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# Fatigue damage in copper polycrystals subjected to ultrahigh-cycle fatigue below the PSB threshold

Anja Weidner<sup>1,a</sup>, Dorothea Amberger<sup>2,b</sup>, Florian Pyczak<sup>2,3c</sup>, Bernd Schönbauer<sup>4,d</sup>, Stefanie Stanzl-Tschegg<sup>4,e</sup> and Hael Mughrabi<sup>2,f</sup>

 <sup>1</sup>Technische Universität Dresden, Institut für Strukturphysik, 01062 Dresden, Germany
<sup>2</sup>Universität Erlangen-Nürnberg, Department of Materials Science and Engineering, General Materials Properties, Martensstr. 5, 91058 Erlangen, Germany
<sup>3</sup>now at: GKSS-Forschungszentrum GmbH, Max-Planckstr. 1, 21502 Geesthacht, Germany
<sup>4</sup>Universität für Bodenkultur, Department für Materialwissenschaften und Prozesstechnik, Peter Jordanstr. 82, 1190 Vienna, Austria

<sup>a</sup>aweidner@physik.tu-dresden.de, <sup>b</sup>dorothea.amberger@ww.uni-erlangen.de, <sup>c</sup>Florian.Pyczak@gkss.de, <sup>d</sup>bernd.schoenbauer@boku.ac.at, <sup>e</sup>stefanie.tschegg@boku.ac.at, <sup>f</sup>mughrabi@ww.uni-erlangen.de

Corrresponding Author:	Hael Mughrabi, <u>mughrabi@ww.uni-erlangen.de</u> ,
	Tel. +49 9131 8527482, Fax +49 9131 8527504

## Abstract

In High-Cycle Fatigue (HCF) of materials like copper, the most common failure modes originate from cyclic strain localization in persistent slip bands (PSBs). The latter form only when the loading amplitudes exceed the PSB thresholds. In contrast to this well-known HCF behaviour, it is shown in the present study that fatigue damage (cyclic strain localization, surface roughening, stage I crack initiation) develops even at loading amplitudes well below the PSB threshold in copper subjected to UltraHigh-Cycle Fatigue (UHCF). These findings are attributed to the accumulation of very small cyclic slip irreversibilities over very large numbers of cycles (>  $10^{10}$ ).

**Keywords**: UltraHigh-Cycle Fatigue (UHCF), fatigue thresholds, dislocation microstructure, cyclic strain localization, surface roughness, fatigue crack initiation.

### **1. Introduction**

There is currently an increased interest in understanding the specific damage and failure mechanisms which occur in the UltraHigh-Cycle Fatigue (UHCF) or Very High Cycle Fatigue (VHCF) range above about  $10^8$  to  $10^9$  cycles. In this work, as in previous related publications by one

of the authors [2, 3, 4], the term UltraHigh-Cycle Fatigue (UHCF) will be used. Most current studies on the UHCF behaviour have focused on high-strength materials containing microstructural heterogeneities, in which internal fatigue failure frequently occurs in the form of so-called "fisheye" fracture, which is initiated at internal heterogeneities such as inclusions, compare [1]. In contrast, the present work is part of an experimental study of the UHCF behaviour of pure ductile single-phase face-centred cubic (fcc) materials such as copper. In these materials, the most common High-Cycle Fatigue (HCF) failure modes originate from cyclic strain localization in persistent slip bands (PSBs). The general belief is that a necessary prerequisite for cyclic strain localization in PSBs is that the so-called PSB threshold amplitudes [5] must be exceeded. However, it had been postulated in earlier work that, even at very low amplitudes, cyclic slip still retains a small but nonnegligible irreversible component which, accumulated in a random fashion over a very large number of cycles in the UHCF regime, can lead to surface roughening (and irreversible changes of the dislocation substructure) and, ultimately, perhaps to PSB formation at the sites of local stress concentration and fatigue crack initiation. In that picture, fatigue damage can be expected to develop in the UHCF regime even below the PSB threshold [5], as illustrated in Fig. 1 ( $\sigma$ : stress, specimen axis vertical).

