MA-423: Experiment Report

Background

Cracks are three-dimensional, and they interact with the microstructure of the material, which is also three-dimensional. For example, intergranular stress corrosion cracking of stainless steels is influenced by the structure and properties of the individual grain boundaries. It is also driven by the development of intergranular strains, which depend on the relative crystallographic orientations of the grains. These are sensitive to the texture of the materials, as well as the connectivity and distribution of grain boundary types. These, in turn, are influenced by the thermo-mechanical processing history (i.e. the heating and forging processes use to fabricate the components). The interaction of fatigue cracks with grain boundaries and the nucleation of intergranular cracks in ceramics are other examples where the behaviour of short cracks is very sensitive to microstructure.

Since crack growth rates generally increase with crack length, the majority of the lifetime of a crack in structural components is spent when the crack is short. Uncertainties in short crack propagation behaviour therefore have a strong influence on lifetime prediction. Unfortunately, this is also the regime within which the study and modelling of crack behaviour is most difficult. Uncertainty leads to conservatism in engineering design and can result in unnecessary and uneconomic repair of structures in which the early stages of cracking are identified in service. Better models for the early stages of crack nucleation are therefore required, verified by experimental observations.

Objectives

Our aim was in-situ, three-dimensional observation of the polycrystalline microstructure of single phase and duplex metals, and its role in short fatigue crack propagation. Due to a limit on the number of samples that could be examined in the available beam time, we chose to study just one microstructure, which was a coarse grain size, cast Magnesium alloy.

Our objective was to use DCT mapping and fiducial marks to locate principal grains for crack initiation in suitable samples. These are larger surface grains that extend into the bulk, in which tensile cyclic loading gives high resolved shear stress on a slip plane. Crystallographic stage I fatigue cracks propagate readily on such planes. We aim to evaluate the relative tilt/twist from this plane to slip planes with high resolved shear stress in all the adjacent grains. This is a predicted measure of the boundary resistance to fatigue crack propagation. We had the objective of selecting several principal grains, surrounded by a range of boundary strengths, and by FIB-milling, introduce a stress-concentrating slot parallel to the maximum resolved shear stress slip plane.

Method

Diffraction contrast tomography was used to map the orientations and shapes of the grains in the polycrystalline sample. Tracking was performed using the monochromatic beam provided by the ID19 Bragg-Bragg monchomator ($\Delta E/E \sim$

10^{-4}), and an energy of 20.5 keV. The optics were selected to give an effective pixel size of 1.75 μm, and sample was positioned ~5 mm from the detector. Under these conditions, the first four diffraction families were visible. A few spot pairs from the fifth and sixth families were also observed. The sample was illuminated using a beam size of 1.15x0.43mm, to restrict the total number of grains observed. Diffraction spots with diffraction angles close to η=90°/270° degrees (normal to the rotation axis) where observed to extend across two images on average, indicating a intragranular orientation spread of around 0.1° per grain.

A number of fiducial markers (small Ni spheres) were fixed to the outside of the sample, above and below the volume scanned, and used to find the positions of grains for the placement of FIB notches. During the fatigue part of the experiment, the sample was loaded between 15-85N (approximately 20-110 MPa). Between intervals of cycling tomograms were recorded at a pixel size of 0.7 μm, using the multilayer beam to provide higher flux and hence shorter counting times.

After reconstruction of the DCT grain map and the absorption contrast crack tomograms, the datasets where aligned using the positions of the fiducial markers attached to the specimen, allowing crack paths to be related to the grain microstructure.

Current Status of Analysis

The main objectives of the experiment were achieved. A method has now been established for the use of DCT to study short fatigue crack growth.

Mapping the microstructure of a cast Magnesium alloy in 3D by DCT allowed us to select individual grains, and then to cut a fine notch within each of them using focussed ion-beam (FIB) milling (Figure 1). We then stressed the sample with cyclic fatigue loading, while making successive observations in 3D by computed tomography (CT) (Figure 2). This allowed us to observe the deceleration and acceleration of the crack as it interacted with individual grain boundaries, and showed how the crack grew along particular crystallographic planes (i.e. stage I growth) (Figure 3). Under certain conditions, it adopted a non-crystallographic path (i.e. stage II growth). These unique observations help us to study the transition from stage I to stage II behaviour, which is fundamental to the role of microstructure in short fatigue crack propagation.

Methods are now being developed to calculate the local crack growth rate, and this will be related to the grain orientations, the mode of cracking and the interaction with grain boundaries. A preliminary visualisation of the crack growth rate for one crack (the largest developed) is shown in Figure 4.

