Development of fatigue damage in ultrafine-grained copper

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Abstract. Equal channel angular pressing is one of severe plastic deformation methods often used for preparation of ultrafine-grained (UFG) materials in bulk. Mechanical and, particularly, fatigue properties of UFG structures has been a subject of recent intensive research. The fatigue lifetime of UFG materials is closely related to the fatigue crack initiation. For conventionally grained (CG) materials the crack initiation mechanism is well described and understood. However, this is not the case of UFG structures. Furthermore, the knowledge obtained on CG materials cannot be straightforwardly transferred to UFG structures just from the reason of the grain size, which is smaller than the characteristic dimension of dislocation structures, which develops due to fatigue in CG materials.

Copper is one of the most thoroughly studied model materials as regards the investigation of fatigue crack initiation mechanisms. The cyclic slip bands, which develop on the surface of cycled Cu and which are sites of crack initiation exhibit very similar features in CG and UFG material. However, the mechanism of the cyclic slip localization known from the CG Cu cannot work in UFG structure. The characteristic dislocation structures formed by fatigue and known from CG Cu cannot simply develop in UFG material unless the grain coarsening takes place.

This contribution brings results of an experimental investigation of fatigue crack initiation in UFG copper. Development of fatigue slip bands was studied by means of focused ion beam (FIB) technique in scanning electron microscope. The observation of cyclic slip bands and material microstructure just beneath them shows that the grain coarsening is not a necessary prerequisite for crack initiation and development of surface relief. Observation of dislocation structure on thin foils prepared by FIB reveals well-developed cell structure below slip bands. The technique of ion-channeling contrast indicates that slip bands develop in regions of grains, which exhibit only very small mutual disorientation (low angle boundary regions). Based on these observations the mechanism of fatigue crack initiation in UFG Cu is discussed and compared with that known from CG copper.

Introduction

Increasing market demands for so-called “advanced materials” having special properties and acceptable cost is a significant feature, which strongly manifests itself during the last years. There is a tendency to substitute “older” materials in constructions and in manufacture of advanced components with the aim to increase their performance and to keep economic costs. UFG materials prepared by severe plastic deformation (SPD) methods, including equal channel angular pressing (ECAP), represent a promising group of such advanced materials. They have got the grain size in the range of 100 nm to 1 μm and represent a transition between CG materials and nano-materials. In comparison with CG materials UFG ones exhibit higher tensile properties and in the case of stress-controlled fatigue loading also higher fatigue strength [1] - [4].

A formation of cyclic slip bands on the material surface during fatigue loading is a decisive stadium of a fatigue life of homogeneous materials [5] - [7]. The cyclic slip bands in UFG Cu were investigated by means of scanning electron microscopy (SEM) since the first studies focused on the fatigue behavior of UFG structures. They were called similarly to the bands observed on CG Cu persistent slip bands (PSBs) [5], later on also shear bands [8]. The introduction of focused ion beam technique (FIB) [9], [10], which is nowadays a technique often available in SEM, made it possible to observe in addition to surface relief also areas and structure just underneath the cyclic slip bands. Another advantage of FIB is a phenomenon known as ion-channeling, which enables to observe local crystallographic orientation of microstructure together with the surface relief.

Fatigue behaviour and mechanisms of cyclic plastic localization during fatigue in CG materials are well described in a great number of publications and reviews, e.g. [11] - [13] but mechanisms taking place in UFG materials are still not sufficiently understood. The crack initiation and early crack propagation in CG Cu is closely related to the development of PSBs. Extrusions and intrusions, which forms during the cyclic loading on the free surface, play an important role. Under the surface relief appears a specific dislocation structure. In the case of low-amplitude loading the ladder-like structure, during high-amplitude loading layers of dislocation cells. By contrast in UFG materials inside the fatigued specimens, with a few exceptions, it was difficult to see marked changes of the dislocation structure [14].

The aim of this study was to investigate the development of cyclic slip bands and their role in the fatigue crack initiation. FIB technique and transmission electron microscopy (TEM) were used to reveal the microstructure under the surface relief of the slip bands and in the nearest vicinity of early fatigue cracks.

Material and experiments

Copper of 99.9 % purity processed by ECAP was used. Cylindrical billets of 20 mm in diameter and 120 mm in length were produced by 8 passes through the die by B_C route (after each pass the billet was rotated by 90°). Samples of 16 mm in diameter and 100 mm in length were turned from the billets. From these samples cylindrical specimens for fatigue tests were manufactured.

