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STRENGTH AND PLASTICITY

Effect of Temperature–Strain-Rate Conditions of Deformation on Structure Formation in Commercially Pure Copper Deformed in Bridgman Anvils

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Abstract—Commercially pure copper (99.9 wt % Cu) subjected to high-pressure torsion at room temperature is investigated. A relation between the copper structure and temperature–strain-rate conditions has been established. It has been shown that impurity dragging prevents grain growth during postdynamic recrystallization. This has allowed us to determine the conditions under which either the hardening of the deformed material, which is accompanied by a continuous increase in the hardness and structural refinement, or the dynamic recrystallization resulting in the stabilization of the hardness and the average grain size predominantly occurs in commercial-purity copper compared to high-purity copper (99.99 wt %). It has been shown that, under the hardening conditions, the structure of the investigated copper is determined by the true strain, whereas under dynamic recrystallization conditions, by temperature–compensated strain rate.

Keywords: deformation, structure, hardness, dynamic recrystallization, copper

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INTRODUCTION

There are different views on the mechanisms of the formation of a disperse misoriented structure upon large plastic deformation [1–7]. One of the mechanisms is considered to be dynamic recrystallization (DR). Different authors found signs of dynamic recrystallization in metals and alloys, for which a deformation temperature of ~20°C is both the high [8–9] and low homologous temperature [10]. In these studies, the dependence of the structure and properties of materials on the true strain were determined as is accepted when studying cold deformation. In [11], the other approach for analyzing the structure evolution has been proposed, which is usually applied to hot deformation, when structural development is considered to be dependent on the temperature–strain-rate conditions of deformation. This approach allowed the staged character of structure formation to be revealed during DR. In [11], high-purity copper (99.99 wt %) was studied, a significant influence on the structure formation of which belongs to postdynamic recrystallization (PDR), which stimulates the growth of individual very coarse grains. It is difficult to establish the laws of dynamic recrystallization development, since the structure of this material does not stop changing after deformation ended. It is known that alloying with a small amount of impurities is an effective way to retard the grain growth [12]. Therefore, the aim of this work is to study the staged character of structure formation under dynamic recrystallization in copper of commercial purities.

EXPERIMENTAL

Copper samples (99.99% purity) 5 mm in diameter and 0.3 mm thick were deformed by high-pressure torsion at 6 GPa and room temperature, at anvil rotation rates of 0.3 and 1 rpm (revolutions per minute). The angle of rotation of the anvil was 15° to 15 rev. The true strain was calculated from the formula

$$e = e_{sh} + e_{up} = \ln(1 + (\varphi r/h_{ir})^2)^{1/2} + \ln(h_0/h_{ir}), \quad (1)$$

where e_{sh} is the shear strain; e_{up} is the upsetting strain; φ is the angle of rotation of the anvil; and h_0, h_{ir} are the thicknesses of the sample before and after deformation, respectively, at distance r_i from the center. The maximum true strain achieved $e = 12$, with an experimental error of ±0.2 [13].

The microhardness was measured in two mutually perpendicular sample diameters using a PMT-3 hardness-testing machine under a load of 0.25 and 0.125 N. In truth, this characteristic is hardness if the size of structural elements is less than 1 μm. To construct the averaged hardness dependence as a function of the true strain, all values from different samples

