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Microstructural Features of Ultrafine Grained Copper under Severe Deformation

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In this work the microstructural features of pure copper were studied using two methods of severe plastic deformation: equal-channel angular pressing (ECAP) and hard cyclic viscoplastic (HCV) deformation.

During the first step the metal was severely deformed up to 10 B_c routes of ECAP. The ultrafine grained microstructure was received. The elongated laminar substructure has low angle and diffuse grain boundaries, but high dislocation density. Metal shows high hardness and strength but low ductility at tension straining. During the second step – HCV deformation – the strain amplitude of tension-compression cycles was stepwise increased from 0.2 % up to 2.5 % for 30 cycles and for five series. The results show, that under HCV deformation the ultrafine grained microstructure with high-angle grain boundaries was formed. The mechanism of microstructure evolution contains the elongated (ECAP processed) subgrains fracture under shear stresses by atomic layers of crystals and new microstructure with high-angle grain boundaries forming. By this the density of dislocation ribbons was decreased (from $\sim 4.3 \times 10^{14}$ m^{-2} to $\sim 2.1 \times 10^{14}$ m^{-2}) mainly inside of grains and the dislocation density was increased on/near new high-angle grain boundaries. The samples with such microstructure show relative stabile viscoplastic behavior, high level of tension-compression stresses and large uniform elongation under tension straining.

Keywords: severe plastic deformation, ultrafine grained microstructure, copper.

1. INTRODUCTION

At present [1, 2], extensive research on the methods of severe plastic deformation (SPD) for ultrafine grained (UFG) and nanocrystalline (NC) microstructure forming in soft metals, alloys and other materials has been carried out. Depending on the collected strain [3] these materials have different grain sizes (GS) of microstructure and consequently, different mechanical properties. They show usually higher tensile stress and hardness but lower ductility as compared to coarse-grained counterparts. In [4] is shown, that the toughening of UFG pure copper (Cu) is possible via high-angle grain boundaries (HAGBs) and low dislocation density microstructure forming. The UFG Cu ductility was increased by microstructure recrystallization via annealing in vacuum [5]. Unfortunately, during annealing the microhardness was decreased depending on temperature and heating time. In the other case, the nanostructured titanium (Ti) show outstandingly high impact toughness at cryogenic temperatures [6]. Contrarily, these UFG and NC metals and alloys show superplastic properties at lower temperatures then their coarse-grained (CG) counterparts [7]. In addition, in [7] it is shown that the superplastic UFG metals microstructure coarsened significantly (from 200 nm to 2 μm in mean) during superplastic tension and acquired the higher true strength. Such microstructure coarsening with true tensile strength increase show the Cu, which was preliminarily heat treated at low temperature up to 200 °C with low heating rate of 1 °C/min [5]. It means [7], that the important features of superplastic behavior of nanostructured materials is “improvement” of their granular structure associated with formation of a typical UFG structure with equiaxed grains and clearly defined grain boundaries. The strain-rate sensitivity is found to be proportional to the inverse of hardness [8]. In other works the fatigue properties [9 – 11] and shear bands forming [12, 13] under cyclic loading are studied. The results in [11] show that no significant changes of the microstructure due to fatigue loading. Only a small tendency to develop “shaken down” dislocations structures were observed by TEM after loading by highest stress amplitudes. The fatigue damage of UFG metals is controlled by large-scale banding process. Investigation of viscoelastic and viscoplastic behavior of UFG metals at increased strain amplitudes, under tension-compression cyclic loading are studied in [14 – 18]. The optimized parameters of SPD results in UFG microstructures excellent cyclic stability in strain controlled fatigue tests. These presented relationships between the SPD parameters and accumulated stress levels, microstructures with LAGBs or HAGBs, cyclic loading frequency and amplitude, strain rate and so on are very complicated in UFG metals and need future investigations.

The present study focuses on cyclic tension-compression response of ECAP-processed UFG Cu to microstructure forming that finally determines the mechanical properties characteristics. For performance of these properties of ECAP-processed UFG Cu the hard cyclic viscoplastic (HCV) deformation test method [17, 18] was used.

