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In situ high resolution synchrotron x-ray tomography of fatigue crack closure micromechanisms

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Abstract
Fatigue crack closure has been identified as an important factor in determining crack growth rates. However, the methods of measurement of crack closure remain the subject of ongoing controversy. To date, computed finite element models, analytical models and widely established compliance-based experimental methods have offered limited micromechanical insight and/or direct information on the active crack tip region within bulk material. To understand the absolute contributions of crack closure mechanisms, such as plasticity-induced and roughness-induced closure, to fatigue properties, an internal, three-dimensional insight into crack behaviour during loading and unloading is clearly of value. In this work, synchrotron radiation x-ray microtomography is carried out at a high resolution of 0.7 μm to provide unique three-dimensional *in situ* observation of steady state plane strain fatigue crack growth in a 2024-type Al alloy (Al–Cu–Mg–Mn). Using such high resolution imaging (additionally exploiting the phase contrast effect in interface imaging), the details of fatigue cracks are readily observed, along with the occurrence of closure. A novel microstructural crack displacement gauging method is used to quantify the mixed mode character of crack opening displacement and the closure effect. A liquid gallium grain boundary wetting technique is used in conjunction with the microtomography to visualize the correlation between the three-dimensional structure of the grains and fatigue crack behaviour. Subsequently, electron backscattering diffraction assessment of the grain orientation on the samples provides a uniquely complete 3D description of crack–microstructure interactions.

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1. Introduction

Fatigue crack closure has been recognized for many years as a significant extrinsic factor in controlling fatigue crack growth rates via the shielding of external cyclic loads to the 'active' crack tip region [1–3]. Various mechanisms of closure, such as plasticity-induced crack closure (PICC) and roughness-induced crack closure (RICC), have largely been discussed separately in the literature, on the basis of relatively simple two-dimensional (2D) interpretations of the phenomena. Whilst crack closure has been widely investigated over the last 20–30 years, measurement methods remain the subject of ongoing controversy, with the most widely established methods (compliance based) offering little micromechanical insight, if indeed any direct information on the crack tip region. Recent work from Parry and co-workers [4] has provided novel 2D models of combined RICC and PICC contributions to crack closure in simple, regularly deflecting crack geometries, highlighting the role of local asperity deformations in closure processes. To understand the absolute contribution of crack closure mechanisms to fatigue properties, the extension of 2D understanding, which has been considered within the current literature, to 3D is clearly valuable. Nevertheless, within the available experimental literature, the 3D viewpoint has been largely neglected. 3D crack closure behaviour is problematic to assess, either by post-failure analyses (such as fracture surface observation), or by conventional *in situ* surface observations. In view of this, it would appear that *in situ* synchrotron x-ray tomography provides a unique possibility for understanding the contributions of crack closure mechanisms such as PICC and RICC to fatigue properties. Guvenilir *et al* [5] have demonstrated the use of this technique in this application; however, they only achieved a voxel resolution of 6.0 μm.

2. Experimental methods

Given the importance of establishing steady state crack growth conditions in assessing long crack closure [3], macroscopic three-point bend fatigue tests were carried out on commercial airframe aluminium alloy 2024-T351, at a load ratio of 0.1 and frequency of 50 Hz in an L–T orientation (crack growth in the rolling plane but perpendicular to the rolling direction). A single-edge notched (SEN) specimen was used, with specimen width, thickness and length corresponding to 20, 5 and 130 mm. Specimens were pre-cracked under a constant amplitude loading.

To perform high resolution microtomographic imaging at the submicrometre level, a rather small crack tip sample was needed. The small samples were prepared from the crack tip at the original SEN sample centre using a slow speed diamond saw to preserve the crack profile ($\sim$1 mm $\times$ 1 mm in cross-section, 25 mm in length). Finally, a pair of T-shape brass tabs were bonded at each end of the sample to provide gripping shoulders for *in situ* loading in the synchrotron experiments. Care was taken in experimental design to ensure a reasonable degree of elastic constraint of the plastic enclave around the crack (to allow crack closure to occur).

Tomography was performed at beamline ID-19 of the European Synchrotron Radiation Facility (ESRF) in Grenoble, France. *In situ* straining was performed on the small sample to different load levels in a specially designed mechanical load cell. Image slices were reconstructed using a conventional filtered backprojection algorithm [6] with an isotropic voxel resolution of 0.7 μm.

A liquid gallium (Ga) grain boundary wetting technique was then applied to the sample prior to further scanning to characterize 3D grain structure [7]. This was followed by *post-mortem* sectioning and electron backscattering diffraction (EBSD) analysis of the Ga charged samples to give grain orientation information and also to confirm the Ga wetting results.

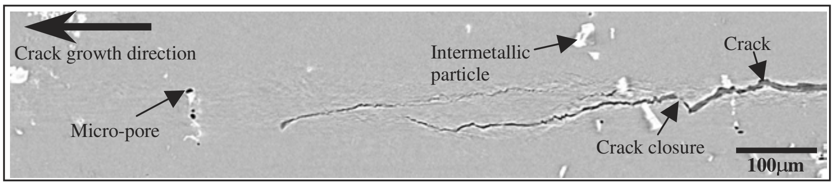

Figure 1. A 2D slice of a reconstructed volume illustrating the crack morphology and microstructural features along the crack growth direction.