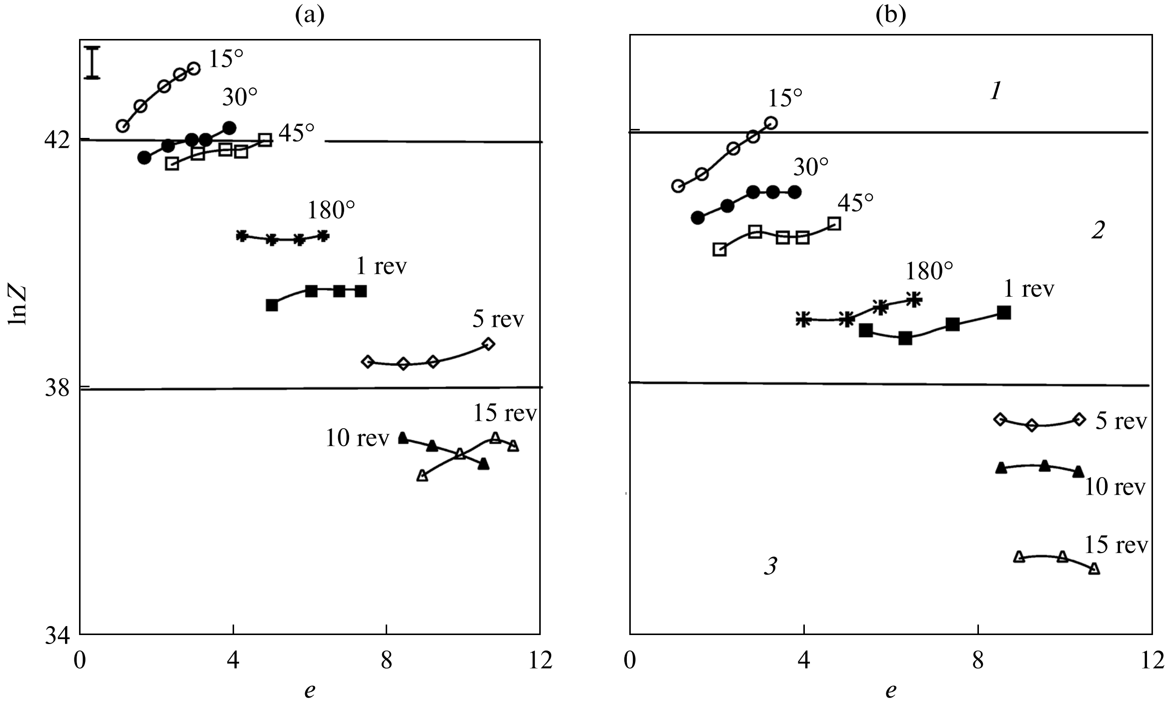

Fig. 1. Temperature–strain-rate conditions of the deformation of commercial-purity copper at various rates of anvil rotation. (a) ω = 1 rpm and (b) ω = 0.3 rpm.

were divided according to the ranges $\Delta e = 4$ and in each range the hardness values were averaged again.

The structure was investigated using a JEM 200CX electron microscope at a distance of 1.5 mm from the sample center. The size of structure elements was determined from the bright- and dark-field electron microscopy images in reflection $(111)_\gamma$ using at least 200 measurements; the error being no more than 10% [14]. The coefficient of variation of linear grain sizes was calculated as the ratio of standard deviation to the average grain size. STATISTICA software was utilized to plot the histograms of the size distribution of structural elements. The maximum, minimum, and most probable size of structural elements were determined.

The temperature–strain-rate conditions of deformation was characterized by the Zener–Hollomon parameter (Z–H) [15, 16] in the form:

$$\ln Z = \ln \dot{e} + \Delta H / RT, \qquad (2)$$

where $\dot{e}$ is the true strain rate, s^{-1}; ΔH is the activation energy of high-temperature deformation, the value of which is close to the activation energy of self-diffusion; R is the gas constant; and T is the deformation temperature, K. We took $\Delta H = 107$ kJ/mol [17]; $R = 8.31$ J (mol K)$^{-1}$; $T = 300$ K. When calculating the true rate of deformation, it was supposed that in each deformed sample the structural changes first were similar to those in the samples deformed with smaller angles of anvil rotation, and the differences arose upon increasing the true strain in the course of subsequent rotation. Therefore, the true rate of deformation was calculated by the equation

$$\dot{e} = \Delta e / \Delta \tau, \qquad (3)$$

where Δe is the true strain at which the structural changes occur compared to that of the samples deformed with a smaller angle of the anvil rotation and $\Delta\tau$ is the time interval required to gain the corresponding true strain increment.

The spread of the Z–H parameter, which was associated with the reproducibility of the test results when changing one series of the samples to the other, did not exceed 1. Therefore, hereafter the absolute measurement error was taken to be $\Delta \ln Z = \pm 0.5$.