#### insert: Figure 1

In order to test this hypothesis, the UHCF behaviour of commercial purity copper polycrystals that had been ultrasonically fatigued up to more than  $10^{10}$  cycles had already been investigated quite thoroughly. In this earlier work, a detailed study of the surface features was conducted by scanning electron microscopy (SEM) and atomic force microscopy (AFM). The most important results of this work have been published [6, 7, 8] and are summarized as follows:

- The "traditional" axial PSB threshold stress amplitude was found to be ca. 63 MPa [6, 7, 8], with a corresponding axial plastic strain amplitude threshold of  $6.1 \times 10^{-6}$ . As will be discussed later, these values differ markedly from the threshold values found at conventional frequencies, namely ca. 56 MPa and  $2.5 \times 10^{-5}$  [9].
- Well below the PSB threshold, marked cyclic slip localization occurred in intense slip bands after very large numbers of cycles and intensified with increasing numbers of cycles [6, 7].
- The slip bands were shown to be persistent [8] in the sense that, when the surface was electropolished after fatigue, they reappeared at the same sites on the surface when fatigue

was resumed. Hence, in analogy to [10], they are considered as "persistent" slip bands (PSBs).

- At the sites of emerging PSBs at the surface, extrusions and intrusion-like deepenings (microcracks) formed [6, 7, 8].
- The observations showed clearly that the magnitude of the PSB threshold (and the fatigue limit) is not constant but depends on the number of cycles, being the lower the higher the number of cycles [6, 7, 8], in accord with the predictions of the model described above [2, 3, 4].

The present work focuses on the microstructural analysis of the fatigue-induced features and damage at the surface and in the bulk, as studied with SEM and the focused ion beam (FIB) technique. The dislocation microstructures were analyzed in surface grains and in interior grains by the electron channeling contrast (ECC) technique in the SEM [11]. These studies provided convincing evidence that *different kinds of fatigue damage occurred in the UHCF regime below the PSB threshold*. In the following, these recent observations will be presented and discussed.

#### 2. Experimental

A polycrystalline rod of commercial purity copper (mean grain size: ca.  $60 \ \mu$ m) of 7 mm diameter and 80 mm in length was fatigued ultrasonically at a frequency of 19 kHz with pulse-pause sequences, as described in more detail previously [6, 7, 8]. The axial stress amplitude at the location of highest stress was ca. 61.5 MPa. Fatigue loading was interrupted repeatedly in order to perform surface studies by scanning electron microscopy (SEM) and atomic force microscopy (AFM) after different numbers of cycles [6, 7]. All studies reported here were performed on that specimen after it had been fatigued for a total of  $1.59 \times 10^{10}$  cycles. For the following, it is important to note that the distribution of the axial stress amplitude along the specimen was sinusoidal with the site of maximum stress amplitude located at a distance of 48 mm from the free end and 32 mm from the clamped end of the rod. Hence, by studying different sections of the specimen at defined distances from the position of maximum stress, it was possible to investigate on one and the same specimen locations that had experienced different stress amplitudes (ranging from 0 to 61.5 MPa).

The studies with the FIB technique were performed on a Zeiss CrossBeam 1540 ESB instrument. In these studies, the following procedure was adopted. Almost all surface grains of the fatigued rod

