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# 21st European Conference on Fracture, ECF21, 20-24 June 2016, Catania, Italy Microstructure-based study of the crack initiation mechanisms in pure copper under high cycle multiaxial fatigue loading conditions

Komlan Agbessi<sup>a</sup>, Nicolas Saintier<sup>a,\*</sup>, Thierry Palin-Luc<sup>a</sup>

<sup>a</sup>Arts et Metiers ParisTech, I2M, CNRS, Esplanade des Arts et Metiers, 33405 Talence Cedex, France

#### Abstract

This paper aims to contribute in understanding the fatigue crack initiation mechanisms in metallic materials under high cycle multiaxial fatigue loadings. It addresses proportional and non-proportional multiaxial loading conditions with the analysis and observation of the cyclic plasticity development (mainly persistent slip band) until crack initiation (especially short cracks) on a pure oxygen-free high conductivity (OFHC) polycristalline copper. Observation and analysis techniques are based mainly on optical microscopy and scanning electron microscopy (SEM). It has been observed that the plastic slip multiplicity in grains seems more important for multiaxial loadings at a stress level corresponding to the same median fatigue strength at 10<sup>6</sup> cycles of the material. A multiaxial loading conditions investigated, although most of the grains exhibits single slip activated, analysis of the preferential crack initiation sites and modes show a higher probability of intragranular microcrack initiation in the multiple slip grains (with more than two slip systems activated). Most multiple slip grains and higher probability of crack initiation in these grains were observed especially for non-proportional multiaxial loadings. Finally, the effects of the biaxiality ratio and the phase shift on the fatigue crack initiation was highlighted.

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Keywords: high cycle multiaxial fatigue; persistent slip band; cyclic plasticity; multiple slip; intragranular crack initiation

# 1. Introduction

Fatigue remains today one of the essential causes of mechanical components and structures failure in service under cyclic loading. It has been well understood for decades that two main steps characterize the fatigue failure which are the crack initiation and propagation. In high cycle fatigue (HCF) regime, a large part of life is spent in the crack initiation phase (more than 90 % of the total life, see Klesnil and Lukas (1992)). The fatigue life of the material depends primarily on this crack initiation phase (mainly stage I). In this regime, crack initiation is controlled by the formation and development of persistent slip bands (PSB) at the surface for pure metallic materials without internal defects.

<sup>\*</sup> Corresponding author. Tel.: +33 5 56 84 53 61. ; fax: +33 5 56 84 53 66.

*E-mail address:* nicolas.saintier@ensam.eu

PSB formation has been widely studied especially for face centrered cubic (FCC) materials (see the references for example Mughrabi (1978); Wang and Mughrabi (1978); Figueroa and Laird (1983); Basinski and Basinski (1992); Brown and Brown (2000); Trochidis et al. (2000)). These authors have shown that they are formed at the surface of the material, on the grains favorably oriented for plastic slip consecutive to the dislocations motion. Hence, the emergence of PSB is linked to the slip systems activation in the grain. The accumulation of irreversible cyclic plastic strain within these PSB structures leads to the fatigue microcracks initiation, as studied by Mughrabi (2009, 2013a,b). This irreversibility of plastic slip is the key cause of damage and later fatigue crack initiation. However, these studies are mostly undertaken under uniaxial loading and often in low cycle fatigue (see Villechaise et al. (2002); Marinelli et al. (2009)) where plastic strain amplitude is high; HCF and very high cycle fatigue regimes have to be more investigated especially under multiaxial loading conditions.

If irreversible shear strain is considered as the driving force for fatigue crack initiation, the presence of normal stress (or hydrostatic stress) on highly sheared material facets assists the growth of microcracks from stage I to II. When comparing crack initiation mechanisms under tension and torsion loading conditions, the effect of the absence of such an opening load in torsion is highlights. Basically, this is understood as possible explanation of the longer stage I characterized by the microcraks development in pure mode II under torsion when under tension mixed mode I and II is observed. The crack initiation mode can therefore be influenced by the loading type and especially by the non-proportionality in case of multiaxial loading conditions Agbessi (2013). In this way, it has already been shown the effect of the non-proportionality of loading on the plastic activity and the additional hardening that occurs in the material, as studied by Lamba and Sidebottom (1978a,b); Busso and Cailletaud (2005). Even if the efficiency of the critical plane based HCF multiaxial criteria under development for decades (see Fatemi and Shamsaei (2011)) is not questioned in this paper, the analyses of the accurate statistical analysis of microcracks initiation under multiaxial proportional and non proportional loading conditions can allow to discuss the main assumption based on one slip system activated per grain.