RESULTS AND DISCUSSION

For high-purity copper, it was established in [11] that dynamic recrystallization begins when the true strain achieves $e = 2$ and its further development depends on parameter $\ln Z$. In the range $34 < \ln Z < 38$ dynamic recrystallization takes place in the entire volume of the material (stage of complete DR). At $\ln Z > 42$, no signs of DR are detected (stage of cellular structure). At intermediate values of $38 < \ln Z < 42$, volume fraction of the structure that undergoes dynamic recrystallization decreases with increasing $\ln Z$. Figure 1 demonstrates the Z–H parameter for the commercial-purity copper calculated at various distances from the center of the samples deformed to various angles of anvil rotation at two rotation rates. In

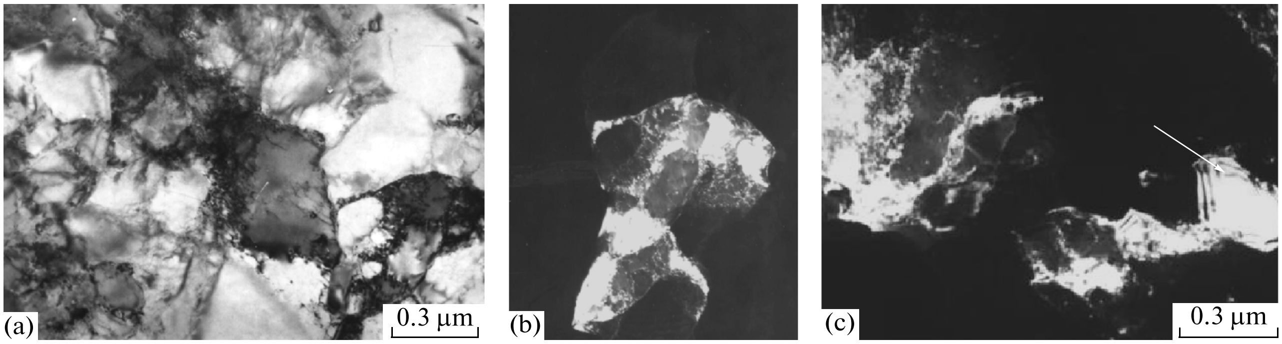

Fig. 2. Microstructure of commercial-purity copper at the stage of hardening ($\ln Z > 42$): (a), (b) $\varphi = 15°$, $e = 1.9$, $\ln Z = 42.7$, $\omega = 1$ rpm and (c) $\varphi = 45°$, $e = 2.9$, $\ln Z = 41.8$, $\omega = 1$ rpm.

the figure, the boundaries of the stages of structural states are also shown, which were defined in [11] for high-purity copper. Experimental results show that $\ln Z$ can both vary along the sample radius and remain the same. It can be seen that curves shift toward the lower values of $\ln Z$ with a decrease in the rate of anvil rotation from 1 to 0.3 rpm.

In the case when the rate of anvil rotation ω = 1 rpm and angles of rotation of 15° and 30°, temperature–strain-rate conditions correspond to region (1), where DR does not develop in the high-purity copper, whereas at $\omega = 0.3$ rpm the curves for these angles already lie in the intermediate region (2), where separate DR grains must be formed. Upon deformation to 5 rev, depending on the rate of rotation, the temperature–strain-rate conditions of deformation correspond either to region (2) or region (3) corresponding to complete DR. After 10 rev, DR covers the entire volume of a sample regardless of the rate of anvil rotation.

Previously, it was established in the study [18] of 99.99%-purity copper that the effect of the rate of anvil rotation on the structure became more pronounced if the temperature–strain-rate conditions occur in different regions of structural states upon deformation to the same angles of rotation. Two of the most typical cases can be distinguished at an angle of the anvil rotation of 15° and 180°. So, deformation at $\varphi = 15°$ and different rates of anvil rotation provides close true strain $e = 1.5$–2, but temperature–strain-rate conditions vary greatly. At $\omega = 1$ rpm ($\ln Z = 42.5$), a dislocation cellular structure is formed. As the rate of anvil rotation decreases to 0.3 rpm, $\ln Z$ decreases to 41.5, which corresponds to the transition to the stage of partial DR. Indeed, separate recrystallization nuclei (microcrystallites) are observed against the background of the cellular structure. Deformation at ω = 0.05 rpm is characterized by $\ln Z = 39.5$ and provides the conditions for the formation of a large number of microcrystallites, some of which acquire a geometrically regular shape and a banded contrast at boundaries [18].

