Programmed Strengthening of Crystalline Materials Using Copper and Aluminum as an Example

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Abstract—Fundamental propositions of the well-known method of programmed strengthening of crystalline solids are described. It is shown that when mechanical (deformation) and thermal (tempering, annealing, and aging) actions are combined, the loading rate is determined by the rate of diffusion and microshear processes of relaxation of internal stresses in regions of defect accumulation and by the formation of stable complexes as a result of directional diffusion of point defects.

INTRODUCTION

Annealing in a stressed state is one of the ways of realization of the diffusion and microshear strengthening of crystalline materials. The thermal and mechanical activation of diffusion and microshear mechanisms of relaxation of local stresses favors the formation of an energetically equilibrium structure of materials that is more homogeneous in stresses. As this takes place, it is important to detect in crystals regions of low internal stresses and to strengthen them. It turned out that this problem can be solved by changing the load upon annealing of the material or the construction. The character of changes in the magnitude of load should take into account at each time instant the rate of the processes that ensure relaxation of overstresses and strengthening of weak sites.

The mechanical properties of a crystal in the macroelastic region of deformations are determined by its structure. Of special interest from the viewpoint of investigation of the dislocation-structure evolution is the case where no dislocation multiplication in the whole volume of the crystal takes place. In this instance, there occur only displacements, annihilation, and escape of separate unpinned dislocations to grain boundaries or generation of dislocations in local overstressed regions at early stages of loading (deformation).

FEATURES OF PHYSICAL PROCESSES UPON PROGRAMED STRENGTENING

In real crystals, there exists a broad range of critical stresses and potential barriers for moving dislocations, which are elementary carriers of shear deformation. The stresses at which the elementary acts of plastic deformation start in the crystal substantially depend on the character of the internal-stress distribution in the magnitude of potential barriers both in height and in spatial arrangement. Figure 1 depicts a theoretical curve of the distribution of potential barriers in height [1], and Fig. 2 displays the obtained experimental values of start stresses for dislocations [2–4].

The nucleation and displacement of dislocations in some regions of defect crystals are possible at stresses that are lower than the magnitude of the yield strength. The yield strength is defined as the stress at which an intense dislocation multiplication begins and the dislocations overcome obstacles by the «by-passing» or «cutting» mechanism (Fig. 3).

Investigations of the microplasticity of crystals make it possible to clarify the nature of sources and laws of the formation of dislocation pileups, stress relaxation as a result of their interaction with each

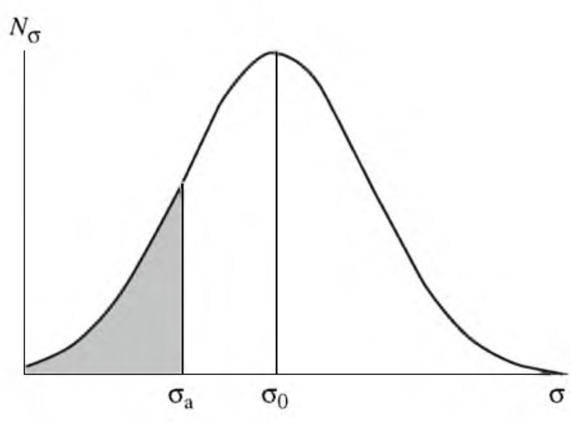

Fig. 1. Distribution of plastic elements N_σ in magnitude of critical stresses σ (Gaussian distribution).

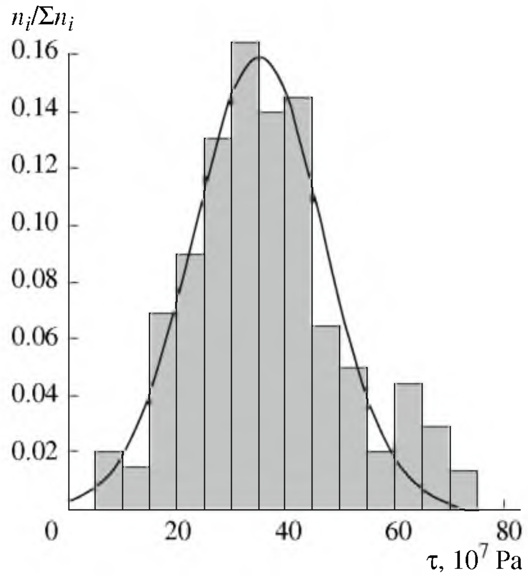

Fig. 2. A typical histogram of start stresses τ of dislocations in crystals.