The work presented in this paper is focused on the analysis of cyclic plastic slip development and fatigue crack initiation modes under multiaxial proportional and non-proportional loading conditions. We focus mainly on microstructuraly short cracks or microcracks (crack at the scale of one or maximum two grains). A statistical analysis of plastic activity in grains leading to the identification of the preferential microcrack initiation sites under different multiaxial loading conditions is proposed and discussed in relation to the literature. The role of multiple slip in the fatigue cracks initiation is investigated based on an analysis of the proportion of cracked grains exhibiting single and multiple slip for all the studied loading conditions.

### 2. Experimental procedures

#### 2.1. Material and fatigue testing conditions

The studied material is an oxygen-free high conductivity (OFHC) polycrystalline pure copper (FCC structure, purity 99,995%) obtained by hot rolling. Before machining, the material was annealed in order to relax internal stresses (230°C for one hour). The microstructure is shown on Fig. 1a with equiaxed grains of 35  $\mu$ m mean size. The specimen geometry used for all the fatigue tests undertaken in this work is shown in Fig. 1b. Prior testing, specimens were mechanically polished then electro-polished to allow accurate observation of PSB using optical microscopy or scanning electron microscopy (SEM).

Fatigue tests were carried out at room temperature under load control using an electro-dynamic tension-torsion Bose fatigue testing machine at a loading frequency of 20 Hz. The loading ratio was  $R_{\sigma} = R_{\tau} = -1$  (stress ratio,  $R_{\sigma} = \sigma_{min}/\sigma_{max}$ ) for all tests. The normal and shear stress amplitudes corresponding to the median fatigue strength at 10<sup>6</sup> cycles were determined for each loading conditions investigated here (see Tab. 1). Three phase shift were investigated in case of combined loading conditions:  $\beta = \{0^\circ\}$  for proportional multiaxial loading conditions and  $\beta$ =  $\{45^\circ, 90^\circ\}$  for non proportional ones. Microplasticity development, slip activity and microcrack initiation analyses were carried out on unfailed specimens (i.e. with no visible macrocrack).

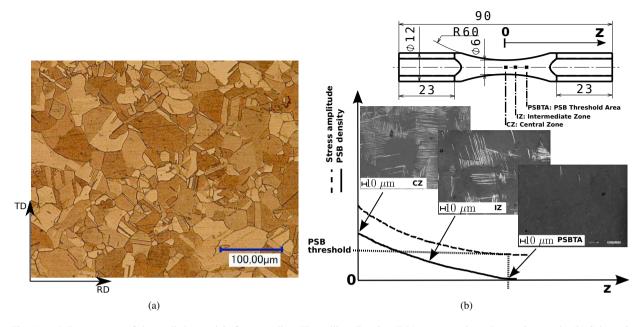


Fig. 1. (a) Microstructure of the studied material after annealing. The rolling direction (RD) corresponds to the specimen axis. (b) Schematic illustration of plasticity development at the specimen surface after  $10^6$  cycles under fully reversed torsion at the median fatigue strength at  $10^6$  cycles ( $\tau_a = 58$  MPa).

Table 1. Stress levels applied for fatigue tests (R = -1, f = 20 Hz) without macroscopic failure at 10<sup>6</sup> cycles.

Load cases	$\sigma_a$ (MPa)	$\tau_a$ (MPa)	$\sigma_a/ au_a$	$\beta$ (°)
Tension $(\sigma_a)$	85	-	-	-
Torsion $(\tau_a)$	-	58	-	-
Tension-torsion ( $\sigma_a/\tau_a = 2.0(0^\circ)$ )	68	34	2.0	0
Tension-torsion ( $\sigma_a/\tau_a = 0.5(0^\circ)$ )	24	48	0.5	0
Tension-torsion $(\sigma_a/\tau_a = 2.0(45^\circ))$	68	34	2.0	45
Tension-torsion ( $\sigma_a/\tau_a = 0.5(45^\circ)$ )	24	48	0.5	45
Tension-torsion ( $\sigma_a/\tau_a = 2.0(90^\circ)$ )	68	34	2.0	90
Tension-torsion ( $\sigma_a/\tau_a = 0.5(90^\circ)$ )	24	48	0.5	90

# 2.2. SEM observations

Specimens tested at 10<sup>6</sup> cycles have been analysed using SEM surface observations on three different zones as shown on Fig. 1b. Zone 1, named the PSB threshold area (PSBTA), corresponds to the threshold stress of PSB appearance at the surface of the cycled specimen. Zone 2, named the intermediate zone (IZ), corresponds to the development of PSB without microcrack initiation. And zone 3, named the central zone (CZ), corresponds to the development of PSB with possible microcrack initiation. Grains with single and multiple slip marks are both analysed in these zones for each loading conditions and correlated to the microcracks initiation.

