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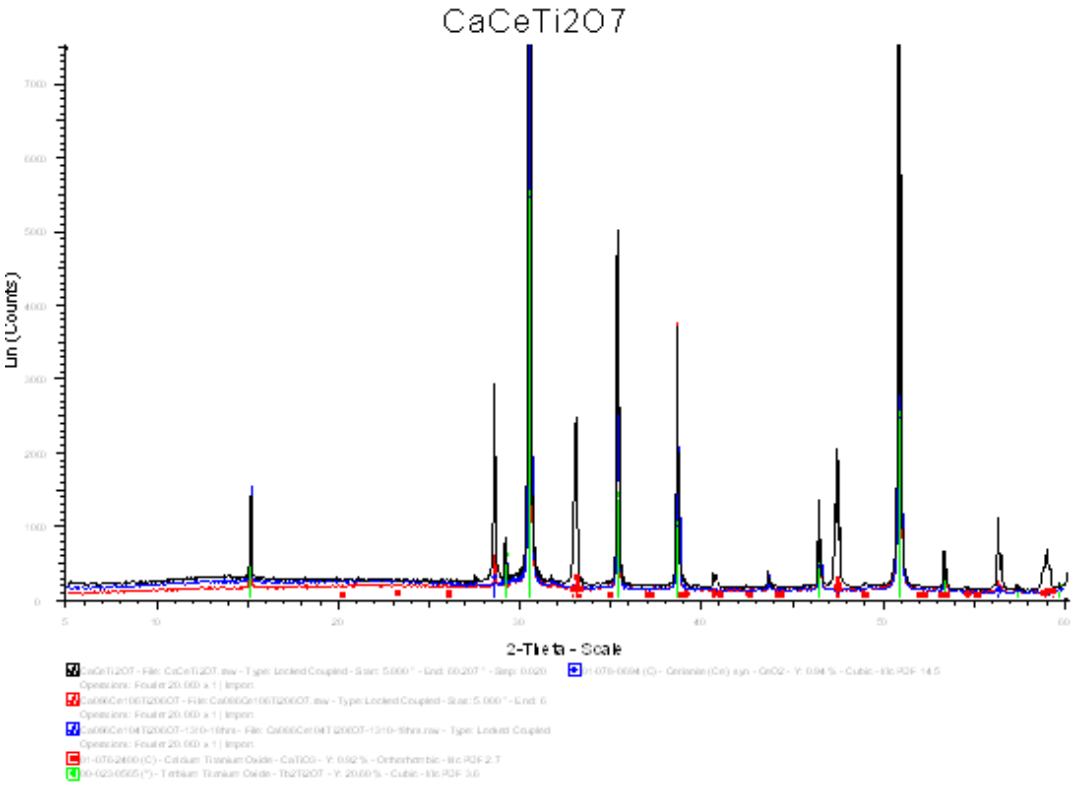
XRD APPLICATION NOTE: QUANTITATIVE PHASE DETERMINATION AND STRUCTURE REFINEMENT OF MATERIALS USING X-RAY DIFFRACTION

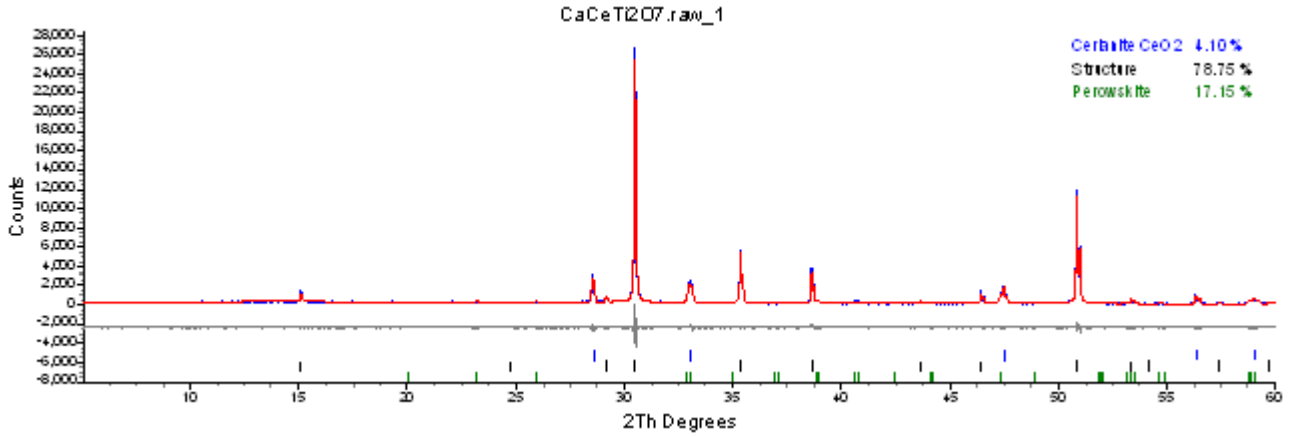
Quantitative X-Ray Diffraction (XRD) is a typical technique for routine quality control of powders and materials discovery. This application note shows how quantitative XRD and full structure refinement can be used to characterise materials and information to enable the starting material to be modified in order to produce a new single phase composition. To illustrate the process, producing a single phase calcium cerium titanate was targeted. An initial attempt to form the stoichiometric phase formed excess perovskite ($CaTiO_3$) and cerianite (CeO_2). From the phase composition, the starting chemistry was altered to produce a single phase product.

