaller more evenly distributed particles was seen for $[\text{B}_2\text{O}_3]$ up to 15 mol% and $[\text{MoO}_3]$ at least 2.5 mol%. These microscopic changes were concurrent to surface-bulk diffusion of Ca and/or Mo ions, where the direction of diffusion depended on $[\text{B}_2\text{O}_3]$ with a barrier identified at about 20 mol%, as well as cross-phase diffusion of the ions. These modifications occurred partly through the formation of distorted ring structures within the borosilicate network, which enabled the increased dissolution of isolated $(\text{MoO}_4)^{2-}$ units. Au-irradiation therefore could increase the solubility of molybdenum and alter the structure and composition of secondary phases with the extent of modification varying with $[\text{MoO}_3]$ and $[\text{B}_2\text{O}_3]/[\text{SiO}_2]$, although glasses notably remained heterogeneous. The collective results suggest that radiation and composition can both be used as design tools to modulate the domain size and distribution of separated phases in heterogeneous glasses.

213350

POLARONIC CONDUCTION AND SWITCHING PHENOMENA OF AMORPHOUS $\text{Li}_2\text{O-CuO-P}_2\text{O}_5$ Mirzayi M - *Miyaneh, Islamic Azad University***J.Aust.Ceram.Soc.** 57, No.3, 2021, p.849-855

The electrical conductivity of bulk phosphate glasses, containing Li_2O and CuO , prepared by melt quenching was studied. Static current-voltage curves at high fields for memory switching behaviour were examined. XRD of prepared films revealed their amorphous nature. The activation energy

of the samples lies in the range of 0.53-0.98 eV, depending on composition. The conductivity at room temperature showed a minimum at a certain ratio of Li₂O/CuO. The conduction mechanism and observed minimum were explained by small polaron hopping and interaction between polarons and ions. The I-V characteristics at a high electric field showed memory switching. The mean value of the threshold voltage was dependent on the composition, decreasing with increasing CuO content. Values of the threshold voltage and activation energy were obtained for the investigated compositions. The observed switching phenomenon was explained using a thermal model.

213351

PHYSICAL, STRUCTURAL, AND GAMMA RAY SHIELDING STUDIES ON NOVEL (35+x) PbO-5TeO₂-20Bi₂O₃-(20-x)MgO-20B₂O₃ GLASSES

Al-Yousef H A; Alotiby M; Kumar A; Alotaibi B M; Alsaif N A M; Sayyed M I; Mahmoud K A; Al-Hadeethi Y - *Riyadh, Princess Nora Bint Abdul Rahman University; Riyadh, King Abdulaziz City for Science & Technology; Patiala, Punjabi University; Amman, Isra University; Dammam, Imam Abdulrahman Bin Faisal University; Ekaterinburg, Ural Federal University; Cairo, Nuclear Materials Authority; Jeddah, King Abdul Aziz University*
J. Aust. Ceram. Soc. 57, No.3, 2021, p.971-981

A novel glass system with the composition (35+x)PbO-5TeO₂-20Bi₂O₃-(20-x)MgO-20B₂O₃ (x = 0, 5, 10, 15 or 20 mol%) was synthesised by melt quenching. Amorphous behaviour and the presence of various vibration and stretching modes was confirmed by XRD and FTIR, respectively. The radiation shielding parameters of the glasses were analysed using MCNP5 simulation and the effects of PbO on the MCNP5 parameters were investigated in detail. The mass attenuation coefficient (MAC) was simulated via the MCNP5 code; it was found that the MAC values all follow the same trend as data from the XCOM software program, implying that the two simulations show good agreement. The linear attenuation coefficient (LAC) was calculated for all the glasses. The sample with 55 mol% PbO showed the greatest LAC at any energy, e.g. 0.317 at 10 MeV (the lowest investigated energy). Other parameters such as transmission factor (TF), lead equivalent thickness (d(lead)) and half-value layer (HVL) were derived from the LAC values. The TF results showed that the glass systems became more effective as their thickness increased. The sample with 35 mol% PbO recorded the highest TF at all energies due to the low PbO content, e.g. 15.333% for 1 cm thickness and 6.122% for 1.5 cm thickness at 0.3 MeV. The radiation protection efficiency (RPE) was also determined, and it was found that glasses with the greater PbO content and least MgO content have the highest RPE. Based on the RPE values, glasses with the greater PbO provide the best radiation shielding.

213352

GLASS-BASED SEALANTS FOR SOLID OXIDE FUEL CELLS

China, University of Mining & Technology; South Carolina, University; Thapar, Institute of Engineering & Technology; Bhabha Atomic Research Centre; Lille, Unite de Catalyse et de Chimie du Solide

Am. Ceram. Soc. Bull. 100, No.7, 2021, p.22-23

The high operating temperatures of solid oxide fuel cells (SOFCs) places stringent durability requirements on materials used in them, such as sealants. Glass and glass-ceramics have properties which make them of great interest as SOFC sealants. Recently published papers on the topic are briefly summarised, including one on the potential of a feldspar-based silicate sealant, one on the use of alkaline earth modified borosilicate glasses and one on the effect of adding V₂O₅ to a barium aluminosilicate sealing glass. Full references to the papers are provided.

213353

ORIGAMI TECHNIQUES UNFOLD NEW OPPORTUNITIES FOR COMPLEX GLASS SHAPING

Zhejiang, University

Am. Ceram. Soc. Bull. 100, No.7, 2021, p.24-25

Researchers from Zhejiang University have studied the possibility of shaping glass using origami techniques. Origami consists of converting a planar sheet into 3D geometries. With the delicate molecular design of the precursor composite, it is possible to introduce mechanisms that make it deform in such a way that permits origami-shaping of transparent glass. The composite sheet was fabricated by curing a silica nanoparticle-filled liquid precursor, the sheet was cut into the desired shape and then folded using manual techniques similar to actual paper origami. Further pyrolysis and sintering removed the polymer binder and converted the 3D object into glass. While manual folding was carried out in the study, the process could be automated. Examples of shapes created are shown.

213354

Er³⁺/Yb³⁺ CO-DOPED OXYFLUORO TELLURITE GLASSES: ANALYSIS OF OPTICAL TEMPERATURE SENSING BASED ON UP-CONVERSION LUMINESCENCE

Dagupati R; Klement R; Galusek D - *Trencin, Alexander Dubcek University; Trencin, Joint Glass Center of the IIC SAS, TnUAD & FChPT STU*

Int. J. Appl. Glass Sci. 12, No.4, 2021, p.462-471

Excitation and luminescence spectroscopy were used to illustrate the influence of Yb³⁺ co-doping on the relaxation dynamics of excited states of Er³⁺ ion in oxyfluoro tellurite (TZYN) glasses. The up-conversion luminescence spectra were investigated as a function of temperature in the 298-633 K range, and the fluorescence intensity ratio (FIR) for green bands, owing to the 2H_{11/2} 4I_{15/2} and 4S_{3/2} 4I_{15/2} transitions of Er³⁺ ion, was calculated versus temperature. The FIR method was used to investigate the temperature sensing capability of TZYN:Er³⁺/Yb³⁺ glasses up to 633 K. The maximum absolute (Sa) and relative (Sr) sensitivity of TZYN:0.5Er³⁺/1.0 Yb³⁺ glass were 0.00246 K⁻¹ at 413 K and 0.72 % K⁻¹ at 323 K, respectively.

213355

STRUCTURE AND FLUORESCENCE PROPERTIES OF Dy-DOPED ALKALINE-EARTH BOROPHOSPHATE GLASSES

Griebenow K; Munoz F; Tagiara N S; Klement R; Prnova A; Wolfrum B; Kamitsos E I; Duran A; Galusek D - *Trencin, University; BARCELONA, CSIC; GREECE, NATIONAL HELLENIC RESEARCH FOUNDATION; Trencin, Joint Glass Center of the IIC SAS, TnUAD & FChPT STU*

Int. J. Appl. Glass Sci. 12, No.4, 2021, p.472-484

The melt-quenching process is used to create dysprosium-doped borophosphate glasses containing the divalent cations Mg²⁺, Ca²⁺, Sr²⁺, Ba²⁺, and Zn²⁺. Raman, infrared, and nuclear magnetic resonance (NMR) spectroscopies were used to study the structure of the glasses, and fluorescence characteristics are determined. The lifetime of Dy³⁺ emission rose with increasing ionic field intensity, reaching a maximum in glasses containing Mg²⁺ and Zn²⁺. This result can be explained by the greater M-O bonding, which induces a reorganisation of the borophosphate network and the formation of a local Dy³⁺ environment with poor symmetry. This is in agreement with the Raman spectra and the evolution of the molar

volume. In a second series of glasses, the impact of B/P variation on the emission characteristics of Dy³⁺ was studied, and the emission lifetime was shown to be maximal at 10 mol% B₂O₃.

213356

DENSIFICATION OF GLASS POWDER STUDIED BY MASTER SINTERING CURVE AND MASTER KINETIC CURVE METHODS

Talimian A; Wondraczek L; Galusek D - *Trencin, Alexander Dubcek University; Jena, University, Otto-Schott-Institut; Trencin, Joint Glass Center of the IIC SAS, TnUAD & FChPT STU*

Int.J.Appl.Glass Sci. 12, No.4, 2021, p.541-550

In mol %, the master sintering curve technique was used to examine the sintering and crystallisation behaviour of 15Na₂O.10CaO.75SiO₂ (SLS) and 17Na₂O.33CaO.50SiO₂ (NC2S3) glasses. The master sintering curve (MSC) technique was used to effectively assess sample densification; it yields activation energy $Q = 310-340$ kJ.mol⁻¹, which is in excellent agreement with the anticipated activation energy of the viscous flow. While the calculated activation energy allowed for the prediction of SLS glass sintering behaviour, the actual findings deviated from the expected data for NC2S3. The activation energy of NC2S3 crystallisation, as measured by building Master Kinetic Curves (MKC), was lower than the sintering activation energy, limiting MSC's capacity to forecast NC2S3 glass.

213357

NEW GLASS-BASED BINDERS FROM ENGINEERED MIXTURE OF INORGANIC WASTE

Hujova M; Monich P R; Kankova H; Lucas H; Xakalash B; Friedrich B; Kraxner J; Galusek D; Bernardo E - *Trencin, Alexander Dubcek University; Padua, University; Aachen, University; MINTEK; Trencin, Joint Glass Center of the IIC SAS, TnUAD & FChPT STU*

Int.J.Appl.Glass Sci. 12, No.4, 2021, p.570-580

The current article is concerned with the conversion of waste-derived glass into new binders for the construction industry using both "conventional melting" and "smelting" methods. In the first example, red mud was mixed with trash to create a reactive glass (CMG), which forms stable gels following activation in an alkaline aqueous solution. The second method involved subjecting red mud to a thermal treatment in a reductive environment, indicating the separation of molten iron alloy. According to its chemical composition (CaO and Al₂O₃-rich), the residual glassy slag gelled by simple contact with pure water, without the need of an alkaline activator, resulting in the formation of a new "glass cement."

213358

THERMODYNAMIC MODEL OF ZnO-Nb₂O₅-P₂O₅ GLASSES-PARAMETERISATION AND VALIDATION

Liska M; Machacek J; Chromcikova M; Holubova J; Cernosek Z - *Trencin, Joint Glass Center of the IIC SAS, TnUAD & FChPT STU; Prague, University of Chemistry & Technology; Pardubice, University*

Int.J.Appl.Glass Sci. 12, No.4, 2021, p.581-587

Shakmatkin and Vedishcheva's ZnO-Nb₂O₅-P₂O₅ thermodynamic model included ten system components based on experimental structural data: ZnO, Nb₂O₅, P₂O₅, ZnP₄O₁₁, Zn(PO₃)₂, Zn₂P₂O₇, Zn₃(PO₄)₂, NbO(PO₃)₃, (NbO)₄(P₂O₇)₃, and NbOPO₄. The FACT database was used to obtain the molar Gibbs energies of ZnO, Nb₂O₅, P₂O₅, and Zn₃(PO₄)₂. The unknown Gibbs energies of the remaining components were determined by minimising the sum of squares of discrepancies between the calculated and observed equilibrium molar quantities of system components for the sequence xNb₂O₅50ZnO(50-x)P₂O₅. The Multivariate Curve Resolution (MCR) and Malfait's study of Raman spectra of xNb₂O₅(50-x)ZnO50P₂O₅ glasses supported the concept. The MCR analysis for three components produced loadings that were in good agreement with the partial Raman spectra obtained by Malfait's decomposition. On the level of experimental error, both the MCR and the Malfait's decomposition reproduced the experimental Raman spectra quite well.

213359

TWO-STEP ION-EXCHANGED SODA LIME SILICATE GLASS: EFFECT OF SURFACE COMPRESSION ON SILVER ION RELEASE

Talimian A; Nowicka A; Kankova H; Sani G; Galuskova D; Wondraczek L; Galusek D - *Trencin, Alexander Dubcek University; Jena, University, Otto-Schott-Institut; Trencin, University*

Int.J.Appl.Glass Sci. 12, No.4, 2021, p.601-612

Although glass is used extensively in medical facilities, such as worktops and medical equipment interfaces, where chemical and mechanical endurance is critical, less emphasis has been devoted to producing long-lasting anti-microbial surfaces. Ion exchange was used to create silver-containing surfaces in soda-lime silicate float glass, and the effects of residual surface compression caused by ion exchange on the release of silver ions were studied. A sequential two-stage ion-exchange procedure in pure potassium nitrate, at 450 C for 4-24 h, and then in KNO₃+ 1 wt% AgNO₃, at 400 C for 10-30 min, was used to create silver-doped surfaces. Silver ions were discovered to penetrate 3-5 μm into the glass surface, causing only a little reduction in surface compression caused by potassium ions. According to Ag⁺ ion leaching experiments, the silver-rich layer offers the necessary Ag⁺ leaching for anti-microbial applications. Surface compression can influence silver release.

213360

RAMAN SPECTROSCOPIC STUDY OF CORRODED HISTORICAL GLASS

Hruska B; Nowicka A; Chromcikova M; Greiner-Wrona E; Smolik J; Soltezs V; Liska M - *Trencin, Alexander Dubcek University; Trencin, Joint Glass Center of the IIC SAS, TnUAD & FChPT STU; Bratislava, Institute of Inorganic Chemistry; Krakow, AGH University of Science & Technology; Pardubice, University; VUEZ*

Int.J.Appl.Glass Sci. 12, No.4, 2021, p.613-620

Micro-Raman spectroscopy was used to examine the deteriorated surfaces of historical glass pieces. The spectra obtained from lateral and stratified mapping were analysed using principal component analysis, which provided the number of linearly independent spectral components, and multivariate curve analysis, which produced a spectrum of native glass as well as spectra of various corrosion products (loadings) and their relative abundances (scores) in each measured spot. The acquired findings were qualified by comparing stratified mapping results to lateral mapping results. The lateral distribution of various corrosion products was determined using dominating scores. The spectral database only provided an approximate estimate of the quality of corrosion products (S.T. Japan spectral database).

213361

EFFECTS OF COMPOSITION AND PHASE RELATIONS ON MECHANICAL PROPERTIES AND CRYSTALLISATION OF SILICATE GLASSESKilinc E; Bell A M T; Bingham P A; Hand R J - *Sheffield, Hallam University; Sheffield, University; LAV Gurallar***J. Am. Ceram. Soc.** 104, No. 8, 2021, p. 3921-3946

Crystallisation, mechanical properties and workability are important for the commercialisation and optimisation of silicate glass compositions, but there has been little research on the inter-relations of these properties as a function of glass composition. Soda-lime-silica glasses with Na₂O-MgO-CaO-Al₂O₃-SiO₂ compositions relevant to commercial glass manufacture were experimentally studied and multiple liquidus temperature and viscosity models were used to complement the experimental results. Liquidus temperatures of the fabricated glasses were measured by the temperature gradient technique, and Rietveld refinements were applied to XRD data for devitrified glasses, enabling quantitative determination of the crystalline and amorphous fractions and the nature of the crystals. Structural properties were studied by Raman spectroscopy. Acoustic echography, micro-Vickers indentation and SENB testing were used to measure Young's moduli, hardness and fracture toughness, respectively. It was shown that it is possible to design lower-melting soda-lime-silica glass compositions without compromising their mechanical and crystallisation properties. Unlike Young's modulus, brittleness is highly responsive to the composition in soda-lime-silica glasses, and low brittleness values can be obtained in glasses with compositions in the wollastonite primary phase field, an effect that is more pronounced in the silica primary phase field. The measured bulk crystal fractions of the glasses subjected to devitrification at the lowest possible industrial conditioning temperatures indicated that soda-lime-silica glass melts can be conditioned close to their liquidus temperatures within the compositional ranges of the primary phase fields of cristobalite, wollastonite or their combinations.

213362

ANALYTICAL MODEL OF THE NETWORK TOPOLOGY AND RIGIDITY OF CALCIUM ALUMINOSILICATE GLASSESYang K; Hu Y; Li Z; Krishnan N M A; Smedskjaer M M; Hoover C G; Mauro J C; Sant G; Bauchy M - *California, University at Los Angeles; Indian Institute of Technology; Aalborg, University; Arizona, State University; Pennsylvania, State University***J. Am. Ceram. Soc.** 104, No. 8, 2021, p. 3947-3962

Topological constraint theory (TCT) enables various properties of oxide glasses to be predicted as a function of their composition and structure, but its application relies on accurate knowledge of the network structure and topology. A fully analytical model describing the topology of the calcium aluminosilicate [(CaO)_x(Al₂O₃)_y(SiO₂)_{1-x-y}, CAS] ternary system was derived based on classical molecular dynamics simulations. This model gave the state of rigidity (flexible, isostatic or stressed-rigid) of CAS systems as a function of composition and temperature. The results showed the existence of correlations between network topology and glass-forming ability. The study suggested that the glass-forming ability is encoded in the network topology of the liquid state rather than that of the glassy state.

213363

OPTICAL PROPERTIES OF NOVEL OXYFLUORIDE GLASSES ON THE SYSTEMS OF LaF₃-LaO₃/2-NbO₅/2 AND LaF₃-LaO₃/2-NbO₅/2-AIO₃/2Chung J; Inoue H; Yoshimoto K; Masuno A; Watanabe Y - *Tokyo, University; Nikon Corp.; Hiroasaki, University***J. Am. Ceram. Soc.** 104, No. 8, 2021, p. 3963-3972

Oxyfluoride glasses of xLaF₃-(60-x)LaO₃/2-40NbO₅/2 (x = 0, 5, 10, 35) and xLaF₃-(60-x)LaO₃/2-30NbO₅/2-10AlO₃/2 (x = 0, 10, 20, 30) were prepared by levitation. The glass transition temperature, T_g, and onset crystallisation temperature, T_c, were both lowered by substituting part of the oxygen with fluorine in the glasses. An appropriate amount of fluorine maximised the difference between the temperatures, indicating the improvement in the glass-forming ability. The atomic packing densities of the glasses were approximately 60%, which gradually increased with the fluorine content. The absorption edge of the glasses shifted towards the shorter wavelength region in the UV spectra and towards the longer region in the IR spectra by fluorine substitution. In one of the oxyfluoride glasses, a wide transparency from 307 nm to 9.2 micron was also found. The glass had superior optical properties, with a combination of a high refractive index, n_d, of 2.020 and low wavelength dispersion, v_d, of 30.1. The effect of fluorine substitution on the n_d and its v_d was analysed using the Lorentz-Lorenz dispersion formula.

213364

CHEMICAL DURABILITY OF BOROSILICATE PHARMACEUTICAL GLASSES: MIXED ALKALINE EARTH EFFECT WITH VARYING [MgO]/[CaO] RATIOYang R; Liu H; Ding Z; Zheng J; Mauro J C; Kim S H; Zheng Q - *Jinan, Qilu University of Technology; Pennsylvania, State University***J. Am. Ceram. Soc.** 104, No. 8, 2021, p. 3973-3981

Glass for pharmaceutical packaging requires high chemical durability. The dependence of the chemical durability on alkaline earth oxide concentrations is not well understood in borosilicate pharmaceutical glasses which typically contain a mixture of different modifier ions (alkali or alkaline earth). A series of borosilicate glasses was designed with systematic substitutions of CaO with MgO while maintaining their total concentrations at 13 mol% and a fixed Na₂O concentration of 12.7 mol%. The glasses were used to investigate the effect of R = [MgO]/([MgO] + [CaO]) on the resistance to aqueous corrosion at 80 C for 40 days. It was found that this type of borosilicate glass underwent both leaching of modifier ions through an ion exchange process and etching of the glass network, leading to dissolution of the glass surface. Based on the concentration analysis of the Si and B species dissolved into the solution phase, the dissolved layer thickness was found to increase from about 100 to about 170 nm as R increased from 0 to 1. Depth profiling analysis of the glasses retrieved from the solution showed that the concentration of modifier ions (Na⁺, Ca²⁺ and Mg²⁺) at the interface between the solution and the corroded glass surface decreased to about 40-60% of the corresponding bulk concentrations, regardless of R and the leaching of modifier cations resulted in a silica-rich layer in the surface. The leaching of Ca²⁺ and Mg²⁺ ions occurred within about 50 and < 25 nm, respectively, from the glass surface and this thickness was not a strong function of R. The leaching of Na⁺ ions varied monotonically, the thickness of the Na⁺ depletion layer increasing from about 100 nm at R = 0 to about 200 nm at R = 1. Vibrational spectroscopy analysis suggested that the partial depletion of the ions may have caused some degree of the network re-arrangement or re-polymerisation in the corroded layer. Overall, the results suggested that for the borosilicate glass, replacing [CaO] with [MgO] deteriorated the chemical durability in aqueous solution.

213365

SEEDED STAGE III GLASS DISSOLUTION BEHAVIOUR OF A STATISTICALLY DESIGNED GLASS MATRIXCrum J V; Reiser J T; Parruzot B P; Neeway J J; Bonnett J F; Kerisit S N; Cooley S K; Ryan J V; Smith G L; Asmussen R M - *Pacific Northwest National Laboratory***J.Am.Ceram.Soc.** 104,No.8,2021,p.4145-4162

The dissolution rate of some glasses accelerates after a prolonged time spent at a slow, residual dissolution rate (Stage III behaviour). The acceleration in the glass dissolution rate linked to Stage III behaviour is significant and may be the most important effect on the long-term performance of glass in a repository for radioactive waste. An attempt to understand the effect of glass composition on Stage III behaviour was made to add a level of technical defensibility to glass disposal. A set of 24 glass compositions were statistically designed, in which eight glass components (SiO₂, B₂O₃, Al₂O₃, CaO, Na₂O, SnO₂, ZrO₂ and others) were independently varied to study the individual effects of each component. The glasses were subjected to static dissolution tests at 90 C in deionised water and then seeded with zeolite Na-P2 28 days into the testing to induce Stage III behaviour. The response of the glasses to the zeolite seeds fell into four primary types: (1) no response to seeds; (2) an immediate linear sustained acceleration in the rate; (3) an immediate linear acceleration in the rate followed by a decrease; and (4) a progressive acceleration in the rate that is concurrent with the addition of the seeds. The main glass components which affected these behaviours were CaO, Al₂O₃, B₂O₃ and ZrO₂, where CaO influenced which glasses showed a Stage III response to seeds (high CaO: types 2, 3 and 4) or did not respond to seeds (low CaO: type 1), (2) Al₂O₃ and B₂O₃ influenced which glasses showed a sustainable Stage III response (high Al₂O₃: types 2 and 4) versus transitory response (low Al₂O₃ and high B₂O₃: type 3) and (3) ZrO₂ concentration influenced whether glasses showed a linear (high ZrO₂: type 2) versus progressive (low ZrO₂: type 4) response to seeds.

Glass-ceramics

See also Abstract(s): 213300 213327 213352 213442 213452 213454 213588

213366

STRUCTURE FORMATION IN CAST SPINELIDE-PYROXENE GLASS-CERAMIC MATERIALS UNDER NON-EQUILIBRIUM CRYSTALLISATIONIgnatova A M; Vereshchagin V I - *Perm, National Polytechnic Research University; Tomsk, Polytechnical University***Glass Ceram.** 78,No.3/4,2021,p.115-119

Image analysis was used to determine how non-equilibrium solidification circumstances (contact with air or a mould, no contact) impact the macrostructural characteristics of glass-ceramic spinelide-pyroxene composites. It was discovered that structural zoning is determined by non-equilibrium circumstances. A mechanism for microzoning of structure is proposed: during the crystallisation process, a spinelide reaches a threshold size at which it obtains its own "zone of influence" - a specific volume of melt equal to the size of the spinelide zone creating an epitaxial shell around the spinelide.

213367

PHYSICAL, MECHANICAL, AND ELECTROPHYSICAL PROPERTIES OF POROUS GLASS COMPOSITE WITH SILICON CARBIDE ADDITIVESSemenova V I; Kazmina O V; Dorozhkin K V; Suslyayev V I; Sudarev E A; Mitina N A - *Tomsk, Polytechnical University; Tomsk, State University***Glass Ceram.** 78,No.3/4,2021,p.140-144

The impact of silicon carbide (10 - 40 wt%) on the physicomechanical and electrophysical properties of composites was investigated. The impact of an additive on the material's radio absorption capacity was measured by measuring the coefficients of absorption, transmission, and reflection of electromagnetic radiation, as well as the material's dielectric constant. Due to the reinforcing effect of silicon carbide needle crystals, it was discovered that including 30% wt. silicon carbide into the mixture improves the compression strength to 2.4 MPa. With the addition of 30% silicon carbide, the increased dielectric losses and average pore size of the composite result in an absorption coefficient of electromagnetic radiation ranging from 95 to 98 percent in the frequency range 120 - 250 GHz. The findings of this study broaden the applications of porous glass composites as well as the raw material foundation for producing the material.

213368

ULTRA-LOW TEMPERATURE PREPARATION OF MULLITE GLASS-CERAMICS WITH HIGH TRANSPARENCY SINTERED FROM EMT-TYPE ZEOLITEZhao Y; Zhou B; Qiu P; Fan Y; Zheng Q; Luo W; Cheng X; Wang L; Jiang W - *Shanghai, Donghua University; Shanghai, Fudan University***J.Am.Ceram.Soc.** 104,No.7,2021,p.3158-3166

Nanocrystalline mullite glass-ceramics are ideal optical window materials due to their excellent thermal shock resistance, low dielectric constant and good high-temperature strength. However, it is still difficult to fabricate high-purity mullite glass-ceramics at a low temperature. Highly transparent mullite glass-ceramics were here prepared at an ultra-low temperature (about 800 C) by the spark plasma sintering (SPS) of EMT-type zeolite. Unlike mullite glass-ceramics made by conventional sintering, these had high transparency both in the visible and IR regions. The sintering activity and linear thermal shrinkage behaviour of samples during SPS were investigated. With ultra-small mullite nanocrystals, the derived glass-ceramic sintered at 950 C had a high Vickers hardness (7.0 GPa), Young's modulus (86.6 GPa) and modified small punch strength (123.2 MPa), better mechanical properties than conventional aluminosilicate or silica glass.

213369

SINGLE-COMPONENT-AT-A-TIME VARIATION STUDY FOR GLASS-CERAMIC WASTE FORMSKissinger R M; Crum J V; Riley B J - *Pacific Northwest National Laboratory***J.Am.Ceram.Soc.** 104,No.7,2021,p.3738-3749

A 51-sample composition variation study was carried out on glass-ceramic waste forms for a raffinate waste stream from the aqueous reprocessing of used nuclear fuel containing high fractions of Mo, alkalis, alkaline earths (AEs) and rare earths (REs). The study was designed with a single-component-at-a-time (SCAT) variation approach off a centroid composition. The components which were varied included Al, B, Ca, Li, Mo, Na, REs, Si, Zr and others (containing minor components). Data analysis included crystallisation curves, microstructure and phase compositions. A number of components (i.e. Li₂O, B₂O₃, REO_x, MoO₃, Na₂O and ZrO₂) significantly affected the concentration and chemistry of the phases, especially the primary phases of oxyapatite (i.e. Ca₂RE₈(SiO₄)₆O₂) and powellite (i.e. AEMoO₄), precipitated in the slow-cool heat-treated waste forms. Minor phases included cerianite (i.e. CexZr(1-x)O₂), Ba-molybdate (i.e. Ba(Gd_{0.67}Mo_{0.33}O₃), noble metals, pollucite (i.e. CsAlSiO₄) and

RE-borosilicate (i.e. RE₃BSi₂O₁₀). The data can be used to help explain the compositional effects on the microstructure, chemistry and phase distribution of glass-ceramic waste forms and will aid future formulation of glass-ceramic waste forms.

213370

SIMPLE APPROACH TO DESIGN FLUORAPATITE GLASS-CERAMIC COATINGS ON THE SURFACE MODIFIED Ti6Al4V SUBSTRATES FOR BIOMEDICAL APPLICATIONS

Haghighat P J; Majidian H; Mobasherpour I; Banijamali S - *Iran, Materials & Energy Research Centre*

J.Aust.Ceram.Soc. 57, No.3, 2021, p.673-685

An attempt was made to prepare fluorapatite glass-ceramic coatings on Ti6Al4V substrates by dip-coating and subsequent heat treatment. The substrate surfaces were modified in order to adjust the quality and bioactivity of the coatings. For this purpose, rough and polished substrates, some of which were subjected to surface oxidation, were coated with a bioactive SiO₂-Al₂O₃-P₂O₅-K₂O-CaO-ZnO-Li₂O-F system glass layer; the coated specimens were then heat-treated at 750 C and 800 C. Heat-treated specimens were characterised by XRD, FESEM and microhardness measurements. Among the tested specimens, samples obtained after coating for three times on a sandblasted substrate were found to be optimal. Fluorapatite crystals with a needle-like morphology were seen in the glass-ceramic matrix. The bioactivity of the optimised glass-ceramic coating was monitored in-vitro by immersion in SBF for different time intervals (7, 14, 21 and 28 days). SEM and XRD confirmed the homogeneous distribution of hydroxyapatite crystals.

213371

EFFECT OF MnO ON THE CRYSTALLISATION, MICROSTRUCTURE, AND PROPERTIES OF MgO-Al₂O₃-SiO₂ GLASS-CERAMICS

Li B; Xia Q; Wang Z - *China, University of Electronic Science & Technology*

J.Aust.Ceram.Soc. 57, No.3, 2021, p.927-932

Mn-doped MgO-Al₂O₃-SiO₂ (MAS) glass-ceramics were prepared by melt quenching, and the effect of MnO on the crystallisation, microstructure and properties was studied. XRD showed that MnO promoted a phase transformation from indialite to Mg_{0.6}Al_{1.2}Si_{1.8}O₆ and enstatite. Crystallisation kinetics suggested that MnO inhibited the precipitation of indialite. Both the crystallisation index and SEM showed that bulk crystallisation played an important role in the Mn-doped MAS system. EDS analyses indicated that the large grains corresponded to Mg_{0.6}Al_{1.2}Si_{1.8}O₆ while the small grains consisted of a magnesium-rich phase. The flexural strength was enhanced from 146 to 247 MPa and the thermal expansion coefficient was increased from 3.52 x 10⁻⁶/C to 5.41 x 10⁻⁶/C with 0.5 wt% MnO addition.

213372

ASPECTS OF DEVELOPMENT AND PROPERTIES OF DENSELY SINTERED OF ULTRA-HIGH-FREQUENCY RADIO-TRANSPARENT CERAMICS OF CORDIERITE COMPOSITION

Zaichuk A; Amelina A; Kalishenko Y; Hordieiev Y; Saltykov D; Sribniak N; Ivchenko V; Savchenko L - *Ukrainian State University of Chemical Technology; Dnipropetrovsk, Oles Honchar Dnipro National University; Sumy, National Agrarian University*

J.Korean Ceram.Soc. 58, No.4, 2021, p.483-494

Part of the components in the composition of cordierite ceramics was introduced using relatively low-melting MgO-Al₂O₃-B₂O₃-SiO₂ (MABS) glass. After firing at 1375 C, densely sintered materials with low values of the linear coefficient of thermal expansion (17.7-18.5) x 10⁻⁷/deg were obtained, which predetermined their high thermal stability (not lower than 900 C). The only crystalline phase of the experimental ceramics was alpha-cordierite, which formed its structural matrix. Crystals of alpha-cordierite 1-2 micron in size were tightly interconnected by thin layers of the residual glass phase. Cordierite ceramics were characterised by zero values of water absorption and open porosity, and high values of mechanical compressive strength. The dense microstructure also allowed consistently high dielectric values (epsilon = 4.9, tan delta = 0.0014) to be achieved in an ultra-high-frequency electromagnetic field (10 exp(10) Hz). The synthesised ceramics can be used as radio-transparent materials, including structural ones.

213373

STRUCTURAL AND OPTICAL PROPERTIES IN Tm³⁺/Tm³⁺-Yb³⁺ DOPED NaLuF₄ GLASS-CERAMICS

Velazquez J J; Balda R; Fernandez J; Gorni G; Sedano M; Duran A; Galusek D; Pascual M J - *Trencin, Alexander Dubcek University; Bilbao, Superior School of Engineering, Basque Country University; San Sebastian, Donostia International Science Centre; Madrid, Ceramic and Glass Institute (ICV-CSIC); Trencin, Joint Glass Center of the IIC SAS, TnUAD & FChPT STU*

Int.J.Appl.Glass Sci. 12, No.4, 2021, p.485-496

Transparent NaLuF₄ glass-ceramics (GCs) doped with Tm³⁺ and Tm³⁺/Yb³⁺ were created by melt-quenching and thermal treatment at temperatures close to the glass transition temperature. X-ray diffraction (XRD) and high-resolution transmission electron microscopy were used to investigate the crystallisation process (HRTEM). The sole crystalline phase was NaLuF₄ nanocrystals (NCs) ranging in size from 9 to 30 nm, with crystal size rising with dopant concentration. Tm³⁺ and Yb³⁺ inclusion in the NCs is confirmed by energy dispersive X-ray (EDX) studies. The optical characterisation included an examination of up-conversion (UC) and near-infrared (NIR) luminescence. Tm³⁺ and Yb³⁺ NIR emission spectra in co-doped materials indicated an efficient energy transfer between both ions. Tm³⁺ single-doped glass and GCs showed no UC emissions. After stimulation at 975 nm, Yb³⁺ incorporation promoted the Tm³⁺-Tm³⁺ UC processes, resulting in Tm³⁺ blue, yellowish-red, and NIR UC emissions. After Tm³⁺ excitation at 791 nm, blue UC emission was also detected in the codoped samples. These effects were more pronounced in the GCs than in the base glasses, indicating the incorporation of RE ions in the NCs. As a consequence, by selective stimulation, these GCs may be utilised to adjust UC emission from NIR to blue.

213374

GLASS-CERAMIC Ce³⁺-DOPED YAG-Al₂O₃ COMPOSITES PREPARED BY SINTERING OF GLASS MICROSPHERES

Akusevich A; Parchovianska I; Parchoviansky M; Prnova A; Lofaj F; Vojtko M; Klement R - *Trencin, Alexander Dubcek University; Trencin, Joint Glass Center of the IIC SAS, TnUAD & FChPT STU; Kosice, Institute of Materials Research*

Int.J.Appl.Glass Sci. 12, No.4, 2021, p.497-508

To produce bulk glass/glass-ceramic luminous materials from quickly quenched glass microspheres in the system Y₂O₃-Al₂O₃ doped with Ce³⁺ ions, viscous flow sintering in a "kinetic window" between T_g and T_x temperatures was utilised. Glass microspheres were made in a high-temperature methane-oxygen flame using sol-gel synthesised precursor powders. Differential scanning calorimetry and high-temperature X-ray

diffraction were used to examine the crystallisation of the glasses. The crystallisation behaviour of quenched glass microspheres was discovered to be highly dependent on the composition of the glass. The glass microspheres were consolidated into bulk material using a hot-pressing method at temperatures up to 1100 C and pressures up to 40MPa. The optimised time-temperature-pressure regime allowed for the production of bulk glass and glass-ceramic with a high proportion of crystalline Ce³⁺-doped YAG phase embedded in an Al₂O₃-enriched glass matrix. Under UV and/or blue spectral excitation, the glass/glass-ceramics generate intense blue/yellow light.

213375

ALKALI-FREE PROCESSING OF ADVANCED OPEN-CELLED SINTER-CRYSTALLISED GLASS-CERAMICS

Elsayed H; Monich P R; Savio G; Hartmann M; Boccaccini A R; Galusek D; Kraxner J; Bernardo E - *Padua, University; Cairo, National Research Centre; Erlangen-Nurnberg, University; Trencin, Joint Glass Center of the IIC SAS, TnUAD & FChPT STU*

Int.J.Appl.Glass Sci. 12, No.4, 2021, p.531-540

The synthesis of an example of alkali-free bioactive glass, easily converted into glass-ceramics featuring two silicate phases, coupled also with kermanite (Ca₂MgSi₂O₇), was determined by the cooling of a melt corresponding to the eutectic between wollastonite (CaSiO₃) and diopside (CaMgSi₂O₆), by sinter-crystallisation of fine glass powders at 1000 C. The fabrication of scaffolds by digital light processing of glass powders suspended in a photo-curable, sacrificial binder is a well-established technique; the current paper aims to reveal novel approaches concerning scaffold topology, offering components with exceptional strength, particularly in bending conditions. Glass-ceramic foams were created as an alternative by burning porous precursors produced from the gelation of glass powder suspensions in alkali-free basic aqueous solution.

213376

Eu²⁺-DOPED OXYFLUORIDE GLASS-CERAMIC WITH NEPHELINE AS AN EFFICIENT 400 nm UV-LED COLOUR CONVERTER

Lee H; Chung W J - *Kongju, National University*

J.Am.Ceram.Soc. 104, No.8, 2021, p.4024-4032

SiO₂-Na₂O-Al₂O₃-LaF₃ glass-ceramics doped with Eu²⁺ were synthesised to give an efficient inorganic colour converter for 400 nm UV-LEDs. When Eu²⁺ formed within the glass matrix, the obtained glass showed cyan emission under 400 nm excitation, but its emission peak drastically shifted to greenish yellow upon heat treatment. With heat treatment the glass-ceramic also showed highly increased emission intensity, and the colour coordinate of the glass-ceramic shifted to yellow. When it was mounted on top of a 400 nm UV-LED, it showed high colour conversion efficiency and practical feasibility as a UV-LED colour converter. To vary the colour coordination, the heat-treatment conditions and the thickness of the glass-ceramic were adjusted. The resulting ceramic had a high quantum yield of up to 78%, comparable to conventional ceramic phosphors. The spectral change in the glass-ceramic was attributed to the formation of nepheline and LaF₃ crystalline phases. XRD, TEM-EDS and cathodoluminescence were used to investigate the mechanism of Eu²⁺-doped nepheline crystal formation and its effect on the spectral change with heat treatment.

DOMESTIC CERAMICS

213377

OPTIMISED ETCHING OF PORCELAIN AND POLYCRYSTALLINE ALUMINA WITH A GLASS PHASE

Whipkey S C; Modugno M C; Lee H; Carty W M - *New York, Alfred University*

J.Eur.Ceram.Soc. 41, No.6, 2021, p.3761-3768

Chemical and thermal etching techniques are commonly used in ceramography to enhance microstructural features. While thermal etching works well for high purity ceramic systems, this technique may not be appropriate where glass is present. Porcelain and industrial alumina both contain a glassy phase, typically 40-60 vol% and 4-30 vol%, respectively, and their glass chemistries are proposed to be similar. Chemical etching of porcelain is common, but the images published in the literature are frequently over-etched. When glass is present at the grain boundaries of alumina, thermal etching can cause the glass to disappear or to recrystallise, obscuring the microstructure. Because of this, it was proposed that both chemical and thermal etching are necessary to prepare an industrial alumina microstructure for grain size measurements. It was also observed that chemical etching is sensitive to the residual stress in the glassy phase, becoming more aggressive when there is residual tension in the glass.

Glazes, colours and decoration

213378

GLAZE RECIPE

Bloomfield L

Ceram.Rev. No.311, 2021, p.79-83

Celadon, a pale grey-green glaze, is coloured by iron oxide fired in reducing conditions. Celadons are traditionally fired in wood or gas kilns, but by adding a reducing agent such as silicon carbide, they can be fired in electric kilns. However, if too much SiC is used, the glaze can turn black. Several potters have been collaborating on the online database glazy.org to test such glazes and find the optimum composition. A number of these glaze compositions are given with examples of the colours achieved.

213379

DEVELOPMENT OF COMPOSITIONS OF PIGMENTED COLOURED LEAD-FREE GLAZES FOR IMPROVING CERAMIC PRODUCT DESIGN

Papulova G N; Kvasnikov M Yu - *Moscow, State Technological University "Stankin"; Moscow, Mendeleev Russian Chemical Technology University*

Glass Ceram. 78, No.3/4, 2021, p.129-132

The surface structure of several varieties of ceramic is determined, and their comparative properties are provided. The use of coloured glazes to decorate faience is investigated, and the formulation of a lead-free pigmented faience glaze is disclosed.

Tableware

- 213380
DRAMA OF DEMAND AND SUPPLY
 Goodfellow V - *G & G Goodfellows Ltd.*
Tableware Int. 143, No. 3, 2021, p. 34-35
 Factors affecting supply and demand in the tableware industry, specifically in the UK, are discussed. These factors include a surge in demand created by the opening up of hospitality venues, reduced workforce availability, supply chain problems due to raw material shortages and continued high retail demand. Many of the problems stem from Covid-19 and Brexit. The importance of good communication with customers when products are in short supply to maintain brand loyalty is emphasised.

Tiles

See also Abstract(s): 213455

- 213381
EFFECT OF (MgO/CaO) MOLAR RATIO ON GLASSY PHASE VISCOSITY AND PYROPLASTIC DEFORMATION IN FLOOR TILES
 Selli NT - *Gebze, Technical University*
J.Ceram.Process.Res. 21, No. 6, 2020, p. 632-639
 Starting with a conventional floor tile body, modified body compositions were prepared by adding different amounts of talc and calcite. The conventional body had a Na₂O:K₂O molar ratio of 0.905, and a MgO:CaO molar ratio of 4.990. The MgO:CaO molar ratios of the experimental bodies were in the range 4.450-6.505. Specimens were prepared by uniaxial pressing, and the sintering temperatures determined, defined as the temperature giving the maximum densification rate. Specimens were fired at 1160 C and characterised by microstructural studies, and by determinations of phase composition, firing shrinkage, water absorption, colour, flexural strength, and pyroplastic index. The viscosity of the glassy phase was calculated from the pyroplastic index measurements. The viscosity decreased with increasing MgO:CaO ratio, such that the body with a ratio of 6.505 had a sintering temperature approximately 20 C below that of the conventional body.

INTERMEDIATE PRODUCTS AND AUXILIARIES

See also Abstract(s): 213314 213317 213318 213375 213458 213479 213480 213499 213512 213513 213581 213618 213634 213635 213637
 213654 213671

- 213382
EFFECT OF SURFACTANTS ON THE PERFORMANCES OF CERAMIC SLURRY BY MATERIAL EXTRUSION AND PHOTO-POLYMERISATION COMBINED MOULDING PROCESS
 He X; Xu J; Ji W - *Jiangnan University; Beijing, Space Engineering University*
J.Ceram.Soc.Jap. 129, No. 8, 2021, p. 489-495
 Various surfactants were evaluated for the modification of alumina suspensions to be used for additive manufacturing by extrusion and photopolymerisation. Suspensions containing different concentrations of spherical alpha alumina were prepared with additions of 0.0-0.30% of two grades of polyvinyl pyrrolidone (PVP) or oleic acid surfactants, with 1,6-hexanediol diacrylate monomer and diphenyl(2,4,6-trimethylbenzoyl)-phosphate oxide photoinitiator. The suspensions were characterised by measurements of the rheological properties, and of stability by sedimentation testing. Formability was assessed by extruding fibres across a measured gap and measuring the slump. Following debinding, specimens were sintered at 1650 C. The suspension containing 0.15% oleic acid had better rheological properties and stability compared with those containing PVP. The deformation of the extrudate was <0.25%. A sintered density of 95.48% of theoretical was achieved.

- 213383
DISPERSANTS FOR SILICON CARBIDE IN WATER- A COMPARATIVE STUDY
 Foghmoes S; Jorgensen J H; Agersted K; Nielsen M; Negra M D - *Denmark, Technical University; LiqTech International A/S*
Int.J.Appl.Ceram.Technol. 18, No. 4, 2021, p. 1164-1173
 A comparative study was made on a pool of 12 dispersants for the deagglomeration and stabilisation of silicon carbide in aqueous suspensions with solids loadings relevant for dip coating applications. Since SiC slurries may include sintering aids, different functional groups, molecular weight, and stabilisation mechanisms were considered for the dispersants to be able to stabilise both slurry components. The pH effect, toxicity, additive compatibility and foaming properties were determined, giving all the necessary information for developing new aqueous formulations of SiC suspensions, including the advantages and disadvantages of the different candidates. Different deagglomeration procedures, powder surface area and calcination temperature were also considered in order to study the effect of SiC surface properties. Slurry stabilisation provided by an alkaline environment at a pH > 8-9 was significantly more effective than slurry stabilisation by any of the tested dispersants. Alkaline environments facilitate a negative surface charge on SiC particles and provide a stable electrostatic stabilisation mechanism not seen in neutral or acidic environments. One of the dispersant candidates (FA 4404) seems to broaden slightly in the range of stability toward the acidic regime. Anionic surfactants or block co-polymers tested showed no significant interaction with the SiC particles.

Binders

See also Abstract(s): 213357 213605 213623 213627

- 213384
EFFECT OF TEMPERATURE ON THE INITIAL PROPERTIES OF CALCIUM SULPHOALUMINATE BINDERS SYNTHESISED AT 1100 C
 Gallardo-Heredia M; Magallanes-Rivera R X; Almanza-Robles J M; Maldonado Y G; Martinez-Sanchez E - *Mexico, Universidad Autonoma de Coahuila; CINVESTAV; CENAM*
Adv.Appl.Ceram. 120, No. 4, 2021, p. 240-247
 The influence of curing temperature on the mechanical characteristics of pastes produced using calcium sulphoaluminate cements synthesised from industrial wastes, at a comparatively low temperature of 1100 C was investigated in this study. Raw materials included fluorgypsum, fly ash,

aluminium slag, and calcium carbonate. Calcium sulphoaluminate and gehlenite were the primary components of production. Pastes were made with various water/cement ratios, 15-25 wt% hemihydrate, and cured at 20 and 40 C. It was discovered that when the overall amount of sulphates increased and the temperature rose, compressive strength increased in the initial days of curing and setting periods reduced. Ettringite was discovered to be the primary reaction product that formed dense formations over time. The hydration of calcium sulphates altered the ettringite production, producing variations in strength development; nonetheless, in certain cases, the strength exceeded 40 MPa after 28 days. SEM and XRD were used to investigate the microstructures and hydration products.

Fibres and whiskers

See also Abstract(s): 213305 213335 213337 213392 213460 213583 213589 213602 213623 213693

213385

MATERIAL CONSTRAINT-BASED LASER PERFORMANCE ESTIMATION OF Yb³⁺-DOPED PHOSPHATE FIBRES

Yan S; Chen L; Du Y; Tao Y; Zhang L; Feng S; Zhou Q; Chen D; Zhang L - *Shanghai, Institute of Optics & Fine Mechanics; Beijing, University of Chinese Academy of Sciences*

J.Am.Ceram.Soc. 104, No.7, 2021, p.3289-3302

Ytterbium ion is an excellent optical gain material for high-power lasers due to its small quantum defect. High gain single-frequency lasers can be obtained from Yb³⁺-doped phosphate fibres with a short linear cavity. Based on the material properties of 47 Yb³⁺-doped phosphate fibres of different sizes and lengths, a laser performance estimation model was established to estimate the laser wavelength just above the threshold (λ_{th}), the laser threshold (P_{th}) and the laser slope efficiency (η). The model was solved using a numerical computing method with an accuracy of 0.01. Under the material constraints of this model, λ_{th} , P_{th} and η were studied by sensitivity analysis and Monte Carlo numerical simulation. It was shown that short fibres with a small core diameter could more easily produce a shorter λ_{th} . For constant material properties, fibres with a longer λ_{th} could have a lower P_{th} and higher η than other fibres. Verifying this, an output power of 8.7 W with 35% enhancement in slope efficiency was obtained from an optimised phosphate fibre for an optical path in which the output of the short-wave laser was inhibited. This model could be extended to simulate the lasing wavelength of multi-composition fibres, providing a theoretical basis for special laser bands, laser material preparation and fibre structure design.

213386

MICROSTRUCTURED MULTIMATERIAL FIBRES FOR EFFICIENT OPTICAL DETECTION

Dai Y; Du M; Feng X; Zhang W; Zhou S - *Guangzhou, South China University of Technology*

J.Am.Ceram.Soc. 104, No.8, 2021, p.4058-4064

Flexible optoelectronic fibres are important for systems of wearable electronics and advanced functional textiles. Thermal drawing gives the possibility of scalable construction of optoelectronic fibres, but they are generally characterised by a continuous cylindrical structure and poor electrical properties. Flexible structured multimaterial fibres thermally drawn from the combination metal (Cu)-semiconductor (Se)-polymer (PMMA) were fabricated and characterised, and their inner structure was controlled by fluid Plateau-Rayleigh instability. A trapezoidal electrical connection configuration with the semiconductor spheres clamped by two metal electrodes inside the fibre was successfully achieved based on this fluid instability. The obtained structured multimaterial fibre had better photoresponse compared to that of the as-drawn multimaterial fibre. The findings give new opportunities to develop fibre devices for efficient optical detection.

RELATED TO PARTICULAR INDUSTRIES AND FIELDS OF USE

See also Abstract(s): 213329 213598

Aerospace

See also Abstract(s): 213372 213417 213418 213420 213494 213602 213620 213647 213653

Iron and steelmaking

See also Abstract(s): 213598 213625 213627 213632 213636 213637 213640

Agriculture and horticulture

See also Abstract(s): 213301 213425

Military

See also Abstract(s): 213594 213670

Chemical (including filtration)

See also Abstract(s): 213438 213464 213491 213528 213530 213548 213669

213387

ZnO-SnO₂ NANOCUBES FOR FLUORESCENCE SENSING AND DYE DEGRADATION APPLICATIONS

Kumar R; Umar A; Kumar R; Chauhan M S; Al-Hadeethi Y - *Himachal Pradesh, University; Himachal Pradesh, Govt. College Rajgarh; Najran, University; Dasuya, Jagdish Chandra DAV College; Jeddah, King Abdul Aziz University*

Ceram.Int. 47, No. 5, 2021, p. 6201-6210

ZnO-SnO₂ nanocubes were investigated as promising materials for the efficient sensing of p-nitrophenol and faster photocatalytic degradation of dyes including methyl orange (MO), methylene blue (MB) and acid orange 74 (AO74). ZnO-SnO₂ nanocubes were prepared by the facile solution process at 50 °C using Zn(NO₃)₂·6H₂O and SnCl₂·2H₂O as a precursor in the presence of ethylenediamine. The morphology, structural, crystalline, optical, vibrational and compositional studies were assessed using FE-SEM, XRD, FTIR, XPS and photoluminescence spectroscopy. F-SEM revealed the formation of well-defined ZnO-SnO₂ nanocubes and structural examination revealed the formation of a crystalline tetragonal rutile phase for SnO₂ with some crystal sites doped with Zn. The as-synthesised nanocubes were explored for their photocatalytic activity towards three different dyes MO, MB, and AO74. Complete degradation of AO74 was observed within 4 minutes of photo-irradiation in the presence of 0.05 g ZnO-SnO₂ nanocubes. However, 97.17% and 41.63% degradation were observed for MB and MO within 15 and 60 minutes, respectively. All the dye degradation processes followed the pseudo-first-order kinetic model. Moreover, the as-synthesised nanocubes were used to fabricate highly sensitive and selective fluorescent chemical sensors for the detection of p-nitrophenol (PNP). ZnO-SnO₂ nanocubes showed a very low detection limit of 4.09 µM for the detection of PNP as calculated according to the 3σ IUPAC criteria. The as-synthesised ZnO-SnO₂ nanotubes were also highly selective for p-nitrophenol as compared to the other two isomers.

213388

FABRICATION OF DIRECT Z-SCHEME FeIn₂S₄/Bi₂WO₆ HIERARCHICAL HETEROSTRUCTURES WITH ENHANCED PHOTOCATALYTIC ACTIVITY FOR TETRACYCLINE HYDROCHLORIDE PHOTODEGRADATION

Shangguan X-Y; Fang B-L; Xu C-X; Tan Y; Chen Y-G; Xia Z-J; Chen W - *Taizhou, University*

Ceram.Int. 47, No. 5, 2021, p. 6318-6328

A series of direct Z-scheme FeIn₂S₄/Bi₂WO₆ hierarchical heterostructures, with intimate interfacial contact, were synthesised by the in-situ growth route and characterised. All the prepared FeIn₂S₄/Bi₂WO₆ nanocomposites showed enhanced photocatalytic activity towards photodegradation for the removal of tetracycline hydrochloride (TCH) compared with FeIn₂S₄ and Bi₂WO₆. The highest photocatalytic degradation activity was achieved by modulating the amount of FeIn₂S₄ in the nanocomposites and the optimised component ratio of FeIn₂S₄ to Bi₂WO₆ was 10 wt%. The enhanced photocatalytic activity was ascribed to efficient separation between photogenerated holes and electrons based on the construction of a direct Z-scheme system. The high photocatalytic stability of the resultant 10 wt% FeIn₂S₄/Bi₂WO₆ nanocomposites was revealed through six successive recycling reactions. The main intermediate generated during TCH photodegradation was investigated. The direct Z-scheme photocatalytic mechanism was confirmed by band position analysis, ESR and active species capture experiments.

213389

PHOTOCATALYTIC PERFORMANCE OF TiO₂ NANOTUBE STRUCTURE BASED ON TiN COATING DOPED WITH Ag AND Cu

Zhao Y; Wang C; Hu J; Li J; Wang Y - *Harbin, Normal University; Beijing, University of Chinese Academy of Sciences; Ningbo, Inst. of Material Technology & Engineering*

Ceram.Int. 47, No. 5, 2021, p. 7233-7240

TiN, TiN-Ag and TiN-Cu coatings were prepared by multi-arc ion plating and TiO₂ nanotube arrays were constructed on the coatings using an anodisation reaction at room temperature. By building TiO₂ nanotube arrays on the coating the performance of the hard coating itself was retained and this reduced the damage to the TiO₂ nanotube structure. Non-metallic N doping and co-doping of metallic Ag, Cu and non-metallic N resulted in nanotubes catalysed by visible light. The specific surface area of the TiO₂ could be altered by changing the anodising voltage. Results showed that an anodic oxidation voltage of 15V with Cu, N codoping resulted in optimum TiO₂ photocatalytic degradation.

213390

REVIEW MXenes AS A NEW TYPE OF NANOMATERIAL FOR ENVIRONMENTAL APPLICATIONS IN THE PHOTOCATALYTIC DEGRADATION OF WATER POLLUTANTS

Feng X; Yu Z; Sun Y; Long R; Shan M; Li X; Liu Y; Liu J - *Chengdu, Southwest Petroleum University; Mianyang, Normal University; Qufu, Normal University; Beijing, University of Chemical Technology*

Ceram.Int. 47, No. 6, 2021, p. 7321-7343

This review aims to design typical, cost-effective heterojunctions and Schottky junctions and their progress, mechanisms and trends in environmental organic pollutant degradation and detailed the heterogeneous catalytic mechanism of MXene-based photocatalysts for the degradation of organic pollutants. Ways to improve the photocatalytic performance of MXene by constructing heterojunctions and Schottky junctions are discussed. The surface properties, catalytic performance and pollution management of various MXene-based catalysts were compared and some issues and application strategies of MXene development were analysed. This review could open up ideas for new approaches and provide valuable clues for designing MXenes as a co-catalyst to develop more effective photocatalysts for practical application in environmental pollution management.

213391

SOLVOTHERMAL PREPARATION AND CHARACTERISATION OF ORDERED-MESOPOROUS ZrO₂/TiO₂ COMPOSITES FOR PHOTOCATALYTIC DEGRADATION OF ORGANIC DYES

Wang F; Pan K; Wei S; Ren Y; Zhu H; Wu H-H; Zhang Q - *Henan, University of Science & Technology; Beijing, University of Science & Technology; Xiamen, University*

Ceram.Int. 47, No. 6, 2021, p. 7632-7641

ZrO₂/TiO₂ composites, with an ink-bottle mesoporous structure, were synthesised by a mild solvothermal method without high-temperature calcination. The cavities in the ink-bottle structure can adsorb organic groups, making them suitable as catalytic materials. The effects of ZrO₂ additions and particle size on the morphology and catalytic performance of composite powders were investigated. Comparative experiments on changing the particle size and content of ZrO₂ showed that the TiO₂-10 wt% ZrO₂ composite had outstanding photocatalytic performance under

simulated sunlight, particularly when the particle size of as-prepared ZrO₂ was on the submicron scale. By mixing two different n-type semiconductors, ZrO₂ and TiO₂, a new heterostructure was formed to hinder the autonomous recombination of excited electron-hole pairs, which improved the photocatalytic activity.

213392

FACILE SYNTHESIS OF TiO₂-SUPPORTED Al₂O₃ CERAMIC HOLLOW FIBRE SUBSTRATES WITH EXTREMELY HIGH PHOTOCATALYTIC ACTIVITY AND REUSABILITYMagnone E; Kim M K; Lee H J; Park J H - *Dongguk, University***Ceram.Int.** 47, No. 6, 2021, p. 7764-7775

TiO₂ oxide was deposited on a microstructured alpha-Al₂O₃ ceramic hollow fibre substrate by a simple one-step solution-immersion process using TEOS as a binder. The effects of the starting coating solution's composition on the photocatalytic properties of TiO₂ powders, deposited on a substrate, was determined using TiO₂-supported Al₂O₃ ceramic hollow fibre substrates fabricated from coating solutions with different compositions. The substrates' effect on the methylene blue (MetB) degradation reaction under UV light was investigated. A strong correlation was observed between the initial coating solution compositions and the final photocatalytic characteristics of the TiO₂-supported Al₂O₃ ceramic hollow fibre substrates. Under optimal conditions, the MetB removal efficiency reached about 91% in a few minutes and is thought to be the highest and most rapidly attained MetB removal efficiency reported for TiO₂-supported Al₂O₃ ceramic hollow fibre substrates. Furthermore, apart from attaining an extremely high photocatalytic activity within minutes, the fabricated TiO₂-supported Al₂O₃ ceramic hollow fibre substrates had high photocatalytic stability even after several cycles.