# 3. Results and discussions

Due to the specimen geometry, the stress level varies along the longitudinal axis of the specimen and the degree of plasticity developing at the specimen surface depends on the location of the observed zone as shown on Fig. 1b. SEM observations of the specimen surface show that the plasticity development is most pronounced in the center of the specimen and slowly vanishes when observing away from the center. A statistical analysis carried out on the

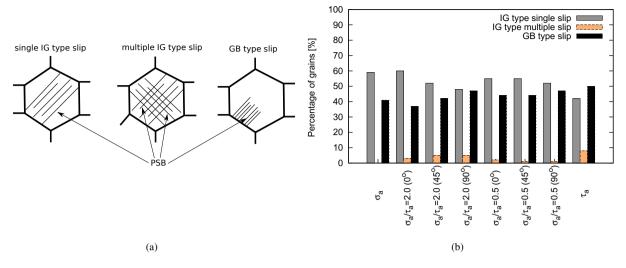


Fig. 2. (a) Schematic representation of the observed main plastic slip modes in the grains. (b) Percentage of activated grains in the intermediate zone (IZ), with single or multiple intragranular plastic slip (IG slip type) and at the grain boundary (GB slip type) for different applied loads. About 110 grains are considered for each loading condition.

PSB appearance modes in the PSBTA show that the proportion of PSB onset in the grain or at grain boundaries (Fig. 1a) are similar for each loading conditions (average of 60 % inside the grain and 40 % at the grain boundaries). No influence of the loading conditions has been observed concerning the PSB onset modes. Over a total of about 160 grains analysed in the PSBTA, an average of 20 activated grains (grain with at least one visible slip marking) are considered for each loading conditions.

#### 3.1. Microplasticity development

The microplasticity development on the tested specimen surface was analysed based on an average of 110 grains in the intermediate zone. In this area, there are two kind of PSB onset within the grain: (i) single slip (one type of PSB in the grain) and (ii) multiple slip (at least 2 PSB cross in the grain). These two configurations are shown schematically in Fig. 2a. Note that in the PSBTA (or transition zone), we do not distinguish multiple slip. It seems that multiple slip occurs in areas subjected to a stress amplitude higher than the PSB thresholds stress amplitudes for the considered loading condition. As the notion of single slip was associated only to the observed slip mark at the surface of the specimen, the corresponding slip directions cannot be characterized. Therefore, single slip activity is relative to one slip plane of {111} family activated (even if there is three possible slip directions).

The percentage of activated grains (grains with visible slip marking at their surface) having PSB at grain boundaries and the intragranular modes (cf. Fig. 2a) within the grain is shown in Fig. 2b. The trend is similar in the PSBTA. In uniaxial tension loading, the percentage of activated grains remains almost similar in the IZ and PSBTA. It has been observed under tension that there is no multiple slip at the tested stress levels corresponding to a lifetime of 10<sup>6</sup> cycles. For combined loadings, at same biaxiality ratio, the phase shift induced a slight increase of the proportion of grains with the PSB development contained at the grain boundaries. In general for all loads investigated, single slip remains predominant. The percentage of grains with multiple slip for multiaxial loadings remains low in the IZ ( $\leq 10\%$ ). The proportion of grains with multiple slip in the IZ is more important in simple torsion and combined tension-torsion with a biaxiality ratio  $\sigma_a/\tau_a = 2.0$ .

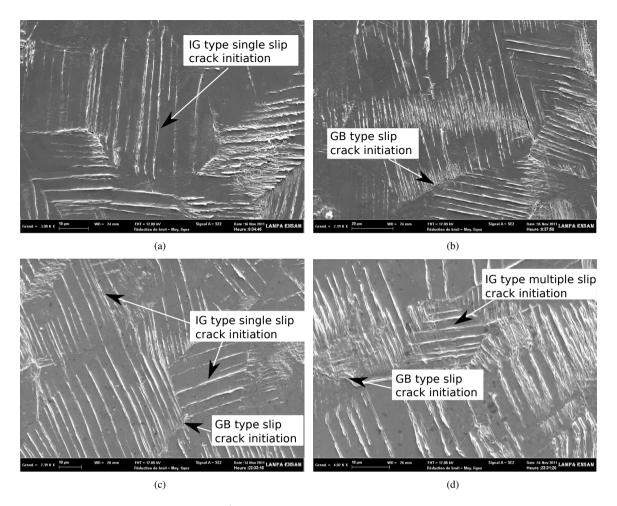


Fig. 3. SEM observation of the specimen after  $10^6$  cycles under (a,b) torsion loading condition ( $\tau_a = 58$  MPa) and (c,d) combined tension-torsion loading condition ( $\sigma_a/\tau_a = 2.0, \beta = 45^\circ, \sigma_a = 68$  MPa). Illustration of the different modes of fatigue microcrack initiation.