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specimen were covered more or less densely with the traces of a large number of "PSBs". For the FIB studies, grains were selected in which the slip band traces lay roughly perpendicular to the direction of the rod (stress) axis. In that case, it could be assumed that the plane of the PSBs would lie approximately under 45° to the direction of the stress axis and that the active Burgers vector **b** would lie roughly in the plane perpendicular to the PSB traces (and in the PSB planes). Next, a thin platinum layer was deposited on a small area covering the PSB traces. Then, the FIB micromachining technique was used to cut by ion sputtering a plane perpendicular to the PSB traces through the platinum layer into the copper material. This plane was then studied in the scanning mode of the FIB. The surface topography of the fatigued specimen was clearly recognizable as a sharp line delineating the interface between the platinum layer and the copper. Then the whole rod was electrocoated with a 0.5 mm thick copper layer to protect the surface and was then cut parallel to the axis into two halves. After chemical and electrolytic polishing of the cut plane, the dislocation microstructures were studied by ECC/SEM at different sites near and further away from the surface in a Zeiss Ultra55 FESEM (field emission scanning electron microscope), equipped with an In-lens-SE-detector (SE: secondary electron) and an angular selective 4-quadrant backscatter electron (BSE) detector. The original specimen surface was easily recognizable as the borderline between the specimen and the electrocoating.

#### **3.** Observations and discussion

#### 3.1 FIB observations of surface roughness and stage I (shear) cracks

Figures 2 and 3 show examples of SEM micrographs obtained on the ion-machined surface that was approximately perpendicular to the surface traces of the PSBs. Figure 2 was taken at a surface site at a distance from the location of maximum stress where the local stress amplitude had been ca. 57 MPa, i.e. about 6 MPa below the "traditional" PSB threshold stress amplitude of ca. 63 MPa, which had been determined after 10<sup>6</sup> cycles in ultrasonic fatigue tests [6,7.8], thus being higher than the PSB threshold stress of ca. 56 MPa at conventional frequencies [9]. In Fig. 2, the original PSB surface markings and the deposited platinum layer are clearly visible. In addition, the following features are noteworthy. At the interface between the platinum coating and the copper specimen, there is a marked surface roughness profile which is more clearly recognizable at higher magnifications. Then there is a family of roughly parallel cracks, aligned under an angle of about 45° to the stress axis. These cracks are regarded as mode II stage I shear cracks. Fig. 3 was taken at

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higher magnification at the location of maximum stress amplitude (61.5 MPa), i.e. ca. 1.5 MPa below the PSB threshold. Here, the surface roughness can be recognized much more clearly in addition to the stage I shear cracks. It can be seen that some cracks have been initiated at stress raisers in the surface roughness.

#### insert: Figures 2 and 3

The surface roughness lends itself to a semi-quantitative evaluation as described below. If it is assumed that the roughness profile has been generated by random slip, it can be characterized by the root mean square displacement (rms), measured parallel to the Burgers vector **b** [6, 7, 8]:

$$\sqrt{\langle \mathbf{x}^2 \rangle} = K \sqrt{4 N \gamma_{\text{pl,loc}} p b h} = (rms)$$
(1)

Here, N is the number of cycles,  $\gamma_{pl,loc}$  is the local shear strain amplitude, b is the modulus of the Burgers vector and p is the so-called irreversibility of slip which is defined as the fraction of cyclic slip (0 ) that is irreversible [2, 3, 4, 5]. The quantity*h*has two possible meanings. When oneconsiders the root mean square displacement between neighbouring atomic slip planes, then h is the glide plane spacing and the constant K in the equation is equal to 1. When one refers, however, to the peak-to-valley roughness, then h is the length along which the peak-to-valley roughness is measured, and the constant K is approximately equal to 1.2 [12]. In the present context, this latter case is of interest. From Fig. 3, a peak-to-valley roughness of ca. 150 nm, measured parallel to b (b =  $2.5 \times 10^{-10}$  m) over a distance h  $\approx 2 \mu$ m, can be evaluated. The local plastic shear strain amplitude can be written as  $\gamma_{pl,loc} = M \times \Delta \varepsilon_{pl,loc}/2$ , where M is an orientation factor (taken as the Sachs factor = 2.24) and  $\Delta \varepsilon_{pl,loc}/2$  the local axial plastic strain amplitude. The value of  $\Delta \varepsilon_{pl,loc}/2$  is taken to be the PSB threshold value of the axial plastic strain amplitude, as measured at ultrasonic frequency [7,8], i.e.  $\Delta \varepsilon_{pl,loc}/2 = 6.1 \times 10^{-6}$ . Then, one can obtain from Eq. 1 an estimate for the cyclic slip irreversibility p which is the only unknown quantity in Eq. 1 and which is not well known and generally difficult to assess. Inserting all quantities, one obtains  $p \approx 0.000036$  which is very small, as expected. However, for the present very large number of cycles N in the UHCF regime (i.e.  $N = 1.59 \times 10^{10}$ cycles), this small irreversibility gives rise to a substantial cumulative irreversible plastic shear strain  $\gamma_{pl,irr,cum} = 4N \cdot \gamma_{pl,loc} \cdot p \approx 31.3 !!$  The microstructural origin of cyclic slip irreversibility and the implications in the present case will be discussed in a later section.