Electron microscopic examination of commercial pure copper showed that the type of the structure corresponds to the temperature-rate conditions of deformation. At the first stage, the structure consists of dislocation cells with a low-angle misorientation gradually changed from cell to cell (the structure of copper deformed to 15° and 30° at a rate of anvil rotation of 1 rpm corresponds to these $\ln Z$).[1] Figures 2a and2b show electron microscopy images of such a structure. Figure 2b demonstrates dark-field image taken in reflection $(111)_\gamma$ with a continuous change in orientation within neighboring cells. Under these temperature-strain-rate conditions, main structure-forming processes are work hardening and dynamic recovery; the latter seems to act at all stages of deformation. First, elastically distorted microcrystallites with high angle boundaries (indicated by an arrow in Fig. 2) are observed in the structure when $\ln Z$ decreases below 42 (41.8 for $\varphi = 45°$ at ω =1 rpm and 41.5 for $\varphi = 15°$ at $\omega = 0.3$ rpm) and during transition to the second stage. Such microcrystallites are nuclei of DR. Similar structure elements are also formed upon the action of rotational modes of deformation at cryogenic temperatures excluding the development of DR [19, 20]. Perhaps, upon deformation in Bridgman anvils, there are no significant differences in the mechanisms of the formation of microcrystallites upon work hardening and DR. Further deformation upon work hardening generates structure distortions accumulated in a microcrystallite; as a result, new high-angle boundaries separate their internal volume, which causes the refinement of structure elements. Upon DR, this process is accompanied by thermally activated movement of high angle boundaries of microcrystallites due to difference in the dislocation density inside the microcrystallite body and outside in the surrounding matrix. As a result, the average DR grain size can be stabilized. However, some researchers do not attach much

[1] Here and below, $\ln Z$ are calculated at a distance $r = 1.5$ mm from the axis of sample rotation.

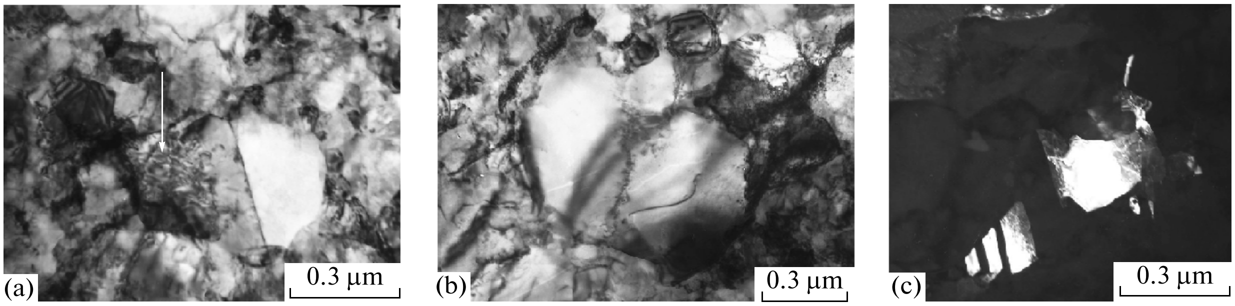

Fig. 3. Microstructure of commercial-purity copper at the stage of individual dynamically recrystallized grains ($38 < \ln Z < 42$). (a) $\varphi = 360°$, $e = 5.0$, $\ln Z = 39.5$, $\omega = 1$ rpm; (b) $\varphi = 180°$, $e = 5.0$, $\ln Z = 40.4$, $\omega = 1$ rpm; and (c) $\varphi = 45°$, $e = 3.5$, $\ln Z = 40.3$, $\omega = 0.3$ rpm.

importance to the difference between processes of strain hardening and DR [21].

