Observation of nano-structure cluster formation of rare earth ions in wide band gap fluorine dielectric crystals using transmission electron microscopy with atomic resolution

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Abstract

157nm laser lithography enable the production of microelectronic circuits with dimensions below 100 nm.

Fluorides (CaF_2, SrF_2 and KY_3F_{10}), doped with trivalent rare earth ions could be used as optical elements in the vacuum ultraviolet region.

The refractive index variation of material should be lower than 10^{-6} =>

the optical elements should be prepared from ultra high purity materials.

Objectives

to test the homogeneity on the nano or even atomic level

Methods

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EDXS, HRTEM, HAADF/STEM

EDXS

Perform sets of point analysis using different beam diameter.

Compare the spread of the results

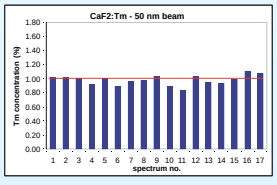
- **If Tm is evenly distributed:**

Re. St. Deviation of the results should be comparable depending on counting statistics, but not on beam size)

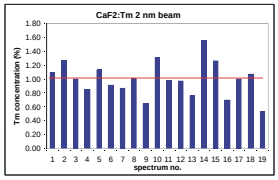
- **If Tm is forming nano-sized clusters :**

Re. St. Deviation should be inversely reciprocal to the beam diameter.

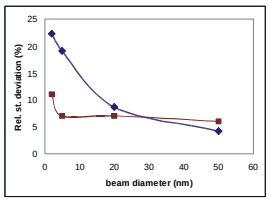
Results



Set of quantitative EDXS results obtained using 50nm beam diameter



Set of quantitative EDXS results obtained using 2nm beam diameter



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

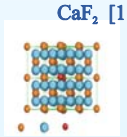
Spread of the results from quantitative EDXS analysis using beam diameter from 2-50nm indicates the presence of nano-meter sized region with non-uniform composition

Direct observation on atomic level

HRTEM

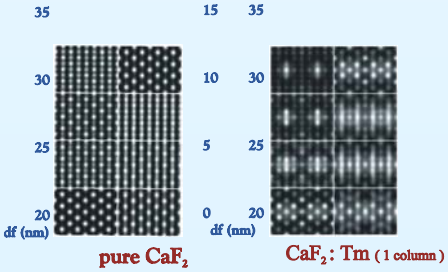
Procedure: Calculate the simulated images on CaF_2 with clusters present and compare the images to the experimental images.

CaF_2 [110]



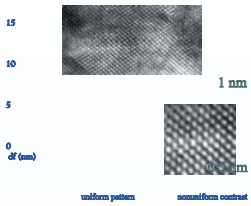
Ca F Tm

SIMULATED

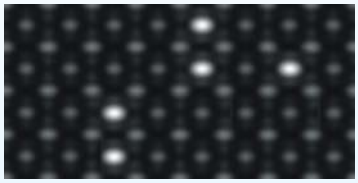


pure CaF_2 CaF_2 : Tm (1 column)

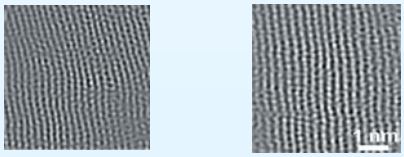
EXPERIMENTAL



In experimental HRTEM image could be found that are similar to those for simulated images where clusters of Tm ions line precipitates) were taken into account



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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