2. EXPERIMENTAL

The specimens used in this study were made of annealed commercial purity polycrystalline copper (Cu). This metal was subjected to equal-channel angular pressing (ECAP) at different passes number (up to ten) by B_c route. Rods were processed up to different ECA pressing numbers for obtaining of different ultrafine grained (UFG) microstructures. The ECAP processed rods with dimensions of 16 mm in diameter and 130 mm – 140 mm in length were subjected to machining from these the

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tensile test samples. The samples have reduced part with dimensions: diameter of 8 mm, length of 15 mm and fillet radiuses of 10 mm. The materials testing system Instron 8516, provided with a strain controller extensometer and personal computer that was used for the computerized testing and data acquirement. The tests were conducted at room temperature. All samples were tested at identical regimes by HCV deformation [17, 18]. The cycling loading was conducted at constant strain amplitudes of ±0.05 %, ±0.2 %, ±0.5 %, ±1 %, ±1.5 %, ±2 % and ±2.5 % for 30 cycles, respectively. The frequency of 0.5 Hz was chosen as constant. As the strain amplitudes were step-by-step increased, the strain rate was increased accordingly. The tension- and true stress were measured before (ECA pressed material properties) and after HCV deformation. The observation of microstructure evolution was performed with a transmission electron microscope (TEM). Under TEM investigation the slices were mechanically polished on both sides and jet thinned in a solution. For dislocation density and NC size measure the X-ray diffraction (XRD) test method was used. The samples for microstructure study were prepared from the ECA pressed- and reduced part of HCV deformed samples in diametric surface and mainly parallel to the stress axis. The microindentation of samples was conducted on Mikromet-2001 and Zwick Z2.5/TS1S installations [19]. The tests were conducted according to "Standard EVS-EN ISO 14577-1:2003: Metallic materials – Instrumented indentation test for hardness and materials parameters – Part 1: Test method".

3. RESULTS AND DISCUSSION

During the very first ECAP passes the microhardness was increased sharply from 65 $HV_{0.5}$ (in recrystallized condition) up to 125 $HV_{0.5}$. The maximal microhardness of 145 $HV_{0.5}$ has Cu after 8 – 9 passes by route B_c of ECAP. Also the Martens hardness [19] was increased from HM_S 30/11/300 = 371 N/mm^2 to HM_S 30/11/300 = 1352 N/mm^2 as well as tension stress was increased up to 435 MPa at strain of ~2 %.

TEM investigation shows that during ECAP for 10 passes by route B_c the ultrafine grained (UFG) microstructure in pure annealed Cu was formed. Material has very high dislocations density ($\sim 4.2 \times 10^{14}$ m^{-2}) and microstructure with low angle (~10°) grain boundaries (LAGBs). As a result of ECAP the GS was decreased in mean from 250 µm to 250 nm (up to ~1000 times!). The ECAP processed microstructure of pure Cu (UFG_{ECAP}) is shown in TEM picture in longitudinal (Fig. 1) and in cross-section (Fig. 2) of sample. The ECA pressed Cu have elongated grains with thickness of about 200 nm. XRD investigation of the same specimen shows that nanocrystallites (NC) have the mean size of about 80 nm.

A new SPD technique, so called as HCV deformation has been proposed for characterizing the viscoplastic behaviour of nanometals [18]. As it is shown in TEM pictures the microstructure (during HCV deformation) was appreciably changed in longitudinal (Fig. 3) as well in cross-section (Fig. 4). The elongated UFG_{ECAP} grains (see Fig. 1) were shattered and new equal-axed grains are formed.

