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One flash and it's done

Flash sintering technology has electrifying potential for advanced nuclear fuel manufacture, as **David Pearmain** and **Carolyn Grimley** explain

LUCIDEON, THE INTERNATIONAL MATERIALS TECHNOLOGY company, recently joined the UK Advanced Fuel Cycle Programme (AFCP) as a research and development (R&D) partner. Lucideon's UK facilities join a network of over 100 British and global organisations headed by the UK National Nuclear Laboratory (NNL) and funded by the Department for Business, Energy and Industrial Strategy (BEIS).

Central to the partnership is Lucideon's multi-million-pound investment in flash sintering (FS) technology at its Stoke-on-Trent headquarters. Its expertise allows AFCP to significantly improve the production of advanced nuclear fuels through new developments in the structure and performance of materials.

What is flash sintering?

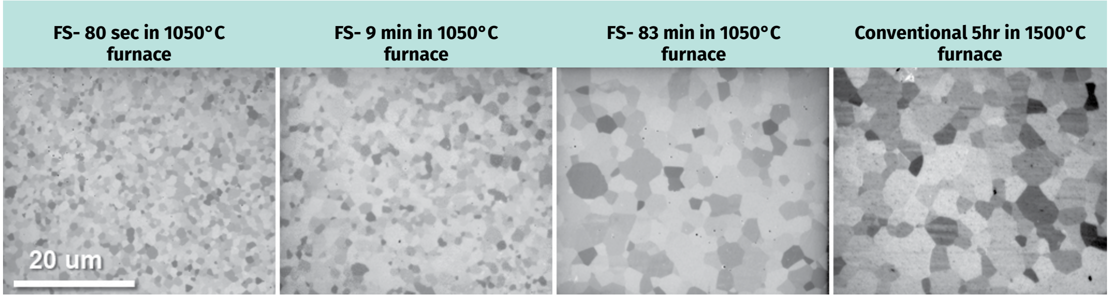
Sintering is a key process in manufacturing the next generation of advanced nuclear fuels. Nuclear fuels are initially produced as powders, which must be densified by sintering before they can be used. This is an energy intensive and time-consuming process. Flash sintering is an innovative technique which incorporates an electric field into the sintering process, offering a more efficient and robust way to densify these promising fuels.

Sintering occurs by atomic inter-diffusion, ie transport of matter from grains into neighbouring pores. The free surface of pores represents a high energy state and, given sufficiently high temperatures, the atoms of a system will rearrange themselves to minimise this energy by reducing the amount of surface area. Conventional furnaces used in sintering processes rely on convection systems to heat materials, with three process-control variables — heating rate, time and temperature. In flash sintering, the electric field is added and consequently add a host of other variables, such as type of field (DC/AC), frequency, voltage, current, time and rate of change in power.

The field is applied to a free-standing body, either by physically contacting electrodes or by a contactless electrode head. With direct dissipation of heat in the ceramic body, as opposed to furnace elements, heating occurs more quickly and more efficiently. In combination with other proposed field enhancing mechanisms, this results in faster densification. Both furnace temperature and time are reduced, with potentially huge productivity gains and a reduction in energy consumption relative to conventional processing.

Furthermore, these new sets of variables can be optimised for individual materials, composites, or dissimilar material systems. When appropriately controlled, they offer a fine level of control over the systems microstructure, and so its properties. An example of this is shown in *Figure 1*.

This advancement represents a pinnacle in the development of new sintering techniques, as depicted in *Figure 2*. Traditionally, sintering has been enhanced by addressing parameters such as pressure or chemical sintering aids, but these offer limited reductions in processing time and temperature. The technique which preceded flash sintering, and is closest to it, is spark plasma sintering (SPS). It also uses electric fields to reduce sintering requirements, and is carried out in a large uniaxial press with an evacuated graphite die, as opposed to a free-standing body in atmosphere (ambient or other



Above Figure 1: **Micrographs of YSZ ceramic structure showing that differing microstructures can be achieved with a variety of optimised FS field parameters, sintering to full density. Far smaller grain size control across the whole sample can be achieved vs the conventional sintering process**

gases, if desired). There are key differences between spark plasma and flash sintering that limit the former's applicability to insulating ceramic materials and large-scale products.

Flash sintering does not require application of uniaxial pressure during processing, and so theoretically lends itself to multiple, or larger, sample sizes.

During spark plasma sintering of most ceramic materials, the current predominantly flows through the conducting die and not through the sample. A greater electric current is therefore needed and the indirect heating lowers densification rate. On the other hand, this type of heating removes the requirement for pre-heating, which may have positive implications for scalability of batch processes.

The presence of a graphite die alters the local redox environment during sintering, creating a more reducing environment in the vicinity of the die. This can lead to spatial variation in the phases present in the sintered product.