Fatigue tests were carried out under controlled load in symmetrical push-pull cycling. Two testing systems were used. A servohydraulic testing machine with frequency of cycling of about 10 Hz was applied for tests in high-cycle region. The diameter of the gauge section the specimens was 6 mm. An ultrasonic testing system with the frequency of 20 kHz was used for giga-cycle fatigue tests. Specimens with diameter of 4 mm in the gauge section were used in this case. The specimens were cooled by flow of air to keep the temperature below 50°C.

Before fatigue testing the gauge section of specimens was mechanically and electrolytically polished. Surface relief of specimens after fatigue loading and microstructure was investigated in scanning electron microscope (SEM) Tescan Lyra 3XMU® equipped with FIB. Furthermore, using FIB, thin foils were prepared and observed in transmission electron microscope Philips CM12 TEM/STEM.

Results

Cyclic slip localization resulting in development of cyclic slip bands on material free surface takes place during fatigue loading of UFG Cu in high-cycle and giga-cycle region. An example of a surface relief developed after giga-cycle fatigue with controlled stress amplitude σ_a = 130 MPa can be seen in Fig. 1. As shown in Fig. 2, obtained by means of FIB and using ion-channeling contrast, the cyclic slip bands appear in areas, where the disorientation of neighbouring grains is low. The shades of gray differ only slightly in the region where the slip bands were formed. In areas, which are characteristic by large grain disorientation, only very short and slightly developed bands appear. The length of the cyclic slip bands in areas with small disorientation substantially exceeds the average grain size. Identical observation was obtained also in the case of high-cycle fatigue loading. Cyclic slip bands developed in UFG Cu after fatigue loading with σ_a = 170 MPa for 6.2×10^6 cycles are shown in Figs. 3 and 4.

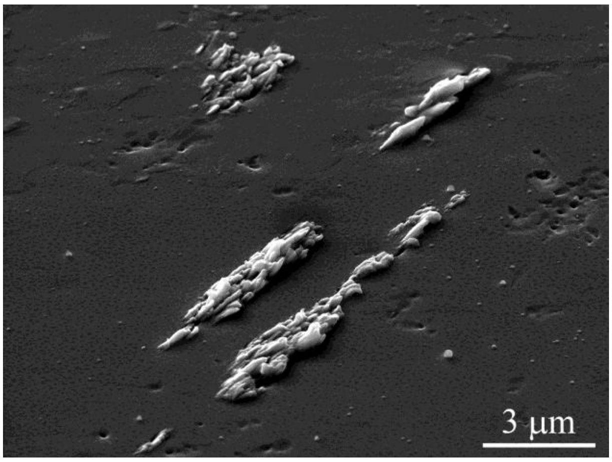

Fig. 1: Cyclic slip bands developed after fatigue loading with σ_a = 130 MPa for 2.3×10^{10} cycles.

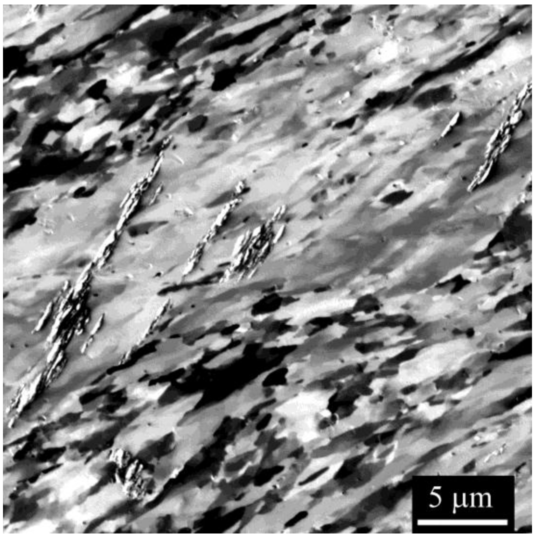

Fig. 2: Cyclic slip bands in the zone of near-by oriented grains after gigacycle fatigue loading.

The appearance of the slip bands changes along the specimen circumference. They develop on the whole circumference, however their orientation, length and frequency of occurrence vary according to the position on the specimen surface, i.e. according to the location of the particular place on the intersection of the last ECAP plane with specimen surface. Figs. 3 and 4 show examples of the slip bands on two different places of the specimen circumference, which differ by an angle of 90°. The loading axis is horizontal in both figures.

