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A quantitative three-dimensional in situ study of a short fatigue crack in a magnesium alloy

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ABSTRACT

A previous four-dimensional in situ study of a short crack in a magnesium alloy King et al. (2011), Elektron 21, used synchrotron X-ray computed micro-tomography to follow its three-dimensional development with progressive fatigue cycling through the microstructure, which had been mapped by diffraction contrast tomography to measure grain shapes and crystal orientations in three dimensions. In the present work, very high-resolution post-test examination of the same sample by Serial Block Face Scanning Electron Microscopy (SBFSEM) provided three-dimensional fractographs to investigate the influence of microstructural features on the measured crack propagation rates. Digital volume correlation, applied to the X-ray computed micro-tomography datasets, measured the three-dimensional crack opening displacements and hence the crack opening modes. The short fatigue crack in magnesium propagated with mixed mode opening. Basal plane fracture is a dominant mechanism; hence, boundaries that disrupt the continuity of the basal plane are proposed to influence the crack propagation rate.

1. Introduction

Cracks have three-dimensional (3D) geometries; they also interact with the three-dimensional microstructure of the material as they develop. These interactions are particularly important for *microstructural short cracks*, which are short relative to microstructure length scales, such as grain size, since the crystallographic orientations of the grains can strongly affect stage I cracks that follow particular crystallographic slip planes [1]. The fatigue lifetime of engineering components can be dominated by the short crack regime; hence, understanding of short crack behaviour is quite important. However, the short crack regime represents a field of experimental study and modelling of 3D crack behaviour that is most difficult, with detailed understanding and data most limited despite significant developments in recent decades (e.g. [2,3]). A key goal of research into short fatigue cracks is predicting the probability of crack growth to a critical size within a given interval of life. The intrinsic material factors on which this depends include crystallographic texture, grain shape and size. In order to implement these in predictive models such as [3], knowledge is required of crack growth rates within grains and the resistance to crack growth presented by features such as grain boundaries.

Three-dimensional in situ observation of crack development within materials has become possible with the wider availability of high-resolution X-ray computed micro-tomography (μXCT) [4–9], aided particularly by the brilliance of synchrotron sources that allow sequential observations in a reasonable time. Recently, non-destructive 3D grain mapping techniques such as synchrotron X-ray diffraction contrast tomography (DCT) have complemented μXCT by providing a description of grain shape and crystal orientation in polycrystalline materials [10]. DCT, which maps the microstructure by reconstructive analysis of the diffraction spots from individual grains, has been used to characterise the microstructure prior to crack development, showing, for the first time, the interaction of cracks with grains and grain boundaries [9–12]. In one recent study [13], short fatigue crack growth behaviour in a cast magnesium alloy (Elektron 21) was observed in 3D using a combination of DCT and μXCT at the European Synchrotron Radiation Facility (ESRF). The evolution of a fatigue crack from an artificial notch, smaller than one grain, was studied in situ during interrupted fatigue cycling. Crack growth occurred preferentially on the basal plane of the magnesium hexagonal close packed crystal, with local crack growth rates varying between 4 and 40 nm/cycle. By combining DCT and μXCT, it was possible to relate the orienta-

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tion of the local crack plane to the grain orientations and, hence, to the crack propagation behaviour. Higher crack growth rates were associated with basal plane propagation and lower rates tended to be associated with non-crystallographically oriented regions of the crack. Crystallographic crack propagation was observed to be retarded at boundaries with large tilt/twist misorientations of the basal slip plane. However, the resolution of the μXCT observations was insufficient to discern the fine topography of crack propagation (i.e. fractography); this could have determined, for instance, whether the crystallographic to non-crystallographic transition was associated with a change from a flat topography to the "staircase" morphology of alternating propagation planes [14].

The Schmid factor is a fundamental element in models that are used to interpret observations of crystal deformation and also in predictive models for fatigue initiation and propagation; it is a measure of the resolved shear stress on the slip systems of a grain. Crystallographic fatigue crack growth is observed to occur along slip planes of high Schmid factor [15], and models for fatigue assume such cracking to be driven by the shear modes of deformation [16]. The transition from crystallographic to non-crystallographic fatigue crack propagation is considered to be accompanied by a transition from shear to tensile modes of crack opening (i.e. mode II to mode I) as multiple slip systems are activated [17]. In simpler fatigue models, the Schmid factor may be estimated with an assumption of uniaxial tension [18,19], whilst more sophisticated crystal plasticity models consider the local stresses and strains from three-dimensional grain interactions [3,20]. In the case of crack propagation, the geometry and orientation of the three-dimensional crack must also be taken into account [21]. These define the local magnitudes and modes of the crack opening displacements that, to our knowledge, have never been measured, at least in three dimensions, for a short fatigue crack. Since the above-mentioned study on Elektron 21 was conducted, two novel techniques have become available that can provide the necessary insight into the fractography and opening modes of three-dimensional cracks: these are (i) very high resolution, large volume ultramicrotomy with low-voltage scanning electron microscopy, and (ii) digital volume correlation of μXCT datasets.