213393

PREPARATION OF MULLITE WHISKER REINFORCED SiC MEMBRANE SUPPORTS WITH HIGH GAS PERMEABILITYXu X; Liu X; Wu J; Zhang C; Zhang Q; Tian K - *Wuhan, University of Technology***Ceram.Int.** 47, No. 6, 2021, p. 8150-8160

Mullite whisker-reinforced porous SiC membrane supports were fabricated by an in-situ reaction bonding technique using SiC particles as the skeletal materials, fine kaolin and alpha-Al₂O₃ powder as bonding agents, activated carbon as a pore-forming agent and MoO₃ as additive, followed by pressureless firing between 1350 and 1520 C for 2 h in air. The effects of firing temperature and MoO₃ addition content on the properties of the membrane supports, including open porosity, bending strength, microstructure, phase composition, thermal shock resistance and air permeability were investigated. Results showed that the morphology of the mullite whiskers and properties of the porous SiC membrane supports were affected by the firing temperature and MoO₃ content. The membrane support with 8 wt% MoO₃, fired at 1450 C for 2 h, had outstanding properties, including a bending strength of 24.69 plus or minus 0.86 MPa, an open porosity of 43.30 plus or minus 0.58%, a corresponding strength retention rate of 95.22% after 30 cyclic thermal shocks and Darcian permeability coefficient k₁ and non-Darcian permeability coefficients k₂ of 6.18 x 10⁻⁹ m² and 1.50 x 10⁻³ m, respectively.

213394

ECO-FRIENDLY SYNTHESIS OF A NOVEL MAGNETIC Bi₄O₅Br₂/SrFe₁₂O₁₉ NANOCOMPOSITE WITH EXCELLENT PHOTOCATALYTIC ACTIVITY AND RECYCLABLE PERFORMANCEWang H; Xu L; Wu X; Zhang M - *Sichuan, Normal University; Chongqing, University***Ceram.Int.** 47, No. 6, 2021, p. 8300-8307

To improve the catalytic activity and recovery efficiency of photocatalysts, Bi₄O₅Br₂ was decorated on SrFe₁₂O₁₉ via a microemulsion method to synthesise a novel magnetic Bi₄O₅Br₂/SrFe₁₂O₁₉ nanocomposite. The microstructures, morphological characteristics, photoelectrochemical properties, magnetic performance and photocatalytic mechanism of catalysts were investigated. The photocatalytic experiments showed that the Bi₄O₅Br₂/SrFe₁₂O₁₉ photocatalyst with 5 wt% of SrFe₁₂O₁₉ had the optimum capacity for the degradation of Rhodamine B (RhB). The activity of the photocatalyst was increased by the optimised energy band structure and the formation of an internal electric field in the type-II heterojunction. Moreover, h(+) and O₂(-) were the primary active constituents in the photocatalytic reaction as shown by energy-band structural analysis and radical scavenging experiments. The saturation magnetisation (Ms) and the recovery rate of composite photocatalyst were 2.76 emu/g and 88.2% respectively, which confirmed that the nanocomposite had excellent magnetic properties. The photodegradation rate was still 84.6% after five circulating tests, which showed that the sample had superior photostability. A new approach for the synthesis of composite magnetic photocatalysts based on Bi₄O₅Br₂ is provided with some guidance on the photocatalytic degradation mechanism.

213395

Tm, Yb CO-DOPED URCHIN-LIKE Cs_xWO₃ NANOCCLUSERS WITH DUAL FUNCTIONAL PROPERTIES: TRANSPARENT HEAT INSULATION PERFORMANCE AND ENHANCED PHOTOCATALYSISYang J; Liu J; Shi F; Ran S; Liu S - *Dalian, Polytechnic University***Ceram.Int.** 47, No. 6, 2021, p. 8345-8356

Tm³⁺, Yb³⁺ co-doped Cs_xWO₃ ((Tm, Yb)-Cs_xWO₃) urchin-like nanoclusters were synthesised in-situ by a solvothermal reaction followed by heat treatment. The effects of Tm, Yb co-doping on the crystal structure, adsorption/photocatalysis, visible transmittance and NIR absorption of the Cs_xWO₃ particles were investigated. Results indicated that the as-prepared (Tm, Yb)-Cs_xWO₃ samples had improved photocatalytic activity while maintaining excellent visible transmittance and NIR shielding properties. The sample 2T-10YC with 2 mol% Tm/10 mol% Yb co-doped Cs_xWO₃ had the highest photocatalytic activity with the photocatalytic degradation efficiency of Rhodamine B up to 69.86%, and its films, prepared from powder dispersions, exhibited high transparent heat shielding performance with visible transmittance of 71.4% and a NIR shielding rate of 90.8%, respectively. The enhanced photocatalytic activity of the sample was ascribed to lattice distortion and a decreased band gap caused by Tm, Yb co-doping, and the increased light utilisation due to the formation of an impurity energy level and multiple light reflection caused by hierarchical structure could also enhance the photocatalytic activity. The introduction of Tm and Yb could also refine the Cs_xWO₃ grains, which is conducive to improving the visible transmittance. Therefore, the as-prepared bi-functional (Tm, Yb)-Cs_xWO₃ particles could have potential application prospects in the field of energy-saving and environmental purification.

213396
SUPERHYDROPHOBIC BETA-SiAlON-MULLITE CERAMIC MEMBRANES WITH HIGH PERFORMANCE IN WATER TREATMENT
 Wang F; Dong B; Ke N; Yang M; Qian R; Wang J; Yu J; Hao L; Yin L; Xu X; Agathopoulos S - *China, University of Electronic Science & Technology; Ioannina, University*
Ceram.Int. 47, No. 6, 2021, p. 8375-8381
 Beta-SiAlON membranes, with high mechanical strength and suitable porosity, were prepared by pressureless sintering of samples obtained via phase-inversion and tape-casting and methods. Their surface was then modified with mullite whiskers using vapour-phase growth technology. The mullite whiskers, with a diameter of about 100 nm and a high aspect ratio of about 30, were formed at the surface of the asymmetrically structured membranes. The prepared membranes showed high performance for the separation of an aqueous emulsion of oil, (average diameter of oil droplets was 1.62 micron, oil rejection reached 94%). The mullite whiskers increased the surface roughness of the membranes, which led, after surface modification, to superhydrophobicity (water contact angle was 165 degrees), and to membrane distillation operational stability with 4% NaCl solution, containing 50 ppm humic acid, for more than 400 h. Results showed that the prepared beta-SiAlON-mullite membranes could have applications in the field of water treatment.

213397
HIGHLY EFFICIENT SYNERGETIC PIEZO/PHOTOCATALYTIC DEGRADATION IN NOVEL M_{0.5}Bi_{2.5}Nb₂O₉ (M = Li, Na, K) FERROELECTRIC NANOSHEETS
 Lu L; Liang N; Li X; Sun H; Zhang Q; Hao X - *Inner Mongolia, University of Science & Technology*
Ceram.Int. 47, No. 6, 2021, p. 8573-8583
 A series of novel bismuth layered structure ferroelectrics: M_{0.5}Bi_{2.5}Nb₂O₉ (M = Li, Na, K) nanosheets were prepared by one-step hydrothermal method. The fabricated nanosheets, with an average thickness of about 32 nm (Li_{0.5}Bi_{2.5}Nb₂O₉), 45 nm (Na_{0.5}Bi_{2.5}Nb₂O₉), 108 nm (K_{0.5}Bi_{2.5}Nb₂O₉) possessed excellent photodegradation performance to various organic dye pollutants with a high concentration of 20 mg/L [Rhodamine B (RhB), methyl orange (MO) etc.]. The degradation rate constants of RhB and MO could be further improved by polarisation-induced charge separation under ultrasonic-assisted illumination conditions, compared with those of light illumination alone, and it increased by about 2.5 times for the Na_{0.5}Bi_{2.5}Nb₂O₉ nanosheets. Results showed that bismuth layered ferroelectrics have potential applications in wastewater treatment as a photocatalyst.

213398
Dy₂BaCuO₅/Ba₄DyCu₃O_{9.09} S-SCHEME HETEROJUNCTION NANOCOMPOSITE WITH ENHANCED PHOTOCATALYTIC AND ANTIBACTERIAL ACTIVITIES
 Yousefi S R; Ghanbari M; Amiri O; Marzhooseyni Z; Mehdizadeh P; Hajizadeh-Oghaz M; Salavati-Niasari M - *Kashan, University; Ranya, University of Raparin; Erbil, International University; Kashan, University of Medical Sciences; Esfaryan University of Technology*
J.Am.Ceram.Soc. 104, No. 7, 2021, p. 2952-2965
 Semiconductor heterogeneous photocatalysis is of great research interest. The combination of two semiconductors with various energy diagrams can dramatically enhance the lifetime and separation of the charge carriers, restrain photogenerated electron-hole recombination and considerably enhance photocatalytic performance compared with other single or binary components. Dy₂BaCuO₅/Ba₄DyCu₃O_{9.09} nanocomposites, active photocatalysts below UV radiation, were prepared by hydrothermal synthesis and applied as catalysts to treat water containing organic pollutions and microorganisms. Dy₂BaCuO₅/Ba₄DyCu₃O_{9.09} nanocomposites degraded methyl orange about 87.0% after 120 min. The nanocomposites also showed antimicrobial activity against Gram-positive species, including a pathogenic strain of *Enterococcus faecalis* and *Staphylococcus aureus*, and a Gram-negative species, including *Klebsiella pneumonia* and *Escherichia coli*.

213399
ENHANCED INTERFACIAL ELECTRONIC TRANSFER OF BiVO₄ COUPLED WITH 2D g-C₃N₄ FOR VISIBLE-LIGHT PHOTOCATALYTIC PERFORMANCE
 Xu M; Zhu Y; Yang J; Li W; Sun C; Cui Y; Liu L; Zhao H; Liang B - *Qinhuangdao, Yanshan University; Hebei, Normal University of Science & Technology; Edinburgh, University*
J.Am.Ceram.Soc. 104, No. 7, 2021, p. 3004-3018
 A BiVO₄/2D g-C₃N₄ direct dual semiconductor photocatalytic system was fabricated by the electrostatic self-assembly of BiVO₄ microparticles and graphite-like carbon nitride (g-C₃N₄) nanosheets. Experimental measurements and first-principle calculations showed that the formation of a built-in electric field and the opposite band bending around the interfacial region in BiVO₄/2D g-C₃N₄ and the intimate contact between BiVO₄ and 2D g-C₃N₄ led to high separation efficiency of the charge carriers. The intensity of built-in electric field was greatly enhanced due to the ultrathin nanosheet structure of 2D g-C₃N₄. As a result, BiVO₄/2D g-C₃N₄ had excellent photocatalytic performance with 93.0% Rhodamine B removal after 40 min visible light irradiation and the photocatalytic reaction rate was about 22.7 and 10.3 times as high as that of BiVO₄ and 2D g-C₃N₄, respectively. BiVO₄/2D g-C₃N₄ also had enhanced photocatalytic performance in the degradation of tetracycline. The work should provide insights into the understanding the significant role of built-in electric field in heterostructure and fabricating highly efficient direct dual semiconductor systems.

213400
DEVELOPMENT OF MAGNETIC RECYCLABLE SPINEL PHOTOCATALYSTS WITH ENHANCED SUNLIGHT-DRIVEN DEGRADATION OF INDUSTRIAL DYES
 Tangcharoen T; T-Thienprasert J; Kongmark C - *Bangkok, Kasetsart University*
J.Am.Ceram.Soc. 104, No. 7, 2021, p. 3695-3714
 Nanocrystalline nickel and copper-substituted zinc aluminate spinel powders (Ni_xZn_{1-x}Al₂O₄ and Cu_xZn_{1-x}Al₂O₄) with different additional ion concentrations (x = 0-1) were synthesised by sol-gel auto combustion using diethanolamine as fuel. The structures, chemical bonds, morphologies, composition, surface area, optical properties and magnetic behaviour of the samples were studied by XRD, FTIR, SEM, EDS, BET method, UV-vis diffuse reflectance spectroscopy and vibrating sample magnetometry. Results confirmed that a single-phase spinel structure was obtained for all calcined aluminate powders with various crystallite sizes and lattice constants. The band gap energy of all modified aluminates was in the range 2.99-3.15 eV, much lower than that of the pure sample (5.60 eV). The results indicated that the Ni²⁺ and Cu²⁺-substituted ZnAl₂O₄ samples could be effectively photoexcited by both the UV and visible light. The photocatalytic activity was determined by investigation of the way the four main

pollutant types (rhodamine B, methylene blue, methyl orange and methyl red) decomposed when irradiated by sunlight. For all optimum samples of organic dyes, the efficiency of the photocatalytic degradation achieved 96% after 150 min. Each of the modified photocatalysts could be reused and showed a high degree of stability. Magnetic measurements showed that the S-shaped curve of ferrimagnetism could arise in samples with the optimum concentration, although pure ZnAl₂O₄ had diamagnetic properties. Compared to pure ZnAl₂O₄, the modified samples showed enhanced remanent magnetisation, indicating that it is easy to recover those magnetic photocatalysts through the use of an external magnetic field application. The findings confirm that ZnAl₂O₄ powders substituted by both Ni²⁺ and Cu²⁺ may have potential in environmentally beneficial harvesting of solar energy.

213401
SUPERHYDROPHOBIC CERAMIC HOLLOW FIBRE MEMBRANES FOR TRAPPING CARBON DIOXIDE FROM NATURAL GAS VIA THE MEMBRANE CONTACTOR SYSTEM

Saud I H; Othman M H D; Hubadillah S K; Aziz M H A; Pauzan M A B; Ismail A F; Jaafar J; Rahman M A - *Malaysia, Universiti Teknologi; Basra, Technical Engineering College*

J. Aust. Ceram. Soc. 57, No. 3, 2021, p. 705-717

The membrane contactor system is one of the most important technologies for trapping CO₂ from natural gas. To apply this technology, hollow fibre membranes with a superhydrophobic surface must be used. The membranes are prepared from kaolin clay via the phase inversion/sintering technique and modified by three types of fluoroalkylsilane (FAS) molecules (C6, C8, C10) for different immersion times (6, 24, 48, 72 h) to capture CO₂ from natural gas by contacting the gas-liquid system. Kaolin was chosen due to its abundance and affordable price, as well as to the high amount of OH groups in the surface which easily react with FAS during the grafting process. Superhydrophobicity was distinguished by FTIR, SEM, liquid entry pressure of water (LEP_w) measurement and contact angle measurement. The lowest pore size of the grafted membrane obtained for C8 was about 1.32 micron; this was regarded as the perfect target for high membrane resistance. The chosen membrane was tested for CO₂ capture via the membrane contactor system. With increasing immersion time, hydrophobicity gradually increased; 48 h was found to be sufficient for obtaining the desired superhydrophobicity so as to avoid pore wetting. Based on sufficient LEP_w and contact angle, the perfect type of FAS for separating CO₂ was C8. A reduction in pH (from 6.6 to 4.3) within 1 h was seen after assessing the performance when using water as an absorbent.

213402
PREPARATION OF NANO-TiO₂/PURIFIED DIATOMITE COATING AND ITS PHOTOCATALYTIC PROPERTIES OF FORMALDEHYDE DEGRADATION

Wang L; Zhao J; Wang Y; Zheng S - *Luoyang, Institute of Science & Technology; China, University of Mining & Technology*

J. Ceram. Process. Res. 21, No. 6, 2020, p. 751-756

Diatomite was purified by calcination at 550 C followed by immersion in 50% sulphuric acid. The washed diatomite was dispersed in water and dilute sulphuric acid added to adjust the pH to 3. Titanium sulphate solution was added drop-wise, followed by ammonium sulphate and a mixture stirred for 30 min. The temperature was then increased to 40 C and the pH adjusted to 5.5. Following 1 h continuous reaction the precipitate was washed, dried and calcined at 650 C. A core-shell structure with a diatomite core and a nanotitania shell was obtained. The prepared material was used to produce a photocatalytic coating by dispersion in media with additions of pigments and fillers. The catalytic activity for the degradation of formaldehyde gas on exposure to white light was assessed. The catalytic activity met the requirements of the appropriate Chinese standard, and the degradation efficiency was attributed to a synergistic effect of adsorption and photocatalytic degradation.

213403
PHOTOCATALYTIC PERFORMANCE OF Fe-SUBSTITUTED ZnAl₂O₄ POWDERS UNDER SUNLIGHT IRRADIATION ON DEGRADATION OF INDUSTRIAL DYES

Tangcharoen T; T-Thienprasert J; Kongmark C - *Bangkok, Kasetsart University*

Int. J. Appl. Ceram. Technol. 18, No. 4, 2021, p. 1125-1143

A sequence of Fe-substituted zinc aluminate, ZnF_xAl_{2-x}O₄, powders with varied Fe³⁺ ion concentrations (x = 0-2) were prepared by the sol-gel auto combustion method using diethanolamine as the fuel. The structure, chemical bonds, morphologies, composition, surface area, and optical and magnetic properties of the samples were studied by XRD, FTIR, TEM, SEM, EDS, BET, UV-DRS and VSM. A single-phase spinel structure with different interplanar spacing and crystallite sizes was obtained for the calcined aluminate powders. The bandgap energy of the Fe-substituted aluminates was in the range of 2.04-3.14 eV, which was much lower than that of the pure sample (5.60 eV). Thus, Fe³⁺-substituted ZnAl₂O₄ samples could be successfully photoexcited using both UV and visible light, as suggested by the results. The photocatalytic activity was assessed on contaminants including rhodamine B, methylene blue, methyl orange and methyl red. Photodegradation reached 98% after 150 min for all optimal dye samples. Each of the altered photocatalysts could be recycled and displayed high stability. Magnetic measurements indicated ferrimagnetism, although pure ZnAl₂O₄ is diamagnetic. The substituted samples show a great improvement in remanent magnetisation compared to pure ZnAl₂O₄, indicating that magnetic photocatalyst recovery is easily achieved by applying an external magnetic field. Fe³⁺-substituted ZnAl₂O₄ powders may help in the ecologically friendly collection of solar energy.

213404
CONCURRENT AND MODULATED SEPARATION OF CO₂ AND O₂ BY A FLUORITE/PEROVSKITE-BASED MEMBRANE

Fabian-Anguiano J A; Ortega-Lugo R; Ramirez-Moreno M J; Zeifert B; Gomez-Yanez C; Ortiz-Landeros J - *Mexico, Instituto Politecnico Nacional*

Int. J. Appl. Ceram. Technol. 18, No. 4, 2021, p. 1307-1320

A novel dense ceramic-molten carbonate membrane used for the selective separation of CO₂ and O₂ at elevated temperature (850-950 C) was fabricated. A fluorite/perovskite ceramic oxide with mixed ionic-electronic conductivity and with the general formula Ce_{0.9}Pr_{0.1}O_{2-δ}/Pr_{0.6}Sr_{0.4}Fe_{0.5}Co_{0.5}O_{3-δ} (CP-PSFC, 60:40 wt%) was synthesised by the citrate EDTA route. The resulting product was used to prepare a partially sintered, disk-shaped porous ceramic support, which was then infiltrated with a ternary mixture of molten salts with the composition Li₂CO₃/Na₂CO₃/K₂CO₃. Permeation measurements at high temperature suggested a concurrent separation of both species, CO₂ and O₂. The system exhibited high permeance of CO₂ and O₂, rising to maximum values of 2.17 x 10⁻⁷ and 0.69 x 10⁻⁷ mol/m².s.Pa, respectively, at 950 C. It may be possible to modulate the permeate CO₂:O₂ ratio by changing the fluorite to perovskite proportion in the membrane composition. The stability of the obtained membrane was studied under long-term permeation tests and was found to display remarkable chemical and thermal stability for 110 h at 875 C.

213405
DEFLUORIDATION OF DRINKING WATER USING A CERAMIC FILTER DECORATED WITH IRON OXIDE-BIOCHAR COMPOSITES
 Mandoreba C; Gwenzi W; Chaukura N - *Zimbabwe, University; Kimberley, Sol Plaatje University*
Int. J. Appl. Ceram. Technol. 18, No. 4, 2021, p. 1321-1329
 Most of the effective defluoridation methods that have been developed are expensive and difficult to use on a household level. One safe, low-cost, decentralised water treatment method which has attracted interest is the use of ceramic filters (CFs). Few studies have explored the use of biochar composites in CFs for defluoridation of water, with the bulk of them being based on batch and column experiments. An attempt was made to develop and evaluate a laboratory-scale, low-cost CF using clay, sawdust and iron oxide-biochar (FBC) for fluoride removal in synthetic and actual water samples. Three CFs with varying FBC content (0, 5 and 10%) were fabricated. Water absorption, apparent porosity and flow rate decreased with decreasing FBC content, while the impact strength increased with decreasing FBC content. The CFs reduced the fluoride concentration of both groundwater and synthetic water to below the WHO recommended limit (1.5 mg/L). While further studies are required to optimise the CF, the results show that a CF decorated with FBC composites is effective for the defluoridation of drinking water.

213406
LOW-ALKALI BOROSILICATE GLASS MICROSPHERES FROM WASTE CULLET PREPARED BY FLAME SYNTHESIS
 Larionau P; Hujova M; Michalkova M; Mahmoud M; Svancarkova A; Galuskova D; Parchoviansky M; Bernardo E; Galusek D; Kraxner J - *Belarus, State Technological University; Trencin, Alexander Dubcek University; Trencin, Joint Glass Center of the IIC SAS, TnUAD & FChPT STU; Padua, University*
Int. J. Appl. Glass Sci. 12, No. 4, 2021, p. 562-569
 Despite the fact that glass recycling is the preferred technique of glass waste management, fine fractions of container soda-lime glass or cullet of other compositions are nevertheless landfilled. This occurs despite the fact that alternatives exist. Success may lie in sophisticated upcycled goods that provide greater economic motive for industrial deployment, although they are frequently linked to different methods of product synthesis. We show how waste glass may be recycled by creating glass microspheres (GM) from specialised low-sodium aluminoborosilicate-based glasses via flame synthesis (FS). SEM, simultaneous thermal analysis combined with differential thermal analysis (STA-DTA), and image analysis were used to analyse GM and the precursors extracted from colourless medicinal vials or glass fibres. To examine corrosion kinetics products, a dynamic corrosion test was performed and analysed using ion-coupled plasma with optical emission spectroscopy (ICP-OES). FS has shown to be a quick way of converting waste glass into GM. Aside from analysis of the starting material and final products, this article also indicates the potential of processing for various landfilled waste glasses and addresses the production of GM for water filters and fillers for polymers.

213407
DESIGNING EFFECTIVE AND STABLE S-SCHEME RGO/AgVO₃/AgBr HYBRID WITH ENHANCED PHOTOCATALYTIC PERFORMANCE
 Abulizi A; Zhang T; Kadeer K; Tursun Y; Talifur D - *Xinjiang, University*
J. Am. Ceram. Soc. 104, No. 8, 2021, p. 4095-4108
 The low separation of photogenerated electron-hole pairs and cycle stability restricts the development of photocatalytic technology for water purification. Reduce graphene oxide (RGO)/AgVO₃ composites were fabricated by photo-ultrasonic assisted reduction and AgBr nanoparticles were assembled on the surface of the RGO/AgVO₃ via in-situ ion exchange. A series of characterisation and experimental results indicated that the introduction of RGO affected the growth of the crystal phase for the AgVO₃ nanorods, with the result that AgVO₃ nanorods became thicker and shorter with increasing RGO content. RGO could act as a bridge to promote the migration of electrons, also improving photocatalytic activity. In-situ growth of AgBr on the surface of AgVO₃ nanorods could prevent its agglomeration and exfoliation, thus improving the photocatalytic activity and cycle stability of the composites. RGO1%/AgVO₃/AgBr30% exhibited excellent photocatalytic activity and stability for methylene blue (MB) degradation due to its unique structure, and its removal ratio reached at 96.2% within 50 min. The separation of photogenerated electron-hole pairs of AgVO₃ was markedly improved due to the introduction of RGO and AgBr. Based on the trapping experiments and theoretical calculation of band gap, a possible S-scheme photocatalytic mechanism for improved photocatalytic activity is proposed.

213408
ROBUST CERAMIC NANOFIBROUS MEMBRANES WITH ULTRA-HIGH WATER FLUX AND NANOPARTICLE REJECTION FOR SELF-STANDING ULTRAFILTRATION
 Jiang J; Ni N; Xiao W; Zhao X; Guo F; Fan X; Ding Q; Hao W; Xiao P - *Shanghai, Jiao Tong University*
J. Eur. Ceram. Soc. 41, No. 7, 2021, p. 4264-4272
 Ultra-strong Y₃Al₅O₁₂-Al₂O₃ electrospun nanofibrous membranes (ENMs) were fabricated using graphene oxide (GO) as a sacrificial additive. The optimised membranes had tensile strengths of 5.63 plus or minus 0.31 MPa after sintering at 900 C and 3.48 plus or minus 0.44 MPa at 1000 C. The high strength of the membranes ensured that they could be used independently under a pressure of up to 160 K Pa. Excellent nanoparticle filtration performance was achieved with a high permeation flux of 5718-8494 L/m²/h/bar and high rejection efficiencies above 99.75 % in both neutral and acidic water conditions. This work showed a facile route for the effective strengthening of ceramic ENMs with potential harsh-environment self-standing ultrafiltration applications.

Engineering (including machining, tool tips and abrasives)

See also Abstract(s): 213609 213618 213643

Energy applications

See also Abstract(s): 213395 213400 213403 213482 213488 213513 213530 213553 213558 213567 213570 213578 213586 213591 213639

213644 213645 213649

213409

MONOLITHIC ALL-PEROVSKITE TANDEM SOLAR CELLS: RECENT PROGRESS AND CHALLENGESAhn Y J; Ji S G; Kim J Y - *Seoul, National University***J. Korean Ceram. Soc.** 58, No. 4, 2021, p. 399-413

All-perovskite tandem solar cells (TSCs) have potential to achieve efficiencies beyond the Shockley-Queisser limit due to their excellent photovoltaic properties and cost effectiveness. The current status of all-perovskite TSCs and recent research progress are reviewed, focusing on three main strategies: optimisation of the interconnecting layer (ICL); suppression of tin oxidation in low-bandgap perovskites; and prevention of halide segregation in wide-bandgap perovskites. The development of triple-junction all-perovskite devices and remaining issues facing all-perovskite TSCs are also discussed. If the efficiency and stability can be further improved, all-perovskite TSCs are promising for flexible solar cells and transparent solar cell applications that cannot be implemented by silicon-based TSCs.

Nuclear including disposal of nuclear waste

See also Abstract(s): 213349 213365 213369 213497

213410

PHYSICAL, THERMAL, OPTICAL, STRUCTURAL AND NUCLEAR RADIATION SHIELDING PROPERTIES OF Sm₂O₃ REINFORCED BOROTELLURITE GLASSESKilic Issa S A M; Ilk E; Kilicoglu O; Gokhan Issever U; El-Mallawany R; Issa B; Tekin H O - *Eskisehir, Osmangazi University; Assiut, Al-Azhar University; Tabuk, University; Menoufia, University; Sharjah, University***Ceram. Int.** 47, No. 5, 2021, p. 6154-6168

The effect of samarium (III) oxide reinforcement on the physical, thermal, optical, structural and nuclear radiation attenuation properties of a group of novel borotellurite glasses were investigated using experimental and numerical methods. FTIR spectra of powdered samples were taken at 250-4000 /cm and the transmittance and absorption characteristics, optical band gaps and Urbach energies were measured. The glass transition temperatures, crystallisation temperatures and melting temperatures of the samples were determined. Nuclear radiation shielding properties were determined for gamma-ray, neutrons and heavy charged particles. The lowest transmittance and highest absorbance were reported for the TBVS1.5 sample with the highest Sm₂O₃ content. The results from the nuclear radiation shielding calculations showed that the TBVS1.5 sample had superior nuclear radiation shielding properties against gamma-ray, neutron and heavy charged particles. Increased Sm₂O₃ contents improved the nuclear radiation attenuation properties by keeping other material properties within useable limits.

213411

THEORETICAL AND EXPERIMENTAL GAMMA-RAYS ATTENUATION CHARACTERISTICS OF WASTE SODA-LIME GLASS DOPED WITH La₂O₃ AND Gd₂O₃Kurtulus R; Kavas T; Akhurt I; Gunoglu K - *Afyon Kocatepe, University; Isparta, Suleyman Demirel University; Isparta, University of Applied Science***Ceram. Int.** 47, No. 6, 2021, p. 8424-8432

The gamma-ray shielding characteristics of waste soda-lime glass (SLG) containing lanthanum oxide (La₂O₃) and gadolinium oxide (Gd₂O₃) were investigated using theoretical and experimental methods. For this purpose, both La₂O₃ and Gd₂O₃ were separately added at 0.005, 0.05 and 0.5 wt% in place of waste SLG. The glass series SLGL and SLGG were fabricated via conventional melting techniques. Some physical analyses were carried out while radiation shielding parameters were assessed using gamma-ray spectroscopy and XCOM computations on the produced glass specimens. The synthesised glasses were transparent, defect-free and the glass density showed an increasing trend with the addition of both oxides, but the SLGG series was denser. Experimental measurements showed that both oxide additions had the ability to increase the linear attenuation coefficient (LAC) values. The LAC was improved from 0.151 to 0.242 /cm for Gd₂O₃ doping and 0.148 to 0.202 /cm for the La₂O₃ series. Good agreement was achieved between the experimental measurements and theoretical calculations. Based on the measured LAC values, other important parameters including mass attenuation coefficient (MAC), half-value layer (HVL), and mean free path (MFP) were evaluated for the series of glasses. It was concluded that the SLGG3 glass could compete with commercial radiation shielding glasses.

213412

SYNTHESIS, STRUCTURE, AND CHARACTERISATION OF THE THORIUM ZIRCONOLITE CaZr_{1-x}Th_xTi₂O₇ SYSTEMBlackburn L R; Sun S-K; Gardner L J; Maddrell E R; Stennett M C; Corkhill C L; Hyatt N C - *Sheffield, University; Foshan, University; Workington, National Nuclear Laboratory***J. Am. Ceram. Soc.** 104, No. 7, 2021, p. 2937-2951

A series of zirconolite ceramics with composition CaZr_{1-x}Th_xTi₂O₇ (Δx = 0.10) were reactively sintered at 1350 C for 20 h, in air (x = 0-0.60) and 5% H₂/N₂ (x = 0-0.40). A sample with composition corresponding to x = 0.20 was also produced by hot isostatic pressing (HIP) at 1300 C and 100 MPa for 4 h. Th⁴⁺ immobilisation was most readily achieved under oxidising conditions, with Th⁴⁺ preferentially incorporated within a pyrochlore-structured phase in the range x = 0.10-0.50. However, formation of the zirconolite-4M polytype was not seen. Single-phase pyrochlore with nominal composition CaZr_{0.40}Th_{0.60}Ti₂O₇ when targeting x = 0.60 was synthesised. Th⁴⁺ incorporation under reducing conditions produced a secondary Th-bearing perovskite, comprising 24.2 plus or minus 0.6 wt% of the phase assemblage when targeting x = 0.40, alongside 8.8 plus or minus 0.3 wt% undigested ThO₂. Under reducing conditions, powder XRD data were consistent with zirconolite adopting the 3T polytype structure. The sample produced by HIP had a nonequilibrium phase assemblage, yielding a major phase of zirconolite-2M alongside accessory Th⁴⁺-bearing phases ThTi₂O₆, ThO₂ and perovskite. The efficacy of Th⁴⁺ as a Pu⁴⁺ surrogate was therefore demonstrated, with implications for the formation of Zr-stabilised Th-pyrochlore phases as matrices for waste with elevated Th⁴⁺ content.

213413

DERIVATION OF THE STRUCTURAL INTEGRITY OF RESIDUAL (SIR) GLASS MODEL FOR THE ENHANCEMENT OF WASTE LOADINGMcClane D L; Amoroso J W; Hsieh M C; Fox K M; Kruger A A - *Savannah River National Laboratory; US, Dept. of Energy***J. Am. Ceram. Soc.** 104, No. 7, 2021, p. 3235-3246

A new model based on glass structure to allow for enhanced waste loading in nuclear waste glass while maintaining chemical durability was proposed. The model was derived by splitting the molar concentrations of the targeted starting glass composition into theoretical crystalline phases

anticipated during devitrification and a residual glass. An empirically derived relationship based on maintaining the residual glass structure, determined from a calculated non-bridging oxygen content, was shown to successfully screen glasses for acceptable durability. This model could identify durable glass compositions containing 20-35 wt% Al₂O₃, a concentration that would significantly increase the projected waste loading in glasses processed at the Hanford Tank Waste Treatment and Immobilization Plant.

213414
PROCESSING OF FULLY CERAMIC MICROENCAPSULATED FUELS WITH A SMALL AMOUNT OF ADDITIVES BY HOT-PRESSING
 Kim G-D; Kim Y-W - *Seoul, University*
J.Eur.Ceram.Soc. 41, No.7, 2021, p.3980-3990
 The effects of processing parameters on the properties of SiC matrices and fully ceramic microencapsulated (FCM) fuel pellets were investigated, using a small amount of additives and hot pressing. Increased applied pressure and sintering time improved the thermal conductivity of the SiC matrix and FCM pellets. Tristructural isotropic (TRISO) particles without an outer pyrolytic carbon layer are not suitable for processing FCM pellets due to the corrosion of the chemical vapour deposited SiC layer by the additive-derived liquid phase during sintering. The FCM pellet sintered with 2.71 wt% AlN-Y₂O₃-Sc₂O₃-MgO additives at 1870 C for 4 h under 30 MPa in argon had excellent thermal conductivity (61.7 W/m K at a TRISO particle content of 43.3 %), due to reduced amounts of additives, a high sintered density (residual porosity of about 0.4 %) and a highly thermally conductive SiC matrix (105.0 W/mK).

APPLICATIONS

See also Abstract(s): 213351 213485 213514 213555

Adhesives and sealants

See also Abstract(s): 213352

213415
NOVEL B₂O₃/B₄C-MODIFIED COMPOSITE ADHESIVE WITH WIDE OPERATIVE TEMPERATURE RANGE FOR ALUMINA FIBRE FABRIC BONDING
 Liu P; Zhang X; Xue Y; Guo A; Liu L - *Tianjin, University*
Ceram.Int. 47, No.5, 2021, p.6643-6652
 A heat resistant adhesive, with a wide operational temperature range, was developed for bonding alumina fibre fabrics using an organic-inorganic modification. PVA modified by B₂O₃ generated a complex crosslinked network connected by B-O-C bonds, which could enhance the bonding strength of the adhesive after heat treatment between RT and 400 C. The addition of B₄C increased the bonding strength after heat-treatment between 400 C and 800 C due to the formation of molten B₂O₃ and borosilicate glass. The appropriate addition of B₄C resulted in a denser structure without transforming the fracture mode of the bonding joints, which enhanced the strength of the bonding joints after sintered at 800 C. But excessive additions of B₄C transformed the fracture mode of the bonding joints to brittle fracture, which degraded the strength of the bonding joints.

Films and coatings

213416
DESIGN, THERMAL SHOCK RESISTANCE OF DENSE BaO-Al₂O₃-SiO₂ GLASS/Si₃N₄-BARIUM FELDSPAR COATING FOR POROUS Si₃N₄ CERAMIC
 Tong Z; Liu Z; Yu H; Zhang B; Li X; Su D; Ji H - *Tianjin, University*
J.Am.Ceram.Soc. 104, No.7, 2021, p.2981-2991
 Silicon nitride-monoclinic barium feldspar (Si₃N₄-m-BAS) composites have good dielectric properties, low density and low thermal expansion coefficient (CTE). Preparing dense Si₃N₄-m-BAS coatings on porous Si₃N₄ ceramic can improve its water resistance and ensure its dielectric performances, but such coatings have not yet been reported, due to the difficulty of synthesising m-BAS and the very high temperatures required for densification of Si₃N₄-BAS composites. BaO-Al₂O₃-SiO₂ glass/Si₃N₄-BAS coatings were fabricated by a manual spray method and pressureless sintering at 1450 C. Combining the influence of Si⁴⁺ on the crystal phase composition of BAS and the volume expansion effect of silicon in N₂, an effective coating structure design scheme was proposed. By changing the content of silicon powder, the CTE and horizontal shrinkage of the coating during sintering were controlled. The prepared coatings had low water absorption and high bonding strength. During thermal shock tests, SiO₂ produced by the oxidation of Si₃N₄ healed the cracks in the coating, thus delaying the degradation of the properties. The coating prepared should have application as radomes in extreme service environments.

213417
CORROSION BEHAVIOUR OF CALCIUM-MAGNESIUM-ALUMINOSILICATE (CMAS) ON SINTERED Gd₂SiO₅ FOR ENVIRONMENTAL BARRIER COATINGS
 Kim S-H; Nagashima N; Matsushita Y; Kim B-N; Jang B-K - *Kyushu, University; Ibaraki, Nat.Inst.for Materials Science*
J.Am.Ceram.Soc. 104, No.7, 2021, p.3119-3129
 The high-temperature corrosion behaviour of calcium-magnesium-aluminosilicate (CMAS) on sintered Gd₂SiO₅ for environmental barrier coatings (EBCs) was studied. Gd₂O₃-SiO₂ powder was spark plasma sintered at 1400 C for 20 min to fabricate the sintered Gd₂SiO₅. CMAS was deposited on the sintered Gd₂SiO₅ surface and exposed for 2, 12 and 48 h at 1400 C by isothermal heat treatment. The main corrosion factor was Ca, and a Ca₂Gd₈(SiO₄)₆O₂ phase was formed by reaction with Gd₂SiO₅. The elongated morphology of the Ca₂Gd₈(SiO₄)₆O₂ particles observed in the reaction area became thicker with increasing heat treatment time as the CMAS was dissolved. High-temperature XRD and DSC showed that CMAS melted at 1243 C or a higher temperature formed the reaction area. The Ca₂Gd₈(SiO₄)₆O₂ phase was recrystallised and grew due to the reaction of Gd₂SiO₅ and Ca of the CMAS components.

213418
TAILORABLE THERMAL EXPANSION IN LEUCITE-POLLUCITE MATERIALS DERIVED FROM GEOPOLYMERS FOR ENVIRONMENTAL BARRIER COATINGS
 Steveson A J; Kriven W M - *Illinois, University at Urbana Champaign*
J. Am. Ceram. Soc. 104, No. 7, 2021, p. 3397-3410
 An attempt was made to develop the understanding of the thermal behaviour of mixed-cation potassium and caesium geopolymer-derived ceramics by studying the atomic mechanism of the thermal expansion and phase transition in the leucite-pollucite system. The K[AlSi₂O₆]-Cs[AlSi₂O₆] pseudo-binary system was synthesised by geopolymer crystallisation. The thermal expansion properties of the materials were studied by in-situ high-temperature XRD to characterise thermal expansion behaviour for potential application as environmental barrier coatings for composite materials for turbine engines. Tailorable thermal expansion by changing the cation stoichiometry gave reduced thermal expansion mismatch with SiCf/SiC composites compared to rare-earth-based coatings.

213419
THERMOMECHANICAL AND THERMOCHEMICAL STABILITY OF HfSiO₄ FOR ENVIRONMENTAL BARRIER COATING APPLICATIONS
 Ridley M J; Opila E J - *Virginia, University*
J. Am. Ceram. Soc. 104, No. 7, 2021, p. 3593-3602
 The thermomechanical and thermochemical stability of hafnion (HfSiO₄) was evaluated for its use as an environmental barrier coating (EBC) for SiC/SiC CMCs. High-temperature XRD showed that hafnion has an excellent coefficient of thermal expansion (CTE) match with SiC and minimal CTE anisotropy, which supports hafnion as an EBC candidate. However, high-velocity water vapour testing at 1200-1400 C showed large amounts of silica depletion from chemical reaction at all velocities and excessive material erosion at steam velocities > 190 m/s. HfSiO₄ is therefore not suitable as an EBC for turbine applications, due to insufficient thermochemical stability in water vapour-containing combustion environments.

213420
PREPARATION AND HIGH-TEMPERATURE OXIDATION RESISTANCE OF MULTILAYER MoSi₂/MoB COATING BY SPENT MoSi₂-BASED MATERIALS
 Zhu L; Wang X; Ren X; Kang X; Akhtar F; Feng P - *China, University of Mining & Technology; Lulea, University of Technology*
J. Am. Ceram. Soc. 104, No. 7, 2021, p. 3682-3694
 The properties of molybdenum make it a useful material for aerospace applications. However, its oxidation resistance at high temperature needs to be improved which can be achieved with an anti-oxidation coating. Spent MoSi₂ and MoB were used as raw materials to prepare multilayer MoSi₂/MoB coatings on molybdenum by a two-step method (slurry deposition and spark plasma sintering). Dense MoSi₂/MoB coatings were formed after sintering while penetrated cracks appeared in the MoSi₂ coating due to coefficient of thermal expansion mismatch between the Mo substrate and the coating. After sintering the MoSi₂/MoB coatings, MoB and Mo₂B diffusion layers were formed between the MoB transition layer and the Mo substrate without defects, exhibiting good metallurgical bonding. The high-temperature oxidation behaviour of the coatings (1500 C) was also examined. After oxidation for 50 h at 1500 C, the lowest mass gain (0.035 mg/cm²) was obtained for the MoSi₂/MoB coating, and the oxide scale was dense and complete without voids, inhibiting the oxygen diffusion at elevated temperature. Compared with MoSi₂ coatings under the same oxidation conditions, relatively thinner silica oxide scale was obtained by the MoSi₂/MoB coating because of the reduction of cracks, and the multilayer coating showed better anti-oxidation properties at high temperature.

213421
IN SITU OBSERVATION OF ENVIRONMENTAL BARRIER COATING AGAINST CALCIUM-MAGNESIUM-ALUMINA-SILICATE ATTACK AT ELEVATED TEMPERATURE
 Yang H; Zhang Y; Li Y; Huang X; Liu Y - *Changsha, Central South University*
Int. J. Appl. Ceram. Technol. 18, No. 4, 2021, p. 1281-1287
 Environmental barrier coatings (EBCs) play a crucial role in protecting ceramic matrix composites exposed to harsh environments. An in-situ observation method was used to systematically study the resistance of EBCs to calcium-magnesium-alumina-silicate (CMAS) phase attack at elevated temperatures (1200, 1300 and 1500 C). Melting and spreading of the CMAS on the surface of the EBC could be seen using the method. The erosion of the EBC by the CMAS phase at the different temperatures was analysed using SEM-EDS. The findings provide guidance for modifying the EBCs.

213422
ENHANCEMENT OF THE Ti-6Al-4V ALLOY CORROSION RESISTANCE BY APPLYING CrN/CrAlN MULTILAYER COATING VIA ARC-PVD METHOD
 Azizi-Malekabadi M; Bakhshi H; Shahbazi H; Nosrati H - *Tehran, Tarbiat Modares University; Ontario, Brock University; Shiraz, University of Technology; Aarhus, University Hospital*
Int. J. Appl. Ceram. Technol. 18, No. 4, 2021, p. 1288-1296
 A Ti-6Al-4V substrate was coated by CrN-CrN/TiN-TiN and CrN/CrAlN multilayer coatings by cathodic arc physical vapour deposition. Potentiodynamic polarisation (PDP) measurements revealed that the double-layer (0.16 microamps/Cm²) and TiN (0.51 microamps/Cm²) samples had the lowest and highest corrosion current density, respectively, indicating the higher corrosion resistance of double-layer coating. FESEM, XRD, open circuit potential, PDP and electrochemical impedance spectroscopy were used to characterise the coatings and evaluate their corrosion behaviour. The application of a double-layer coating significantly improved the corrosion protection of the Ti-6Al-4V alloy compared to the sample coated with TiN.

Electrical applications

See also Abstract(s): 213386 213409 213517 213518 213522 213530 213532 213533 213544 213558 213571 213572 213573 213575 213578

213579 213592

213423

HIGH-VOLTAGE SURGE PROTECTION BY A VARISTOR-FILLED AIR GAPYang P; Gomez C A; Andrews S; Sorenson J D; Chen K S - *Sandia National Laboratories***J.Am.Ceram.Soc.** 104, No.7, 2021, p.3247-3259

The electrical discharge across a ZnO-based varistor granule-filled air gap under a fast-rising voltage pulse was studied for surge protection applications. The effects of temperature and pressure on the arc and the electrical conduction were analysed by the characteristic changes in voltage waveforms triggered by a fast-rising high voltage pulse. In addition to the gap size, experimental results showed that competing mechanisms among arc conduction, conduction through the varistor granule network, thermionic emission from Joule heating at granule-to-granule contact points and the magnitude of the switching voltage dictated the maximum surge protection voltage for the filled air gap. It was shown that accumulated degradation was created at small contact points between the varistor granules by repetitive assaults from longer duration, high voltage pulses. The uniqueness of using varistors over other dielectric granules in an air gap for surge protection was identified and is discussed.

Optical, photographic and scientific equipment

See also Abstract(s): 213341 213376 213386 213463 213527 213575 213585 213597 213666

213424

HIGH LUMEN DENSITY OF Al₂O₃-LuAG:Ce COMPOSITE CERAMIC FOR HIGH-BRIGHTNESS DISPLAYZhang Q; Zheng R; Ding J; Cui P; Wang Z; Lv P; Wei W - *Nanjing, University of Posts & Telecommunications; Nanjing, Institute of Advanced Laser Technology***J.Am.Ceram.Soc.** 104, No.7, 2021, p.3260-3268

Thermally robust and highly efficient green-emitting luminescent ceramics are promising phosphors for use in high-brightness laser phosphor displays to reduce serious speckle noise and high cost. However, lumen density still seriously restricts their potential applications, especially under high-power density laser due to insufficient absorption of blue laser and significant thermal quenching. An Al₂O₃-LuAG:Ce composite ceramic phosphor (CCP) for high-brightness laser phosphor displays was developed. Due to the good optical properties and high thermal conductivity of Al₂O₃, the Al₂O₃-LuAG:Ce CCP had high photoluminescence quantum yield (79.6%), low thermal quenching (only 3.2% loss in luminescence at 200 °C) and high thermal conductivity (18.9 W/m.K). The Al₂O₃, as scattering centres, enhanced the Rayleigh-Mie scattering of the blue laser, and hence the absorption of the Al₂O₃-LuAG:Ce CCP showed a remarkable improvement (about 2.3 times) at 450 nm. With optimised thickness (0.3 mm) of Al₂O₃-LuAG:Ce CCP, an excellent luminous efficiency (216 lm/W) and outstanding lumen density (6129 lm/mm²) of the green-emitting light source was obtained by driving under a high power density (28.33 W/mm²) blue laser. The Al₂O₃-LuAG:Ce CCP is therefore suitable as a green-emitting colour converter for high-brightness display.

213425

FAR-RED-EMITTING YAl₃(BO₃)₄:Cr³⁺ PHOSPHORS WITH EXCELLENT THERMAL STABILITY AND HIGH LUMINESCENT YIELD FOR PLANT GROWTH LEDsShi M; Yao L; Xu J; Liang C; Dong Y; Shao Q - *Nanjing, Southeast University; Jiangsu Bree Optronics Co.Ltd.***J.Am.Ceram.Soc.** 104, No.7, 2021, p.3279-3288

Phosphor-converted LEDs (pc-LEDs) are excellent artificial light sources for indoor plant cultivation, in which the far-red-emitting component (700-780 nm) is important in regulating the photomorphogenesis of plants. Highly efficient and thermally stable far-red-emitting phosphors are thus needed to develop high-performance plant cultivation pc-LEDs. Far-red-emitting YAl₃(BO₃)₄:Cr³⁺ (YAB:Cr³⁺) phosphors were synthesised by solid-state reaction, and their photoluminescence, thermal quenching, quantum yield (QY) and application in pc-LEDs were studied. The YAB:Cr³⁺ phosphor had an intense broadband absorption to the blue light, simultaneously exhibiting the sharp-line 2E emission and the broadband 4 T₂ emission of Cr³⁺ with a QY of about 86.7%. The far-red broadband emissions of YAB:Cr³⁺ centred at about 735 nm were very similar to the active-state absorption of plant phytochrome. The YAB:Cr³⁺ phosphor had thermally enhanced luminescence at 303-393 K and near-zero thermal quenching up to 423 K. The anomalous thermal enhancement was attributed to the temperature-dependent repopulation between 2E and 4 T₂ states. A pc-LED device was fabricated with the YAB:Cr³⁺ phosphor and blue chip, exhibiting light out power of about 50.6 mW and energy conversion efficiency of about 17.4% at 100 mA drive current. The exceptional photoluminescence properties, including suitable excitation/emission wavelengths, suppressed thermal quenching and the high QY made YAB:Cr³⁺ phosphors very promising for applications in plant growth pc-LEDs.

213426

COMPOSITION ENGINEERING OF RED-EMITTING Eu³⁺-DOPED Ca_{10.5}(PO₄)₇-TYPE SOLID SOLUTION PHOSPHORS AND APPLICATION IN LEDWang J; Huang S; Yan Y; Shang M - *Shandong, University; Qingdao, University***J.Am.Ceram.Soc.** 104, No.7, 2021, p.3365-3375

Using Ca_{10.5}(PO₄)₇ as the structural model, a number of Eu³⁺-doped Ca₉Na₃xY_{1-x}(PO₄)₇ (CNYP-I, x = 0-1/2) from Ca_{10.5}(PO₄)₇ to Ca_{9+y}Na_{3/2-y}Y_{1-y}/2(PO₄)₇ (CNYP-II, y = 0-1) phosphors were designed and synthesised through the heterovalent substitution of Y³⁺ and Na⁺ to Ca²⁺. The substitution mechanism, composition structure, luminescence performance and thermal stability of Eu³⁺-doped CNYP-I (x = 0-1/2) and the solid solutions of CNYP-II (y = 0-1), are discussed. The morphology and elemental composition of CNYP-I (x = 0-1/2) and CNYP-II (y = 0-1) solid solutions were analysed by SEM and EDS. The photoluminescence spectra of the specimens contained the predominant red peak of emission at 612 nm caused by the transition of 5D₀-7F₂, indicating that Eu³⁺ occupied the low-symmetry centre. The site symmetry Eu³⁺ occupied changed with the x/y value. The luminous intensity of Eu³⁺-doped CNYP-I (x = 0-1/2) and CNYP-II (y = 0-1) phosphors at 150 °C maintained about 60% of that at room temperature. The representative compound CNYP-I (x = 1/3) was used as the red phosphor to prepare a near-UV based white LED along with Ra of 80.9 and CCT of 4100 K.

213427

EFFECTS OF THE SOLUTION REACTANT CONCENTRATION AND TEMPERATURE ON THE PREPARATION OF Pr³⁺:BaF₂ TRANSPARENT CERAMICSLiu X; Zhou Z; Li W; Yang Y; Mei B - *Wuhan, University of Technology; Guangxi, University of Science & Technology***J.Am.Ceram.Soc.** 104, No.8, 2021, p.3862-3872

BaF₂ crystals are efficient inorganic scintillators with potential for high-energy physics, industrial inspection and other fields due to the fast component of the decay time (0.6 ns) and high radiation resistance. However, there are two major drawbacks which limit its practical application: a slow decay time of about 600 ns is derived from self-trapping excitons; and the absolute light yield from the fast luminescence component is not competitive. The introduction of rare earth ions and preparation of BaF₂ polycrystalline ceramics can help to solve these problems. Pr³⁺ is very suitable as the activated ion of scintillation materials, which has emission peaks in the visible band and faster 5d-4f transition. Highly sinterable Pr³⁺:BaF₂ precursor powder was synthesised by coprecipitation by adjusting the reactant concentration and temperature. The morphology and microstructure of as-synthesised powders were characterised using SEM and TEM. A 5 at% Pr³⁺:BaF₂ transparent ceramic with a transmittance of 50.7% at a wavelength of 500 nm was fabricated by hot pressing the as-prepared powders at 900 C for 4 h under an axial pressure of 50 MPa. SEM images of the ceramic cross-section showed that the residual pore was the main light scattering source. The absorption and emission spectrum of the ceramic samples are discussed.

213428

ENHANCED LUMINESCENCE AND THERMAL STABILITY OF (Sr,Ca)AlSiN₃:Eu²⁺ VIA SUPERFICIAL ORGANIC CARBON MODIFICATION

Tian J; Zhuang W; Liu R; Liu Y; Chen G; Chen S; Jiang Z - *GRINM Group Co.Ltd.; Griem Advanced Materials Co.Ltd.; Beijing, General Res.Inst.Non-ferrous Metals; Beijing, University of Science & Technology*

J.Am.Ceram.Soc. 104, No.8, 2021, p.4014-4023

Eu²⁺-activated nitride phosphors are widely used in solid-state lighting, but applications in high-power white-light-emitting diodes (wLEDs) require higher thermal stability of the luminescent materials. Luminescence loss during operation is due to the oxidation of Eu²⁺ and the damage of the nitride host in the Eu²⁺-activated nitride phosphors. A superficial organic carbon modification was carried out on the red-emitting (Sr,Ca)AlSiN₃:Eu²⁺ phosphor via the incorporation of organic carbon by solution mixing and thermal post-treatment under an N₂-H₂ atmosphere. After the modification, the oxidation of Eu²⁺ and the formation of impurity phases on the phosphor surface were effectively reduced. When the superficial organic carbon modified sample was treated in 2 wt% sucrose solutions, the relative brightness was strengthened by 2.15%, the thermal quenching characteristic was improved by 8.9% at 300 C and ageing test results showed excellent thermal stability. The superficial organic carbon modification is therefore a promising technique to enhance the thermal stability of analogous Eu²⁺-activated nitride phosphors.

213429

CHARGE TRANSFER BAND EXCITATION OF La₃NbO₇:Sm³⁺ PHOSPHORS INDUCED ABNORMAL THERMAL QUENCHING TOWARD HIGH-SENSITIVITY THERMOMETERS

Hua Y; Kim J-U; Yu J S - *Kyung Hee, University*

J.Am.Ceram.Soc. 104, No.8, 2021, p.4065-4074

Different luminescent behaviour of La₃NbO₇:Sm³⁺ phosphors under the excitation of a charge transfer band (CTB, 250 nm) and the featured absorption peak (6H_{5/2} to 4H_{7/2}, 405 nm) of Sm³⁺ ions was demonstrated. Under an excitation wavelength of 405 nm, the optimal La₃NbO₇:0.1Sm³⁺ phosphor showed an orange-red emission, while the chromatic coordinate was (0.609, 0.387), which also showed the excellent thermal performance, exhibiting an emission intensity of about 90.67% at 423 K with respect to 303 K. In the case of CTB excitation, the La₃NbO₇:0.1Sm³⁺ phosphor emitted an orange-yellow region with the chromaticity coordinate of (0.540, 0.443), and the emission intensity was stronger than that above (λ_{ex} = 405 nm) even though the optimised sample would be changed to the La₃NbO₇:0.05Sm³⁺ phosphor. With increasing temperature, the sample had an abnormal thermal quenching phenomenon between the emission peak of the host material and the emission transition of 4G_{5/2} to 6H_{9/2} under an excitation wavelength of 250 nm, which could be suggested to turn into a pair of thermal-couple levels. The sensing sensitivity of the obtained sample was further investigated based on fluorescence intensity ratio theory. The absolute and relative sensing sensitivities of the La₃NbO₇:0.01Sm³⁺ phosphor were estimated to be as high as 5.379 × 10 exp(-2)/K and 1.60%/K, respectively.

213430

EFFICIENT RED AND BROADBAND NEAR-INFRARED LUMINESCENCE IN Mn²⁺/Yb³⁺-DOPED PHOSPHATE PHOSPHOR

Dong L; Zhang L; Xu Y; Yin S; You H - *Changchun, Institute of Applied Chemistry; China, University of Science & Technology*

J.Am.Ceram.Soc. 104, No.8, 2021, p.4109-4118

A novel phosphor Li₃Sc₂(PO₄)₃ (LSPO):Mn²⁺ exhibiting red emission at about 616 nm and satisfactory broad NIR emission at about 800 nm with a FWHM of 112 nm is reported. The structure and spectra showed that the doped manganese ions occupied two kinds of Sc sites forming Mn1 and Mn2 emission centres, which were responsible for red and NIR emission, respectively. XPS and low-temperature fluorescence spectra revealed that both red and NIR emissions came from the Mn²⁺ ions. NIR luminescence was improved by doping Yb³⁺ in LSPO:Mn²⁺, leading to the broadened NIR emission range (700-1100 nm) and enhanced luminescent thermal stability. The results suggest that the prepared LSPO:Mn²⁺ and LSPO:Mn²⁺, Yb³⁺ phosphors have potential applications as red and NIR components in phosphor-converted white-light-emitting diodes (pc-WLED) and broadband NIR pc-LED. A new way to design novel broadband NIR phosphors is also given.

