DEVELOPMENT OF CYCLIC SLIP BANDS IN UFG COPPER IN GIGACYCLE FATIGUE

Lucie Navrátilová[1,2)], Ludvík Kunz[2)], František Nový[3)], Rastislav Mintách[3)]*
[1)]Faculty of Mechanical Engineering, Institute of Material Science and Engineering, Brno University of Technology, Technická 2, 616 69 Brno, Czech Republic
[2)]Institute of Physics of Materials, Academy of Sciences of the Czech Republic, Žižkova 22, 616 62 Brno, Czech Republic
[3)]Faculty of Mechanical Engineering, University of Zilina, Univerzitná 1, 010 26 Žilina, Slovakia

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[*]Corresponding author: e-mail: lunavra@google.com, Tel.: +420 532 290 464, Academy of Sciences of the Czech Republic, Institute of Physics of Materials, Žižkova 22, 616 62 Brno, Czech Republic

Abstract
Ultrafine-grained (UFG) copper studied in this paper was produced by equal channel angular pressing (ECAP) method and tested in gigacycle fatigue region. In contrary to conventional grain size copper, the appearance of cyclic slip bands is quite rare and surface relief does not form well below the fatigue limit. The cyclic slip bands develop in regions of near-by oriented grains, where the neighbouring grains have very simmilar disorientation. Length of the cyclic slip bands substantially exceeds the average grain size of UFG Cu. No grain coarsening due to cycling was observed. Development of damage, which finally results in fatigue crack initiation, was observed below the surface relief.

Keywords: ultrafine-grained copper, gigacycle fatigue, cyclic slip bands

1 Introduction

Fatigue properties of conventional grain (CG) size materials in low- and high-cycle region has been studied very extensively since a long time. The knowledge is described in a number of textbooks and review publications, e.g. [1-3]. It holds partially also for ultrafine-grained (UFG) materials prepared by ECAP method [4-7], which has been studied intensively since the last two decades, see e.g. [7–14]. By contrast, fatigue properties in gigacycle region (alternatively called ultrahigh-cycle or very high cycle region) have been studied to substantially lesser extent, though fatigue failure of engineering components may appear after a number of cycles of 10^{10} or even higher [15].

Recent studies [16-20] concerning gigacycle fatigue behaviour of CG metals and alloys distinguish two kinds of materials. The first one, called Type I, includes pure fcc materials like copper. To the second Type II belong materials containing microstructural heterogeneities; high-strength bearing steels are an example. These two types differ by mechanism of fatigue crack initiation. In the case of the Type I materials, fatigue loading in gigacycle region causes surface roughening and subsequent persistent slip band or cyclic slip marking formation, followed by fatigue crack initiation. The fatigue failure of the Type II materials most frequently occurs in the

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form of so-called "fish-eye" fracture, originating from inclusions or heterogeneities and followed by propagation of an internal crack. Research studies of fatigue behaviour in gigacycle region performed up to now were focused exclusively CG materials. The knowledge on fatigue behaviour of UFG materials in gigacycle fatigue region is missing.
The aim of this work is to investigate the fatigue behaviour of UFG Cu, which belongs to the type I materials. Particularly, the localization of cyclic plasticity in gigacycle region and the mechanism of fatigue crack initiation was studied.

2 Material and experiments

Copper of commercial purity (99.9 %) was prepared by equal channel angular pressing (ECAP). The number of passes was equal to 8 by the B_C route (90° rotation in the same direction after each pass). Chemical composition of investigated Cu is given in **Table 1**. Specimens for fatigue tests with a diameter of 4 mm in a gauge section were machined from ECAPed billets. The gauge length of the specimens was fine-grounded, mechanically polished and finally electropolished. Fatigue tests were performed under constant stress amplitude in symmetrical push-pull cycling on an ultrasonic machine with loading frequency of 20 kHz. The specimens were cooled by flow of air to keep the temperature below 50 °C.
Surface relief, sub-surface and microstructure of fatigued specimens were investigated by means of scanning electron microscope (SEM) Philips XL30 and focused ion beam (FIB) equipped scanning electron microscope TESCAN LYRA\FEG.

