3D Laue microdiffraction: characterization of dislocation structures at grain boundaries during fatigue cycling

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Abstract

Fatigue damage is of outmost importance for our daily lives, as it limits the lifetime of almost all engineering components. Most of the knowledge on fatigue damage prior to crack initiation was obtained by transmission electron microscopy (TEM), which effectively images microstructural features such as dislocation cells and cell walls. However, TEM is limited to thin samples and, therefore, cannot investigate damage processes occurring in buried grains and grain boundaries. Laue microdiffraction (μLaue) experiments can then be used to study fatigue damage (1). However, due to the high density of stored dislocations next to a grain boundary during cyclic loading, this 2D method was not suitable for bi-crystals. To deconvolute volume integrated signals in Laue microdiffraction, an additional aperture was added to an existing μLaue beamline, which extends the μLaue technique to "Differential Aperture X-Ray Microscopy" (DAXM) (2,3), allowing for 3D subvolumetric reconstruction. Using this approach, the local crystallographic phase, orientation, and the full elastic strain tensor are determined with a three-dimensional resolution below $1\mu m^3$ voxel.

The experiments were conducted at the BM32 beamline of the European Synchrotron (ESRF) during fatigue cycling of copper bi-crystal microcantilevers ($5x5x25\mu m^3$) prepared by focused ion beam (FIB) machining. Grain boundaries were selected based on known properties, such as penetrability, particularly regarding slip transfer systems. Through the analysis of the local deviatoric strain tensor and local orientation (KAM), we observed and quantified the unexpected cyclic storage and annihilation of dislocations at an impenetrable grain boundary during low-cycle fatigue loading (up to 10 cycles). For comparison, a penetrable grain boundary was also studied in the same way. This grain boundary is expected to show continuous slip irreversibility and requires a long time (more than 10 cycles) before a steady state of the dislocation structure is reached. The combined DAXM - in situ deformation setup (3) offers unique possibilities to study the influence of specific grain boundaries on cyclic slip of dislocations and the eventual subsequent formation of dislocation structures during fatigue.

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