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Effect of Internal Stresses on Copper Creep

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The effect of shear stress on the creep kinetics of Cu (99.95 %) in the temperature range from 373 to 923 K is considered. A coincidence of internal stress estimates obtained using different methods was found, which confirmed the correctness of the creep kinetic equation chosen for data analysis and also made it possible to exclude the effect of deformation point defects, impurity atoms, and piel up of dislocations on the internal stress. It is shown that in the region of relatively high stresses, where the exponential dependence of the creep rate on stress is fulfilled, creep is controlled by self-diffusion, and at high temperatures in the region of the power dependence, it is controlled by diffusion along dislocation cores. In the intermediate temperature region, a change in the slope of the creep rate dependences on stress is observed, while the activation parameters decrease: the self-diffusion energy to diffusion along dislocation cores, and the activation volume value decreases by 3 times. It is concluded that the mechanism of plastic deformation of copper at intermediate test temperatures changes depending on the temperature, the magnitude of the applied stress and the magnitude of the internal stress caused by strain hardening.

Keywords: shear stresses, dislocation density, activation energy, activation volume, self-diffusion, diffusion along dislocation cores, deformation mechanism.

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Introduction

Owing to the smallness of Peierls barriers, the mobility of dislocations in FCC metals is known [1] to be governed largely by local obstacles that could be introduced prior to deformation (e.g., impurity atoms) or formed in the process of deformation (dislocations, dislocation pile up, the precipitation of impurity atoms and point defects at dislocations).

When pure metals are tested in the intermediate temperature region, the dislocation structure is formed at the first stage of creep and induces long-range counter internal stresses τ_i for dislocations moving at the steady-state creep stage [2]. This is the reason why steady-state creep rate $\dot{\varepsilon}$, which varies exponentially with applied shear stress τ, is written in the following form within the range of moderate test temperatures ($(0.3{-}0.5)T_m$) [2]:

$$\dot{\varepsilon} = \dot{\varepsilon}_0 \exp\Big(-\frac{H_0 - V(\tau - \tau_i)}{kT}\Big), \quad (1)$$

where $\dot{\varepsilon}_0$ is the frequency factor, which may be set to $10^7\,\mathrm{s}^{-1}$ [3]; k is the Boltzmann constant; H_0 is the activation energy, which is close to self-diffusion energy Q_{SD} [4] or the boundary diffusion energy [5] in dislocation models; $(\tau - \tau_i)$ is the acting stress; and V is the activation volume.

It has been demonstrated [6] that the change from the low-temperature deformation mechanism associated with conservative dislocation motion to the high-temperature one at test temperature $T \approx 0.5T_m$ is induced by diffusion processes and occurs at stress $\tau = \tau_i$. At the same time, experimental data [7] reveal that dependences $\log\dot{\varepsilon} - \tau$ feature kinks, the nature of which has not yet been fully clarified, at intermediate test temperatures 473–763 K.

The present study is a continuation of research into the influence of internal stresses on the type of dependence of creep rate on stress and the kinetic parameters of Eq. (1). Its aim is to examine the influence of dislocation density on the deformation resistance of copper. The data on the dependence of copper creep rate within a wide range of test temperatures under uniaxial tension reported in [7,8] were used in the present study.

1. Estimation of the magnitude of internal stresses in copper

In the region of high test temperatures, we used the values of stress sensitivity (n) and activation energy (Q) determined earlier in [7] according to the dependence

$$\dot{\varepsilon} = A\Big(\frac{\tau}{G}\Big)^n \exp\Big(-\frac{Q}{kT}\Big), \quad (2)$$

where G is the shear modulus and τ is the shear stress.

