# DETECTION of RARE EARTH DOPANTS at the ATOMIC LEVEL in YAG-based OPTICAL CERAMICS

*Optimizing specific properties of materials frequently involves the addition of small quantities of 'dopants', the detection of which may require an investigation at the atomic level. This is the case of optical, luminescent ceramics (such as YAG polycrystals) doped with rare earth elements. We demonstrate here the positive quantitative analysis of Yb dopants in such a YAG polycrystals by means of high resolution HAADF-STEM imaging in the TITAN, 300 kV transmission electron microscope of the IMR.*

The research activity on advanced optical materials for various applications, such as lasers, scintillators, is greatly increasing with the availability of sintered polycrystalline ceramics. Understanding their optical properties requires a detailed investigation of their microstructure, especially regarding the exact location of the required dopants (e.g. rare earth elements such as $Ce^{3+}$, $Nd^{3+}$ or $Yb^{3+}$). For example, it was recently shown by careful Transmission Electron Microscopy (TEM) observations that Ce strongly segregates at grain-boundaries within YAG (Yttrium Aluminium Garnet $Y_3Al_5O_{12}$) [1].

Identifying dopants at the atomic level by Electron Microscopy techniques is now possible according to the technological progress, especially in the so-called STEM-HAADF mode [2].

STEM-HAADF imaging (Scanning TEM in High Angle Annular Dark Field) consists in scanning the electron probe on the sample, and collecting on an annular detector the primary electrons scattered at high angle. In such a process, the scattered signal is roughly proportional to $Z^2$, where Z is the atomic number of the probed species. Then, heavy atoms (such as rare-earth elements) give rise to a brighter contrast that lighter species (such as O, Al and even Y in the YAG structure). Atomic resolution is achieved if the probe size is smaller than the interatomic distance within the material of interest under some specific viewing crystallographic directions.

We have performed both High Resolution (HR) and HAADF imaging on pure YAG and Yb-doped YAG polycrystals, using a TITAN FEI electron microscope, operating at 300 kV. On the one hand, this instrument is equipped with a $C_s$ aberration corrector on the objective lens, allowing a resolution down to 0.1 nm to be obtained in the HREM mode. On the other hand, it is also equipped with an annular detector collecting electrons in a 70-210 mRad angular range. Nanoprobe chemical analysis was also simultaneously performed using an EDAX EDX (Energy-Dispersive X-ray) analyser mounted on the microscope. Whereas Cs-corrected HREM failed to reveal the distribution of Yb-containing atomic columns, the STEM-HAADF imaging mode appeared to be more efficient owing to its sensitivity to Z. Figure 1 shows a comparison of a 1.4 at.%Yb-doped and a pure YAG samples when observed along the [001] azimuth. A high density of brighter columns is observed for the doped material, which can be consistently analysed in terms of statistics as imaging Yb-containing atomic columns. Dedicated

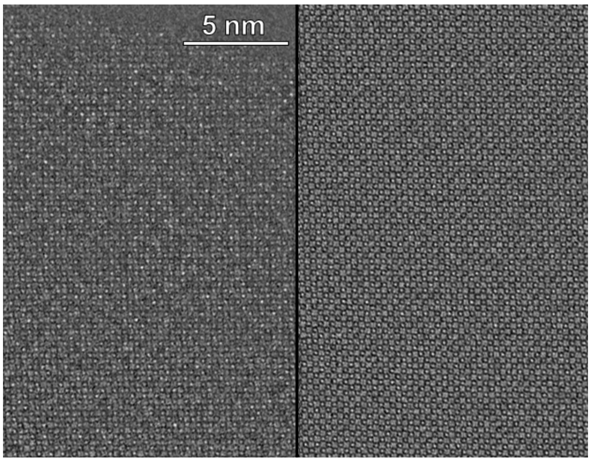


Fig. 1 HAADF-STEM images of two regions of similar thicknesses (as attested by low-loss EELS spectra) from a 1.4 at. % Yb-doped sample (left) and a pure YAG sample (right).

HAADF-STEM image simulations confirm this finding. This work shows that no segregation, neither clustering of Yb ions occurs in polycrystalline YAG.

**References**

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**Key Words**

Optical ceramics, Yb-doped YAG, atomic STEM-HAADF

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