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Applications of Electron Channeling Contrast Imaging (ECCI) in Failure Analysis of In-Situ Synchrotron X-Ray Diffraction Deformation Experiments

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Significant advances in high pressure deformation of Earth materials have been made in the last decade using in-situ synchrotron X-ray diffraction deformation experiments. The high pressure D-DIA apparatus [1], used for many of these experiments allows monitoring the internal stress within the sample via synchrotron X-ray diffraction. Polycrystalline α-alumina is commonly used as material for deformation pistons in sample assemblies for the D-DIA apparatus. Despite the higher strength of alumina relative to geological materials under study, the alumina pistons occasionally shorten during compression, resulting in an underestimate of the strength of the samples. In order to investigate the failure mechanism of alumina pistons microstructural evaluation is necessary. The primary goal of this study is to showcase the value of using electron channeling contrast imaging (ECCI) in a field emission scanning electron microscope (FE-SEM) for analysis of the deformation mechanisms operating in plastically deformed polycrystalline α-alumina. Improving our understanding of the plastic deformation of the alumina piston material will lead to improved reproducibility in D-DIA data sets and will also contribute to the general understanding of deformation mechanisms in alumina ceramics.

We performed microstructural analysis on an alumina piston that was shortened by 17% at ~7.5 GPa and 730 ºC. ECCI was performed at a 30 keV electron beam energy and a working distance of 3.5-4 mm using a Zeiss GeminiSEM 300. EBSD crystal orientation mapping was performed at a 20 keV electron beam energy and a working distance of 19.4 mm using a JEOL JSM-6700 FE-SEM. The sample was carbon coated prior to imaging and mapping in SEM using a EMS 150T carbon coater.

Examining the sample at low magnification with ECCI, we identified two groups of grains; those that contain deformation twins and grains that exhibit rotation contours. Rotation contour contrast (RCC) indicates the gradual rotation of the lattice across the grain [2], which is diagnostic of the presence of dislocations. Figure 1 shows ECCI micrographs of twinned grains. Figure 1a shows multiple parallel twins across a grain in addition to transgranular cracks and surface porosity. The serrations of the twin boundaries are caused by local grain growth that occurred after twinning. Figure 1b shows a higher magnification micrograph displaying recrystallized grains 10-50 nm in diameter at the twin boundaries. EBSD crystal orientation mapping was performed on the twinned grains. A $\{01\bar{1}2\}$ rhombohedral twinning law was confirmed by comparing the experimental pole figures with a theoretical pole figure. The host and twins were related to each other through the rhombohedral twinning symmetry. For the second group of grains containing RCC, we used high resolution ECCI for dislocations characterization and slip systems identification. First we identified grains with a single RCC in the form of one band of contrast across the width of the grain. An example is displayed in Figure 2. We performed a series of stage tilts with a 0.5º step size in order to find the optimum stage position for dislocation imaging. The tilt series for three stage positions is shown in Figure 2a-c. The edge-on dislocations are revealed at the top corner of the grain indicated with black arrows. We performed EBSD crystal orientation mapping after ECCI to identify the dislocations Burgers vectors and confirm the invisibility criterion in the tilt series.

The importance of this work is two-fold. First, analysis of dislocations and twins in deformed polycrystalline alumina has been historically performed using transmission electron microscope (TEM) [3]. We were able to characterize twins and dislocation in plastically deformed alumina using ECCI in FE-SEM for the first time. Second, taking advantage of a large field of view in SEM and statistically reliable data in the case of heterogeneous microstructures, we conclude that the porosity in sintered polycrystalline alumina could lead to large variations in stress distribution across the piston during compression and ultimately lead to failure of the piston.