# 3.2. HCF microcracks initiation

Fatigue microcracks are statistically analysed in the central area shown on Fig. 1b. The chosen approach is to make a SEM high resolution micrograph on areas where we see microcracks and to count the different types observed. Microcracks initiation modes are classified into three main categories (illustrated, as example, on Fig 3a,b in case of torsion loading and Fig. 3c,d for combined tension-torsion with  $\sigma_a/\tau_a = 2.0$  and  $\beta = 45^{\circ}$ ):

- intragranular crack initiation in grains with single slip system activated (IG type single slip crack initiation).
- intragranular crack initiation in grains with multiple slip systems activated (IG type multiple slip crack initiation).
- intergranular crack initiation with PSB located at the grains boundaries (GB type slip crack initiation).

For all the investigated loading conditions, microcracks considered are about the size of one grain. The identification of the microcracks is relatively delicate and due to surface observation, it is not always unsure that the microcrack is developed across the grain (i.e. in the thickness, as it could have been observed by performing Focused Ion Beam measurements).

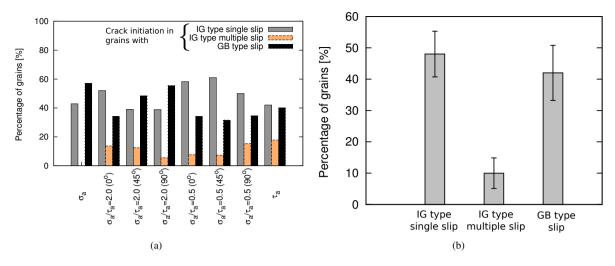


Fig. 4. (a) Representation of the proportion of intragranular crack initiation (in grains with one and at least two identified slip systems activated) and intergranular (at grain boundary). (b) Proportion of grains with intragranular crack initiation and intergranular averaged over all loading conditions. About 60 grains are considered from a total of 250 grains analyzed for each loading condition.

### 3.2.1. Statistical analysis of fatigue microcracks

The Fig. 4a represents the proportion of grains according to the different categories of crack initiation observed for each loading condition. In tension, we observe that cracks initiate more often at the grain boundaries (57 % of cases). Deformation incompatibilities at the grain boundaries seem to play a significant role in the development of damage under uniaxial tension in HCF regime. In the case of torsion loading, where PSB grow more homogeneously in several grains, the proportion of crack initiation at grain boundaries is 40 % (~ 15 points less compared to the tension loading case). Thus, under torsion, the plastic activity in the grain is most important than the one at the grain boundary. Intragranular crack initiation seems to be clearly promoted under torsion and combined tension-torsion  $\sigma_a/\tau_a = 0.5$  loading conditions.

It has to be noted that these observations were made for equivalent loads corresponding to the same fatigue life of  $10^6$  cycles. In the case of equivalent loads according to von Mises in cyclic tension and torsion (in LCF regime with a different lifetime), it was observed in the work of Doquet Doquet (1997) that intergranular cracks initiation is more prominent in torsion (about 45%) than in tension (about 26%). Indeed, under Von Mises equivalent tension and torsion loading conditions, it is not possible to have the same fatigue lifetime. The ratio between tension and torsion median fatigue limit (see table 1) differ from  $\sqrt{3}$ . These results highlight the striking difference of HCF crack initiation mechanisms depending on the considered lifetime and the stress equivalence chosen when comparing different applied loading conditions.

Regarding all the load types together, the majority of observed cracks are initiated within grains with single slip (~ 50%, cf. Fig. 4b). The crack initiation at grain boundaries represent about 40 %. A proportion of about 10% of grains is with at least two visible slip cross marking. The fatigue cracks initiation is mainly intragranular on OFHC pure copper studied. Relatively lower percentage of intragranular cracks initiation in the grains with multiple slip is observed. However, the effect of grain boundaries (including twin boundaries) remains important regarding the higher percentage of intergranular crack initiation observed in this study.