Powders with nominal compositions of $CaCeTi_2O_7$, $Ca_{0.86}Ce_{1.06}Ti_2O_7$ and $Ca_{0.86}Ce_{1.04}Ti_2O_7$ were synthesised by solid state reaction of starting powders $CaCO_3$, CeO_2 and TiO_2 at 1300 °C. Data was collected in Bragg-Brentano mode from 5 to 60° two theta in the first instance as a coupled theta-two theta scan. For full structure refinement of the data, an extended collection range up to 120° two theta was used.

Figure 1 shows the various compositions overlaid. It can clearly be seen that $CaCeTi_2O_7$ is tri-phasic comprising of a pyrochlore, CeO_2 and a perovskite phase. For the phase $Ca_{0.86}Ce_{1.06}Ti_2O_7$, the reduced Ca content has resulted in the absence of the perovskite phase $CaTiO_3$ from the products. For $Ca_{0.86}Ce_{1.04}Ti_2O_7$ a 2 atom% reduction of the CeO_2 content in the starting material has afforded a single phase pyrochlore. The structure of this phase has been refined. Figure 2 and table 1 present the resulting Rietveld refinement for phase quantification of the $CaCeTi_2O_7$ composition and the refined contents of the various compositions respectively.



XRD traces outlining the changing phase assemblage with nominal composition.



Quantitative Rietveld refinement of $CaCeTi_2O_7$.

Composition	$CaCeTi_2O_7$	$Ca_{0.86}Ce_{1.06}Ti_2O_7$	$Ca_{0.86}Ce_{1.04}Ti_2O_7$
Pyrochlore wt%	78.8%	99.2%	100%
CeO_2 wt%	4.1%	0.8%	Nd
Perovskite wt%	17.1%	Nd	Nd
Pyrochlore unit cell size (Å)	10.14735 Å	10.14754 Å	10.14762 Å

Table 1 - Quantitative phase assemblage for each nominal composition

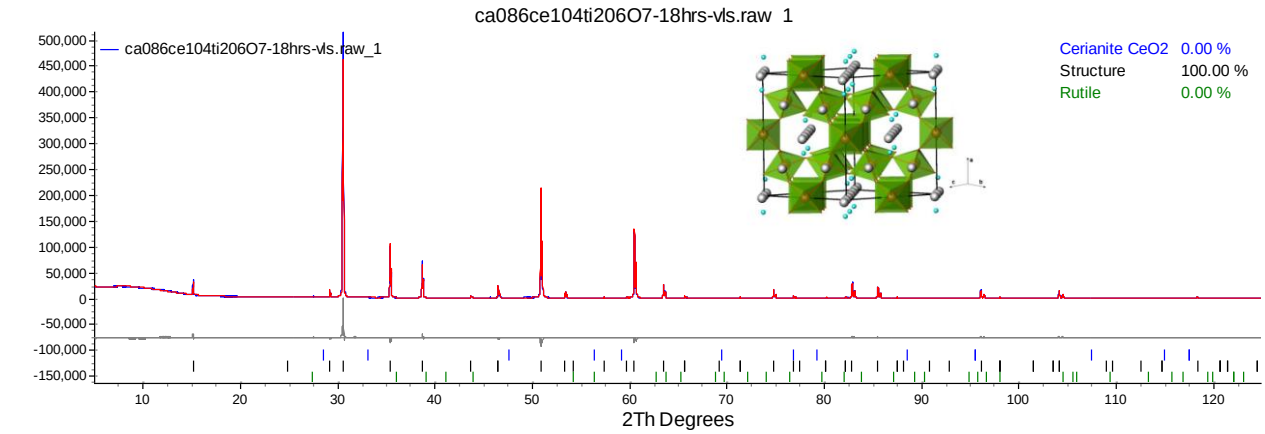


Figure 2 - Structural refinement of single phase Ca0.86Ce1.04Ti2O7 with a representation of the refined structure shown.

XRD has proven to be an invaluable tool in the quantification and characterisation of solid materials. Furthermore, with precise quantification target compositions and structures can be synthesised by "back engineering". The tools are vital in understanding the phase composition in complex systems including electro ceramic components.