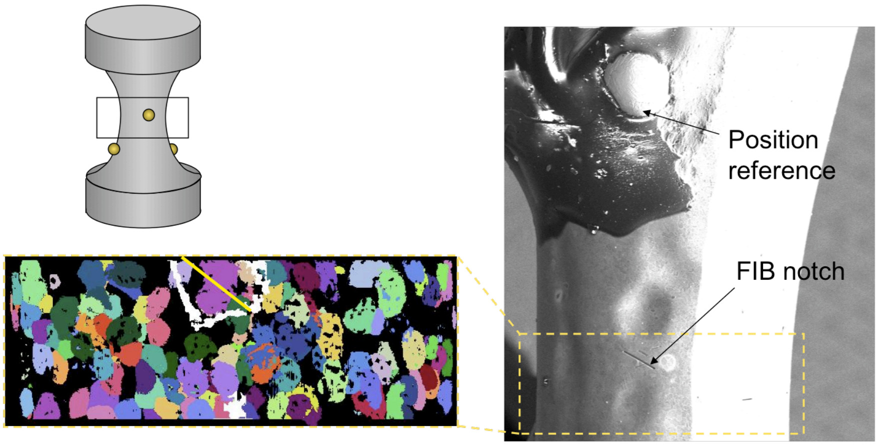

Figure 1: Selection of grains for FIB notch cutting using DCT grain map

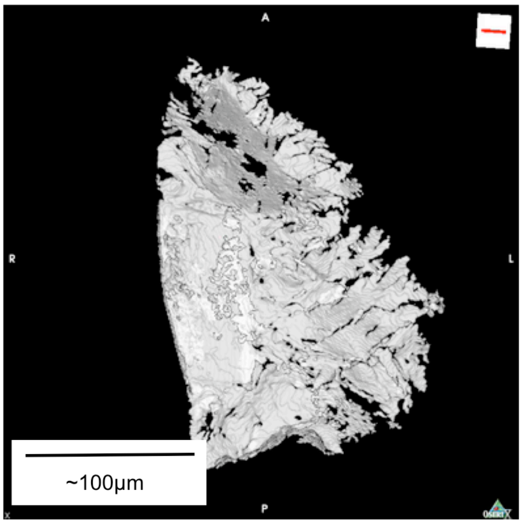

Figure 2: 3D tomography of crack after 10,500 cycles

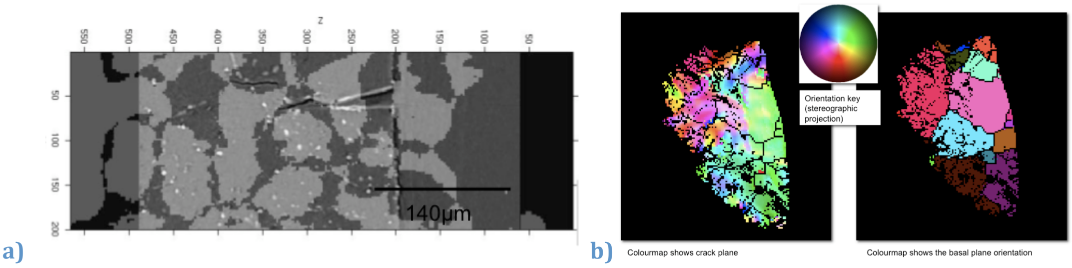

Figure 3: Example data from fatigue of Magnesium, A) 2D section through the 3D dataset, showing the crack propagating from right to left, after initiating at the V-shaped notch. The background grains in the microstructure is shown in grey, b) Surface plot of the crack developed after 10,500 cycles. The colours show the actual orientation of the crack plane (left) and the orientation of the basal crystallographic plane (right). Similar colours at the same location show where the crack is close to the basal plane.

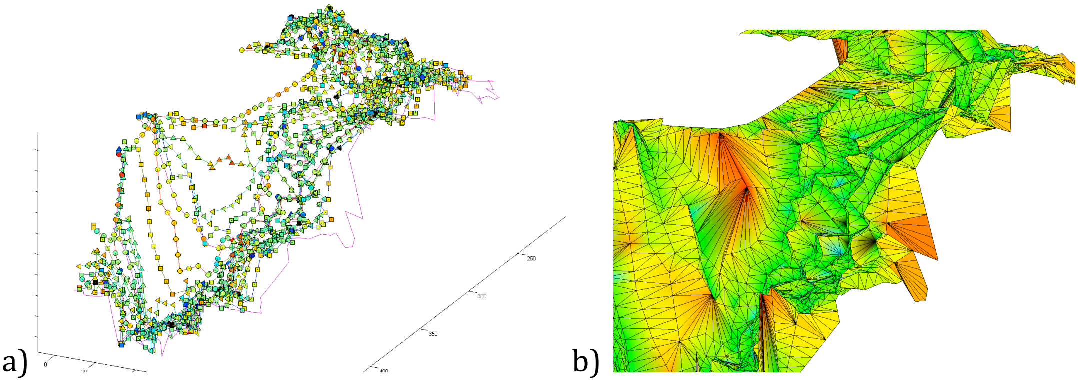

Figure 4: Local crack growth rates, using a) identification of crack tip position at each observation and b) calculation of velocity between successive observations.