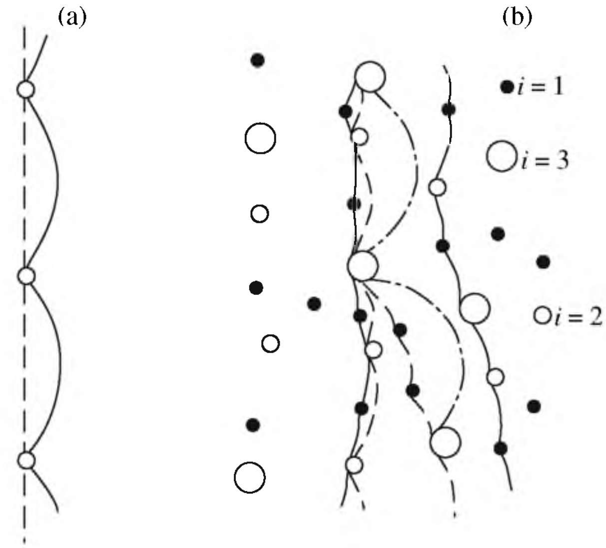

Fig. 3. A schematic of dislocation motion under the action of an applied stress and break-away from pinning points in the case of (a) one and (b) three types of barriers.

other, and their transition into a more advantageous energetic state. The processes of microdeformation determine the magnitude of the macroscopic yield strength, tendency to brittle fracture of materials, and the behavior of materials upon creep, relaxation, and fatigue tests.

The suppression of processes of microplasticity favors an enhancement in the yield strength and an improved reproduction of the shape of articles upon multiple loadings at loads below the yield strength. Thermal, mechanical, radiation, electromagnetic, and other types of actions disturb the initial quasi-equilibrium state of crystalline solids and favor the occurrence of a variety of physical processes that result in changes and redistribution of internal stresses.

The relaxations of local stresses and microplasticity in the process of annealing, tempering, and aging of materials and articles made of them proceed appreciably more rapidly under the action of a continuously increasing load in the macroelastic range of deformation. The loading rate in this case must correspond to the rate of diffusional and microshear relaxation of local stresses. Such a thermomechanical action was given the name of programmed loading [5–8].

Upon programed loading, owing to a slowly increasing external load, on the one hand, there arises plastic deformation in the region of weak sites and, on the other hand, at a certain tempering temperature and a loading rate there occurs strengthening of these sites at the expense of diffusional flow of point defects that contribute to a partial redistribution and pinning of dislocations. Simultaneously, annihilation of mobile dislocations takes place.

The main condition for the programmed strengthening is that the equality between a growing external load and the internal resistance to deformation of the material be maintained at each level of the load. The change in the magnitude of the applied load $\sigma(t)$ can be written as

$$\sigma(t) = \int_0^t \sigma_n dt, \tag{1}$$

where σ_n is the rate of increasing stress upon programmed loading.

The employment of this method of strengthening is connected with difficulties of determining the precise details of the loading process.

The rate of programmed loading can be determined from the character of the behavior of internal friction, electrical resistance, and other properties that are unambiguously correlated to the rate and degree of the occurrence of diffusion processes in metals under load. This can be effected on a special setup for programmed loading with feedback [9]. It is known from the literature data that in order to enhance the structural strength of high-pressure vessels, the programmed loading was performed at a rate proportional to the rate of changes in the heat content and specific volume of the material [10–12].