In the region of low temperatures, the values of activation volume V, effective (apparent) activation energy H_{eff}, and current activation energy $\mathrm{H}(\tau)$ were estimated:

$$V = kT\frac{\Delta \ln \dot{\varepsilon}}{\Delta\tau}, \quad (3)$$

$$H_{\mathrm{eff}} = kT\,\frac{\ln \dot{\varepsilon}_0}{A}, \quad (4)$$

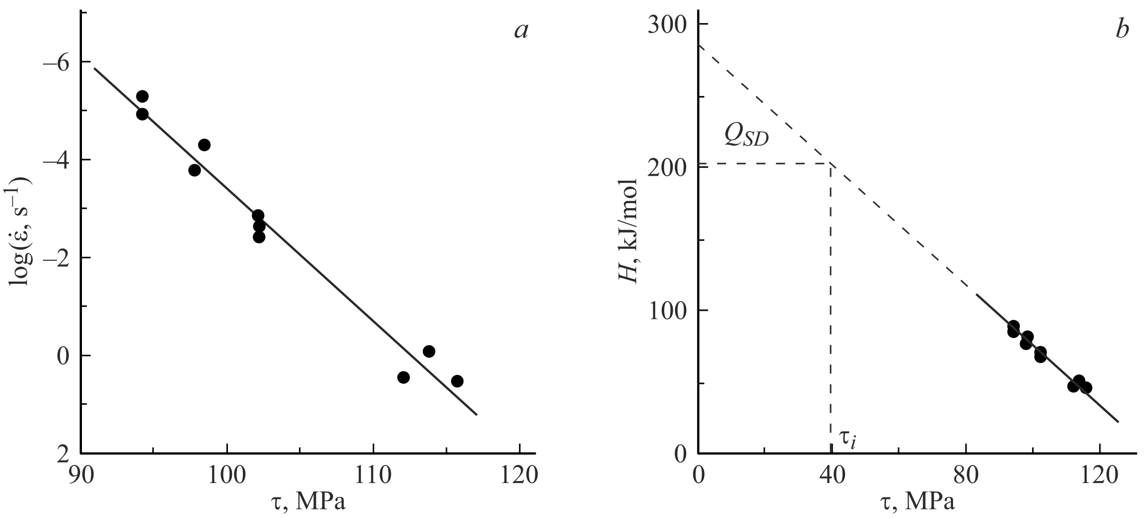

Figure 1. Dependences of creep rate (a) according to [7] and activation energy (b) on the magnitude of shear stress for copper with a grain size of $100\,\mu$m at a test temperature of 373 K.

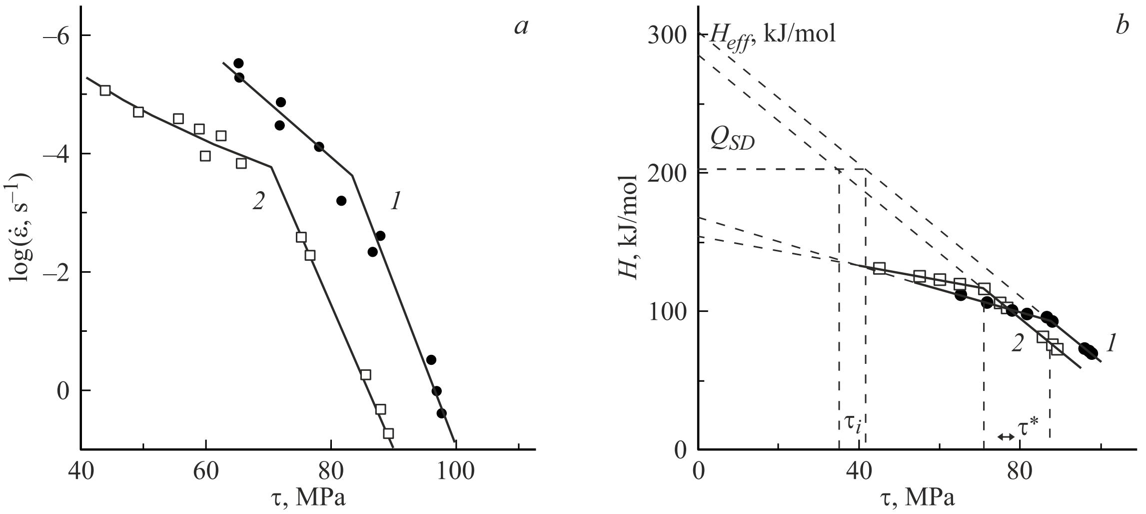

Figure 2. Dependences of creep rate (a; data from [7]) and activation energy (b) on shear stress for copper at test temperatures of 473 (1) and 548 K (2).