Insulation (non-electrical)

See also Abstract(s): 213301 213310 213325 213620 213639

213431

FABRICATION AND CHARACTERISATION OF A NOVEL FOAM CERAMIC MATERIAL BASED ON COAL GASIFICATION SLAGS

Yuan H; Yin H; Tang Y; Xin Y; Pu X; Zhang H - *Xian, University of Architecture & Technology*

J.Ceram.Process.Res. 21, No.6, 2020, p.640-646

Mixtures of coal gasification slag and clay, containing 15-22% clay, and 3% SiC as a foaming agent, were prepared by planetary ball milling in aqueous media. Granules were formed by spray granulation and sieved into three particle size distributions: 1.4-2.36 mm; 1.0-1.4 mm; and 0.25-1 mm. The materials were characterised by TGA and DSC. The specimens were then heated to various temperatures in the range 1140-1160 C at a rate of 5C/min. The sintered specimens were characterised by microstructural studies, and by measurements of density, compressive strength, and phase composition. A foaming mechanism is proposed, based on the oxidation of SiC, and involving reaction with residual carbon, the formation of a protective shell of SiO₂, and the subsequent degradation of the shell by a liquid phase. It was established that the optimum particle size range was 0.25-1 mm, with specimens containing 77% slag and 20% clay, sintered at 1160 C, having a density of 0.21 g/cm³, a thermal conductivity of 0.05

W/m.K and a compressive strength of 1.18 MPa. The prepared materials are concluded to be suitable for the fabrication of insulation board for construction applications.

Building parts, structures, flooring and roofing

See also Abstract(s): 213299 213305

213432

FULL-SCALE MODELS FOR THE STUDY OF REPAIR TECHNIQUES ON BRICK MASONRY SURFACES

Binda L; Baronio G; Cantoni F; Rocca P - *Milan, Polytechnic; CNR-ICITE*

Ceram.Modern Technol. 3, No.1, 2021, p.10-15

A systematic approach developed by the authors for studying the durability of wall surface treatments, based on accelerated ageing tests and subsequent measurement of the surface deterioration of treated and untreated specimens, has been extended to full-scale facades. Three small constructions designed by them, consisting of modular facades made of sandstone and/or soft-mud facing bricks, was built in the open. A continuous content of water in the subsoil can be provided so that the capillary rise into the masonry is assured; soluble salts can also be provided from the soil. Some of the facades were treated with protective materials while others were treated after deterioration. Environmental data and data relating to moisture and salt movement in the walls were collected. Deterioration of the external surfaces and the influence of mortar joints on moisture distribution and of the position and exposure of the material on the deterioration were taken into account. Comparisons were made between in-situ and laboratory results in order to adjust the ageing test conditions to the natural environment.

Medical, dental and veterinary application

See also Abstract(s): 213614

213433

SIMPLE, LOW COST, AND TEMPLATE-FREE METHOD FOR SYNTHESIS OF BORON NITRIDE USING DIFFERENT PRECURSORS

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Ceram.Int. 47, No.5, 2021, p.5977-5984

Hexagonal boron nitride (h-BN) was synthesised using a simple, low cost and template-free method with urea-boric acid (UB), melamine-boric acid (MB), and melamine-urea and boric acid (MUB) precursors, followed by the pyrolysis and heat treatment in a nitrogen atmosphere at 1050 C. Samples were characterised using XRD, FE-SEM, XPS, Raman Spectroscopy, FTIR, and BET techniques. The specific surface areas obtained for h-BN synthesised by UB, MB, and MUB precursors were 87.43, 573.07, and 1005.7 m²/g, respectively. The average diameters of the pores, using the Barrett, Joyner, and Halenda (BJH) model, were 37.78, 3.68, and 2.13 nm, respectively. TGA showed a wider range of decomposition temperatures after using three precursors for the synthesis of h-BN. Crumpled, whisker, and flower-like morphologies for UB, MB, and MUB precursors were respectively found using FE-SEM. The formation of h-BN within the MUB sample was confirmed by XPS analysis with measured peaks of 398.5 and 190.6 eV belonging to N 1s and B 1s, respectively. Raman spectroscopy revealed a high-intensity peak at 1366 cm⁻¹ related to the E_{2g} mode for h-BN synthesised with MUB. Results showed that the method used could increase the potential use of h-BN porous powder, with a high specific surface area, as a lubricant, thermal insulation filler, anti-corrosion filler in paint coatings, adsorbent for various gas and hydrocarbon molecules and as drug-delivery nanocarriers.

213434

PHOTOTHERMAL EFFECT OF 3D PRINTED HYDROXYAPATITE COMPOSITE SCAFFOLDS INCORPORATED WITH GRAPHENE NANOPATELETS

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Ceram.Int. 47, No.5, 2021, p.6336-6340

Graphene nanoplatelets were incorporated into apatite/gelatin composites to construct porous scaffolds using 3-D printing. Under near infrared laser irradiation, the composite scaffolds showed high photothermal conversion efficiency. The temperature of the scaffolds could be increased to 43 C by controlling the time and power of the laser irradiation, and then subsequently cooled to room temperature. Mild photothermal treatment (40-43 C) was applied to MC3T3-E1 cells cultured on the scaffolds and after 3 cycles of treatment the proliferation of MC3T3-E1 cells was significantly accelerated. It was suggested that the incorporation of graphene nanoplatelets into 3-D printed hydroxyapatite composite scaffolds could have the potential to accelerate bone regeneration after photothermal treatment for malignant bone tumours.

213435

MECHANICAL AND TRIBOLOGICAL ASSESSMENT OF COMPOSITE AlCrN OR a-C:Ag-BASED THIN FILMS FOR IMPLANT APPLICATION

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Ceram.Int. 47, No.5, 2021, p.6736-6752

A comparison of cathodic arc deposited AlCrN (ternary coating) and Ag alloyed a-C (amorphous carbon base coating), on chrome nitride (CrN) medical grade 316 LVM stainless steel, is reported. The morphological, structural, nanomechanical and tribological properties in physiological simulated body fluid (SBF) lubrication, following conditions pertaining to a simulated hip joint, were investigated. Results showed that H/E, H₃/E₂ and Ecoating/Esubstrate affected the nanomechanical and tribological properties of the coatings. While a-C:Ag/CrN exhibited a better Ly value compared to AlCrN/CrN due to the improved surface quality, the latter had a higher Lc₂ value during nanoscratch testing due to a lower H₃/E₂ and higher plastic work done. In spite of a lower friction coefficient, a-C:Ag/CrN had a higher wear rate during simulated tribotests attributed to low hardness, separate graphitic structure due to Ag doping and a sudden increase in the friction coefficient ascribed to severe abrasive delamination of the a-C:Ag top layer. The wear mechanism observed by SEM indicated severe adhesion of the Ti6Al4V counterbody on the AlCrN/CrN coated surface. The size of the wear debris obtained using the AlCrN/CrN-Ti6Al4V tribopair was larger compared to a-C:Ag/CrN-Ti6Al4V tribopair. Despite inferior surface quality and lower Ly value and larger wear debris size, the AlCrN/CrN coating performed better in nanoscratch tests (at Lc₂ value) and had reduced wear in a simulated tribotest in physiological SBF conditions.

213436

BIOCOMPATIBILITY AND CYCLIC FATIGUE RESPONSE OF SURFACE ENGINEERED Ti6Al4V FEMORAL HEADS FOR HIP-IMPLANT APPLICATION

Samanta A; Rane R; Jhala G; Kundu B; Datta S; Ghosh J; Joseph A; Mukherjee S; Roy S; Mukhopadhyay A K - *Gandhinagar, Institute for Plasma Research; Central Glass & Ceramic Research Institute; Kattankulathur, SRM Institute of Science & Technology; Manipal, University*
Ceram.Int. 47, No.5, 2021, p.6905-6917

The development of a wear, corrosion and fatigue resistant Ti6Al4V alloy based femoral head (FH) using a duplex surface engineering (DSE) technique is reported. The DSE based FHs were developed by a novel combination of plasma nitridation (PN) and a Ti/TiN multilayer coating (MLC) deposited by magnetron sputtering. The Ti6Al4V based FHs were denoted as Ti and the plasma nitrided FHs were TiPN. The DSE based FHs were denoted as TiPNML. The corrosion resistance was studied using Hank's solution and the sliding wear resistance was studied in simulated body fluid (SBF). The biocompatibility was assessed using the standard MTT assay technique and the cyclic fatigue resistance behaviour, up to one million walking cycles, was studied in SBF in a HIP simulator with UHMWPE acetabular cups used as the counter bodies in the articulation. Results revealed that the performance of the TiPNML and TiPN FHs was better than that of the Ti based FHs due to microstructural factors, wear mechanisms and surface roughness. The implications of the current results in terms of futuristic FH developments for hip implant applications are discussed.

213437

LOW-TEMPERATURE FABRICATION OF CALCIUM DEFICIENT HYDROXYAPATITE BONE SCAFFOLD BY OPTIMISATION OF 3D PRINTING CONDITIONS

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Ceram.Int. 47, No.5, 2021, p.7005-7016

A novel low-temperature fabrication process for calcium-deficient hydroxyapatite (CDHA) 3-D scaffolds, for bone tissue regeneration, using a combination of material extrusion type 3-D printing process and bone cement chemistry. The CDHA scaffolds with a high porosity of 70% had excellent mechanical properties with 25 MPa compressive strength, without sintering after 3-D printing. Using this method, the final structures did not show any shrinkage, which must be considered in conventional ceramic sintering processes. Therefore, both size and 3-D architecture-controlled porous CDHA scaffolds could be fabricated with high accuracy. The resulting ceramic cement scaffolds were not brittle and could be machined without chipping or fracturing the scaffold. The complete process took place under physiological conditions (37 C, pH = 7), and heat sensitive drugs and biomolecules could be directly loaded onto the raw powder and achieve homogeneous distribution throughout the CDHA scaffold. Various key factors to complete the low-temperature 3-D printing fabrication of calcium phosphate, including the pre-treatment conditions of the particles, liquid-to-powder ratio, relationship between strut sizes, infill of structures, setting temperatures and the properties of 3-D printed scaffolds are discussed. The low-temperature fabrication of calcium phosphate scaffolds developed in this study has potential use in bone tissue engineering.

213438

MIXED Zn-Ni SPINEL FERRITES: STRUCTURE, MAGNETIC HYPERTHERMIA AND PHOTOCATALYTIC PROPERTIES

Manohar A; Chintagumpala K; Kim K H - *Yeungnam, University; Vellore, Institute of Technology*
Ceram.Int. 47, No.5, 2021, p.7052-7061

The synthesis of narrow size distributed, small diameter, high-quality Zn_{1-x}Ni_xFe₂O₄ (x = 0.1, 0.3 and 0.5) nanoparticles, with increased saturation mass magnetisation for localised superparamagnetic hyperthermia and photocatalysis applications, is reported. Narrow size distributed samples, with about 10 nm average diameter, were synthesised in the presence of a surfactant by the solvothermal reflux method. All the samples showed good colloidal stability through electrostatic stabilisation with zeta potentials of about -60 mV. The heat capacity or specific heat generation rate (SHR) of the synthesised superparamagnetic nanoparticles was determined by calorimetry. The x = 0.1, 0.3 and 0.5 samples had a SHR = 372, 399 and 410 W g⁻¹ of nanoparticles, respectively, under 35.28 kA/m and 316 kHz field parameters. The SHR values were higher than those of samples synthesised by other methods. Similarly, the saturation mass magnetisation value increased with the Ni²⁺ concentration which was attributed to the preferential octahedral ligand site occupation by Ni²⁺ ions in the spinel lattice due to high crystal field stabilisation energy. The photocatalytic activity of all the nanoparticles was studied for methylene blue (MB) dye degradation under visible light. Data showed that the degradation efficiency increased with the Ni²⁺ concentration. All the samples were characterised using XRD, TEM, FTIR, zeta potential distribution curve, TGA, vibrating sample magnetometer (VSM), transient temperature curves, and UV-VIS-NIR photo-absorption spectra.

213439

EFFECT OF BaTiO₃ TEMPLATES ON THE ELECTRICAL CHARACTERISTICS OF (Ba, Ca)(Ti, Sn, Hf)-BASED CERAMICS UNDER THE REDUCING ATMOSPHERE FOR ACTUATOR APPLICATIONS

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Ceram.Int. 47, No.5, 2021, p.7207-7217

A cylindrical rotary linear piezoelectric motor was fabricated using a textured (Ba_{0.968}Ca_{0.032})(Ti_{0.96}Sn_{0.005}Hf_{0.035})O₃ lead-free piezoelectric plate as the motor vibrator in the first bending vibration mode. The d₃₁ and Q_m values of the proposed ceramics were the key factors affecting the ultrasonic motors. The tape casting method was used to prepare textured (Ba_{0.968}Ca_{0.032})(Ti_{0.96}Sn_{0.005}Hf_{0.035})O₃ + 2 mol% MnCO₃ + 2 mol% Li₂CO₃ (BCTSHML) ceramics at 1280 C under 10 exp(-7) atm oxygen pressure. The maximum figure of merit for the -d₃₁ x Q_m value occurred with the addition of a 3 wt% BT textured template. Values of -d₃₁ = 137 pC/N, Q_m = 426 for the proposed samples were obtained. The dynamic characteristics of the linear piezoelectric motors based on the proposed samples were also studied. The output characteristics of the fabricated devices had a left-pull velocity = 2.38 mm/s, a right-pull velocity = 2.03 mm/s and a thrust = 2.37 N at 63.6 kHz for an applied voltage of 200 Vp-p. The fatigue behaviour of the P-E measurements for the BT template-doped BCTSHML ceramics, after 105 bipolar cycles, were also shown. The polarisation deviation achieved the smallest change rate of 9.1% for the samples with 3 wt% BaTiO₃ templates. To suitably inject insulin in diabetes patients, the modified piezoelectric motor could launch a small amount of insulin aqueous solution in a limited time with limited linearity error (<2.3%). This is the first reported result on the use of textured lead-free piezoceramics under a reduced atmosphere with sintering temperatures <1300 C for high-power piezoelectric devices.

213440
LASER SURFACE TEXTURING OF CERAMICS AND CERAMIC COMPOSITE MATERIALS - A REVIEW
 de Zanet A; Casalegno V; Salvo M - *Turin, Polytechnic*
Ceram.Int. 47, No. 6, 2021, p. 7307-7320
 This review provides an overview of most of the technological aspects considered relevant for the laser surface texturing of ceramics and CMCs, and includes the fundamentals of laser-material interactions and a summary of the equipment used and their parameters. Furthermore, most of the techniques related to the modifications of surface material induced by a laser are reviewed and the new horizons that are opening up, in the context of the modification of surfaces to improve the performance of materials for several applications, are discussed.

213441
FEA EVALUATION OF MATERIAL STIFFNESS CHANGES FOR A POLYMER ASSISTED 3D POLYCAPROLACTONE/BETA-TRICALCIUM PHOSPHATE SCAFFOLD IN A MANDIBULAR DEFECT RECONSTRUCTION MODEL
 Lowe B; Huotilainen E; Laitinen M; Henell A-M; Ye Q; Troulis M J; Walsh L J - *Queensland, University; MASSACHUSETTS GENERAL HOSPITAL; Harvard School of Dental Medicine; Disior Ltd.*
Ceram.Int. 47, No. 6, 2021, p. 8075-8081
 A novel polymer-assisted method was used to fabricate a three-dimensional (3D) polycaprolactone/beta-tricalcium phosphate scaffold. The compressive modulus was measured and the data was used for finite element analysis. The scaffolds achieved a maximum compressive modulus of 151 MPa, close to that of cortical bone. Further computational simulations were performed to determine the stresses and local scaffold adaptation profile, using data from computed tomography scans of the mandible. Local stresses were simulated based on the density changes in the new bone forming in the scaffold at different stages of healing. The stress distribution in the mandible, scaffold centre and scaffold interface were assessed for a static load of 200 N, which corresponded to the load during adult mastication near the incisors. Analysis revealed that the maximum cross-sectional stress at the scaffold centre and at the scaffold interface were 2.7 and 4.12 MPa respectively. The majority of the stress was localised in the bone of the mandible, with the scaffold bearing minimal loading at the start, but more over time as infiltration of more new bone progressed.

213442
SUITABILITY OF BIOSILICATE GLASS-CERAMIC POWDER FOR ADDITIVE MANUFACTURING OF HIGHLY POROUS SCAFFOLDS
 Elsayed H; Colombo P; Crovace M C; Zanutto E D; Bernardo E - *Padua, University; Cairo, National Research Centre; Pennsylvania, State University; Sao Carlos, Federal University*
Ceram.Int. 47, No. 6, 2021, p. 8200-8207
 The suitability of Biosilicate for the manufacture of highly porous scaffolds (porosity of 50-80 vol%) using additive manufacturing technologies, including direct ink writing (DIW) and digital light processing (DLP), was investigated. Both techniques could be applied to fine powders of Biosilicate mixed with fugitive binders. Significant densification of the struts, despite limited powder packing, could be achieved using liquid-assisted sintering which was triggered by the phosphate-enriched residual glass phase already at 1000 C. The strength-to-density ratio could be tuned (from 1.5 to 9.5 MPa cm³/g), especially using DLP-derived samples, by adjusting both the firing temperature and the scaffold topology.

213443
EFFECT OF B2O3 ON THE STRUCTURAL AND IN VITRO BIOLOGICAL ASSESSMENT OF MESOPOROUS BIOACTIVE GLASS NANOSPHERES
 Bai N; Chen W; Luo L; Tong W; Wen C; Zhan X; Sa B - *Fuzhou, University; Fujian, Medical University; Fujian Institute of Microbiology*
J.Am.Ceram.Soc. 104, No. 7, 2021, p. 3058-3072
 Boron-containing mesoporous bioactive glass (MBG) nanospheres were synthesised by a modified sol-gel method assisted by surfactants (CTAB combined with ethyl acetate), and the effect of boron substitution on the structure and bioactivity was evaluated by combining experiments and ab initio molecular dynamics (AIMD) simulations. All the samples had a regularly uniform mesoporous spherical microstructure with an average size of about 60 nm, and the boron-containing MBGs had higher specific surface areas with values up to 416.20 m²/g. Simulated body fluid immersion tests confirmed that the deposited hydroxyapatite clearly increased with increasing boron content, indicating that the biological behaviour was significantly improved, as a result of the incorporation of boron. The results also showed that B₂O₃ substitution had a positive impact on the cell proliferation of human periodontal ligament cells at lower extracted concentration. AIMD simulation was used to understand the relationship between the structural changes and in-vitro bioactivity in terms of structural information, especially the boron coordination number. The results showed that the boron-containing MBG nanospheres with excellent bioactivity have great potential for biomedical applications.

213444
RELATIONSHIP BETWEEN VALENCE OF TITANIA AND APATITE MINERALISATION BEHAVIOUR IN SIMULATED BODY ENVIRONMENT
 Miyazaki T; Imanaka S; Akaike J - *Kyushu, Institute of Technology*
J.Am.Ceram.Soc. 104, No. 7, 2021, p. 3545-3553
 Titania-based materials are attractive for hard tissue repair due to their bone-bonding ability induced by apatite formation in the body environment. Various surface treatments have therefore been developed to produce a hydrated titania layer on Ti and its alloys. Titania takes various valences, such as TiO (Ti²⁺) and Ti₂O₃ (Ti³⁺), as well as typical TiO₂ (Ti⁴⁺), but there is no comprehensive study of structural effects on the apatite-forming ability of these titanias. Apatite formation on titania powders with various valences was studied in simulated body fluid. Anatase- and rutile-type TiO₂ formed apatite in simulated body fluid within 7 days, but TiO and Ti₂O₃ did not. In contrast, when the titania powders were treated with NaOH solution, the surface converted to tetravalent titania and all samples formed apatite. It is proposed that the surface electrical states of TiO and Ti₂O₃ are strongly affected by their bulk conductivity and that these behaved like pure Ti metal, which has poor apatite-forming ability. Apatite formation was favourable when the titania had a high absolute value and showed high fluctuations of zeta potential during initial stages in simulated body fluid, due to adsorption of large amounts of Ca²⁺ and HPO₄(²⁻).

213445
HYDRAULIC REACTIVITY AND CEMENT FORMATION OF BAGHDADITE
 Holzmeister I; Weichhold J; Groll J; Zreiqat H; Gbureck U - *Wurzburg, University; Sydney, University*
J.Am.Ceram.Soc. 104, No. 7, 2021, p. 3554-3561

Baghdadite ($\text{Ca}_3\text{ZrSi}_2\text{O}_9$) has application as a biomaterial. The hydraulic reactivity and cement formation of baghdadite was studied. The material was synthesised by sintering a mixture of CaCO_3 , SiO_2 and ZrO_2 which was then mechanically activated by planetary milling. This led to a decrease in particle and crystallite size and a partial amorphisation of baghdadite, as shown by XRD and laser diffraction measurements. Baghdadite cements were formed by the addition of water at a powder to liquid ratio of 2.0 g/ml. Maximum compressive strengths were about 2 MPa after 3-day setting for a 24-h ground material. ICP-mass spectrometry measurements showed an incongruent dissolution profile of the set cements with a preferred dissolution of calcium and only marginal release of zirconium ions. Cement formation occurred under alkaline conditions, whereas the unground raw powder led to a pH of 11.9 during setting, while prolonged grinding increased pH values to about 12.3. Baghdadite cements were shown to have potential either in bone replacement or, due to the inherent X-ray opacity, as endodontic fillers.

213446

COMPARATIVE STUDY ON PHYSICO-MECHANICAL AND BIOACTIVITY PROPERTIES OF BETA-WOLLASTONITE DERIVED FROM RICE HUSK ASH AND CALCINED LIMESTONE DRYING THROUGH FREEZE-DRIED AND INCUBATOR TECHNIQUE

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J.Aust.Ceram.Soc. 57, No.3, 2021, p.755-766

Beta-wollastonite powder was synthesised by the solid-state route using rice husk ash and calcined limestone as precursors at a SiO_2 :CaO ratio of 45:55. The resulting mixture was autoclaved and sintered at 950 C for 2 h to yield single-phase beta-wollastonite. The beta-wollastonite powder was mixed with a phosphate buffer at a liquid-to-powder ratio of 0.5. The samples were dried using two different techniques, viz. freeze-drying at -40 C for 12 h or incubation in an incubator at body temperature (36.7 C) for 48 h. The physical and mechanical properties were determined and the in-vitro bioactivity was assessed by immersion in SBF. The density increased from 3.02 to 3.12 g/cm³ while the porosity decreased from 63.9 to 56.1% for samples dried in an incubator and for freeze-dried samples, respectively. The compressive strength of freeze-dried samples (0.83 plus or minus 0.05 MPa) was higher than that of incubated samples (0.69 plus or minus 0.13 MPa). SEM and EDX revealed the presence of hydroxyapatite-like microstructures for both samples. After 21 days of soaking, amorphous calcium phosphate and calcium-deficient hydroxyapatite were formed, with the molar ratio ranging between 1.63 and 1.76 for both samples. Thus, freeze-dried beta-wollastonite has good density and compressive strength but poor bioactivity.

213447

ANTIFUNGAL EFFECTS OF ZnO-TiO₂/Au NANOSTRUCTURES ON ASPERGILLUS FLAVUS

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J.Aust.Ceram.Soc. 57, No.3, 2021, p.793-802

An attempt was made to synthesise ZnO-TiO₂/Au (Zn/Ti weight ratio of 1/1; Au concentration of 5-15 wt%) nanostructures using zinc acetate, titanium isopropoxide and tetrachloroauric acid via the sol-gel route. Physicochemical in-vitro antifungal properties along with morphological characterisation were investigated by MIC (minimal inhibitory concentrations), MFC (minimal fungicidal concentrations) and ROS (reactive oxygen species) tests. ZnO-TiO₂/15 wt% Au showed higher antifungal activity against *A. flavus* (MIC = 0.625 micrograms/ml) than ZnO-TiO₂ alone (MIC = 78 micrograms/ml). Based on the findings, all ZnO-TiO₂/Au samples showed higher ROS production, leading to the induction of oxidative stress which was the reason for the higher fungicidal effect with Au-containing samples. It was concluded that the Au-doped samples with enhanced chemical reactivity offer great potential as a fungicide in clinical applications.

213448

IN VITRO STUDY OF FLUORIDE RELEASE OF CALCIUM FLUORIDE-PORCINE BONE-DERIVED HYDROXYAPATITE COMPOSITE

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J.Aust.Ceram.Soc. 57, No.3, 2021, p.803-808

Fluoride-releasing materials are helpful in the remineralisation of enamel in teeth damaged by erosion due to caries. They include glass ionomers, composites and amalgams, and are usually incorporated with filler materials. A composite with the potential to be a fluoride-releasing component in dental materials was synthesised using two biocompatible components, viz. calcium fluoride (CaF₂) and porcine bone-derived hydroxyapatite (HAp). The synthesised material was characterised based on its fluoride release in water. The HAp was obtained from porcine bones by calcination and mixed with CaF₂ by manual grinding. The resulting powder mixture was pelletised and sintered at 900 C to form a disc-shaped green compact. FTIR analysis of the pellet showed that the HAp and CaF₂ formed fluorapatite (FA), given the absence of OH peaks. The XRD pattern confirmed that a proportion of HAp transformed into FA and that there are three phases (HAp, FA and CaF₂) present in the pellet. Immersion tests showed that the line of the best-fit model describing the amount of fluoride released over time is a linear relationship. It was also found that there is an initial high release of fluoride due to surface wash-off. Furthermore, the synthesised pellets were hydrolytically stable due to the very small weight loss seen even after 16 days of immersion. With the capability to release fluoride and its stability, the synthesised material has the potential to be a cheap alternative material in dental applications.

213449

HEPARIN AS A BIOMIMETIC TEMPLATE ON NANOAPATITE RODS WITH TUNABLE ASPECT RATIO: SYNTHESIS AND BIOCOMPATIBILITY

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J.Aust.Ceram.Soc. 57, No.3, 2021, p.825-834

Heparin was used as a natural organic template to prepare nanoscale needle- or rod-like (nanorod) carbonate-based apatite. The characteristic phase and SAED patterns confirmed that the nanorods grew in the preferred crystallographic orientation, i.e., the [0001] direction of the c-axis of apatite. The morphological variations indicated that the heparin was potentially incorporated with the Ca²⁺ ions, thereby inhibiting a-axis growth, increasing the aspect length-to-width ratio of the apatite nanorods, and effectively changing the precipitate form into uniform nanoscale rod-like shaped apatite. FTIR spectra showed that the hexagonal structure template mediated by heparin was B-type carbonated calcium-deficient hydroxyapatite (CDHA), indicating that heparin regulated growth in the processed template of carbonated CDHA. Cell viability analysis showed that cytotoxicity was caused by the influence of specific nanoscale aspect ratios of the rod-like apatite morphology or by tuning a large aspect ratio of rod-like shaped apatite with a large quantity of heparin incorporation, resulting in rapid ion dissolution by rapidly raising the pH. Overdosed heparin used as a template to prepare apatite nanoparticles led to highly negatively charged nanorods that caused cytotoxicity. The optimal group of synthetic HAp@Hep25K within the proper heparin concentration, showing the intended morphology of apatite nanorods and exhibiting

biocompatibility, is proposed.

213450

EFFECT OF MgO ADDITION ON THE SINTERABILITY, MECHANICAL PROPERTIES AND BIOLOGICAL CELL ACTIVITIES OF SINTERED SILICON-SUBSTITUTED HYDROXYAPATITE

Le B T; Ramesh S; Long B D; Son N A; Munar M; Shi X - *Hanoi, University of Science & Technology; Malaysia, University; Brunei, University of Technology; Kyushu, University; Jiangsu, University of Science & Technology*

J.Aust.Ceram.Soc. 57, No.3, 2021, p.857-868

The effect of 1 wt% MgO as a sintering aid on the sintering behaviour, microstructure, mechanical properties and bioactivities of 0.4 and 1.6 wt% silicon-substituted hydroxyapatite (MgO-0.4Si-HAp and MgO-1.6Si-HAp) was studied. The powders were synthesised by the wet chemical route and compacts were pressureless sintered in air between 1150 and 1250 C for 2 h. It was shown that MgO-0.4Si-HAp promoted the formation of the secondary phase, beta-TCP, when sintered at 1250 C, whilst the stability of the hydroxyapatite phase was not disrupted for the MgO-1.6Si-HAp. MgO was beneficial in suppressing grain growth of Si-HAp when sintered at 1250 C; however, it did not promote densification at lower temperatures (1150 and 1200 C). It was seen that, despite the lower densification, the Vickers hardness and compressive strength of the MgO-Si-HAp were higher than those of undoped Si-HAp samples. The beneficial effect of MgO in enhancing the biocompatibility of Si-HAp was shown; MgO-doped samples dramatically enhanced the osteogenetic differentiation of MC3T3-E1 cells, as shown by; the higher cell proliferation, alkaline phosphatase activity and bone-like nodule formation when compared to pristine HAp.

213451

Zn-DOPED HYDROXYAPATITE IN BIOMEDICAL APPLICATIONS

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J.Aust.Ceram.Soc. 57, No.3, 2021, p.869-897

The structure of hydroxyapatite ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$; HAp) enables ionic substitutions that change its characteristics, especially in terms of biological, antibacterial and mechanical properties. Zinc, an essential trace element in the body, is an important dopant for biomedical applications due to its positive effect on the biological and antibacterial properties of HAp. Thus, Zn-doped HAp has been extensively studied as a coating material as well as a constituent of composites and tissue engineering scaffolds. This review summarises the synthesis methods, sintering parameters and morphology of crystals formed in Zn-doped HAp. The lattice parameters, crystal size and phase composition and specific FTIR bands detected in Zn-doped HAp were determined. The proliferation and differentiation of stem cells and the functional activities of osteoblastic cells were generally shown to be enhanced on Zn-doped HAp when compared with pure HAp. Since the number of studies on the mechanical properties of Zn-doped HAp is limited, more studies will be needed in the future. The co-doping of Zn^{2+} with Ag^+ , Sr^{2+} , Mg^{2+} , Cu^{2+} , Fe^{3+} , Si^{2+} , Zr^{4+} , F^- , Cl^- , CO_3^{2-} , SeO_3^{2-} and SiO_4^{4-} ions into the structure of HAp was covered. Computational studies to predict the locations of substitution of Zn^{2+} and mechanical properties such as bulk modulus, shear modulus, Young's modulus and Poisson's ratio were also discussed.

213452

BIOACTIVE GLASS-CERAMIC SYNTHESIS USING NANO AND NORMAL SILICA: A COMPARATIVE STUDY

Ghosh T K; Das S K - *Calcutta, University*

J.Aust.Ceram.Soc. 57, No.3, 2021, p.933-946

Bioactive glass-ceramics are promising materials for soft and hard tissue repair due to their high biocompatibility and bioactivity. Bioactive glass-ceramics in the $(38-X)\text{normal-SiO}_2\text{-}28\text{CaO-}18\text{Al}_2\text{O}_3\text{-}12\text{CaF}_2\text{-}4\text{P}_2\text{O}_5\text{-}X\text{nano-SiO}_2$ (mol%) system, where $X = 0, 19$ or 38 , were synthesised by melt quenching. Their thermal behaviour was assessed by DTA. The glass transition temperature (T_g) was high in the case of a mixture of 19 mol% normal/19 mol% nano-SiO₂. However, it was lower in 38 mol% normal or 38 mol% nano-SiO₂ samples. As the nano-SiO₂ content increased, the peak crystallisation temperature (T_p) increased. XRD showed that fluorapatite, mullite and wollastonite were the main crystalline phases present in all three batches. The crystals were needle-like and irregular shaped in all sintered specimens as shown by SEM. Surface cracks were evident in the case of high sintering temperatures. The elemental composition was analysed by EDX before and after immersion in SBF solution. FTIR spectra showed the characteristic peaks for Si-O-Si, Si-O-Ca, C-O stretching vibration, O-H bending vibration, carbonate, and phosphate groups. TEM confirmed the presence of nanoparticles (2.58-8.15 nm) in the prepared glass-ceramics. A hydroxycarbonate apatite (HCA) layer formed after 28 days immersion in modified SBF. Nano-SiO₂ addition led to an increase in density, Vickers hardness and acid-base resistance.

213453

FABRICATION, CHARACTERISATION, AND IN VITRO BIOACTIVITY EVALUATION OF FREEZE-CAST HIGHLY POROUS HARDYSTONITE CERAMIC REINFORCED BY GRAPHENE OXIDE AS A NOVEL BONE SCAFFOLD

Azizi M; Kalantar M; Nezafati N; Zamanian A - *Yazd, University; Karaj, Materials & Energy Research Centre*

J.Aust.Ceram.Soc. 57, No.3, 2021, p.947-960

A major challenge in bone tissue engineering is the preparation of highly interconnected porous scaffolds with suitable mechanical properties. Synthetic scaffolds used in medicine are usually made of single-phase ceramic or polymer. However, the combination of these materials with graphene-based nanofillers can produce scaffolds with improved mechanical and biological properties. Highly porous (up to 85%) and lamellar hardystonite-graphene oxide (0-1.5 wt% GO) composite scaffolds were prepared by freeze casting and sintering for 5 h at 1150 C. Microstructural analysis showed that using higher amounts of GO increases the porosity and decreases the shrinkage. Optimum mechanical properties were obtained for HT-1 wt% GO ($E = 71.77$ plus or minus 2.40 MPa; $\sigma = 1.8$ plus or minus 0.16 MPa; and $K = 47.87$ MJ/m³). Biological tests were performed on the HT-1 wt% GO scaffold and HT scaffold as optimal and control samples, respectively. Apatite formed on the surfaces of HT and HT-1 wt% GO specimens soaked in SBF for 14 days. The HT-1 wt% GO scaffold sample showed the best attachment and proliferation of osteoblastic cells. The biocompatibility of the HT-1 wt% GO samples was assessed in-vitro using methyl thiazole tetrazolium (MTT) assays. The alkaline phosphatase (ALP) activity and proliferation rate of cells on HT-1 wt% GO was higher compared with pure HT.

213454

Ag-DOPED BIOACTIVE GLASS-CERAMIC 3D SCAFFOLDS: MICROSTRUCTURAL, ANTIBACTERIAL, AND BIOLOGICAL PROPERTIES

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J.Eur.Ceram.Soc. 41, No.6, 2021, p.3717-3730

Ag-doped sol-gel derived bioactive glass-ceramic particles (Ag-BG) were used to fabricate highly porous scaffolds with advanced antibacterial properties and the formation of an apatite-like layer. Heat treatment for the development of the 3D Ag-BG scaffolds was selected after characterisation of the thermal behaviour of Ag-BG particles using DTA, TGA, and hot stage microscopy. The structural characteristics of the scaffolds were studied using optical microscopy, SEM, EDS, Micro-CT, XRD, FTIR, MAS-NMR and TEM to correlate how the characteristics in the hierarchical structure of the Ag-BG scaffolds affected their antibacterial performance and apatite forming ability. Methicillin-resistant staphylococcus aureus (MRSA) was used to evaluate the antibacterial response of the Ag-BG scaffolds. The observed characteristics make these Ag-BG scaffolds attractive candidates for biomedical applications.

213455

SOLID STATE FIELD-ASSISTED SILVER ION EXCHANGE IN PORCELAIN STONEWARE: A NEW ROUTE TOWARD ANTIMICROBIAL TILES?

Biesuz M; Giopato P; Tessarolo F; Nollo G; Bortolotti M; Sglavo V M; Soraru G D - *Trento, University; INSTM Consortium*

J.Eur.Ceram.Soc. 41, No.6, 2021, p.3755-3760

A field-assisted ion exchange process was used to functionalise porcelain stoneware by introducing silver into the material surface using a solid silver electrode. The application of a small direct electric current (10 mA/cm²) at 380 C allowed silver diffusion. The penetration of silver into stoneware appeared homogeneous and primarily involved an exchange between silver and sodium ions under the effect of the external electric current. Penetration depths approaching 120 micron could be obtained in 5 min. Preliminary characterisation revealed that the Ag-exchanged stoneware possessed strong biocidal action against Staphylococcus aureus.

213456

DESIGN, CHARACTERISATION AND MECHANICAL PROPERTIES OF NEW Na⁺, CO₃(²⁻)-APATITE/ALGINATE/C60 FULLERENE HYBRID BIOCOMPOSITES

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J.Korean Ceram.Soc. 58, No.4, 2021, p.422-429

Nanoparticles (20-50 nm) of Na⁺, CO₃(²⁻)-containing calcium phosphate (1.49 wt% Na and 1.53 wt% C) with apatite-type structure were prepared by precipitation from aqueous solution. FTIR spectroscopy showed that the partial substitution of phosphate by carbonate (B-type) was achieved in the apatite-type structure. Obtained Na⁺, CO₃(²⁻)-hydroxyapatite (HAP) was used to prepare hybrid biocomposites with alginate (Alg) with weight ratio HAP:Alg = 1:1 or 2:1 and C60 fullerene (0.2-4 wt% C60) and their mechanical properties were determined. It was found that the sample with weight ratio HAP:Alg = 2:1 and containing 4.0 wt% of C60 had the highest Young's modulus (429 MPa) compared with other samples. Structure modelling of the system showed that the formation of triple complexes Na⁺, CO₃(²⁻)-HAP-Alg-C60 was stabilised by solvophobic and stacking interactions. The created biocomposites can be used as implants for bone restoration.

213457

EFFECT OF SINTERING REGIMES AND THICKNESS ON OPTICAL PROPERTIES OF ZIRCONIA CERAMICS FOR DENTAL APPLICATIONS

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Int.J.Appl.Ceram.Technol. 18, No.4, 2021, p.1354-1364

In dentistry, monolithic zirconia restorations have been preferred over all-ceramic restorations in recent years. Translucency, which can be identified by the translucency parameter, TP, is an aesthetic requirement in dental restorations. Zirconia thickness, Y₂O₃ content and sintering conditions are important parameters affecting the translucency of restorations. It is crucial to investigate monolithic ZrO₂ ceramics under different sintering regimes and reveal the critical parameters for dental restorations. An attempt was made to determine the optical properties and microstructure of monolithic ZrO₂ ceramics containing different amounts of Y₂O₃ prepared under different sintering regimes and thicknesses. A conventional zirconia CopranZr (CZI) and two monolithic zirconia materials, CopraSupreme (CSP) and CopraSmile (CSM), were used. Slow, normal, speed and translucency sintering regimes with thicknesses of 0.7, 1.0 and 1.3 mm were selected. The TP of the specimens was calculated and statistical analyses were performed using one-way ANOVA and Tukey-Kramer post hoc tests. The specimens were characterised by XRD, SEM and EDX. The results showed that the effect of different sintering regimes is more critical for CSP and CSM in terms of translucency variations. The translucency sintering regime led to the greatest grain growth.

213458

INFLUENCE OF POLYACRYLIC ACID (PAA)/Na₂HPO₄ MIXTURE ON BIPHASIC CALCIUM PHOSPHATE CEMENT: ENHANCING STRENGTH AND CELL VIABILITY

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Int.J.Appl.Ceram.Technol. 18, No.4, 2021, p.1365-1378

The effect of a combined liquid phase mixture of disodium hydrogen phosphate (Na₂HPO₄) and polyacrylic acid (PAA) on the compressive strength, setting time, bioactivity and cytotoxicity of biphasic calcium phosphate cement was studied. PAA is recognised as a water-soluble and biocompatible polymer for improving the mechanical performance of bone cement, but it usually inhibits phase conversion to hydroxyapatite after the cement has set. An attempt was made to evaluate the effect of incorporating the PAA/Na₂HPO₄ mixture into biphasic cement. The crucial concentration of PAA/Na₂HPO₄ for enhancing mechanical strength and cell viability and for maintaining bioactivity was found to be 30:70 v/v%. The phase composition and crosslinking reaction between PAA and alpha-tricalcium phosphate (alpha-TCP) powder determined by XRD and FTIR. Beta-TCP was added to the formulation to achieve biphasic cement, which was composed of beta-TCP and apatite/calcium deficient-hydroxyapatite (CD-HAp) in the final product. A commercial biphasic granule product was used for comparison in cell viability tests. The cement was shown to be non-toxic. The results suggest that PAA/Na₂HPO₄ may be beneficial for further clinical applications.

213459

AKERMANITE GLASS MICROSPHERES: PREPARATION AND PERSPECTIVES OF SINTER-CRYSTALLISATION

Dasan A; Talimian A; Kraxner J; Galusek D; Elsayed H; Bernardo E - *Trencin, Alexander Dubcek University; Cairo, National Research Centre; Padua, University*

Int.J.Appl.Glass Sci. 12, No.4, 2021, p.551-561

The flame synthesis process was used to create glass microspheres with the precise stoichiometry of kermanite ($\text{Ca}_2\text{MgSi}_2\text{O}_7$), one of the most promising contemporary bioceramics. The unusually fast cooling rate was discovered to avoid crystallisation; the size of amorphous microbeads could be linked with the size of partly crystallised precursor powders derived from traditional melt quenching and milling. In preparation for applications in additive manufacturing of kermanite-based scaffolds, the glass microspheres were studied in terms of crystallisation and sintering behaviour. Merwinite ($\text{Ca}_3\text{MgSi}_2\text{O}_8$) was found to be the principal product of glass devitrification; however, merwinite only interacts with the residual glass in a second stage, yielding kermanite. Rapid crystallisation, indicating restricted viscous flow sintering, was investigated as a means of producing components with complicated porosity distribution.

213460

POLYMER (PCL) FIBRES WITH Zn-DOPED MESOPOROUS BIOACTIVE GLASS NANOPARTICLES FOR TISSUE REGENERATION

Nescakova Z; Kankova H; Galuskova D; Galusek D; Boccaccini A R; Liverani L - *Trencin, Alexander Dubcek University; Trencin, Joint Glass Center of the IIC SAS, TrnUAD & FChPT STU; Erlangen-Nurnberg, University*

Int.J.Appl.Glass Sci. 12, No.4, 2021, p.588-600

Electrospinning was used to create composite fibrous membranes made of poly(-caprolactone) (PCL) and mesoporous bioactive glass nanoparticles (MBGNs). MBGNs and Zn-doped MBGNs were effectively integrated into polymeric fibres with diameters of 240 and 385 nm for undoped and Zn-doped PCL MBGNs fibres, respectively. Thermal investigation revealed that the concentration of MBGNs in Zn-doped MBGNs reached a high of about 21 wt%. After immersion in simulated bodily fluid, both PCL MBGNs and PCL MBGNs Zn composite membranes demonstrated bioactivity (SBF). The development of composite membranes was demonstrated using X-ray diffraction (XRD) and Fourier transform infrared spectroscopy (FTIR), illustrating the production of hydroxyapatite (HAp). The breakdown products of membranes, on the other hand, had no effect on the survival and proliferation of murine stromal cells (ST-2) and hence the new fibre architectures offer a favourable environment for cell attachment. As a result, the inclusion of mesoporous glass nanoparticles doped with therapeutically active Zn^{2+} ions into PCL fibres opens the door to the development of a multifunctional biomaterial appropriate for drug delivery and tissue engineering.

TECHNICAL CERAMICS

See also Abstract(s): 213308 213341 213372 213412 213418 213426 213427 213430

213461

STRUCTURE DESIGN INFLUENCING THE MECHANICAL PERFORMANCE OF 3D PRINTING POROUS CERAMICS

Mei H; Tan Y; Huang W; Chang P; Fan Y; Cheng L - *Xian, Northwestern Polytechnical University*

Ceram.Int. 47, No.6, 2021, p.8389-8397

A novel process is reported for fabricating 3-D porous ceramic lattices with excellent mechanical properties by bridging 3-D printing and SiC chemical vapour infiltration. The effects of coordination number, rotation angle, hollow structure and sintering temperature on the performance of ceramic lattices were also investigated. Results showed that the cubic lattices possessed the highest compressive strength and work-of-fracture, 41.93 MPa and 2126.7 kJ/m², respectively. Among different hollow lattices, the stability and bearing capacity of triangular structures was superior to that of square and honeycomb structures. The compressive strength of the triangular hollow lattices could reach 31.20 MPa and the work-of-fracture of the 3-D porous lattices without rotation could be 7 times that of lattices with a rotation angle of 30 and 45 degrees. As the sintering temperature increased, the compressive strength of the 3-D porous ceramics increased while the shrinkage tends to be more serious. Additionally, the work-of-fracture of 3-D porous ceramics reached its peak at a sintering temperature of 1400 C. The approach developed in this work provides a simple, economic, and effective way to fabricate 3-D porous ceramics, which may lead to applications in future functional devices.

213462

INFLUENCE OF THE ANIONIC COMPOSITION OF INITIAL SALTS ON OBTAINING ZINC ORTHOSTANNATE BY THE SOL-GEL METHOD

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Glass Ceram. 78, No.3/4, 2021, p.148-152

The anionic composition of zinc salts in interaction with tin (IV) chloride was studied in the production of zinc orthostannate using the sol-gel technique. The lowest temperature for the production of zinc orthostannate was determined to be 1000 C. It was discovered that when tetraamminezinc chloride is used, chloride is produced, which decomposes when fired.

213463

ELASTIC AND THERMODYNAMIC PROPERTIES OF CERIUM-DOPED YTTRIUM ALUMINIUM GARNETS

Goncharov V G; Wei N; Lau M L; Ennaceur S M; Migliori A; Xu H; Long M; Guo X - *Washington State, University; Los Alamos National Laboratory; Sichuan, University; Boise, State University; Alamo Creek Engineering*

J.Am.Ceram.Soc. 104, No.7, 2021, p.3478-3496

Cerium-doped yttrium aluminium garnets ($\text{Y}_3\text{-xCeAl}_5\text{O}_{12}$, Ce:YAGs) are promising yellow light-emitting components of solid-state white LEDs. Despite numerous studies examining the effects of Ce concentrations on the luminescent properties of $\text{Y}_3\text{-xCeAl}_5\text{O}_{12}$, the effect of the Ce dopant on the elastic and thermodynamic properties is not well understood. Resonant ultrasound spectroscopy (RUS) was here used to determine the effects of Ce doping (0.025, 0.1, 1 at%) on the elastic and thermodynamic properties of $\text{Y}_3\text{-xCeAl}_5\text{O}_{12}$. The elastic moduli calculated by the Voigt-Reuss-Hill (VRH) method showed that low Ce dopant concentrations (up to 0.1 at%) induced negligible effects on the elasticity of the YAG host matrix, while a high Ce concentration (1 at%) gave significant softening. RUS and SEM suggested that the elastic softening originated from microstructural differences induced at higher Ce dopant concentrations. An increase in elastic anisotropy at higher Ce concentrations was also found, which further elucidated the correlations between structure and elasticity of $\text{Y}_3\text{-xCeAl}_5\text{O}_{12}$. Debye temperatures (θ_D), heat capacities (C_p) and thermal conductivities (κ) were calculated for Ce:YAGs through the relations of RUS-derived parameters (sound velocities, elastic moduli) and previously determined thermal expansion coefficients. Ce:YAG had a significant reduction in θ_D , C_p and κ at Ce concentrations at least 1 at%. Extrapolation of C_p and κ to higher temperatures allowed the modelling of the thermal stress experienced by $\text{Y}_3\text{-xCeAl}_5\text{O}_{12}$ discs up to 1073.15 K.

213464

HETEROGENEOUS PHOTOCATALYTIC REDUCTION OF HEXAVALENT CHROMIUM BY MODIFIED Ag, Cu CO-DOPED TUNGSTEN OXIDE NANOPARTICLESBoonprakob N; Channei D; Zhao C - *Uttaradit,Rajabhat University; Phitsanulok,Naresuan University; Guangdong,University of Technology*
J.Aust.Ceram.Soc. 57,No.3,2021,p.743-754

A highly efficient photocatalyst of 5.0 mol% Ag, 0.5 mol% Cu co-doped WO₃ nanoparticles was synthesised by a simple co-precipitation route at low temperature. The nanoparticles had a high specific surface area (SSA), rectangular shape and controlled size. The Ag metal ions acted as a loading on the surface. The Cu metal ions disappeared into the WO₃ structure, enhancing electron trapping and reducing the bandgap energies. The intense, narrow peak in the XRD patterns corresponded to the strongly crystalline monoclinic WO₃. The particle size was 30-40 nm. The high SSA (34.0 m²/g) was key to enabling Cr⁶⁺ ions to adsorb on the large active sites of the catalyst. TEM of the modified WO₃ confirmed deposition of the hemispherical Ag particles on the WO₃ surface and that the Cu²⁺ may be incorporated into the WO₃. XPS showed a drastic shift in binding energy for all oxidation states, consistent with EDX and the results of spontaneous elemental mapping. The DRS-UV-Vis spectrum of annealed Ag, Cu-WO₃ revealed a red-shift to a wavelength of ca. 475 nm, with a bandgap energy of 2.61 eV, helping to increase the rate of electron generation. The photoreduction of Cr⁶⁺ over the optimum catalyst showed an extremely fast decay: 82.38% in just 15 min under low-energy visible light radiation. The optimum sample decayed a percentage of Cr⁶⁺ which was around 8 times that achieved with pure WO₃.

Borides

213465

ULTRA HIGH TEMPERATURE HIGH-ENTROPY BORIDES: EFFECT OF GRAPHITE ADDITION ON OXIDES REMOVAL AND DENSIFICATION BEHAVIOURBarbarossa S; Orru R; Garroni S; Licheri R; Cao G - *Cagliari,Universita degli Studi; Sassari,University; Burgos,University*
Ceram.Int. 47,No.5,2021,p.6220-6231

The introduction of 0.5-1.0 wt% graphite to the powders, prepared by self-propagating high-temperature synthesis (SHS), was beneficial for the removal of oxide impurities (from 2.7-8.8 wt% to 0.2-0.5 wt%) during the spark plasma sintering (1950C/20 min, 20 MPa) of (Hf_{0.2}Mo_{0.2}Ta_{0.2}Nb_{0.2}Ti_{0.2})B₂ and (Hf_{0.2}Mo_{0.2}Ta_{0.2}Zr_{0.2}Ti_{0.2})B₂ ceramics. The consolidation level achieved increased from about 92.5 and 88%, respectively, to values exceeding 97%. Increased graphite content slightly improved the densification and the final products became progressively richer in unreacted carbon. It was assumed that graphite played a double role during SPS, not only as a reactant during the carbothermal reduction of oxide contaminants but also as a lubricating agent for the powder particles. The latter phenomenon could have been responsible for the improved densification when 3 wt% or larger amounts of additive were used. Another positive effect was the crystallite size refinement of the high-entropy phases with the progressive abatement of oxides, which confirmed that their presence promoted grain coarsening during sintering.

213466

RAPID SYNTHESIS OF CaB₆ POWDERS BY COMBINING INDUCTION HEATING WITH CARBOTHERMIC REDUCTION AND THEIR ANTIOXIDANT PROPERTIESXu H; Wang X; Zhang W; Deng N - *Jiujiang,University; Wuhan,University of Science & Technology*
J.Aust.Ceram.Soc. 57,No.3,2021,p.919-926

CaB₆ powders were synthesised by combining induction heating (IH) with carbothermic reduction from a CaO-B₂O₃-C system. Cubic single-phase CaB₆ powders with a mean particle size of 12 micron were successfully synthesised at 1700 C after 10 min in an Ar atmosphere (the reaction time can be shortened). The liquid phase of the reaction system can be rapidly formed using IH. Under the synergistic effects of high temperature and an induced field, mass migration is easily promoted, leading to the rapid formation of CaB₆. Oxidation test results showed that synthesised CaB₆ powders incorporated into MgO-CaO-C refractory materials provided much better oxidation resistance than conventional SiC.

213467

MECHANICAL PROPERTIES OF BOROTHERMALLY SYNTHESISED ZIRCONIUM DIBORIDE AT ELEVATED TEMPERATURESMurchie A C; Watts J L; Fahrenholtz W G; Hilmas G E - *Missouri,University of Science & Technology*
Int.J.Appl.Ceram.Technol. 18,No.4,2021,p.1235-1243

The mechanical properties of a nominally phase pure ZrB₂ ceramic were determined up to 2300 C in an argon atmosphere. ZrB₂ samples were hot pressed at 2000 C using borothermally synthesised powder obtained from high purity ZrO₂ and B raw materials. The relative density of the ceramics was about 95%, with a mean grain size of 8.8 micron. The room temperature flexural strength was 447 MPa, decreasing to 196 MPa at 1800 C before increasing to 360 MPa at 2300 C. The strength up to 1800 C was probably controlled by a combination of effects, namely, surface damage from oxidation of the specimens, stress relaxation, and decreases in the elastic modulus. The strength above 1800 C was controlled by flaws consistent with maximum grain sizes and the largest pores. The fracture toughness was 2.3 MPa.m^{exp(1/2)} at RT, rising to 3.1 MPa.m^{exp(1/2)} at 2200 C. The use of higher purity starting materials improved the mechanical behaviour in the ultra-high temperature regime compared to previous studies.

Carbides

See also Abstract(s): 213383 213414

213468

COMPUTATIONAL THERMODYNAMIC STUDY OF SiC CHEMICAL VAPOUR DEPOSITION FROM MTS-H₂Peng J; Jolly B; Mitchell D J; Haynes J A; Shin D - *Oak Ridge National Laboratory*
J.Am.Ceram.Soc. 104,No.7,2021,p.3726-3737

Computational thermodynamic analysis of CVD of SiC from the methyltrichlorosilane-hydrogen (MTS-H₂) was performed using up-to-date thermodynamic databases. High-resolution computation was carried out with fine intervals of temperature and pressure at various H₂/MTS ratios to investigate the deposition condition range (800-1600 C, 0-26664 Pa and H₂/MTS ratios of 0.1 to 100). The effect of deposition parameters on the compositions and phase stabilities of the deposit and gas phase relevant to vapour processing was determined. Low pressure and medium

temperatures (1000-1400 C) were found to be beneficial to reaching a higher SiC deposition efficiency and gave an optimal window to prepare a high-purity (> 99 wt% SiC) deposit. This optimal processing window significantly expanded with increasing H₂/MTS ratio (< 20). The results were supported by previous theoretical and experimental observations. The mass fraction of SiC in the deposit was proposed as an additional perspective to understand the discrepancy between thermodynamic calculation and experimental observation of pure CVD SiC at low H₂/MTS ratios.

213469

FORMATION MECHANISM OF Ti₂InC BY PRESSURELESS SINTERING AND OPTIMISATION OF SYNTHESIS PARAMETERS

Li S; Liu Y; Zhang Y; Wang L; Zhang P; Pan L; He W; Tian W; Sun Z - *Nanjing, Southeast University; Nanjing, Institute of Technology*

J.Aust.Ceram.Soc. 57, No.3, 2021, p.911-917

Ti₂InC is expected to be promising in many fields owing to its excellent conductivity and stability, and large-scale synthesis of high-quality Ti₂InC is crucial to exploring its various potential applications. Ti₂InC powder was synthesised starting from elemental Ti/In/C powder mixtures by pressureless sintering. The composition of the starting powder and sintering parameters were optimised. Ti₂InC phase was easily obtained by sintering a mixture of 2Ti/1In/0.95C (molar ratio) at 1250 C for 1.5 h, greatly shortening the preparation time. The purity of the Ti₂InC in the sintered product reached 94 wt%, as determined by the relative intensity ratio (RIR) method, and unreacted C was confirmed as a major impurity phase. DSC, XRD and SEM revealed the reaction path of Ti₂InC formation, and the formation mechanism was discussed. TiC was not formed in the process.

213470

FABRICATION OF SILICON CARBIDE PARTICLES FROM RECYCLED POLYSILICON PHOTOVOLTAIC CELLS

Oh A H; Lee H S; Kim B-G; Choi S-C; Jung Y-G; An G S - *Hanyang, University; Changwon, National University*

J.Ceram.Process.Res. 21, No.4, 2020, p.400-406

A method for recovering polysilicon (poly-Si) wafers from end-of-life poly-Si photovoltaic (PV) cells, and the synthesis of high-purity silicon carbide using the recovered wafers, is reported. An end-of-life poly-Si PV module was etched using a mixture of HF and HNO₃ to recover the wafer. The purified wafer was then milled by high-energy milling to form Si powders. Fine SiC powder was then synthesised by spark plasma sintering a mixture of the poly-Si powder and carbon black. The optimum temperature for synthesising the fine SiC powder was determined by analysing the morphology and crystal structure of powders synthesised at different reaction temperatures. After milling, the poly-Si and SiC powders were washed using an HF solution to remove oxide impurities. A fine SiC powder with an oxygen content of 0.11% was obtained.

213471

COMPLEX GEOMETRY MACROPOROUS SiC CERAMICS OBTAINED BY 3D-PRINTING, POLYMER IMPREGNATION AND PYROLYSIS (PIP) AND CHEMICAL VAPOUR DEPOSITION (CVD)

Baux A; Jacques S; Allemand A; Vignoles G L; David P; Piquero T; Stempin M-P; Chollon G - *Laboratoire des Composites Thermostructuraux; CEA-DAM; CANOE*

J.Eur.Ceram.Soc. 41, No.6, 2021, p.3274-3284

The manufacture of SiC ceramics based on 3-D printing, polymer impregnation and pyrolysis and chemical vapour deposition (CVD) is reported. Green porous elastomer structures were initially prepared by fused deposition modelling (FDM) 3-D printing using a composite polyvinyl alcohol/elastomer wire and soaking in water, followed by impregnation with an allylhydridopolycarbosilane preceramic polymer. After crosslinking and pyrolysis, the polymer-derived ceramics were reinforced by the CVD of SiC using CH₃SiCl₃/H₂ as a precursor. The multiscale structure of the SiC porous specimens was examined by X-ray tomography and SEM and their oxidation resistance was improved by the pure and dense CVD-SiC coating.

213472

FABRICATION OF CERAMIC PARTICLES FROM PRECERAMIC POLYMERS USING STOP FLOW LITHOGRAPHY

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J.Eur.Ceram.Soc. 41, No.6, 2021, p.3314-3320

This work aims to expand the palette of materials suitable for stop flow lithography (SFL) processing by investigating the use of UV-crosslinkable preceramic polymers to prepare ceramic particles. Commercially available methacrylated-polysiloxane was used as the preceramic polymer and was mixed with 2.5 wt% Irgacure 651 photoinitiator. A simple SFL system was assembled to continuously fabricate the UV-crosslinked preceramic polymer particles in the shape of hexagons, triangles and gears with diameters ranging from 100 to 200 micron and thicknesses of 74 micron plus or minus 4 micron. The particles were harvested from the excess preceramic solution, cleaned and then pyrolysed at 1000 C to transform them into silicon oxycarbide particles. Particle shape was maintained during pyrolysis despite approximately 80% linear shrinkage due to the removal of acryl and methyl side groups, as confirmed via FTIR. After pyrolysis the outer diameters of the SiOC particles ranged from 20 to 40 micron with thicknesses of 10 to 12 micron and the pyrolysed particles were recovered and dispersed in water. This work demonstrated a robust path for the fabrication of ceramic particles with specific shapes from preceramic polymers via SFL.

