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On the reversibility of dislocation slip during small scale low cycle fatigue

C. Kirchlechner,[a,b,*] P.J. Imrich,[c,1] W. Liegl,[c,2] J. Pörnbacher,[b,1] J.-S. Micha,[d,e,3] O. Ulrich[d,e,3] and C. Motz[f,4]

[a] *Max Planck Institut für Eisenforschung, Max-Planck-Straße 1, 40237 Düsseldorf, Germany*
[b] *Department of Materials Physics, Montanuniversität Leoben, Jahnstraße 12, A-8700 Leoben, Austria*
[c] *Erich Schmid Institute of Materials Science, Austrian Academy of Sciences, Leoben, Jahnstraße 12, A-8700 Leoben, Austria*
[d] *CEA-Grenoble/Institut Nanosciences et Cryogénie, BP 220, 38043 Grenoble Cedex 9, France*
[e] *CRG-IF BM32 at ESRF, BP 220, 38043 Grenoble Cedex 9, France*
[f] *Department of Materials Science, Universität des Saarlandes, Postfach 15 11 50, D-66041 Saarbrücken, Germany*

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Abstract—The evolution of low cycle fatigue damage in copper is studied by *in situ* micro Laue diffraction. Free standing single crystalline micro-cantilevers with a cross-section of $10 \times 10\ \mu m^2$ were loaded in displacement controlled mode with a surface strain amplitude up to 5%. The point to point misorientation and the diffraction peak width as a measure of geometrically necessary dislocation density was analyzed locally during deformation and globally after 0, 1/4, 1/2, 3/4, 1 and multiples up to a maximum of 22 full cycles. Two different behaviors were observed (i) samples geometrically suppressing cross-slip show a steady state deformation pattern with dislocations in a pile-up. (ii) The sample with cross-slip does not reach a steady state with dislocations accumulating at the neutral plane.

Keywords: Size effect; Synchrotron diffraction; Micromechanics; Copper; Fatigue

1. Introduction

Fatigue is the cumulative damage of materials caused by cyclic loading, often leading to fracture and catastrophic failure of components. During the last two centuries, numerous studies have focused on the responsible mechanisms and their quantitative description. Fatigue damage accumulates in metals when irreversible plastic flow – i.e. irreversible dislocation motion – occurs. Sources of such irreversible behavior can be manifold as recently reviewed by Mughrabi [1] and strongly depend on the microstructure of the underlying material.

The formation of characteristic dislocation patterns and the subsequent crack initiation and growth plays a central role in fatigue damage and is well documented in the literature [1,2]. In macroscopic sized, face centered cubic materials, dislocation veins, persistent slip bands (PSBs) or dislocation cells are typically formed [2]. Their microstructural size is in the order of a few micrometers. In this size regime the deformation behavior of metals generally starts to differ from bulk, as the availability of dislocation sources, the distribution of dislocation source sizes, the probability of dislocation–dislocation interactions and the influence of interfaces or free surface become dominating factors in the deformation mechanisms [3–6]. Below both critical length scales, the microstructural length scale associated with fatigue dislocation structures in bulk as well as the microstructural length scale responsible for size effects in strength, the fatigue properties and mechanisms are expected to change.

In the past, several studies on thin metal films on various substrates [7–9] demonstrated the size dependency of fatigue damage accumulation. Thereby, a significant increase of the required stress amplitude for damage initiation was found for a decrease in film thickness [7,9]. The formed dislocation patterns vary with grain size and film thickness as proposed in a damage mechanism map for copper [8]. Below 8 μm grain size the formation of PSBs is rare [10] but dislocation walls and cells structures still exist [8,11]. Underneath 3 μm film thickness the macroscopic dislocation patterns are not observed anymore [12]. In a transition regime from 1 to 3 μm tangled dislocations and diffuse, cell-like structures are found. Below 1 μm thickness dislocation patterns are

* Corresponding author at: Max Planck Institut für Eisenforschung, Max-Planck-Str. 1, 40237 Düsseldorf, Germany. Tel.: +49 211 6792 970; fax: +49 211 6792 360; e-mail addresses: kirchlechner@mpie.de; peter.j.imrich@gmail.com; Wolfgang.Liegl@bohler-forging.com; josef.poernbacher@stud.unileoben.ac.at; micha@esrf.fr; ulrich@esrf.fr; motz@matsci.uni-sb.de

[1] Tel.: +43 3842 804 112.
[2] Tel.: +43 3862 20 7418.
[3] Tel.: +33 476 88 24 20.
[4] Tel.: +49 681 302 5108.

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Table 1. Summary of the tested samples.