The inert atmosphere is necessary to protect the graphite die, but it removes oxygen from the ceramic, dramatically altering the properties of oxide materials.

Flash energy consumption is typically an order of magnitude less than in spark plasma sintering due to the lower applied current required for a given sample.

Greater control over the field enhanced sintering effect is available with FS, and therefore greater control over properties and enhanced ceramic performance.

Applying flash sintering to nuclear fuels

Basic experiments have been performed on nuclear fuels in the past, usually focusing on uranium but including thorium-based fuel as well. In these studies, samples were successfully densified up to 95% theoretical density at temperatures several hundreds of degrees below the conventional requirement.

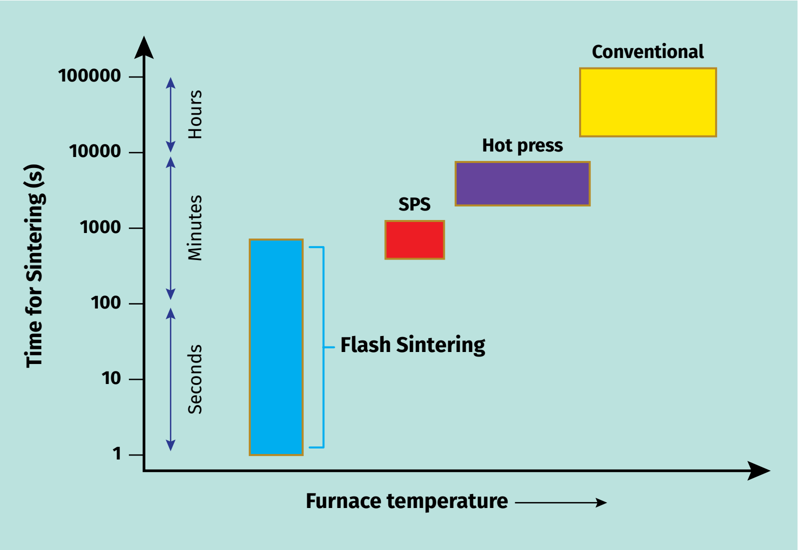
However, a persistent issue in this application has been obtaining homogeneous densification throughout the entire sample. During the process, the path of the electrical current can localise to small regions due to, for example, defects within the monolithic structure. This is encountered in preliminary experiments in a variety of applications and results in a ceramic component which is fully dense in some regions and fairly porous in others.

Clearly, additional work is required to optimise the results and to expand into novel fuel types and high-volume manufacture. In order to develop a robust and repeatable sintering process with complete sintering using flash technology, the application of the field has to be adjusted in real time. Lucideon has developed expertise in applying the technique, including real-time control software to ensure that it can be scaled up and reliable. A simple schematic of this system is shown in *Figure 3*.

Functional ceramics of varying sizes, such as alumina and YSZ, have been sintered using this system using a range of optimised electric field parameters. Full density is achieved for all samples, with far smaller grain size for flash-sintered samples, in comparison with conventional sintered samples. This improves material performance. Ceramic strength and optical improvements have already been demonstrated via this technique, and sintering fuel pellets to optimal microstructures for performance on longevity of life may have a significant impact in advanced reactor fuels.

UK Advanced Fuel Cycle Programme

Lucideon is mapping the full potential of flash sintering in collaboration with NNL, under AFCP. Innovation across

