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Microstructural Analysis of Polycrystalline Er:YAG using Automated Crystal Orientation Mapping

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Erbium doped YAG (Er:YAG) is a laser material which emits at 1645nm when excited at either 1473nm or 1532nm, all of which are in an eye safety region. This behavior relies on the crystal operating in a quasi-three-level system, which leads to strong temperature dependence and a strong up-conversion process, which is dopant concentration dependent. [1] To evaluate the microstructure of these ceramic materials, electro-transparent sample preparation was performed by Focus Ion Beam (FIB), Zeiss Crossbeam 340, working with 30, 5 and 2kV gallium ions. The crystalline structure of the lamella was studied using a Scanning/Transmission Electron Microscopy (S/TEM) JEOL ARM 200F microscope operating at 200 kV equipped with an ASTAR system capable of perform Scanning Precession Electron Diffraction (S-PED), for crystal orientation mapping of the Er:YAG sample. The use of PED increases the quality of the indexed maps as the technique reduces dynamical diffraction effects encounter in normal SAED/NBD patterns. With precession the intensities of the diffraction spots are proportional to the square modulus of the structure factor of the material. During the indexing process with the theoretical simulations this will result in higher correlation ratios. The precession angle can be increased to obtain a more kinematical diffraction pattern, but the probe will suffer from beam broadening and the resolution of the map will degrade. For that reason, is important to find a balance between these two variables [1]. In this work, the diffraction patterns were recorded every 5 nm in an automated way by scanning the precessed beam 0.3° at 50 Hz along the sample over an area of 3.5 µm x 2.1 µm under nanobeam diffraction (NBD) conditions using a nominal spot size of 2.0 nm. Then, the set of diffraction patterns were indexed with the ASTAR software considering the Er:YAG structure, until reconstructed an orientation map.

In Figure 1, after all the diffraction patters have been collected, a virtual bright field image (VBF) is produced from the intensity of the central beam. The insets on Figure 1a shows the type of diffraction over the grains. The next three images represent the orientation maps combined with the orientation reliability map to improve image contrast, and to show the degree of confidence. The map illustrates with colors the orientations calculated by the software. The ‘Z’ direction (Figure 1d) is the one familiar with during the TEM data acquisition. The blue color over the bottom right grain at Figure 1d confirms the alignment of the grain over the [111] zone axis. The misorientations between this grain and its neighbors range between 40 to 56 degrees.

Our work proposes that the high quantum efficiency seen in polycrystalline ceramics is due to the chemical segregation of Er at the grain boundaries. Figure 2a shows a Z-contrast image, registered using the high angle annular dark field detector (HAADF) in STEM mode. HAADF-STEM image confirms higher concentration of Er (higher atomic number) at the GB compared with grains I and II in which Er:YAG alloy present the same contrast. Energy dispersive X-ray spectrometry (EDS) was carried out over the GB and in grain II to quantify Er concentration (EDS spectrum in Figure 2b). The reported quantification of Erbium and Yttrium shows a higher concentration of Er at the GB [3].

References:

[1] Jun Zhou, *et al*, Ceramics International. **37** (2011), p. 513.
[2] F. Ruiz-Zepeda, *et al*, Microsc. Res. Tech. **77** (2014), p. 980.
[3] This project was supported by Department of Defense #64756-RT-REP and the National Institute on Minority Health and Health Disparities of the NIH (G12MD007591).

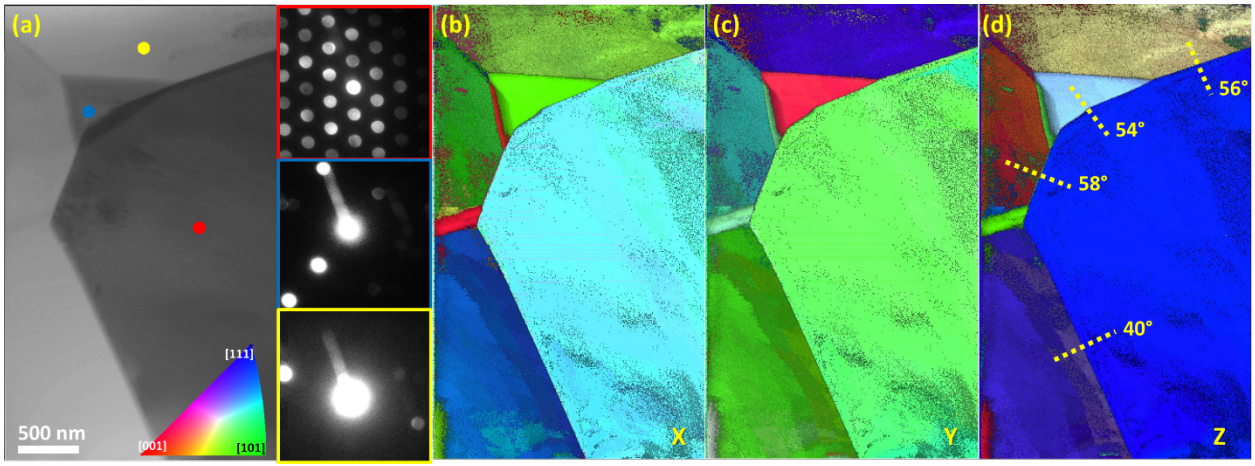

Figure 1. (a) Virtual Bright Field (VBF) image, the insets correspond to the type of DP recorded over those areas. Maps of orientation plus reliability maps of the indexed crystals over the X (b), Y (c), and Z (d) directions, the color diagram on (a) serves to correlate the maps with their orientations. As highlighted in the last figure, misorientations angles can be obtained.

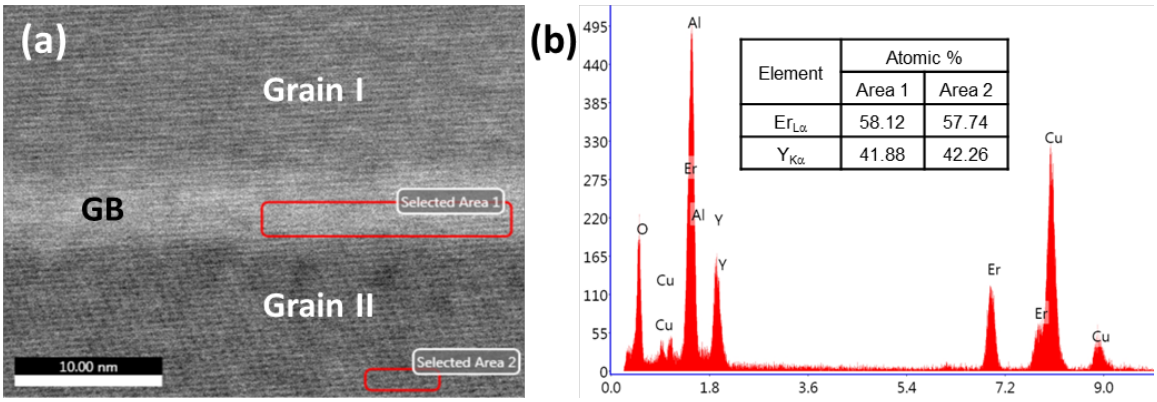

Figure 2. (a) HAADF-STEM image indicating the selected regions for data acquisition, (b) EDS spectrum with a semiquantitative analysis of the EDS spectra collected in selected areas 1 and 2 marked in (a).

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