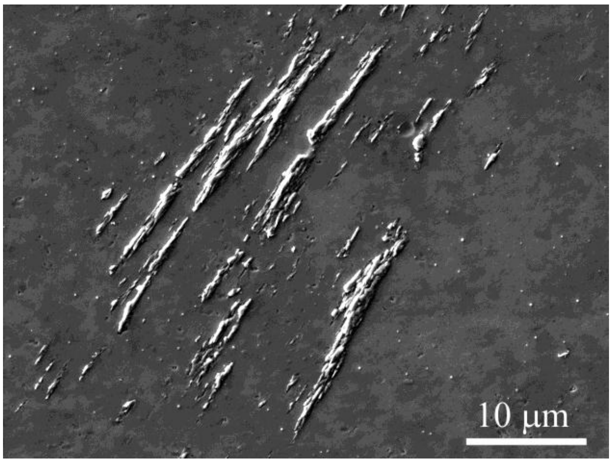

Fig. 3: Cyclic slip bands produced by high-cycle fatigue. The loading axis is horizontal.

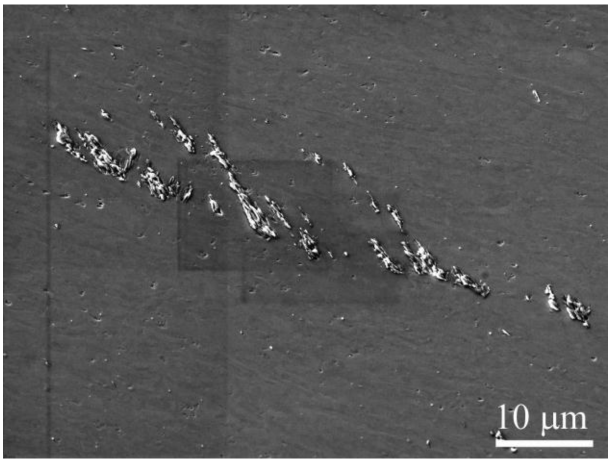

Fig. 4: Cyclic slip bands on the same specimen as in Fig. 3, however turned around the circumference by an angle of 90°.

Figs. 5 and 6 document the development of the cyclic slip bands during fatigue. Fig. 5 shows the surface relief after 9.5×10^5 cycles at the stress amplitude σ_a = 170 MPa. The view of the same area after 6.2×10^6 cycles at the same stress amplitude, Fig. 6, indicates that the slip bands do not develop continuously during the fatigue life. The majority of the bands visible in Fig. 5 remained without any marked change. Only some of them increased slightly in the length and marginally in the width. This means that the cyclic slip activity in bands, which start to be active at the beginning of the fatigue tests, exhausts. On the other hand, new bands appeared on the surface in areas where no apparent slip activity was visible after the first 9.5×10^5 cycles.
Fig. 6 shows the region with one of the fatigue cracks, which appeared on the gauge section of the specimen. The displayed area is a region where the crack initiated. From the comparison with Fig. 5 it can be concluded that the crack surprisingly passes through the areas where after the first 9.5×10^5 cycles only very short or even no slip bands were present.

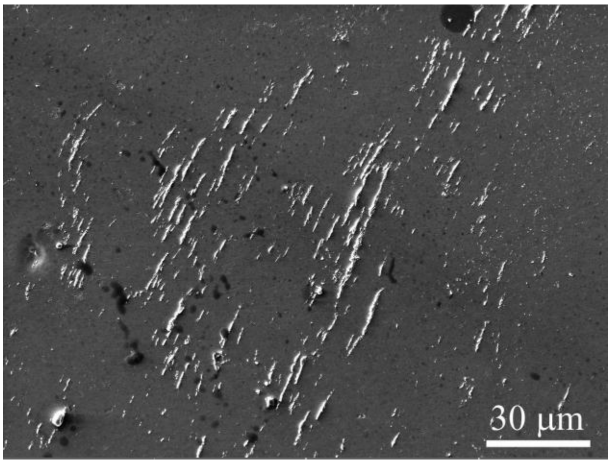

Fig. 5: Surface relief after 9.5×10^5 cycles at the stress amplitude σ_a = 170 MPa.