where $\ln A$ is the creep rate with extrapolation of straight line $\log\dot{\varepsilon} - \tau$ to stress $\tau = 0$;

$$H(\tau) = kT \ln \frac{\dot{\varepsilon}_0}{\dot{\varepsilon}(\tau)} + V\tau. \qquad (5)$$

The internal stress was determined based on (1) and (4):

$$\tau_i = \frac{H_{\text{eff}} - Q_{\text{SD}}}{V}, \qquad (6)$$

since it follows from Eq. (1) that $H_{\text{eff}} = Q + V\tau_i$.

The calculated τ_i values were compared with the internal stresses calculated from independent measurement data on dislocation density ρ [2]:

$$\tau_i = \alpha G b \sqrt{\rho}, \qquad (7)$$

where $\alpha = (0.2{-}0.4)$ is an athermal constant and b is the Burgers vector. This constant for copper is $\alpha = 0.31$ [9].

2. Estimation of activation parameters

Figures 1–4 present the data on the dependence of creep rate on stress for copper (99.95 %) obtained in [7] and the current values of creep activation energy as functions of the magnitude of shear stress and test temperature. The samples were pre-annealed in vacuum at a temperature of 953 K for 1 h and tested within the temperature range from 373 to 923 K in a helium atmosphere. The grain size after annealing was $100\,\mu$m.

Let us consider the data for copper tested at $T = 373$ K (Fig. 1, a). It is evident that $\dot{\varepsilon}$ depends exponentially on τ; activation volume $V = 2.1$ kJ/(mol·MPa). Having processed the data, we found (Fig. 1, b) that the effective activation energy is 286 kJ/mol. Thus, with self-diffusion activation energy for copper $Q_{\text{SD}} = 203$ kJ/mol, we obtain $\tau_i = 39.5$ MPa.

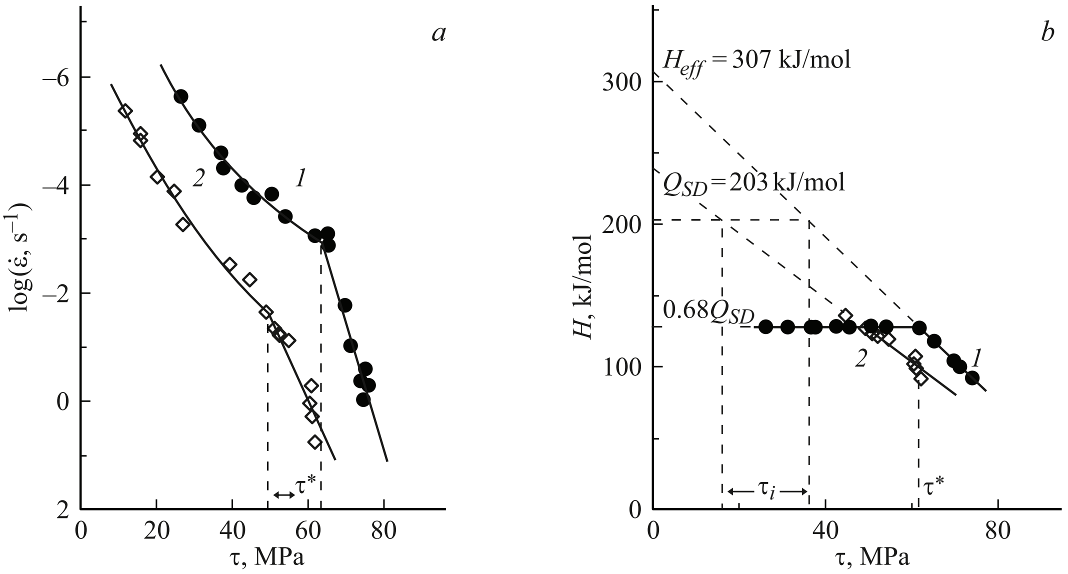

Figure 3. Dependences of creep rate (*a*; data from [7]) and activation energy (*b*) on shear stress for copper at test temperatures of 623 (*1*) and 763 K (*2*).