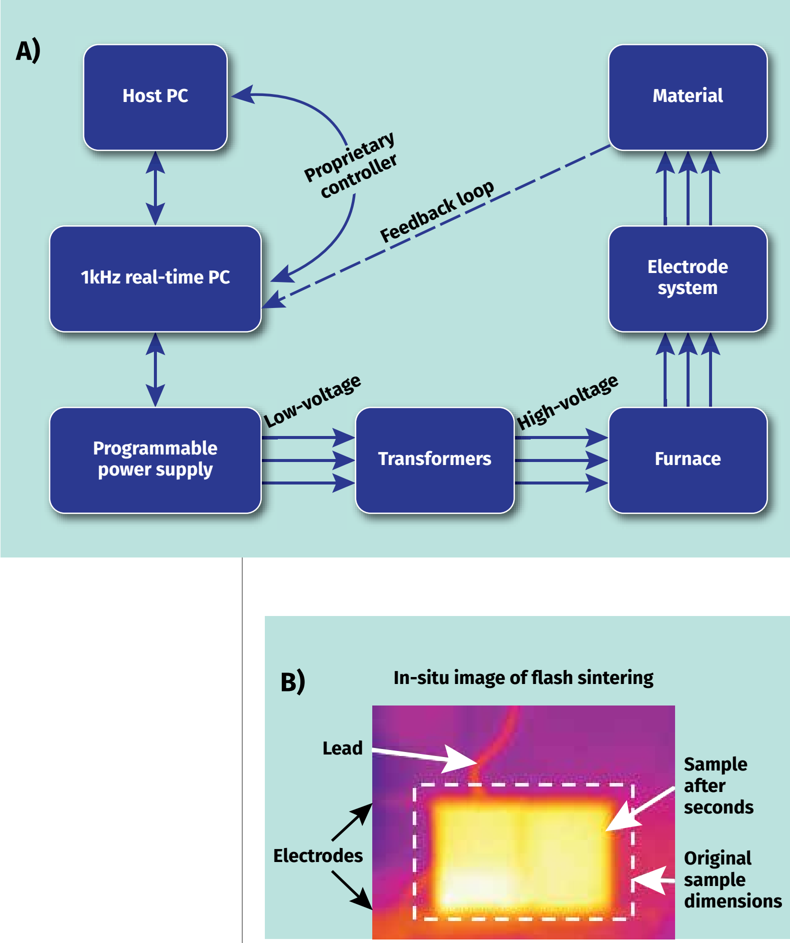


Above Figure 2: **A simplified graphical summary of sintering techniques represented in terms of indicative operating furnace temperatures and times taken to sinter**

AFCP aims to amplify the role of advanced fuels and recycling in a future Net Zero UK. Funded by BEIS, as part of the UK's Nuclear Innovation Programme, AFCP unites nuclear sector innovators. Lucideon's flash sintering expertise aligns with the programme's four strategic outcomes: collaboration, capability, capacity and cost reduction.

The objective of Lucideon's AFCP-supported work is to demonstrate a scalable, robust and optimised manufacturing route for the next generation of advanced reactor fuels. It will develop flash sintering processes to densify nuclear fuels homogeneously, at relatively high speed and with a range of different microstructures. Because flash sintering is faster than conventional routes it may allow an increased production rate, and as a consequence a reduction in factory footprint.

The technology provides a green rapid sintering technique that tolerates fewer pre-sinter processing steps, simplifying the formation route and minimising safety considerations. It may also have elements of microstructural control that allow for improved performance or fuel longevity.



Above Figure 3: **a) Diagram representing FS hardware at Lucideon. Real-time control software essential for technology scale up and reliability due to the requirement to manage inherent instabilities in the FS process. b) Image of a pellet inside the FS apparatus during processing** Source: Lucideon

In manufacturing advanced nuclear fuels, the process begins with contact flash sintering. Initially the fuel pellet material sits between two contacting electrodes (a variety of diameters and thicknesses can be accommodated). For this project, cerium oxide (CeO_2) will initially be used to mimic active material.

Upon completion of successful trials, Lucideon will work alongside a team of AFCP researchers at the University of Manchester using uranium-based active material. The goal of this stage is to successfully demonstrate the feasibility, reliability and robustness of flash sintering at Lucideon, and to show the same capability of flashing fuel pellets of active material at UoM.

A team of subject matter experts will develop and drive Lucideon's proprietary control system, which will be utilised to make real-time decisions about the electric field applied to the material (within given tolerances or instruction). The process will be optimised and evidenced for rapid sintering of pellets, and will demonstrate how the technology should be used to sinter fuels of differing green microstructures or starting grain sizes.

Uniaxial pressure could be incorporated into some experiments to examine whether this additional aid is needed to densify these materials.

This will indicate the ability to scale up and optimise the fuel manufacturing line, including the amount of pre-processing the material needs prior to sintering.

Nuclear, aerospace and the future

When both cerium oxide and uranium dioxide materials have been demonstrated through AFCP, the next step is a clear route to use flash sintering on high Pu-content MOX fuel in a replicated process line at the NNL central laboratory. This will require a glove box to be integrated into the design and will be performed in parallel with the work above, so that scalability challenges are resolved by the end of the project.

The other important element of this delivery will be automation, in forming ceramic pieces and loading material in and out of the furnace. Safety considerations have to take account of best practice around high voltage systems; these are accounted for during development of the technology. The use of robotics and automated systems is already commonplace within the ceramics industry, and it is suggested that these could easily be incorporated into the production method being developed.

Through collaborative programmes such as AFCP, Lucideon is working alongside academics and manufacturers in order to develop the next generation of ceramic processing technologies and demonstrate pilot line scale processing.

The future lies both in the scale-up of bulk flash sintering and in its adaptation to new areas and products.

One particular area of growth is contactless flash sintering. This has lower furnace temperature requirements and it shows great promise for improving the manufacture of metal substrates with ceramic coatings. Contactless electrodes are key to this application. Currently this is performed by rastering the contactless electrode head over the ceramic coating surface and it is under development for solid oxide fuel cell applications. Also, improving the bonding between ceramics and metals would also bring significant benefit to a range of products and industries including advanced reactors, aerospace, and defence. ■