Table 1 Chemical composition of UFG Cu

Cu	Impurities (%), no more than										
	Bi	Sb	As	Fe	Ni	Pb	Sn	S	O	Zn	Ag
min. 99.9%	0.001	0.002	0.002	0.005	0.002	0.005	0.002	0.004	0.05	0.004	0.003

3 Results

Fatigue tests in gigacycle regime were conducted in a stepwise manner. It was observed that cyclic slip markings appear on the surface since certain value of the stress amplitude. Moreover, the examination of the surface indicates that their occurrence is mainly localized in the crack vicinity. Their density on the rest of the specimen surface is very low. The example of fatigue crack is shown in **Fig. 1**, detail of crack with the slip markings is in **Fig. 2**. The specimen was loaded at first with the stress amplitude of 100 MPa for 1×10^{10} cycles and after observation of the polished surface by SEM the loading was continued at the higher stress amplitude of 120 MPa for 8.64×10^{9} cycles. No cyclic slip markings were found on the surface. Subsequent fatigue loading on the higher stress amplitude of 130 MPa resulted in failure after next 5.18×10^{9} cycles and the formation of cyclic slip markings on the surface. SEM micrograph of fatigue slip markings developed after loading with the stress amplitude of 130 MPa is shown in **Fig. 3**. The same area, but displayed by ion channelling contrast technique, can be seen in **Fig. 4**. It is obvious that the surface slip markings developed in the area with the low grey contrast of neighbouring grains, which shows that the disorientation between grains is small. The zone can be called "zone of near-by oriented grains". Outside this zone the disorientation between the grains is clearly higher. The grain size determined by the transmission electron microscopy in previous work of one the author is 300 nm [9]. It is obvious that the length of the cyclic slip

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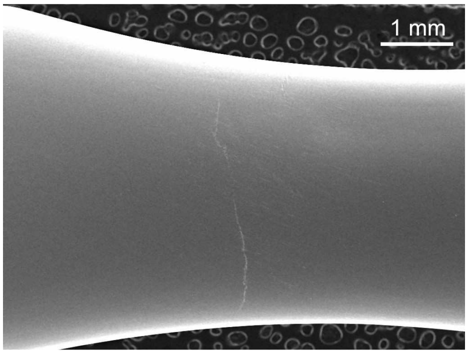

Fig.1 Sample with fatigue crack

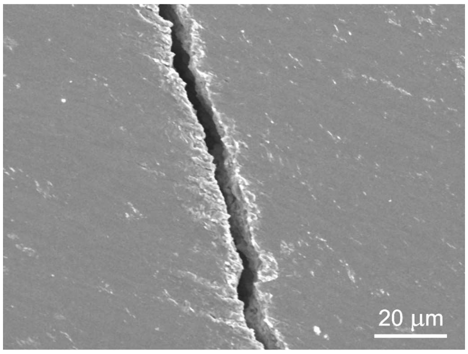

Fig.2 Detail of fatigue crack with cyclic slip markings

markings clearly exceeds this average grain size. Further, it is evident that the fatigue loading does not cause any substantial microstructure changes in terms of the grain size. The same conclusion can be drawn from **Figs. 5** and **6**, where the ultrafine-grained microstructure is well visible just beneath the cyclic slip markings.

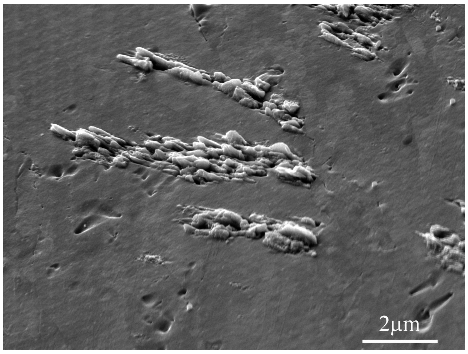

Fig.3 SEM micrograph of cyclic slip markings on the surface

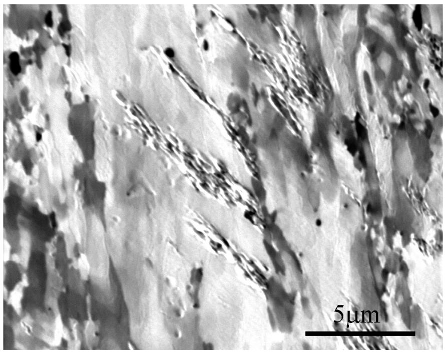

Fig.4 Cyclic slip markings and structure observed by means of FIB

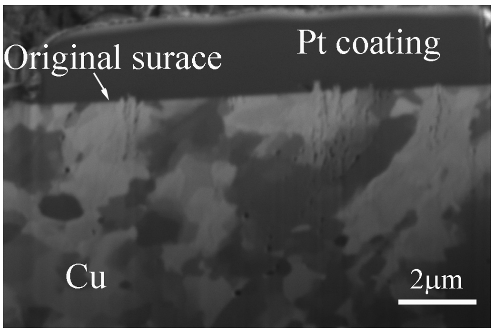

Fig.5 Sub-structure under cyclic slip bands observed by means of FIB.