Deformation of commercial-purity copper under temperature–strain-rate conditions in the range $41.5 > \ln Z > 38.0$, regardless of the rate of anvil rotation, is accompanied by the appearance of recrystallized grains (Figs. 3a, 3b), the volume fraction of which grows with decreasing $\ln Z$ upon deformation. Dynamically recrystallized grains that already contain regions have arisen with an increased density of dislocations formed during deformation after the nucleus (indicated by the arrow in Fig. 3), and regions with low density of defects generated after a moving high-angle boundary. In the grains formed in high-purity copper during postdynamic recrystallization, annealing twins are frequently observed [11]. In commercial-purity copper investigated in this work, annealing twins are observed much less frequently (Fig. 3c). The presence of twins in the recrystallized grains is an important structural feature of postdynamic recrystallization. On the contrary, in the case of dynamic recrystallization, twinning is complicated [22]. In this way, the presence of impurities in the commercial-purity copper, considerably complicates PDR, but it does not lead to its complete suppression.

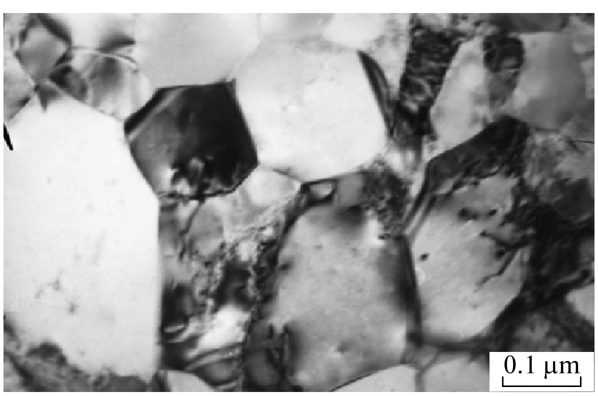

Fig. 4. Microstructure of commercial-purity copper at the stage of the complete dynamic recrystallization (10 rev, $\ln Z = 37.2$, $e = 8.1$, $\omega = 1$ rpm).

When the deformation conditions correspond to the region $\ln Z < 38.0$, DR covers the entire volume of the material. Since there are many nuclei of recrystallization, collision between growing grains occurs rapidly to form fine similar-in-size recrystallized grains, which often have a geometrically regular shape (Fig. 4).

Figure 5 shows the histograms of the size distribution of structural elements (dislocation cells, microcrystallites, and recrystallized grains) of copper at various stages of deformation. Tables 1 and 2 list the main characteristics of the size distribution of structure elements, namely, average d_{av} and most probable d_{pr} sizes, the coefficient of variation of linear grain sizes K calculated as the ratio of standard deviation to the average grain size, as well as the corresponding true strains, and temperature–strain-rate conditions.

The size distribution at all stages is unimodal. At the stage of work hardening (stage 1) and even at the beginning of stage 2, when the first microcrystallites arise, an increase in the true strain leads to the refinement of structure elements and improvement of their size uniformity (Figs. 5a, 5b). At the second stage, no refinement of structure elements is observed, the distribution shows a "tail" in the region of coarse sizes (Fig. 5d), and the coefficient of variation of linear sizes increases to 0.7–0.8. The coefficient of variation characterizes the dimensional uniformity of the structure: for a uniform structure $K = 0.5$–0.6; an increase in its values to 1.2 indicates the appearance of abnormally coarse grains and substantial dimensional structure nonuniformity [23]. Earlier, in [11], it was shown that in pure copper a tail in the area of the grain size 5–7 μm is a result of postdynamic recrystallization. In the commercial-purity copper studied in this work, the tail observed in the range of grain sizes 1.0–1.5 μm can be caused by both PDR and DR-induced grain growth. Figure 3b support the latter. It shows a substructure inside a fairly coarse grain, which divides the grain into blocks. This substructure could only appear