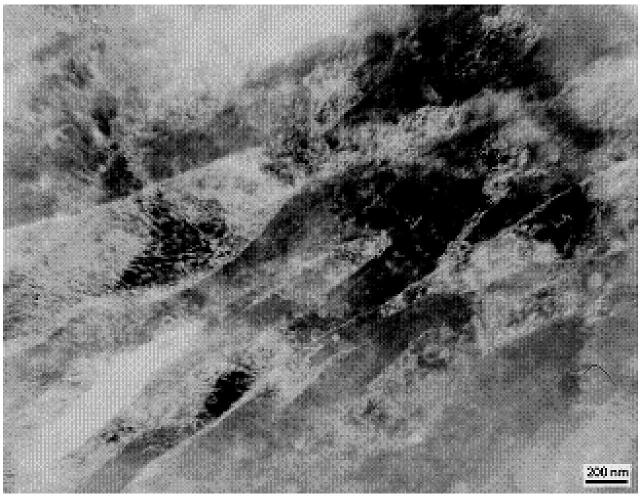

Fig. 1. TEM picture in longitudinal section (transverse plane) of UFG_{ECAP} Cu

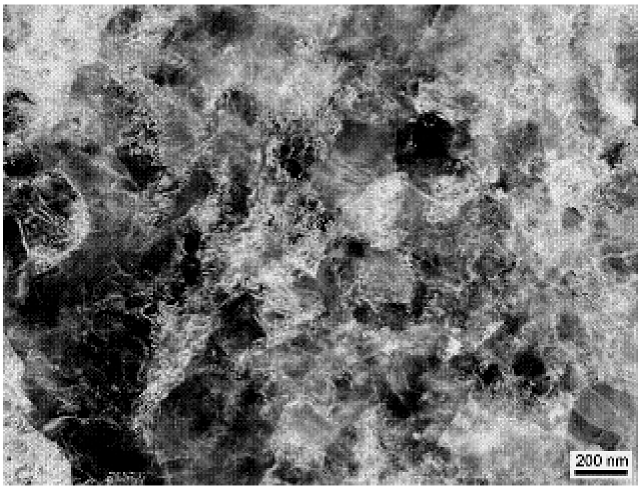

Fig. 2. TEM picture in cross-section of UFG_{ECAP} Cu (10 passes by B_c route)

New rhombohedral in view of grains are oriented by sharp corners in direction of cyclic load applied under HCV deformation. Dislocation ribbon densities in grains were merged as compared to UFG_{ECAP} (see Fig. 1 and Fig. 2). Contrary to Kunz et al. [11] during HCV deformation the microstructure evolution is so large. Draw a parallel between microstructures in TEM pictures in Figs. 1, 2 and Figs. 3, 4. Elongated subgrains with lattice structure were changed to rhombohedral grains with HAGBs.

During hard viscoplastic cycling the dislocations density was decreased significantly, mainly in grains. By this the dislocation density was increased via sliding in fracture regions of equiaxed grain boundaries (Figs. 5 and 6). Such UFG equal-axed HAGBs microstructure in Fig. 3 (compare with Fig. 1) was formed under cyclic loading at strain amplitudes of ±(1 – 1.5) %. Investigations established that during HCV deformation at stabile stress amplitude the grain size had not changed in dimensions as the cycle's number is limited up to 20 – 30 as maximal in this study.

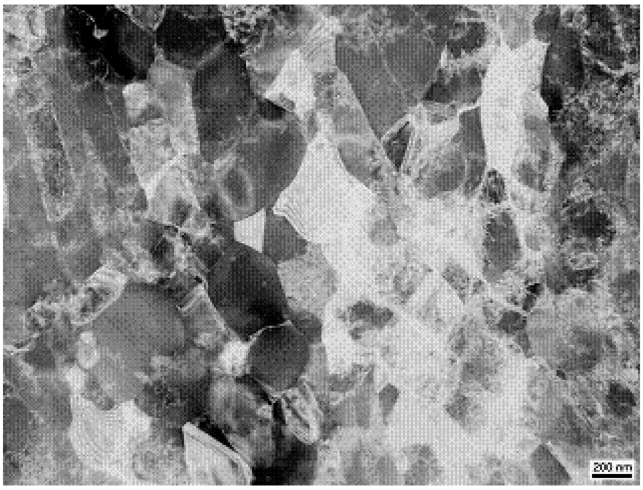

Fig. 3. TEM picture in longitudinal section of HCV deformed Cu followed by ECAP

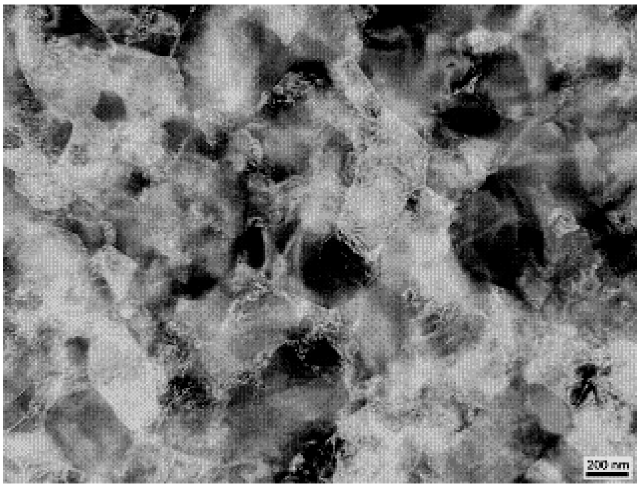

Fig. 4. TEM picture in cross-section of HCV deformed Cu followed by ECAP

The mechanism of becoming refined is illustrated in Figs. 5 and 6. The TEM and XRD investigations shows that during HCV deformation testing the microstructure changing via dislocation density decrease during at very first cycle's number is main mechanism for UFG metals mechanical properties damage.