	Width [μm]	Height [μm]	Bending length l_b [μm]	Surface stress [MPa]	Cycles [1]	Mesh scans [1]	Meshes at cycle	Laue patterns [1]
A	10.33	9.71	50.2	192	4	6	0, 1/4, 1/2, 3/4, 1, 2	8204
B	13.84	11.58	46.1	179	3	13	0, 1/4 + N, 1/2 + N, 3/4 + N, 1 . . . 3	12604
C	13.32	10.25	62.4	173	12	6	0, 2, 4, 6, 9, 12	10514
D	14.74	8.93	40.1	174	8	7	0, 1/4, 1/2, 1, 3, 7, 10	15244
E	10.94	10.56	32.6	184	2	2	0, 2	7984
F	9.90	11.67	45.3	141	22	7	0, 1, 2, 4, 10, 20, 22	2366

entirely replaced by single dislocations [8,11]. The aforementioned studies – except [10] – are all thin film systems still attached to their substrates, and thus, with constraints on one face. This complicates data interpretation and might cause extra effects as also stated in these publications.

The first micro-fatigue experiment on single crystalline copper bending beams was performed by Kiener and co-workers [13]. Their *in situ* SEM experiments were accompanied by discrete dislocation dynamics (DDD) simulations on similarly oriented samples. The DDD experiments showed the formation of distinct dislocation pile-ups at the neutral fiber during loading, with subsequent reversible backflow of the dislocations. Even though most of the dislocation pile-up dissolved in the straight beam, some dislocations were left behind leading to an increase of dislocation density, as independently shown by Demir and Raabe [14] with post mortem electron backscatter diffraction (EBSD). Unfortunately, these DDD simulations cannot easily be extended to high cycle numbers and larger crystals, and thus the formation of dislocation patterns in this size regime remains unknown.

Methods for analyzing the dislocation structure in micron sized bending cantilevers *in situ* are rare. X-ray microdiffraction Laue experiments (μLaue) can be used to measure sample orientation, deviatoric strain and density of geometrically necessary dislocations (GNDs) within one recorded pattern [15–17]. The acquisition time needed to record a μLaue pattern at modern beamlines is continuously decreasing, reaching the order of 0.1 s at the moment, making real *in situ* experiments with high frame rate possible. Recently μLaue has been applied to study the defect structures in metallic micropillars [18,19] and tensile samples [20]. These experiments require dedicated deformation setups [21,22] which are able to deform micron sized samples without obstructing the primary and secondary X-ray beams.

The aim of this study is to apply *in situ* μLaue diffraction during cyclic loading of copper micro-cantilevers in the low cycle fatigue regime to investigate the accumulation and global patterning of dislocations in micron sized samples.

2. Experimental setup and data evaluation

2.1. Sample manufacture

Micro bending cantilevers were produced similar to the approach in [23]: copper rods with $1 \times 1 \times 20\ mm^3$ with a nominal 1 1 2 crystal direction parallel to the beam length axis were cut from a macroscopic single crystal using a diamond wire and then etched to a needle. The cone angle was in the order of 10° with a top radius of 5 μm. The etched needle was glued to a sample holder with a well-defined loading axis as described in the supporting online material of [24]. Subsequently, bending cantilevers with a nominal width and height given in Table 1 were produced at the end of the needles by focused ion beam milling (FIB) in a Zeiss 1540XB crossbeam microscope with a final polishing current of 500 pA.

2.2. In situ μLaue experiments

The mechanical tests were performed at the CRG-IF BM32 [25] of the European Synchrotron Radiation Facility (ESRF) with a user built *in situ* device as described in [22]. The piezo actuated, displacement controlled device is able to record tensile and compressive forces with 10 μN load resolution. Successive tensile and compressive loading steps normal to the cantilever were performed with constant displacement rates leading to cyclic bending of the beam. The sharp edge of a razor blade was FIB milled to produce two parallel knife edges separated by a roughly 25 μm sized slit to be used as counter body (see schematic in Fig. 1). This approach is comparable to the one used by Kiener and co-workers in [13] and does not require sample manipulation between the two loading directions, which favors the approach for *in situ* μLaue. However, as the gap in between the slotted counter body and the sample needs to be corrected subsequently, a slight force-step can be seen in the force–displacement curve (Fig. 2d). Furthermore, using the thin razor blade assures a taper-free and therefore parallel cut of the opposing knife edges.

As the used SSD [22] does not have any surface approach routine the first point of contact can only be analyzed with an accuracy of roughly 25 nm *in situ*.[5] Therefore, the displacement during unloading is subjected to an uncertainty which leads to not fully straightened bending beams in the unloaded state after back-bending. This uncertainty will further be denoted as "inaccurate backbending" of the cantilever. To assure a symmetric loading of the bending beam the displacement amplitude was increased as visible in Fig. 2a and d.

The beamline [25] is equipped with a 2D-CCD Detector for recording Laue patterns, an energy dispersive X-ray point-detector for fluorescence scans and an optical microscope for the coarse sample alignment. The synchrotron beam is focused by a set of Kirkpatrick–Baez mirrors to 700 nm full width at half maximum (FWHM) in horizontal and 1200 nm in vertical direction. The photon energy varies from 5 keV to 27 keV. Due to the high penetration depth of the X-rays with respect to the sample dimensions the

[5] The point of contact can be measured during post mortem data analysis but the same is not possible online during the experiment at the full accuracy provided by the machine.