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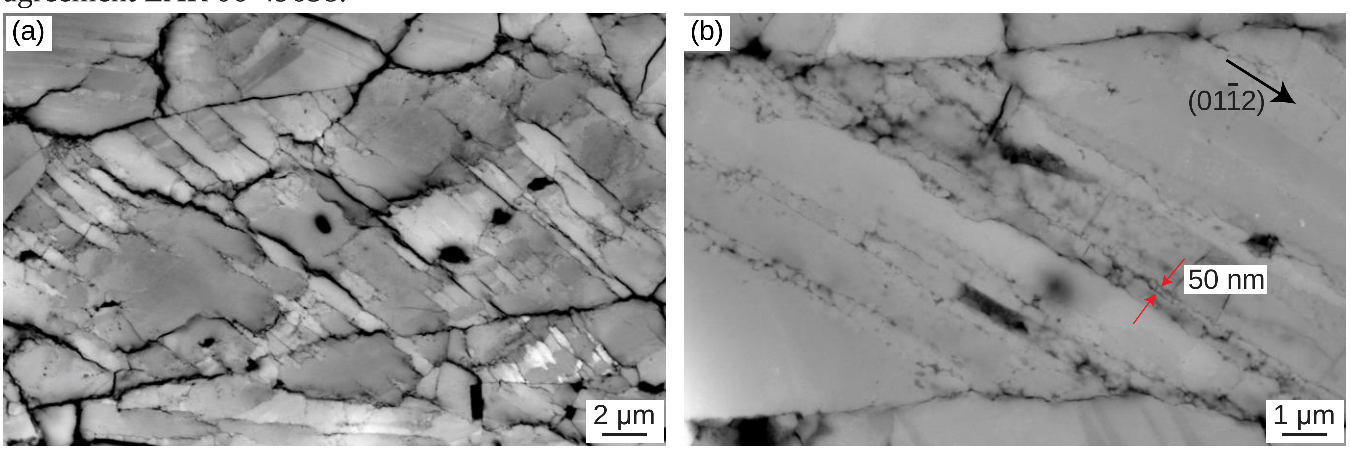

Figure 1. Electron channeling contrast imaging (ECCI) of a twinned alumina grain shortened by 17% at 7.5 GPa and 730 ºC containing (a) multiple parallel twins in addition to transgranular cracks and surface porosity. (b) Electron backscatter diffraction (EBSD) analysis confirmed the $\{01\bar{1}2\}$ rhombohedral twinning law.

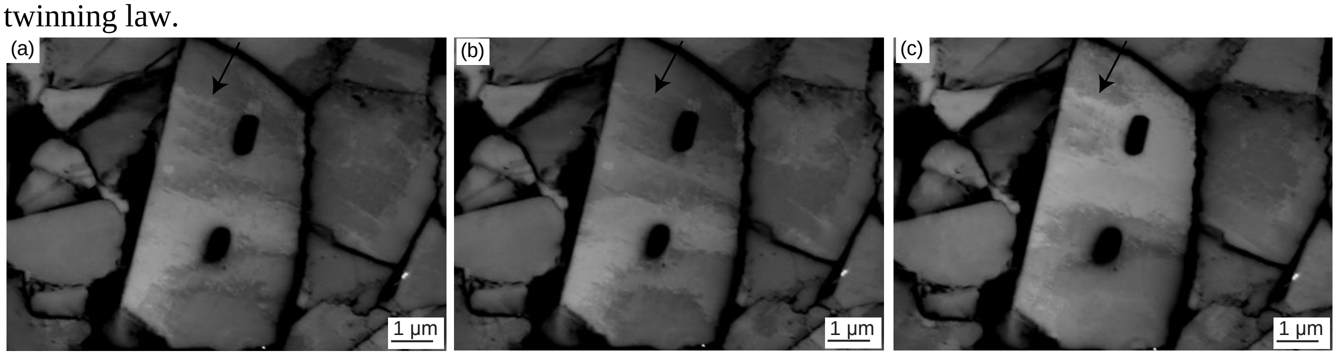

Figure 2. Tilt series with a 0.5º step size on a grain with a rotation contour contrast (RCC). Edge-on dislocations are indicated with black arrows in the top corner of the grain.

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