#### 3.2 ECC/SEM observations of dislocation patterns and fatigue damage

Figures 4a and 4b are examples of the ECC/SEM imaging of a surface site at a distance from the location of maximum axial stress amplitude (local stress amplitude ca. 54 MPa, i.e. about 9 MPa below PSB threshold). Figure 4a, taken with the In-Lens-SE detector, shows particularly clearly the original specimen surface as the interface between the electrocoating (at the top) and the specimen, showing a marked surface roughening and, in addition, several sites of stage I crack initiation in the valleys of the roughness profile. These findings confirm the FIB observations and substantiate the conclusion that surface roughening and stage I crack initiation occurred at stress amplitudes well below the PSB threshold. Figure 4b, taken with the BSE detector, does not show the features of surface roughening as clearly as Figure 4 a, but instead provides complementary information regarding some details about the dislocation microstructures, showing that cyclic slip occurred in a localized fashion in slip lamellae aligned close to 45° with respect to the stress axis.

#### insert Figures 4 and 5

Similar dislocation patterns, indicative of localized deformation in slip lamellae, aligned very roughly under an angle of 45° to the stress axis, were also observed in interior grains, as shown in Fig. 5, both at the location of maximum stress (Fig. 5a) and also in areas of significantly smaller stress amplitude (Fig. 5b). Figure 6a shows another example of such localized slip lamellae in an area of even lower local stress amplitude (ca. 48 MPa, i.e. ca. 15 MPa below the PSB threshold). The dislocation microstructure in the slip lamellae observed was mainly an elongated cell structure. Sometimes slip lamellae with ladder-like dislocation patterns, looking like remnants of PSBs with the classical ladder structure, next to vein-like dislocation patches, were also found in such areas of lower stress amplitude. Figure 6b shows an example (ca. 54 MPa, i.e. ca. 9 MPa below the PSB threshold). In regions of lower stress amplitude levels such as in Fig. 6a, the surface roughening was much less pronounced than in areas of higher stress amplitude. Figure 7 shows an example of a surprising observation in an interior grain at the location of maximum stress amplitude, showing clearly what looks like a family of microcracks, some microns long, lying roughly under an angle of 45° to the stress axis, looking very much like "interior" stage I shear microcracks. Similar

"microcracks" were also found in interior grains in areas of much lower stress amplitude but much less frequently.

#### insert: Figures 6 and 7

Finally, Fig. 8 shows another surprising observation made in interior grains. Quite commonly, the grain boundaries were heavily fragmented, indicative of grain boundary displacements up to about one micron. While these displacements look irregular, they seem to be spatially correlated to the slip lamellae at the same time.

insert: Figure 8

#### 4. Concluding Discussion of Main Features Observed

#### 4.1 Evolution of dislocation microstructure and fatigue damage

In agreement with the earlier results[6, 7, 8], it is now clear that, after a sufficiently large number of cycles in the UHCF range, fatigue damage develops at loading amplitudes below the traditional PSB threshold amplitudes. This result had already been anticipated by Hessler et al. [13]. In another earlier study, Polák and Vašek [14] had observed localized cyclic slip in copper fatigued below the fatigue limit. In view of the fact that the fatigue tests were performed at ultrasonic frequency, the frequency dependence of the PSB threshold amplitudes must be taken into account. The damage observed in the present study occurs in the form of cyclic strain localization, surface roughening and stage I shear "microcrack" initiation at the surface and at sites where the loading amplitude was maximal and decreased markedly at sites of lower loading amplitudes and further away from the surface. The lamellae of localized cyclic slip differ noticeably in appearance from the dislocation patterns in the form of PSBs with the ladder structure embedded in a matrix structure of veins which are observed at conventional frequencies (and numbers of cycles) in cyclic saturation in HCF. The fragmentation of the grain boundaries is a phenomenon that has not been encountered before. In the following, details of the behaviour observed will be discussed.