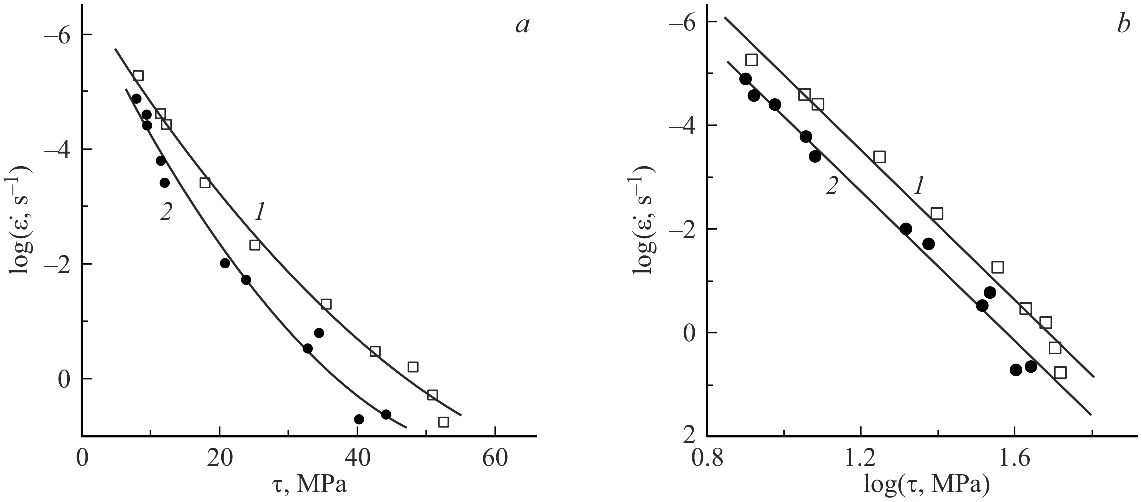

Figure 4. Dependences of creep rate on shear stress for copper plotted based on the data from [7] in semilog (*a*) and log-log (*b*) coordinates at test temperatures of 863 (*1*) and 923 K (*2*).

The test data for copper samples at temperatures of 473 and 548 K are presented in Fig. 2,*a*. It can be seen that the dependences of creep rate on stress obtained at higher test temperatures are significantly more complex in nature: kinks emerge at stresses $\tau \approx \tau^*$, and activation volume V decreases by a factor of approximately 3 in the case of testing at $T = 473$ K. Note that the average value of activation energy under stress $\tau \approx \tau^*$ was $0.5Q_{\mathrm{SD}}$. Figure 2,*b* presents the results of processing of the data from Fig. 2,*a*, which suggest that the values of internal stresses were 41.6 and 35.5 MPa at 473 and 548 K, respectively.

Having processed the data obtained at stresses $\tau < \tau^*$, we found that the effective activation energy was 167 and 153 kJ/mol at 473 and 548 K, respectively.

With a further increase in temperature to 623 and 763 K (Fig. 3), kinks also emerge in dependences $\log\dot{\varepsilon} - \tau$. At stresses $\tau > \tau^*$, the creep rate depends exponentially on stress; at $\tau < \tau^*$, the dependence is a power-law one with coefficient $n = 7$ and creep activation energy $Q = 128$ kJ/mol (according to the data from [7], it is close to the activation energy of pipe diffusion). Our estimates revealed that the magnitude of internal stresses is 34 and 16 MPa at 623 and 763 K, respectively, in the case of an exponential $\dot{\varepsilon} - \tau$ dependence. It follows from Fig. 3,*b* that the transition from an exponential dependence of the creep rate on stress to a power-law one is found at $\tau \approx \tau^*$.

At temperatures exceeding significantly the level of $0.5T_m$ (863 and 923 K), only a power-law dependence of the creep rate on stress is observed in copper (Fig. 4,*a*) with stress sensitivity coefficient $n = 7$ (Fig. 4, *b*). This value is typical of coarse-grained copper under such conditions where creep is effected by the motion of dislocations and is governed by diffusion along dislocation pipes with an activation energy independent of stress, $Q = 128$ kJ/mol [10,11] (Fig. 5).

