**PHYSICAL FOUNDATIONS OF STRENGTH AND PLASTICITY**

# **Plastic Deformation of Ultrafine-Grained Metallic Materials**

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**Abstract**—The micromechanisms of plastic deformation of ultrafine-grained metallic materials are analyzed using copper, the nanostructure of which is produced by equal-channel angular pressing, as an example. Slip traces are studied at various deformation stages, and their parameters are estimated. The change in the gran ular structure during deformation has been studied.

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#### 1. INTRODUCTION

Plastic deformation of ultrafine-grained (UFG) materials has been extensively studied in recent years. The purpose of these studies is to find the character of flow curves  $\sigma = f(\varepsilon)$ , where  $\sigma$  is the flow stress and  $\varepsilon$  is the strain, to discuss the problems of fulfillment of the Hall–Petch relation for fine grains, and to determine operating deformation mechanisms [1–14].

The main change in the mechanisms of plastic deformation when the average grain size of UFG materials increases is the transition from the grain boundary effects related to the transition of free and constraint volume, grain-boundary diffusion, and grain-boundary sliding to dislocation slip in the grain volume when the Burgers vector increases gradually from partial dislocations to perfect dislocations [14]. In the other words, main deformation occurs along grain boundaries if their density is high and in the grain body if the boundary density is not high. However, the quantitative characteristics of an UFG material defect structure that change with deformation have not been adequately studied. Because of this, such studies should be continued. This paper presents some results of studying the micromechanisms of plastic deforma tion in UFG copper.

# 2. EXPERIMENTAL

Specimens of high-purity copper with an UFG structure were produced by equal-channel angular pressing (ECAP) at room temperature without interme diate annealing. During ECAP, initial  $14 \times 14 \times 60$ -mm workpieces with a grain size of 300 μm were subjected to shear deformation by repeated compression in two channels of the same diameter intersecting at an angle of 90 $^{\circ}$ . The true plastic strain upon ECAP was  $e \approx 4$ . From the workpieces,  $4 \times 4 \times 6$ -mm specimens were

cut; the specimens were subjected to uniaxial com pressive deformation at room temperature on an Instron testing machine. Deformation was carried out at a rate of  $1.4 \times 10^{-3}$  s<sup>-1</sup> to  $\varepsilon = 2.6, 8.6, 29, 53,$ and 80%.

The structure of copper was studied on foils and high-resolution carbon replicas using an EM-125K electron microscope equipped with a goniometer at an accelerated voltage of 125 kV. The carbon replicas were prepared from the central part of the side surface of the deformed specimens. The replicas were coated with platinum at a low angle to the surface. Foils were prepared using a standard technique. Plates for the foils were cut from the central part of the deformed specimens perpendicular to the compression axis. The sizes of grains, subgrains (fragments), and dislocation cells in the foils were measured by the linear-intercept method. The replicas were used to measure the shear in slip traces from the step height on the surface, the distances between slip traces, and the number of slip systems.

## 3. RESULTS AND DISCUSSION

#### *3.1. Types of Grains in UFG Copper*

As was found in [11, 15], UFG copper subjected to ECAP contains the following three types of grains with different dislocation structures: dislocation-free grains, grains having a chaotic dislocation structure, and grains containing a dislocation substructure (cells or fragments) (Fig. 1). The sizes of grains of each type increase from dislocation-free grains to grains with cells and fragments (Table 1). Such a grain structure and a grain size distribution are due to the specific fea tures of formation of a nanostructured polycrystalline aggregate during ECAP [1, 16]. For example, defor mation is stopped at some moment; because of this,



**Fig. 1.** Electron-microscopic images of three types of grains: (a) dislocation-free, (b) with chaotically arranged dislocations or dislocation networks, and (c) with disloca tion cells or fragments.

the structure contains dislocation-free fine grains and moderate-size grains with a developed dislocation structure. The coarsest grains of the nanocrystalline aggregate contain cells, which are gradually trans formed to fragments, i.e., subgrains bounded by low angle boundaries. During further deformation, the fragments become new nanograins.

# *3.2. Slip Pattern in UFG Copper at Various Deformation Stages*

The dependence  $\sigma = f(\varepsilon)$  for UFG copper under study exhibits a fairly specific feature (Fig. 2) [11]. The copper has a high yield point  $(\approx 390 \text{ MPa})$  followed by



**Fig. 2.** Stress and strain-hardening coefficient vs. strain for UFG copper.

a fairly short  $(1-2\%$  of the strain) stage with a high constant coefficient of strengthening θ = *d*σ/*d*ε (stage II). This stage is changed by a small portion (to  $\varepsilon = 5\%$ ), where strengthening is close to a parabolic process (stage III). Then, at a stress of ≈500 MPa, a prolonged portion almost without strengthening is observed (stage IV).