213473

SYNTHESIS OF ULTRAFINE SILICON CARBIDE NANOPARTICLES USING NONTHERMAL ARC PLASMA AT ATMOSPHERIC PRESSURE

Wang C; Zhou J; Song M; Lu Z; Chen X; Zheng Y; Xia W - *China, University of Science & Technology*

J.Am.Ceram.Soc. 104, No.8, 2021, p.3883-3894

The scalable production of ultrafine SiC nanoparticles with sizes < 10 nm remains a challenge. A novel process based on atmospheric nonthermal arc plasma for the continuous synthesis of ultrafine SiC nanoparticles was proposed. This low-cost and scalable technique allows SiC nanoparticles with small size (5-9 nm) and narrow size distribution to be prepared by hexamethyldisilane (HMDS) decomposition in an argon/hydrogen plasma environment. The as-synthesised products were carbon-rich beta-SiC nanoparticles with plentiful functional groups on the surface. The addition of hydrogen in the plasma gas could tune the product characteristics, such as decreasing the particle size, improving the crystallinity and reducing the carbon and oxygen contents. The as-prepared beta-SiC nanoparticles had a high band gap (about 2.5 eV), and their photoluminescence peak showed a clear blue shift relative to that of bulk beta-SiC, which was mainly attributed to the quantum confinement effect induced by their ultrafine size. According to the spectral information of arc plasma, the formation of SiC nanoparticles in the plasma is discussed.

Nitrides

See also Abstract(s): 213428 213526

213474
SCALABLE PRODUCTION OF BORON NITRIDE NANOSHEETS IN IONIC LIQUIDS BY SHEAR-ASSISTED THERMAL TREATMENT
 Sun G; Bi J - *Shandong, University*
Ceram.Int. 47, No. 6, 2021, p. 7776-7782
 A scalable method to exfoliate boron nitride nanosheets (BNNs) in ionic liquids (ILs) via shear-assisted thermal treatment is reported. Few-layer BNNs, with well-preserved structural integrity, were prepared by this method. The synergistic effects of strong physical adsorption and intercalation of IL molecules, chemical interactions between hydrogen fluoride (HF) and h-BN, activation energy provided by heat treatment and shear forces generated by a repetitive stirring effect contributed to the exfoliation of BNNs.

213475
SYNTHESIS AND THERMAL EVOLUTION OF POLYSILAZANE-DERIVED SiCN(O) AEROGELS WITH VARIABLE C CONTENT STABLE AT 1600 C
 Zambotti A; Biesuz M; Camprotrini R; Carturan S M; Speranza G; Ceccato R; Parrino F; Soraru G D - *Trento, University; Padua, University; Fondazione Bruno Kessler*
Ceram.Int. 47, No. 6, 2021, p. 8035-8043
 The possibility of producing aerogels belonging to the SiCN system from polysilazane mixtures, namely perhydropolysilazane (PHPS) and a methyl/vinyl-containing polysilazane, namely Durazane 1800 and thus changing the C/Si ratio of the amorphous pyrolysed products was investigated. The chemical composition of the ceramic aerogel was shown to affect the main properties of the porous materials, including thermal stability and specific surface area (SSA). Results showed that the presence of carbon in the aerogels inhibited the crystallisation of Si₃N₄ up to 1600 C in N₂ and allowed a SSA of about 90 m²/g to be maintained up to this temperature.

213476
STRUCTURE AND PERFORMANCE CONTROL OF POROUS Si₃N₄ CERAMICS FABRICATED BY FREEZE-DRYING PROCESS
 Sun M; Yang S; Gao X; Man P; Qu J; Zhang W; Yin S; Cheng L - *No. 52 Inst. of China North Industries Group Corp.; Beijing, Institute of Technology; Xian, Northwestern Polytechnical University*
Ceram.Int. 47, No. 6, 2021, p. 8169-8174
 The effects of glycerine and silica sol on the structure and performance of porous Si₃N₄ ceramics, prepared via freeze-drying, were studied. Glycerol additions affected the growth process of the ice crystals, changed the pore structure from bimodal to triple peak distribution and a formed regular honeycomb structure. This improved the mechanical properties and electromagnetic wave transmission properties of the Si₃N₄ ceramics and changed the mesoscopic morphology of the material. For porous Si₃N₄ ceramics with 72.5% porosity, the flexural strength was 38 MPa, the compressive strength was 42.6 MPa and the dielectric constant was 1.75 in the 12.4-18 Hz frequency range as the amount of added glycerol increasing to 5 wt%. By reducing the sintering eutectic point of the system, the addition of silica sol promoted sintering shrinkage and the formation of new phases. Therefore, the pore size distribution was changed from bimodal to unimodal and the dielectric constant of samples increased from 2.25 to 3.5. Results showed that the structure and performance of the porous Si₃N₄ ceramics were controlled by additions of glycerol and silica sol.

213477
ESTIMATE OF THE POROUS STRUCTURE PARAMETERS OF REACTION-BONDED SILICON NITRIDE
 Kryuchkov Yu N - *Gzheh, State University*
Glass Ceram. 78, No. 3/4, 2021, p. 157-160
 On the basis of the author's techniques and structural model of monodisperse systems in combination with percolation theory, the parameters of the porous structure of reaction-bonded silicon nitride are calculated as functions of the pore-forming agent content. When the thermal conductivity of ceramics was evaluated, it was discovered that the analytical and experimental findings for silicon nitride without a pore-former are in good agreement, but there is a significant discrepancy for samples with a pore-former. It is suggested that the significant decrease in thermal conductivity seen after the insertion of a pore-former is caused by the separation of silicon particles by the bigger particles of the pore-former.

213478
COARSE-GRAINED BETA-Si₃N₄ POWDERS PREPARED BY COMBUSTION SYNTHESIS
 Cui W; Li F; Tian Z; Zhang J; Du S; Chen Z; Chen K; Liu G - *Beijing, Tsinghua University*
J. Am. Ceram. Soc. 104, No. 7, 2021, p. 2919-2923
 Coarse-grained beta-Si₃N₄ powders were prepared by combustion synthesis under N₂ pressure of 6 MPa, with a low diluent content of not more than 10 wt% and a high reaction temperature of > 1900 C, well beyond the melting point of Si. Beta-Si₃N₄ was obtained as the major phase in the products, except for a small amount of residual Si. The addition of carbon black reduced the residual Si, but beta-SiC formed if too much carbon black was used. The coarse-grained beta-Si₃N₄ powders consisted of beta-Si₃N₄ crystals with an average thickness of > 10 micron, some crystals being > 20 micron. The growth mechanism of the coarse beta-Si₃N₄ crystals is discussed, associated with the particular reaction conditions in combustion synthesis.

213479
SYNTHESIS OF MONOPHASE TWO-DIMENSIONAL ALPHA-Si₃N₄ NANOPATELETS VIA AN IONOTHERMAL ROUTE
 Wang S; Liang F; Li S; Tian L; Zhang S; Jia Q; Chang S; Yan S; Zhang H - *Wuhan, University of Science & Technology; Exeter, University; Zhengzhou, High Temperature Ceramics Institute; Shenzhen, University*
Int. J. Appl. Ceram. Technol. 18, No. 4, 2021, p. 1183-1191
 Single phase alpha-Si₃N₄ was prepared by a salt melt strategy using Si as a starting material. It was found that molten salt enhanced the conversion of Si to monophasic alpha-Si₃N₄, and some as-synthesised alpha-Si₃N₄ nanoparticles had a hexagonal platelet morphology. These nanoplatelets had a mean lateral dimension of about 170 nm and a thickness of about 5.2 nm. The nitridation of Si molten salt was investigated by a thermal quenching method, and the silicon nitride intermediate products were detected by XPS. Silicon halides possessing higher reactivity facilitated the

nitridation of Si and nucleation of α -Si₃N₄. Furthermore, the lattice mismatch between α -Si₃N₄ and the salts was calculated, suggesting that the lateral growth of α -Si₃N₄ nanoplatelets is guided by the salt crystal structure. The formation mechanism for the nanoplatelets is proposed.

213480
ENHANCED MECHANICAL AND THERMAL PROPERTIES OF AlN CERAMICS VIA A CHEMICAL PRECIPITATION PROCESS
 Nie G; Sheng P; Li Y; Zuo F; Bao Y; Wu S - *Guangdong, University of Technology; China Building Materials Academy*
Int. J. Appl. Ceram. Technol. 18, No. 4, 2021, p. 1255-1268
 A uniform dispersion of sintering aids is crucial for improving the thermal and mechanical properties of AlN ceramics. A Y₂O₃-coated AlN composite powder was prepared by chemical precipitation (CP), thereby improving the homogenisation of Y₂O₃ in AlN green compacts. The precipitation coating behaviour of the Y₂O₃ precursor was investigated by FTIR and TG-DSC, and the corresponding equation for the reaction was proposed. TEM, XRD and XPS indicated that a uniform amorphous Y₂O₃ layer fully covered the AlN powder surface. The microstructures and phase compositions of the sintered AlN samples prepared by CP and conventional ball milling (BM) processes were compared. The CP process decreased the oxygen content in the AlN grains, facilitating the formation of desirable isolated second phases and strengthening the grains and grain boundaries of the AlN ceramics, thereby enhancing the thermal conductivity, bending strength and fracture toughness by 9.43%, 10.56% and 18.50%, respectively, compared to those of the BM processed samples.

213481
STUDYING THE IN VITRO CORROSION RESPONSE OF NANOSTRUCTURED TaN COATINGS IN HANK'S PHYSIOLOGICAL SOLUTION
 Babaei K; Fattah-alhosseini A; Elmkhah H; Imantalab O; Ghomi H
Int. J. Appl. Ceram. Technol. 18, No. 4, 2021, p. 1269-1280
 An attempt was made to determine the effect of N₂ to Ar pressure (P(N₂)/P(Ar)) ratio on the microstructure and corrosion behaviour of nanostructured TaN coatings deposited by reactive DC-magnetron sputtering. The microstructure was studied by XRD and SEM, and the elemental distribution was studied by EDS. The corrosion behaviour was investigated by performing potentiodynamic polarisation and electrochemical impedance spectroscopy measurements on Hank's physiological solution. The test results revealed that the coating with a P(N₂)/P(Ar) content of 17.6% consisted of hexagonal and orthorhombic TaN phases and had a denser microstructure with open pores. This coating also showed superior corrosion behaviour compared to the other samples. Its corrosion resistance increased by increasing the immersion time from 48 to 168 h.

213482
RESISTANT TRANSITION-METAL-NITRIDES BASED COATINGS FOR SOLAR ENERGY CONVERSION
 Wang X; Li K; Cheng X - *Jinzhong, University; Taiyuan, University of Technology; Wuhan, University of Technology*
J. Eur. Ceram. Soc. 41, No. 7, 2021, p. 4076-4085
 A four-layered Cr/CrAlN/CrAlON/CrAlO tandem coating was annealed at 500 C in air for up to 1000 h. The absorption increased from 0.90 to 0.92 while the emittance decreased from 0.20 to 0.14 after the annealing treatment. Microstructural analysis suggested that the enhanced selectivity was caused by the formation of AlN, CrN and Cr₂N nanocrystallites in the CrAlN and CrAlON amorphous matrices and Al₂O₃ and Cr₂O₃ nanocrystals in the CrAlO antireflection layer after annealing. The excellent thermal tolerance was primarily ascribed to the self-passivation of Al atoms, which could partially oxidise during annealing in air which resulted in the formation of an aluminium oxide layer covering these nanoparticles. Results suggested that the incorporation of an amorphous-phase structure which consisted of transition-metal-nitrides embedded in amorphous matrices is an attractive strategy to obtain high-performance cermet-based solar absorbers for photothermal conversion.

213483
HOT ISOSTATIC PRESSING OF TRANSPARENT AlON CERAMICS ASSISTED BY DISSOLUTION OF GAS INCLUSIONS
 Li J; Zhang B; Tian R; Mao X; Zhang J; Wang S - *Shanghai, Institute of Ceramics; Beijing, University of Chinese Academy of Sciences*
J. Eur. Ceram. Soc. 41, No. 7, 2021, p. 4327-4336
 Transparent AlON ceramics were fabricated using hot isostatic pressing (HIP) assisted by the dissolution of gas inclusions. The effect of the additive content, pre-sintering, HIPing and annealing parameters on the sintered AlON ceramics were investigated and optimised. The obtained transparent AlON ceramics had a high transmittance of 85.8 % and a low loss coefficient of 0.005 /cm at 2000 nm after annealing. Compared with different sintering additives and pre-sintering atmospheres, the densification mechanism showed that SiO was formed from SiO₂ during pre-sintering in a N₂ atmosphere occupying micropores which were then solidified and dissolved into the AlON matrix during HIPing. The transformation and dissolution of gas into the AlON ceramics made it possible to anneal the HIPed ceramics without degradation of the microstructure and transmittance.

Oxides

See also Abstract(s): 213307 213377 213382 213389 213392 213395 213400 213429 213584

213484
LUMINESCENCE PERFORMANCE OF YTTRIUM-STABILISED ZIRCONIA CERAMICS DOPED WITH Eu³⁺ IONS FABRICATED BY SPARK PLASMA SINTERING TECHNIQUE
 Stepanov S; Khasanov O; Dvilis E; Paygin V; Valiev D; Ferrari M - *Tomsk, Polytechnical University; CNR, Ist. Fotonica e Nanotecnologie*
Ceram. Int. 47, No. 5, 2021, p. 6608-6613
 Yttrium stabilised zirconia (YSZ) ceramics doped with Eu³⁺ ions were fabricated by spark plasma sintering and the effects of the europium concentration and post-annealing process on the structural, optical and luminescent properties of the ceramics were studied. Increased europium concentration, from 0.1 to 3 wt%, did not lead to significant changes in the transmission spectra. However, annealing in air between 700 and 1300 C affected the transmission spectrum due to the formation of oxygen vacancy defects. Analysis of the photoexcitation and photoluminescence spectra showed that the main excitation bands were determined by the direct excitation of the 7F₀ ground state of Eu³⁺ ions to the higher 4f energy levels with further radiation transitions from these states. Moreover, the Eu³⁺ ion in the obtained ceramics occupied low-symmetry sites without an inversion centre. The luminescence decay kinetics were described by a double exponential function with a decay time Tau₁ of about 20 ns and Tau₂ approximately 90 ns for intrinsic emission centres and millisecond (Tau = about 1.4 ms) for Eu³⁺ emission, for all the investigated ceramics. The

luminescence spectra in the nanosecond time region were characteristic of yttrium-stabilised zirconia and were caused by oxygen vacancies in the presence of heavy cations (Y³⁺ and Eu³⁺).

213485

COMPARATIVE STUDY OF Yb:Lu₃Al₅O₁₂ AND Yb:Lu₂O₃ LASER CERAMICS PRODUCED FROM LASER-ABLATED NANOPOWDERS

Basyrova L; Loiko P; Maksimov R; Shitov V; Serres J M; Griebner U; Petrov V; Aguilo M; Diaz F; Mateos X - *Tarragona, Universidad Rovira i Virgili; St. Petersburg, ITMO University; Caen, Normandie University; Ekaterinburg, Ural Federal University; Ekaterinburg, Institute of Electrophysics; Berlin, Max-Born-Institut*

Ceram.Int. 47, No.5, 2021, p.6633-6642

A comparative study of two Lu-based oxide ceramics doped with Yb³⁺ ions, namely Yb:Lu₃Al₅O₁₂ (garnet) and Yb:Lu₂O₃ (sesquioxide), which are promising materials for thin-disc lasers. The ceramics were fabricated using nanopowders of 3.6 at% Yb:Lu₂O₃ and Al₂O₃ produced by laser ablation: Yb:Lu₃Al₅O₁₂ - by vacuum sintering at 1800 C for 5 h with the addition of 1 wt% TEOS as a sintering aid, and Yb:Lu₂O₃ - by vacuum pre-sintering at 1250 C for 2 h followed by hot isostatic pressing at 1400 C for 2 h under Ar gas pressure of 207 MPa. The comparison included the structure, Raman spectra, transmission, optical spectroscopy and laser operation. The crystal-field splitting of Yb³⁺ multiplets was revealed for Lu₃Al₅O₁₂. A continuous-wave (CW) Yb:Lu₃Al₅O₁₂ ceramic microchip laser generated 5.65 W at 1031.1 nm with a slope efficiency of 67.2%. In the quasi-CW regime, the peak power was scaled up to 8.83 W. The power scaling for the Yb:Lu₂O₃ ceramic laser was limited by losses originating from residual coloration and inferior thermal behaviour.

213486

STRONTIUM HEXAALUMINATE FORMATION IN ALUMINA AND ALUMINA-ZIRCONIA MATRICES

Kuzmin R; Cherkasova N; Bataev A; Veselov S; Ogneva T; Ruktuev A; Felofyanova A - *Novosibirsk, State Technical University*

Ceram.Int. 47, No.5, 2021, p.6854-6859

The formation mechanism of strontium hexaaluminate (SrAl₁₂O₁₉) in Al₂O₃-SrO and Al₂O₃-SrO-ZrO₂ composites was investigated. Strontium oxide (SrO) powder was added to the initial suspensions in various quantities and high-purity submicron powders were used during the experiments. The powder compositions were heat treated between 900 and 1500 C for 5 h and the stepwise formation of SrAl₁₂O₁₉ was established using X-ray phase analysis. The intermediate compound, SrAl₂O₄, formed in the 900-1200 C temperature range and the onset of SrAl₁₂O₁₉ formation occurred at 1200 C and was completed between 1400 and 1500 C. After sintering between 1300 and 1500 C for 1 h, the samples were assessed. Density values indicated a decreasing trend for increased amounts of SrO and the density of alumina ceramics sintered at 1500 C was 3.65 plus or minus 0.2 g/cm³. However, the alumina ceramics contained 15 wt% SrAl₁₂O₁₉ and their density was 3.35 plus or minus 0.4 g/cm³. The reduced density level was explained by the platelet shape of the SrAl₁₂O₁₉ grains, which prevented additional contact points from generating between the matrix grains and led to porosity formation. The effect of a reduction in the alumina grain size was established with an increase in the content of the initial SrO and SrAl₁₂O₁₉ powders. SEM showed that the SrAl₁₂O₁₉ platelets comprised one to three layers fused together along the basal planes and zirconia grains inhibited the lateral growth of the SrAl₁₂O₁₉ platelets.

213487

TRIBOLOGICAL MECHANISM OF MICRO-ARC OXIDATION COATINGS PREPARED BY DIFFERENT ELECTROLYTE SYSTEMS IN ARTIFICIAL SEAWATER

Guo H; Liu Z; Wang Y; Li J - *Lanzhou, Northwest Normal University; Ningbo, Inst. of Material Technology & Engineering*

Ceram.Int. 47, No.6, 2021, p.7344-7352

Microarc oxidation (MAO) coatings were prepared in four different electrolyte systems, including mixed acid, phosphate, phosphate-aluminate and phosphate-silicate electrolytes. The friction and wear properties of the MAO coatings in ambient air, seawater and four groups of saline solutions related to seawater were investigated. Results showed that the addition of silicate to phosphate could increase the density of the coating. The phosphate-aluminate ceramic layer had the lowest wear rate in different environments. Additionally, the friction coefficient and wear rate of the MAO coatings in seawater were lower than those in ambient air due to the boundary lubrication effect of seawater. Meanwhile, the presence of divalent metal salts in seawater resulted in it having better lubricity than other salt solutions.

213488

REACTION INDUCED MULTIFUNCTIONAL TiO₂ ROD/PARTICLE NANOSTRUCTURED MATERIALS FOR SCREEN PRINTED DYE SENSITISED SOLAR CELLS

Selvapriya R; Sasirekha V; Vajeeston P; Pearce J M; Mayandi J - *Coimbatore, Avinashilingam University for Women; Oslo, University; Michigan, Technological University; Aalto, University; Madurai Kamaraj University*

Ceram.Int. 47, No.6, 2021, p.8094-8104

The potential of utilising multifunctional nanostructured materials for efficient light trapping and electron transport in solar cells by combining titanium dioxide (TiO₂) rods and nanoparticles was investigated. A simple solvothermal method was used for the synthesis of the coupled morphology by adopting the desired precursor with a constant concentration and temperature. The reaction time (12, 24, 36 and 48 h) was varied and the materials resultant physical, optical and structural characteristics determined the nature of the prepared material. The crystallographic phase of the synthesised samples was determined using XRD and Raman analysis. From experimental data it was hypothesised that the surface plane of anatase (105) was involved in the deformation of the structure and the formation of the rutile phase. To further investigate the formation of mixed phase in the prepared sample a computation study was performed using density functional theory coupled with Hubbard U correction (DFT + U) as a function of volume in both the anatase and rutile phases. The relative stability of the O-Ti-O networks was explored starting from ultrathin materials of four different sizes, of anatase and rutile nanorods separately. Finally, the synthesised TiO₂ materials were used to prepare screen printed dye sensitised solar cell (DSSC) devices and their respective properties were quantified.

213489

HYDROTHERMALLY GROWN ZnO NSs ON BI-DIRECTIONAL WOVEN CARBON FIBRE AND EFFECT OF SYNTHESIS PARAMETERS ON MORPHOLOGY

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Ceram.Int. 47, No.6, 2021, p.8208-8217

ZnO nanostructures were hydrothermally grown on woven carbon fibre under varying processing parameters. The effects of the process parameters, including pH and molar concentration of the precursor solution, growth duration and growth temperature on the morphology and dimensions of the ZnO nanostructures were assessed using FE-SEM, EDS and UV-VIS-NIR spectroscopy. Different ZnO nanostructures, nanopallet-like, nanoflower-like and nanoflake-like, were achieved at pH > 7 but nanorod-like and nanowire-like morphology were achieved at a low pH. In addition, the effect of the chemical reactions, seeding and growth process on the ZnO nanostructures on woven carbon fibres are discussed.

213490

DENSITIES OF LIQUID LANTHANOID SESQUIOXIDES MEASURED WITH THE ELECTROSTATIC LEVITATION FURNACE IN THE ISS

Koyama C; Ishikawa T; Oda H; Saruwatari H; Ueno S; Oshio M; Watanabe Y; Nakata Y - *Japan Aerospace Exploration Agency; Sokendai, Graduate University for Advanced Studies; Ibaraki, Advanced Engineering Service*

J.Am.Ceram.Soc. 104, No.7, 2021, p.2913-2918

The densities of several liquid lanthanoid sesquioxides (Ln_2O_3 , Ln = Er, Ho, Tb, Gd) were measured over the temperature range from 2700 K (the approximate melting point of these materials) to 3200 K. The measurements were carried out using the Electrostatic Levitation Furnace onboard the International Space Station (ISS-ELF), which enabled precise density values to be obtained over a wide temperature range without contamination of the samples. Based on the Coulomb force between the charged samples and the surrounding electrodes and using a rapid feedback control process, specimens were stably levitated and subsequently melted by high power lasers. The molten oxides had spherical morphologies and their volumes were readily calculated from magnified images. The densities of the oxides could be determined by subsequently weighing the samples on earth. The densities of Er_2O_3 , Ho_2O_3 , Tb_2O_3 and Gd_2O_3 at their melting temperatures (T_m) were 8170, 8035, 7451 and 7268 kg/m³, respectively, and the densities showed a linear correlation with temperature. The molar volumes of the oxides at their T_m values were calculated and compared with those of other sesquioxides (Al_2O_3 , Ga_2O_3 and B_2O_3). The molar volumes of the nonglass-forming sesquioxides (Er_2O_3 , Ho_2O_3 , Tb_2O_3 , Gd_2O_3 , Al_2O_3 and Ga_2O_3) showed linear correlations with the cubes of their cation radii, whereas those of the glass-forming oxide (B_2O_3 , As_2O_3 and Sb_2O_3) showed different correlations.

213491

DOPING-INDUCED COLOURATION IN TITANIA

Mane R B; Sahoo R; Reddy B K S; Ravula V; Panigrahi B B; Borse P H; Chakravarty D - *Hyderabad, Int. Advanced Res. Centre for Powder Metallurgy & New Mats.; Indian Institute of Technology*

J.Am.Ceram.Soc. 104, No.7, 2021, p.2932-2936

Thermal decomposition of the MAX phase Ti_3GeC_2 at 1773 K gave an orange-coloured titania powder. Micro-XRD of the powder under oscillation mode revealed a pure rutile phase (space group $P4_2/mnm$). XPS confirmed substitutional doping of Ge in the titania lattice. The presence of the Ti-O-Ge bond was seen in the O 1s spectrum and confirmed by the shift in binding energy in Ti 2p_{3/2} and Ge 3d peaks. UV-vis DRS of the Ge-doped titania powder showed wide absorption in the visible region (380 to 650 nm) yielding a bandgap of 2.83 eV, which is desirable for photocatalytic applications. Defect states formed due to Ge doping led to lowering of the titania conduction band, inducing an orange coloration in the powder.

213492

LASER-PULSE MELTING OF CALCIUM OXIDE AND SOME PECULIARITIES OF ITS HIGH-TEMPERATURE BEHAVIOUR

Bgasheva T; Falyakhov T; Petukhov S; Sheindlin M; Vasin A; Vervikishko P - *Moscow, Joint Institute of High Temperatures*

J.Am.Ceram.Soc. 104, No.7, 2021, p.3461-3477

Despite numerous experiments, the exact melting point (MP) of CaO still remains quite uncertain due to three factors associated with it: a high melting temperature, an optical semitransparency and a high vapour pressure in the vicinity of the melting point. A method based on laser-pulse heating with CO₂- and Nd:YAG-lasers is reported. It was designed specifically to eliminate the possible ambiguity due to variance in absorbance in the specimen bulk. Temperature measurements were performed with advanced multichannel pyrometry combined with probe laser reflectometry. This technique enabled both the melting and the solidification points of CaO to be determined. The final measurement of the melting point gave a value of 3160 plus or minus 10 K.

213493

SYNTHESIS METHOD COMPARISON OF COMPOSITIONALLY COMPLEX RARE EARTH-BASED RUDDLESDEN-POPPER n = 1 T'-TYPE CUPRATES

Musico B L; Wright Q; Delzer C; Ward T Z; Rawn C J; Mandrus D G; Keppens V - *Tennessee, University; Oak Ridge National Laboratory*

J.Am.Ceram.Soc. 104, No.7, 2021, p.3750-3759

The multicomponent approach was expanded to the Ruddlesden-Popper structure with the synthesis of two different high-entropy cuprate compositions ((La_{0.2}Nd_{0.2}Gd_{0.2}Tb_{0.2}Dy_{0.2})₂CuO₄ and (La_{0.2}Pr_{0.2}Nd_{0.2}Sm_{0.2}Eu_{0.2})₂CuO₄). The effect of the synthesis method was examined using both solid-state reaction and polymeric steric entrapment (PSE) methods. It was found that PSE led to more randomly distributed cation species, providing an advantageous method of synthesis for the growing field of high entropy oxides. In-situ high-temperature XRD followed the amorphous to crystalline phase transformation in (La_{0.2}Nd_{0.2}Gd_{0.2}Tb_{0.2}Dy_{0.2})₂CuO₄ powder, synthesised using PSE. Using the high-temperature XRD data, a method for obtaining information on the kinetic behaviour was also applied. Magnetometry of both compositions indicated ferrimagnetic behaviour at low temperatures.

213494

2D-HARECXs ANALYSIS OF DOPANT AND OXYGEN VACANCY SITES IN Al-DOPED YTTRIUM TITANATE

Ohtsuka M; Oda K; Tanaka M; Kitaoka S; Muto S - *Nagoya, University; Japan Fine Ceramics Center*

J.Am.Ceram.Soc. 104, No.7, 2021, p.3760-3769

A Y₂Ti₂O₇/Al₂O₃ multilayer, with high thermal reflectivity and oxidation resistivity, has potential as a functional surface coating for advanced gas turbine blades. Slight Al doping of Y₂Ti₂O₇ has been found to significantly improve the structural stability of the multilayer, but the detailed mechanism is still unclear. Site occupancies of dopant Al were studied using high-angular resolution electron channelling X-ray spectroscopy (HARECXs) with 2D rocking of the incident electron beam. A statistical linear regression analysis of a set of observed ionisation channelling patterns (ICPs) of fluorescent X-rays quantitatively confirmed that Al³⁺ preferentially occupied the Ti⁴⁺ site, rather than the Y³⁺ site, because of the large

difference in the ionic radii of Al^{3+} and Y^{3+} . Comparison of theoretical and experimental X-ray ICPs showed that oxygen vacancies VO were introduced at the 48f site, the first-nearest neighbour of the Ti site, consistent with the hypothesis that oxygen vacancies compensate for the local charge imbalance associated with preferential substitution of Al^{3+} for Ti^{4+} . The findings can help to improve the thermal properties of environmental barrier coatings for SiC/SiC composites for advanced jet engines and also demonstrate the use of beam-rocking schemes for investigating dopant effects in various functional materials.

213495

TAILORING PORE STRUCTURES AND MORPHOLOGIES OF HIGHLY ORDERED CUBIC MESOPOROUS SILICA PREPARED IN MILD CONDITIONS: THE EFFECTS OF REACTION PARAMETERS

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J.Aust.Ceram.Soc. 57, No.3, 2021, p.663-672

A novel two-step method for the reactive synthesis of highly ordered cubic mesoporous silica with tailored pore structures and morphologies is described. The method is conducted at relatively low temperatures (20-60 C) using a single type of surfactant, Pluronic F-127. The effects of reaction parameters such as pH, hydrolysis and condensation times, surfactant concentration, and ageing temperature were investigated by SAXS, analysis of surface area and porosity by adsorption of N_2 (BET isotherms), and SEM/TEM. The results showed that, at 20 C, the particle shape is sensitive to changes in surfactant concentration, implying that it is possible to tune spherical to polyhedral particles simply by increasing the surfactant concentration. In addition, at low concentrations of F-127, the particle size was temperature-dependent, with an inversely proportional pattern of behaviour. The protocol also enabled the production of well-ordered pores, particularly with 3D-cubic symmetries $\text{Fm}\bar{3}\text{m}$ and $\text{Im}\bar{3}\text{m}$, and some hexagonal, as well as to control the pore size.

213496

SINTERING BEHAVIOUR OF NANO YTTRIA POWDER COMPACTS FABRICATED BY VARIOUS FORMING PROCESSES

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J.Ceram.Process.Res. 21, No.4, 2020, p.450-455

The sintering behaviour of 10 nm-sized, nano yttria powder was investigated using powder compacts prepared by various aqueous forming methods. A well-dispersed nano yttria slurry and gelation conditions were determined by examining the rheological behaviour according to the pH and dispersant content. The slurry prepared under optimum dispersion conditions was slip cast. For comparison, compacts were prepared by uniaxial pressing the fine nanopowder and gel powder. When a granular-type gel powder was applied, dry pressing was conducted with less agglomeration and the green density was improved, unlike the case when using nanopowder. The slip cast sample had the most homogeneous microstructure. As a result of sintering at 1650 C for 2 h in ambient air, the green slip cast sample showed the highest densification (98% relative density with 41% shrinkage). Fractures and circular pores were present in the intergranular region, unlike the case of press-formed samples. The powder compact obtained using a gel powder showed the highest sintered density of 93% compared with the press-formed compact obtained using nanopowder.

213497

SINGLE-STEP, HIGH PRESSURE, AND TWO-STEP SPARK PLASMA SINTERING OF UO_2 NANOPOWDERS

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J.Eur.Ceram.Soc. 41, No.6, 2021, p.3655-3663

Three different spark plasma sintering (SPS) treatments were applied to highly sinter-active, near-stoichiometric UO_2 nanocrystalline (5 nm) powders produced by U(IV) oxalate hydrothermal decomposition at 170 C. The sintering conditions for reaching 95 % theoretical density (TD) during regular SPS, high pressure SPS (HP-SPS), and two-step SPS (2S-SPS), were determined. Densification to 95 % TD was achieved at 1000 C during regular SPS (70 MPa applied pressure), 660 C in HP-SPS (500 MPa), and 650-550 C in 2S-SPS (70 MPa). To minimise grain growth during densification, the sintering treatments were optimised to favour densification over coarsening, and the final microstructures obtained were compared. Equally dense UO_2 samples with different grain sizes, ranging from 3.08 micron to 163 nm, were produced. Room-temperature oxidation of the powders could not be avoided due to their nanometric dimensions and a final annealing treatment was designed to reduce hyper-stoichiometric samples to UO_2 .

213498

HIGHLY ORIENTED ALPHA- Al_2O_3 TRANSPARENT CERAMICS SHAPED BY SHEAR FORCE

Chen H; Shimai S; Zhao J; Mao X; Zhang J; Zhou G; Wang S - *Shanghai, Institute of Ceramics; Beijing, University of Chinese Academy of Sciences*

J.Eur.Ceram.Soc. 41, No.6, 2021, p.3838-3843

Alumina platelets were arranged horizontally in submicron alumina particles by shear force in the flow of slurries during casting. The obtained alumina green bodies with platelets were pressureless sintered in vacuum, which produced ceramics with oriented grains and high transmittance. The effects of sintering parameters on the densification, microstructural evolution and degree of orientation of the alumina ceramics were investigated. Results showed that the densification, grain size, degree of orientation and in-line transmittance increased with increased sintering temperature. The enhanced degree of orientation was mainly coherent with grain growth. The grain-oriented samples had a higher in-line transmittance (at 600 nm) of 61 % than that of the random grain sample (29 %). Moreover, the transmission remained at a high level in the UV range (<300 nm).

213499

EFFECT OF THE PROCESSING CONDITIONS OF RETICULATED POROUS ALUMINA ON THE COMPRESSIVE STRENGTH

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J.Korean Ceram.Soc. 58, No.4, 2021, p.495-506

Reticulated porous ceramics have a 3D network structure with high porosity and permeability simultaneously. However, in general the compressive strength of reticulated porous ceramics is low, limiting their applications. To increase their compressive strength, the strut walls of the polyurethane foam need to be completely coated with alumina slurry to form a thin ceramic coating layer after optimising the processing conditions, specifically the

alumina slurry composition and the coating condition. An attempt was made to optimise the composition (thickener (methyl cellulose), dispersant (DARVAN C-N or Dolapix CE 64) and binder (PVA)) of the alumina slurry. Reticulated porous alumina was studied as there has been little research to date. The optimised processing conditions were determined to improve the compressive strength of reticulated porous alumina. The conditions, specifically the composition of the alumina slurry and the pore density of the polyurethane foam, are discussed.

213500

EFFECT OF DISPERSION STATES (ELECTROSTATIC/ELECTROSTERIC STABILISATION) ON PARTICLES ARRANGEMENT IN THE YTTRIA-STABILISED ZIRCONIA SEDIMENTS

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Int. J. Appl. Ceram. Technol. 18, No. 4, 2021, p. 1174-1182

Particle arrangement and packing in the sediment of a zirconia suspension were studied. Aqueous suspensions of YSZ nanoparticles were prepared in different states of dispersion in order to evaluate particle settling. In one state, Dolapix CE64 was used as a dispersant to provide an electrosteric interaction mechanism; in another, the suspension pH was adjusted at 4 to provide an electrostatic interaction mechanism; while in a third state, the dispersant was combined with adjustment of the pH, resulting in the most stable suspension. The stability of all the dispersion states was evaluated by zeta potential, sediment volume and height, viscosity, and packing density measurements. The sediment layers of all the suspensions were then characterised. The incorporation of an electrostatic mechanism led to a decrease in viscosity with high surface charges, while the electrosteric mechanism caused a lower sedimentation of particles. The fall velocities of the particles/agglomerates were estimated, and the influence of dispersion state on these velocities was determined. Microstructural analysis revealed homogeneous packing of particles in the sediment layer of the stable suspension, demonstrating the proper dispersion of particles. Dolapix CE64 and pH adjustment resulted in a uniform arrangement of particles without agglomeration, and spherical and regular granules with a uniform shape.

213501

PROPERTIES OF MAGNESIUM-ALUMINATE SPINEL DERIVED FROM BAUXITE AND MAGNESIA

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Int. J. Appl. Ceram. Technol. 18, No. 4, 2021, p. 1205-1212

Magnesium-aluminate spinel was prepared from bauxites and magnesias as starting materials with different molar weight ratios ($\text{Al}_2\text{O}_3:\text{MgO}$) of 0.6, 1.0 and 3.0 by solid-state sintering at 1350-1500 C. The effects of different weight ratios and sintering temperatures on the phase composition, densification behaviour, shrinkage, flexural strength and microstructure of the synthesised materials were studied. It was found that, as the bauxite content decreased, the strength first decreased before increasing. When $n(\text{Al}_2\text{O}_3)/n(\text{MgO})$ was 1, spinel was the main phase and the sample was dense. The strength reached its highest value (106.48 MPa) when the sintering temperature was 1450 C.

213502

THETA-PHASE STABILISATION BY RARE-EARTH AND ALKALI INCORPORATION IN SOL-GEL DERIVED ALUMINA

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Int. J. Appl. Glass Sci. 12, No. 4, 2021, p. 509-518

Bottom-up synthesis of alumina suspensions doped with trivalent (Eu^{3+}) or monovalent (K^+) ions has been described as a method of producing crystalline transition-phase alumina with improved thermal stability. The stability of precursor sols was tracked using dynamic light scattering, which demonstrated how dissolved cations contribute to the stabilising double layer and particle separation in the liquid phase. The addition of alkali or rare-earth dopants to the alumina structure affects the heat conversion and crystallisation behaviour even more. The Kissinger activation energy of the phase transition in Al_2O_3 reveals that Eu^{3+} and K^+ have an inhibitory impact on the development of the stable phase, which is further verified by X-ray diffraction. To confirm this result, the intensity ratio of the 7F2/7F1 characteristic photoemission bands of Eu^{3+} was utilised to investigate the local symmetry as a function of calcination temperature.

213503

RELATIONSHIP OF STRUCTURE AND MECHANICAL PROPERTY OF SILICA WITH ENHANCED SAMPLING AND MACHINE LEARNING

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J. Am. Ceram. Soc. 104, No. 8, 2021, p. 3910-3920

In terms of molecular structure and the corresponding mechanical properties between those of ordered and disordered states, partially ordered silica states have not been well explored in the literature due to little appearance in experiments and simulations. This lack of knowledge significantly hinders the understanding of the inherent mechanism of mechanical properties and limits the applications in many engineering fields. The complex interdependent relations of the structural properties of silica were here studied over a wide range of free energy surfaces and a machine learning-based prediction model was established via high-throughput molecular dynamics simulations coupled with an enhanced sampling method. Each scale of structure information of samples with varying crystallinity was analysed. Descriptors of silica structures were identified and selected as inputs to a deep neural network (DNN). The results indicate that the DNN-based approach could accurately predict the bulk modulus, shear modulus and tensile strength of the silica samples. The generalisability of the machine learning model was verified on the prediction tasks for much larger silica systems, as well as silica quenched at varying cooling rates. Overall, the enhanced sampling method could reliably accelerate the exploration of free energy surfaces and collection of training samples, and machine learning methods were effective in generating accurate and reliable predictions of mechanical properties of materials over the free energy surface.

213504

MECHANICAL PROPERTIES STUDY OF VO₂ MICRO-BEAM ACCORDING TO METAL-INSULATOR TRANSITION

Kim Y; Cho H H; Bae J K; Lee J; Lee S H; Dong X; Asghar G; Choi J-Y; Yu H K - *Ajou, University; Korea, Basic Science Institute; Sung Kyun Kwan University*

J. Am. Ceram. Soc. 104, No. 8, 2021, p. 4183-4189

There have been many attempts to develop applications using the metal-insulator transition (MIT) phenomenon of VO_2 , but the difference in the densities of the metal and insulator phases can result in deterioration or disuse during the phase transformations. For micro-sized or nano-sized devices, this can be critical. An attempt was made to measure the mechanical properties when the two phases co-existed, as well as for an individual phase, via in-situ control of the temperature of plate-shaped VO_2 . The lamellar structure was formed during MIT. At this time, the stress

was applied by the density gradient and the residual strain could easily occur at the interface of each phase. Therefore, the co-existing state was thought to be the most vulnerable during the MIT. The change in mechanical properties of VO₂ during phase transition was also simulated by the finite element method.

213505

EFFECT OF Eu-DOPING AND GRAIN BOUNDARY PLANE ON COMPLEXION TRANSITIONS IN MgAl₂O₄Marvel C J; Krause A R; Harmer M P - *Lehigh University; Florida University***J.Am.Ceram.Soc.** 104, No.8, 2021, p.4203-4213

Eu-doped MgAl₂O₄ has been used to evaluate the kinetics of equilibrium grain boundary transformations (complexion transitions) by monitoring abnormal grain growth induced by the nucleation of highly mobile complexions. The assumption had been that abnormal grain growth can be charted using time-temperature-transformation (TTT) diagrams to reflect the complexion transition nucleation and growth kinetics. A model depending on doping concentration, grain size and abnormal area fraction has been recently developed to estimate excess grain boundary coverage, and thereby predict complexion types depending on microstructural descriptors, but this model has not been validated using atomic-resolution characterisation. An attempt was made to directly validate the grain boundary excess model and complexion TTT diagrams by applying aberration-corrected electron microscopy to characterise grain boundary structures and compositions (i.e. complexions) in Eu-doped MgAl₂O₄. Eu doping concentrations of 100 ppm and 500 ppm were used and bulk samples were annealed at 1600 C for different times (0 and 2 h for 500 ppm and 2 and 8 h for 100 ppm). Forty-five distinct grain boundaries were characterised. At least five grain boundary complexion types were identified and the grain boundary excess model was validated. Interpretability of the grain boundary excess model and correlations between grain boundary structures and compositions are discussed.

213506

INFLUENCE OF CARBON ON THE MICROSTRUCTURE AND WEAR RESISTANCE OF ALUMINAMarder R; Ghosh P; Reimanis I; Kaplan W D - *Technion-Israel Institute of Technology; Colorado, School of Mines***J.Am.Ceram.Soc.** 104, No.8, 2021, p.4214-4225

The effect of carbon as a dopant on the grain growth and wear resistance of polycrystalline alumina was studied. Carbon was introduced into alumina by sintering in a carbon-rich environment (graphite furnace under flowing He) and/or by residual carbon from organic binders used during green body consolidation. Samples were sintered at 1600 C for 2 h. Doping alumina with carbon resulted in a reduced grain size after sintering, correlated to solute-drag, and graphite particle-drag for high concentrations of carbon (about 3 wt%). The material response to abrasive wear was quantified by measuring the sample area cut for a defined time using a diamond wafering saw, as a function of grain size and carbon content. Sintering alumina with carbon resulted in a significant increase in wear resistance, as a result of the reduced grain size.

213507

RESEARCH PROGRESS AND PROSPECTS OF RARE-EARTH DOPED SESQUIOXIDE LASER CERAMICSLiu Z; Ikesue A; Li J - *Shanghai, Institute of Ceramics; Beijing, University of Chinese Academy of Sciences; World-Lab.Co.Ltd.***J.Eur.Ceram.Soc.** 41, No.7, 2021, p.3895-3910

The fabrication of rare-earth ion doped-sesquioxide ceramics, including the synthesis of raw nanopowders and ceramic sintering is reviewed. Results showed that the ceramics fabricated by vacuum sintering plus hot isostatic pressing from well-dispersed nanopowders possessed high transparency with uniform microstructures but further investigation is required. Progress on the laser performance of the corresponding transparent ceramics revealed that rare-earth active ion-doped sesquioxide ceramics have great potential in efficient high power and ultrashort pulse laser applications for about 1 to 3 micron.

213508

HIERARCHICALLY POROUS ALUMINA CATALYST CARRIER WITH BIOMIMETIC VEIN STRUCTURE PREPARED BY DIRECT INK WRITINGHuo C; Tian X; Chen C; Zhang J; Nan Y; Zhong Q; Huang X; Hu J; Li D - *Xian Jiaotong University; PetroChina Co.Ltd.***J.Eur.Ceram.Soc.** 41, No.7, 2021, p.4231-4241

A novel structural bionic, 3-D printing strategy and a pseudoboehmite-based slurry for the fabrication of hierarchical alumina ceramic carriers, with biomimetic vein structures, is reported. A tuneable hierarchical porosity from nm to mm scales, high specific surface area, and superior strength were achieved by combining the dehydroxylation and peptisation of pseudo-boehmite, direct ink writing (DIW) and post-sintering processes. The effects of post-sintering temperature and impregnation treatment on the crush strength, porosity and specific surface area were investigated. The optimised porous ceramics had a crush strength of 54.453 plus or minus 7.359 N/cm, a specific surface area of 109.870 plus or minus 0.143 m²/g and a porosity of 60.03 plus or minus 2.72%. A catalytic test for the dry reforming of methane was conducted on biomimetic catalysts loaded with nickel (Ni) and phosphorus (P). The biomimetic catalyst showed a methane conversion of 41.27%, in addition to mass transfer and coke inhibition effects. Using the integrated biomimetic design of macro-micro structures, monolithic alumina catalyst carriers, with biomimetic vein structures, prepared by DIW could be used more efficiently in industrial catalytic reactions.

213509

MECHANICALLY ISOTROPIC ALUMINA PREPARED BY SPARK PLASMA SINTERING: THE ROLE OF PYROLYTIC CARBON AND MULTILAYER GRAPHENEFan X; Ni N; Wang X; Hao W; Guo F; Zhao X - *Shanghai, Jiao Tong University; Konca Solar Cells Co.Ltd.***J.Eur.Ceram.Soc.** 41, No.7, 2021, p.4242-4251

The SPS preparation of isotropic alumina by adding pyrolytic carbon, with and without multilayer graphene, as sintering additives is reported. Anisotropy in the grain morphology, elastic modulus, microhardness and fracture toughness (KIC) was evaluated by comparing measurements along directions parallel and perpendicular to the external electric field in SPS. Results showed that anisotropy in the first three parameters was insignificant except for KIC. {0001} fibre texture contributed to the anisotropy in pure alumina. With pyrolytic carbon additions, equiaxed grains in alumina were refined mainly due to a pinning effect and KIC increased due to induced microcracking. The anisotropy of KIC in the composite was reduced due to scattering of the {0001} fibre texture. When multilayer graphene, with a micron in-plane size was further introduced, anisotropy in KIC was reduced at the cost of embrittlement.

213510
EMULSION PREPARATION OF ULTRALIGHT TiO₂ FOAMS FOR SELECTIVE OIL ABSORPTION
 Chuang C-Y; Zhang M-H; Tseng W J - *Taiwan, National Chung-Hsing University*
J.Eur.Ceram.Soc. 41, No.7, 2021, p.4349-4354
 Ultralight TiO₂ foams were prepared by an emulsion route using mesoporous TiO₂ microbeads (MTMs) as a raw material in water followed by mechanical frothing and calcination at elevated temperatures. The MTMs were modified by the preferential adsorption of hexadecylamine in ethanol prior to the emulsion practice so that the surface became hydrophobic for facilitating the formation of stable air-in-water bubbles. The TiO₂ foams had cellular spherical macropores with an interconnected pore structure with a pore-size range from about 10 to 400 micron and a porosity ranging from 85.8 to 95.6 % (corresponding to a bulk density of 0.06 to 0.24 g/cm³). The foams could float on oil/water mixtures and absorb oil selectively after short period of time without noticeable degradation under recycled use, e.g., the absorption of soybean oil with up to 15 times the foam weight was obtained in less than 30 s after the third use.

213511
HIERARCHICALLY CELL-WINDOW STRUCTURED POROUS CORDIERITE PREPARED BY PARTICLE-STABILISED EMULSIONS USING POTATO STARCH AS A MODIFIER
 Luan X; Li J; Wang Z; Feng W; Huang K; Liu S - *Shenyang, University; China, University of Geosciences*
J.Eur.Ceram.Soc. 41, No.7, 2021, p.4369-4380
 Porous cordierite ceramics (PCCs) with hierarchical microstructures were prepared by the particle-stabilised emulsion method using potato starch (PS) as a modifier. The formation of the hierarchical structure was ascribed to the removal of oil bubbles and the consumption of PS during the sintering process. The volume shrinkage of samples tended to be stable with a solids content of 40 vol%. When the PS content was 25 wt%, PCCs with a controlled hierarchical pore structure of up to 91.4% porosity, a compressive strength of 5.76 MPa and a pore size of 6.19 micron was obtained. The thermal conductivity of the PCCs was optimised at 0.4325 W/m K for a pore size distribution showing a single peak at the same time. The chemical corrosion of PCCs in strong acid and alkaline media was inhibited by the stable cordierite phase. This method of preparing PCCs, with a controlled hierarchical structure, has potential applications in catalyst carriers and filtration.

ABRASIVES

See also Abstract(s): 213333

ELECTROCERAMICS

See also Abstract(s): 213386 213390 213398 213399 213400 213404 213409 213423 213493 213504 213671 213692

213512
THERMALLY STABLE PIEZOELECTRIC PERFORMANCE IN LOW-TEMPERATURE SINTERED Pb_{0.95}Ba_{0.01}Sr_{0.04}(Zr_{0.53}Ti_{0.47})O₃ CERAMICS WITH A LOW LOSS FACTOR
 Du Z-Z; Liu Y-X; Wei W-Q; Liu H; Jiang S-D; Fang J-Z - *Chinese Academy of Sciences; Beijing, Tsinghua University; Medcaptain Medical Technology Co., Ltd.*
Adv.Appl.Ceram. 120, No.4, 2021, p.209-214
 Pb_{0.95}Ba_{0.01}Sr_{0.04}(Zr_{0.53}Ti_{0.47})O₃ is used in this case. At a low firing temperature of 900 C, O₃ piezoelectric ceramics containing 0.25 wt% MnCO₃, 1 wt% LiBiO₂, and 0.06 wt% CuO (PBSZT-LBCu-Mn ceramics, abbreviated as Mn0.25) were produced using a standard technique. The phase structure as well as the temperature-dependent electrical characteristics were thoroughly studied. The following attributes were included in the product composition: normalised strain (d₃₃^{*}) 362 pm/V, Curie temperature (T_c) 336 C, mechanical quality factor (Q_m) 568, loss factor (tan δ) 0.0058, and relative dielectric constant (ε_r) 872. The remnant polarisation (P_r), maximum polarisation (P_{max}), and coercive field (E_c) reduced as temperature increased as the strain (S_{pol}) increased. Furthermore, d₃₃^{*} values of 362 and 411 pm/V were obtained at 25 and 120 C, respectively, with a change ratio of 14%. Mn0.25 ceramics have low ε_r and tan δ values, resulting in low average power dissipation, which is advantageous for high-frequency actuator applications.

213513
NOVEL INTERMEDIATE TEMPERATURE SOLID OXIDE FUEL CELL BASED ON La-DOPED Bi₄V₂O₁₁ ELECTROLYTE
 Pasciak G; Chmielowiec J - *Wroclaw, Institute of Low Temperature & Structural Research*
Adv.Appl.Ceram. 120, No.4, 2021, p.215-221
 It was explored if an electrolyte-supported intermediate temperature solid oxide fuel cell (IT-SOFC) based on Bi₄V₂O₁₁ (BIVOX) ceramic stabilised by La doping might be used. The excellent oxygen ion-conductive phase has been stabilised by adding La to the basic BIVOX composition. Due to the obvious improved thermodynamic stability of the BIVOX electrolyte in decreasing (H₂ - fuel) environment, the addition has considerably prolonged the lifespan of SOFC. Open circuit voltage (OCV), maximum power density (P), and current density (J) in the temperature range 500-800 C, as well as running duration under constant electronic load, were measured for fuel cells. The results of these electrical parameter tests for BILAVOX-based single cells as a function of operating time and cyclic load variations were also displayed. This is the first study on the design and electrical characteristics of BIMEVOX (BIVOX doped with METal) ceramic fuel cells.

213514
DRYING STABILITY ENHANCEMENT OF RED-PEROVSKITE COLLOIDAL INK VIA LIGAND-DERIVED COATING FOR INKJET PRINTING
 Yoo J H; Jeong S G; Choi S H; Kwon S B; Song Y H; Yoon D H - *Suwon, Sungkyunkwan University; Korea Photonics Technology Institute*
Ceram.Int. 47, No.5, 2021, p.6041-6048
 The drying stability of red perovskite colloidal ink was improved by inducing aggregation during the drying process. A silane ligand was used to induce the aggregation and a thin coating layer was formed on the perovskite surface. The synthesised red-perovskite, using silane ligands, was well dispersed in the solvent as in a colloidal state and an emission intensity above 50% was maintained after drying. The as-prepared perovskite ink was successfully printed on an ethyl cellulose film with 338 dpi while maintaining the transparency of the film. These results are beneficial not only

for display applications but also security applications including double anti-counterfeiting.

213515

EFFECTS OF Sn⁴⁺ DOPING AND OXYGEN VACANCY ON MAGNETIC AND ELECTRICAL PROPERTIES OF YTTRIUM IRON GARNET PREPARED BY SOL-GEL METHOD

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Ceram.Int. 47, No. 5, 2021, p. 6442-6452

Sn-substituted yttrium iron garnet samples, Y₃Fe_{5-x}Sn_xO₁₂ (x = 0-0.1, step 0.02), were prepared using a citrate sol-gel method followed by sintering. XRD, XANES, SEM and FTIR were used to investigate the structural parameters, valence state of Fe, oxygen vacancies and lattice distortion in the samples. The magnetic properties of the samples were measured using SQUID and VSM. Sn substitution and oxygen vacancies caused the transformation of Fe³⁺ to Fe²⁺ which led to a reduced Curie temperature and a slight increase in the saturation magnetisation. The temperature dependence of the resistivity in the 300-573 K range was investigated to elucidate the conduction mechanism in the samples. The resistivity of the sol-gel derived samples was nine orders of magnitudes lower than the value for the bulk sample prepared by the flux-grown method. The effects of Fe²⁺ centres, lattice dislocation, porosity and grain boundaries on the resistivity are discussed. This study indicated that Sn-substituted yttrium iron garnets could have applications in sensor elements that operate based on electrical signals.

213516

FABRICATION OF ELECTROCHROMIC DEVICES BY LASER PATTERNING OF SPIN-SPRAYED TRANSPARENT CONDUCTIVE Ga:ZnO FILMS

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Ceram.Int. 47, No. 5, 2021, p. 6470-6478

A novel spin-spraying method for growing Ga-doped ZnO (Ga:ZnO) films on glass substrates is reported. The optimum Ga doping concentration of the spin-sprayed Ga:ZnO (SSedGZO) films was determined from the electrical and optical properties of the films. The feasibility of using UV laser patterning to produce patterned SSedGZO films for application as transparent electrodes (TEs) in optoelectronic devices was evaluated, and the optimum laser power required for effective patterning was determined by analysis of the surface microstructures of the patterned films. Finally, to confirm the applicability of the SSedGZO films as TEs, electrochromic devices (ECDs) comprising the SSedGZO TEs were fabricated. The laser-patterned SSedGZO TEs functioned successfully in the fabricated ECDs, and displayed reversible colour changes during electrochromic switching.

213517

STRAIN MEDIATED ENHANCEMENT IN MAGNETOELECTRIC PROPERTIES OF SONOCHEMICALLY SYNTHESISED PIEZOELECTRIC AND PIEZOMAGNETIC COMPOSITES

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Ceram.Int. 47, No. 5, 2021, p. 6496-6504

Three-dimensional magnetoelectric (ME) nanocomposites, with the general formula (1-x)CoCr_{0.3}Fe_{1.7}O₄(CCFO)+(x)BaTiO₃(BTO) (x = 0.0, 0.2, 0.4, 0.6, 0.8 and 1.0), were obtained using piezoelectric-BTO and piezomagnetic-CCFO phases. The individual CCFO and BTO phases were synthesised separately by ultrasonic irradiation-assisted sonochemical and sol-gel routes. XRD confirmed the crystalline nature of both phases. BTO and CCFO phases were under tensile strain as confirmed by the variation in lattice constants with varying proportions of BTO and CCFO. EDS confirmed the phase purity of the samples and stoichiometric concentration of the elements. Magnetic properties were investigated by M-H loop measurements and dielectric properties using a RF impedance analyser. The dielectric constant increased with increased BTO percentage. The maximum ME coefficient (24.7 mV/cm Oe) was observed for the 60%CCFO + 40%BTO sample. The results are discussed in the light of the grain size, strain and the basic properties of the individual phases. The prepared materials could have electronic applications where a high magneto-electric coefficient is desirable.

213518

SOLUTION-PROCESSED La-Zr-O DIELECTRIC AT A LOW TEMPERATURE FOR HIGH-PERFORMANCE In-Ga-O TRANSISTORS: ENGINEERING A PRECURSOR SOLUTION

Lee S; Lee S-H; On N; Jeong J K - *Hanyang, University*

Ceram.Int. 47, No. 5, 2021, p. 6918-6927

The effect of the solution stirring temperature and H₂O₂ loading on the structural and electrical properties of lanthanum zirconium oxide (LZO) dielectric films was investigated. The surface roughness, mass density and purity of H₂O₂-assisted LZO improved with increased stirring temperature which resulted in a low leakage current of 4 x 10⁻⁸ A/cm² at 2 MV/cm, a high-breakdown electric field of about 5.8 MV/cm, and relatively high permittivity (>12) at a low photochemical activation temperature of 180 C for optimised LZO dielectric films. This could be attributed to the synergetic interaction of oxygen radicals introduced by H₂O₂ oxidants during deep UV photochemical annealing, which caused hydrolysis of metal precursors, the removal of organic residue and the formation of a metal-oxide lattice. The fabricated indium gallium oxide thin-film transistor, on a polyimide substrate at a maximum processing temperature of 180 C, exhibited a high mobility of 14.5 cm²/Vs, a high I(ON/OFF) ratio of 10^{exp(7)}, negligible hysteresis, and a small driving voltage range (-2 to 2 V).

