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Evolution of intragranular stresses and dislocation densities during cyclic deformation of polycrystalline copper

Jun Jiang,[a,b,*] T. Benjamin Britton[b] and Angus J. Wilkinson[a]

[a] *Department of Materials, The University of Oxford, Park Road, Oxford OX1 3PH, UK*

[b] *Department of Materials, Imperial College London, Royal School of Mines, Exhibition Road, London SW7 2AZ, UK*

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Abstract—We have used the cross-correlation based high resolution electron backscattering diffraction (HR-EBSD) technique to evaluate at high spatial resolution the spatial patterning of the type III intragranular residual stresses and geometrically necessary dislocation (GND) density within polycrystalline Cu after cyclic deformation. Oxygen free high conductivity (OFHC) polycrystalline copper samples were cyclically deformed under stress-control at a load ratio of 0.1 and EBSD measurements were made at points throughout the early stages of fatigue when strain amplitudes and cyclic creep rates are changing most significantly, namely at 0 cycles, 2 cycles, 200 cycles and 2000 cycles. Statistical analysis is presented showing that moderate correlations exist between stored GND density, residual intragranular stress and distance from the nearest grain boundaries and/or triple junctions.

Keywords: Fatigue deformation; Geometrically necessary dislocations (GNDs); Intragranular stress; High resolution electron backscattering diffraction (HR-EBSD)

1. Introduction

Research on fatigue failure has a long history with perhaps the earliest report given in 1837 by Wilhelm Albert who studied iron chain subjected to repeated loading [1]. It has been stated that fatigue accounts for at least 90 percent of all service failure due to mechanical causes [2], and that the cost of fatigue failure is high (4% of US gross national product [3]) and can lead to catastrophic incidents. Many studies have been carried out over the past ~150 years to improve our understanding as this is one of the most critical materials failure modes.

Significant progresses of fatigue deformation development especially fatigue crack formation have been made on single crystalline materials by characterising dislocation slip, interaction and accumulation using transmission electron microscopy (TEM) [4–9]. These provide significant insight into very local mechanisms but necessarily avoid any effects of constraint imposed by neighbour grains in a polycrystal. The study of single crystals is an important building block but the majority of engineering structural materials e.g. metal alloys, ceramics and natural rock consist of aggregates of grains with characteristic crystallographic orientations.

Design and safety cases typically use physical insight from single crystals to inform empirical equations for life and failure tolerance and detailed microstructural variation is not taken into account. This drives a safety culture which is very conservative and limits cost savings. Movement towards physically motivated design rules, informed from accurate and high fidelity microstructural investigations, will enable a transformation towards ‘sufficient but not excessive’ design, enabling improved life and a reduction in material used within components, thereby imparting significant savings in, for example, fuel consumption and CO_2 emissions in transport applications.

Extension of knowledge towards polycrystalline materials is constrained by limited supporting experimental evidence. In polycrystals the complexity of microstructure (combination of grain size and orientations) gives rise to heterogeneous deformation distribution. Therefore observations must not only consider the macroscopic loading conditions, such as an externally applied component level stress, in combination with stress risers such as structural defects like notches, but also consider microstructural effects on the distribution of cyclic slip and stress variations. Where ‘weakest-link’ failure modes exist, such as in fatigue, the local combination of grains shapes and orientations may elevate the likelihood of failure. To understand this, high spatial resolution characterisation techniques, with high fidelity, must be employed in order to resolve the inhomogeneous dislocation density, stress or strain

* Corresponding author; e-mail: jun.jiang@imperial.ac.uk

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distributions within grains and also across large areas to map sufficient number of grains to understand how aggregates of grains deform together to accommodate cyclic deformation.