The study of the slip pattern of UFG copper at var ious deformation stages shows that the main mecha nisms contributing to the form change of the speci mens at room temperature are intragrain dislocation glide and grain-boundary sliding. Figure 3 shows the typical pattern on the surface of a deformed specimen of the UFG copper. We can observe thin slip traces inside grains and rougher ones along grain boundaries. Of course, intragrain dislocation glide and grain boundary sliding correlate with each other. Slip occurs not only along the boundary of one grain but often includes several boundaries of neighboring grains. Table 2 gives the generalized characteristic of a slip pattern obtained from the observation and measure ment of the shears in the slip traces and the deforma tion micromechanisms at various stages of strain hard ening. Let us comment this table.

It should be noted that rougher slip traces along high-angle grain boundaries are easily observed from the start of deformation using the replica method. It is more difficult to observe the pattern of intragrain slip, which is performed by small dislocation groups.

**Table 1.** Average sizes of grains of various types in UFG copper produced by ECAP

Type of grains	$d, \text{nm}$	$P_V$
Dislocation-free	$83 \pm 33$	0.02
With chaotic dislocationstructure	$105 \pm 17$	0.10
With dislocation cells	$231 \pm 45$	0.88
Averaged over material	$210 + 100$	10

*d* is the grain size and  $P_V$  is the volume fraction of grains.



**Fig. 3.** Typical image of the surface of the deformed UFG copper sample surface observed in replicas at  $\varepsilon =$  (a) 29 and (b) 53%. Rough slip traces along grain boundaries (indi cated by arrows) and thin traces inside grains are visible.

The pattern is quantitatively identified only at stage IV  $(\epsilon > 20\%)$ . Although the resolution of the replica method used in this work (1.5–2 nm) is fairly high, the shears performed by groups of six or less dislocations cannot be observed. They perform a significant part of deformation. The study of the deformation relief shows that the main contribution to the deformation of UFG copper is due to uniform slip of small disloca tion groups, with the number of dislocation  $n \leq 6$  in a group. First, one slip system is observed. At  $\epsilon \geq 50\%$ , we observe two slip systems. The average shear in a slip trace observed at  $\varepsilon \approx 53\%$  is  $\approx 6$  nm. A visible part of slip

is one-fourth/one-third of the entire deformation. The other part of deformation falls on the slip of small dislocation groups, which give traces below the resolu tion of the replica method. In this case, grain-bound ary sliding is one-tenth/one-fifth of the entire defor mation.

It can be suggested that intragrain dislocation slip in copper with a submicrometer grain size leads to strengthening and grain-boundary sliding leads, more probably, to softening. If the grain-boundary sliding is easy, these two mechanisms act simultaneously. Then, the strain-hardening coefficient at stage IV is small or even zero. This is the situation that takes place in UFG copper (Fig. 2). It is known  $[17-21]$  that the activation energy of grain-boundary processes, namely, grain boundary sliding and grain-boundary migration, is significantly lower in UFG materials than in coarse grained materials. Because of this, these processes occur easily, which explains the existence of stage IV with a zero strain-hardening coefficient.

An analysis of these results shows that a grain boundary source starts to work at the yield point only in the largest grains. This implies that other smaller grains are deformed only elastically at such stresses. From the start of deformation, intense slip is observed along one-fifth of the boundaries of the largest grains, and the average shear is 2.5 nm. Dislocations are emit ted only in the third-type coarse grains  $(d = 600 \text{ nm})$ . Deformation at the yield point and stage II is provided by grain-boundary sliding and the emission of small dislocation groups ( $n \leq 5$ ) in the largest grains with cells and fragments. At stages II and III, replicas only allow the observation of grain-boundary sliding. In going to stage III, small dislocation groups begin to slip in 450–500-nm grains. The fast strengthening at stage II is related to the disappear of the contribution of coarse grains and their boundaries to deformation. In going to stage III, grain-boundary sliding covers more and more boundaries. In this case, the local shear increases quickly. To the end of stage III, the flow stresses are due to grain-boundary shear; the shear is 8–10 nm. Hereafter, the fraction of grain boundaries in which sliding takes place is unchanged; however, the fraction of grains in which the slip of dis location groups with  $n \ge 10$  takes place increases.