213519

STRUCTURAL ANALYSIS AND MICROWAVE DIELECTRIC PROPERTIES OF A NOVEL Li₂Mg₂Mo₃O₁₂ CERAMIC WITH ULTRA-LOW SINTERING TEMPERATURE

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Ceram.Int. 47, No. 5, 2021, p. 7081-7087

Single orthorhombic phase Li₂Mg₂Mo₃O₁₂ ceramics, as novel ultralow sintering temperature ceramics, were investigated and synthesised using a solid-state method. The optimum sintering temperature was 650 C, with a high relative density of 97.86%. The structure of the Li₂Mg₂Mo₃O₁₂ ceramic was characterised by Rietveld refinement, XPS, Raman spectroscopy and FTIR. Rietveld refinement and XPS results showed that Mo⁶⁺ was located in the near-ideal MoO₄ tetrahedrons and Mg²⁺ and Li⁺ were distributed in the centre of three types of AO₆ polyhedrons. Raman and

FTIR studies confirmed the internal vibrational features of the $\text{Li}_2\text{Mg}_2\text{Mo}_3\text{O}_{12}$ ceramics. Correlations between the structural and microwave dielectric losses of the $\text{Li}_2\text{Mg}_2\text{Mo}_3\text{O}_{12}$ ceramics were analysed by Raman spectroscopy. The lower FWHM of the Raman peak indicated a lower intrinsic dielectric loss. The $\text{Li}_2\text{Mg}_2\text{Mo}_3\text{O}_{12}$ ceramics exhibited optimal microwave dielectric properties when sintered at 650 C with $\epsilon_r = 9.26$, $Q \times f = 58583$ GHz, and $\tau_f = -51$ ppm/C. $\text{Li}_2\text{Mg}_2\text{Mo}_3\text{O}_{12}$ ceramics may have ULTCC applications.

213520

WSe₂/2D ELECTRON GAS HETEROJUNCTION ON KTaO₃ FOR ROOM-TEMPERATURE GIANT PHOTOCONDUCTIVITYZhou Z; Huang G; Shen J; Gong S; Zhou P; Yin L; Zhao R; Liu G; Zhang J; Li Y; Ma C; Jiang Y - *Suzhou, University of Science & Technology***Ceram.Int.** 47, No. 6, 2021, p. 7425-7429

A Van der Waals (vdW) heterojunction was fabricated between WSe₂ and 2DEG on KTaO₃. Under visible light illumination, giant photoconductivity was observed with a bias voltage applied at room temperature. Such a device exhibited an instantaneous photoelectric response to on/off light illumination and the ratio of on/off currents reached $10 \exp(4)$. In the 405-808 nm wavelength range, the photoresponsivity was greater at shorter wavelengths and reached a maximum of 1.84 A/W. Moreover, even without a bias voltage, the WSe₂/2DEG heterojunction still generated a short-circuit photocurrent due to the photovoltaic effect, which implied a self-powered photodetector. This work paves the way towards high-performance photoelectric devices based on vdW heterojunctions of 2D-material/2DEG.

213521

STABILISING NANOCRYSTALLINE Cu₂O WITH ZnO/rGO: ENGINEERED PHOTOELECTRODES ENABLES EFFICIENT WATER SPLITTINGShanmugasundaram A; Ali Johar M; Boppella R; Kim D-S; Jeong Y-J; Kim J Y; Hassan M A; Ryu S-W; Lee D W - *Chonnam, National University; Illinois, University at Urbana Champaign; Indian Institute of Chemical Technology***Ceram.Int.** 47, No. 6, 2021, p. 7558-7570

A highly effective photoelectrode based on Cu₂O decorated with ZnO and rGO, for efficient photoelectrochemical (PEC) water splitting, was prepared. Initially, different thicknesses of Cu₂O were sputtered on the FTO substrate (FC). The PEC performance of the FC photoelectrode was further improved by depositing ZnO and rGO protection layers (FCZG). The fabricated photoelectrodes were investigated for their morphological and crystal structure using AFM, FE-SEM, TEM, XPS, XRD and Raman, UV-Vis DRS and photoluminescence (PL) analyses. The FCZG hybrid photoelectrode had a photocurrent density of 4.94 mA/cm² at 0 V vs. reversible hydrogen electrode (RHE), which was 1.5 times higher than the unmodified photoelectrodes. The improved PEC performance of the FCZG hybrid photoelectrode was due to the high surface roughness, larger electrochemical active surface area and reduced radiative recombination rate of the photogenerated charge carriers.

213522

DIRECT INK WRITING OF BISMUTH MOLYBDATE MICROWAVE DIELECTRIC CERAMICSGoulas A; Chi-Tangye G; Zhang S; Wang D; Ketharam A; Vaidhyathan B; Reaney I M; Cadman D A; Whittow W; Vardaxoglou J C; Engstrom D S - *Loughborough, University; Sheffield, University***Ceram.Int.** 47, No. 6, 2021, p. 7625-7631

Additive manufacturing via direct ink writing and microwave dielectric characterisation of commercially produced low sintering temperature bismuth molybdenum oxide ceramics were performed for the first time, following a powder-to-product holistic approach. It was shown that direct ink writing is an excellent candidate for producing dielectric substrates for wireless telecommunication applications operating at microwave (MW) frequencies, with great repeatability and properties comparable to ceramics fabricated via conventional processing routes. The optimum density (relative density of about 93%) of the 3-D printed test samples was obtained after sintering at 660 C for 2 h, which resulted in a relative permittivity $\epsilon_r = 35.7$, a dielectric loss $\tan \delta = 0.0004$ and a microwave quality factor $Q \times f = 14928$ GHz. Sintering at higher temperatures resulted in increased porosity due to mismatched grain growth mechanisms and phase decomposition, that hindered the test samples' microwave dielectric performance in terms of achievable relative permittivity (ϵ_r) and dielectric loss ($\tan \delta$).

213523

HIGH-DENSITY SOL-GEL DERIVED, COLD-ISOSTATICALLY PRESSED La_{0.67}Ca_{0.27}Sr_{0.06}MnO₃ POLYCRYSTALLINE CERAMICS AND THEIR ROOM-TEMPERATURE TCR IMPROVEMENTLiu Y; Dong G; Zhang S; Liu X - *Kunming, University of Science & Technology***Ceram.Int.** 47, No. 6, 2021, p. 7674-7682

High-density La_{0.67}Ca_{0.27}Sr_{0.06}MnO₃ (LCSMO) ceramics were obtained by the sol-gel method followed by cold isostatic pressing (CIP) under 250 MPa. The effects of sintering temperature (TS, TS = 1250 C to 1450 C) on the structure, surface chemical, electrical and optical properties of as-obtained LCSMO ceramics were investigated using XRD, XPS, FTIR, Raman microscopy and UV-VIS spectrophotometry, respectively. All the samples had an orthorhombic structure with a Pnma space group. As TS increased, the grain size increased from 2.71 to 7.30 micron. XRD refinement, Raman and FTIR showed a constant bond angle and length of Mn-O with no lattice distortion. The optimum electrical properties were obtained at 1450 C, where resistivity (ρ) fell below 0.008 Ohm cm and the temperature coefficient of resistivity (TCR) reached 13.0% /K at 289.2 K (room temperature). The electrical performance was improved due to the high density of the samples processed by CIP and the change in TS from 1250 to 1450 C influenced the electrical behaviour but had little effect on the structural and optical properties of the LCSMO ceramics.

213524

IMPROVING OPTICAL AND ELECTRICAL PERFORMANCES OF ALUMINIUM-DOPED ZINC OXIDE THIN FILMS WITH LASER-ETCHED GRATING STRUCTURESHuang L-J; Zhao L; Li B-J; Zhang Y; Wang Y-I; Wang Y-y; Ren N-f; Song J - *Jiangsu, University***Ceram.Int.** 47, No. 6, 2021, p. 7994-8003

A laser etching method was used to achieve the dual purpose of fabricating grating structures and laser annealing on aluminium-doped zinc oxide (AZO) thin films, to improve the photoelectric performance of the films. Different line spacings and laser fluences were adopted to explore the optimal laser etching conditions. Too narrow line spacings or too high laser fluences led to light reflections at the grating external surface which caused more light dissipation, and too wide line spacings or too low laser fluences resulted in relatively small total grating lateral areas being detrimental to multiple internal light reflections. Moreover, too narrow line spacings brought about laser-caused lattice disorder and too high laser fluences produced laser-ablated spots or overburned traces. Therefore, using the medium line spacing and laser fluence, e.g. 40 micron and 0.6

J/cm² in the present work, was more suitable for realising grating structure fabrication and laser annealing. The corresponding AZO film had a maximum figure of merit of $2.89 \times 10 \exp(-2)$ /Ohm, which was 1.6 times that of the untreated AZO film. This study is expected to improve methods for TCO films and promote the application of laser-etched grating structures.

213525

THREE-PHASE BORATE SOLID SOLUTION WITH LOW SINTERING TEMPERATURE, HIGH-QUALITY FACTOR, AND LOW DIELECTRIC CONSTANT

Peng R; Li Y; Su H; Lu Y; Shi L; Yu G; Wang G; Gan G; Yu C - *Chengdu, University of Electronic Sci. & Technology; Huzhou, University of Electronic Sci. & Technology; Hangzhou, China Jiliang University*

J.Am.Ceram.Soc. 104, No.7, 2021, p.3303-3315

The sintering and microwave dielectric properties of a ceramic material based on the mixing of Mg₃B₂O₆ and Zn₃B₂O₆ were studied using first-principles calculations and experimental solid-state reactions. Characterisation methods included network analysis, XRD, Raman diffraction, SEM, EDS, DTA and thermomechanical analysis. Increasing Mg²⁺ content resulted in the appearance of Mg₂B₂O₅ and ZnO, and the mutual substitution (Mg²⁺ and Zn²⁺) phenomenon emerged in Zn₃B₂O₆ and Mg₂B₂O₅. The mechanisms were explained using DFT calculations. The bond parameters and electron distributions of the ZnO₄ tetrahedron and MgO₆ octahedron were modified due to substitution. The sintering, substitution and phase formation properties were analysed quantitatively through the energy parameters. The best dielectric properties ($\epsilon_{\text{r}} = 6.47$, $Q \times f = 89600$ GHz (15.2 GHz), $\tau_{\text{f}} = -48.6$ ppm/C and relative density = 96.7%) were obtained for $x = 0.20$ sintered at 950 C. The mixing of Zn₃B₂O₆ and Mg₃B₂O₆ ceramics can thus be performed to obtain a ceramic with low sintering temperature and excellent dielectric properties.

213526

DISCOVERY OF THE GRIFFITHS PHASE IN THE QUATERNARY NITRIDES Ge_{1-x}Sn_xNFe₃

Liu C; Kan X; Liu X; Feng S; Hu J - *Anhui, University*

J.Am.Ceram.Soc. 104, No.7, 2021, p.3387-3396

The Griffiths phase (GP) and transport behaviour in the quaternary antiperovskite nitrides Ge_{1-x}Sn_xNFe₃ ($x = 0-1$) were studied using a series of systematic magnetic susceptibility and thermoelectric behaviour measurements. All the corresponding fitting exponent parameters $\lambda_{\text{GP}} = 0.852$ and 0.854 completely satisfied $\lambda = 0-1$ in the GP regions for Ge_{0.7}Sn_{0.3}NFe₃ and Ge_{0.8}Sn_{0.2}NFe₃, respectively, indicating the different ferromagnetic concentrations in the system. An observed structural transition from distorted tetragonal structure (I4/mcm) to cubic antiperovskite (Pm3m) with increasing Sn doping concentration should be responsible for the GP in Ge_{1-x}Sn_xNFe₃, which resulted in a magnetic phase transition from ferromagnetism (FM) to antiferromagnetism (AFM). Studies of electrical transport properties indicated that the Ge_{1-x}Sn_xNFe₃ series of compounds performed a Fermi liquid behaviour at low temperature while the stronger electron-phonon scatterings were found in the high-temperature region. The intensive electron-electron correlation was also determined with the value of the lattice thermal conductivity being much higher than that of electronic thermal conductivity.

213527

HIGHLY LUMINESCENT CdS NANOPARTICLES SYNTHESISED USING MICROWAVE IRRADIATION OF DITHIOCARBAZATE LIGAND AS A SINGLE MOLECULAR PRECURSOR SOURCE

Sharma R; Singh R; Goswami Y C; Kumar V; Kumar D - *Gwalior, ITM University; Gwalior, SMS Government Science College*

J.Aust.Ceram.Soc. 57, No.3, 2021, p.697-703

The microwave-assisted synthesis of highly luminescent cadmium sulphide nanocrystals using the dithiocarbazate Cd(II) chloride complex single-molecule precursor route is reported. The precursors were heated by microwave irradiation to obtain CdS nanocrystals. The irradiation of the precursor in a DMSO environment played an important role in reducing the reaction time, minimising the possibility of side reactions, and leading to good quality particles. The obtained samples were analysed. XRD confirmed the presence of a crystalline hexagonal phase. HREM and TEM revealed rod-shaped structures with a mean particle size of 80 nm. A sharp absorption edge at 320 nm showed that the particles were of good quality. Strong luminescence peaks in the visible wavelength range of 450-490 nm were seen in PL spectra. Another luminescence peak at 680 nm for excitation at 320 nm indicated the involvement of deep donor and acceptor levels in the crystals. The luminescence spectra show a strong dependence on excitation wavelength, making these particles suitable for various optoelectronic applications.

213528

STRUCTURAL AND MORPHOLOGICAL CHARACTERISATION OF THE PEROVSKITE LaFe_{0.2}Cr_{0.8-x}CoxO₃ (X = 0.0, 0.2, 0.4, 0.6, 0.8) FOR SELECTIVE OXIDATION OF CO

Rativa-Parada W; Gomez-Cuaspad J A; Schmal M; Cruz-Pacheco A F; Vera-Lopez E - *Colombia, Universidad Pedagogica y Tecnologica; Rio de Janeiro, Federal University; Medellin, Universidad de Antioquia*

J.Aust.Ceram.Soc. 57, No.3, 2021, p.767-781

Five compositions in the LaFe_{0.2}Cr_{0.8-x}CoxO₃ ($x = 0.0, 0.2, 0.4, 0.6, 0.8$) system were synthesised by a combustion route to investigate the structure and morphology of doped LaCrO₃ perovskite-type oxides. The optimal conditions for synthesising the materials from citrate precursors at low temperatures were determined by TGA-DTA. XRD and SEM/TEM of the calcined oxides confirmed the formation of a single perovskite structure in the materials. Incorporation of cobalt into the structure was associated with the evolution from a rhombohedral R3-c to an orthorhombic Pnma 62 structure, with nanoparticle sizes and relevant surface properties. Raman and XPS results showed that the chemical composition and electronic state of the cations were in accordance with the proposed system and method of synthesis. The conversion of CO and O₂ was between 83.5 and 100% for all oxides, depending on the cobalt content, and the positive effect of temperature on the conversion and selectivity for CO₂ formation. Stability tests confirmed a stable conversion of CO and O₂, with high initial values around 96% plus or minus 0.97, falling to 83.5% plus or minus 0.87 for the less reactive materials.

213529

EFFECT OF ANNEALING TEMPERATURE ON HYDROTHERMALLY GROWN SISAL-LIKE ZnO MICROSTRUCTURES

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J.Aust.Ceram.Soc. 57, No.3, 2021, p.993-1002

Sisal-like ZnO microstructures were fabricated and characterised by FESEM, XRD, FTIR and Raman spectroscopy. An HMT aqueous solution

containing zinc nitrate tetrahydrate as the crystalline zinc source was used to synthesise the samples via a controlled hydrothermal route, and the effect of annealing temperature on the morphology and structure was investigated. FESEM showed a transition from hexagonal to pointed microrods with sisal-like microstructures as the temperature increased. XRD indicated considerably high purity for the synthesised microstructures. The crystallinity of the samples was estimated using various X-ray line broadening methods including Debye-Scherrer and Williamson-Hall techniques. The optical properties were studied using UV-Vis DRS measurements. The results showed improved light harvesting related to both enhanced absorption intensities and decreased band-gap energies with increasing annealing temperature.

213530

3D ORDERED NANO-ELECTRODES FOR ENERGY CONVERSION APPLICATIONS: THERMOELECTRIC, PIEZOELECTRIC, AND ELECTROCATALYTIC APPLICATIONS

Kim K; Tiwari A P; Novak T G; Jeon S - Korea, *Advanced Institute of Science & Technology*

J. Korean Ceram. Soc. 58, No. 4, 2021, p. 379-398

Although many methods have been suggested to improve the performance of materials in various applications by applying new physical and chemical properties at the nanometre scale, in the form of nanodots, nanowires and nanofilms, most of the proposed methods are difficult to apply to industrial settings due to their size limitations. The realisation of 3D nanostructured materials is significant for the practical use of nanotechnology. The continuous 3D nanostructuring ensures the maximum utilisation of materials efficiency and improves the stability through well-ordered structures. 3D nanostructures of materials can be useful for energy conversion applications such as thermoelectric, piezoelectric and electrocatalytic applications. 3D nanofabrication methods to convert the materials in the 3D nanostructures and the advantages of 3D ordered nanoelectrodes for high-performance energy conversion applications are reviewed. Conversion techniques include atomic layer deposition, electroplating, electroless plating and wet and dry chemistry.

213531

EFFECT OF EXCESS Ge AND Te ON THERMOELECTRIC PERFORMANCE OF GeTe

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Int. J. Appl. Ceram. Technol. 18, No. 4, 2021, p. 1144-1152

GeTe is an excellent medium-temperature thermoelectric material, the performance of which is affected by the carrier concentration generated by Ge vacancies. The effect of excess Ge or Te on the thermoelectric performance was studied. $GexTey$ ($x:y = 1:1.08, 1:1.06, 1:1.04, 1:1, 1.05:1, 1.075:1$ and $1.1:1$) were fabricated by high-pressure sintering (HPS) and spark plasma sintering (SPS), respectively, to study the effects of different Ge/Te atomic ratios and preparation routes on the thermoelectric properties of polycrystalline GeTe. The composition and microstructure were analysed by XRD and FESEM. The thermoelectric performance was tested in the range 303-703 K. The Seebeck coefficient of $GexTey$ increased and the conductivity decreased with decreasing Te content or increasing Ge content. $Ge1Te1$ showed the highest power factor since its Seebeck coefficient and conductivity are at an average level. Due to the presence of pure Ge and the decrease in Ge vacancies, the lattice thermal conductivities of samples with excess Ge are higher than that of $Ge1Te1$. $Ge1Te1$ sintered by HPS had the highest ZT(max) value, reaching 1.37 at 723 K.

213532

FAST RESPONSE AND HIGH STABILITY Mn-Co-Ni-Al-O NTC MICROBEADS THERMISTORS

Li H; Zhang H; Chang A; Ma X; Xie J; Yang L - Xinjiang, *Technical Institute of Physics & Chemistry; Beijing, University of Chinese Academy of Sciences; Xinjiang, University*

J. Am. Ceram. Soc. 104, No. 8, 2021, p. 3811-3817

Al-doped $Mn_{1.95}Co_{0.21}Ni_{0.84}O_4$ (MCN)-microbead structures were prepared by in-situ ink-jet printing. The effects of Al_2O_3 doping on the electrical properties of the MCN ceramic microbeads are discussed. The R_{25} , $B_{25/50}$ and E_a values of NTC ceramic thermistors ranged from $14.14 \times 10 \exp(3) - 63.21 \times 10 \exp(3)$ ohm, 3635-3773 K and 0.3133-0.3252 eV, respectively. The ageing coefficient ($\Delta R/R$) of the (Mn-Co-Ni-Al-O) MCNA ceramics was $< 1.1\%$, compared to a value $> 4.2\%$ for the undoped MCN microbeads. The thermal time constant of the doped microbeads was small (33 ms) compared to 8 s for the MCN ceramic chip. It was found that the grain boundaries were important in the overall electric behaviour of the ceramics.

213533

NEW LOW-EPSILONR, TEMPERATURE STABLE $Mg_3B_2O_6-Ba_3(VO_4)_2$ MICROWAVE COMPOSITE CERAMIC FOR 5G APPLICATION

Zhou T; Liu Y; Song K; Xue L; Xu P; Khesro A; Wang D; Liu B; Mao M; Shi F; Sun S - Hangzhou, *Dianzi University; Mardan, Abdul Wali Khan University; Shenzhen, Institute of Advanced Technology; Jinan, Qilu University of Technology; Foshan, University*

J. Am. Ceram. Soc. 104, No. 8, 2021, p. 3818-3822

Novel low-epsilon_r, thermal and phase stable $(1-x)Mg_3B_2O_6-xBa_3(VO_4)_2$ ($x \text{ mol}\% = 51, 53, 55, 57, 59$) microwave composite ceramics were fabricated by solid-state reaction. XRD, SEM, EDX and Raman spectroscopy confirmed the coexistence of both phases without other phases. A near-zero temperature coefficient of resonant frequency (τ_{f0} approximately $+1.2 \text{ ppm/C}$) was obtained for the $0.43Mg_3B_2O_6-0.57Ba_3(VO_4)_2$ composite ceramic, with epsilon_r of 8.8 and Qxf of 45420 GHz, making it a promising candidate for 5G applications.

213534

HIGH THERMOELECTRIC PERFORMANCE OF HIGH-MOBILITY Ga-DOPED ZnO FILMS VIA HOMOGENOUS INTERFACE DESIGN

Zhou Z; Zou M; Xu Y; Lan J; Liu C; Ahmad A; Lin Y-H; Nan C-W - Beijing, *Tsinghua University; Beijing, University of Chemical Technology*

J. Am. Ceram. Soc. 104, No. 8, 2021, p. 3992-3999

Ga-doped ZnO (GZO) thin films grown on sapphire substrates are promising transparent thermoelectric (TE) materials. However, due to the large lattice mismatch and thermal expansion between the sapphire substrate and GZO film, strain-induced lattice distortion impedes the transport of electrons, leading to low carrier mobility. ZnO homo-buffer layers with different thicknesses were inserted between sapphire substrates and GZO films, and their effect on the TE properties was studied. A thin ZnO interlayer (10 nm) effectively reduced the lattice mismatch of the GZO film and improved the carrier mobility, which helped to greatly enhance the electrical conductivity. Simultaneously, energy filtering occurred at the interface between GZO and ZnO, resulting in a relatively high density of states effective mass and maintaining a high Seebeck coefficient compared to that of the unbuffered GZO films. Consequently, the GZO film with a 10 nm thick ZnO buffer layer had a high power factor value of 449 $\mu\text{W/m.K}^2$ at 623

K. The study provides a facile and effective method to optimise the TE performance of oxide thin films by synergistically improving their carrier mobility and enhancing their effective mass.

213535

FERROMAGNETIC INSULATING BEHAVIOUR AT LOW TEMPERATURE INDUCED BY Sn DOPING IN THE CERAMIC SrRuO₃Liu Y; Zhang Z; Lv J; Qian P; Cai H; Wu X - *Nanjing, University***J.Am.Ceram.Soc.** 104, No.8, 2021, p.4086-4094

The effects of Sn doping at the Ru site on the structural, magnetic and transport behaviour of polycrystalline SrRu_{1-x}Sn_xO₃ (x up to 0.1) were studied. Substitution of Sn⁴⁺ for Ru⁴⁺ had the same crystal symmetry as that of Sn-free SrRuO₃, while it induced Ru(Sn)O₆ octahedral distortions. Samples with a low doping concentration (x up to 0.08) showed a metallic behaviour at high temperature, while a metal to insulator transition occurred at low temperature. However, an insulator behaviour was detected for the sample with x = 0.1, which followed an Arrhenius-type process in the temperature range 80-140 K and a Mott's variable range hopping model in the temperature range 140-300 K. It was also found that Sn⁴⁺ had a significant effect on the magnetic behaviour of Sn doping in SrRuO₃ where the ferromagnetic transition temperature and magnetic moment decreased rapidly due to octahedral distortion and site dilution.

213536

INVESTIGATION OF ELECTRICAL AND AGEING PROPERTIES OF Bi-MODIFIED (Zn_{0.4}Ni_{0.6})_{1-x}Na_xO CERAMIC THERMISTORSGao C; Li Z; Yang L; Peng D; Zhang H - *Changsha, Central South University***J.Eur.Ceram.Soc.** 41, No.7, 2021, p.4160-4166

Bi-modified (Zn_{0.4}Ni_{0.6})_{1-x}Na_xO (denoted as xZNN, x < 0.1) ceramics were prepared by a conventional solid-state reaction process. The xZNN ceramics had a rock-salt type structure. Besides the rock-salt phase, there was a monoclinic Bi₂O₃ secondary phase coexisting in the Bi-modified xZNN ceramics. The ceramics exhibited a typical characteristic with a negative temperature coefficient (NTC) of resistivity. The NTC material constants (B values) of all the prepared ceramics were higher than 4000 K. The room temperature resistivity and B values could be adjusted over a large range by altering either or both contents of Na-ions and Bi-ions. Bi₂O₃ additions enhanced the sinterability and reduced the sintering temperature from 1350 to 1150 C and improved the ageing stability by reducing the drop resistance drift rate from 237 to 1.8 %. The ageing characteristics are discussed based on models of space charge distribution and interfacial barriers.

213537

NOVEL CORE-SHELL STRUCTURE NTC CERAMIC WITH HIGH STABILITY FABRICATING BY AN IN-SITU INK-JET PRINTING METHODLi H; Zhang H; Chang A; Ma X; Rong J; Yang L - *Xinjiang, Technical Institute of Physics & Chemistry; Beijing, University of Chinese Academy of Sciences; Xinjiang, University***J.Eur.Ceram.Soc.** 41, No.7, 2021, p.4167-4174

Mn_{1.95}Co_{0.21}Ni_{0.84}O₄ (MCN) microsphere-based core/Al₂O₃-shell structures were prepared using in-situ ink jet printing technology. The ceramic inks consisted of MCN and Al₂O₃ nanocrystallites with a particle size of about tens of nanometres with excellent printability. The effects of the Al₂O₃ shells and the interfacial region on the electrical properties compared to the MCN core ceramic are discussed. Moreover, the R₂₅, B_{25/50}, and E_a values of the core-shell composites were from 8.3 x 10 exp(3) to 4.963 x 10 exp(4) Ohms, 3448-3636 K, 0.2972-0.3134 eV, respectively. The ageing coefficient (DeltaR/R) of the composites was <0.54 %, while the ageing coefficient exceeded 2.22 % for the conventional MCN microspheres. The diffusion layer (about 2-4 micron) played an important role in the overall electric behaviour of the composites.

Ferrites

See also Abstract(s): 213438

213538

INNOVATIVE METHODOLOGY FOR CO-TREATMENT OF MILL SCALE SCRAP AND MANGANESE ORE VIA OXIDISATION ROASTING-MAGNETIC SEPARATION FOR PREPARATION OF FERRITE MATERIALSLiu B; Zhang Y; Han G; Zhang B - *Zhengzhou, University; Changsha, Central South University***Ceram.Int.** 47, No.5, 2021, p.6139-6153

The co-treatment of mill scale scrap and manganese ore via the oxidation roasting-magnetic separation process was investigated for the synchronous preparation of higher-value materials and the recovery of valuable metals. Thermodynamic and magnetic analyses indicated that a high temperature (>1100 C) and a MnO₂/Fe₂O₃ molar ratio of 0.75-1 were essential for the preparation of manganese ferrite. Experimental validation revealed that soft magnetic manganese ferrite powders, with a purity of 97.5 wt%, were obtained when the test was conducted at 1300 C for 120 min, followed by a two-stage grinding and magnetic separation process; the corresponding yield and the Mn and Fe recovered were 78.99 wt%, 86.14 wt%, and 84.60 wt%, respectively. During the oxidation process, [Fe²⁺]O was initially oxidised into the anti-form spinel-type structure of [Fe³⁺][Fe²⁺+Fe³⁺]O₄, and thereafter, it reacted with the decomposition product of [Mn³⁺][Mn²⁺+Mn³⁺]O₄ to form a hybrid spinel-type structure [Me₂+xMe₃+1-x][Me₂+1-xMe₃+1+x]O₄ (Me refers to Mn and Fe) via Mn²⁺/Fe²⁺+Mn³⁺/Fe³⁺ ion exchange at the tetrahedral and octahedral sites. Moreover, the as-purified ferrite could be used as an ingredient for the preparation of high-performance MnZn ferrites.

213539

EVOLUTION OF STRUCTURE AND IMPROVEMENT IN DIELECTRIC PROPERTIES OF PRASEODYMIUM SUBSTITUTED YFeO₃ NANOMATERIALS SYNTHESISED VIA A SOL-GEL AUTO-COMBUSTION METHODAsif M; Khan M A; Atiq S; Alshahrani T; Mahmood Q; Kattan N A; Manzoor A - *Bahawalpur, Islamia University; Lahore, University of the Punjab; Riyadh, Princess Nora Bint Abdul Rahman University; Dammam, Imam Abdulrahman Bin Faisal University; Saudi Arabia, Taibah University; Faisalabad, GC University***Ceram.Int.** 47, No.5, 2021, p.6663-6674

Y(1-x)Pr(x)FeO₃ (x = 0-0.16) ceramics were fabricated via the auto-combustion sol-gel method and XRD, SEM/EDS and FTIR were used for structural and morphological analyses. The substitution of praseodymium (Pr) modified the orthorhombic structure to a hexagonal one and traces of Fe₂O₃ and Y₂O₃ appeared in all the samples. The lattice constant 'a' was increased from 3.523 to 5.530 Angstrom after substitution up to x = 0.12,

afterward which it decreased from 5.530 to 5.529 Angstrom. Pure orthorhombic YFeO₃ (o-YFeO₃) had a cell volume of 223.61 (Angstrom)³, while the cell volume of Pr-substituted hexagonal YFeO₃ (h-YFeO₃) showed a decreasing trend (125.61-125.43 Angstrom). The bulk density decreased from 5.009 to 4.634 (g/cm³) with increased Pr concentration. The frequency dependence of the dielectric behaviour was investigated at room temperature over the 1 MHz to 3 GHz frequency range. SEM showed an average grain size of 149.28 nm, 66.78 nm, and 69.36 nm for the 0.00, 0.08, and 0.16 samples, respectively. o-YFeO₃ was confirmed by two main absorption bands ν_1 (449.86 /cm) and ν_2 (567.12 /cm) which indicated a clear shift after Pr addition. The force constant of the absorption bands at tetrahedral and octahedral sites showed an increasing trend between 1.633dyn/cm² x 10 exp(5) and 1.912dyn/cm² x 10 exp(5) and 2.818dyn/cm² x 10 exp(5) to 8.832dyn/cm² x 10 exp(5), respectively. The substitution of Pr enhanced the dielectric constant and the shifting of peaks at higher frequencies showed that increased dopant concentration resulted in the shifting of peaks that may follow the Maxwell-Wagner-Sillars polarisation mechanism. Pr-substituted YFeO₃ exhibited microwave frequency response in the 1.6 GHz-2.9 GHz range which could be suitable for high-frequency applications.

213540

EFFECTS OF MECHANOCHEMICAL ACTIVATION ON THE STRUCTURAL AND ELECTRICAL PROPERTIES OF ORTHORHOMBIC LuFeO₃ CERAMICS

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J.Am.Ceram.Soc. 104, No.7, 2021, p.3019-3029

Orthorhombic LuFeO₃ is a rare-earth orthoferrite with various potential applications. Pure phase LuFeO₃ ceramics were prepared by mechanochemical activation-assisted solid-state reaction (MAS) and conventional solid-state reaction (CSS) for comparison. XRD showed that the mechanochemical activation process could lower the starting reaction temperature and substantially shorten the time to complete the reaction of LuFeO₃ ceramics. More homogeneous and highly denser LuFeO₃ ceramics could be obtained using MAS as shown by density measurements and SEM. XRD and Raman measurements revealed that there was a small increase in lattice constant in the MAS sample compared to the CSS sample. Complex impedance spectra showed that the MAS sample had a much higher resistance compared to the CSS sample, which was caused by the higher density and fewer oxygen vacancies, verified by XPS. It is hoped that the study will contribute to solving the common leakage problems in ReFeO₃ ceramics and investigation of its multiferroic properties.

213541

SMOOTHING METHOD TO DIRECTLY DENOISE TERAHERTZ SIGNALS IN RARE-EARTH ORTHOFERRITE ANTIFERROMAGNETS

Zeng X; Liang Y; Zhang H; Xi X; Cao J; Li B; Zhou J - Beijing, University of Science & Technology; Shenzhen, Tsinghua University; Beijing, Tsinghua University

J.Am.Ceram.Soc. 104, No.7, 2021, p.3325-3333

Rare-earth orthoferrites with weak ferromagnetism have potential application in future information technologies, but there is not yet an appropriate and direct way to reduce the noise in their intrinsic but weak resonances. A numerical smoothing method was developed to directly denoise the detected terahertz (THz) responses of orthoferrites using a B-spline algorithm. The Savitzky-Golay smoothing method, a widely used numerical smoothing method, was also used to process the data for comparison. The thickness-dependent signals of DyFeO₃ were processed by these methods. LaFeO₃, HoFeO₃ and DyFeO₃ were chosen and prepared, and their thermally tunable signals were processed. The B-spline signal smoothing algorithm was shown to have stability and robustness to noise and was more effective than the Savitzky-Golay smoothing method at reducing noise. This work on smoothing thickness- and temperature-dependent THz signals may help provide a promising approach to reduce noise in the intrinsic but weak resonances of rare-earth orthoferrites.

213542

ENHANCED ROOM-TEMPERATURE MAGNETOELECTRIC COUPLING EFFECTS IN c-AXIS ORIENTED POLYCRYSTALLINE

BaSrCo_{2-x}Mg_xFe₁₁AlO₂₂

Chen X-H; Zhai K; Qian G-Y; Fu Q-S; Chakrabarti C; Li C-L; Yin H-X; Qiu Y; Tian Z-M; Yuan S-L - Huazhong, University of Science & Technology; Qinhuangdao, Yanshan University; Peking, University; Xinyang, Normal University

J.Am.Ceram.Soc. 104, No.7, 2021, p.3334-3343

The effect of the applied magnetic field during annealing and of Mg doping on the room-temperature magnetoelectric coupling effects in BaSrCo_{2-x}Mg_xFe₁₁AlO₂₂ (x = 0-1.5) were experimentally studied using magnetisation, magnetodielectric and magnetoelectric current measurements. Hexaferrite samples of Co₂Y were found to be highly oriented by an applied magnetic field during annealing, leading to enhanced room-temperature magnetoelectric coupling effects. Although the substitution of nonmagnetic Mg ions in Co sites tended to reduce the ferromagnetism at macroscopic scale, a suitable amount of Mg doping content facilitated the superexchange interaction between the adjacent magnetic blocks, while modulating the magnetic anisotropy in the samples. An appropriate adjustment of the competition between the anisotropy and the superexchange could enhance the magnetoelectric coupling at room temperature, which could be confirmed by the magnetic-field-induced dielectric constant and current density study.

213543

ELECTROCHEMICALLY MANIPULATING BiFeO₃ PARTICLES VIA Bi³⁺ ION EXTRACTION

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J.Am.Ceram.Soc. 104, No.7, 2021, p.3354-3364

Multifunctional ceramics are widely used in electronics, so manipulating the composition and structure of electronic ceramics can improve the performance of the target material. However, composite materials with multiphases and multicomponents can be achieved by mixing nanomaterials using various preparation methods or technologies, which generally requires experimental design to obtain the target products. A novel electrochemical approach to prepare functional ceramic materials via ion extraction from the A site of perovskite BiFeO₃ (BFO) ceramic particles was demonstrated, whereby a single-phase perovskite BFO could be evolved into multiphase and multicrystalline homogeneous composites. With gradual changes in material particle size, composition, structure and morphology upon electrochemical Bi³⁺ ion extraction, the magnetic and electromagnetic properties of the decomposed BFO and the generated multiphase and multicrystalline composites would show pronounced evolutionary behaviour, compared with the original BFO. Such a titration method upon electrochemical ion extraction could be used to manipulate the properties of the functional ceramics. The simple strategy of electrochemical manipulation of BFO could be applied to certain materials with

electrochemical activity to achieve the target performance.

213544

ENHANCED MICROWAVE ABSORPTION PROPERTIES OF Y-Co2Z/PANI HEXAFERRITES COMPOSITES IN THE FREQUENCY RANGE OF 0.1-18 GHz

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J. Am. Ceram. Soc. 104, No. 7, 2021, p. 3376-3386

Ba_{3-x}Y_xCo₂Fe₂₄O₄₁ (Y-Co₂Z, x = 0, 0.2 and 0.4) was prepared by solid-state reaction. Y-Co₂Z and polyaniline (PANI) composites (Y-P0, Y-P2 and Y-P4) were prepared by in-situ polymerisation. The Y-doping was important in the variation of lattice parameters, a and c. The combination of Y-doping and PANI modified the magnetic properties of the composites, as seen by the change of the saturation magnetisation and coercivity. This combination also affected the electromagnetic properties of the composites through the measurements of complex permittivity and permeability. Using transmission line theory, the reflection loss (RL) of the composites was calculated with thickness varying from 1.00 to 2.50 mm. The composites tuned the minimum RL from the X band (RL = -29.6 dB at 11.4 GHz for Y-P2) to the Ku band (RL = -16.3 dB at 15.7 GHz for Y-P4 and RL = -26.4 dB at 16.6 GHz for Y-P4). For maximum effective bandwidth, the composites covered a huge range from the S and C bands (Y-P0 with 3.9 GHz in the range of 3.4-7.3 GHz) through the X band (Y-P2 with 3.9 GHz in the range of 9.0-12.9 GHz) to the Ku band (Y-P4 with 4.0 GHz in the range of 13.8-17.8 GHz). These properties demonstrated that the composites could act as promising absorbers in the S, C, X and Ku bands.

213545

ELECTRICAL AND MAGNETIC PROPERTIES OF BiFeO₃ NANOPARTICLES SUBSTITUTED WITH HIGH CONCENTRATIONS OF COBALT

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J. Aust. Ceram. Soc. 57, No. 3, 2021, p. 643-650

Co-substituted (10 and 20% Co) BiFeO₃ (BFO) nanoparticles were prepared by coprecipitation and their microstructure, phase composition and electrical and magnetic properties were studied. BFO, Bi₂₅FeO₄₀, CoFeO₄ and Bi₂Fe₄O₉ phases formed in Co-substituted BiFe_{1-x}O₃ (x = 0.0, 0.1 and 0.2). The lattice parameter, 'a', decreased and 'c' increased for BFO with Co substitution, thereby increasing the lattice cell volume. The surface morphology and grain size and shape were studied by SEM. Enhanced dielectric properties were seen in the Co-substituted BFO nanoparticles. At low frequency, BFO showed a dielectric constant of 40, increasing to 52 and 402 for 10% and 20% Co-substituted BFO samples, respectively. The electrical conductivity in the temperature range 300-700 C range showed semiconducting behaviour. Remanence (Mr), saturation magnetisation (Ms) and coercivity (Hc) values for BFO were 0.123 emu/g, 6.178 emu/g and 108.59 G, respectively. The Ms, Mr and Hc values for 10% Co- and 20% Co-substituted BFO were 5.742 emu/g, 0.213 emu/g and 124.86 G and 42.777 emu/g, 24.255 emu/g and 4127.55 G, respectively. It was concluded that the synthesis of pure BiFeO₃ is not possible and an increase in magnetic parameters is due to the formation of cobalt ferrite.

213546

RESISTIVITY AND MAGNETISATION BIMODAL IMPROVEMENT IN Ni FERRITE NANOPARTICLES BY Mg SUBSTITUTION

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J. Aust. Ceram. Soc. 57, No. 3, 2021, p. 719-729

Ferrites still attract research interest as well as achieving significant global sales due to their large number of applications. A series of Mg-doped Ni ferrites with the composition Ni_{1-x}Mg_xFe₂O₄ were prepared by the conventional ceramic route. All compositions crystallised in the spinel form with a single-phase cubic spinel structure. FTIR spectral frequencies of the vibrational bands for different Mg concentrations were reported. IR spectral analysis confirmed the formation of the spinel structure. The magnetic susceptibility was determined as a function of temperature. The Curie temperature increased slightly together with the susceptibility up to x = 0.4. The resistivity was improved as a result of Mg doping. At x = 0.04, the resistivity reached 97% with 40% Mg²⁺ at the expense of Ni²⁺, with an observable enhancement in the susceptibility values and a shift of the Curie point towards higher values.

213547

SYNTHESIS, STRUCTURAL AND MAGNETIC PROPERTIES OF NiFe_{2-x}La_xO₄ (X=0-0.1) NANOPARTICLES

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J. Aust. Ceram. Soc. 57, No. 3, 2021, p. 809-818

Lanthanum substituted nickel ferrite, NiFe_{2-x}La_xO₄ (X=0-0.1) powders were synthesised for the first time by the combustion technique using glycine as the fuel. All the ferrites crystallise in the Fd3m space group with cubic symmetry. Very small peaks of both Fe₂O₃ and LaFeO₃ were also detected, the intensity of which increases with increasing La content. No systematic trend in the lattice parameter with La doping was noticed. Room temperature magnetisation studies obtained from a vibrating sample magnetometer show a decrease in saturation magnetisation with La doping. The cation distribution obtained from magnetic measurements shows that La doped nickel ferrites have an inverse spinel structure in which the octahedral sites are mainly occupied by Ni²⁺ ions.

213548

NOVEL SUCROSE CHELATED VISIBLE-LIGHT SENSITIVE AFO NPs: PREPARATION, CHARACTERISATION, PHOTOCATALYTIC ACTIVITY, AND REACTION MECHANISM

Singh H; Rajput J K; Dogra N; Jain G; Gupta A; Garg S - *Jalandhar, DAV University; Jalandhar, Dr. B R Ambedkar National Institute of Technology*

J. Aust. Ceram. Soc. 57, No. 3, 2021, p. 835-848

AgFe₂O₄ nanoparticles (AFO NPs) were prepared by a novel auto-combustion route using sucrose as a chelating agent. The AFO NPs were categorised into AFO-U NPs and AFO-C NPs, based on the calcination conditions. The synthesised products were characterised by XRD, SEM and FTIR and UV-Vis spectroscopy. A detailed comparison was made of the structure and optical properties of the two products. AFO-C NPs were found to be superior to AFO-U NPs and were successfully used as a heterogeneous photocatalyst for the degradation of the industrial pollutant, methylene blue, under solar radiation. The degradation efficiency reached 99.3%. AFO-C NPs showed remarkable performance in recyclability up to five consecutive runs without any noticeable loss in activity.

213549
EFFECT OF TiN CONTENT ON PROPERTIES OF NiFe₂O₄-BASED CERAMIC INERT ANODE FOR ALUMINIUM ELECTROLYSIS
 Zhang Z; Wang Y; Zu G; Cao Z; Liu J; Wang C - *Shenyang, Northeastern University; Texas, University at Dallas*
Int. J. Appl. Ceram. Technol. 18, No. 4, 2021, p. 1114-1124
 TiN nanoparticle-doped NiFe₂O₄-based ceramic inert anodes for aluminium electrolysis were prepared by a two-step cold-press sintering process to investigate how TiN affects the sintering behaviour and properties of the composites. DSC-TG, XRD and microstructural analysis showed that Ti and N were evenly distributed in the NiFe₂O₄ matrix to form a solid solution. The maximum linear shrinkage and shrinkage rate were enhanced with increasing content of TiN nanoparticles, while the activation energy of sintering in the initial stage fell from 382.63 to 279.58 kJ/mol for TiN contents of 0-9 wt%. When the TiN content was 7 wt%, the relative density, bending strength and elastic modulus attained maximum values of 97.24%, 73.88 MPa and 3.77 GPa, respectively, whereas the minimum static corrosion rate of 0.00114 g/cm².h was obtained; this was attributed to the relatively dense and stable microstructure of the NiFe₂O₄-based ceramic. The electrical conductivity of the ceramics showed a clear ascending trend with increasing TiN content and elevated temperature, due to the increased concentration and migration rate of the carrier.

Titanates, zirconates and niobates

See also Abstract(s): 213397 213585

213550
ATHERMAL ELECTRIC FIELD EFFECTS IN FLASH SINTERED ZIRCONIA
 Dong J; Biesuz M; Sglavo V M; Kermani M; Su X; Saunders T; Hu C; Grasso S - *Southwest Jiaotong University; Trento, University; London, Queen Mary University*
Adv. Appl. Ceram. 120, No. 4, 2021, p. 193-201
 A new approach for quantifying the effect of grain development on current density during zirconia (3YSZ) flash sintering (field set at a notional current limit of 150 mA mm²) was presented. This was accomplished by inserting a notch (0.5 mm deep and 1.5 mm broad) in the centre of a dog-bone specimen, which allowed for an unequal current distribution (varying from 3 to 670 mA mm²) while maintaining a reasonably uniform temperature (1420-1500 C). Localised overheating was verified by microstructure studies at the notch tip. The modelling technique separated thermal and electrical field effects, revealing previously unknown athermal electric field effects in flash sintering. Increased current density appeared to weakly restrict grain development, speed densification, enhance defect repair (notches, fractures, and pores), and promote inter-particle link formation. These findings may help to explain the athermal effects observed during FS and flash joining.

213551
INFLUENCE OF THE SINTERING METHOD ON DENSIFICATION, MICROSTRUCTURE AND ELECTRICAL CONDUCTIVITY OF 12Ce-TZP
 Ferreira L A S; Muccillo E N S - *IPEN/CNEN/SP*
Adv. Appl. Ceram. 120, No. 4, 2021, p. 202-208
 Zirconia ceramics containing 12 mol% cerium oxide were created using a solid state reaction and the traditional, rapid fire, and two-step sintering techniques. The sintering temperature and duration were adjusted in order to find the best sintering profile for these techniques. Dense specimens' monoclinic and tetragonal phase compositions, microstructure, and electrical conductivity were studied. The linear shrinkage was found to be insignificant up to 1100 C, with the greatest rate of shrinkage at 1150 C. For typical sintered specimens, densification increased by up to 2 h. The tetragonal phase was stabilised in non-isothermally sintered specimens at a high temperature (1500 C). Specimens sintered in two steps achieved excellent density and fine grain sizes. For each of the sintering techniques utilised, optimal sintering profiles were identified.

213552
EFFECT OF ANTIMONY CONTENT ON ELECTRICAL AND STRUCTURAL PROPERTIES OF 0.98(K_{0.48}Na_{0.52})_{0.95}Li_{0.05}Nb_{1-x}Sb_xO₃-0.02Ba_{0.5}(Bi_{0.5}Na_{0.5})_{0.5}ZrO₃ CERAMICS
 Carreno-Jimenez B; Villafuerte-Castrejon M E; Reyes-Montero A; Lopez-Juarez R - *Mexico, Universidad Nacional Autonoma*
Bol. Soc. Esp. Ceram. Vidrio. 60, No. 4, 2021, p. 266-272
 Lead-free 0.98(K_{0.48}Na_{0.52})_{0.95}Li_{0.05}Nb_{1-x}Sb_xO₃-0.02Ba_{0.5}(Bi_{0.5}Na_{0.5})_{0.5}ZrO₃ (KNLNS_x-BBNZ) solid solutions with x between 0.04 and 0.08 were prepared by solid-state reaction. Samples were sintered at 1120 C for 4 h. The effect of the Sb⁵⁺ content on the phase structure, microstructure, ferroelectric, dielectric and piezoelectric properties of the KNLNS_x-BBNZ ceramics was studied. The phase transition of the ceramic was determined by the temperature dependence of the dielectric properties, while the structural properties, such as phase coexistence, were studied by XRD. It was found that ceramics with x between 0.06 and 0.08 had an orthorhombic (Amm²) and tetragonal (P4mm) phase coexistence. The best piezoelectric properties were obtained in ceramics with x = 0.07: d₃₃ = 282 pC/N, -d₃₁ = 103 pC/N, k_p = 46%, ε_{nr} = 1820, tan δ = 3% and T_c = 271 C. This composition also had good thermal stability, up to 200 C on d₃₃ piezoelectric constant, indicating that it has great potential for application as sensors and actuators from room temperature until this temperature limit.

213553
IMPROVED STRAIN AND TRANSDUCTION VALUES OF LOW-TEMPERATURE SINTERED CuO-DOPED PZT-PZNN SOFT PIEZOELECTRIC MATERIALS FOR ENERGY HARVESTER APPLICATIONS
 Kim B S; Ji J-H; Koh J-H - *Chung-Ang, University*
Ceram. Int. 47, No. 5, 2021, p. 6683-6690
 The effects of low-temperature sintering and improved piezoelectric properties of CuO-doped 0.69Pb(Zr_{0.47}Ti_{0.53})O₃-0.31[Pb(Zn_{0.4}Ni_{0.6})_{1/3}Nb_{2/3}]O₃ ceramics were investigated. At high temperatures, sintered Pb(Zr,Ti)O₃-Pb(Zn,Ni)NbO₃ had excellent piezoelectric properties with a high piezoelectric charge coefficient (d₃₃), high electromechanical coupling coefficient (k_p) and high relative dielectric permittivity (ε_{nr}). However, low-temperature sintering of functional ceramics is important for industrial device applications and for sensor applications, sintering or fabrication temperature is important since the processing temperatures could interfere with material compatibility. By employing CuO dopant to Pb(Zr,Ti)O₃-P(Zn,Ni)NbO₃ material, the strain and transduction values were increased while reducing the sintering temperature.

213554
REVISITING STRUCTURAL EVOLUTION, DIELECTRIC AND FERROELECTRIC PROPERTIES IN (Pb_xBa_{1-x})ZrO₃ SYSTEM (x = 0.5-1.0)
 Li F; Long M; Wang C; Zhai J - *Anhui, University; Shanghai, Tongji University; Huizhou, University*
Ceram.Int. 47, No. 6, 2021, p. 7430-7437
 The relationship between the phase structure, dielectric and ferroelectric performance in the (Pb,Ba)ZrO₃ system was investigated. The confusing characteristics included the incorporation of barium which reduced the phase transition temperature and the orthorhombic-orthorhombic and rhombohedral-pseudocubic phase transition sequence was determined for a new sample and more complicated transitions for poled ones. Macroscopically, antiferroelectric-ferroelectric and antiferroelectric-ferroelectric relaxor -Debye medium states at room temperature were deduced, evidenced by multi-dimensional analysis. The observed ferroelectric behaviour originated from synergistic ferroelectric and antiferroelectric domain switching for x = 0.90-0.75 compositions, which were justified by single-bipolar-cycle measurements. Antiferroelectric and ferroelectric <to/from> ferroelectric <to/from> relaxor phase boundaries were built in (Pb_xBa_{1-x})ZrO₃, where polarisation and strain value reached a maxima at x = 0.75 and 0.65 compositions. Finally, the phase diagram in the (Pb_xBa_{1-x})ZrO₃ binary system (x = 0.5-1.0) was constructed and provided a guideline for compositional design in this system.

213555
MULTILEVEL LUMINESCENCE OF Er³⁺/Pr³⁺ CO-DOPED Ca₂Nb₂O₇ CERAMICS AND COMPOSITE FILMS FOR OPTICAL ANTI-COUNTERFEITING
 Lu Z; Tang J; Du P; Li W; Liu Z; Wang J; Luo L - *Ningbo, University; Shanghai, Institute of Ceramics; Fuzhou, University of Minjiang*
Ceram.Int. 47, No. 6, 2021, p. 8248-8255
 Ca_{1.997-x}Nb₂O₇:xEr³⁺+0.003Pr³⁺ (x = 0.002, 0.006, 0.01, 0.015, 0.02) ferroelectric ceramics were prepared by a solid-state reaction method. Four-modal luminescence comprising colour-tunable (red/yellow/green) photoluminescence (PL), up-conversion luminescence (UCL), photostimulated luminescence (PSL) and thermoluminescence (TL) were observed in the prepared ceramics. The corresponding mechanisms are also discussed and proposed. PSL using NIR light excitation was initially reported in this material. The as-synthesised phosphors and commercial ZnS:Cu²⁺ were incorporated into a polydimethylsiloxane (PDMS) matrix to fabricate a multifunctional optical device which provided multidimensional codes with colourful (red/pink/white/cyan/green/blue) and multimodal (upconverted/downshifted and photon-/thermo-/mechano-driven) emission, showing the promising prospects for optical information storage and anti-counterfeiting.

213556
MICROWAVE-HYDROTHERMAL SYNTHESIS AND PHOTOCATALYTIC PROPERTIES OF Cr-DOPED SrTiO₃ POWDERS
 Lopez-Juarez R; Perez-Juache T J; Hernandez-Cristobal O; Razo-Perez N; Cipagauta S - *Mexico, Universidad Nacional Autonoma; Iztapalapa, Universidad Autonoma Metropolitana*
Ceramics-Silikaty. 65, No. 2, 2021, p. 113-119
 SrTi_{1-x}CrxO₃ (x = 0, 0.02, 0.04 and 0.1) powders were synthesised by the microwave-hydrothermal route. Pure phase SrTi_{0.96}Cr_{0.04}O₃ powders were obtained at 200 C after 30 min. XRD and Raman analyses showed that chromium formed a solid solution within the SrTiO₃ lattice. The mean crystal size of the synthesised powders was close to 60 nm, as determined by XRD and confirmed by TEM. The specific surface was measured and the values ranged from 27 plus or minus 2.8 to 37 plus or minus 3.7 m²/g. The photocatalytic properties were evaluated for H₂ evolution from methanol solution; the best performance was seen for x = 0.04, for which one of the highest surface areas was obtained (34 m²/g) among the investigated compositions.

213557
SINTERING BEHAVIOUR, MICROSTRUCTURE AND MICROWAVE DIELECTRIC PROPERTIES OF Li₂Zn₃Ti₄O₁₂ CERAMICS DOPED BY LiF
 Chen J; Shen Y; Tang B - *China, University of Electronic Science & Technology*
Ceramics-Silikaty. 65, No. 2, 2021, p. 170-175
 LiF is widely used as a sintering aid for reducing the sintering temperature of microwave dielectric ceramics. LiF-doped Li₂Zn₃Ti₄O₁₂ ceramics were prepared by the solid-state route and their sintering behaviour, microstructure and microwave dielectric properties were studied. When a small amount of LiF is added, the LiF occurs as a pure phase coexisting with the Li₂Zn₃Ti₄O₁₂ under co-firing conditions, reducing the sintering temperature to 900 C without adversely affecting the microwave dielectric properties. With increasing LiF content, the apparent density increased from 3.81 g/cm³ to 4.17 g/cm³, the dielectric constant decreased from 17.9 to 16.4, and the Q x f value first increased from 2800 GHz to 47983 GHz before decreasing to 24944 GHz. The temperature coefficient of resonant frequency slightly decreased from -48 ppm/C to -54 ppm/C. Excellent combined microwave dielectric properties were obtained for the composition containing 1 wt% LiF when sintered at 900 C for 4 h: epsilon(r) = 17.9; Q x f = 47983 GHz; and tau(f) = -48.3 ppm/C.

213558
HYDROSTATIC-PRESSURE-INDUCED DEPOLARISATION OF (Pb_{1-1.5x}Lax)(Zr_{0.80}Ti_{0.20})O₃ FERROELECTRIC CERAMICS
 Xie M; Bao Y; Li S; Nie H; Wang G; Dong X - *Shanghai, Institute of Ceramics; Beijing, University of Chinese Academy of Sciences*
J.Am.Ceram.Soc. 104, No. 7, 2021, p. 3269-3278
 Pulse power energy conversion materials with ultrafast discharge processes and ultrahigh power densities have wide use in the defence, energy, medical and mining fields. The pressure-driven depolarisation in ferroelectric materials is significant and accounts for the discharge processes. Pressure-induced depolarisation in (Pb_{1-1.5x}Lax)(Zr_{0.80}Ti_{0.20})O₃ (PLZT) (x = 0-0.07) ceramics, and their corresponding phase structure, dielectric properties, ferroelectric properties and thermal depolarisation performances, was studied. Although a satisfactory pulse power energy conversion performance has been achieved in Pb(Zr_{0.95}Ti_{0.05})O₃ materials, poor temperature stability negatively affects their use. The static charge densities of PLZT (x = 0.04, 0.06) decreased from 29.11 microC/cm² and 31.53 microC/cm² to 19.76 microC/cm² and 6.56 microC/cm² under 400 MPa hydrostatic pressure, respectively, due to a pressure-driven ferroelectric-antiferroelectric phase structural transition. The temperature stability of PLZT (x = 0.06) materials was up to 87 C. This work may help further development of pulse power energy conversion devices.

213559
QUENCHING-CIRCUMVENTED ERGODICITY IN RELAXOR $\text{Na}_{1/2}\text{Bi}_{1/2}\text{TiO}_3\text{-BaTiO}_3\text{-K}_0.5\text{Na}_0.5\text{NbO}_3$
 Wei Q; Riaz A; Zhukov S; Hofmann K; Zhu M; Hou Y; Rodel J; Venkataraman L K - *Darmstadt, Technical University; Beijing, University of Technology*
J.Am.Ceram.Soc. 104, No.7, 2021, p.3316-3324
 Quenching alkaline bismuth titanates from sintering temperatures results in increased lattice distortion and consequently higher depolarisation temperature. The effect of quenching on the ergodicity of relaxor $\text{Na}_{1/2}\text{Bi}_{1/2}\text{TiO}_3\text{-BaTiO}_3\text{-K}_0.5\text{Na}_0.5\text{NbO}_3$ was studied. A distinct departure from ergodicity was observed from the increase in remanent polarisation and the absence of frequency dispersion in the permittivity response of the poled samples. The samples showed enhanced negative strain when an electric field was applied, indicating a proclivity towards correlated polar nanoregions, corroborated by the enhanced tetragonal distortion. As a result, ergodic relaxor $\text{Na}_{1/2}\text{Bi}_{1/2}\text{TiO}_3\text{-BaTiO}_3\text{-K}_0.5\text{Na}_0.5\text{NbO}_3$ had a depolarisation temperature of 85 C with a 60% increase in remanent polarisation and approximately a threefold increase in remanent strain upon quenching. Quenching-induced changes in the local environment of Na^+ and Bi^{3+} cations hindered the development of ergodicity promoted by the A-site disorder. The results provide new insight into tailoring ergodicity of relaxor ferroelectrics.