#### 4.2 Frequency dependence of the PSB threshold amplitudes

As noted before, the values of the PSB threshold amplitudes of stress and plastic strain, measured at ultrasonic frequency, differ markedly from the corresponding values at conventional frequency that

had been reported earlier [7,8]. According to two of the co-authors of this study [15], it seems possible that these discrepancies arise, at least in part, from the different forms of testing and measurement, see [15] for details. On the other hand, an explanation of the differences observed can also be offered in terms of the effect of frequency (via the strain rate) on the dislocation behaviour in cyclic deformation [16]. Thus, the higher value of the ultrasonically measured axial PSB stress threshold of 63 MPa, compared to the value of ca. 56 MPa at lower conventional frequencies [9], can be attributed to the higher strain rate at ultrasonic frequency and can be explained semiquantitatively by considering the strain-rate sensitivity of cyclically deformed copper. However, it is not immediately obvious, why the axial plastic strain PSB threshold amplitude (6.1×10<sup>-6)</sup> at ultrasonic frequency should be lower by a factor of about 4 than the corresponding value of  $2.5 \times 10^{-10}$ <sup>5</sup> at conventional frequency. A tentative explanation is offered as follows [16]. A necessary prerequisite for the formation of PSBs in cyclically hardened fcc metals is that the initially formed dipolar vein structure becomes unstable and then breaks down on a local scale. Thereupon, the glide to-and-fro and, in a dynamic process of dislocation annhibilation and dislocations multiplication, finally settle down in the form of the well-known wall ladder structure of PSBs. This process bears some resemblance to the destabilization of the dipolar loop patches in the Neumanntype strain bursts [17]. Here, it is of interest to note that Neumann-type strain bursts had been observed initially at 50 Hz. In later work, it was shown that, at lower frequencies like ca. 1 Hz, the occurrence of strain bursts was largely absent [18]. These results suggest that the *local* destabilization of the dipolar vein structure will only spread, if the sequence of the destabilizing dislocation processes is so fast that any locally triggered instability will not have enough time to "heal out" before the next destabilizing glide event. In other words, the condition for spreading of the instability would be that the "decay time"  $\tau$  of the instability must be (much) longer than the time interval  $\Delta t \approx 1/v$  (v: frequency of fatigue test) between destabilizing glide events. This condition, i. e.  $\Delta t \approx 1/v \ll \tau$ , is much more easily fulfilled at higher frequency than at lower frequency. It hence appears very plausible that the condition for the spreading of a local instability, as a prerequisite for PSB formation, is more probable at higher (ultrasonic) than at lower (conventional) frequency and can hence occur already at a lower plastic strain amplitude at ultrasonic than at conventional frequencies.

#### 4.3 Lamellae of localized shear deformation

These lamellae are reminiscent of "old" PSBs in which the ladder structure has been transformed into a cell structure due to the accumulated effect of slip irreversibilities and slowly increasing secondary slip, as shown earlier for copper single crystals that acquired a very long life during fatigue in vacuum [19]. This shows clearly that, even in so-called cyclic "saturation", the small fraction of slip that is irreversible, which is caused, for example, by mutual dislocation annihilations [5], leads to a slow persistent modification of the dislocation distribution. In the present case, it is proposed that a classical vein matrix structure probably forms first but then continues to harden very slowly so that some form of PSBs would develop even below the PSB threshold. The fact that in regions of lower local stress amplitude remnants of a wall structure were sometimes seen suggests that, at an earlier stage, the originally formed PSBs may have had the ladder structure. These PSBs would then also harden very slowly and would eventually be replaced by new ones which would suffer the same fate, etc. [19]. In this manner, provided that cyclic deformation is performed to very large numbers of cycles, most of the grains that are favourably orientated (for slip on a "dominant" glide plane) would gradually become filled with the lamellae of localized slip observed in this study. It is assumed that, because of the different ages and prehistories of these lamellae, they would differ in their glide activities.