In addition to the above data for copper with a grain size of 100 μm, we examined the data for copper samples with a grain size of 250 μm. Figure 6 shows the dependences of creep rate on the magnitude of shear stress within the test temperature range of 623–823 K that were plotted based on the data from [8]. At stress $\tau > 20$ MPa, the experimental data follow exponential dependence (1) with effective activation energies of 205, 201, 202, 210, and 215 kJ/mol corresponding to five different curves that illustrate an increase in temperature from 623 to 823 K. Activation volume $V = 0.6$ kJ/(mol·MPa) does not depend on temperature and stress. Therefore, at an average value of activation energy of (207 ± 4) kJ/mol, internal stresses are no greater than 8 MPa.

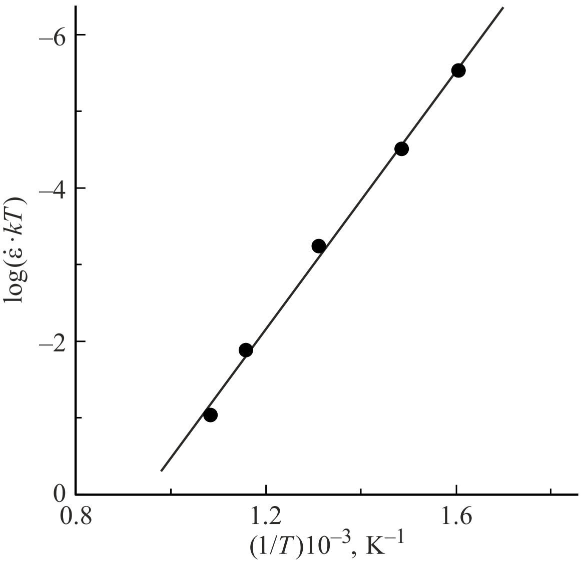

Figure 5. Dependence of strain rate on reverse temperature for copper under stress $\tau/G = 1 \cdot 10^{-3}$.

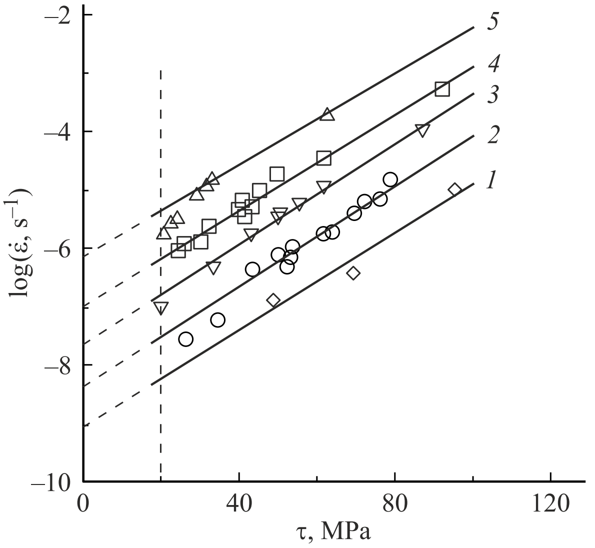

Figure 6. Dependences of creep rate on shear stress for polycrystalline copper (99.999 %) with a grain size of 250 μm at different test temperatures: *1* — 623, *2* — 673, *3* — 723, *4* — 773, *5* — 823 K.

Dependence of the magnitude of internal stresses in copper on test temperature and grain size

Grain size, μm	Test temperature, K	τ_i, MPa
100 [7]	373	39.5
	473	41.5
	548	35.5
	623	34
	763	16
250 [8]	623–823	8

The magnitudes of internal stresses are listed in the table as functions of temperature and grain size. It is evident that the value of τ_i at $d = 100\,\mu$m is virtually independent of temperature within the range of 373–623 K, but decreases sharply at a test temperature of 763 K. At a larger grain size $d = 250\,\mu$m, τ_i is also constant, but significantly smaller in magnitude. Thus, it follows from the presented data that strain hardening caused by the interaction of dislocations in the region of exponential creep has a noticeable effect on the magnitude of effective activation energy of copper creep only in the case of small grain sizes.