213560
EFFECT OF SINTERING TEMPERATURE ON THE DEPOLARISATION BEHAVIOUR OF $(\text{Bi}_0.5\text{Na}_0.5)\text{TiO}_3$ -BASED CERAMICS
 Peng P; Nie H; Zheng C; Wang G; Dong X; Yang C - *Fujian, University of Technology; Shanghai, Institute of Ceramics*
J.Am.Ceram.Soc. 104, No.7, 2021, p.3344-3353
 $(\text{Bi}_0.5\text{Na}_0.5)\text{TiO}_3$ (BNT)-based ferroelectric ceramics have excellent electrical properties and interesting depolarisation behaviour, but the poor thermal stability of their electrical properties limits their practical application. The effect of sintering temperature (T_s) on the depolarisation behaviour of BNT-based ceramics was studied. It was found that the depolarisation temperature T_d determined from pyroelectric measurement tended to decrease with increasing T_s , indicating that lower T_s deferred the ferroelectric-relaxor (FE-RE) phase transition. However, for samples sintered at higher T_s (e.g. 1180 C), although the T_d was reduced, the thermal stability was better compared with the sample sintered at lower T_s (1100 C) because the diffuse behaviour of the FE-RE phase transition was suppressed. According to the results, it was proposed that the thermal stability of electrical properties for BNT-based ceramics is not only related to high T_d , but also to the diffuse degree of phase transition.

213561
ASYMPTOTIC ANALYSIS OF RADIAL VIBRATION OF THIN PIEZOELECTRIC DISCS
 Xiong X; Li X - *Princeton, University; Wuhan, University*
J.Am.Ceram.Soc. 104, No.7, 2021, p.3411-3423
 The 1-D radial vibration model of piezoelectric discs has been widely used to determine the relevant material coefficients from admittance measurements. However, it assumes infinitely thin discs, and therefore cannot predict their axial displacements. The 1-D model was extended by performing an asymptotic analysis of the axisymmetric radial vibration of thin discs. The asymptotic expansions included the asymptotic axial displacement and the second-order corrections to the admittance and the radial displacement in the 1-D model. The asymptotic expansions and the 1-D model were verified by the Chebyshev tau method. In the 1-D model, the frequencies of the maximum admittance f_n in the first and second radial modes were accurate to 1% for Pz27 discs with thickness-to-diameter ratios of 0.15 and 0.065, respectively. For a general piezoelectric disc in the forced vibration, the error of f_n in the 1-D model could be estimated from the second-order correction of the asymptotic resonance frequency in the free vibration.

213562
STRUCTURAL AND MICROSTRUCTURAL FEATURES OF LEAD-FREE BNT-BT THIN FILMS: NANOSCALE ELECTROMECHANICAL RESPONSE ANALYSIS
 Mendez-Gonzalez Y; Ferri A; Lima E C; Hamieh A; Remiens D; Pelaiz-Barranco A; Silva A C; de los Santos Guerra J - *Uberlandia, Federal University; Havana, University; Lens, Universite d'Artois; Tocantins, Universidade Federal; Valenciennes, Polytechnic University of Hauts-de-France*
J.Am.Ceram.Soc. 104, No.7, 2021, p.3665-3681
 $\text{Bi}_0.506\text{Na}_0.46\text{Ba}_0.08\text{-}3x/2\text{La}_x\text{TiO}_3$ ($x = 0, 2$ and 3 at%) (BNLBT- x) lead-free thin films were synthesised by a sol-gel method and deposited on Pt(111)/ $\text{TiO}_2/\text{SiO}_2/\text{Si}$ substrates by spin coating. Their structural, microstructural and electromechanical properties were studied at the nanoscale as a function of lanthanum concentration. Structural characterisation by XRD and Raman spectroscopy confirmed the formation of the perovskite structure and suggested the coexistence of both antiferroelectric (tetragonal) and ferroelectric (rhombohedral) phases. The surface morphology, characterised by AFM, showed a dense and crack-free nanostructured surface for all the compositions, noting that the increase in the lanthanum content promoted a decrease in both the grain size and surface roughness. PFM imaging showed the ferroelectric domain structure over the surface, and the presence of non-piezoelectric regions attributed to the antiferroelectric phase. PFM spectroscopy revealed a reliable switching behaviour for locally probed ferroelectric domains, yielding a noticeable local piezoactivity, which showed an increase with increasing doping content. The static domain wall was also analysed in terms of the nanoscale domain structure, and the obtained dimensionality indicated a local electric field-induced structural disorder. The clear local piezo/ferroelectric nature highlighted on the studied materials indicates the potential for applications of the BNLBT- x films for integration into advanced electronic nanodevices.

213563
DOMAIN MORPHOLOGY OF NEWLY DESIGNED LEAD-FREE ANTIFERROELECTRIC $\text{NaNbO}_3\text{-SrSnO}_3$ CERAMICS
 Ding H; Zhang M-H; Koruza J; Molina-Luna L; Kleebe H-J - *Darmstadt, Technical University*
J.Am.Ceram.Soc. 104, No.7, 2021, p.3715-3725
 Reversible antiferroelectric-ferroelectric phase transitions have been seen in a series of SrSnO_3 -modified NaNbO_3 lead-free antiferroelectric materials, exhibiting well-defined double polarisation hysteresis loops at ambient conditions. TEM was used to investigate the crystallography and domain configuration of this newly designed system via electron diffraction and centred dark-field imaging. It was confirmed that antiferroelectricity was maintained in all the compositions, as shown by the characteristic $1/4$ superlattice reflections in the electron-diffraction patterns. Investigation of the antiferroelectric domains and domain boundaries in NaNbO_3 showed that antiphase boundaries were present and their irregular periodicity was responsible for the streaking features along the $1/4$ superlattice reflections in the electron-diffraction patterns. The signature domain blocks seen in pure NaNbO_3 were maintained in the SrSnO_3 -modified ceramics, but disappeared when the amount of SrSnO_3 reached 7 mol%. In particular, a well-defined and distinct domain configuration was seen in the NaNbO_3 sample modified with 5 mol% SrSnO_3 , which presented a

parallelogram domain morphology.

213564

STRUCTURAL INVESTIGATION AND IMPROVEMENT OF MICROWAVE DIELECTRIC PROPERTIES IN $\text{Ca}_{1-x}\text{Ba}_x\text{TiO}_3$ LOW LOSS CERAMICS

Uddin S; Zaman A; Rasool I; Akbar S; Kamran M; Mehboob N; Ali A; Ahmad A; Nasir M F; Iqbal Z - *Peshawar, Government College Hayatabad; Islamabad, Riphah International University; Peshawar, University*

J.Ceram.Process.Res. 21, No.6, 2020, p.745-750

A series of calcium barium titanates of composition $\text{Ca}_{1-x}\text{Ba}_x\text{TiO}_3$ ($x = 0.0-0.7$) was synthesised by solid state reaction of powder mixtures of calcium carbonate, barium carbonate and titania, and 1000 C. Cylindrical specimens were prepared by uniaxial pressing and sintered at 1300 C. The specimens were characterised by microstructural studies, and by determinations of phase composition, bulk density and microwave dielectric properties at 3 GHz. The sintered materials were single phase orthorhombic structure. The dielectric constant decreased from 145 to 52 as x increased from 0.0 to 0.7, whilst the near zero value of the temperature coefficient of resonant frequency decreased from 705 to 80 ppm/C.

213565

($\text{Bi}_0.5\text{K}_0.5$) TiO_3 LEAD-FREE FERROELECTRIC CERAMICS: PROCESSING, PROPERTIES, AND COMPOSITIONAL MODIFICATIONS

Hagiwara M - *Keio, University*

J.Ceram.Soc.Jap. 129, No.8, 2021, p.496-503

Studies at Keio University of the fabrication and properties of bismuth potassium titanate (BKT) ferroelectric ceramics are reviewed. Synthesis studies have been focused on hydrothermal synthesis, and a two-stage process was developed, in which an aqueous solution of potassium hydroxide containing anatase and bismuth hydroxide was first heated at 160 C, followed by cooling to 110 C and maintaining this temperature for 6 h. This avoided the formation of a titanium-rich fibrous secondary phase. The product was readily sintered to a relative density of about 95%, and exhibited less weight loss than conventionally-synthesised powder. The good sinterability is attributed to a covering of Bi_2O_3 nanoparticles on the surface of the BKT particles. The prepared materials have been characterised by measurements of the dielectric, ferroelectric and piezoelectric properties, including the influence of grain size. Solid solutions with strontium titanate have been developed as a means of stabilising the relaxor state.

213566

EFFECT OF Li CONCENTRATION ON THE IONIC CONDUCTIVITY OF $\text{Li}_x\text{La}_{(1-x)}\text{3Nb}_0.80\text{Ta}_0.20\text{O}_3$ SOLID SOLUTIONS

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J.Ceram.Soc.Jap. 129, No.8, 2021, p.535-539

Solid solutions of $\text{Li}_x\text{La}_{(1-x)}\text{3Nb}_0.80\text{Ta}_0.20\text{O}_3$ ($x = 0.00-0.30$) were synthesised by calcining powder mixtures of Li_2CO_3 , La_2O_3 , Nb_2O_5 and Ta_2O_5 at 800 C in air, followed by the sintering of pressed specimens of the calcined and ground powders at 1100-1300 C for 24 h in air. The specimens were evaluated by determinations of chemical composition, phase composition, density, Raman spectra, and resistivity by complex impedance measurements over the frequency range 1 Hz to 10 MHz, and from room temperature to 225 C. The sintering temperature to yield a solid solution rapidly decreased with increasing x . A single phase product was obtained for x in the range 0.00-0.25, and the crystal structure changed from orthorhombic to tetragonal and then to cubic with increasing x . The resistivity decreased as x increased from 0.10 to 0.30, attributed to a decrease in the number of vacancies and an increase in the activation energy. The optimum composition is concluded to be $\text{Li}_{0.10}\text{La}_{0.29}\text{Nb}_0.79\text{Ta}_0.20\text{O}_2.96$.

213567

HIGH PYROELECTRIC RESPONSE OVER A BROAD TEMPERATURE RANGE IN NBT-BZT: SiO_2 COMPOSITES FOR ENERGY HARVESTING

Shen M; Hu L; Li L; Zhang C; Xiao W; Zhang Y; Zhang Q; Zhang G; Jiang S; Chen Y - *Hubei, University; Huazhong, University of Science & Technology; Nanjing, University*

J.Eur.Ceram.Soc. 41, No.6, 2021, p.3379-3386

SiO_2 networks, with low heat capacities, were constructed in NBT-BZT ceramics. The networks improved the heat transfer (dT/dt) and broadened the pyroelectric temperature region of the composites by reducing the heat absorption capacity which led to high pyroelectric energy density and stability. The temperature range of the NBT-BZT composite with 0.1 wt% SiO_2 for a pyroelectric coefficient higher than $20 \times 10 \exp(-4) \text{ C/m}^2\text{K}$ was increased to 20 C which resulted in the high thermal stability of energy harvesting. In addition, the NBT-BZT: 0.1 wt% SiO_2 composites had an optimised pyroelectric energy density of 110 $\mu\text{J/cm}^3$, nearly three times that of pure NBT-BZT ceramics. This work is beneficial for the application of high-performance pyroelectric materials for devices used in energy harvesting.

213568

THERMAL CONDUCTIVITY OF PLASMA-ENHANCED ATOMIC LAYER DEPOSITED HAFNIUM ZIRCONIUM OXIDE DIELECTRIC THIN FILMS

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J.Eur.Ceram.Soc. 41, No.6, 2021, p.3397-3403

The thermal conductivity of plasma-enhanced atomic layer deposited (PEALD) hafnium zirconium oxide (HZO) thin films is reported. Steady-state thermoreflectance was used to measure the effective thermal conductivity of undoped and yttrium-doped HZO films and their interfaces. The effective thermal conductivity of the undoped film was 0.75 W/mK , which is comparable to those reported previously for thermal ALD HZO films with a similar composition. With increased yttrium doping level, the effective thermal conductivity slightly decreased to 0.67 W/mK due to the dopant scattering of phonons. The PEALD HZO films were nanocrystalline as observed by grazing-incidence XRD and TEM.

213569

NEW HIGH TEMPERATURE DIELECTRICS: Bi-FREE TUNGSTEN BRONZE CERAMICS WITH STABLE PERMITTIVITY OVER A VERY WIDE TEMPERATURE RANGE

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J.Eur.Ceram.Soc. 41, No.6, 2021, p.3416-3424

Potential high-temperature Class II capacitor material, free from Bi or Pb ions, developed by doping $\text{Sr}_2\text{NaNb}_5\text{O}_{15}$ with Ca^{2+} , Y^{3+} and Zr^{4+} ions,

according to the formulation $\text{Sr}_{2-2z}\text{Ca}_z\text{Y}_z\text{NaNb}_{5-z}\text{Zr}_z\text{O}_{15}$, is reported. For the sample composition $z = 0.025$, ϵ_r values were 1565 plus or minus 15 % (1 kHz) from -65 C to 325 C. At a slightly higher doping level, $z = 0.05$, the ϵ_r values were 1310 plus or minus 10 % from -65 C to 300 C. Values of the dielectric loss tangent, $\tan\delta$, were up to 0.025 from -60 C to 290 C, for $z = 0.025$, with $\tan\delta$ increased to 0.035 at 325 C. Microstructural analyses excluded core-shell mechanisms being responsible for the flattening of the ϵ_r -T response.

213570

EFFECT OF UNIAXIAL STRESS ON ENERGY HARVESTING, STORAGE AND ELECTROCALORIC PERFORMANCE OF BZT CERAMICSPatel S; Yadav H; Kumar M - *Indian Institute of Technology; Jaipur, Malaviya National Institute of Technology***J. Korean Ceram. Soc.** 58, No. 4, 2021, p. 437-444

A systematic approach of waste (thermal/mechanical) energy harvesting and storage potential in $\text{Ba}_{0.85}\text{Zr}_{0.15}\text{TiO}_3$ (BZT) ceramics was studied. The effect of stress on the energy storage density (harvesting/storage) and electrocaloric performance was also studied. Polarisation-electric field hysteresis loops were recorded at various temperatures and uniaxial compressive stress. The Olsen cycle and electromechanical cycle were used for direct waste heat or mechanical energy to electrical energy conversion. A thermal energy-harvesting density of 42 kJ/m³ per cycle was obtained when the Olsen cycle was operated between 296-343 K and 0.25-1.5 MV/m. The electromechanical cycle-based energy harvesting was estimated as 78 kJ/m³ under an applied stress of 5-160 MPa and an electric field of 0.25-1.5 MV/m. The energy storage density was 39 kJ/m³ at zero stress field and 343 K, which increased to 53 kJ/m³ under the biased stress of 80 MPa in the wide operating temperature range 296-328 K. The high energy storage was found to be a result of the reduction of the hysteresis loss. The electrocaloric temperature was 0.16 K and 0.18 K under 0 and 80 MPa stress fields, respectively. These findings will help to understand the stress effect on BZT materials, which offer high performance for energy harvesting and storage-based applications, and can help to improve the energy storage density and electrocaloric effect via stress confinement.

213571

CHARACTERISING ELECTRICAL BREAKDOWNS UPON REOXIDATION ATMOSPHERE FOR RELIABLE MULTILAYER CERAMIC CAPACITORSLee D; Song K; Jung D; Cha B; Park M; Kim J; Lee J H - *Samsung Electromechanics Co. Ltd.***J. Korean Ceram. Soc.** 58, No. 4, 2021, p. 445-451

Electrical breakdown of multilayer ceramic capacitors (MLCCs), shown as an increase in leakage current, was characterised as a function of atmospheric reoxidation. The atmospheric reoxidation was controlled with respect to the theoretical pO₂ for the oxidation of Ni internal electrodes. The breakdown was characterised by a Maxwell-Wagner polarisation technique, which showed space-charge-limited and Poole-Frenkel currents for all the samples. The threshold voltage for the transition between these two conduction modes was suggested as an index for the robustness of the grain boundary resistance of BaTiO_3 , therefore the breakdown voltage. The reoxidation atmosphere, which prevented Ni oxidation, increased the threshold voltage, dramatically enhancing the breakdown voltage and insulation resistance. Impedance spectroscopy and STEM-EDS revealed that the cation distribution throughout the BaTiO_3 grains and grain boundaries changed during the reoxidation, including Ni cations from the internal electrodes, which affected the grain boundary resistance and determined the breakdown voltage of the MLCCs with Ni internal electrodes. The reoxidation should therefore be concurrently optimised in terms of the cation redistribution and elimination of oxygen vacancies.

213572

ANTIFERROELECTRICS: HISTORY, FUNDAMENTALS, CRYSTAL CHEMISTRY, CRYSTAL STRUCTURES, SIZE EFFECTS, AND APPLICATIONSRandall C A; Fan Z; Reaney I; Chen L-Q; Trolier-McKinstry S - *Pennsylvania, State University; Sheffield, University***J. Am. Ceram. Soc.** 104, No. 8, 2021, p. 3775-3810

The history of antiferroelectric (AFE) materials, used in high-energy density capacitors, is reviewed, and the characteristics of antiferroelectricity and the phase transition of an AFE material are described. AFEs are energetically close to ferroelectric (FE) phases, and thus both the electric field strength and applied stress (pressure) affect the nature of the transition. As there are comparable energetics between the AFE and FE phases, there can be a competition and frustration of these phases, and either incommensurate and/or a glassy (relaxor) structures may be seen. The phase transition in AFEs can also be affected by the crystal/grain size, particularly at nanometric dimensions, and may be tuned through the formation of solid solutions. There have been extensive studies on the perovskite family of AFE materials, but many other crystal structures host AFE behaviour, such as $\text{CuBiP}_2\text{Se}_6$. AFE applications include DC-link capacitors for power electronics, defibrillator capacitors, pulse power devices and electromechanical actuators. Future needs and opportunities with respect to discovery, science and applications of AFE are also discussed.

213573

NOVEL RELAXOR (Bi,Na,Ba)(Ti,Zr)O₃ LEAD-FREE CERAMIC WITH HIGH ENERGY STORAGE PERFORMANCEBilal M K; Wang J; Bashir R; Liu H; Asif S U; Xie J; Hu W - *Yunnan, University***J. Am. Ceram. Soc.** 104, No. 8, 2021, p. 3982-3991

Energy storage ceramic capacitors have increased high power density and working voltage, but it is difficult to obtain simultaneously large recoverable energy density (W_{rec}), high energy efficiency (η) and good thermal stability. To achieve this, a novel lead-free ceramic system $(1-x)(\text{Bi}_{0.5}\text{Na}_{0.5})\text{TiO}_3-x(\text{BaZr}_{0.3}\text{Ti}_{0.7}\text{O}_3)$ [(1-x)BNT-xBZT] was developed by tailoring the ferroelectric relaxor states. The introduction of BZT gradually promoted the transformation of ferroelectric states into relaxor states at about room temperature for $x = 0.3-0.5$ that presented a pinched P-E loop. The optimised composition of $x = 0.45$ had a large W_{rec} of up to 2.6 J/cm³ and ultrahigh η of 94%, with only a small variation (plus or minus 8%) in W_{rec} and the high η (90%) over a broad temperature range (-30 to 180 C), demonstrating superior performance compared to many existing lead-free ceramics. The remarkable advantages of these novel BNT-BZT lead-free ceramics are promising for high-efficiency and temperature-stable energy storage capacitor applications.

213574

ENHANCED HYBRID IMPROPER FERROELECTRICITY IN Fe/Nb COSUBSTITUTED Ca₃Mn₂O₇ CERAMICSChen B H; Sun T L; Wei L Y; Liu X Q; Wen W; Tian H; Li J Y; Chen X M - *Zhejiang, University; Zhejiang, University of Technology; Shenzhen, Institute of Advanced Technology; Shanghai Advanced Research Institute; Shenzhen, Southern University of Science & Technology***J. Am. Ceram. Soc.** 104, No. 8, 2021, p. 4000-4013

The prototypical Ruddlesden-Popper compound $\text{Ca}_3\text{Mn}_2\text{O}_7$ has been predicted to have hybrid improper ferroelectricity, where the polarisation is induced by the condensation of two oxygen octahedral distortion modes. However, it is a challenge to switch the polarisation at room temperature in

Ca₃Mn₂O₇ since the presence of the intermediate nonpolar Acaa phase generally leads to a complex domain morphology. The effects of Fe/Nb cosubstitution on hybrid improper ferroelectricity in Ca₃Mn₂O₇ were studied, and easy polarisation switching at room temperature was achieved in Ca₃[Mn_{0.5}(Fe_{0.5}Nb_{0.5})_{0.5}]₂O₇. The ferroelectric phase transition occurred directly from I4/mmm to A21am at a temperature far above room temperature without the intermediate nonpolar Acaa phase. The distinct transition pathway formed the alternating 180 degree-type ferroelectric domains rather than the irregular 90 degree-type ferroelastic domains stacked along [001], resulting in easy polarisation switching at room temperature. The enhanced ferroelectric polarisation (Pr about 2.0 microC/cm²) was obtained due to the increased anti-ferrodistortive displacements of Ca cations at the A-site, arising from the larger amplitudes of oxygen octahedral distortions. Chemical pressure was important for the tunability of phase transition, domain morphology and ferroelectric characteristics, and it provided a useful approach for designing and creating high-performance improper ferroelectrics.

213575
SIGNIFICANT PHOTOSTRICTIVE RESPONSE IN LEAD-FREE Bi_{0.5}Na_{0.5}TiO₃ CERAMICS UNDER VISIBLE LIGHT ILLUMINATION
 Fang H; Chen C; Zhang F; Cao M; Yi Z - *Wuhan, University of Technology; Beijing, University of Chinese Academy of Sciences; Shanghai, Institute of Ceramics*

J.Am.Ceram.Soc. 104, No.8, 2021, p.4033-4040

The photostrictive effect, the deformation of a material under illumination, has great potential for wireless remote-control technology. Compared with the intensively studied lead lanthanum zirconate titanate (PLZT) ferroelectrics, Bi_{0.5}Na_{0.5}TiO₃ (BNT) ceramics are more environmentally friendly. A significant photostrictive response for BNT ceramics under visible light illumination was found. The UV-vis diffuse reflectance spectrum indicated that BNT was a direct bandgap semiconductor (E_g = 2.93 eV) with a long tail absorbance up to a wavelength of about 560 nm. TEM revealed nanoscale domains and a fluctuated local structure in the BNT ceramics. A significant photostriction ΔL/L about 0.08% was seen for BNT when irradiated by a 405-nm laser at a power of 20 kW/m². The nonuniform shifts of the XRD peaks under external laser illumination indicated that the photostriction was mainly due to the light-induced distortion of the crystalline lattice. Such a large deformation, approaching that of piezoelectrics/ferroelectrics, induced by an electric field gives great potential for photo-driven multifunctional applications.

213576
BISMUTH LAYER-STRUCTURED FERROELECTRICS WITH NON-SHEET-LIKE POLYHEDRAL MICROSTRUCTURES
 He X; Chen C; Zeng H; Li Y; Yang Q; Yi Z - *Shanghai, Institute of Ceramics; Beijing, University of Chinese Academy of Sciences*

J.Am.Ceram.Soc. 104, No.8, 2021, p.4041-4048

Bismuth layer-structured ferroelectrics (BLSFs) are promising for a variety of applications because of their high Curie temperature and environmental friendliness compared to lead-containing equivalents. With a mica-like grain growth habit and spontaneous polarisation along the crystallographic a-b plane, it is difficult to obtain excellent ferroelectric properties for most BLSFs fabricated by the conventional ceramic route. A new approach to obtain Bi₂WO₆ and Bi₄Ti₃O₁₂ BLSF ceramics with non-sheet-like polyhedral microstructures is reported. The ferroelectric hysteresis behaviour, dielectric tunability and domain structures were observed. The Bi₂WO₆ ceramic with a unique single-domain structure in each grain showed a typical ferroelectric polarisation-electric field hysteresis loop with a remanent polarisation of about 17.8 microC/cm² and a coercive field of about 43 kV/cm, values never before obtained in Bi₂WO₆ ceramics with sheet-like microstructure. The Bi₄Ti₃O₁₂ ceramic with irregular-shaped and curved tiny domains showed antiferroelectric-like behaviour, which was significantly different from the reported Bi₄Ti₃O₁₂ counterparts with plate-like anisotropic grains. The study would stimulate microstructure design and property modulation for other layer-structured materials.

213577
ELECTRICAL PROPERTIES AND TEMPERATURE STABILITY OF SrTiO₃-MODIFIED (Bi_{1/2}Na_{1/2})TiO₃-BaTiO₃-(K_{1/2}Na_{1/2})NbO₃ PIEZOCERAMICS

Song G; Zhang F; Liu F; Liu Z; Li Y - *Shanghai, Institute of Ceramics; Beijing, University of Chinese Academy of Sciences; Melbourne, RMIT University*

J.Am.Ceram.Soc. 104, No.8, 2021, p.4049-4057

SrTiO₃-modified lead-free piezoelectric ceramics, (0.93-x)Bi_{0.5}Na_{0.5}TiO₃-xSrTiO₃-0.06BaTiO₃-0.01 K_{0.5}Na_{0.5}NbO₃ ((BNT-xST)-BT-KNN, x = 0-0.06), were prepared by solid-state reaction. XRD structural analysis and electrical property characteristics revealed the ST-induced phase transformation from the ferroelectric phase to the relaxor phase and their coexistence state. Benefiting from the ST-destroyed ferroelectric long-range orders, a high normalised strain value (600 pm/V) was obtained in the (BNT-0.02ST)-BT-KNN ceramic at 5 kV/mm. The ST-generated relaxor phase helped to improve the temperature stability and restrain the hysteresis of the electric-field-induced strain. The normalised strain of (BNT-0.06ST)-BT-KNN ceramics could maintain a high value of about 337 pm/V at elevated temperatures up to 120 C.

213578
LOW THERMAL CONDUCTIVITY OF SrTiO₃-LaTiO₃ AND SrTiO₃-SrNbO₃ THERMOELECTRIC OXIDE SOLID SOLUTIONS
 Zhang Y; Cho H J; Sugo K; Mikami M; Woo S; Jung M-C; Zhuang Y-H; Feng B; Sheu Y-M; Shin W; Choi W S; Han M J; Ikuhara Y; Ohta H - *Hokkaido, University; Nagoya, Nat. Inst. of Advanced Industrial Science & Technology; Sung Kyun Kwan University; Korea, Advanced Institute of Science & Technology; Taiwan, National Yang Ming Chiao Tung University; Taiwan, National Chiao-Tung University; Tokyo, University*

J.Am.Ceram.Soc. 104, No.8, 2021, p.4075-4085

Electron-doped SrTiO₃ has potential as an oxide thermoelectric material to convert waste heat into electricity. The power factor of the electron-doped SrTiO₃, including SrTiO₃-LaTiO₃ and SrTiO₃-SrNbO₃ solid solutions, has been clarified, but their thermal conductivity (kappa) has not been clearly identified to date. Only a high kappa (> 12 W/m.K) has been assumed from the electron contribution based on Wiedemann-Franz law. It was here shown that the kappa of the electron-doped SrTiO₃ was lower than the assumed kappa, and its highest figure of merit (ZT) was > 0.1 at room temperature. The kappa slightly decreased with the carrier concentration (n) when n was < 4 x 10 exp(21)/cm³. In the case of SrTiO₃-SrNbO₃ solid solutions, an upturn in kappa was seen when n was > 4 x 10 exp(21)/cm³ due to the contribution of conduction electron to the kappa. However, kappa decreased in the case of SrTiO₃-LaTiO₃ solid solutions, probably due to lattice distortion, which scattered both electrons and phonons. The highest ZT was 0.11 around n = 1 x 10 exp(21)/cm³. These results are useful for the future design of electron-doped SrTiO₃-based thermoelectric materials.

213579

SIMULTANEOUSLY ACHIEVED HIGH-ENERGY STORAGE DENSITY AND EFFICIENCY IN (K,Na)NbO₃-BASED LEAD-FREE FERROELECTRIC FILMSHuang Y; Shu L; Zhang S; Zhou Z; Cheng Y-Y-S; Peng B; Liu L; Zhang Y; Wang X; Li J-F - *Beijing, Tsinghua University; Beijing, National Institute of Metrology; Xian, Xidian University; Jinan, Qilu University of Technology***J. Am. Ceram. Soc.** 104, No. 8, 2021, p. 4119-4130

Lead-free dielectric capacitors with excellent energy storage performance are desirable for environmental protection, but it is a challenge to achieve both large energy storage density and high efficiency simultaneously. The energy storage performance of sol-gel-processed (K,Na)NbO₃-based lead-free ferroelectric films on silicon substrates with compositions of 0.95(K_{0.49}Na_{0.49}Li_{0.02})(Nb_{0.8}Ta_{0.2})O₃-0.05CaZrO₃-x mol% Mn (KNN-LT-CZ5-x mol% Mn) was studied. An appropriate amount of Mn-doping facilitated the coexistence of orthorhombic and tetragonal phases, suppressed the leakage current and considerably enhanced the breakdown strengths of the KNN-LT-CZ5 films. Consequently, large recoverable energy storage density up to 64.6 J/cm³ with a high efficiency of 84.6% under an electric field of 3080 kV/cm was achieved in the KNN-LT-CZ5-5 mol% Mn film, which is thought to be superior to most lead-based and lead-free films on silicon substrates and thus demonstrates great potentials of (K,Na)NbO₃-based lead-free films as dielectric energy storage materials.

Fuel cell and sensor applications

See also Abstract(s): 213352 213387 213691

213580

SYNTHESIS OF MESOPOROUS HOLLOW CARBON MICROCAGES BY COMBINING HARD AND SOFT TEMPLATE METHOD FOR HIGH PERFORMANCE SUPERCAPACITORSHu F; Lin Y; Qiu Y; Wen B; Zheng Y; Yang H - *Shaanxi, University of Science & Technology***Ceram. Int.** 47, No. 5, 2021, p. 5968-5976

Nitrogen-doped mesoporous hollow carbon microcages (N-MHCC) were prepared by combining hard and soft template methods. The hierarchical porous structure (meso- & micro-pore) of N-MHCC contained a large number of active centres, sufficient space and reaction interface, which promoted the rapid diffusion of electron and electrolyte ion transport. By comparing the electrochemical performance of nitrogen-doped hollow carbon microcages (N-HCC) and N-MHCC, it was shown that N-MHCC had a high specific capacitance of 210.66 F/g at 0.5 A/g while that of N-HCC was 132.6 F/g. The cyclic retention rate of N-MHCC was as high as 96.92% at 5 A/g after 4000 cycles. Furthermore, the simple preparation method and attractive performance make N-MHCC a promising candidate for high performance supercapacitors.

213581

SOL-GEL SYNTHESIS OF NANO Li_{1.2}Mn_{0.54}Ni_{0.13}Co_{0.13}O₂ CATHODE MATERIALS USING DL-LACTIC ACID AS CHELATING AGENTChen C; Wu H; Zhou D; Xu D; Zhou Y; Guo J - *Guizhou, University; Guizhou, Institute of Technology***Ceram. Int.** 47, No. 5, 2021, p. 6270-6278

Lithium-rich cathode materials Li_{1.2}Mn_{0.54}Ni_{0.13}Co_{0.13}O₂ (LMNCO) were prepared by the sol-gel method using DL-lactic acid as a chelating agent. The effect of pH on the crystal structure, morphology, particle size and electrochemical properties of the cathodes were studied using XRD, SEM, nanoparticle analysis, charge-discharge tests and electrochemical analysis. The Li_{1.2}Mn_{0.54}Ni_{0.13}Co_{0.13}O₂ cathodes had well-ordered layered structures consisting of hexagonal LiMO₂ and monoclinic Li₂MnO₃ with smooth surfaces and well-crystallised particles (100-500 nm). LMNCO-7.0 had a smaller particle sizes than LMNCO-5.5 and LMNCO-8.5 and improved electrochemical performance. The first discharge capacity and Coulombic efficiency of LMNCO-7.0 were 232.31 mAh/g and 73.2%, respectively. After 50 cycles, the discharge capacity of LMNCO-7.0 decreased to 194.93 mAh/g. The LMNCO-7.0 cathode had superior discharge capacity and rate performance due to its low charge transfer impedance and the small average quasi-spherical particle diameter.

213582

INFLUENCE OF Zn-SUBSTITUTION ON STRUCTURAL, MORPHOLOGICAL, ELECTRICAL, AND GAS SENSING PROPERTIES OF Zn_xAl₂O₄ (x = 0.1 TO 0.5) SYNTHESISED BY A SOL-GEL AUTO-COMBUSTION METHODTikare R R; Powar R R; Parale V G; Park H-H; Shinde T J; Kanase D G - *Sangli, Dr. Patangrao Kadam Mahavidyalaya; Titave, Shahid Virpatni Laxmi Mahavidyalaya; Yonsei, University; Islampur, Kanya Mahavidyalaya***Ceram. Int.** 47, No. 5, 2021, p. 6779-6789

The nanosphere decorated needle-like morphology of zinc-substituted aluminate, with the general formula Zn_xAl₂O₄ (x = 0.1, 0.2, 0.3, 0.4, and 0.5) (ZAN), were synthesised by a sol-gel auto-combustion method. The phase formation and thermal stability were confirmed by TGA-DTA and XRD confirmed the formation of a cubic spinel structure in the samples. The effect of Zn-substitution on structural and morphological properties of aluminate were investigated using XRD, FTIR, TEM, FE-SEM and EDX. The dc electrical resistivity of the ZAN samples revealed that the resistance decreased with increased temperature which confirmed their semiconducting nature. The nanospheres on the micro-needles of the zinc-substituted aluminate gas sensor showed sensitivity to several gases including H₂S, Cl₂, CH₃OH, SO₂, and NO₂ between room temperature and 300 C. The Zn_{0.4}Al₂O₄ gas sensor produced the highest response at an operating temperature of 200 C to 100 ppm H₂S. Results showed that the prepared nanosphere decorated needles of the ZAN sensor were sensitive and selective to H₂S gas.

213583

SILICON OXYCARBIDE-CARBON HYBRID NANOFIBRES: A PROMISING ANODE FOR ULTRALONG-CYCLE LITHIUM ION BATTERIES WITH HIGH RATE CAPABILITYLi H; Yan X; Ma Z; Zhang Y; Li C; Xiao W; Jiang Y - *Yangtze, University***Ceram. Int.** 47, No. 5, 2021, p. 6867-6874

Silicon oxycarbide-carbon hybrid nanofibres were synthesised using an economically efficient approach which involved several steps: electrospinning the PAN nanofibres (PNFs), grafting polysiloxanes onto the PNFs and thermal conversion of the silicone/PNFs (SPNFs). The obtained 1D nanostructures resulted in the hybrid electrodes having rapid electron transfer kinetics, robust structural stability, fast lithium ion conduction and an efficient electronic pathway. When tested as an active electrode material, the hybrid electrode delivered a reversible specific capacity of 715.8 mAh/g at 1 C (1 C = 372 mA/g), excellent high rate performance (422.1 mAh/g at 20C) and outstanding capacity retention capability of 95.4% after

2000 cycles at 10 C. Results showed the potential application of silicon oxycarbide-carbon hybrid nanofibre anodes for the next-generation of lithium ion batteries.

213584
OXYGEN VACANCY-RICH BLACK TiO₂ NANOPARTICLES AS A HIGHLY EFFICIENT CATALYST FOR Li-O₂ BATTERIES
Ge J; Du G; Kalam A; Bi X; Ding S; Su Q; Xu B; Al-Sehemi A G - *Shaanxi, University of Science & Technology; Abha, King Khalid University*
Ceram.Int. 47, No.5, 2021, p.6965-6971
Coloured titanium dioxide nanoparticles were synthesised by calcination in argon and Raman spectroscopy and XPS confirmed the existence of more oxygen vacancies and Ti³⁺ in black TiO₂ than the grey oxide. HR-TEM revealed defect layers of 2-3 nm on the TiO₂ surface. Due to the high electrical conductivity and good catalytic activity promoted by oxygen vacancies and Ti³⁺ ions, the black TiO₂-based cathodes had good electrochemical activity and enhanced cycling stability for lithium-oxygen batteries (LOBs). Compared with grey TiO₂ (4.3 V), the black TiO₂ nanoparticles showed a low over-potential with a charge voltage of 3.7 V. The charging voltage was reduced to approximately 600 mV, which reduced the battery's side reactions and polarisation and thus enhanced the cycling performance of the battery. When the cut-off capacity was 500 mAh/g, it could run for 108 cycles without significant change in terminal voltage, which indicated that black TiO₂ is an efficient catalyst for LOBs.

213585
INVESTIGATION ON BAROMETRIC AND HYDROSTATIC PRESSURE SENSING PROPERTIES OF Pb[(Mg_{1/3}Nb_{2/3})_{0.7}Ti_{0.3}]O₃ ELECTRO-CERAMICS
Singh C; Thakur V N; Kumar A - *New Delhi, National Physical Laboratory; Gaziabad, Academy of Scientific & Innovative Research*
Ceram.Int. 47, No.5, 2021, p.6982-6987
A two-stage solid-state reaction-based intermediate columbite phase technique was used to fabricate single phase Pb[(Mg_{1/3}Nb_{2/3})_{0.7}Ti_{0.3}]O₃ (PMN-0.3 PT) electroceramics. Rietveld refinement of XRD data suggested the presence of a monoclinic crystal structure with a Cm space group. The microstructure of the single phase bulk ceramics was void-free with well-defined grain boundaries. The pressure and temperature-dependent dielectric and impedance properties were investigated to assess possible applications as a pressure transducer. Well defined square hysteresis with large polarisation confirmed the high quality ferroelectric and piezoelectric properties. The pressure sensing properties of the PMN-0.3 PT ceramics in the 25-250 kPa range of barometric pressure, using dead weights and 10-50 MPa hydraulic pressure (oil medium), were studied. A gradual change in capacitance and capacitive reactance, with a percentage sensitivity of 0.10 /MPa in the hydraulic pressure range (10-50 MPa) and with a percentage sensitivity of 0.02 /kPa in the barometric pressure range (25-250 kPa), were observed. Results demonstrated the suitability of PMN-0.3 PT ceramics as pressure transducers.

213586
SYNTHESIS OF MANGANESE (IV) OXIDE AT ACTIVATED CARBON ON REDUCED GRAPHENE OXIDE SHEETS VIA LASER IRRADIATION TECHNIQUE FOR ORGANIC BINDER-FREE ELECTRODES IN FLEXIBLE SUPERCAPACITORS
Qahtan T F; Cevik E; Gondal M A; Bozkurt A; Akhtar S; Hassan M - *King Fahd, University of Petroleum & Minerals; Dammam, Imam Abdulrahman Bin Faisal University*
Ceram.Int. 47, No.6, 2021, p.7416-7424
Binder-free, high-purity conductive composites comprising activated carbon (AC), manganese dioxide nanorods (MnO₂), and reduced graphene oxide sheets (rGO), for flexible supercapacitors with outstanding electrochemical performance, are reported. UV pulsed laser irradiation of the GO-based composite dispersion (AC/GO or MnO₂@AC/GO) in ethanol aqueous medium was used to induce the photocatalytic reduction of GO and simultaneous anchor the AC particles or AC loaded MnO₂ nanorods (MnO₂@AC) on the reduced GO sheets (rGO) at room temperature and atmospheric pressure. The rGO sheets served as a large surface area, conductive binder to enhance the ion adsorption, electrical conductivity and mechanical flexibility of the supercapacitor electrodes. The laser-induced photocatalytic reduction method was used to prepare two different rGO-based colloidal composites AC/rGO (CG) and MnO₂@AC/rGO (MCG). The prepared rGO-based colloidal composites were used to fabricate symmetrical supercapacitors (CG//CG and MCG//MCG) and asymmetric supercapacitors (MCG//CG) in which MCG was the positive electrode and CG was the negative one. All the prepared rGO-based supercapacitors had improved electrochemical performance compared with rGO-free AC based supercapacitors. The enhanced electrochemical properties of the rGO-based supercapacitors could be attributed to the intrinsic characteristics of rGO, including high surface area, excellent electrical conductivity and mechanical flexibility. This method is a one-step, scalable, cost-effective synthesis technique to produce binder-free AC/rGO based composites for flexible energy-storage devices.

213587
PREPARATION AND GAS-SENSING PERFORMANCE OF GO/SnO₂/NiO GAS-SENSITIVE COMPOSITE MATERIALS
Jiang L; Tu S; Xue K; Yu H; Hou X - *Lanzhou, University of Technology; Lanzhou, General Hospital*
Ceram.Int. 47, No.6, 2021, p.7528-7538
A binary SnO₂/NiO composite was prepared via a co-precipitation method followed by calcination at 400 C for 2 h which resulted in good crystallisation. A ternary graphene oxide (GO)/SnO₂/NiO composite was then prepared by a hydrothermal method, in which SnO₂/NiO showed secondary growth on the GO surface. XRD showed that SnO₂/NiO had good crystallinity and proved the existence of a chemical bond, Sn-O-C, due to the formation of a chemical bond between GO and SnO₂/NiO. Finally, GO/SnO₂/NiO was prepared and deposited on the surface of a gold electrode for gas sensitivity tests. Good response to acetone gas in the 10-500 ppm concentration range at 350 C was determined. Compared with SnO₂/NiO, GO/SnO₂/NiO had improved response time, recovery time and sensitivity. At 350 C, the sensitivity of acetone at a concentration of 50 ppm was 21.11, the response time was only 5 s and the recovery time was 150 s. GO/SnO₂/NiO comprised two structures, a chemical bond and a p-n junction, which exerted a synergistic effect and could have applications for acetone gas detection.

213588

ELECTRICAL CHARACTERISATION OF GLASS-CERAMIC SEALANT-METALLIC INTERCONNECT JOINED SAMPLES UNDER SOLID OXIDE ELECTROLYSIS CELL CONDITIONS; INFLUENCE ON THE MICROSTRUCTURE AND COMPOSITION AT THE DIFFERENT POLARISED INTERFACESJaved H; Herbrig K; Sabato A G; Ferrero D; Santarelli M; Walter C; Seacetto F - *Sunfire GmbH; Barcelona, Catalonia Institute for Energy Research; Turin, Polytechnic***Ceram.Int.** 47, No. 6, 2021, p. 8184-8190

The electrical resistivity of a glass-ceramic sealant was evaluated at 850 C, for 2800 h under an applied voltage of 1.6 V. The glass-ceramic sealant was sandwiched between two Crofer22APU plates to produce Crofer22APU/glass-ceramic/Crofer22APU samples. The Crofer22APU/glass-ceramic/Crofer22APU joints had an electrical resistivity of about $10 \exp(6)$ - $10 \exp(7)$ Ohm cm, significantly high to ensure the insulation between two conductive interconnect plates. SEM-EDS post mortem analysis showed good thermomechanical compatibility between the glass-ceramic and the Crofer22APU substrates, which excluded any detrimental interaction with the metallic interconnect under a high applied voltage. XRD analysis of the glass-ceramic confirmed the presence of crystalline phases with suitable CTEs, after electrical resistivity under harsh conditions.

213589

FABRICATION OF CONTINUOUS LINEAR PORES IN AN SOFC ANODE USING UNIDIRECTIONAL CARBON FIBRES AS SACRIFICIAL TEMPLATESChoi S; Cheung C; Graeve O A - *California, University at San Diego***J.Am.Ceram.Soc.** 104, No. 7, 2021, p. 3030-3041

A solid-oxide fuel cell anode of NiO-8 mol% Y₂O₃-stabilised ZrO₂ was manufactured with micro-scale continuous linear pores (CLPs) and nano-scale interparticle pore structures, achieved through thermal decomposition of unidirectional amorphous carbon fibres as sacrificial templates. The CLP structure prepared by this method was characterised by its controllable uniform size, a tortuosity (i.e. uniformity) of 1.003 and a coefficient of variation of 0.59. These highly regular CLPs should minimise Knudsen diffusion, resulting in enhanced mass transport of hydrogen gas at the active sites (triple-phase boundary sites). Mass transport in the systems was determined by simulations using the Lattice Boltzmann method. An optimum diameter of 3 micron and an interparticle pore size of 185 nm were shown to maximise the acceleration of mass transport of H₂ and maintain the number of triple-phase boundary sites to minimise concentration overpotential. The proposed porous design can therefore increase the energy efficiency of a solid-oxide fuel cell primarily by reducing the concentration overpotential.

213590

FABRICATION AND CHARACTERISATION OF Al₂O₃-TiB₂ NANOCOMPOSITE POWDER BY MECHANOCHEMICAL PROCESSINGAydin H; Elmusa B - *Kutahya, Dumlupinar University***J.Aust.Ceram.Soc.** 57, No. 3, 2021, p. 731-741

Alumina-titanium diboride nanocomposite powder was synthesised by subjecting a mixture of TiO, B₂O₃ and Al powders to high-energy ball milling. The effect of milling time on the structural evolution and phase transformation of the powder particles was studied by XRD and SEM. After only 2 h milling, the Al/TiO/B₂O₃ reacted in a self-sustaining mode to form Al₂O₃-TiB₂ nanocomposite powder. As far as is known, this fabrication route has not been previously studied. In particular, similar previous studies have used TiO₂ instead of TiO as the titanium source; the main benefit is that the replacement of elementary reagents by compounds usually leads to the formation of a structure with finer particles and requires a shorter milling time for powder synthesis. In the final stage of milling, the crystallite size of the Al₂O₃ and TiB₂ was found to be < 20 nm. The minimum calculated particle size was 15.7 nm. FTIR revealed that the Al-O and Ti-B bonds had peaks between 409.16 and 673.9/cm and between 739.03 and 740.45, respectively. Peak values were 1017.9 and 1025.01/cm. Increasing the milling time up to 4 h had no significant effect other than reducing the crystallite size.

213591

PREPARATION AND CHARACTERISATION OF WTiO₂ NANOTUBES DECORATED WITH PRUSSIAN BLUE NANOPARTICLES AND NANOCUBES WITH ENHANCED PHOTOELECTROCHEMICAL PROPERTIESMomeni M M; Avazverdi M - *Isfahan, University of Technology***J.Aust.Ceram.Soc.** 57, No. 3, 2021, p. 961-969

The SILAR (successive ionic layer adsorption and reaction) method was used to decorate Prussian blue (PB) on tungsten-doped titanium oxide (WTiO₂) nanotubes. The morphology, phase composition, elemental composition and optical properties of the prepared samples were analysed by SEM, XRD, EDX and UV-Vis-DRS. The samples were used as photoanodes in photoelectrochemical water splitting to yield hydrogen. Their morphology, optical properties and photoelectrochemical performance were greatly affected by the SILAR deposition cycle. The sample with PB deposited in nine SILAR cycles showed the highest photocurrent (0.118 mA/cm²) under illumination. This was mostly due to visible light harvesting and reduced recombination of photo-generated electron-hole pairs, resulting from the synergistic effect of the heterojunction and the well-organised morphology of WTiO₂.

213592

DIELECTRIC CHARACTERISTICS OF COMPLEX PEROVSKITE CERAMIC AT MICROWAVE FREQUENCIES FOR APPLICATION IN DIELECTRIC RESONATOR ANTENNA TEMPERATURE SENSOR NETWORKLi C-H; Chen Y-C; You Y-C; Kuo C-C - *National Taiwan University of Science & Technology; Lunghwa, University of Science & Technology***J.Aust.Ceram.Soc.** 57, No. 3, 2021, p. 983-992

The microwave dielectric characteristics of La(Mg_{0.5-x}CaxSn_{0.5})O₃ ceramics for use in dielectric resonator antenna temperature sensor networks were studied. La(Mg_{0.49}Ca_{0.01}Sn_{0.5})O₃ ceramics sintered at 1600 C for 4 h have a bulk density of 6.57 g/cm³, a dielectric constant of $\epsilon_s 19.9$, a quality factor of 94,300 GHz and a temperature coefficient of resonant frequency of -87 ppm/C. Sensitivities of -88.96, -93.40 and -91.02 ppm/C were achieved for sensors 1, 2 and 3, respectively. The linearities of sensors 1, 2 and 3 were 6.6%, 6.7% and 6.3%, respectively.

213593
ENHANCED THE ELECTROCHEMICAL PERFORMANCES OF Ni-RICH LiNi_{0.8}Co_{0.1}Mn_{0.1}O₂ CATHODE BY ZrO₂ COATING
 Jo S-J; Kim H-S; Hwang D-Y; Jin B-S; Sim S-J; Shin J-S; Lee S-H - *Daejeon, University; Korea, Electrotechnology Research Institute*
J.Ceram.Process.Res. 21, No.6, 2020, p.731-735
 The hydroxide precursor of nickel-rich nickel cobalt manganese oxide was prepared by coprecipitation. The spherical precursor was mixed with lithium hydroxide in the molar ratio of 1.05:1 and calcined at 480 C, to obtain LiNi_{0.8}Co_{0.1}Mn_{0.1}O₂ cathode material. Samples of the prepared material were coated with different proportions of ZrO₂ and calcined at 500 C in air. The resulting powders were characterised by morphology studies and determinations of crystalline phase composition. Cathodes were prepared by mixing the nickel-rich powder with carbon black and polyvinylidene fluoride and used to produce 2032 coin cells, which were evaluated by measurements of the electrochemical properties. The ZrO₂ coating did not affect the crystalline structure, particle size or shape. It stabilised the structure and enhanced the electrochemical properties, attributed to decreased side reactions and an increase in the electrochemical reaction kinetics of the lithium ion, so improving the cycling performance.

213594
INFLUENCE OF TEMPERATURE ON PERFORMANCE OF CuV₂O₆ CATHODE FOR HIGH VOLTAGE THERMAL BATTERY
 Roh H C; Kim I Y; Ahn T Y; Cheong H-W; Yoon Y S - *Gachon University; Korea, Agency for Defence Development*
J.Korean Ceram.Soc. 58, No.4, 2021, p.507-518
 Thermal batteries are used as primary batteries for military missiles. The effect of heat treatment on the material properties and electrochemical properties of CuV₂O₆, a candidate cathode for thermal batteries, was evaluated, measuring the discharge capacity and open-circuit voltage. The properties of CuV₂O₆ calcined at different temperatures were determined using XRD, SEM, FTIR spectroscopy, XPS, TGA, TEM and EDS. CuV₂O₆ was synthesised from NH₄VO₃ and CuCl₂ by the hydrothermal method, and the XRD pattern was consistent with PDF#01-074-2117. The synthesised CuV₂O₆ was calcined at different temperatures, and the effect of calcination on the structures was analysed by SEM and TEM which showed that CuV₂O₆ was formed as rod-shaped crystals that gradually grew with increasing calcination temperature. However, upon calcination at 600 C, the morphology of CuV₂O₆ was not maintained and the compound decomposed irregularly. XPS showed that CuV₂O₆ was changed to Cu₂V₂O₇ at higher calcination temperatures. Less than 5% weight change was seen in the TGA curves, including an increase in the weight at a higher temperature. The capacity of the CuV₂O₆ samples was about 300-350 mAh/g, as determined from the discharge curve, and CuV₂O₆ calcined at 550 C had a higher activation energy than the other samples. Calcination at a higher temperature could provide enhanced battery capacity. The data indicated that CuV₂O₆ may be a viable candidate material for thermal battery cathodes.

213595
UNVEILING THE PRESENCE OF MIXED OXIDATION STATES OF EUROPIUM IN Li₇+DELTA Eu_xLa₃-DELTA Zr₂-DELTA O₁₂-DELTA GARNET AND ITS IMPACT ON THE Li-ION CONDUCTIVITY
 Salimkhani H; Erdem E; Gursel S A; Yurum A - *Istanbul, Sabanci University*
J.Am.Ceram.Soc. 104, No.8, 2021, p.4257-4271
 Lanthanides have recently been studied to examine their impact on the structure and properties of Li₇La₃Zr₂O₁₂ (LLZO) garnets. Eu₂O₃-doped LLZO (Li₇+delta Eu_xLa₃-delta Zr₂-delta O₁₂-delta) solid electrolytes demonstrating a cubic phase with the symmetry of Im-3m (No.230) at room temperature were developed. Different concentrations of Eu (0.1-0.6 atoms per formula unit) were doped into Li₇La₃Zr₂O₁₂ to evaluate the effect of Eu on the stability of the cubic phase and thereby the ionic conductivity. The results showed that, upon doping Eu³⁺ ions, the Eu²⁺ state was also formed and was then self-doped into the structure in which Rietveld refinement coupled with XPS, EPR and solid-state NMR suggested that Eu³⁺ ions most probably partially occupied the Zr⁴⁺ (16a) site, the Eu²⁺ ions occupied the La³⁺ (24d) site and the Li⁺ ions occupied two different sites (24d and 96h). It was further found that such a site preference induced distortion at the LaO₈ polyhedra opening up the neck for Li-ion diffusion, thereby enhancing the ionic conductivity. It was also shown that Li-ions probably hopped from 96h to 24d and then to the 96h site to generate the Li-ion movement. By introducing Eu ions into the LLZO structure, an enhanced bulk ionic conductivity of 0.30 x 10⁻³ S/cm at 298 K with a minimum electronic conductivity of 2.547 x 10⁻⁹ S/cm at 298 K was achieved.

213596
STRENGTH AND HYDROTHERMAL STABILITY OF NiO-STABILISED ZIRCONIA SOLID OXIDE CELLS FUEL ELECTRODE SUPPORTS
 Khajavi P; Frandsen H L; Gremillard L; Chevalier J; Hendriksen P V - *Denmark, Technical University; Lyon, Institut National des Sciences Appliquees*
J.Eur.Ceram.Soc. 41, No.7, 2021, p.4206-4216
 The strength, Young's modulus and low-temperature degradation (LTD) of several zirconia-based SOCs supports were studied. Results showed that by replacing 3YSZ with a tetragonal zirconia compound, with a lower stabiliser content, could improve the strength of porous supports. An enhancement of up to 30 % over the state-of-the-art support (NiO-3YSZ) could be achieved using NiO-2.5YSZ. It was further shown that tetragonal zirconia-based SOC components (both Y-doped and Ce-Y codoped) were susceptible to LTD in the studied grain size range (approximately 200 to 300 nm). The addition of a small amount of alumina (0.5 wt%) increased the LTD resistance of the porous supports. The susceptibility to LTD should be considered when designing materials for SOCs, also during material processing, storage, handling and operation where a dry atmosphere is recommended.

ENGINEERING CERAMICS

See also Abstract(s): 213417 213420

213597
SURFACE MODIFICATION OF K₂TiF₆:Mn⁴⁺ PHOSPHOR WITH SrF₂ COATING TO ENHANCE WATER RESISTANCE
 Fang Z; Lai X; Zhang J; Zhang R - *Guilin, University of Technology*
Int.J.Appl.Ceram.Technol. 18, No.4, 2021, p.1106-1113
 K₂TiF₆:Mn⁴⁺ is an attractive narrow-band red-emitting phosphor for warm white light-emitting diodes (LEDs). Nevertheless, the hexafluoride phosphor is liable to deliquesce in moist environments, leading to a sharp deterioration in luminescent performance. The phosphor surface was modified with an SrF₂ coating, using a KHF₂ transition layer to moderate the lattice mismatch. The reaction mechanism and the effect of the SrF₂ coating on the luminescence intensity are discussed in detail. The coating is able to prevent hydrolysis of the [MnF₆]²⁻ group, and the luminescence intensity retains > 90% of its initial value after immersion in distilled water for 2 h. LED devices fabricated with commercial Y₃Al₅O₁₂:Ce³⁺ and

as-modified $K_2TiF_6:Mn^{4+}$ phosphors exhibit bright white light with tunable chromaticity coordinate, correlated colour temperature, and colour rendering index. The method described is a convenient way of enhancing the moisture resistance of Mn^{4+} doped fluoride phosphors for commercial white LEDs.

213598

HIGH-TEMP, HIGH PERFORMANCE - TECHNICAL CERAMICS HANDLE THE HEATMelrose B - *International Syalons (Newcastle) Ltd.***Interceram.** 70, No.3, 2021, p.16-19

Technical ceramics are used where conventional materials are prone to failure, or to refine equipment for greater throughput and profitability. Their thermomechanical properties have made them important in metal works, foundries and other ultra-high temperature sectors. Some technical ceramics offer long-lasting performance over multiple cycles, even when in direct contact with molten metals. Applications of high-performance technical grades of Si_3N_4 , ZrO_2 and sialon from International Syalons are discussed, including break rings in the horizontal continuous casting of steel, shielding thermocouples in foundries and aluminium smelting, and immersion and radiant heater tubes, die casting riser tubes and level sensors in the aluminium smelting industry. Syalon 110 is a Si_3N_4 -BN composite with excellent corrosion resistance to molten steel.

213599

SAFE DEBINDING AND SINTERING OF TECHNICAL CERAMICSHaigh P; Ohnweiller T; Piening R; Schenk C; Schneider S; Uhlemann-Koelly F; Weiss J - *Carbolite Gero Ltd.; Carbolite Gero GmbH & Co.KG***Interceram.** 70, No.3, 2021, p.23-25

The various manufacturing processes for technical ceramics have become significantly more extensive due to the possibilities of additive manufacturing. However, the challenges of subsequent debinding and sintering are often underestimated. Systems required for 3D printing and their safety concept to prevent explosions are discussed. Solutions for safe debinding and sintering available from Carbolite Gero for large and small systems are described.