#### 4.4 Evolution of surface roughness, cyclic strain localization and crack initiation

Equation 1 is based on the assumption that surface roughness develops as a result of the irreversible component of random slip [2, 3, 4]. This statement holds only for the early stages of cyclic deformation, before severe strain localization sets in. Hence, the roughness seen could be partially due to random slip in the early stages and, to the other part, it could be a result of the localized slip with extrusion formation, etc. Crack initiation at the surface could be promoted by either form of roughness. However, it is important to note that lamellae of cyclic strain localization were observed not only in surface but also in interior grains. Hence, cyclic strain localization can be expected to develop in surface (and in other) grains even in the absence of the surface roughening proposed in the model [2, 3, 4] and would by itself lead to extrusions, etc., and contribute to fatigue crack initiation.

#### 4.5 Stage I shear microcracks

It is difficult to imagine that the "microcracks" that have been seen and that lay without exception roughly under 45° to the stress axis are anything else but stage I shear cracks. This assumption applies not only to those cracks that originated from stress raisers in the roughness profile but also to the "interior" stage I cracks, although it is not readily understood how such cracks could form inside bulk grains. It may have played a role that the copper investigated was of commercial purity. During cyclic deformation point defects, in particular vacancies, are formed continuously. In copper, these vacancies can migrate at room temperature. Hence, it is conceivable that in regions of high glide activity foreign atoms can diffuse and segregate to "potential crack sites" and promote "interior" crack initiation. To verify this hypothesis, more rigorous examination is necessary, for example by checking whether interior cracks form also in high-purity material.

Whether, after further cycling, the stage I cracks observed would grow and turn into mode I stage II cracks at a later stage, and perhaps, ultimately, lead to failure remains an open question. As elaborated by two of the co-authors [15], it can be argued, based on fracture mechanics measurements of threshold stress intensities for crack growth, that the stage I cracks are nonpropagating cracks. These authors showed that when the threshold stress intensities for crack growth are plotted in a Kitagawa diagram and correlated with the ultrasonically measured endurance limit of 93 MPa, a critical "short crack" length of 0.34 mm is obtained which is considerably larger than the stage I crack lengths of ca. 10 µm observed. On the other hand, this raises the question why the stage I cracks did grow at all to a length of ca. 10 µm. In addition, it is noted that the fracture mechanics considerations mentioned above were based on mode I stage II crack growth, whereas in the present case, strictly speaking, these considerations should have been made for mode II stage I crack growth. Finally, it is interesting that Petit and co-workers [20, 21] showed that, in single crystals, stage I cracks can grow considerably faster than stage II cracks, indicating that the threshold stress intensities for stage I crack growth (and hence the critical "short crack" lengths) can be considerably smaller than those for stage II crack growth. Hence, the question, whether the stage I cracks observed would be able to continue to grow at even higher numbers of cycles than N = $1.59 \times 10^{10}$ , cannot be answered conclusively before the questions raised above are clarified satisfactorily.

#### 4.6 Local displacements and fragmentation of grain boundaries

It has been observed that, at higher temperatures, grain boundaries undergo discrete displacements during each cycle of deformation [22, 23, 24]. The magnitude of these displacements depends on the character of the grain boundaries. The displacements are thermally activated and have been suggested to be driven by the driving force for grain coarsening [23, 24]. In the present case, one can hardly speak of high temperature fatigue. Nonetheless, vacancies are produced and can migrate. They can hence assist very small thermally activated displacements of the grain boundaries per cycle which, accumulated over the very large numbers of cycles to which the specimen has been exposed, could lead to measurable displacements. In the present case, the main driving force for the irregular grain boundary displacements which are correlated spatially with the locations of the slip lamellae is suggested to lie in the unequal dislocation fluxes on either side of the grain boundaries, caused by different orientation factors of the grains and different forms of slip localization within the grains.