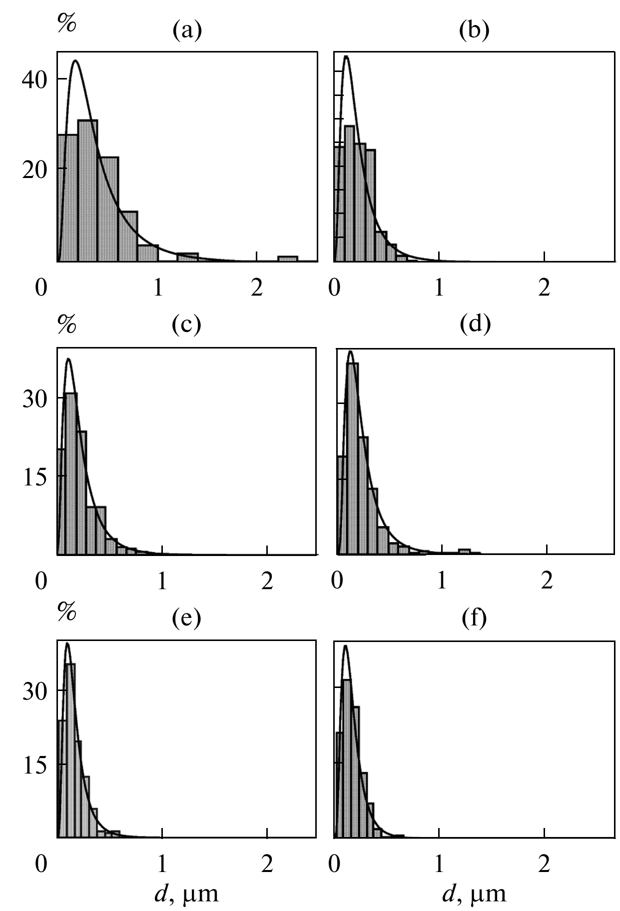

Fig. 5. Histograms of size distribution of structure elements: (a) $\varphi = 15°$, $e = 1.9$, $\ln Z = 42.7$, $\omega = 1$ rpm; (b) $\varphi = 45°$, $e = 2.9$, $\ln Z = 41.8$, $\omega = 1$ rpm; (c) $\varphi = 60°$, $e = 4.8$, $\ln Z = 40.2$, $\omega = 0.3$ rpm; (d) $\varphi = 180°$, $e = 5.8$, $\ln Z = 39.2$, $\omega = 0.3$ rpm; (e) 10 rev, $e = 8.1$, $\ln Z = 37.2$, $\omega = 1$ rpm; and (f) 15 rev, $e = 9.0$, $\ln Z = 35.2$, $\omega = 0.3$ rpm.

because of the deformation of a growing recrystallized grain.

At the third stage, a structure is formed that is uniform in size, where $K = 0.6$. The shape and parameters of grain size distribution do not depend on the true strain and rate of anvil rotation (Fig. 5e, 5f; Tables 1, 2).

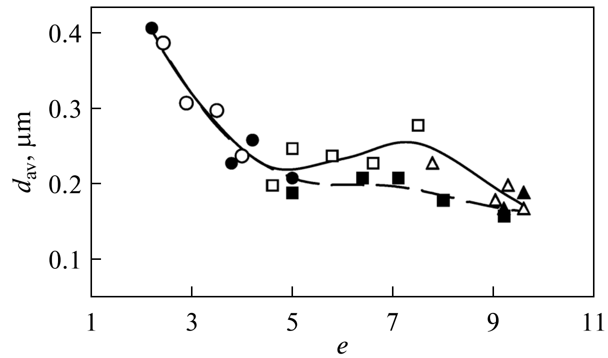

Fig. 6. Average size of structure elements as a function of true strain for two rates of anvil rotation: 0.3 rpm (open symbols) and 1 rpm (solid symbols); (△), (▲) $\ln Z < 38$; (□), (■) $38 < \ln Z < 40$; and (○),(●) $\ln Z > 40$.

Figure 6 shows the dependence of the average size of structural elements on the true strain e for two rates of anvil rotation. The structure undergoes refinement with increasing e regardless of the rate of anvil rotation; then, after reaching $e = 5$, the dependence of d_{av} on ω is observed. At $\omega = 1$ rpm, d_{av} stabilizes at the level 0.19 ± 0.02 μm. At $\omega = 0.3$ rpm, the average size increased to 0.28 μm in the strain range $e = 5$–8 and $\ln Z = 38$–40, and then at $e > 9$ ($\ln Z < 38$) structure elements are refined again to values that correspond to a high strain rate.