According to these results, even at stage IV, a grain with the average size ( $\langle d \rangle = 210$  nm) in an ensemble in an UFG copper polycrystal is deformed via the emission of small dislocation groups consisting of no more than five dislocations from one boundary and their absorption by another boundary. In addition, at all degrees of deformation beginning from stage II, grain boundaries migrate and grains grow. These factors determine the possibility of intragrain dislocation activity in coarse grains with markedly larger sizes than the average size. Fine grains, the sizes of which are smaller than the average size, do not contribute to a

Deformation	Deformation mechanisms and the sizes of the grains in which they operate			
stages	grain-boundary sliding	intragrain dislocation slip	transformation of fragments into grains	
Yield point	Sliding along the boundaries of the coarsest grains $d = 600$ nm	Small dislocation groups $n = 1-5$	N <sub>0</sub>	
Stage II	Sliding along the boundaries of coarse grains $d = 500-600$ nm	Small dislocation groups $n \approx 6$	Beginning of the transformation of fragments into grains	
Stage III	Increase in the shear and slip trace length, sliding along sev- eral coarse-grain boundaries $d = 450 - 600$ nm	Small dislocation groups $n \leq 6$	Transformation of grain substructure and the formation of new grains and fragments	
Stage IV	Sliding along every second grain boundary $d = 300-600$ nm, shear $\sim$ 2 nm	Dislocation groups $n \ge 6$ in 40% grains	Active transformation of grain sub- structure, formation of new grains and fragments, and grain growth	

**Table 2.** Grain sizes, micromechanisms, and stages of deformation of UFG copper

change of the specimen shape. However, their role in strengthening UFG copper is undoubtedly significant.

### *3.3. Substructure of UFG Copper after Plastic Deformation*

The change in the distribution of grain sizes during plastic deformation characterizes the intragrain and



**Fig. 4.** Grain size distribution and its variation during deformation of UFG copper.

grain-boundary processes in more detail. According to the data generalized in Fig. 4, the grain size distribu tion function is smeared as the degree of plastic defor mation increases. During deformation, the main types of grains (dislocation-free grains, grains with disloca tions, and grains with cells and fragments) are retained, but their relative fractions are changed. After deforma tion at  $\epsilon = 80\%$ , the grain size is  $310 \pm 205$  nm. A comparison of the grain size distribution in the initial state and after  $\varepsilon = 80\%$  shows that the grains of all types undergo changes. The fraction of the smallest grains  $(d < 100$  nm) increases by a factor of almost two. This shows that, during deformation, new grains nucleate from fragments and grow. Simultaneously, coarse grains with fragments grow intensely. Their fraction decreases but the average size increases by a factor of more than two (from 230 to 485 nm). The average size of grains with dislocations also increases almost twice. Only the average size of dislocation-free grains remains almost unchanged during deformation.

An analysis of these results shows that the change in the grain-size spectrum has a quasi-oscillating charac ter. This is doubtlessly due to the fact that a large part of deformation develops at a constant flow stress. Doubtlessly, the plastic deformation of UFG copper at room temperature occurs under dynamic recrystalli zation. This conclusion follows from a comparison of the grain sizes in the distribution tails shown in Fig. 4. After  $\varepsilon = 80\%$ , there are grains with a size of 1000 nm  $(1 \mu m)$ .

#### 4. CONCLUSIONS

A significant role of the grain size distribution in the deformation of UFG copper was revealed. It was found that the slip of small dislocation groups begins in the coarsest grains at the yield point. Simultaneously, grain boundary sliding takes place. Further deformation is provided by the involvement of fine grains into intra-

grain slip and grain-boundary sliding. Dislocation-free grains do not take part in deformation.

The contribution of moderate-size grains to defor mation is insignificant. It is due to the slip of individ ual dislocations from one boundary to another (through the grain body) and grain-boundary sliding. The grains whose sizes are larger than the average size make the main contribution to deformation. The sig nificant contribution of coarse grains to deformation is provided by substructural transformations, in particu lar, the formation of high-angle boundaries and sub boundaries and new grains from fragments, which were arranged in old coarse grains earlier.

The main micromechanisms can be arranged in an order of decreasing their contributions to the relative deformation as follows: intragrain slip of dislocation groups in one plane and of individual dislocations (in grains with near-average sizes), an increase in the number of active slip planes from one to two, grain boundary sliding, and grain-boundary migration (for coarse grains). The contribution related to grain boundary diffusion is also not excluded.