#### 5. Summary

The present work, in combination with the earlier studies on the UHCF behaviour of polycrystalline copper at ultrasonic frequencies [6, 7, 8], confirms that, provided the number of cycles is large enough, *substantial fatigue damage does develop at loading amplitudes well below the PSB threshold* in the form of

- lamellae of localized cyclic shear [6, 7, 8], containing elongated dislocation cells, which probably have developed out of initially formed PSBs with the well-known wall ladder structure.
- a pronounced fatigue-induced surface roughening [6, 7], and
- initiation of stage I (mode II) shear cracks which, interestingly enough, were found not only at the surface but also in interior grains.
- The severity of all features of fatigue damage decreased with increasing distance from the surface and with decreasing local stress amplitude.

In addition, substantial local grain boundary displacements were noted. The fatigue damage observed is more complex than predicted by the earlier model [2, 3, 4]. In view of the fact that the fatigue limit lies above the PSB threshold [11] and since it could not be verified so far whether the stage I cracks formed are non-propagating cracks or not, it remains an open question whether the

fatigue damage observed can finally lead to fatigue failure. In the discussion of details of the results obtained in ultrasonic fatigue tests, the effect of the frequency must also be considered. In future work, the newly found microstructural features should be studied further as a function of numbers of cycles N, and the results should be implemented in a more refined model.

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# FIGURE CAPTIONS: Next Page

#### **Figure Captions**

Fig. 1: Schematic illustration of the different stages of development of fatigue damage as a result of surface roughening caused by accumulated irreversible slip. a) initial state, b) early stage of surface roughening and c) PSB formation at sites of local stress concentration at later stage. After [4].

Fig. 2: FIB micrograph, showing original surface slip bands, platinum coating and micro-machined plane roughly perpendicular to the plane of the slip bands with fatigueinduced surface roughness visible at the interface between copper specimen and platinum coating. Note the "family" of stage I shear cracks. About 6 MPa below PSB threshold. Specimen axis horizontal.

Fig. 3: FIB micrograph showing surface roughness at interface between platinum layer and fatigued copper specimen and stage I microracks. About 1.5 MPa below PSB threshold. Specimen axis horizontal.

Fig. 4: Fatigue-induced surface roughness and stage I crack initiation from a location where the local stress amplitude was about 57 MPa, i.e. ca. 6 MPa below the PSB threshold. a) image taken with In-Lens-SE detector. b) same area, backscattered electron SEM/ECC image. Specimen axis horizontal.

Fig. 5: ECC/SEM images of localized cyclic shear lamellae in interior grains. a) at the location of maximum stress amplitude (61.5 MPa, i.e. ca. 1.5 MPa below PSB threshold). b) away from the location of maximum stress amplitude (local stress amplitude about 54 MPa, i.e. ca. 9 MPa below PSB threshold). Specimen axis horizontal.

Fig. 6: ECC/SEM: Regions of localized cyclic shear deformation in slip lamellae. a) surface grain, ca. 15 MPa below PSB threshold. b) Interior grain, with ladder-like slip lamella, ca. 9 MPa below PSB threshold. Specimen axis horizontal.

Fig. 7: Stage I "microcracks" in interior grain, region of maximum stress amplitude (ca. 61.5MPa, i.e. ca. 1.5 MPa below PSB threshold). ECC/SEM (In-Lens-SE detector). Specimen axis horizontal.

Fig. 8: ECC/SEM image of fragmented grain boundary in interior grain in region of maximum stress amplitude (61.5 MPa, i.e. ca. 1.5 MPa below PSB threshold). Specimen axis horizontal.