Borides

See also Abstract(s): 213647 213684

Carbides

See also Abstract(s): 213659 213684 213689 213693

213600

DEVELOPMENT OF MULTILAYER GRADED CEMENTED CARBIDES WITH Ti-Zr CARBONITRIDE MISCIBILITY GAPSLi N; Wang Q; Zhang W; Du Y; Lv J; Liu Y; Li Y - *Shandong, University; Changsha, Central South University; Jiangxi University of Science & Technology***Ceram.Int.** 47, No.6, 2021, p.7521-7527

WC-Co-Ti(C, N)-ZrC cemented carbides with Ti-Zr carbonitride miscibility gaps were investigated under denitriding and nitriding conditions. Based on thermodynamic calculations, various ZrC contents were added to the samples to control the phase composition. The evolution of the graded layers was analysed using elemental distribution and concentration profiles. Results showed that two layers, namely, a fcc-free layer and a Ti-enriched layer, could be obtained in all the samples during the denitridation process. The first layer (the fcc-rich layer) and the second layer (the fcc-free layer) were observed in all the samples sintered under the nitridation process. However, a new third layer (Zr-enriched layer) was observed in samples with a high Zr content and the thickness of each layer was gradually reduced with increased ZrC addition. The diffusion behaviour of various elements and the mechanism of multilayer formation were clarified. This work could provide a new strategy for the development of novel graded cemented carbides.

213601

COMPLEX OPTIMISATION OF ARC MELTING SYNTHESIS FOR BULK Cr₂AlC MAX-PHASESobolev K; Pazniak A; Shylenko O; Komanicky V; Provino A; Manfrinetti P; Peddis D; Rodionova V - *Kaliningrad, Immanuel Kant Baltic Federal University; CRNS, Institut Pprime; Kosice, P.J.Safarik University; CNR-SPIN***Ceram.Int.** 47, No.6, 2021, p.7745-7752

Optimisation of the arc melting synthetic approach was performed to enhance the phase purity of Cr_2AlC MAX-phase in the bulk form. Optimisation steps included the initial ratio of 2Cr: xAl: 1C, variation through a change in the Al content (x ranged from 1 to 1.5), tuning duration of the post-annealing thermal treatment and adjustment of the melting chamber pressure. The use of Cr_3C_2 , as a precursor in place of a Cr and C mixture, was also tried. Cr_2AlC MAX-phase formed as the predominant phase when a stoichiometric ratio of 2Cr: 1.3Al: 1C was used. By maintaining this stoichiometry, the melting chamber pressure could be adjusted to further improve the phase purity of the samples. The use of Cr_3C_2 was not an appropriate way to improve sample purity and promote homogeneity in carbon distribution. The establishment of an optimised arc fusion protocol for parental Cr_2AlC is a necessity for further mass synthesis of the substituted $(Cr_1-xMnx)_2AlC$ MAX-phase.

213602

SUBCRITICAL CRACK GROWTH MODELS FOR STATIC FATIGUE OF HI-NICALON-S SiC FIBRE IN AIR AND STEAMHay R S; Robertson S J; Ruggles-Wrenn M B; Piper M; Shillig T; Mitchell R; Kroeger B; Gumucio L - *Wright-Patterson Air Force Base***J.Am.Ceram.Soc.** 104, No.7, 2021, p.3562-3592

Three subcritical crack growth (SCG) laws (a power law, a reaction-rate law and a bond-energy law) were used to model strain rates and failure times for the static fatigue of Hi-Nicalon-S SiC fibre tows in air and $Si(OH)_4(g)$ -saturated steam. The models were fitted to tow failure times and steady-state strain rates for brittle creep measured at 700-1100 C under initial applied stresses of 260-1260 MPa. The laws were used to describe SCG that caused sequential filament failure and ultimately tow failure. Two versions of each model were developed, one allowing access of chemisorbed species to flaws throughout the fibre (mode 1) and the other only allowing access to flaws at the SiC-SiO₂ interface (mode 2). The

stress increase on intact filaments as others fractured and as filaments oxidised and the increase in stress intensity geometric factors as the crack size increased were incorporated in the models. The fits to data were compared for the different models using both regression analysis and orthogonal distance regression (ODR) analysis. Faster convergence and more consistent results were achieved using ODR analysis. Regression analyses found parameters for all models with similar error in data fits, so the validity of a model could not be distinguished by regression analysis alone. For all models, the stress dependence of SCG rates was much stronger in steam than in air, and for most models activation energies were between 300 and 420 kJ/mol, regardless of environment. For the steam environment, the bond-length parameter for the bond-energy model was very close to the lattice parameter of beta-SiC (0.436 nm), but in air it was significantly lower at 0.25-0.26 nm, but still larger than the Si-C bond length of 0.189 nm. Other factors suggested that either a bond-energy based law or a modified version of a reaction-rate law are the best choices for a SCG law. Filament strength distributions initially described by Weibull distributions could not be described by such distributions after application of the models. SCG mechanisms are discussed.

213603

SYNTHESIS AND CHARACTERISATION OF TiC POWDERS BY SOL-GEL METHODGao L; Zhang Y; Yang X; He Y B; Song L H - *Changzhi, Medical College***J.Ceram.Process.Res.** 21, No.6, 2020, p.615-621

Titanium butyrate was added dropwise to a methyl alcohol solution of acetyl chelating agent. An aqueous solution of fructose was then slowly added, forming a sol. A gel was obtained by vigorously stirring the sol at 80 C. The gel was freeze dried under vacuum, ground and calcined at 1400 C in an argon atmosphere to obtain TiC powder by carbothermal reduction. The precursor materials and the TiC were characterised by FTIR and Raman spectroscopy, XRD and SEM. Single phase TiC, containing some titanium and carbon was obtained. The powder was equiaxed with an average grain size of approximately 1 micron.

213604

DENSIFICATION AND JOINING OF A (HfTaZrNbTi)C HIGH-ENTROPY CERAMIC BY HOT PRESSINGSun K; Yang Z; Mu R; Niu S; Wang Y; Wang D - *Tianjin, University; Tianjin, Key Lab. of Advanced Ceramics & Machining Technology***J.Eur.Ceram.Soc.** 41, No.6, 2021, p.3196-3206

Bulk (Hf_{0.2}Ta_{0.2}Zr_{0.2}Nb_{0.2}Ti_{0.2})C high-entropy ceramics (HECs), with a high density, were prepared by hot pressing (HP), and using a robust joining technique, large-sized pieces were fabricated. Hot-pressed carbide HECs, with a single-phase and homogeneous composition, were obtained at sintering temperatures between 1800 and 1950 C for 30 min under a pressure of 30 MPa. The effect of the sintering temperature on the mechanical properties of the HECs was investigated, and the flexural and compressive strengths are reported. Additionally, the feasibility of active brazing this HEC was studied and solid joints with high shear strength were obtained due to atomic diffusion and a chemical reaction at the interface, which provided a key approach to fabricate complex components of HECs.

213605

EFFECT OF PREPARATION CONDITIONS ON GAS PERMEABILITY PARAMETERS OF POROUS SiC CERAMICSXu X; Liu X; Wu J; Zhang C; Tian K; Yu J - *Wuhan, University of Technology***J.Eur.Ceram.Soc.** 41, No.6, 2021, p.3252-3263

The effects of binder content, firing temperature, SiC particle size, activated carbon content and activated carbon particle size on the gas permeability parameters of porous SiC ceramics were investigated according to their pore structure performance and microstructure. The degree of influence of these preparation conditions on the gas permeability parameters was also compared. Results showed that the Darcian permeability parameter and non-Darcian permeability parameter $k(1)$ increased with increased SiC particle size, activated carbon content and activated carbon particle size, but decreased with the binder content and firing temperature. The binder content and activated carbon particle size had more significant effects on $k(1)$ than the other three preparation conditions whose effect in descending order were activated carbon content, SiC particle size and firing temperature. The effect of the preparation conditions on $k(2)$ and $k(1)/k(2)$ were ambiguous, but it was clear that the SiC particle size had the greatest influence on $k(2)$ and $k(1)/k(2)$.

213606

THERMAL CONDUCTIVITY OF SPARK PLASMA SINTERED SiC CERAMICS WITH ALUMINA AND YTTRIAChai Z; Gao Z; Liu H; Zhang X; Glodan G; Xiao P - *Manchester, University***J.Eur.Ceram.Soc.** 41, No.6, 2021, p.3264-3273

Dense SiC ceramics were fabricated at 1650-1750 C between 10 and 60 min by spark plasma sintering (SPS) using 3-10 wt% Al₂O₃-Y₂O₃ as sintering additives. The effects of sintering temperature, sintering additive content and holding time on microstructure and correlations between the microstructure and thermal conductivity were investigated. Increased sintering temperature promoted grain growth. Extending the holding time had little effect on the grain size but resulted in the formation of a continuous network of sintering additive, which increased the interfacial thermal resistance which reduced the thermal conductivity. For SiC ceramics composed of a continuous SiC matrix and a discrete secondary phase (yttrium aluminium garnet, YAG), an increase in the sintering additive content resulted in a smaller grain size and lower thermal conductivity. The lower thermal conductivity of the SiC ceramics with higher sintering additive content was mainly due to the smaller grain size rather than the low intrinsic thermal conductivity of YAG.

213607

VAPOUR-MEDIATED MELT INFILTRATION FOR SYNTHESISING SiC COMPOSITE MATRICESSilverstein R; Zok F W; Levi C G - *California, University at Santa Barbara***J.Am.Ceram.Soc.** 104, No.8, 2021, p.3833-3844

SiC was synthesised in a two-step process involving initial exposure of the carbon surfaces to Si vapour, followed by Si melt infiltration and the mechanisms, kinetics and microstructural evolution were examined. Interrupted DTA of amorphous C and Si powder mixtures and microstructure characterisation were used to generate insight into the stages of the reaction. Exposure to Si vapour gave a SiC layer with nanoscale porosity driven by the volume change associated with the reaction. This formed a continuous pore network that promoted subsequent melt access to the reaction front with the C. While the pores remained open, the vapour phase reaction proceeded at a nearly constant rate and showed strong

temperature sensitivity, the latter due in part to the temperature sensitivity of the Si vapour pressure. The implications for enhancing the reactive melt infiltration process and fabrication of SiC matrices for ceramic composites are discussed.

213608
(W1-x,Mx)C CARBIDES WITH DESIRED COMBINATIONS OF COMPATIBLE DENSITY AND PROPERTIES - A FIRST-PRINCIPLES STUDY
 Liu R; Zhang D; Tang Y; Tang X; Humphries E; Li D - *Alberta, University; Harbin, Engineering University; Weir Minerals Australia Ltd.*
J.Am.Ceram.Soc. 104, No.8, 2021, p.4239-4256
 Tungsten monocarbide (WC) is widely used as a reinforcement in hardfacing overlays, thermal spray coatings, composites and various alloys. However, its large density leads to the inhomogeneous distribution of WC particles in the metal-matrix hardfacing overlays. It is highly desirable to have appropriate reinforcing phases with an optimal combination of high strength, compatible density and physical properties. WC was tailored by partially substituting W with 3d and 4d transition metals through first-principles calculations. Metal substitutions included Sc, Ti, V, Cr, Mn, Fe and Mo. It was shown that WC could be tailored by element-substitution with desired properties. Identified stable (W1-x,Mx)C carbides had lowered density and mechanical properties comparable to those of WC. Physical properties, e.g. the Debye temperature, Gruneisen parameter and thermal conductivity, of the tailored carbides were also studied for extended applications. Attempts were made to generate comprehensive information on metal-substituted (W1-x,Mx)C with explained underlying mechanisms through analysing the corresponding electronic characteristics.

213609
PROCESSING AND MICRO-MECHANICAL CHARACTERISATION OF MULTI-COMPONENT TRANSITION MC CARBIDES IN IRON
 Deillon L; Fornabaio M; Zagar G; Michelet L; Mortensen A - *Ecole Polytechnique Federale de Lausanne*
J.Eur.Ceram.Soc. 41, No.7, 2021, p.3937-3946
 Multi-component transition monocarbides of chosen composition were prepared by arc-melting together a pre-alloy of the transition metals and cast iron. Based on the elements Ti, Ta, V, Nb and W, 51 different binary, ternary and quaternary compositions were produced. The intrinsic hardness H and elastic modulus E of the resulting iron-embedded carbide particles were directly measured using nanoindentation. Of all the compositions tested WC showed the highest modulus while two (Ta,V)C and (Ti,W)C carbides had a hardness that was 15% higher than that of all binary carbides and some (Ti,Ta,V)C compositions displayed interesting combinations of properties. The modulus and hardness variations showed that the valence electron concentration, which had been proposed to be a dominant parameter in predicting the carbide hardness and modulus, was not a useful single predictor of optimal compositions. Other important parameters also governed the hardness and modulus of the MC carbides.

213610
INVESTIGATION OF INTERFACIAL REACTION MECHANISM BETWEEN SiC AND INCONEL 625 SUPERALLOY USING THERMODYNAMIC CALCULATION
 Shi H; Chai Y; Li N; Yan J; Peng H; Zhang R; Li M; Bai D; Chen K; Liu Z; Luo M; Sun Q; Zhu X; Zhang Y; Li R; Zhang B; Dong X - *Sichuan, University; China Nuclear Power Technology Research Institute; Shanghai, University; Chengdu Keningda Materials Co.Ltd.*
J.Eur.Ceram.Soc. 41, No.7, 2021, p.3960-3969
 The interfacial reaction between SiC ceramics bonded using an Inconel 625 superalloy interlayer at different temperatures was investigated. The CALPHAD method was used based on a Ni-Cr-Si ternary thermodynamic database to analyse the reactions between the Inconel 625 superalloy and the SiC ceramics. When the liquid alloy reached a specific composition, the reactions between them stopped. Equilibrium was established between the liquid alloy and SiC during the joining process, regardless of the temperature change. Furthermore, thermodynamic predictions of the reaction products were correct using EBSD and XRD. This work has demonstrated that CALPHAD is a suitable method for effectively investigating this complex reaction system and further promoting precise theoretical analysis.

Nitrides

See also Abstract(s): 213416 213422 213598 213693

213611
MICROSTRUCTURE AND PROPERTIES OF TiAlCrN CERAMIC COATINGS DEPOSITED BY HYBRID HiPIMS/DC MAGNETRON CO-SPUTTERING
 Gui B; Zhou H; Zheng J; Liu X; Feng X; Zhang Y; Yang L - *Lanzhou, Institute of Physics; Anhui, University of Technology*
Ceram.Int. 47, No.6, 2021, p.8175-8183
 TiAlCrN coatings were prepared by a hybrid deposition technique consisting of high power impulse magnetron sputtering (HiPIMS) and direct current magnetron sputtering (DCMS). The chemical composition, phase structure, morphologies, mechanical and tribological properties of the coatings were investigated. Results indicated that the content of Ti increased monotonically from 0 to 22 at% with increased Ti target power. The TiAlCrN coatings had a competitive growth tendency between (111) and (200) crystal plane due to energetic ion bombardment. A higher Ti target power resulted in a stronger compressive intrinsic stress, which suppressed the precipitation of the hcp-AlN phase. With increased ion bombardment, the diffusion energy and nucleation rate of the adatoms on the growing surface increased, which caused a denser structure and an ultra-smooth surface. The hardness and toughness also varied as a function of the Ti target power, with a maximum hardness of 28.3 GPa under a Ti target power of 5 kW. A positive correlation was discovered between the adhesion strength (i.e., the critical load of the scratch test) and H3/E2 ratio which indicated a strong dependence of the adhesion properties on toughness for the TiAlCrN ceramic coatings, which agreed with literature data. As for the tribological behaviour, the lowest wear rate of $8.9 \times 10 \exp(-17) \text{ m}^3/\text{Nm}$ was obtained for the TiAlCrN coating deposited at a Ti target power of 5 kW.

Oxides

See also Abstract(s): 213419 213598 213642 213665 213668 213687 213690

213612
MICROSTRUCTURAL TRANSITIONS IN CAMPHENE-BASED FREEZE CASTED CERIA: EFFECT OF PRIMARY BUILDING BLOCKS
 Mukkavilli R S; Papakollu K; Kumar R - *Chennai, Indian Institute of Technology Madras*
Adv.Appl.Ceram. 120, No.4, 2021, p.248-253
 Porous ceria for high temperature catalytic applications necessitates structural integrity as well as sinter resistance and enhanced gas permeability.

The current state of the art technology is based on complicated synthesis techniques that are not only costly but also lack pore tailorability flexibility. As a result, the creation of porous scaffolds using low-cost methods without sacrificing functionality is necessary. For the first time, porous ceria with an open porosity of 88% was created via camphene aided freeze casting. Microstructural evolution with various building pieces, including micrometre-sized particles and short fibres, was also investigated. Preliminary catalytic activity achieved by temperature controlled reduction exhibited comparable patterns, demonstrating that the starting building blocks had no influence on the activity.

213613

OUTSTANDING SINTERING RESISTANCE IN PYROCHLORE-TYPE $\text{La}_2(\text{Zr}_{0.7}\text{Ce}_{0.3})_2\text{O}_7$ FOR THERMAL BARRIER COATINGS MATERIAL

Che J; Wang X; Liu X; Liang G; Zhang S - *Xian Jiaotong University; Xian, Chang'an University; Beijing, Tsinghua University*

Ceram.Int. 47, No.5, 2021, p.6996-7004

The sintering behaviour of pyrochlore-type $\text{La}_2(\text{Zr}_{0.7}\text{Ce}_{0.3})_2\text{O}_7$ (LZ7C3) was investigated experimentally and using molecular dynamics. Results showed that novel LZ7C3 exhibited higher sintering resistance than host $\text{La}_2\text{Zr}_2\text{O}_7$ (LZ) and 8 wt% yttria-stabilised zirconia (8YSZ) at temperatures up to 1773 K, which indicated that pyrochlore-type LZ7C3 is a promising thermal barrier coating (TBC) candidate to replace conventional 8YSZ at high temperatures. Further study also revealed that the initial stage played a crucial role in sintering process and the sintering mainly occurred in the grain boundary (GB) region. The intrinsic sintering activation energy of LZ7C3 GB (695.248 J/mol) was larger than that of LZ GB (384.171 J/mol) and 8YSZ GB (173.303 J/mol), which resulted in outstanding sintering resistance for LZ7C3. No obvious enrichment of the secondary phase was observed at the GBs of LZ7C3. It was concluded that hindering the atomic diffusion of GBs and the introduction of a foreign atom with larger mass and bond energy could act as an effective strategy to enhance the sintering resistance of TBC materials.

213614

STRUCTURE-PROPERTY RELATIONS FOR A PHASE-PURE, NANOGRAINED TETRAGONAL ZIRCONIA CERAMIC STABILISED WITH MINIMUM CaO DOPING

Arun A; Kumar L; Chowdhury A - *Indian Institute of Technology*

J.Am.Ceram.Soc. 104, No.7, 2021, p.3497-3507

Dense (about 97%) ZrO_2 ceramic was stabilised with 3 mol% CaO (the minimum reported to date) and sintered at a low temperature (about 1200 C). The CaO doping accuracy was confirmed by compositional analysis via XRF. The phase-pure tetragonal structure (characterised by both XRD and Raman spectroscopy) and uniform nanograins (90 nm) of the ceramic ensured that no monoclinic phase evolved, even after vigorous low-temperature degradation experiments (both thermal and hydrothermal ageing for 80-100 h). The sintered ceramic recorded a high hardness (about 15 GPa) and the indentation toughness value was also comparable to a 3 mol% yttria-stabilised zirconia system. The remarkable structure-property correlations in the 3 mol% CaO-stabilised ZrO_2 ceramic suggest that it may be worth examining for suitable future applications such as dental ceramics.

213615

TOUGHENING OF PYROCHLORE $\text{La}_2\text{Zr}_2\text{O}_7$ BY A FERROELASTIC NdAlO_3 SECOND PHASE FOR POTENTIAL THERMAL BARRIER COATING APPLICATIONS

Wang Y; Han J; Du J; Liu R; Wan F - *Changsha, National Univ. of Defence Technology*

J.Am.Ceram.Soc. 104, No.7, 2021, p.3508-3517

The poor fracture toughness of $\text{La}_2\text{Zr}_2\text{O}_7$ severely limits its application as a high temperature thermal barrier coating topcoat material. A ferroelastic second phase, NdAlO_3 , with a Curie temperature of 1367 C was introduced to form $x\text{NdAlO}_3/(1-x)\text{La}_2\text{Zr}_2\text{O}_7$ ($x = 10, 20, 30, 40$ and 50 mol%) composite ceramics by spark plasma sintering to toughen the material. The fracture toughness of the sintered composite ceramic compacts was measured at both room temperature and 1200 C by a single-edge-notch beam test method. It was shown that, at room temperature, the residual compressive stress in the $\text{La}_2\text{Zr}_2\text{O}_7$ matrix was important to toughen the composites. By eliminating this factor, the remaining toughening effects agreed with the measured fracture toughness at 1200 C, suggesting that the other toughening was probably ferroelastic domain switching toughening and that it was still valid at high temperature. The toughening effect arising from ferroelastic domain switching was governed by the overall domain switching zone, which was determined by both individual domain switching zone width and the "concentration" of ferroelastic phases. A relatively high coercive stress of NdAlO_3 and relatively low residual tensile stress in NdAlO_3 second phases contributed to the negligible influence of residual tensile stress on the domain switching zone width, leading to the continuous increase of fracture toughness of the composite ceramics with more NdAlO_3 added at room temperature.

213616

CORROSION RESISTANCE OF NONSTOICHIOMETRIC GADOLINIUM ZIRCONATE COATINGS AGAINST CaO-MgO- Al_2O_3 - SiO_2 SILICATE

Zhang C; Fan Y; Zhao J; Chen H; Sun L; Yang G; Liu B - *Shanghai University; Shenyang, Institute of Metal Research*

J.Eur.Ceram.Soc. 41, No.6, 2021, p.3687-3695

Three gadolinium zirconate coatings, with different Gd/Zr ratios, were prepared via atmospheric plasma spraying using an amorphous feedstock. Their mechanical properties and corrosion resistance were investigated. The Young's moduli and hardness of the as-sprayed coatings were comparable with the gadolinium zirconate coatings reported in previous literature. Furthermore, the higher Gd content promoted the formation of Gd-apatite and the depletion rate of CaO-MgO- Al_2O_3 - SiO_2 (CMAS) corrosion. As a result, the infiltration depth of the $\text{Gd}_{2.3}\text{Zr}_{1.7}\text{O}_{6.85}$ coating, after 24 h annealing, decreased up to 35 % compared with those of $\text{Gd}_{2.0}\text{Zr}_{2.0}\text{O}_{7.0}$ and $\text{Gd}_{1.8}\text{Zr}_{2.2}\text{O}_{7.1}$, which exhibited enhanced long-term corrosion resistance. This work had developed a viable fabrication method to produce non-stoichiometric gadolinium zirconate coatings with tailored CMAS corrosion resistance and is expected to promote the future design of thermal barrier coatings with a long service life.

213617

INTERPLAY OF THE PHASE AND THE CHEMICAL COMPOSITION OF THE POWDER FEEDSTOCK ON THE PROPERTIES OF POROUS 8YSZ THERMAL BARRIER COATINGS

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J.Eur.Ceram.Soc. 41, No.6, 2021, p.3706-3716

Some chemical impurities enhance sintering kinetics of ceramic thermal barrier coatings (TBCs) which can cause their premature failure during operation while others can suppress the resistance to tetragonal to monoclinic phase transformation in 8YSZ, which can also be an important factor

regarding TBC's performance. Most of the impurities and some of the monoclinic phase present in the powder feedstocks can survive into the as-sprayed coating. This paper investigated the role and the relative importance of the chemical and phase composition of the powder feedstock on the properties and performance of thick 8YSZ TBCs.

213618

EFFECTS OF MICRO-SIZED MULLITE ON CRISTOBALITE CRYSTALLISATION AND PROPERTIES OF SILICA-BASED CERAMIC CORES

Zheng W; Chen X; Liu C; Ren S; Zhang L - *Shenyang, Northeastern University*

Int.J.Appl.Ceram.Technol. 18, No.4, 2021, p.1244-1254

Silica-based ceramic cores are extensively used in the investment casting process, during which they must exhibit sufficient flexural strength and deformation resistance. Micron-sized mullite was incorporated as an additive in SiO₂-based ceramic cores in order to optimise their high-temperature properties. Ceramic cores with different amounts of micron-sized mullite were fabricated. XRD showed that micron-sized mullite additions diminished the crystallisation of cristobalite at high temperatures, mainly due to mullite-related compressive stresses on the surfaces of fused silica particles. Three-point bending tests and SEM showed that micron-sized mullite had a more significant effect on the flexural strength of ceramic cores compared with conventional additives; in particular, the fracture mechanism of the cores changed from intergranular to a mixed behaviour consisting of intergranular and transgranular. The mechanical and thermal properties of the cores were all slightly reduced for mullite contents > 4.6 wt%. Thus, to optimise the properties of SiO₂-based ceramic cores, the micron-sized mullite content should not exceed 4.6 wt%.

213619

PREPARATION OF ZrO₂ BEADS BY AN IMPROVED MICRO-DROPLET SPRAY FORMING PROCESS

Chen J; Xue W-Q; Xu C-M; Luo P-F; Cheng J-G; Lu Y-W - *Hefei, University of Technology; Suzhou Nutpool Materials Technology Co.Ltd.; Jiangxi Nutpool Industrial Co.Ltd.; Hefei, Res. Centre for Powder Metallurgy Engineering & Technology*

J.Am.Ceram.Soc. 104, No.8, 2021, p.3855-3861

Monodisperse ZrO₂ ceramic beads > 1 mm in size were prepared by an improved micro-droplet spray forming process, through which a compressor and a dispenser were used to produce droplets continuously. The slurry recipe and drying temperature were optimised to enhance the sphericity and smoothness of the beads. The sintered ZrO₂ ceramic beads had promising mechanical performance, including a relative density of 84.6%, a crushing strength of 256.2 plus or minus 36.6 N and a Vickers hardness of 1344.4 plus or minus 58.3 HV. This procedure has great potential in the mass production of ceramic beads.

213620

ENHANCED THERMAL STABILITY OF HIGH YTTRIA CONCENTRATION YSZ AEROGELS

Olson N S; Hurwitz F I; Guo H; Madden N J; Stokes J L; Rogers R B; Krogstad J A - *Illinois, University at Urbana Champaign; NASA, Glenn Research Center; Universities Space Research Association*

J.Am.Ceram.Soc. 104, No.8, 2021, p.4190-4202

Aerogels are promising for lightweight, high-performance insulation for use in aerospace applications, but their high specific surface area contributes to rapid densification of the structure at elevated temperatures. Upon densification, their low thermal conductivity and low density are lost. Doped metal oxide systems offer a route to the stabilisation of porous structures at high temperatures and a platform to study parameters conducive to thermal stability. Yttria-stabilised zirconia (YSZ) aerogels prepared by a sol-gel method and supercritically dried were investigated. Yttria concentrations from 0 to 50 mol% YO_{1.5} were studied to stabilise the porosity to temperatures of 1200 C and develop an understanding of properties contributing to improved stability. Increased yttria content improved the thermal stability of the pore structure by reducing densification and suppressing crystallite growth, resulting in retention of the mesoporous structure to 1200 C. The improvement in thermal stability was related to the associated reductions in specific surface energy and cation diffusivity at higher yttria concentrations. The work showed that tuning the thermodynamic and kinetic factors is a viable route to improve thermal stability in highly porous structures for use as insulation in extreme environments.

REFRACTORIES

See also Abstract(s): 213298 213303 213304 213309 213598 213683

213621

CHARACTERISATION OF SPINEL BUBBLES AND PREPARATION AND PROPERTIES OF LIGHTWEIGHT CERAMICS

Chen Z; Chen H; Yang H; Wang J - *Zhejiang, University; Zhejiang, California International Nanosystems Institute*

Ceram.Int. 47, No.5, 2021, p.6513-6520

Spinel bubbles were prepared using an electrofusion injection process and their bulk density, volume density and pressure resistance of different sizes were investigated. Lightweight spinel bubble ceramics with different densities were prepared using electrofused spinel bubbles as light aggregates with rho-Al₂O₃ and caustic burnt magnesia powder as matrix powders. The microstructures of different sized spinel bubbles and the lightweight spinel ceramics were assessed by SEM. Results showed that the spinel bubble were complete with good sphericity, perfect grain crystallisation and clear edges and corners. Although there were some fine cracks and cavities the grains were tightly bound together. The compressive strength and bending strength of the lightweight spinel ceramics, with a density between 0.99 and 1.63 g/cm³, were 5.19-36.33 MPa and 3.48-12.84 MPa, respectively.

213622

EFFECT OF MICROPOROUS MAGNESIA AGGREGATES ON MICROSTRUCTURE AND PROPERTIES OF PERICLASE-MAGNESIUM ALUMINATE SPINEL CASTABLES

Yan J; Yan W; Schaffner S; Dai Y; Chen Z; Wang Q; Li G; Jia C - *Wuhan, University of Science & Technology; Connecticut, University; Huaneng Hainan Power Generation Co.Ltd.*

Ceram.Int. 47, No.5, 2021, p.6540-6547

Three lightweight periclase-magnesium aluminate spinel castables, containing microporous magnesia aggregates with a varying apparent porosity (12.8%, 30.8% and 39.3%), were investigated. The effect of the apparent porosity of the aggregates on the phase composition, microstructure,

fracture behaviour and strength of the lightweight castables was investigated using XRD, SEM and three-point bending tests. Large cracks between the aggregates, with an apparent porosity of 12.8%, and the matrix reduced the strength of the castable. For aggregates with an apparent porosity of 30.8%, an excellent interlocking interface with the matrix increased the strength but also reduced the fracture toughness. At the highest level of apparent porosity for the aggregates of 39.3%, the formation of a small number of microcracks between the aggregates and matrix reduced the strength, while the fracture toughness was only slightly affected. Lightweight castables with the best combination of properties were achieved at an apparent porosity for the aggregates of 30.8% since they had a low bulk density of 2.63 g/cm³ and a high compressive and flexural strength of 70.2 MPa and 20.9 MPa, respectively.

213623
EFFECT OF RICE HUSK POWDER AS A BINDER ON MECHANICAL AND THERMAL PROPERTIES OF ZrO₂ HOLLOW-FIBRE REFRACTORY BRICKS
Liu X; Wang T; Kong J; Wong C - *Nanjing, University of Science & Technology; Georgia, Institute of Technology*
Ceram.Int. 47, No.6, 2021, p.8685-8691

ZrO₂ hollow-fibre bricks, with low density and thermal conductivity, have thermal insulating applications but they have low strength and a binder that can overcome these challenges was investigated. Rice husk, containing a large concentration of amorphous SiO₂, was investigated as a suitable binder. After treatment, the rice husk powder was added to the ZrO₂ hollow fibres as a high-temperature binder. The densities and thermal conductivities of these ZrO₂ bricks prepared at 1300 C were unaffected by the rice husk-derived binder, while the compressive strengths increased by 285% compared with that of the Zr-sol binder.

213624
INFLUENCE OF THE PARTICLE SIZE DISTRIBUTION OF COARSE-GRAINED REFRACTORIES ON THE THERMAL SHOCK PERFORMANCE
Fruhstorfer J - *Leoben, Montan-Universitat*
J.Aust.Ceram.Soc. 57, No.3, 2021, p.899-909

The effect of particle size distribution (PSD), characterised by the amounts of fine and coarse particles and by the maximum particle size (1-3 mm) and the compaction pressure (50-100 MPa) of die-pressed refractory bars on the bulk density, cold modulus of rupture (CMOR) and Young's modulus, E, during five thermal shocks (TS) was studied. The highest densities were obtained for wide PSDs with the densest packed coarse aggregates, a slight excess of lubricating fines and a high compaction pressure. The highest CMOR and E were obtained for the same parameters with the exception of a maximum particle size of 1 mm inducing lower flaw sizes. Independent of the compaction pressure, the lowest CMOR losses during TS were obtained for a wide PSD with a high number of aggregates constituted of medium-sized grains and the most densely packed coarse particles. Crack deflection and contact shielding toughening supposedly occurred. During TS, E behaved differently; it was predictably low when there were many flaws. During TS, these led to a large process zone and a high decrease of E whereas, for batches with a higher initial E, a crack could propagate with less microstructural interactions, supposedly giving a smaller process zone and a reduced decrease.

213625
IMPACT OF PREHEATING ON THE MECHANICAL PERFORMANCE OF DIFFERENT MgO-C BRICKS -INTERMEDIATE TEMPERATURE RANGE
Gass S E; Galliano P G; Tomba Martinez A G - *INTEMA; Tenaris Siderca*
J.Eur.Ceram.Soc. 41, No.6, 2021, p.3769-3781

The mechanical behaviour of MgO-C refractories at an intermediate temperature of 1000 C were investigated. Conditions similar to those occurring during the in-service pre-heating of steelmaking ladles were simulated by thermal treatment before mechanical testing. Stress-strain curves were determined under compressive loading at RT and 1000 C. The impact of the thermally activated physicochemical transformations on the mechanical response of the materials was evaluated in terms of parameters including strength, fracture deformation, secant modulus and yield strength. The presence of Al seemed to be deleterious when longer soaking times at 1000 C were used and was attributed to the progressive damage related to in-situ phase formation. But, when heating these types of refractories simultaneously with the application of a low compressive load, the closure of previously formed microcracks (self-healing) was considered to be mainly responsible for their mechanical behaviour.

213626
3D PRINTING OF DENSE AND POROUS ALKALI-ACTIVATED REFRACTORY WASTES VIA DIRECT INK WRITING (DIW)
Coppola B; Tardivat C; Richaud S; Tulliani J-M; Montanaro L; Palmero P - *Turin, Polytechnic; Saint-Gobain CREE*
J.Eur.Ceram.Soc. 41, No.6, 2021, p.3798-3808

Alkali-activated pastes, prepared from refractory waste, were 3D-printed via direct ink writing. Two different aluminosilicates were used: chamotte (CH, mainly composed of corundum, mullite and andalusite) and alumina-zirconia-silica (AZS, mainly baddeleyite, corundum and amorphous silica). Initially, rheological parameters of the pastes were optimised in terms of liquid-to-solid ratio and polyethylene glycol content. Dense prismatic samples and lattice structures were then 3D-printed. PMMA beads, 20 and 60 micron in diameter, were used to generate additional porosity in both dense and lattice structures, which led to hierarchically porous materials. The samples were partially sintered up to 800 C to decompose the PMMA beads and improve the mechanical properties. A flexural strength of approx. 45 MPa was obtained for both AZS and CH dense samples and the addition of 30 vol% PMMA beads led to macroporous samples without affecting their mechanical properties.

213627
PHENOL-FREE REFRACTORY LINING - ANOTHER INNOVATION FOR BETTER WORKPLACE AND ENVIRONMENT
Wonjin Worldwide
Refract.Worldforum. 13, No.3, 2021, p.11-12

A new eco-friendly phenol-free refractory brick has been launched by South Korean company H Steel which significantly reduces odour without negative side-effects including heat resistance and wear resistance compared to traditional resin-bonded refractory products. Historically, resin-bonded bricks were developed to replace pitch-bonded bricks to reduce emission of unfavourable gases. Although resin-bonded bricks are manufactured by drying at about 250 C to remove volatiles, the remaining phenol substances still emit a small amount of chemical substances during preheating with significant odour. The phenol-free technology does not emit any gases significantly other than CO₂, nor require any further treatment.

- 213628
RISE ABOVE DOWNTIME: THREE WAYS SUSPENDED PLATFORMS INCREASE EFFICIENCY FOR VERTICAL VESSEL MAINTENANCE
 Mirisola J - *Bricking Solutions*
Refract.Worldforum. 13, No.3, 2021, p.13-14
 Maintenance and repair of vertical vessels is costly in time and lost productivity, partly because scaffolding is required to access the vertical surfaces which limits efficiency and increases safety risks. Custom-manufactured suspended platforms offer benefits over scaffolding systems. The systems feature a lightweight, heavy-duty metal platform erected inside the vessel and raised or lowered using manual or electric hoists. Benefits include reduced downtime, improved worker safety, easier working conditions, ability to access all parts and improved productivity.
- 213629
REFRACTORIES TESTING AND THE SIGNIFICANCE OF CHEMICAL AND PHYSICAL CHARACTERISTICS. PT.2. THE PHYSICAL PROPERTIES OF REFRACTORIES MATERIALS
 Jarvis D A
Refract.Worldforum. 13, No.3, 2021, p.15-18
 The main aspects of refractories testing were studied with regard to achieving optimum lining configurations. Physical and thermomechanical properties and their test methods are summarised looking at bulk density, apparent porosity, cold crushing strength, refractoriness, refractoriness-under-load, hot modulus of rupture, dimensional change, thermal conductivity, abrasion resistance and corrosion resistance.
- 213630
PROMOTION AND SUPPORT OF REFRACTORY SCIENCE AND ENGINEERING
 Semler C E - *Semler Materials Services*
Refract.Worldforum. 13, No.3, 2021, p.19-20, 22, 24, 26-27
 The 2021 St Louis Refractory Symposium was conducted online. This Zoom paper reviewed the contributions of Professor Richard Bradt (1938-2019) over the years 1965-2000 when he published 320 papers. His research focused on properties of refractories, ceramics, eutectics, minerals and glass, specifically mechanical properties with the application of fracture mechanics. He was also responsible for setting up the UNITECR refractory conferences and the international symposia on the Fracture Mechanics of Ceramics. His research topics are summarised, looking at work on control of particle size grading, routine thermal shock testing, fracture energy testing, raw materials, castables, magnesia-carbon/graphite refractories, thermal shock resistance and optimisation of tests. His legacy for the refractory industry is highlighted.
- 213631
VISCOSITY CHANGES OF AN ALUMINA BASED CASTABLE DURING MIXING AND THE EFFECT OF BIMODAL REACTIVE ALUMINAS
 Dunzen C; Janousch D - *Nabaltec AG*
Refract.Worldforum. 13, No.3, 2021, p.33-37
 Castables with very low water contents exhibit strong dilatancy, although the viscosity at high shear rate is not usually measured. The rheological phenomena of a castable occurring during mixing and therefore at a high shear rate were studied, using a pure alumina ultralow cement castable with a bimodal matrix composed either of two reactive aluminas with D50 values of 2.5 and 0.8 micron or of pre-homogenised bimodal reactive aluminas with the same particle sizes. A setup was created to measure the power consumption of the mixer and enable the viscosity to be determined at higher shear rate. Power consumption plotted against the mixing time resulted in characteristic curves. The mixing process could be divided into distinct stages, such as wet out point, when the particles started to form a coherent mass with very high viscosity, continuous decline of viscosity and then a further increase. The dispersion, coagulation and electrochemical interactions between the cement and deflocculant were studied. It was shown that a bimodal reactive alumina dispersed much faster than a bimodal mix of two reactive aluminas. The power consumption monitoring is proposed as a viable method to ensure that castables are mixed to the optimal viscosity.
- 213632
FORMATION OF HEXA-ALUMINATE SOLID SOLUTION PHASES IN SPINEL CONTAINING CASTABLES - MINERALOGICAL INVESTIGATIONS IN THE SYSTEM CaO-Al₂O₃-MgO
 Klaus S; Buhr A; Schustereder M; Gobbels M; Dutton J - *Almatis GmbH; GeoZentrum Nordbayern*
Refract.Worldforum. 13, No.3, 2021, p.38-44
 Magnesium aluminate spinel can improve the thermal stability of alumina-based refractories. Refractories used for applications such as purging plugs or impact pads show increased thermomechanical stability and high erosion resistance when spinel is included. The formation of ternary hexa-aluminate phases CAM I (C₂M₂A₁₄) and CAM II (CM₂A₈) between 1400 and 1700 C was studied. It was shown that CAM phases grew into sintered alumina grains resulting in interlocking matrix and aggregate compounds. The resulting microstructure contributed to improved hot strength properties, especially thermomechanical resistance. White-fused alumina aggregates showed a weaker reaction with the surrounding matrix, leading to less potential for interlocking.
- 213633
STUDY OF LIME KILN REFRACTORY FAILURE SOLUTION
 Cristante A; de Moraes E M; Carvalho R P B; Mendes R L M; Vernilli F - *Sao Paulo, University; Rio de Janeiro, Lime Plant*
Refract.Worldforum. 13, No.3, 2021, p.45-49
 A post-mortem study was carried out on a lime kiln refractory lining. Three refractory failures (thermal shock, discrepancy in the material properties and excessive abrasion wear) were hypothesised and XRF, XRD, elastic modulus, abrasion tests and SEM used to compare post-mortem refractories to new ones. There was found to be no difference in chemical composition, crystalline phases, modulus of elasticity, abrasion resistance and microstructure between the new and post-mortem bricks. It was concluded that the premature failure of the aluminosilicate bricks was related to limestone abrasion during limestone decomposition. Refractory bricks with higher abrasion resistance were therefore recommended with the new refractory profile design.

213634

SETTING PROPERTIES OF REFRACTORY CASTABLES: INFLUENCE OF IMPURITIES ON COAGULATION AND HYDRATION - A CASE STUDY BASED ON THE ADDITION OF TRACES OF MgCl₂Kasper J; Dannert C; Koch A; Krause O - *Forschungsgemeinschaft Feuerfest Ev; Koblenz, Hochschule***Refract.Worldforum.** 13, No.3, 2021, p.50-58

Refractory castable linings need to be homogeneous to ensure predictable lining lifetime. Modern dispersed castables must demonstrate the same working conditions in terms of rheology and setting behaviour whatever the ambient conditions at the construction site. Ambient temperature can be corrected, but the effect of soluble ions introduced by impurities related to raw materials is more difficult to suppress. Traces of soluble ions can affect the workability, setting kinetics and strength evolution of calcium aluminate cement-containing refractory castables. Model castables were used to study the effect of MgCl₂ on the setting and curing behaviour, measured by sonic velocity and deriving the formation of hydrate phases from the concentration of hydrate bond water. Matrix compositions were used to analyse the effect of MgCl₂ on the time-dependent change of the zeta potential during early setting, enabling the coagulation behaviour to be understood. When 0-0.018 mass% MgCl₂ was dissolved in deionised mixing water and added to a corundum-based model castable, the corresponding matrix composition (grain size < 45 micron), the dispersibility of the refractory castable deteriorated significantly in most cases. The first stiffening and the period of main hydration found to be accelerated retarded significantly, depending on the type of dispersant used.

213635

IMPROVED EXPLOSION RESISTANCE OF LOW CEMENT REFRACTORY CASTABLESPeng H; Myhre B - *Elkem Silicon Materials***Refract.Worldforum.** 13, No.3, 2021, p.59-64

The explosion resistance of low cement castables (LCCs) containing different drying agents (polymer fibres and EMSIL-DRY) has been studied for both laboratory and industrial scale specimens. It was found that the type of drying agent had a significant effect on the flow/workability of the fresh castables and on the explosion resistance of the LCCs during firing. Compared with polymer (polypropylene) fibres, EMSIL-DRY reduced the temperature level for the maximum dewatering rate, thus preventing explosions during the heat-up step. The production of a perfect 400 kg block fired to 850 C at a rate of 75 C/h demonstrated that the LCC with EMSIL-DRY had excellent explosion resistance. The thermal behaviour of 300 mm cubes (about 80 kg) of selected LCCs was studied using Macro-TGA, a macro thermobalance.

213636

EVALUATION OF OXIDATION RESISTANCE OF MgO-C BRICKS IN OXY-COMBUSTION AND AIR-COMBUSTIONZanotelli V H; Ribas L L; Braganca S R - *Rio Grande do Sul, Universidade Federal***Int.J.Appl.Ceram.Technol.** 18, No.4, 2021, p.1392-1403

Oxy-fuel technology offers important advantages over conventional combustion, such as the reduction in fuel consumption and gaseous emissions. However, before employing it in the steel industry, it is essential to be aware that the alteration in the firing system can lead to an increase in wear of the oxide-carbon refractories. Four types of MgO-C bricks were fired under oxy-fuel and air-fuel conditions in a semi-industrial furnace, simulating the heating of a steelmaking ladle. The fired bricks were evaluated in terms of weight loss, thickness of the decarburised layer, and phases and microstructure formed in this layer. The results indicated few differences in the properties of the bricks fired in the two systems. The brick with 12% carbon and Al antioxidant showed the highest oxidation resistance among all the studied bricks. The most important factor for using an oxy-fuel system is the correct selection of refractory microstructure, including the utilisation of antioxidants.

213637

IMPACT OF MgO-C WITH CMA- AND NANOCARBON ADDITION ON STEEL CLEANLINESSGehre P; Preisker T; Dudczig S; Schmidt G; Wohrmeyer C; Parr C; Aneziris C G - *Freiberg, TU Bergakademie; Imerys SA***Interceram.** 70, No.3, 2021, p.26-31

Non-metallic inclusions in molten steel mainly originate from the refractory-slag-steel interactions during continuous casting and considerably affect its cleanliness. A new approach to minimise non-metallic inclusions by interaction with a suitable MgO-C-based lining material was developed. In order to study the efficiency of MgO-C and MgO-CMA-C to reduce endogenous inclusions, immersion tests were carried out in a metal casting simulator at 1650 C. A special automatic SEM was used to investigate the inclusions.

213638

COMPARISON OF THERMAL SHOCK DAMAGE AND SLAG CORROSION IN CASTABLE REFRACTORIESCristante A; Vernilli F - *Sao Paulo, University***Interceram.** 70, No.3, 2021, p.32-37

The thermal shock damage resistance and the slag corrosion resistance of a high-alumina and an alumina-containing andalusite refractory castable were compared. Thermal shock damage resistance was analysed using thermal cycling and thermal shock damage resistance parameters. Thermal shock cycling damage was analysed using the dynamic modulus of elasticity and the modulus of rupture before and after the thermal shock cycles. The alumina-containing andalusite castable had superior thermal shock damage resistance for both parameters. The high-alumina castable had a higher resistance to slag corrosion. The results were linked to material selection for specific industry applications.

213639

POROUS REFRACTORY CERAMICS FOR HIGH-TEMPERATURE THERMAL INSULATION. PT.1. THE SCIENCE BEHIND ENERGY SAVINGSalomao R; Oliveira K S; Fernandes L; Tiba P; Prado U S - *Sao Paulo, University; Sao Paulo, State University; Sao Carlos, Federal University; Lining Representacao Consultoria e Projetos Ltda***Interceram.** 70, No.3, 2021, p.38-45

The use of thermal insulation to prevent undesirable heat exchange and save energy is a growing trend. However, there is little systemic and structured information combining technological and scientific aspects of its development in the literature. A compilation of updated information on the thermal energy consumption per area of human activity is provided, giving examples of industrial processes occurring in each temperature range.

213640
MgO-C REFRACTORIES: A DETAILED REVIEW OF THESE IRREPLACEABLE REFRACTORIES IN STEELMAKING
 Kundu R; Sarkar R - *Rourkela, National Institute of Technology*
Interceram. 70, No.3, 2021, p.46-55
 The raw materials, additives, general properties, oxidation and corrosion of MgO-C refractories, irreplaceable in steelmaking, are reviewed. Because it is a composite refractory, it benefits from the combined properties of magnesia and carbon. High-purity magnesia aggregates (fused and sintered), graphite as a carbon source, additives such as antioxidants (primarily metal powders) and organic binders (resins) are used to manufacture MgO-C refractories. The properties of the refractories may be tuned according to application demands by varying their carbon content. MgO-C refractories are mainly used in steelmaking converters for the entire lining of basic oxygen furnaces, electric arc furnaces, steel ladles and secondary steelmaking.

213641
RELATIONSHIP BETWEEN PHASE COMPOSITION, THERMAL CONDUCTIVITY AND CORROSION RESISTANCE OF FUSED-CAST AZS REFRACTORIES
 Wang R; Gu H; Bai C; Yuan L; Huang A - *Wuhan, University of Science & Technology; Ruitai Materials Technology Co.Ltd.*
Interceram. 70, No.3, 2021, p.56-61
 Fused-cast refractories are important lining materials for glass furnaces. Their high corrosion resistance and low thermal conductivity, determined by the phase structure, are a main focus of their development. The relationship between the phase composition, thermal conductivity and corrosion resistance to glass liquid was studied using numerical simulation combined with a melting test. A phase structure and thermal conductivity corrosion resistant model with a eutectic content of 40-70% and an average equivalent eutectic grain size of 5-25 micron was established. It showed that the uniformity of thermal insulation and corrosion resistance of the material can be achieved by increasing the eutectic content and refining the eutectic grains. Using a computer-controlled melting process, low thermal conductivity and high corrosion resistant fused-cast AZS refractories can be produced, which can improve energy saving as glass furnace insulation and give a safe operation time.

COMPOSITES

General

See also Abstract(s): 213386 213405 213407 213436 213441 213456 213482 213544 213600

213642
MICROSTRUCTURE, HARDNESS AND FLEXURAL STRENGTH OF Ni/Al₂O₃ FGMs BY PRESSURE-LESS SINTERING WITH DIFFERENT COOLING RATES
 Bhaskararao K A; Janardhana G R - *Kakinada, Jawaharlal Nehru Technological University*
Bol.Soc.Esp.Ceram.Vidrio. 60, No.4, 2021, p.255-265
 Four-layered functionally graded materials (FGMs) were fabricated with Ni and Al₂O₃ as the principal materials. Four FGMs of different compositional layers (PLS10, PLS15, PLS20 and PLS25) were prepared by powder metallurgy. Ni/Al₂O₃ FGMs were developed by weighing the elementary powders, blending, stacking and cold compacting followed by pressureless sintering at 1200 C for 90 min with different cooling rates (5 C/min, 15 C/min, 20 C/min and 25 C/min). SEM showed the varying microstructure with different percentages of raw materials and cooling rate. EDS was used to determine the composition of the sintered FGM samples. Green and sintered densities, hardness and flexural strength were also evaluated. The results indicated that the densities decreased steadily with increased alumina composition and the hardness increased with alumina content. The PLS15 layer composition with 20 C/min sinter-cooling rate gave the best results for the FGMs.

213643
MILLING EFFECT OF A NEW HORIZONTAL ATTRITION MILLING IN THE PRODUCTION OF (Ti_{0.7}W_{0.3})C-Ni CERMET
 Ha D; Kim J-H; Seo M - *Seoul, National University; Samsung Engineering Co.Ltd.*
Ceram.Int. 47, No.6, 2021, p.7398-7406
 A new high-energy ball milling technique, horizontal attrition ball milling (HAM), is reported. The milling effect of HAM was superior to that of the conventional planetary and attrition milling techniques in the size reduction of particles (fragmentation) and the impact of milling (mechanical strength). Ultrafine (Ti_{0.7}W_{0.3})C-Ni cermet powders could be obtained by HAM since (Ti_{0.7}W_{0.3})C particle growth was effectively inhibited during carbothermal reduction. Sintered (Ti_{0.7}W_{0.3})C-Ni cermets, prepared by HAM, had excellent mechanical properties including hardness and fracture toughness. Results showed that newly designed HAM is a practical method for the synthesis of ultrafine ceramics and composite powders appropriate for the production of structural ceramics and composites with excellent mechanical properties.

213644
IMPROVING ELECTRIC INSULATION CHARACTERISTICS OF PVA/V₂C MXENE COMPOSITE HIGH-DIELECTRIC-CONSTANT FILMS BY BLENDING CELLULOSE
 Deng Q; Zhou J; Qin B; Bo M; Feng Y - *Chongqing, Yangtze Normal University*
J.Aust.Ceram.Soc. 57, No.3, 2021, p.819-824
 Polymer/conductor composite dielectrics have high permittivity, high conductivity and high dielectric loss, limiting a large increase of energy storage density in them. The relationship between high permittivity and low conductivity is coordinated to achieve high energy density. Ternary PVA/V₂C MXene/cellulose composites with high permittivity and low dielectric loss were prepared. Due to the good water solubility of PVA and the high water dispersibility of V₂C, PVA and V₂C have good interfacial compatibility; that reduces the interface electric leakage conductance, which is conducive to a lower conductivity and dielectric loss. The high interfacial polarisation between PVA and V₂C improved the dielectric response, which improved the permittivity. Compared with binary PVA/V₂C composites, ternary PVA/V₂C/cellulose materials rely on the high insulation of the cellulose filler to increase the insulation, thereby reducing the conductivity and dielectric loss. Good synergy of conductive V₂C and insulating cellulose fillers is important for obtaining a balance between high dielectric response and high insulation trait in ternary composites. The ternary composite containing 6

w% V2C has a high permittivity of about 40, low dielectric loss of about 0.25 at 100 Hz, and a high breakdown strength of about 40 MV/m. Thus, composites promising for dielectric energy storage can be easily prepared.

Ceramic matrix composites

See also Abstract(s): 213387 213396 213398 213408 213424 213440 213533 213537 213543 213567 213583 213586 213587 213598 213615 213684

213645
ONE-STEP IN-SITU LASER IRRADIATION FOR UNIQUE FLOCCULENT CARBON NETWORK-TWINED C/Si/SiC COMPOSITE STRUCTURE
 Luo T; Cao B; Li W; Sun J; Yang C; Yang S - *Jinan, Qilu University of Technology; Jinan, University; Qufu, Normal University*
Ceram.Int. 47, No.5, 2021, p.7101-7105
 Flocculent carbon network-twinning C/Si/SiC nanocomposites were synthesised by a novel in-situ laser irradiation method using SiC targets as both a template and the source of C and Si. The fragmentation, decomposition and reshaping processes of bulk SiC to flocculent C/Si/SiC were simultaneously accomplished in one step, which could provide a more stable structure between an epitaxial Si layer and C layer. Due to the distinct laser-induced loose microstructure and flocculated carbon nanosheets, such nanocomposites have a high specific surface area and hierarchically porous structure.

213646
RELATIVE IMPORTANCE OF Al(V) AND REINFORCEMENT TO THE FLEXURAL STRENGTH OF GEOPOLYMER COMPOSITES
 Samuel D M; Sutrisno A; Kriven W M - *Illinois, University at Urbana Champaign*
J.Am.Ceram.Soc. 104, No.7, 2021, p.3452-3460
 Geopolymer bars (1 cm x 1 cm x 10 cm) were prepared from sodium silicate and six commercial metakaolins, both unreinforced and reinforced with 20 wt% of 55-micron wollastonite (CaO.SiO₂) needles, to evaluate the relative contributions of five-coordinated aluminium in the metakaolin and the presence of a reinforcing phase to the flexural strength of geopolymers. Two metakaolins, with about 20 at% and lower of five-coordinated aluminium content, did not react sufficiently with the processing method and could not be tested. The flexural strengths of the other four geopolymers were similar at about 11-14 MPa unreinforced and 22-29 MPa reinforced. The effect of reinforcement on flexural strength was more significant than the choice of metakaolin as long as the metakaolin was reactive. The geopolymerisation reaction depended on the amount of five-coordinated aluminium present in the metakaolin and was the primary difference between the samples that reacted and those that did not.

213647
EXPERIMENTAL APPROACH TO PROBE INTO MECHANISMS OF HIGH-TEMPERATURE EROSION OF NbB₂-ZrO₂
 Maity T; Gopinath N K; Biswas K; Basu B - *Indian Institute of Technology; Indian Institute of Science*
J.Am.Ceram.Soc. 104, No.7, 2021, p.3518-3530
 Boride-based ultrahigh temperature ceramics have applications in high-speed supersonic aircraft. The mechanisms of high-temperature erosive wear of spark plasma sintered NbB₂-ZrO₂ composites were therefore studied. The solid particle erosion experiments were performed at different temperatures starting from room temperature (25 C) to 800 C using Al₂O₃ particles (50 micron). The air-erodent particle mixture was impinged towards the target surface at normal impact with a velocity of 50 m/s. Microstructural analysis using HRTEM showed the generation and accumulation of a large number of dislocations within NbB₂ and ZrO₂ grains of the eroded surfaces. Such observations indicated the activation of dislocation plasticity during erosion at 800 C. XRD-based analyses gave residual stress-based interpretations for the enhancement of erosion resistance at high temperatures. In contrast, substantial material loss via brittle failure, involving the generation and intersection of the lateral/radial cracks, was recorded after room temperature erosion. In case of erosion at 400 and 800 C, the residual stress relaxed as a result of high-temperature exposure prior to erosion. The accumulation of dislocations near the crater region and shot peening phenomena played a dominant role in decreasing the erosion rate with increasing erosion test temperature.

213648
FLASH SINTERING PRODUCES EUTECTIC MICROSTRUCTURES IN Al₂O₃-LaPO₄ VERSUS CONVENTIONAL MICROSTRUCTURES IN 8YSZ-LaPO₄
 Yang Y; Mumm D R; Mecartney M L - *California, University at Irvine*
J.Am.Ceram.Soc. 104, No.8, 2021, p.3895-3909
 Although monazite (LaPO₄) does not flash sinter even at high fields of 1130 V/cm and temperatures of 1450 C, composite systems of 8YSZ-LaPO₄ and Al₂O₃-LaPO₄ more readily flash sinter. 8YSZ added to LaPO₄ greatly lowered the furnace temperature for flash to 1100 C using a field of only 250 V/cm. Alpha-Al₂O₃ alone also did not flash sinter at 1450 C, even with high fields of 1130 V/cm, but composites of Al₂O₃-LaPO₄ powders flash sintered at 900-1080 V/cm at 1450 C. Al₂O₃-LaPO₄ composites with compositions from 25 to 75 vol% Al₂O₃ were flash sintered with current limits from 2 to 25 mA/mm². Microstructures were evaluated by SEM and TEM. A eutectic microstructure formed in all flash sintered Al₂O₃-LaPO₄ composites. With higher power (higher current limits), eutectic structures with regular lamellar regions coexisted in the channelled region (where both the current and the temperature were the highest) with large hexagonal-shaped alpha-Al₂O₃ grains (up to 75 micron) and large irregular LaPO₄ grains. With lower power (lower current limits), an irregular eutectic microstructure was dominant, and there was minimal abnormal grain growth. These results indicate that Al₂O₃-LaPO₄ is a eutectic-forming system and the eutectic temperature was reached locally during flash sintering in regions. These eutectic microstructures with lamellar dimensions on the scale of 100 nm offer potential for improved mechanical properties.