Traditionally this problem has been tackled using either high spatial resolution techniques, such as TEM, or volume averaged high fidelity techniques such as high energy X-ray diffraction of high energy neutron diffraction. Diffraction contrast TEM observations have been highly influential in characterising dislocation sub-structures such as dislocation veins, persistent slip bands (PSBs), labyrinths, and cell structures that form during cyclic deformation [4,8,9]. However, the observed region of interest using TEM is limited to $\sim 10 \times 10\ \mu m^2$ in a thin foil, which often only contains limited number of grains (potentially less than one in many cases) so that co-operative processes leading to dislocation patterning which spans many grains are typically not captured. High energy X-ray and neutron diffraction have mainly been used to probe macro residual stress (type I) distributions near large-scale component features such as holes and notches using diffraction peak positions [10]. Furthermore peak broadening occurs due to very short range stress fluctuations (type III) caused by dislocations introduced by plastic deformation and has extensive literature associated with it [11–14]. Although short range stress fluctuations are responsible for the effect only the average response over a relatively large volume is obtained and the true spatial distribution cannot be recovered. Innovations in synchrotron-based X-ray diffraction methods have provided routes to measure grain averaged residual stresses (type II) [15–17]. Transmission powder diffraction geometry is used with the sample rotated to generate patterns in which individual diffraction spots can be segmented and assigned to individual grains. A large number of grains are probed in parallel so statistical analysis can be undertaken but the spatial information is of limited resolution. New approaches, such as the reflection micro-Laue geometry, enables probing 3D strain elastic strain tensors (i.e. stress) stored within individual grains using a fine focused white beam and a differential aperture to distinguish the depth of different contributions to the observed patterns [18,19]. While this technique is clearly an exciting innovation, sampling a large number of grains with sufficient spatial information in order to address fatigue deformation problem in polycrystalline materials remains a challenge.

One recent development used high spatial resolution (sub-micro) digital image correlation (HR-DIC) technique to correlate microstructure with surface plastic strain distribution during the fatigue crack formation process [20]. However, typically HR-DIC only captures in-plane total strains, and cannot easily track the driving stresses, out of plane strain and residual deformation such as lattice curvature and residual stress. Many of these studies have been largely qualitative in nature [21] and complement analysis of out of plane slip through slip trace analysis [22].

In contrast, the residual elastic strain state is now accessible with the EBSD technique. Wilkinson et al. in 2006 [23] developed image cross-correlation based high resolution EBSD technique by measuring the shifts of test EBSD patterns with respect to a reference EBSD pattern within a grain. This technique significantly improved angular resolution of the conventional EBSD technique and provided full 3D elastic strain and rotation tensors (with appropriate assumptions). When combined with the existing advantages of EBSD including fine spatial resolution (~40 nm) [24], potential to scan large areas, easy access to complementary SEM imaging modes (e.g. electron channelling contrast image (ECCI) [25] and cathodoluminescence (CL) [26]), and the improvement in angular resolution makes HR-EBSD a powerful technique to study fatigue deformation processes in polycrystalline materials. Considerable development of the method has been made such that the analysis generates rather comprehensive deformation information such as intragranular residual stresses (type III) [27], geometrically necessary dislocation (GND) density [28] and total dislocation density [29]. These quantitative measurements can be readily combined with the microstructural information more commonly available from ‘conventional’ EBSD such as grain orientations, phase distributions, grain size, proximity to grain boundaries and triple junctions to reveal microstructurally sensitive trends.

The aim of this study is to systematically characterise the distribution and evolution of residual stresses, GND density and total dislocation density in fcc polycrystalline copper samples at the early stages of tension–tension fatigue using the HR-EBSD technique. Our study combines qualitative visual inspections of the residual stress and GND density maps and the patterns formed with respect to microstructural features along with quantitative analysis of statistical distribution and development with increasing number of cycles. Furthermore, samples subjected to cyclic and monotonic loading up to approximately the same strain will be compared.

2. Experimental and analytical methods

Four dog-bone-shaped OFHC (99.95% purity) copper specimens were milled from a 2 mm thick copper sheet. These specimens were progressively ground and polished to a 1 μm finish. Colloidal silica was then used to give a final mechanical and chemical polish. These polished specimens were annealed at 550 °C for 15 min and furnace cooled to room temperature to reduce residual cold work from the manufacturing process. Finally electrolytic-polishing in 85% phosphoric acid solution was utilised to remove the oxidation layers and contamination formed on the free surface to give optimised results for EBSD measurements.

A preliminary EBSD map (>1000 grains) of the annealed copper was collected to reveal the general microstructure morphology and orientation distribution as shown in Fig. 1(a). The average grain size is determined as ~10.3 μm as shown in Fig. 1(b) and there is a relatively weak texture existing in the annealed samples as indicated in Fig. 1(c).

Stress controlled fatigue tests with a stress range of ~150 MPa (16.6–166 MPa) at load ratio of 0.1 were performed at a frequency of 1 Hz on these specimens to increasing number of cycles (0 cycle, 2 cycles, 200 cycles and 2000 cycles) as illustrated in Fig. 2(a). Interpolation of life time data presented by Murphy [30] indicates that these loading conditions should correspond to fatigue lifetimes of $\sim 10^6$ cycles. The 0 cycle sample provides a reference annealed state and 2 cycle sample shows the initial deformation state at the start of fatigue deformation process. Significant cyclic hardening has been imposed onto