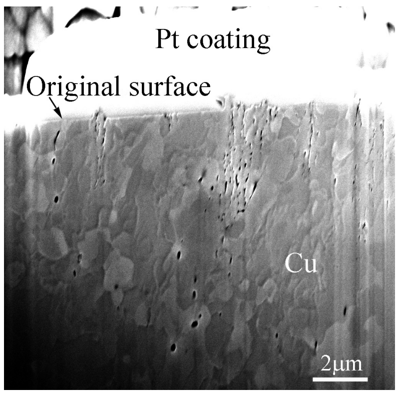

Fig.6 SEM micrograph of the sub-structure under cyclic slip bands

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Fig. 5 shows an example of SEM micrograph of a FIB cut performed perpendicular to surface slip markings displayed by ion-induced secondary electrons. By reason of protecting the surface was coated by platinum layer. After that the material was taken away by focused ion beam. The original surface with cyclic slip markings coated by platinum layer is well visible. Also the grain structure below the surface can be clearly recognized, even in **Fig. 6**, where the sub-structure is displayed by secondary electrons. Further, small approximately parallel cracks, arranged under circa 45° can be seen below the fatigue slip markings and some of them are connected with the surface relief. These cracks can be considered the nuclei of fatigue cracks in UFG Cu.

4 Discussion

Comparison of CG and UFG Cu behaviour under loading in gigacycle fatigue regime shows substantial differences. Recent paper by Stanzel-Tschegg et al. [18] dealing with CG copper after gigacycle fatigue loading describe formation of cyclic slip bands on the specimen surface. Their occurrence was observed not only above, but also below a certain cyclic stress (strain) amplitude value - persistent slip band threshold. Density of the slip markings increases with increasing stress (strain) amplitude and the number of cycles. Nevertheless, failure does not occur even if slip markings cover the whole surface. The very high cycle fatigue threshold for formation of persistent slip bands (VHCF-PSB threshold) [19] obtained for CG copper is $\Delta\sigma/2 \approx 45$ MPa for 2.7×10^8 cycles. It is expected that persistent slip bands and small cracks can be formed at even lower stress amplitudes. Thus, the cyclic slip activity in CG copper has to take place at stress amplitudes below the 0.5 of the VHCF fatigue limit. In the case of UFG Cu no slip activity was observed at the stress amplitude of 120 MPa, whereas an increase of the stress amplitude to 130 MPa leads to development of cyclic slip markings and finally to fatigue failure of the specimen. The ratio is here about 0.9. The explanation of the observed behaviour can be based on the differences in the shape of the stress-strain curves, **Fig. 7**. The plastic strain amplitude (measured over the whole gauge length of the specimen) corresponding to the fatigue limit of CG copper is of the order of 10^{-5}, whereas the value in the case of UFG copper is significantly lower. The corresponding part of the stress-strain diagram for UFG Cu is practically linear and the value is not detectable by conventional methods of plastic strain determination. As shown in **Figs. 5** and **6**, below the cyclic slip markings take place the cyclic slip localization. Dislocation activity in near-by oriented grains leads to a slip of these highly localized regions and subsequently to the generation of surface relief and formation of cavities under the slip markings, which are sites of fatigue crack initiation. It is interesting that similar

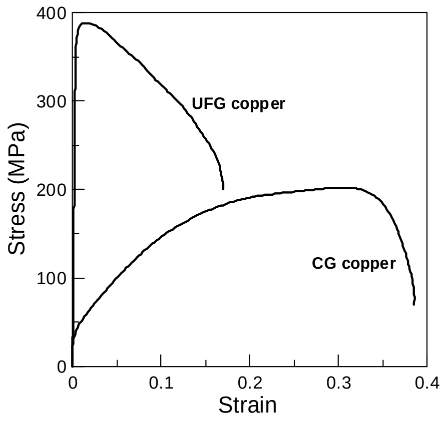

Fig.7 Stress-strain curves of copper with conventional and ultrafine grain size.

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mechanism was described in CG copper in work [20], where the family of roughly parallel cracks under PSB surface markings was also observed and they were considered to be stage I shear cracks.

5 Conclusions

1) Gigacycle fatigue loading of UFG Cu causes formation of surface relief. However, cyclic slip bands appear not until the stress amplitude value reaches 130 MPa. Below this stress amplitude no surface relief development was observed.
2) The observation of area beneath the cyclic slip bands revealed formation of cavities, which causes crack initiation.
3) No changes in microstructure in terms of a grain coarsening after gigacycle fatigue loading were observed by means of SEM or FIB.
4) Cyclic slip markings, which are sites of fatigue crack initiation, are formed in zones of low grain misorientation; these zones can be called zones of near-by oriented grains.
5) Length of the observed fatigue slip markings is significantly larger than the average grain size of material.

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