213649
PREPARATION OF BaTiO₃@NiO CORE-SHELL NANOPARTICLES WITH ANTIFERROELECTRIC-LIKE CHARACTERISTIC AND HIGH ENERGY STORAGE CAPABILITY
 Wang H; Cao M; Huang R; Tao C; Pan W; Hao H; Yao Z; Liu H - *Wuhan, University of Technology*
J.Eur.Ceram.Soc. 41, No.7, 2021, p.4129-4137
 Core-shell structured BaTiO₃@NiO nanoparticles, with excellent energy storage performance, were prepared by coating NiO shell layers onto BaTiO₃ nanoparticles using the sol-precipitation method. The structures, dielectric and energy storage properties of the BaTiO₃@NiO ceramics were

investigated. Results showed that, high-density nanostructures with fine-crystalline and uniform distribution were observed. An excellent energy storage density of 2.72 J/cm³ with low remanent polarisation was achieved at 230 kV/cm in the BaTiO₃@1%NiO ceramics, which also possessed fast discharge features ($\tau = 0.9 < 2$ micro-s), superior thermal stability (20-80 C) and prominent cyclic stability (up to 1×10^5 times). Moreover, the BaTiO₃@NiO ceramics exhibited an antiferroelectric-like features due to the synergistic effect of NiO and BaTiO₃. The antiferroelectric-like dielectric material could be a prospective material for energy storage applications.

Fibre and whisker reinforced ceramic matrix composites

See also Abstract(s): 213393 213418 213419 213607 213693

213650

MULTISCALE MODELLING FOR PREDICTING THE THERMAL EXPANSION BEHAVIOURS OF 3D C/SiC COMPOSITES CONSIDERING POROSITY AND FIBRE VOLUME FRACTION

Sun Z; Shan Z; Shao T; Li J; Wu X - *Beijing, Tsinghua University; Beijing, Key Lab. of Advanced Manufacturing Technology; Nanjing, University of Aeronautics & Astronautics*

Ceram.Int. 47, No. 6, 2021, p. 7925-7936

Representative volume element (RVE) models of microscale, void/matrix and mesoscale developed in this work were used to investigate the coefficients of thermal expansion (CTEs) of 3-D C/SiC composites. A coupled temp-displacement steady-state analysis step was created for assessing the thermal expansion behaviour of the composites by applying periodic displacement and temperature boundary conditions. Three RVE models of cuboid, hexagonal and fibre random distribution were respectively established to comparatively study the effect of fibre package pattern on the CTEs at microscale. Similarly, the effects of different void size, locations and shapes on the CTEs of the matrix were comparatively analysed by the void/matrix models. The predicted results at the mesoscale corresponded closely to the experimental results. The effect of the porosities on the CTEs was studied by the void/matrix RVE models and the voids were effective in lowering the CTE of the 3D C/SiC composites. Furthermore, the effect of fibre volume fractions on the CTE were also taken into consideration. Equal in-plane and out-of-plane CTEs were realised by selecting appropriate fibre volume fractions for the different directions. The multiscale models developed could be used to predict the thermal expansion behaviour of other complex structured composites.

213651

DRY SLIDING TRIBOLOGICAL BEHAVIOUR OF C/SiC UNDER DIFFERENT LOAD AND SPEED

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Ceram.Int. 47, No. 6, 2021, p. 8627-8633

The tribological characteristics of C/SiC and zirconia (ZrO₂) were investigated. The effects of the load (10-100 N) and sliding speed (0.2-0.8 m/s) on the tribological performance of C/SiCs were studied under dry friction and ambient temperature using a pin-on-disc tribometer. The wear surfaces of the pin and disc were examined before and after the experiments to investigate the wear mechanism of the friction pair under different working conditions. Results showed that the contact pressure and sliding speed affected the coefficient of friction (COF) and wear rate (WR) of the pin and disc and the parameters cited decreases as the load and speed increased. A lubricating film, formed by the wear debris, was a key factor affecting the tribological performance. For high loads and speeds, the wear debris size decreased, which contributed to the formation of the protection film and a smooth wear surface during the dry friction process.

213652

DAMAGE MONITORING OF 2D SiC-SiC COMPOSITES UNDER MONOTONIC AND CYCLIC LOADING/UNLOADING USING ACOUSTIC EMISSION AND NATURAL FREQUENCY

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Ceramics-Silikaty. 65, No. 2, 2021, p. 125-131

Tensile damage and fracture in 2D SiC/SiC composites were investigated by damage monitoring of acoustic emission (AE) and natural frequency. Nonlinear damage and fracture are mainly due to matrix cracking, interface debonding and fibre fracture. The monotonic tensile stress-strain curves are divided into three stages based on the analysis of AE count, amplitude and energy. Under cyclic loading/unloading, hysteresis loops appear due to internal frictional slip between the fibre and the matrix, and the natural frequency and composite modulus were obtained for different peak stress values. A micromechanical tensile and cyclic loading/unloading constitutive model was adopted to predict the tensile curves. Micro damage parameters of interface debonding ratio and the fraction of broken fibres were used to characterise tensile damage and fracture. The relationships between natural frequency, composite modulus, interface debonding and fibre fracture were established. Under tensile loading, the AE signal is large and concentrated mainly in the stress range of 50-100 MPa and 150-180 MPa, corresponding to matrix cracking and fibre fracture, respectively. When the degradation rate of the natural frequency approaches 0.01, matrix cracking and interface debonding occur but fibre failure does not occur; when the degradation rate approaches 0.04, the composite modulus decreases by about 47%, the interface debonding ratio approaches 0.8, and the broken fibre fraction is approximately 2.2%.

213653

MELT INFILTRATION STUDIES OF 2D TYRANNO SA3 CERAMIC MATRIX COMPOSITE PREFORMS WITH CrSi₂ INTERMETALLIC ALLOY

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J. Am. Ceram. Soc. 104, No. 7, 2021, p. 2966-2980

2D Tyranno SA3 SiC fibre woven preforms were infiltrated with molten CrSi₂ between 1768 and 1896 K under a vacuum of 1.3×10^{-4} Pa. The infiltration times varied between 1800 and 7200 s, which did not affect the volume fraction of voids filled with the metal. Optical microscopy, SEM, backscattered electron imaging, EDS and Raman spectroscopy were used to characterise the infiltrated preforms. A plot of the volume fraction of open voids infiltrated against the absolute melt infiltration temperature showed a sharp peak at 1773 K with almost complete infiltration of the voids at this temperature. The extent of silicide infiltration of the preforms dropped steeply above 1773 K with an increasing melt infiltration temperature, irrespective of the amount of infiltration time. Above 1805 K, the volume fraction of voids infiltrated with the melt was nearly 0%. It was shown that CrSi₂ did not react with the SiC fibres. The possibility of a resisting force due to contact angle hysteresis influencing the diminishing amount of voids filled with increasing temperature > 1773 K was examined. The resistance force was estimated to be extremely small to be of consequence.

Thermodynamic analyses showed that it was also possible that the CrSi₂ decomposed into Cr(g) and Si(g) in the vacuum melt infiltration furnace. An empirical equation was proposed to calculate the amount of remaining charge left to infiltrate the preforms at the infiltration temperature. It was shown that an initial charge of 10 g would rationalise these observations. While the decomposition of the CrSi₂ appeared to mostly explain these results, some discrepancies were seen, which were inconsistent with the decomposition model.

213654

EFFECTS OF PHENOLIC RESIN CONTENTS ON MICROSTRUCTURES AND PROPERTIES OF C/SiC-DIAMOND COMPOSITESLi J; Liu Y; Chen C; Pan Y; Wang J; Wang N - *Xian, Northwestern Polytechnical University; Changsha, National Univ. of Defence Technology***J. Am. Ceram. Soc.** 104, No. 7, 2021, p. 3424-3442

C/SiC-diamond composites were obtained by CVI and reactive melt infiltration (RMI) and the effects of the phenolic resin content on their microstructure and properties were studied. The results suggested that the phenolic resin content had a significant effect on the pore structure of the composites before RMI, as well as phase composition and density of the matrix after RMI. The mechanical properties of composites correlated with the threshold effect of phenolic resin. The sample prepared with a high phenolic resin content had significantly reduced mechanical properties. However, adjustment of the phenolic resin content yielded samples with maximum room temperature thermal conductivity reaching 14.75 W/m.K. The theoretical thermal conductivity of the composites calculated by the Hasselman-Johnson theoretical model was estimated to be 24.52 W/m.K. The increase in phenolic resin content led to unreacted diamond-C regions and the formation of substantial porosity which reduced the thermal conductivity of the resulting C/SiC-diamond composites.

213655

AMORPHOUS SELF-HEALED, CHOPPED BASALT FIBRE-REINFORCED, GEOPOLYMER COMPOSITESKeane P F; Foltz J S; Chadha V; Marsh C P; Kriven W M - *Illinois, University at Urbana Champaign; US, Army Corps of Engineers***J. Am. Ceram. Soc.** 104, No. 7, 2021, p. 3443-3451

Geopolymer composites containing refractory, chopped basalt fibres and low-melting glass were fabricated and heat-treated at higher temperatures. Potassium-based geopolymers of stoichiometric composition K₂O·Al₂O₃·4SiO₂·11H₂O were produced by high shear mixing from fumed silica, deionised water, potassium hydroxide, (i.e. water glass) and metakaolin. When low-melting glass (T_m about 815 C) was added, the flexural strengths of the composites increased to about 6 MPa after heat treatment above 900 C to 1100 C. Weibull statistical analysis showed how the amorphous self-healing effect of the glass frit significantly improved the flexural strength of the geopolymer and ceramic composites after high-temperature exposure. At temperatures up to 900 C, the geopolymer-basalt composite remained amorphous and the low-melting glass frit flowed into the dehydration cracks in the geopolymer matrix. This type of composite could be described as amorphous self-healed geopolymer (ASH-G). At about 1000 C, the geopolymer converted to primarily a crystalline leucite ceramic, but the basalt fibre remained intact, and the melted glass frit flowed and sealed the cracks developed at that temperature. This type of composite could then be described as amorphous self-healed ceramic (ASH-C). A temperature of 1150 C was found to be optimum as at 1200 C the basalt fibres melted and the strength of the reinforcement was lost. The amorphous self-healing effect of the glass frit significantly improved the room temperature flexural strength of the heat-treated geopolymer-based composites.

213656

ABRASIVE WEAR PERFORMANCE AND WEAR MAP OF ZrB₂-MoSi₂-SiC_w COMPOSITESPaul T R; Mondal M K; Mallik M - *Durgapur, National Institute of Technology***J. Eur. Ceram. Soc.** 41, No. 6, 2021, p. 3227-3251

The effects of SiC_w additions on the abrasive wear performance of ZrB₂-MoSi₂ composites were examined. The coefficient of friction, specific wear rate and surface roughness decreases with the addition of SiC_w. The formation of the self-lubricating tribo-oxide layer caused by a tribochemical reaction during frictional heating enhanced the wear resistance of the composites. The tribological performance of the composites was affected by parametric variation of different experimental conditions which were primarily the sliding speed and applied load. Operational wear regime maps revealed that the workable damage tolerance limits for the applied load were 20-25 N for the ZMSw-5 composite, whereas that for ZMSw-20 composite was about 40 N. The wear mechanisms varied from delamination, cracking and grain pullout at medium applied loads to grain boundary fracture at higher applied loads.

213657

EFFECT OF 1-D AND 2-D CARBON-BASED NANO-REINFORCEMENTS ON THE DRY SLIDING-WEAR BEHAVIOUR OF 3Y-TZP CERAMICSRodriguez-Rojas F; Cano-Crespo R; Borrero-Lopez O; Dominguez-Rodriguez A; Ortiz A L - *Badajoz, Universidad de Extremadura; Seville, University***J. Eur. Ceram. Soc.** 41, No. 6, 2021, p. 3595-3602

The dry sliding-wear behaviour of a zirconia doped with 3 mol% yttria (3Y-TZP) monolith and of two 3Y-TZP composites, the latter with the same ultrafine-grained microstructure as the former but reinforced with either 1-D carbon nanofibres (CNFs) or 2-D reduced graphene oxide (rGO) nanoplatelets, was evaluated using ball-on-disc tests at moderate load and compared. 3Y-TZP, 3Y-TZP/CNFs and 3Y-TZP/rGO underwent mild wear, in the three cases by abrasion with contributions from both plastic deformation and fracture (with varying severities depending on the sample). The wear resistance did not correlate with the hardness or toughness, but with whether or not self-lubricating tribofilms were formed on the contact surfaces. Specifically, once pulled-out, 2-D rGO nano-reinforcements imposed solid-state lubrication that reduced the coefficient of friction (CoF), which provided 3Y-TZP/rGO with superior wear resistance relative to both 3Y-TZP and 3Y-TZP/CNFs. The 1-D CNF nano-reinforcements did not form such tribofilms and had no effect on the CoF and wear resistance. The implications of the dimensionality of the carbon-based nano-reinforcements and the testing conditions for the microstructural design of ceramic composites for tribological applications are discussed.

213658

AMORPHOUS SELF-GLAZED, CHOPPED BASALT FIBRE REINFORCED, GEOPOLYMER-BASED COMPOSITESChadha V; Kriven W M - *Illinois, University at Urbana Champaign***Int. J. Appl. Ceram. Technol.** 18, No. 4, 2021, p. 1097-1105

Geopolymer composites reinforced with refractory, chopped basalt fibres and low melting glass were fabricated and heat-treated at high temperatures. The potassium-based geopolymer with the composition K₂O·Al₂O₃·4SiO₂·11H₂O was produced from water glass (fumed SiO₂,

deionised H₂O and KOH) and metakaolin. Addition of low melting glass (T_m about 815 C) increased the flexural strength of the composites to about 5 MPa after heat treatment between 1000 and 1200 C. Weibull statistical analysis showed that the amorphous self-healing and self-glazing effect of the glass frit significantly improved the flexural strength of the geopolymer and ceramic composites after exposure to 1 hour at high temperatures. At 950-1000 C, the K-based geopolymer converted to primarily a crystalline leucite ceramic, but the basalt fibre remained intact, and the melted glass frit flowed out of the surface cracks and sealed them. The optimum heat treatment temperature was found to be 1150 C since the basalt fibres melted at less than or equal to 1200 C and the strength of the reinforcement was significantly reduced. The amorphous self-healing and amorphous self-glazing effects of the glass frit significantly improved the room temperature flexural strength of the heat-treated geopolymer and ceramic composites.

213659
EFFECT OF SiC NANOWHISKER ADDITION ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF REGENERATED CEMENTED CARBIDES BY LOW-PRESSURE SINTERING

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Int.J.Appl.Ceram.Technol. 18,No.4,2021,p.1222-1234

The hardness and toughness of recycled cemented carbides are, in general, contradictory. Regenerated WC-8 wt%CO cemented carbides reinforced with SiC nanowhiskers were prepared by low-pressure sintering, and the effect of SiCw content on the microstructure and mechanical properties were investigated. The hardness, density, flexural strength and fracture toughness first increased then decreased with SiCw content, reaching 1575 HV, 14.6 g/cm³, 2204 MPa and 16.85 MPa.m exp(1/2), respectively, for a SiCw content of 0.5 wt%; this represented an increase of 14.4%, 0.7%, 12.2% and 17.3%, respectively, compared with recycled cemented carbide alone. The lowest friction coefficient and the best wear resistance was reached for 0.5 wt% SiCw addition. The Fracture in the recycled cemented carbide was both transgranular and intergranular as seen by SEM of fracture surfaces.

213660
PREPARATION AND MECHANICAL CHARACTERISTICS OF FINE-WOVEN CLOTH AND PUNCTURED FELT PREFORM Cf/C-SiC-ZrC COMPOSITE

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Xian, Academy of Aerospace Solid Propulsion Technology

Int.J.Appl.Ceram.Technol. 18,No.4,2021,p.1330-1341

A 3D architecture carbon fibre preform consisting of a specifically fine-woven cloth and punctured felt preform was used to manufacture a novel advanced Cf/C-SiC-ZrC composite. The matrix was produced by CVI combined with precursor infiltration and pyrolysis (PIP), and finalised by CVD of an SiC coating to provide a material with a density of 1.95 g/cm³. The organic precursors to SiC and ZrC had a weight ratio of 4:1 in a xylene solute. The mechanical properties of the composite (tensile, compression, bending, shear and Z-direction loadbearing) were determined and its failure behaviour was monitored by SEM. The results showed that the punctured filament tows are beneficial in allowing the composite to withstand a compressive load up to 308.6 MPa and a shear strength of 18.14 MPa, while the alternatively stacked weave piles and short fibre layers support the punctured bundles as well as holding the composite structure together, thereby enabling the ceramic matrix to withstand high pressures and severe ablation.

213661
TENSILE DAMAGE BEHAVIOUR OF SiCf/SiC-B4C AFTER OXIDATION IN WET ATMOSPHERE BASED ON ACOUSTIC EMISSION PATTERN RECOGNITION

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J.Am.Ceram.Soc. 104,No.8,2021,p.4131-4144

SiCf/SiC-B4C composites were prepared by slurry impregnation and reactive melt infiltration (RMI). Changes of damage behaviour of the composites before and after oxidation were studied using acoustic emission pattern recognition technology to investigate the damage evolution of the composites after oxidation in a wet atmosphere. The results showed that, since the damage behaviour of the composites changed after oxidation, the optimal number of clusters transformed from 3 to 5. As the oxidation time increased, the area of concentration of the damage events was gradually broadened towards lower stress direction due to the decrease of damage threshold of composites. Because the BN interface was gradually oxidised, the density of C3-cracks (axial yarns cracks) near the fracture and the proportion of Cluster-4 (the signals of transverse yarns cracks) increased, for which the increase of interfacial shear stress should be responsible. This research helps to understand the damage evolution of SiCf/SiC-B4C composites after oxidation and the method used can be as guide for studying similar materials.

213662
SYNERGISTIC MODEL OF STRESS AND OXIDATION INDUCED DAMAGE AND FAILURE IN SILICON CARBIDE-BASED CERAMIC MATRIX COMPOSITES

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J.Am.Ceram.Soc. 104,No.8,2021,p.4163-4182

A micromechanics-based modelling approach allowing for the simultaneous consideration of deformation, damage and oxidation associated with each constituent of SiC-based ceramic matrix composites (CMCs), including the fibre, boron nitride fibre coating and matrix, is described. Chemical kinetics models from the literature were combined with a progressive damage model. Rupture predictions of unnotched and notched stress-hold (creep) specimens were compared with experimental measurements from a SiC/SiC CMC to assess the efficacy of the modelling approach. Techniques of improving creep rupture life were examined using the model.

213663
MICROSTRUCTURAL CONTROL AND REINFORCEMENT MECHANISM OF THE M₀ AND B INSERTS IN THE SiCf/SiC COMPOSITES/Ni-BASED SUPERALLOY JOINT

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J.Eur.Ceram.Soc. 41,No.7,2021,p.4025-4036

Two novel composite fillers were designed for the dissimilar joining of SiCf/SiC composites and Ni-based superalloys. The AuCuTi/Mo/AuCuTi filler prevented atomic exchange using a 50 micron thick Mo interlayer and reduced thermal stress due to the multiple layers of the ductile (Au, Cu) solid solution and rigid Mo insert. The addition of B introduced an extra dimensionality to control the diffusion of interstitial atoms (Ni, Cr, and Si) and some TiB whiskers to add extra nucleation sites for the Ni-compounds, which is an easier and more accurate method to control the distribution of metallic atoms. The loadbearing strength of the joints increased by 17.7% when a AuCuTiB/Mo/AuCuTiB filler was used, since the distribution of intermetallic compounds was homogenised and the interfacial region changed from Ti₃SiC₂ to TiC + Ni-compounds.

213664

WET-LAID NONWOVEN BASED CERAMIC MATRIX COMPOSITES: AN INNOVATIVE AND HIGHLY ADAPTABLE SHORT FIBRE REINFORCEMENT FOR CERAMIC HYBRID AND GRADIENT MATERIALS

Kessel F; Klopsch L; Jehle V; Biller N-J; Friess M; Shi Y; Cepli D; Keck M; Jemmali R - *DLR; Reutlingen, University*

J.Eur.Ceram.Soc. 41, No.7, 2021, p.4048-4057

The wet-laid nonwoven process as a preform manufacturing technique for C/C-SiCs, which holds great promise for hybrid and gradient materials, is reported. The wet-laid nonwovens were compatible with liquid silicon infiltration (LSI) and stand alone samples of the wet-laid nonwoven ceramics and hybrid materials were produced. Due to variations in the manufacturing process the formation of short fibre C/C-SiC (69 % Carbon) and SiSiC (68 % SiC) was possible. Characterisation (porosity, phase composition, flexural strength, Young's modulus, coefficient of thermal expansion, specific heat capacity and thermal conductivity) showed similar material properties compared to well-established materials (SGL, Schunk). The wet-laid nonwoven process allowed the fabrication of an in-situ formed hybrid, which eliminated several high-temperature steps used in traditional hybrid manufacturing and reduced costs. The process was demonstrated on a real scale component (ceramic brake disc) with a final material pairing of C/C-SiC and SiSiC.

213665

THERMAL CYCLING BEHAVIOUR OF La₂Zr₂O₇/Yb₂Si₂O₇/SiC COATED PIP Cf/SiC COMPOSITES UNDER BURNER RIG TESTS

Chen P; Xiao P; Li Z; Li Y; Chen S; Duan J; Deng P - *Changsha, Central South University; Changsha, National Univ. of Defence Technology*

J.Eur.Ceram.Soc. 41, No.7, 2021, p.4058-4066

Ytterbium disilicate (Yb₂Si₂O₇) and lanthanum zirconate (La₂Zr₂O₇) powders were synthesised by the sol-gel method. To improve the oxidation resistance of Cf/SiC composites at high-temperature and in high-speed gas scour environment, a tri-layer La₂Zr₂O₇/Yb₂Si₂O₇/SiC EBC was prepared on the surface of Cf/SiC composites using chemical vapour deposition (CVD) and the sol-gel method combined with air spraying. Results showed that the tri-layer La₂Zr₂O₇/Yb₂Si₂O₇/SiC coating had better oxidation resistance than the bi-layer Yb₂Si₂O₇/SiC coating, and could prevent the oxidation of Cf/SiC samples in gas scour environment at 1773 K for 24 thermal cycles (360 min) with a weight loss of 7.06 x 10⁻³ g/cm². Microstructural characterisation revealed that crack deflection phenomena in the tri-layer coating largely avoided the formation of penetrating cracks and further improved the oxidation resistance of the coating. After the corrosion of the tri-layer coating in high-speed and high-temperature gas, a small amount of coating fell off and eventually a corrosion pit formed and coating fracture occurred.

Particle reinforced ceramic matrix composites

See also Abstract(s): 213388 213391 213394 213434 213448 213453 213517 213549 213657

213666

SCREEN PRINTED NOVEL ZnO/MWCNTs NANOCOMPOSITE THICK FILMS

Zargar R A; Arora M; Alshahrani T; Shkir M - *Rajouri, BGSB University; New Delhi, National Physical Laboratory; Riyadh, Princess Nora Bint Abdul Rahman University; Abha, King Khalid University*

Ceram.Int. 47, No.5, 2021, p.6084-6093

Zinc oxide-multiwalled carbon nanotube (ZnO/MWCNT) nanocomposite thick films were prepared via sol-gel screen printing followed by sintering at 550 C. The prepared films were characterised using XRD, SEM, UV-VIS, photoluminescence (PL), FTIR, Raman spectroscopy and the two-probe method. XRD revealed (101) orientation for both ZnO and ZnO/MWCNT thick films with a wurtzite structure. SEM confirmed the porous nature of the ZnO film while the ZnO/MWCNT films contained ZnO particles trapped in the porous MWCNT network and were crack-free. Reflectance spectroscopy showed a direct transition with decreasing band gap whereas the refractive index and absorption index showed appreciable variation within the band gap regime related to the change in the crystallite size. FTIR showed Zn-O stretching and the presence of a carboxylic CDC group. The PL spectra of ZnO and ZnO/MWCNT thick films showed a red shift and exhibited UV, blue and green emissions confirmed from CIE diagram. Raman spectra showed that Raman phonons were shifted and dominated due to the doping of MWCNT in the ZnO matrix. The electrical properties were investigated using a 2-probe method and showed a reduction in the resistance after MWCNT incorporation. The ZnO/MWCNT thick films, deposited by a screen printing process, had enhanced conductivity and colour emission for the fabrication of low cost optoelectronics devices (LEDs) as compared with pure ZnO films.

213667

SOL-GEL SYNTHESIS AND CHARACTERISATION OF SiC-B4C NANO POWDER

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Ceram.Int. 47, No.5, 2021, p.6376-6387

SiC-B₄C nanopowders were synthesised by the sol-gel process using a water-solvent-catalyst-dispersant system and their formation mechanism was evaluated using TEM, SEM, DTA/TGA, BET method, XRD, FTIR and DLS. The nanometric size of the precursor was controlled by dispersing agents and by controlling the pH inside the sol and DLS analysis revealed that the particles were below 10 nm. FTIR results indicated that (Si-O-B) bonds were formed in the dried gel powder due to hydrolysis and condensation reactions and DTA confirmed that the synthesis temperature was below 1400 C. XRD results implied the presence of cubic beta-SiC and rhombohedral B₄C phases, which were formed simultaneously in the SiC-B₄C nanopowder. BET analysis indicated a high surface area of about 171.42 m²/g and that the surfaces of the particles were mesoporous. SEM showed that SiC-B₄C particle size was in the 20-40 nm range with a homogenous morphology. Ultimately, TEM/EDS microstructural analysis showed that the B₄C and SiC particles were formed simultaneously and uniformly in the final product.

213668
FORMATION OF MULLITE AND MULLITE-CORUNDUM COMPOSITES FROM KAOLIN USING SPARK PLASMA SINTERING
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J.Aust.Ceram.Soc. 57, No.3, 2021, p.651-661
 Mullite was synthesised, alone and in a composite with alumina, from natural kaolin. Kaolin powder for the mullitisation process was placed in the apparatus for spark plasma sintering (SPS) as either raw kaolin or as calcined kaolin free of bound water. The thermal properties, microstructure and phase composition of the samples were studied. Calcination led to dehydration of kaolinite and amorphisation. The thermal properties of the dehydrated and as-received kaolinite-based material were very similar. Materials with perfect thermal insulation characteristics were obtained. Dehydrated powders were mixed with corundum powder and fired by SPS. The Vickers hardness and parameters based on instrumented indentation were enhanced in the composite.

213669
FABRICATION AND PHOTOCATALYTIC PROPERTIES OF CERAMIC ZnS NANOCOMPOSITES
 Yoon S-D; Yun J W; Yun Y-H - *Chonnam, National University*
J.Ceram.Process.Res. 21, No.4, 2020, p.479-487
 ZnS nanoparticle-reinforced ceramic nanocomposites were prepared by mechanical processing and one-step sintering of powder mixtures of fly ash, waste glass and ZnS. The ZnS nanoparticles were synthesised by the template-free hydrothermal route. The chemical durability and morphology of samples heat-treated at 800 C with/without acid treatment were evaluated. The photocatalytic activities were estimated using methyl orange (MO), methylene blue (MB), acetaldehyde (ATA) and 2,4-dichlorophenoxyacetic acid (2,4-D) as photodegradation targets. The crystallisation behaviour of the nanocomposites was studied using XRD, FE-SEM and EDS. The compressive and bending strength of the samples were also evaluated. The nanocomposites showed improved optimal photocatalytic reaction and properties regardless of acid treatment when the ZnS nanoparticle content increased from 7.0 to 25.0 wt%. Degrees of photocatalytic decomposition of MO, ATA, 2,4-D, and MB by acid treated nanocomposites containing 25 wt% ZnS were approximately 0.185, 0.121, 0.216 and 0.236, respectively, after UV irradiation for 180 min.

213670
BALLISTIC STUDY OF ALUMINA CERAMIC-STEEL COMPOSITE FOR STRUCTURAL APPLICATIONS
 Sanusi O M; Oyelaran O A; Badmus J A - *Blois, INSA Centre Val de Loire; Oye-Ekiti, Federal University*
J.Ceram.Process.Res. 21, No.4, 2020, p.501-507
 The protection of security/military personnel and their vehicles, aircraft and other security hardware continues to attract attention as the trade-off between weight and protection lingers. Ceramics are used in lightweight armour systems for their ballistic efficiency and light weight but are comparatively expensive. The effectiveness of sintered alumina, derived from corundum, as a laminate component of ceramic-steel composites for structural armour applications was studied. Both armour steel and medium carbon steel were separately impacted by a 7.62 x 51 mm armour-piercing (AP) projectile prior to lamination with the sintered ceramic in order to evaluate the ballistic resistance. The mechanical properties of the sintered Al₂O₃ ceramic compete favourably with commercial CoorsTEK sintered ceramics. Subsequently, varying target configurations of the ceramic and medium carbon steel composites were tested against the same AP projectile in accordance with NIJ Standard-0108.01. Depending on the configuration, the composite structure displays different failure modes. The study confirmed the protective capability of the sintered ceramic by the severity of its interaction with the projectile and delay of penetration when used as a laminate component plate.

213671
INFLUENCES OF Bi_{0.75}Y_{0.25}O_{1.5} ADDITION ON THE MICROSTRUCTURE AND IONIC CONDUCTIVITY OF Ce_{0.8}Y_{0.2}O_{1.9} CERAMICS
 Liang W; Meng B; Xiao Q; Ping X; Zheng Q; Li C; Xia Z - *Kunming, University of Science & Technology*
Int.J.Appl.Ceram.Technol. 18, No.4, 2021, p.1153-1163
 A second phase of Y₂O₃-stabilised Bi₂O₃ (Bi_{0.75}Y_{0.25}O_{1.5}, YSB) was incorporated into Y₂O₃-doped CeO₂ (Ce_{0.8}Y_{0.2}O_{1.9}, YDC) as a sintering aid, and YDC-xYSB (x = 0, 5, 10, 20, 30, 40 wt%) ceramic composites were prepared by sintering in air at 1100 C for 6h. The YDC-xYSB ceramics were composed of both YDC and YSB with the cubic fluorite structure; no other impurity phases were detected by XRD. The relative density first rose for less than or equal to 5 wt% before declining with YSB addition from 5 to 40 wt%. TEM showed that the YSB phase segregates at the grain boundaries of the YDC. The ionic conductivity of YDC-xYSB (greater than or equal to 5 wt%) was lower than that of YDC in the 200-500 C temperature range, while gradually exceeding that of YDC in the 500-750 C range. At 750 C, the conductivity of YDC-30%YSB (6.22 x 10⁻² S/cm) was 1.35 times higher than that of YDC (4.6 x 10⁻² S/cm). Thus, YSB addition improved the ionic conductivity of YDC in the 500-750 C range and decreased its sintering temperature.

213672
HYBRID COMPOSITE FOR STRUCTURAL APPLICATIONS MADE OF RUBBER WASTE TYRES AND CALCIUM PHOSPHATE CEMENT
 Huertas C F R; Vieira C M F; Colorado H A - *Medellin, Universidad de Antioquia; Rio de Janeiro, Universidade Estadual do Norte Fluminense*
Int.J.Appl.Ceram.Technol. 18, No.4, 2021, p.1342-1353
 A novel hybrid composite was prepared using eight different formulations consisting of powdered waste tyres, a polyurethane resin and a wollastonite-based phosphate cement. The phosphate cement was obtained by mixing wollastonite particles with an aqueous phosphoric acid solution, and the waste tyres were processed by mechanical grinding. A GOM Inspect Professional 3D scanner was used to characterise the surface of tiles prepared to be adhered to the phosphate cement. Pull-out tests were conducted on the rubber tile-cement system. SEM was used to examine the microstructure of the tile-cement interface and XRD was used to analyse the mineralogical phases in the composite. Two sample formulations gave good adhesion test results, with binding stress up to 0.274 MPa. Different adhesion mechanisms were identified at the interface, in which the roughness played a significant role in the adhesion of the materials.

213673

OXIDE-SCALE EVOLUTION AND DYNAMIC OXIDATION MECHANISM OF ZrB₂-SiC IN HIGH-ENTHALPY PLASMA WIND TUNNELLiu L; Yang L; Zhao C; Xiao X; Ye Z; Zhang J; Wang G; Wang Y - *Xian, Northwestern Polytechnical University; China Aerodynamics R&D Centre; Changsha, Central South University; Beijing, Institute of Technology***J.Eur.Ceram.Soc.** 41, No.7, 2021, p.3911-3921

A 1 MW plasma wind tunnel was used to study the dynamic oxidation mechanisms of ZrB₂-SiC at a heat flux ranging from about 0.5 to 5.0 MW/m² in reduced pressure. Results show a transition in the uppermost oxide layer from a SiO₂-rich glass to SiO₂ when the heat flux was increased from about 0.55 and 1.70 MW/m² (approximately 1100 C and 1340 C) to about 2.50 MW/m² (about 1550 C). A temperature fluctuation was observed at about 2.50 MW/m². At higher heat flux levels (at least 3.5 MW/m²), thermal instability occurred at about 1650 C. In addition, the ZrO₂ grain structure was temperature dependent, particularly at about 1.70 MW/m² where the ZrO₂ grain evolved from a tiny grain, to an equiaxed grain and then to a columnar grain. At a higher heat flux (at least 2.50 MW/m²), columnar grains were observed prior to the thermal instability. Finally, since the oxidation of ZrB₂-SiC was a diffusion controlled process, the oxidation rate was dependent not only on temperature but also on the grain structure of ZrO₂.

213674

FABRICATION OF RELIABLE ZTA COMPOSITE/Ti6Al4V ALLOY JOINTS VIA VACUUM BRAZING METHOD: MICROSTRUCTURAL EVOLUTION, MECHANICAL PROPERTIES AND RESIDUAL STRESS PREDICTIONWang Y; Liu M; Zhang H; Wen Z; Chang M; Feng G; Deng D - *Chongqing, University***J.Eur.Ceram.Soc.** 41, No.7, 2021, p.4273-4283

Reliable ZTA composite/Ti6Al4V alloy brazed joints were fabricated using a Ag-based filler metal. Microstructural investigation of the joints showed that a thin TiO layer formed at the ZrO₂/(Cu,Al)₃Ti₃O interface, whereas the Al₂O₃ matrix was in contact with (Cu,Al)₃Ti₃O. The thicknesses of the reaction layers and the diffusion zone increased as the brazing temperature increased, but the Ag(s,s) region became smaller. The joint fabricated at 825 C had a maximum shear strength of 54 plus or minus 4 MPa. A three-dimensional finite element model was established to study the residual stresses in the ZTA composite/Ti6Al4V alloy joints. Relatively large tensile residual stresses in the X direction were predicted in the (Cu,Al)₃Ti₃O layer and the Ti-Cu compound region. The simulated maximum tensile residual stress in the Z direction of about 190 MPa was on the vertical edges of ZTA near the ZTA/brazing seam interface.

Metal matrix composites

213675

EFFECT OF THERMAL STABILITY OF Mn+1AX_n PHASES ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF Mn+1AX_n/ZA27 COMPOSITESLi H-Y; Tian Y; Wan D; Bao Y; Zhou Y - *China Building Material Test & Certification Group Co.Ltd.; China Building Materials Academy; Beijing, Jiaotong University***Int.J.Appl.Ceram.Technol.** 18, No.4, 2021, p.1213-1221

The layered structure of ternary Mn+1AX_n phase ceramics endows them with good electrical and thermal conductivity, excellent abrasion resistance and perfect thermal shock resistance. Three kinds of Mn+1AX_n phase ceramics (Ti₃SiC₂, Ti₃AlC₂ and Ti₂SnC) were used to reinforce ZA27 (zinc aluminium) alloys. Mn+1AX_n/ZA27 composites were successfully fabricated using a two-step sintering technology, viz. pressureless sintering at 870 C for 1 h before hot pressing at 500 C for 1 h. The effects of thermal stability of the Mn+1AX_n phase on the microstructure, mechanical properties and frictional properties of the composites were investigated. The different degrees of reaction between the three Mn+1AX_n reinforcements and the ZA27 matrix are ascribed to differences in the chemical bond energies. The results showed that, at a sintering temperature of 870 C, Ti₂SnC completely reacted in the Ti₂SnC/ZA27 composite, Ti₃AlC₂ partially reacted in the ZA27 matrix, while no reaction occurred between Ti₃SiC₂ and ZA27. Hence, the thermal stability for the three Mn+1AX_n phases in the ZA27 matrix is in the order: Ti₃SiC₂ > Ti₃AlC₂ > Ti₂SnC. Ti₃AlC₂/ZA27 composites possessed the best mechanical properties and wear resistance due to interfacial reaction which improved the bonding between the matrix and the reinforcement.

213676

INTEGRATED CORROSION-RESISTANT SYSTEM FOR AZ31B Mg ALLOY VIA PLASMA ELECTROLYTIC OXIDATION (PEO) AND SOL- GEL PROCESSESMerino E; Duran A; Castro Y - *Madrid, Ceramic and Glass Institute (ICV-CSIC)***Int.J.Appl.Glass Sci.** 12, No.4, 2021, p.519-530

An integrated system combining anodising with the deposition of a hybrid SiO₂ sol-gel coating was created in this study to improve the corrosion resistance of AZ31B Mg alloy. The preparation and deposition of a SiO₂ sol obtained by hydrolysis and condensation of TEOS (tetraethoxysilane), GPTMS (3-Glycidyloxypropyl) trimethoxysilane), colloidal SiO₂ nanoparticles, and 1-Methylimidazole were investigated alongside the optimisation of anodising conditions using an alkaline electrolyte (NaOH) modified with different concentrations of sodium metasilicate pen (MI). The surface shape, coating thickness, and content of the coating were all examined. The thickness of the anodised coatings ranged between 1.1 and 1.7 μm, with a composition similar to magnesium oxide and a low silicon component. In a 3.5 percent NaCl solution, the corrosion performance was evaluated. After anodising the Mg alloys, the findings indicated an excellent corrosion resistance performance. However, when the porous PEO layer was encapsulated with a hybrid silica sol-gel film, the corrosion resistance was the greatest. The corrosion current density is reduced by three orders of magnitude between Mg alloy (1.54E-06 A/cm²) and multilayer system (2.80E-8 A/cm²). Furthermore, the polarisation resistance for 8AF+SG samples was extremely high (31546.8 cm²) when compared to Mg alloy (207.3 cm²).

Fibre and whisker reinforced metal matrix composites

213677

EFFECT OF CARBON NANOTUBES GROWN TEMPERATURE ON THE FRACTURE BEHAVIOUR OF CARBON FIBRE REINFORCED MAGNESIUM MATRIX COMPOSITES: INTERLAMINAR SHEAR STRENGTH AND TENSILE STRENGTHZhou J; Zhong K; Zhao C; Meng H; Qi L - *Xian, Northwestern Polytechnical University***Ceram.Int.** 47, No.5, 2021, p.6597-6607

Different amounts of carbon nanotubes (CNTs) were grafted onto the carbon fibre (CF) surface by adjusting the growth temperature using injection

chemical vapour deposition (ICVD). The prepared CF preform grafted with CNTs (CNTs-CF) were used to reinforce magnesium alloy using a squeeze casting process. Their microstructures were investigated using optical microscopy (OM) and SEM, and the interlaminar shear strength (ILSS) and tensile strength of the composites were determined by double-notch shear and tensile tests. Results indicated that a moderate ILSS was more conducive to improving the tensile properties of carbon fibre-reinforced magnesium matrix (Cf/Mg) composites. As compared with Cf/Mg, the tensile strength of the composite with CNTs increased by about 80%. For the Cf/Mg composites with grafted CNTs, the CNTs delayed crack propagation and increased energy consumption due to pullout and bridging mechanisms, which were the main reasons for the improved strength. Analysis of the shear fracture surface showed that the crack propagation path could be optimised by adjusting the amounts of grafted CNTs. The presence of CNTs affected the stress distribution and consequently crack initiation and crack propagation.

Particle reinforced metal matrix composites

See also Abstract(s): 213609

213678

CHARACTERISATION OF AA6061-ZrO₂-C FRICTION STIR WELDED COMPOSITE JOINTS

Pandiyarajan R; Prabakaran M P - *Tamil Nadu, KLN College of Engineering; Erode Sengunthar Engineering College*

J.Ceram.Process.Res. 21, No.6, 2020, p.690-698

Metal matrix composites were prepared by dispersing 6% zirconia and 2% graphite powder in molten aluminium alloy 6061, and plate specimens of 6 mm thickness were produced by casting. Butt welds between plates were produced by friction stir welding, using a tool with a flat shoulder and a cylindrical pin, and welding parameters of: welding speed 50 mm/min; tool rotary speed 600-1000 rpm; tool axial force 4-6 kN. This gave energy inputs of 24.9-62.3 kJ/mm. The welds and parent material were characterised by metallurgical studies, and by measurements of the tensile properties and microhardness. Columnar dendrites and an evenly mixed matrix of fine microstructure were observed in the dynamically recrystallised zone, and grain coarsening was observed in the thermomechanically affected zone. The highest tensile strength and hardness were achieved by welding at a speed of 50 mm/min, using a tool rotary speed of 800 rpm, subjected to an axial force of 5 kN.

213679

EFFECT OF HIGH-ENTROPY ALLOY BINDER CONTENT ON THE MICROSTRUCTURE, MECHANICAL PROPERTIES AND OXIDATION BEHAVIOUR OF Ti(C,N)-TiB₂ CERMETS

Fu P; Li Z; Wen X; Liu C; Chang F; Lin L; Dai P - *Fujian, University of Technology; Xiamen Tungsten Co.Ltd.; China National R&D Centre for Tungsten Technology; Fujian, Provincial Key Lab. Adv. Mater. Process. & Appl.; Xiamen Golden Egret Special Alloy Co.Ltd.*

J.Ceram.Soc.Jap. 129, No.8, 2021, p.504-515

AlCoCrFeNi high entropy alloy was prepared by mechanical alloying. It was used to prepare cermets containing 6-14% alloy, 64.5-70.5% Ti(C,N) and 21.5-23.5% TiB₂, by conventional powder metallurgy processing. The cermets were characterised by determinations of microstructure, phase composition, hardness, flexural strength and fracture toughness. The porosity decreased, and the density and hardness increased with increasing alloy content, whilst the grain size, flexural strength and fracture toughness initially increased and then decreased. Abnormal TiB₂ grain growth was observed in the cermet with an alloy content of 14%. Oxidation resistance was determined by measuring the mass change as a function of time up to 10 h, for specimens maintained at 800-1000 C. The mass gain increased with increasing test temperature. At 800 C, the surface oxidation product was mainly TiO₂, with small amounts of Al₂O₃, FeTiO₃ and Al₂TiO₅. The concentration of the minority oxidation products increased with increasing oxidation temperature. The cermets containing 10% alloy exhibited the highest oxidation resistance at 800 and 900 C, and that containing 14% alloy perform best at 1000 C. Cracks formed during oxidation were filled with Al₂O₃, the effect increasing with increasing alloy content.

213680

DEVELOPMENT OF PRESSURELESS SINTERED AND HOT-PRESSED CNT/ALUMINA COMPOSITES INCLUDING MECHANICAL CHARACTERISATION

Bechteler C; Rubling A; Girmscheid R; Kuhl H - *Nurnberg, Georg-Simon-Ohm-Fachhochschule; Rauschert Heinersdorf-Pressig GmbH*

Int.J.Ceram.Eng.Sci. 3, No.4, 2021, p.237-248

A method for producing carbon nanotube (CNT)/alumina composites using an aqueous solution and without significant pretreatment was devised. The pressureless sintering and hot-pressing of nanocomposites were thoroughly investigated and improved. The effect of various CNT contents, alumina powders, sintering temperature, and pressure on mechanical characteristics was studied. The ideal hot-pressing conditions are 1550 C, 15 min of dwell time, and an applied pressure of 80 MPa. Hot-pressing and pressureless sintering were used to create dense nanocomposites with up to 3 wt.% and 0.5 wt.% CNT content, respectively. Furthermore, a 20% increase in hardness was seen for CNT concentrations less than 1.0 wt.%, which is independent of applied force and alumina matrix. An indentation-based technique found significantly anisotropic fracture toughness as CNT content increased. The proposed technique allows for the manufacture of CNT/alumina composites with better mechanical characteristics with minimal effort, which may potentially be employed in industrial production.

PROCESSING AND TREATMENT (INCLUDING MACHINERY)

Preliminary processes: mining, winning, purification, milling, comminution

See also Abstract(s): 213304 213445 213470 213540 213590 213643 213669

213681

EFFECTS OF MILL SPEED AND AIR CLASSIFIER SPEED ON PERFORMANCE OF AN INDUSTRIAL BALL MILL

Ahmad F; Qayyum J A; Asghar U; Ali A; Masoom A - *Wah, University; Petronas, Technological University*

Ceram.Modern Technol. 3, No.1, 2021, p.28-37

Ball mills are commonly used in cement plants to grind clinker and gypsum. The effect of mill speed and air classifier speed on the milling of cement in a two-compartment ball mill was studied. The aspects investigated included Blaine specific surface area, SO₃ content, mill power, mill residue and mill residence time. It was found that an optimum combination of parameters can reduce power consumption while improving the quality of the

cement. A knowledge of the effects of parametric variations on end product quality could be helpful for quality control; furthermore, proper grinding of the clinker results, in the first place, in a fine Blaine SSA, reducing the need to recycle coarse grains.

213682

ESTIMATION OF CERAMIC MATERIAL PROPERTIES USING SPECIFIC RATE OF BREAKAGE DETERMINED FROM GRINDING TESTS OF NEPHELINE SYENITE

Haner S - *Afyon Kocatepe, University***J.Ceram.Process.Res.** 21, No.6, 2020, p.736-744

Nepheline syenite from a Turkish deposit was dry ground in a laboratory ball mill of 2650 cm³ capacity rotating at 75% of the critical speed. Batches were produced using alloy steel grinding media with diameters of 1/4 in (6.4 mm), 5/16 in (7.9 mm), 3/8 in (9.5 mm), 1/2 in (12.7 mm) and 3/4 in (19.1 mm). The particle size distributions were measured after 0.5-64 min. A size reduction rate, termed "the specific rate of breakage" was determined. The ground nepheline syenite was used to produce five casting slips containing 27% nepheline syenite, 19% silica sand and 54% of various clays. The slips were characterised by measurements of density, viscosity, thixotropy and particle size distribution. Cast specimens were fired at 1200 C and used to determine the firing shrinkage, fired strength, pore size distribution and water absorption. The shrinkage and strength increased, whilst the pore size distribution and water absorption decreased, as the particle size of the nepheline syenite decreased.

213683

NEW GENERATION MIXING TOOL

Krauskopf G - *Krauskopf Maschinentchnik GmbH & Co.KG***Interceram.** 70, No.3, 2021, p.20-22

During the manufacture of refractory products, highly abrasive raw materials cause heavy wear on the mixing blades and tool holders. Tunemixx, a mixing tool for a vertical intensive mixer, allows wear parts to be changed quickly, production shutdown costs to be kept to a minimum and the service life of components to be extended significantly. The wear protection on the new blades consists of a carbide shell with a bonded silicon carbide layer.

Sheet forming including tape casting

See also Abstract(s): 213439

Other shaping processes

See also Abstract(s): 213353 213382 213404 213434 213437 213442 213459 213461 213471 213472 213479 213480 213508 213514 213522 213537 213548 213591 213599 213626 213690

Sol-gel processing

See also Abstract(s): 213374 213400 213443 213447 213462 213502 213515 213523 213539 213581 213582 213603 213620 213665 213666 213667 213676

Coating and impregnating

See also Abstract(s): 213370 213389 213392 213402 213514 213530

Bonding

See also Abstract(s): 213352 213604 213610 213663 213674 213678

213684

MICROSTRUCTURE AND MECHANICAL PROPERTIES OF ZrB₂-SiC/Nb JOINTS BRAZED WITH CoFeNiCrCuTi_x HIGH-ENTROPY ALLOY FILLER

Yang Y; Wang G; He R; Shu D; Tan C; Cao W - *Anhui, Polytechnical University; Beijing, Institute of Technology; Harbin, Institute of Technology; Oulu, University***J.Am.Ceram.Soc.** 104, No.7, 2021, p.2992-3003

ZrB₂-SiC ceramics and Nb alloy were brazed at 1160 C for 60 min with CoFeNiCrCuTi_x high-entropy alloy filler. The effect of Ti content on the interfacial structure and mechanical properties of the ZrB₂-SiC/Nb joint was studied. It was found that the rich-Ti Laves phase was formed due to the addition of large atomic size Ti fill into the filler alloy or brazing joint, its content increasing with Ti content. The joint brazed by high-entropy alloys filler without Ti could be divided into a tooth-shaped Cr₂B reaction layer and a central area composed of a eutectic mixed structure of FCC phase and rich-Nb lamellar Laves phase. Ti and Nb were mutual solid solution elements. The increase of Ti content in the joint caused the FCC phase and the rich-Nb lamellar Laves phase to transform into a big bulk Ti-rich Laves phase and the quadrilateral (Ti, Nb)₃B phase. The tooth-shaped Cr₂B disappeared. The residual stress generated in the joint during brazing tended to cause defects such as holes and microcracks in the bulk Ti-rich brittle Laves phase. The normal temperature performance of the joint decreased from 216 MPa to 52 MPa with the addition of Ti. However, with increasing Ti content, the high-temperature mechanical properties of the joint first decreased, then increased, mainly due to the formation of rich-Ti Laves phase and quadrilateral (Ti, Nb)₃B with excellent high-temperature mechanical properties. When brazing with CoFeNiCrCuTi_{1.5} filler, the high-temperature performance of the joint reached 92% of its room-temperature performance.

Machining, finishing and surface treatment

See also Abstract(s): 213333 213377 213440 213444 213492 213524 213597

Repairing

See also Abstract(s): 213628

Control equipment and computer-aided design

213685

BUILDING INFORMATION MODELLING - THE FUTURE FOR GLASS PLANT DESIGN

Schippan D; Steinberger F - *cm.project.ing GmbH*

Glass Int. 44, No.6, 2021, p.20-22

Building Information Modelling (BIM) is a planning method used in building construction to centrally manage all building characteristics and installations from the outset. New intelligence can be integrated to a virtual model and it is a dynamic process. BIM has been implemented for the design of glass plants. Time and cost can be added to the simulations to provide cost control and reliable scheduling. Errors can be analysed quickly and productivity and quality improved.

213686

HOLISTIC SIMULATION APPROACH TO BUILDING PERFORMANCE

Schippan D; Barnstorf D - *cm.project.ing GmbH*

Glass Int. 44, No.6, 2021, p.24-26

The use of computational fluid dynamics (CFD) to analyse the performance and efficiency of glass factories and in the design of new glass plants is discussed. The plant can be analysed in its entirety or in specific sub-areas. Optimisation of the working conditions, such as ventilation, pollutant dispersion and noise, is carried out as well as optimisation of production. Problems can also be identified at an early stage.

Pressing

See also Abstract(s): 213467 213496 213523

Slip casting

See also Abstract(s): 213496 213682

Drying

See also Abstract(s): 213446 213635

Heat treatment: firing, sintering, furnaces and kilns

213687

RAPID SINTERING OF 3 MOL% Y₂O₃-DOPED ZrO₂ BY A COMBINED RAPID FURNACE HEATING AND SHRINKAGE-CONTROLLED FLASH SINTERING PROTOCOL

Ishino Y; Taguchi K; Kodaira A; Tokunaga T; Yamamoto T - *Nagoya, University*

J.Ceram.Soc.Jap. 129, No.8, 2021, p.551-554

Specimens (3.5 x 3.5 x 15 mm) of zirconia doped with 3 mol% yttria were prepared by uniaxial pressing followed by cold isostatic pressing at 100 MPa. Platinum electrodes were applied and the specimens subjected to furnace heating at a rate of 50 C/min, with application of an AC current at a frequency of 1.0 kHz under an applied field of 100 V/cm. When the current reached 50 mA, the furnace temperature was maintained and the power supply switched to current control. The shrinkage was then maintained at a constant rate of 200 micron/min by progressively increasing the current, to a maximum of 800 mA. The current was maintained at 800 mA for 5 min, and the furnace and power supply switched off. The sintered specimens were characterised by microstructural studies, including grain size measurement, and by measurements of density, Vickers hardness, and fracture toughness. Polycrystalline YSZ of high density and uniform grain size was obtained, requiring a total sintering time of about 40 min. The mechanical properties were similar to those of polycrystalline material obtained by conventional sintering.

213688

EXTENDED LIFE AND TIMES OF AN OXY-FUEL FURNACE

Naveken B - *TECO*

Glass Int. 44, No.6, 2021, p.15-16, 18

The collaboration between TECO and a customer producing glass tubing using a TECO all-electric melter to build an oxy-fuel furnace to replace the melter is discussed. The furnace has been successfully operating for 17 years and its use and modifications to overcome problems over that time are described.

213689

FLASH SINTERING FEASIBILITY STUDY AND LOCALISED DENSIFICATION IN BORON CARBIDE

Rosenberger A; Brennan R E; Fry A L - *Bennett Aerospace; US, Army Research Lab.; Pennsylvania, State University*

J.Am.Ceram.Soc. 104, No.8, 2021, p.3823-3827

The feasibility of flash sintering boron carbide (B₄C) was studied using a DC electric field across different electrodes, field strengths and thermal profiles. The flash behaviour was observed at furnace temperatures as low as 386 C with field strengths of 68-278 V/cm, but only a small channel

of the specimen was densified due to hot spot effects. Application of a 2.2 V/cm.s voltage ramp at a constant temperature of 550 C caused uniform heating, but at temperatures too low for sintering. It is theorised that scalable densification of B4C at low furnace temperatures with flash sintering is possible by applying a higher current density through power supply or specimen modifications.

213690

FLASH SINTERING OF ADDITIVELY MANUFACTURED 3YSZ GEARSYang B; Cho J; Phuah X L; Wang H; Zhang X - *Purdue University***J.Am.Ceram.Soc.** 104, No.8, 2021, p.3828-3832

Complex gear-shaped 3 mol% yttria-stabilised zirconia was fabricated by a combination of additive manufacturing and flash sintering. The gear was printed by direct ink printing. Flash sintering was carried out at an electric field of 150 V/cm and the specimens were rapidly densified within a few seconds at a furnace temperature of 1200 C. The flash-sintered gear had a relative density of 95% and showed no obvious warping or cracking after the rapid densification. Microstructural analysis and hardness measurement showed grain size gradients and hardness variation along both thickness and lateral directions of the flash-sintered gear. This demonstrates a possible route to produce dense complex-shaped parts using flash sintering.

213691

FLASH SINTERING OF CATIONIC CONDUCTIVE CERAMICS: A WAY TO BUILD MULTILAYER SYSTEMSLachal M; El Khal H; Bouvard D; Chaix J-M; Bouchet R; Steil M C - *Grenoble Alpes, University***J.Am.Ceram.Soc.** 104, No.8, 2021, p.3845-3854

Flash sintering, which enables ceramics to be densified at low furnace temperatures in a few seconds, requires a reversible electrochemical reaction to enable the current flow through the electrode/material interface. For Li⁺ pure ionic conductors such as Li_{1.3}Al_{0.3}Ti_{1.7}(PO₄)₃ (LATP), the best material to ensure this fast charge transfer reaction is a mixed Li⁺/e⁻ conductor, such as LiCoO₂ (LCO). The feasibility of flash sintering on LCO between two Pt electrodes and on multilayer systems using LCO or LATP + LCO composite as electrodes was demonstrated. It was shown that a composite electrode both allowed the flash event to occur and prevented the delamination possibly seen with pure LCO by lowering interfacial stresses.

213692

IN SITU DEFECT QUANTIFICATION AND PHASE IDENTIFICATION DURING FLASH SINTERING USING RAMAN SPECTROSCOPYMurray S E; Lv G; Sulekar S S; Cahill D G; Shoemaker D P - *Illinois, University at Urbana Champaign***J.Am.Ceram.Soc.** 104, No.8, 2021, p.3873-3882

Flash sintering is used to rapidly densify or induce phase transformation in materials by applying an electric field. Understanding rapid transformations is challenging in a laboratory setting, where phase and defect populations may change with sub-second time scales. In-situ Raman spectroscopy was used to investigate defect complexes and phase transformations during flash sintering. It was shown that the oxygen vacancy complexes in Ce_{0.85}Gd_{0.15}O_{1.925} and the evolving phases in a SrCO₃ + V₂O₅ reaction can be examined with acquisition times sufficient to monitor stage II of flash sintering. The way in which thermal effects can be separated from effects of the electric field was established and the resolution of the defect concentration was determined. This fast characterisation can be combined with well-developed models of thermal and phase evolution to explain the mechanism of densification during flash sintering.

Vapour deposition, pyrolysis, gunning, flame spraying

213693

LASER-DRIVEN CHEMICAL VAPOUR DEPOSITION FOR HIGH-PERFORMANCE FIBRES AND POWDERSHarrison S; Schaefer M - *Free Form Fibers LLC***Am.Ceram.Soc.Bull.** 100, No.7, 2021, p.38-44

In contrast to conventional CVD techniques, laser-driven CVD (LCVD) uses a focused laser beam with a spot size about 20 micron to deliver the energy required for gas decomposition. The resulting temperature gradient (about 10 exp(5) K/m) drives fibre deposition rates that can range from 30 to 300 micron/s. A large range of materials can be fabricated in fibre format and the parallelisation of input laser beams allows multiple fibres to be grown simultaneously. Advantages of LCVD-based fabrication of fibres are discussed. Free Form Fibers use LCVD to manufacture a wide range of fibre and powder products, including SiC fibres, Si₃N₄ fibres, boron fibres, ultrahigh temperature fibres and high-purity SiC powder. The fibres can be used as composite reinforcement.

Lasers/laser treatment

See also Abstract(s): 213434 213440 213485 213507 213516 213524 213586 213645 213693

PROPERTIES AND TESTING

See also Abstract(s): 213402

Toxicity

See also Abstract(s): 213357 213449

Colloid properties, properties of slips

See also Abstract(s): 213383 213500 213634

Rheological properties

See also Abstract(s): 213382 213496 213499 213626 213631

Adhesion

See also Abstract(s): 213672

Non-destructive testing

See also Abstract(s): 213421 213661

Phase relations

See also Abstract(s): 213554 213632