Destructive, i.e. post-test, techniques of serial sectioning that typically use focused ion beams to reconstruct the microstructure with very high resolution have had limited application in studies of short fatigue cracks [22] since the sectioning of the large volumes required to observe the crack in sufficient grains can be prohibitive. Conversely, although X-ray tomography allows the study of larger volumes, its resolution (typically sub-micron) is generally insufficient for the 3D fractographic characterisation of crack propagation, which is necessary to determine the crack propagation mode with confidence; useful observations have been achieved, though, when the fracture surface roughness is significant [9]. The new destructive technique of ultramicrotomy by Serial Block Face Scanning Electron Microscopy (SBFSEM) [23] allows larger volumes to be studied with very high resolutions achievable by scanning electron microscopy. Although such post-test methods can only investigate damaged and deformed microstructures, thereby losing the damage chronology afforded by μXCT, they may complement lower resolution in situ studies.

Previous X-ray tomography studies have been largely qualitative, taking advantage of 3D visualisation to measure crack morphology and dimensions (e.g. [6–8]). Quantitative measurements can be obtained now by digital volume correlation (DVC), which is a powerful analysis technique applied to sequential three-dimensional observations to map the relative changes in displacement with sub-voxel resolution [24]. With increases in computational power, DVC can be readily applied to the large datasets produced by high-resolution X-ray tomography. Often applied in studies of bulk deformation and plasticity [25–28], it can yield particularly useful insights into crack development by allowing precise measurement of crack opening displacements and modes of loading [30–32].

In this paper, we have returned to the magnesium fatigue sample and X-ray micro-tomography data of the previous experiment [13] in order to investigate the fractography of crack propagation by SBFSEM of the sample and also to measure the modes and magnitude of the crack opening displacements by DVC. Our goal is to demonstrate the additional insights that can be obtained which, in future work, might be used to validate advanced crystal plasticity models for short fatigue crack propagation and the fatigue lifetime prediction of engineering components.

2. Methods and materials

2.1. In situ observation of fatigue crack propagation by X-ray microtomography (μXCT)

A full description of the fatigue experiment has been provided in the previous publication [13]; the relevant details are reported here briefly. The material was an annealed Elektron 21 magnesium alloy (a casting alloy containing nominally 2.6–3.1% neodymium, 1.0–1.7% gadolinium and 0.2–0.5% zinc), with a grain size of approximately 55 μm. Following DCT characterisation of the tensile fatigue specimen (1 mm gauge diameter), a focused ion beam (FIB) instrument was used to introduce sharp notches in selected grains.

The sample was fatigue tested with a sinusoidal waveform at a frequency of 5 Hz with a maximum stress of 130 MPa and R-ratio (minimum/maximum load) of 0.25. The cycling was interrupted at intervals for tomographic scanning from 500 to 10250 cycles. All the μXCT scans were performed with the sample under maximum load. The effective voxel size of the reconstructed tomographs was 0.7 μm. The resolution of the reconstructed DCT grain map was 1.75 μm.

2.2. Digital volume correlation (DVC) analysis of 3D displacements

The μXCT datasets were analysed with respect to a reference dataset (500 cycles; first dataset recorded) using digital volume correlation software (LaVision StrainMaster 8.1.6). This paper presents data for 1000, 3500 and 10250 cycles, referred to as *early*, *intermediate* and *final* data. Cropped regions of the datasets at one notch were examined; each was 320 × 260 × 180 voxels in size. DVC analysis provides a 3D displacement vector field on a discrete grid; the DVC analysis was performed on successively refined grids using with step-wise reducing size interrogation subsets; these were initially 128 × 128 × 128 voxels (with 50% overlap with neighbouring interrogation subsets), then 64 × 64 × 64 voxels (50% overlap) and finally 32 × 32 × 32 voxels (75% overlap). Three passes were performed at each step with each pass informing the interrogation subsets in the subsequent pass. The final displacement field was thus calculated with a grid separation distance of 24 voxels (~17 μm). A vigorous censorship criteria, requiring a correlation coefficient larger than 0.9, was applied to eliminate displacement vectors with low accuracy.

2.3. Post-test Serial Block Face Scanning Electron Microscopy (SBFSEM)

In order to obtain 3D images of the fatigue-tested specimens, sequential sectioning by ultramicrotomy within a scanning electron microscope chamber was performed; this technique is referred to as Serial Block Face Scanning Electron Microscopy (SBFSEM). The specimen was first trimmed with a conventional