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CHARACTERIZATION OF CRYSTALLITE MORPHOLOGY FOR DOPED STRONTIUM FLUORIDE NANOPHOSPHORS BY TEM AND XRD

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M.E. Lee1, J.H. O'Connell1, M.Y.A. Yagoub2, H.C. Swart2 and E. Coetsee2

1Centre for HRTEM, Nelson Mandela Metropolitan University, Port Elizabeth, 2Department of Physics, University of the Free State, Bloemfontein, South Africa

Corresponding author e-mail address: michael.lee@nmmu.ac.za

1. Introduction
   Strontium fluoride (SrF2) is one of the most widely used optical materials due to its optical properties (wide bandgap and low phonon energy) as well as physical properties (low refraction index, high radiation resistance, mechanical strength and low hygroscopicity)[1,2]. Europium doped (optimally ˜2%) strontium fluoride (Eu:SrF2) nanophosphors have been shown to possess improved scintillation properties [3]. Previous work on the characterization of the material by X-ray diffraction (XRD) has produced results on the average dimensions for the nanoparticles [4]. However, analysis of exact morphology and orientation for the nanocrystallites will require alternative techniques such as high resolution transmission electron microscopy (HRTEM). In this paper, complimentary results obtained by HRTEM will be compared to results obtained from XRD.
   Doped and undoped SrF2 samples were prepared by a hydrothermal process described in the literature [5]. The reaction product consists of spherical SrF2 particles having dimensions in the range 0.8-2.0 μm as measured by scanning electron microscopy secondary electron (SEM-SE) imaging. The FIB lamellae produced from the nanophosphor particles were imaged in a TEM. XRD analysis showed the structure of the material to agree with the expected space group Fm3m. The micron-sized particles were assumed to consist of nanocrystallites which are unimodal and single phased. Application of the Scherrer's equation to calculate the crystallite size produced dimensions in the range of 6-8 nm. The accuracy of the Scherrer equation will however depend on a number of factors such as grain size distribution, crystal defects and lattice strain.

2. Results
   The high angle annular dark field (HAADF) STEM imaging of the FIB lamella, clearly demonstrate the nanocrystalline composition of the particles shown in figure 1. The selected area diffraction patterns (SAD) clearly shows the presence of numerous, relatively randomly oriented crystallites. The hollow cone technique is used to produce dark field (DF) micrographs obtained by precessing the indicated diffraction ring through the objective aperture by applying hollow cone precession of the incident beam as shown in figure 2. Exposure time was an integer multiple of the precession period. The hollow cone DF TEM images have demonstrated evidence for a bimodal distribution of crystallite sizes. Firstly, a 5-10 nm size distribution which is in reasonable agreement with the XRD Scherrer calculation was measured. A second distribution of crystallite sizes in the range of 50-80 nm was also observed, as shown in figure 2. Data from the hollow cone TEM technique has therefore shown the crystallite size distribution to be bimodal which was not possible to determine by the XRD Scherrer equation.

3. References
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Primary author: Prof. LEE, Mike (NMMU)

Presenter: Prof. LEE, Mike (NMMU)

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