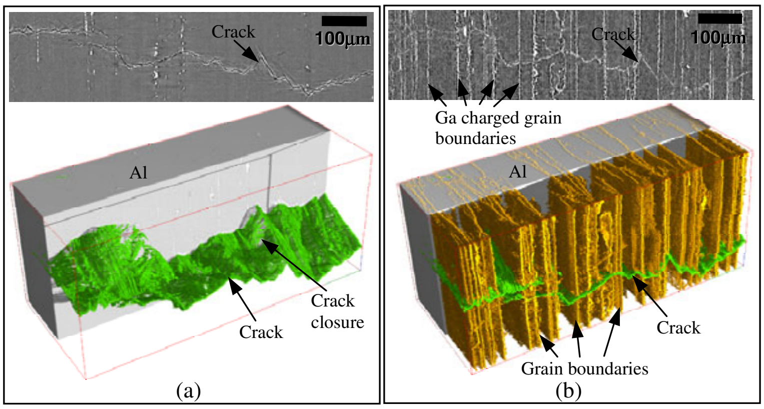

Figure 2. A 2D cross-sectional slice and 3D rendered perspective view of a crack in a tomographic volume (a) without and (b) with application of Ga.

3. Results and discussion

Figure 1 shows a typical 2D cross-sectional crack image from the reconstructed tomography data set. A complex crack morphology can be seen at the centre of the image, with relatively spherical micropores (black) and complex intermetallic particle groups (white) in the surrounding matrix. A conventional RICC 'event' can also be seen at a deflection in the crack wake.

The crack volume was segmented and analysed in 3D. A progressive change in closure area was identified as a function of loading. Closure areas were found to be linked with local topology such as ridges running parallel to the crack growth direction. For a more quantitative analysis of the closure phenomenon, a microstructural displacement gauging method was established to measure the crack displacement. In this technique, pairs of micropores located on each side of the crack were segmented and positions monitored during loading as a displacement gauge. By measuring centroid displacements at different loading levels, mode I, II and III displacements of the crack were obtained (see [8] for details).

Figure 2 shows 2D crack images and 3D rendered volumes that illustrate the differences between tomography images with and without the application of Ga to the sample. With the application of liquid Ga, lines of contrast can be seen running parallel to the loading direction, consistent with the known grain structure of this rolled plate material. A clear visualization of apparent 3D grain boundary structure has been obtained by segmenting the boundaries from the aluminium matrix.

To assess the 3D segmented grain boundary mapping quantitatively, it is compared directly with an EBSD orientation map in figure 3. A misorientation profile is plotted across the *y*–*y*

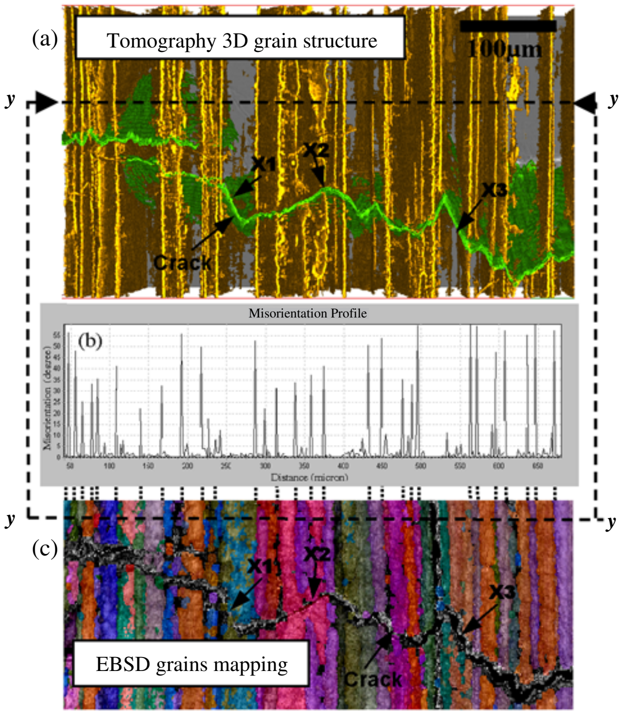

Figure 3. Comparison of the EBSD grains map and tomography data: (a) 3D reconstruction of grain boundaries (yellow) and the crack (green), (b) grain misorientation data from the EBSD data in (c), along the line *y*–*y*, and (c) an EBSD orientation map of the sample at the front plane of the image (a).

section showing the misorientation angles of grain boundaries. From the results, it can be seen that most of the high angle grain boundaries have indeed been picked up reasonably well by the 3D segmentation of the Ga wetted boundaries (i.e. matching with the EBSD orientation map), although several individual boundaries can be identified with little or no Ga effect, presumably associated with special boundary orientations [7]. It is valuable to note that subgrain structure prevalent in such a commercial microstructure is not picked up by the Ga wetting technique (consistent with a low effective surface tension in the low angle boundaries), which significantly clarifies the crack–microstructure imaging. A preponderance of {100} and {111} oriented crack growth is in fact identifiable at locations X1, X2 and X3 in figure 3 (i.e. linking crack plane orientations to the EBSD data), consistent with slip band crack growth occurring (shear decohesion).

4. Summary

High resolution synchrotron x-ray microtomography has been successfully utilized to perform *in situ* observation of fatigue crack closure behaviour in a high strength aluminium alloy. By sampling a small tensile specimen within an *in situ* testing rig (from a standard macroscopic fatigued specimen), high resolution observation (isotropic voxels with 0.7 μm edges) of a crack that has been propagating under steady state, plane strain conditions has been realized. The details of a crack, such as its surface topology, bifurcation and tip geometry, are readily observed by volume rendering, and evidence obtained of the occurrence and mechanical influence of crack closure. The practicality of liquid Ga grain boundary wetting techniques in x-ray tomography study of long fatigue crack behaviour has been shown.

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