3. Discussion

The magnitudes of internal stresses were estimated in accordance with Eq. (1) under the assumption that the mechanism of plastic deformation of copper in the region of average test temperatures is diffusive in nature. It was also assumed that internal stresses are caused only by strain hardening associated with an increase in dislocation density. However, it cannot be ruled out that point defects, which diffuse toward dislocations and impede their motion, and impurity atoms, which tend to pile up at dislocations and form a Cottrell atmosphere, may act as sources of internal stress in copper. Pile up of dislocations, which raise τ_i by a factor of $\sqrt{n'}$ (n' is the number of dislocations in a pile up [2]), may also serve as such sources. Thus, if pile up of 20–40 dislocations, which are necessary for the formation of nucleation pores at the steady-state creep stage [12], are present, the value of τ_i at this stage of creep may increase 5–6 times, potentially compensating for the applied shear stress.

To clarify the nature of internal stresses in copper, we compare the values of τ_i calculated according to Eq. (1) (formula (6)) with the values of internal stresses caused by an increase in dislocation density (Eq. (7)). It can be seen from Fig. 7 that the values of τ_i determined by independent

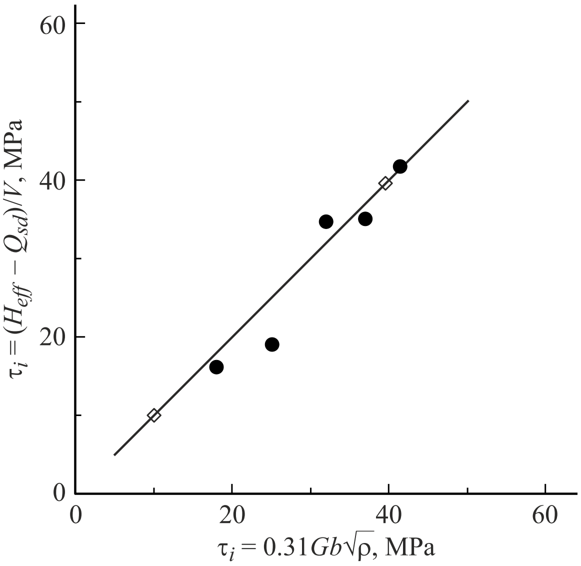

Figure 7. Comparison of the values of internal stresses in copper (with a grain size of $100\,\mu$m) at different test temperatures determined by different methods (●) or based of the value of dislocation density only (◇).

methods match quite well within the entire temperature range where the dependence of $\dot{\varepsilon}$ on τ is exponential. This implies that internal stresses in copper in isothermal tests are apparently governed largely by the increase in dislocation density.

The agreement of the values of τ_i calculated in accordance with Eq. (1) and independent estimates of τ_i based on the change in dislocation density also indicates that Eq. (1) with the specified parameters, pre-exponential factor $\dot{\varepsilon}_0$, self-diffusion energy Q_{SD}, and activation volume V does indeed characterize correctly the behavior of copper samples in the region of intermediate test temperatures.

This conclusion is verified by the experimental data on polycrystalline copper grade M00b [13]. Dislocation substructures (DSs) formed in copper under creep fracture were studied by diffraction electron microscopy. The copper substructure features the following DS types: randomly distributed dislocations (56 %), cellular substructure (36 %), mesh substructure (5 %), band substructure (3 %), dislocation accumulations (3 %), broken subboundaries (2 %); near the fracture zone, the main DS type is the subgrain structure. Scalar dislocation density $\langle\rho\rangle$ was determined separately for each DS type. Its average value was calculated with account for the volume fraction of each DS type in the following way:

$$\langle\rho\rangle = \sum_i P_{\mathrm{Vi}}\rho_i, \tag{8}$$

where ρ_i is the scalar density of dislocations in a certain DS type and P_{Vi} is the volume fraction of a material occupied by this DS type.

It was demonstrated that $\langle\rho\rangle$ is $\sim 2.1\cdot 10^{14}\,\mathrm{m}^{-2}$, $\sim 2.6\cdot 10^{14}\,\mathrm{m}^{-2}$, $\sim 4.4\cdot 10^{14}\,\mathrm{m}^{-2}$, and $\sim 1.1\cdot 10^{14}\,\mathrm{m}^{-2}$ in copper grains and in the cellular substructure, in the band substructure, in the mesh DS, and in the subgrain structure, respectively.

