

Nano-mapping of concentration of Tm^{3+} in wide band gap dielectric crystals with transmission electron microscopy



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Abstract

The concentration of Tm^{3+} ions in wide band gap CaF_2 was mapped with high resolution transmission electron microscopy (HRTEM). The distribution of RE ions was non-uniform for an electron beam size of 2 nm suggesting that Tm ions occupy intersite positions in the form of clusters rather than individual atoms.

Objectives

To test the homogeneity of Tm concentration on the nano / atomic level

Methods

Analytical Electron Microscopy J 2010 F (FEG)

EDXS, HRTEM, HAADF/STEM

EDXS

Questions to be answered.

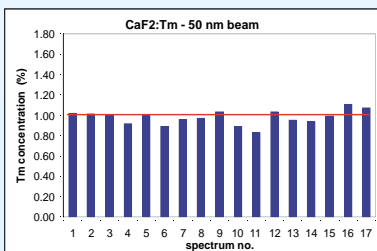
> Even distribution of Tm:

Relative Standard Deviation (RSD) should be comparable depending on counting statistics, (not on beam size).

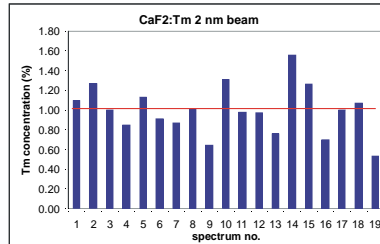
> Formation of Tm nano-sized clusters :

RSD should be inversely proportional to the beam diameter.

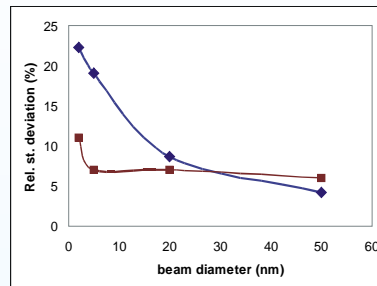
Results



Quantitative EDXS results using 50nm beam diameter



Quantitative EDXS results obtained using 2nm beam diameter



Relative standard deviation of the measured Tm concentration Vs electron beam diameter

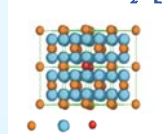
Distribution of results from quantitative EDXS analysis using beam diameter from 2-50nm indicates the presence of nano-meter sized domains with non-uniform composition

Direct observation on atomic level

HRTEM

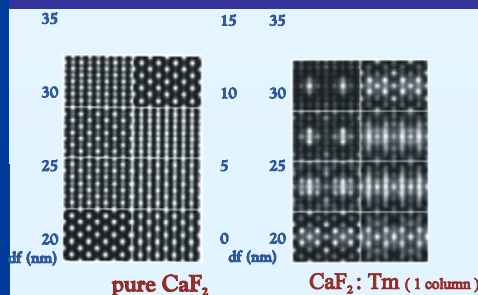
Procedure: Calculate the simulated images on CaF_2 with clusters present and compare the images to the experimental images.

CaF_2 [110]

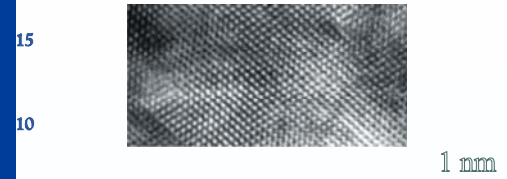


Ca F Tm

SIMULATED

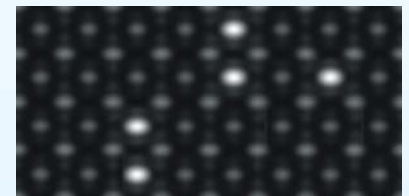


EXPERIMENTAL

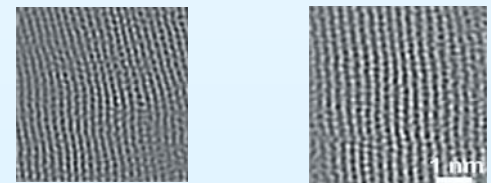


nonuniform contrast

Experimental HRTEM images are similar to simulated ones where clusters of Tm ions were formed.



Simulated HAADF images for CaF_2 in [110] zone with 5 columns of Tm ions



Experimental FFT (filtered) HAADF-STEM images

In experimental high resolution HAADF-STEM images columns with brighter contrast were found

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