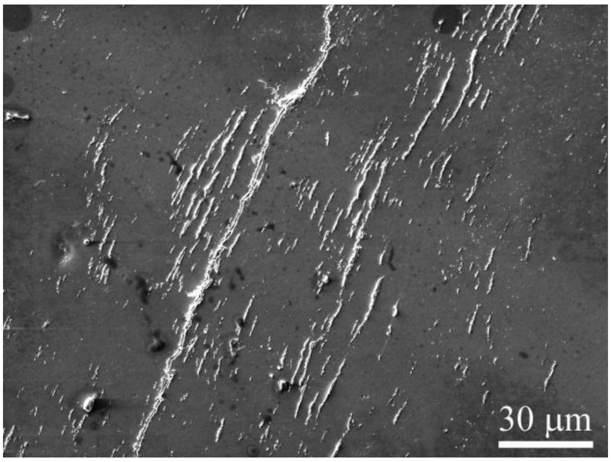

Fig. 6: Surface relief and fatigue crack after 6.2×10^6 cycles at the stress amplitude σ_a = 170 MPa.

Result of the observation of microstructure around an early fatigue crack by means of FIB is shown in Fig. 7. The central part of the crack (indicated by an arrow) is located in the zone of near-by oriented grains. The both crack tips lie in zones with higher grain disorientation. The crack path in the zone of near-by oriented grains is straight, whereas in the regions with higher disorientation is tortuous.
FIB milling technique was used for a preparation of cuts through the fatigue crack in the region of its initiation and for the preparation of foils for TEM. A platinum layer was transversally deposited over the crack. The reason of Pt deposition was protection of the original surface relief in the studied area during the milling process. Fig. 8 shows the cut through the crack shown in Fig. 6. The boundary between the Pt layer and Cu in Fig. 8 corresponds to the original surface of the specimen. The cut is oriented perpendicularly to the specimen surface. Fatigue crack penetrates from the surface into the material interior under the angle of approximately 45°. The crack is substantially opened. The opening makes nearly 1 μm. There is also evident a displacement of the material on both sides the crack. Fig. 8 shows beyond the crack also section through several cyclic slip bands, which are located in the crack vicinity. The surface relief with extrusions and the subsurface damage by fatigue is clearly visible. The orientation of damaged regions in material interior is the same in all cases and is situated along the macroscopic planes of highest shear stress.

The fatigue damage related to the cyclic slip bands is under higher magnification shown in Figs. 9 and 10. The bands were formed by fatigue loading resulting in fatigue failure of specimens either in high-cycle or giga-cycle fatigue regions. Fig. 9 displays a cut through slip bands which were produced by high-cycle fatigue of a sample loaded with $\sigma_a = 170$ MPa for 6.2×10^6 cycles. Extrusions reaching the height of 0.5 μm above the surface can be seen. The damage below the surface relief consists of cavities and failure of material aligned along the traces of the planes inclined to the surface at an angle of about 45°. Similar damage exhibits the sample cycled with $\sigma_a = 130$ MPa for 2.4×10^{10} cycles, Fig. 10. The fatigue damage under the surface is more pronounced in this case. Rows of small approximately parallel "cracks" arranged under circa 45° resembling the shear Stage I cracks can be seen.

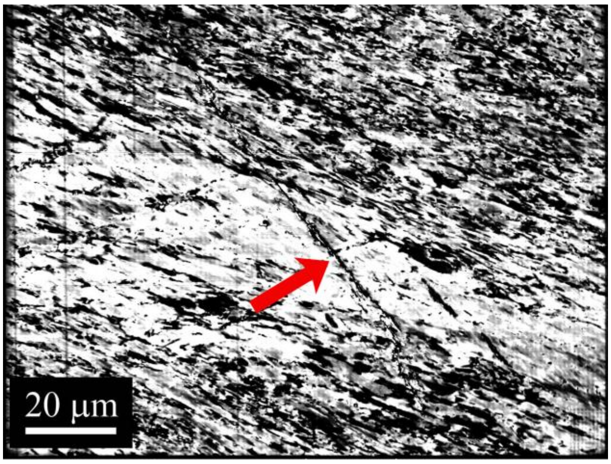

Fig. 7: Fatigue crack and microstructure displayed by means of FIB.

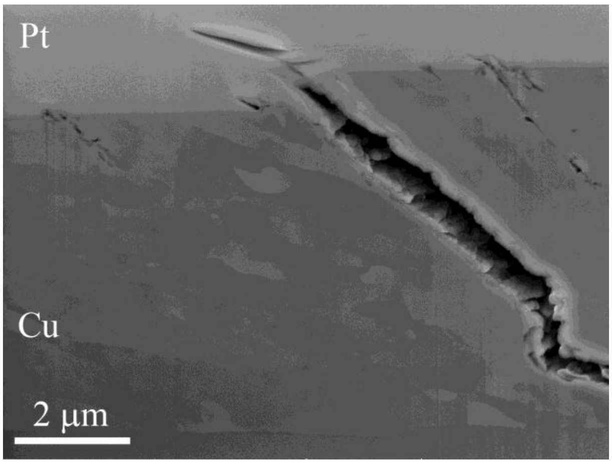

Fig. 8: FIB cut across the fatigue crack perpendicularly to the surface.