Figure 7 presents the dependence of the hardness on the true strain taking into account division into stages according to the temperature–strain-rate conditions, i.e., intervals $\ln Z$. At the first stage ($\ln Z > 42$), the hardness increases with true strain; at the third stage ($\ln Z < 38$) it remains practically unchanged. At the second stage, there is a large scatter in the values of hardness. It has been shown earlier in works [11, 18] dedicated to the study of pure copper that this scatter was associated with grain size nonuniformity observed at the intermediate stage when DR prevailed ($\ln Z < 40$). Therefore, data related to the intermediate stage and presented in Fig. 7 were divided into two parts using $\ln Z$: one data array relates to $40 < \ln Z < 42$ when work hardening predominates, while the second

Table 1. Structural parameters of the copper deformed at an anvil rotation rate $\omega = 1$ rpm

Revolution of the anvil	Stages	$\ln Z$	e	d_{av}, μm	d_{pr}, μm	σ	K
$\varphi = 45°$	1	41.8	2.9	0.23	0.12	0.14	0.6
$\varphi = 60°$	2	41.3	3.2	0.26	0.14	0.18	0.7
$\varphi = 180°$		40.4	5.0	0.21	0.10	0.18	0.8
1 rev		39.4	5.0	0.19	0.10	0.14	0.7
5 rev		38.4	9.2	0.16	0.08	0.10	0.6
10 rev	3	37.1	9.2	0.17	0.10	0.10	0.6
15 rev		36.6	9.6	0.19	0.10	0.13	0.5

Table 2. Structural parameters of copper deformed at the rotation rate of the anvil $\omega = 0.3$ rpm

Revolution of the anvil	Stages	$\ln Z$	e	d_{av}, μm	d_{pr}, μm	σ	K
$\varphi = 15°$	2	41.7	2.4	0.39	0.18	0.27	0.7
$\varphi = 30°$		41	2.9	0.31	0.09	0.29	0.9
$\varphi = 45°$		40.3	3.5	0.31	0.12	0.25	0.8
$\varphi = 180°$		39.2	5.8	0.24	0.13	0.18	0.8
1 rev		38.9	7.5	0.28	0.14	0.20	0.7
5 rev	3	37.3	9.3	0.20	0.10	0.12	0.6
10 rev		36.7	9.6	0.17	0.08	0.13	0.8
15 rev		35.2	9.0	0.18	0.12	0.10	0.6

relates to the region $38 < \ln Z < 40$ when DR plays the leading role. It can be seen that, in the first region, the scatter of hardness values is significantly less than in the second region. In region of work hardening prevalence, the hardness increases with true strain and data from the first ($\ln Z > 42$) and intermediate ($40 < \ln Z < 42$) stages made up one dependence. Data related to the intermediate stage ($38 < \ln Z < 40$) and the third stage ($\ln Z < 38$) also form one dependence, which attains the saturation level. It should be noted that, for the commercial purity copper, the coefficient of variation of linear grain sizes at $\ln Z < 40$ does not exceed 0.8, i.e., there is no significant growth of individual grains during PDR. Therefore, the scatter of hardness values in this interval of $\ln Z$ may be associated with a nonuniform distribution of defects due to the development of DR rather than size nonuniformity of the structure as it is in pure copper [11, 18].

Figure 8 shows the dependence of the average grain size on the temperature–compensated strain rate ($\ln Z$). In this case, a structure formed at $\ln Z > 42$ is not considered because, in this area, DR does not yet begin and new high-angle boundaries do not form. The experimental data can be divided into three groups according to the intervals of $\ln Z$. In the region $\ln Z > 40$, where the main role is played by work hardening, there is no corporate dependence for different rates of anvil rotation. According to Fig. 6, d_{av} depends on the true strain in this region: the higher e, the smaller d_{av}. Since for similar values of $\ln Z$, the strain is greater at a rate of anvil rotation of 1 rpm than that at $\omega = 0.3$ rpm, the dependence for the first rate lies lower than for the second one. Experimental values do not differ from each other within the measurement error in the region of the developed DR ($\ln Z < 38.0$).