#### **REFERENCES**

- 1. R. Z. Valiev and I. V. Aleksandrov, *Nanostructured Materials Produced by Severe Plastic Deformation* (Logos, Moscow, 2000).
- 2. R. A. Andrievskii and A. M. Glezer, "Size effects in nanocrystalline materials: I. Structure characteristics, thermodynamics, phase equilibria, and transport phe nomena," Phys. Met. Metallogr. **88** (1), 45–66 (1999).
- 3. R. A. Andrievskii and A. M. Glezer, "Size effects in nanocrystalline materials: II. Mechanical and physical properties," Phys. Met. Metallogr. **89** (1), 83–102  $(2000)$ .
- 4. R. R. Mulyukov and N. I. Noskova, *Submicrocrystalline Metals and Alloys* (IFM UrO RAN, Yekaterinburg, 2003).
- 5. M. Yu. Gutkin and I. A. Ovid'ko, *Nanocrystalline Materials/Physical Mechanics of Deformed Nanostruc tures* (IPM RAN, St. Petersburg, 2003) Vol. I.
- 6. M. Yu. Gutkin and I. A. Ovid'ko, *Nanolayered Struc tures/Physical Mechanics of Deformed Nanostructures* (IPM RAN, St. Petersburg, 2003), Vol. II.
- 7. *Severe Plastic Deformation: Toward Bulk Production of Nanostructured Materials*, Ed. by B. S. Altan (Nova Sci. Publ., New York, 2006).
- 8. R. Z. Valiev and I. V. Aleksandrov, *Bulk Nanostructural Metallic Materials* (Akademkniga, Moscow, 2007).
- 9. F. Z. Ytyashev, *Modern Methods of Severe Plastic Defor mation* (UGATU, Ufa, 2008).
- 10. A. P. Zhilyaev and A. I. Pshenichnikov, *Superplasticity and Grain Boundaries in Ultrafine-Grained Materials* (Fizmatlit, Moscow, 2008).
- 11. E. V. Kozlov, N. A. Koneva, A. N. Zhdanov et al., "Structure and the resistance to deformation of fcc ultrafinemetals and alloys," Fiz. Mezomekhainka **7** (4), 93–113 (2004).
- 12. R. A. Andrievskii and A. M. Glezer, "Strength of nano structures," Usp. Fiz. Nauk **179** (4), 337–358 (2009).
- 13. G. A. Malygin, "Strength and plasticity of nanocrystal line materials and nanosized crystals," Usp. Fiz. Nauk **181** (11), 1129–1156 (2011).
- 14. E. V. Kozlov, L. I. Trishkina, N. A. Popova, and N. A. Koneva, "Place of the dislocation physics in mul tilevel approach to plastic deformation," Fiz. Mezomekhainka **14** (3), 95–110 (2011).
- 15. E. V. Kozlov, N. A. Popova, Yu. F. Ivanov et al., "Struc ture and sources of long-range stress fields in ultrafine grained copper," Ann. Chim. Fr. **21**, 427–442 (1996).
- 16. V. M. Segal, V. I. Reznikov, V. I. Kopylov, et al., *Pro cesses of Plastic Structure Formation in Metals* (Nauka i Tekhnika, Minsk, 1994).
- 17. R. Monzen and Y. Sumi, "Determination of activation energy for nanometer-scale grain-boundary sliding in copper," Phil. Mag. A **70** (5), 805–817 (1994).
- 18. Y. Umakoshi, W. Fujitani, T. Nakano et al., "The role of dislocation in high-strain-rate superplasticity of an Al– Ni-misch metal alloy," Acta Mater. **46** (13), 4469–4478 (1998).
- 19. B. Calet, Q. P. Kong, L. Lu, and K. Lu, "Low temper ature creep of nanocrystalline pure copper," Mater. Sci. Eng. A **286**, 188–192 (2000).
- 20. B. L. Shen, T. Itoi, T. Yamasaki, and Y. Ogino, "Inden tation creep of nanocrystalline Cu–TiC alloy prepared by mechanical alloying," Scripta Mater. **41**, 893–898 (2000).
- 21. Yu. R. Kolobov, G. P. Grabovetskaya, K. V. Ivanov, and M. B. Ivanov, "Grain boundary diffusion and mecha nisms of creep of nanostructured metals," Interface Sci. **10**, 31–36 (2002).

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