Note that the formation of twin structures was not observed in the examination of substructures of deformed copper. The emergence of twins (just as other types of reorientation) is associated with the formation of local fields of internal stress and the subsequent formation of cracks. Mechanical twinning in copper is one of the predominant deformation mechanisms when slip deformation is hindered (e.g., under shock-wave loading). Specifically, it was demonstrated in [14] that deformation twins form at the early stages of high-speed loading ($\sim 10^4\,\mathrm{s}^{-1}$) along with cells with low-angle boundaries and grains with high-angle boundaries. With further deformation, twins become distorted and form high-angle boundaries of arbitrary orientation.

The force and temperature dependence of creep rate may be characterized formally by the Zhurkov equation [15]:

$$\dot{\varepsilon} = \dot{\varepsilon}_0 \exp\left(-\frac{U_0 - \gamma\sigma}{kT}\right), \tag{9}$$

where the following values of kinetic parameters were obtained for copper in the exponential region [7]: $\dot{\varepsilon}_0 = 10^{12}\,\mathrm{s}^{-1}$, $U_0 = 323.5\,\mathrm{kJ/mol}$, and structural parameter $\gamma = 0.65\,\mathrm{kJ/(mol{\cdot}MPa)}$ for normal stresses and $\gamma = 2.0\,\mathrm{kJ/(mol{\cdot}MPa)}$ for shear stresses. Note that $\gamma = \mathrm{V/m}$ and $\sigma = \tau\cdot m$, where $m = 3.06$ is the Taylor factor.

It has already been concluded in [16] that the behavior of FCC metals under load is governed by shear stresses, the magnitude of which determines the mobility of dislocations, rather than by normal ones. The shear stress acting on a given dislocation segment has two components: applied shear stress τ and internal stress τ_i, which is characterized as a measure of the retarding effect of the structure on a slip dislocation.

It follows from the data presented in [7] that the value of U_0 is comparable with the effective activation energy ($\cong 300\,\mathrm{kJ/mol}$) determined without regard to the effect of internal stress. This implies that the conclusions regarding the mechanism of fracture and plastic deformation made on the basis of expression (9) are insufficiently substantiated. For example, it was concluded [15] that the process of fracture associated with the rupture of interatomic bonds and the subsequent formation of micro-discontinuities is initiated in a uniaxial stress state at the second stage of creep. However, literature data [17–21] are indicative of a different fracture mechanism. It is known [22,23] that the uniaxial stress state in a homogeneous part of the sample changes upon the formation of a neck to a triaxial stress state with average stress σ_m equal to one third of the sum of principal stresses:

$$\sigma_m = (\sigma_1 + \sigma_2 + \sigma_3)/3 \tag{10}$$

where σ_1 is the axial stress, σ_2 is the radial stress, and σ_3 is the tangential stress.

Hydrostatic tensile stress σ_m reduces the vacancy formation energy, potentially promoting the formation of pores along the neck axis, where the σ_m value is estimated [22] to reach its maximum. The coalescence of pores with the formation of a main crack occurs under the condition of overlap of stresses between pores (the distance between the pores becomes equal to the pore size [17,24]).

It has also been demonstrated [18] that when tensile pressure in the neck is compensated by external hydrostatic pressure, the number of pores at the cleavage site decreases. With a further increase in pressure, pores do not form, and a transition from cleavage fracture to shear fracture is observed [25,26].

Note that a texture, which has a noticeable effect on the formation of highly misoriented fragments [27], is produced in the neck under large deformations. It was noted [27] that the identification of the relation between crystallographic texture and fragmentation has been discussed at length in literature [28–35].

It was demonstrated in [36,37] that the initial orientation of grains exerts a significant influence on the deformation-dislocation structure of copper under tension even at a strain degree of 0.3. Copper samples (99.999 %) with a grain size of $190\,\mu$m were pre-deformed at 18 °C to a true strain of 0.22. It was found in the process of subsequent tensioning that the microstructure forms non-uniformly throughout the sample volume and depends on the orientation of grains. Significant heterogeneity (down to the formation of fragments) was observed in grains with orientation [100] parallel to the tension axis. The other microstructure with [111] grains does, unlike the previous one, retain the grain orientation. This structure also features a high dislocation density (50 %−100 % higher than in the [100] grain structure).