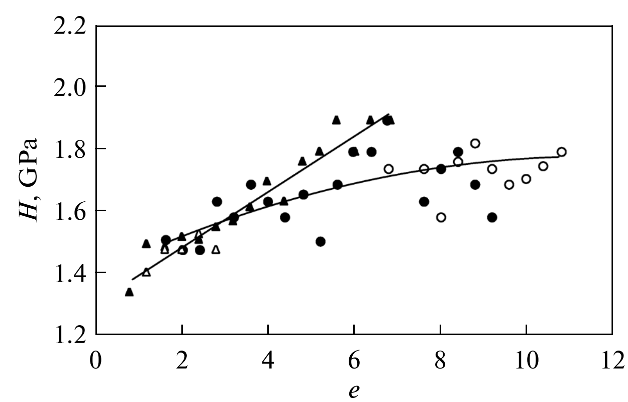

Fig. 7. Dependence of the hardness on the true strain with allowance for stages divided according to $\ln Z$ intervals. (○) $\ln Z < 38$; (●) $38 < \ln Z < 40$; (▲) $40 < \ln Z < 42$; and (△) $\ln Z > 42$.

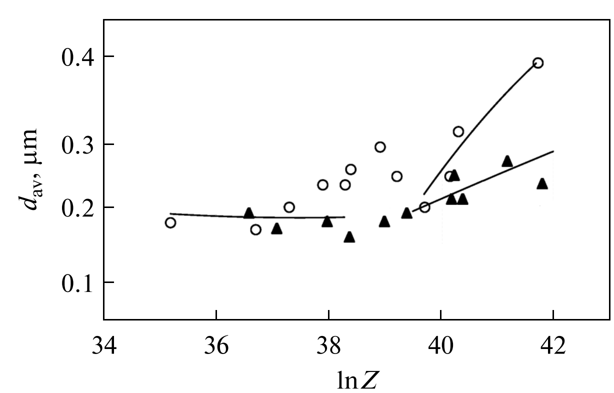

Fig. 8. Average size of structure elements as a function of temperature–compensated strain rate: (○) $\omega = 0.3$ rpm and (▲) $\omega = 1$ rpm.

The grain size stops to change at $40 > \ln Z > 38$ and $\omega = 1$ rpm, whereas the sporadic scatter of values and no noticeable dependence on the deformation conditions are observed at $\omega = 0.3$ rpm. This may be due to the competition between DR and work hardening processes, and PDR that seems to be not suppressed. At a low rate of anvil rotation, the deformed material is long enough under the temperature–strain-rate conditions ensuring DR. On the one hand, this time may be sufficient for a grain to grow in a coarse one during DR until a driving force, which is the gradient of the dislocation density through a moving boundary, is not exhausted. On the other hand, during subsequent PDR, the deformation-induced density of the recrystallization centers capable to grow is likely to lead to the formation of a greater number of grains with sizes coarser than 0.5 μm, which come into collision.

CONCLUSIONS

In commercial-purity copper, impurity dragging prevents grain growth upon postdynamic recrystallization. It allowed the laws of structure formation to be established under conditions of dynamic recrystallization.

Dynamic recrystallization develops at the same true strain ($e = 2$) and in the same range of the Zener–Hollomon parameter as in 99.99 wt %-purity copper. In the range of $35 < \ln Z < 38$, dynamic recrystallization takes place in the entire volume of the material. At $38 < \ln Z < 42$, the volume fraction of the structure that undergoes dynamic recrystallization decreases with increasing $\ln Z$. At $\ln Z > 42$, no signs of dynamic recrystallization were detected. An increase in the rate of anvil rotation impedes the development of dynamic recrystallization. It was established that the hardness and the average grain size depend not only on the temperature–strain-rate conditions of deformation ($\ln Z$), but on the true strain in the commercial-purity copper compared to high-purity copper. Under temperature-rate conditions that ensure work hardening, the hardness increases with strain and structural elements are refined. Under deformation conditions that contribute to dynamic recrystallization, the hardness and structure element sizes achieve the level of saturation.

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