During the dislocation density decrease the stress amplitude took slightly decrease also at very first cycles as the material toughening begins. Due to the XRD method, it was found that the HCV deformation influenced on the UFG metals microstrains to a great extend. The effect of structure softening after HCV deformation was observed at the same time with the high relative microstrain forming in microstructure.

By this the X-ray investigation results show that during HCV deformation of UFG_{ECAP} Cu the CS was decreased from 80 nm to 50 nm – 56 nm (for different specimens) and relative microstrains (RMS) (compare to annealed copper microstrains) were increased from 0.6 to 3.4, respectively. These test results confirm that this advantages method (HCV deformation) is precise, robust and very flexible.

In addition, during HCV deformation is possible to change the metal microstructure from LAGBs to HAGBs. The results show also, that the UFG_{ECAP} metals with LAGBs have high dislocations density and show high tensile strength and hardness while HCV deformed metals with HAGBs have low dislocation density but they are tougher. They show high stability under cyclic loading, good elastic and plastic characteristics, and uniform elongation at tension straining without accompanying significant sacrifice in its hardness and yield strength.

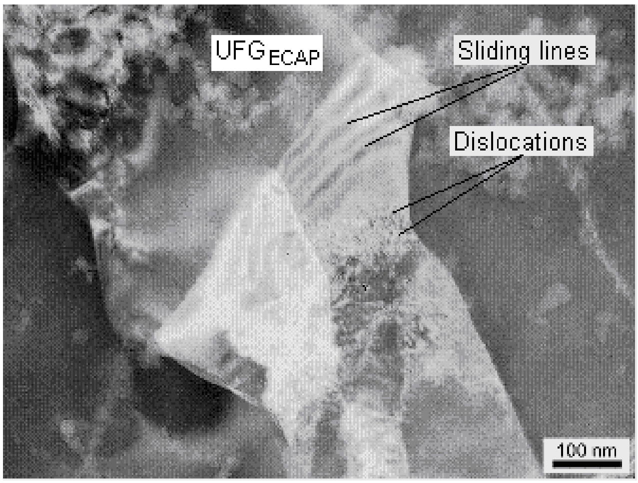

Fig. 5. The destruction mechanism of elongated UFG_{ECAP} grains via sliding under cyclic stresses of HCV deformation

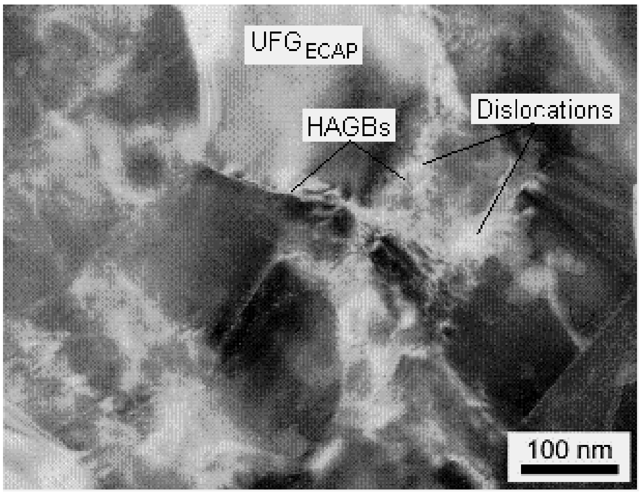

Fig. 6. The HAGBs UFG microstructure forming from elongated UFG_{ECAP} under cyclic stresses of HCV deformation

These results of toughened UFG_{ECAP} Cu via HAGBs and low dislocation density coincide with latest results presented by Zhao et al. [4].

4. CONCLUSIONS

Microstructure evolution under two SPD methods – ECAP and HCV deformation – is described and the results compared.

1. During ECAP the metal (pure Cu) coarse grained microstructure under shear stresses was changed to UFG microstructure with LAGBs. The hardness and tensile stress were increased significantly, but elongation under tension was decreased.

2. During HCV deformation of UFG_{ECAP} Cu the microstructure was reoriented and elongated grains were

fractured to rhombohedral grains in view of transverse plane. HAGBs UFG microstructure was formed under cyclic loads in viscoplastic condition of UFG_{ECAP} metal. Such metal is tougher, show increased plastic characteristics and uniform elongation at tension straining but such changes are accompanimented with lowering of hardness.

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