The evolution of copper microstructure in the process of uniaxial tension under large plastic deformations in the neck region within the range of true strain from 0.45 to 1.15 was investigated in [27]. It was found that a texture consisting of two components forms in copper alongside with the deformation microstructure. A crystallographic texture with grains oriented predominantly in the [100] and [111] directions parallel to the tension axis did form already at a strain of 0.45. As the deformation in the neck increased, the texture became sharper: the maximum pole density increased approximately by a factor of 1.5. It was found that grains belonging to the [100] component deform easily and break into highly ($\sim 15^\circ$) misoriented fragments as the deformation increases. At the same time, grains with the [111] orientation retain a uniform orientation. Thus, it was found that large plastic deformations of copper result in the formation of a microstructure in the neck consisting of low-angle ($> 5^\circ$) dislocation cell boundaries and high-angle fragment boundaries ($\theta > 15^\circ$) of deformational origin, which, according to [28], give rise to micro-discontinuities.

Note that the Zhurkov equation is not the only expression characterizing the dependence of creep rate and life time on stress and temperature. Another empirical relation is the power equation proposed in [38]:

$$\dot{\varepsilon} = A\left(\frac{\tau}{G}\right)^n \left(\frac{b}{d}\right)^p \exp\left(-\frac{Q_c}{RT}\right), \tag{11}$$

where n and p characterize the dependence of creep rate on stress and grain size (d); parameter n varies from 4 to 7; parameter p varies from 0 to 1; and Q_c is the creep activation energy. Data obtained within a wide temperature and microstructure evolution range [39–42] were processed with account for the effective stress $(\tau - \tau_i)$ and the dependence of pre-exponential factor A on the diffusion coefficient and temperature, and it was found that the value of n of most metals becomes equal to $4-5$ at temperatures $T \approx 0.5T_m$, while the creep process is controlled by lattice diffusion. With a coefficient of $n+2$, creep is governed by diffusion along dislocation cores. At lower temperatures and $n > 7$, the creep rate depends exponentially on stress (with the activation energy also depending on stress).

It follows from the above data for copper that the true value of the creep activation energy at test temperatures from 373 to 763 K within the $\tau > \tau^*$ stress range is equal to the self-diffusion energy (when data are processed in the $\log\dot{\varepsilon} - (\tau - \tau_i)$ coordinates). At test temperatures of 623–923 K and stresses $\tau < \tau^*$, the dependence of the creep rate on stress may be characterized by a power law with parameter $n \approx 7$. The activation energy turned out to be equal to the activation energy of pipe diffusion (128 kJ/mol).

At test temperatures of 473 and 548 K, Q_c was found to be equal to (160 ± 7) kJ/mol. A close value of Q_c was obtained for copper under torsion within the same temperature range (from ~ 400 to ~ 600 K). However, the creep activation energy at test temperatures outside the specified range was 128 kJ/mol [43]. It is fair to assume that segregation of mobile non-equilibrium vacancies and impurity atoms to dislocations occurs within the specified temperature range, which leads to anomalous values of the activation energy in the temperature dependence of Q_c.

Conclusions

The examined data on the influence of internal athermal stresses caused by an increase in dislocation density on the kinetics of creep in copper within a wide range of test temperatures suggest the following conclusions:

1. It was demonstrated that the restriction of dislocation mobility as a result of dislocation-strain hardening has a significant effect on the kinetic parameters of copper creep (creep activation energy and activation volume); in other words, it affects the mechanism of plastic deformation.

2. It was demonstrated that the creep activation energy is equal to the self-diffusion energy in the region of temperatures and stresses where the dependence of creep rate on stress is exponential.

3. It was demonstrated that the formation of kinks in the force dependence of creep rate on stress is accompanied by a reduction in activation energy and activation volume.

Conflict of interest

The authors declare that they have no conflict of interest.

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