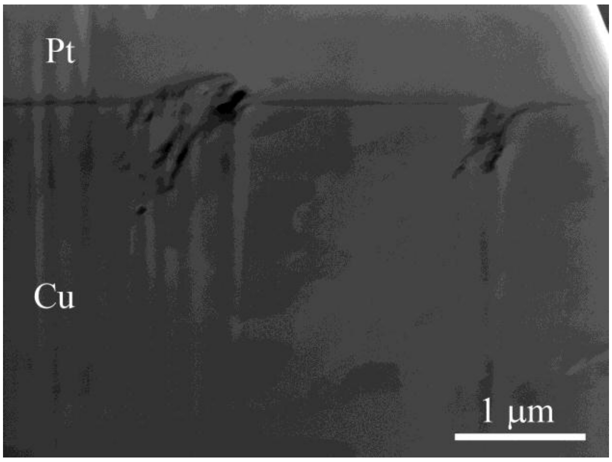

Fig. 9: FIB cut of cyclic slip bands developed during cycling with σ_a = 170 MPa.

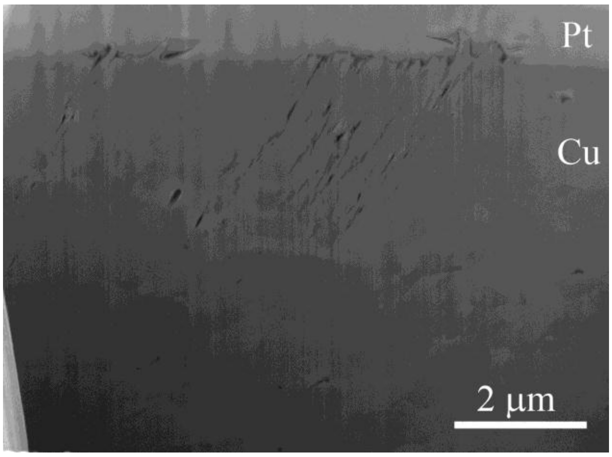

Fig. 10: FIB cut of cyclic slip bands formed after giga-cycle fatigue loading.

With the aim to observe the dislocation structure near the crack a thin foil containing the crack was cut using the FIB method. The FIB cut shown in Fig. 8 was thinned from both sides and finally the foil containing the crack was carefully cut off and observed in TEM. Fig. 11 shows the crack and the dislocation structure in its closest vicinity. The dislocation structure at higher magnification is shown in Fig. 12. The structure consists of slightly disoriented nearly equiaxial dislocation cells with narrow boundaries.

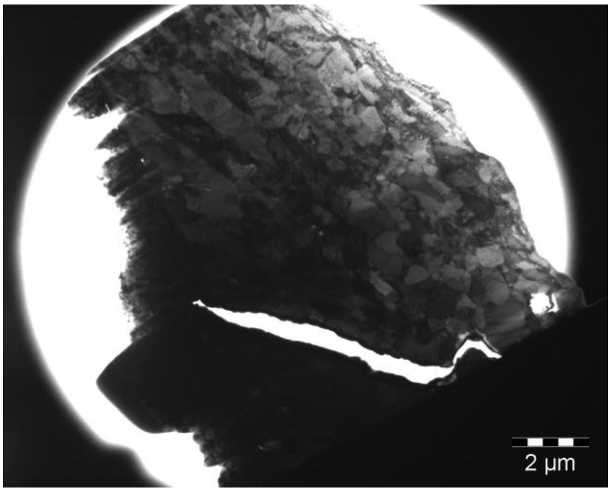

Fig. 11: Thin foil prepared by means of FIB showing the microstructure around fatigue crack. TEM.