#### 3.2.2. Relation plasticity vs crack initiation

The PSB development modes in the IZ of the tested specimens shown in Fig. 2b was statistically compared to the different modes of fatigue cracks initiation in the CZ. It allows relationship identification between evolution of the proportion of activated grains with and without microcracks according to the microplasticity development observed and analysed previously. The proportion of IG type multiple slip grains located in the CZ, presenting microcracks increases for multiaxial loading conditions (cf. Fig. 4a). Regarding the low population, this indicates that the slip multiplicity in grain plays a significant role regarding the intragranular fatigue crack initiation.

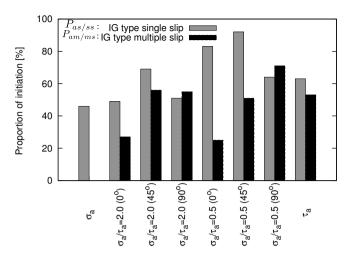


Fig. 5. Proportion of intragranular crack initiation reported to the percentage of activated grains with single and multiple slip.

Focusing only on intragranular crack initiation, we can also establish a link between the plastic activity in grain and the crack initiation modes. In order to analyse this relationship, the proportion of intragranular crack initiation is normalized by the percentage of observed grains with single and multiple slip. Let us introduce  $P_{ss}$  and  $P_{ms}$  the percentage of observed grains with single slip and multiple slip respectively. We define the proportion of intragranular crack initiation grains reported to the proportion of activated grains by:

$$P_{as/ss} = \frac{P_{as}}{P_{ss}}$$

$$P_{am/ms} = \frac{P_{am}}{P_{ms}}$$
(1)

where  $P_{as}$  and  $P_{am}$  are respectively the percentage of intragranular crack initiation on grain with single slip and multiple slip plastic activity.

 $P_{as/ss}$  and  $P_{am/ms}$  can be respectively defined as the number of grains with intragranular crack initiation among grains with single and multiple IG type slip. Their distribution is represented on Fig. 5. It can be seen that, in general, intergranular crack initiation in grains with single slip activated occurs moslty. However, the non-proportional multiaxial loadings and torsion (which is in fact a proportional multiaxial stress condition) have a high probability of crack initiation in activated grain with multiple slip. For the same biaxiality ratio, the out-of-phase loadings conditions induce an increases of +30% of the proportion of intragranular crack initiation on multiple slip grains. These grains exhibits clearly more than one slip system activated. Due to the high probability of crack initiation on these grains, slip multiplicity seems to be a key mechanism that can drive HCF strength of the material. It can be noticed that most of the critical plane multiaxial loading conditions, HCF critical plan criteria predictions can lead to relative important errors, as shown by previous studies of Froustey and Lasserre (1989); Papadopoulos et al. (1997); Morel et al. (2001); Wang and Yao (2004). One of the possible reason can be relative to the single slip hypothesis which may have to be reconsidered especially for such loading conditions.

#### 4. Conclusion

In this paper, the microplastic activity under proportional and non-proportional loading conditions was investigated at the surface of OFHC copper. It was shown that the PSB appearance mode (IG type slip and GB type slip) little depends on the load type. The slip multiplicity effect in the grains seems significant for multiaxial loading conditions. Indeed, it was observed that multiaxial loading induces additional slip multiplicity in the grains compared to the uniaxial loading. The statistical study of the microplasticity development highlighted the dominance of single slip in grains for all the studied loading conditions. The development of plasticity in the grains, is almost characterized by single slip activity for the OHFC polycristalline copper.

Based on the fatigue crack initiation modes (microstructural short cracks), the effects of biaxiality ratio and phase shift on crack initiation were statistically analysed. In uniaxial tension ( $\sigma_a/\tau_a = \infty$ ), we observed more crack initiation at grain boundaries (+15% compared to the torsion loading,  $\sigma_a/\tau_a = 0$ ). This is consistent with: (i) the fact that in tension, PSB distribution is very localized in a few grains (here mostly at grain boundaries or twin boundaries) and (ii) in torsion, PSB are distributed more homogeneous and in several grains. For combined tension-torsion loading conditions, the percentage of crack initiation at the grain boundaries increases with the non proportionality.

Finally, the multiaxial loads promote mostly intragranular crack initiation in grains with multiple slip. It has been found that the probability of cracks initiation in these grains is higher in the case of non-proportional multiaxial loading (about 55%). Accordingly, the multiple slip seems to play an important role on the intragranular fatigue cracks initiation. This is not taken into account in the formulation of most of the critical plane based multiaxial fatigue criteria in the literature and may explain the poor predictions of these criteria for non-proportional loading. The assumption of single slip per grain in the formulation of the critical plane multiaxial fatigue criteria is questioned for these loading conditions.

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