MA-157 Experiment Report (ID11)

Background

Polycrystalline alumina is an engineering ceramic with anisotropic crystal properties and a tendency for intergranular fracture. This is due to the intergranular strains which develop with the differential thermal contraction of adjacent grains from the sintering temperature. In coarse grain size alumina, these strains can be sufficient to nucleate intergranular micro-cracks at room temperature. In finer grades, micro-cracking only develops in response to additional applied tensile stress. These micro-cracks have a significant effect on the behaviour for macroscopic cracks (i.e. >>1 mm), through micro-crack shielding. Such mechanisms give rise to improved fracture toughness (i.e. resistance to crack propagation). However, the failure of ceramic components, particularly bearings, is determined by their resistance to crack nucleation. This is characterised by their fracture strength.

Our detailed understanding of the processes is very limited due to the difficulty of making observations. Sub-critical cracks are small (<<1 mm), and conventional techniques allow only two-dimensional surface observations or destructive post-mortem analysis. The combined techniques of 3D microstructure mapping by diffraction contrast tomography (DCT) and high resolution tomography provide a unique opportunity to study crack nucleation in ceramics for the first time. This experiment aimed to provide the first in-situ observations of intergranular crack nucleation in ceramic materials, and to validate our understanding of the role of intergranular strains. This will ultimately aid the development of ceramic bearings with improved strength and wear resistance.

Objectives

Our aim was to use DCT (Diffraction Contrast Tomography) to map the microstructure of alumina, and then select samples for compression testing. With increasing applied load, less favourably oriented boundaries and those with less tensile or compressive intergranular strains were expected to fail. It was intended to use diametral compression testing of cylindrical samples. Due to limitations of beamtime, and difficulties in the machining of the coarse grain size alumina, only axial compression was done.

Method

The grain shapes and orientations in the alumina specimen (roughly cube shaped, ~1 mm per side) were mapped using diffraction contrast tomography at beamline ID11. The newly available "portable DCT instrument" was used to transfer the technique from ID19. A beam of 40 keV radiation was used, produced by the Laue-Laue monochromator yielding a bandwidth $\Delta E/E \sim 10^{-3}$. Combined with the in-vacuum undulator, this provided sufficient flux that an counting time of one second per image yielded sufficient intensity, allowing a complete DCT scan to be recorded in less than 2.5 hours. Optics were selected to give an effective pixel size of 1.8 μm, and the sample was positioned ~8 mm from the detector. This allowed the first 6-7 families of diffraction spots to be observed, yielding a relatively large number of diffraction spots per grain, in this case around 25 on average. As the diffraction spots are used as projection information for the algebraic reconstruction of the grains, more diffraction spots mean more accurate and reliable determination of grain shapes.

After mapping of the grain structure using DCT, the sample was loaded in-situ using the same experimental conditions, and a series of absorption tomograms recorded at increasing loads (55, 80, 120, 170N). The sample failed shortly after the scan at 170N, and extensive crack was visible in the reconstructed volume. These absorption constrast tomograms were aligned with the DCT grain reconstuctions, showing the tendency for cracks to propagate along grain boundaries, between intergranular porosities.

Results and Current Status of Analysis

Several samples of coarse grain size polycrystalline alumina were mapped by diffraction contrast tomography. The sample was then loaded in compression, whilst observed by computed tomography, until failure occurred. This confirmed that fracture was intergranular, and that DCT had correctly mapped the microstructure (Figure 1). Future work will correlate the fracture path with the orientations of the grains on either side of the grain boundary.

The diffraction patterns obtained in this experiment are exciting. They show the effects of intergranular elastic strains – these change the lattice spacing and hence the diffraction condition. This is illustrated in Figure 2, which shows a single grain moving through the diffraction condition. Future analysis of these data will be used to calculate the magnitude and distribution of elastic strains, and we will then use these to validate three-dimensional models for the development of intergranular strains from the anisotropic elastic and thermal expansion properties of the alumina crystal.

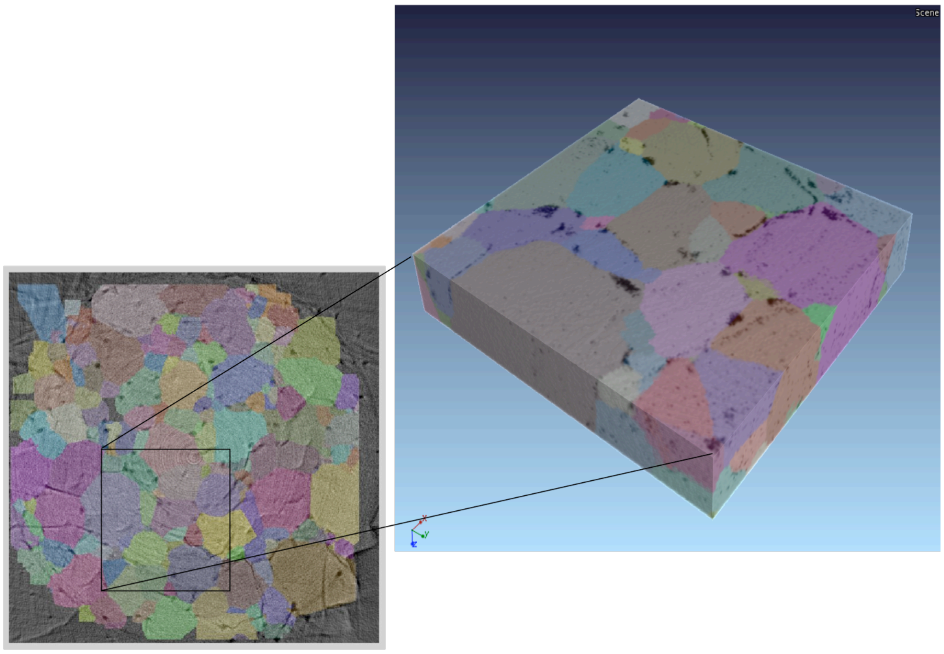

Figure 1: Three-dimensional grain map obtained from polycrystalline alumina. The sample has then been loaded in compression to develop intergranular damage. The sample size is approximately 0.7 mm.

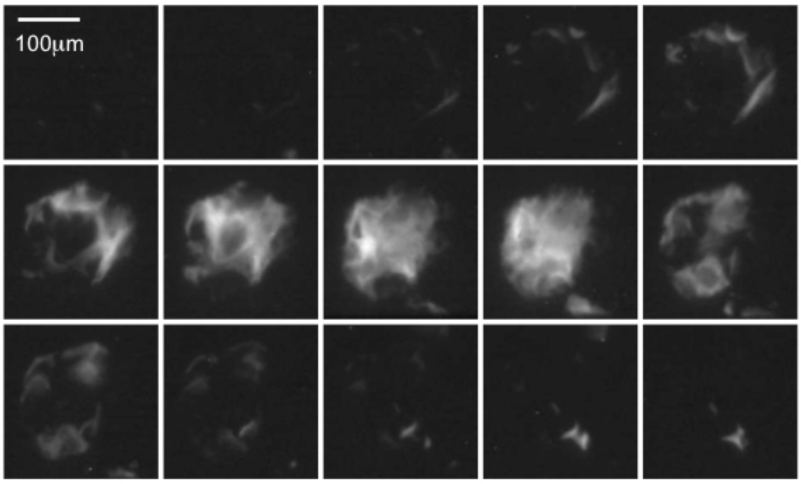

Figure 2: Diffraction spots from a single grain of alumina in a polycrystalline sample, as it moves through the diffraction condition. The images are recorded at intervals of 0.05° rotation.