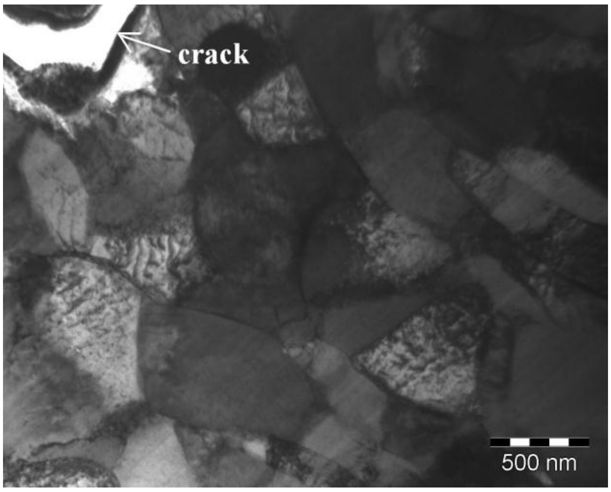

Fig. 12: Dislocation microstructure in the vicinity of the fatigue crack.

Discussion

The mechanism of cyclic slip localization, formation of slip bands and initiation of fatigue cracks is well described and known for CG Cu. Fatigue loading with small stress amplitudes resulting in fatigue failure in high-cycle or giga-cycle region produces slip bands which develop usually on one active slip system in individual grains. Slip bands in CG Cu are long and their dimension is limited by the grain size. The slip bands on the free surface are related to the specific dislocations structure beneath them in material interior.

In the case of UFG Cu the cyclic slip bands are substantially shorter when compared to CG material, but their average length substantially exceeds the average ultrafine-grain size. The cyclic slip activity appears above all in zones with near-by oriented grains. Here forms groups of bands oriented under nearly the same angle to the loading axes and also just here develop the longest bands. The bands do not grow continuously during the cycling. Some of them develop early after the beginning of the cycling and later on its activity ceases. On the other hand, in their vicinity new slip bands appear in the course of the cycling in areas, which were previously without any apparent slip activity. From this point of view the cyclic slip bands in UFG Cu seems to be not persistent. The existing and the new developed bands later on join and form long bands within the near-by oriented regions.

The fatigue damage expresses oneself by the growth of extrusions on the free surface. The extrusion height above the surface can reach up to 0.5 μm. There is no one to one correspondence between the extrusions and intrusions. Fig. 10 shows examples of extrusions, which do not have any intrusions in their neighborhood. FIB sections presented in Figs. 8 - 10, show that in material interior the fatigue damage manifests itself by forming of cavities and quite large voids. They are aligned along the traces of the planes inclined to the surface at the angle of about 45°. Moreover, deep in the material interior isolated cavities and elongated voids can be observed. These isolated voids are often situated in the grain interior. From the comparison of high-cycle and giga-cycle results it is obvious that this effect is most pronounced just in the case of giga-cycle fatigue. The observation of the fatigue damage on FIB sections confirms the idea, that the point defects play an important role. Vacancies produced by dislocation reactions migrate and form cavities and voids, which concentrate along the macroscopic planes with maximum shear stress. The planes along which the cavities and voids concentrate represent "weakened" areas, which later on in the course of fatigue join and form the fatigue crack nucleus. Here it is worthwhile to mention that similarly to the UFG Cu, in the case of giga-cycle fatigue of CG Cu under PSB surface markings were observed families of roughly parallel cracks oriented under the angle of circa 45° to the surface relief [15]. Authors consider these cracks to be stage I shear cracks. In the case of CG Cu, they are situated within one surface grain. In the case of UFG Cu the formation of the similar damage is related to region of the near-by oriented grains.

The mechanism of fatigue crack initiation, which was proposed in literature [8], [16] is based on the instability of UFG Cu under cyclic loading. The high stored energy can result in the dynamic grain coarsening during fatigue and in formation of bi-modal structure. The mechanism of fatigue crack initiation in the coarsened areas can be than similar to that known form CG Cu. From the observation of dislocation structure in TEM, Figs. 11 and 12 no grain coarsening or formation of bi-modal structure can be stated. In other words, the grain coarsening is not a substantial condition for crack initiation and propagation in the UFG Cu, which is in agreement with our previous studies [7], [17].

Conclusions

Fatigue cracks in UFG Cu initiate in cyclic slip bands, which develop in areas, where disorientation of neighbouring grains is small. The point defects generated by the irreversible movement of dislocations in high-cycle and giga-cycle fatigue play an important role in the formation of cavities and voids, which are observed beneath the cyclic slip band. The cavities and voids are situated on the macroscopic planes of maximum shear stress and represent the areas where the fatigue cracks nucleate. TEM observation of the dislocation structure near the crack indicates that the grain coarsening is not a necessary condition for the fatigue crack initiation in UFG Cu.

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