For experimentally determining the regime of programmed loading, the method of step-by-step relaxation with return to the start of the steady-state stage at each step of loading can be used. The line that was drawn through the initial points of the steady-state stage of curves of step-by-step relaxation corresponds to temporal changes in the load that are necessary upon the strengthening regime of loading (Fig. 4). It is

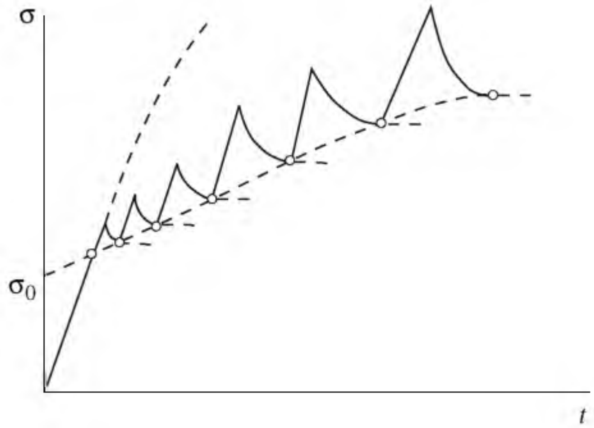

Fig. 4. Determination of the rate of programmed loading using the step-by-step stress relaxation with return to the start of the steady-state stage at each step of loading: σ_0 is the initial stress above which the loading should follow this program; and t is the time.

important to note that this method, simultaneously, registers the level of stress σ_0 beginning from which the loading should be performed at a rate corresponding to the program of loading for the metal under study.

EXPERIMENTAL INVESTIGATIONS

The experiments were performed on samples of an oxygen-free copper and a polycrystalline aluminum of purity 99.995%. The samples of the first batch were loaded to $0.9\sigma_{0.2}$ ($\sigma_{0.2}$ is the 0.2 offset yield strength) at temperatures of 300 K for aluminum and at 400K for copper, with subsequent stress relaxation. The samples of the second batch were loaded starting from $0.5\sigma_{0.2}$ to a value that is not higher than the yield strength of metal at these temperatures, with stress relaxation after each step of loading. The third batch was treated using the above-suggested method. The residual deformation in all of the cases did not exceed 0.3–0.4%.

The results of measurements of the relative increment of the yield strength $\Delta\sigma/\sigma_{0.2}$ for the samples that were subjected to different regimes of thermomechanical treatment are given in the table. As follows from these results, the greatest effect of enhancement in the yield strength is observed in the samples that were treated using the above-suggested method.

The temperature range was limited by the conditions of the occurrence of intense diffusion and microshear processes of stress relaxation. In this situation, there should arise stable complexes of point defects in the process of their directional diffusion and redistribution of dislocations in elastic-stress fields. It is known that at high temperatures of loading the diffusion processes become more intense, and the strengthening is reduced, which is connected with a decrease in the probability of the formation of stable complexes of point defects at dislocations. This is confirmed by the results of the experiment performed on polycrystalline aluminum at a temperature of 400 K. No positive effect was found both after accomplishment of relaxations and upon treatment by the suggested method. With decreasing temperature, the diffusion processes are suppressed, and the strengthening of crystals upon programmed loading is mainly affected by microshear processes of the redistribution of dislocations and by the escape of dislocations with small start stresses to the interphase boundary [13].

ANALYSIS OF EXPERIMENTAL DATA

It is known that many dependences of strength characteristics on the strain rate, duration of the load application, and temperature were obtained without taking into account the processes of stress relaxation and recovery of continuity of the materials in the stressed state, which increase the resistance to deformation. For this reason, in metals and alloys in certain temperature ranges there is observed a deviation of the temperature–strain-rate dependence of the yield strength and flow stress from these consistent with the classical concepts on the occurring processes described in the literature.

As is seen from Fig. 5, the critical shear stresses of single crystals of aluminum (Fig. 5a) and copper (Fig. 5b) depend in a complex way on the strain rate. In

Table

Batch	Material	Method of treatment	The number of steps	T, K	$\frac{\Delta\sigma}{\sigma_{02}}$, %
1	Aluminum	Stress relaxation	1	300	8
2	Aluminum	Stress relaxation	5	300	13
3	Aluminum	By the suggested method	–	300	19
4	Copper	Stress relaxation	1	400	17
5	Copper	Stress relaxation	5	400	23
6	Copper	By the suggested method	–	400	32

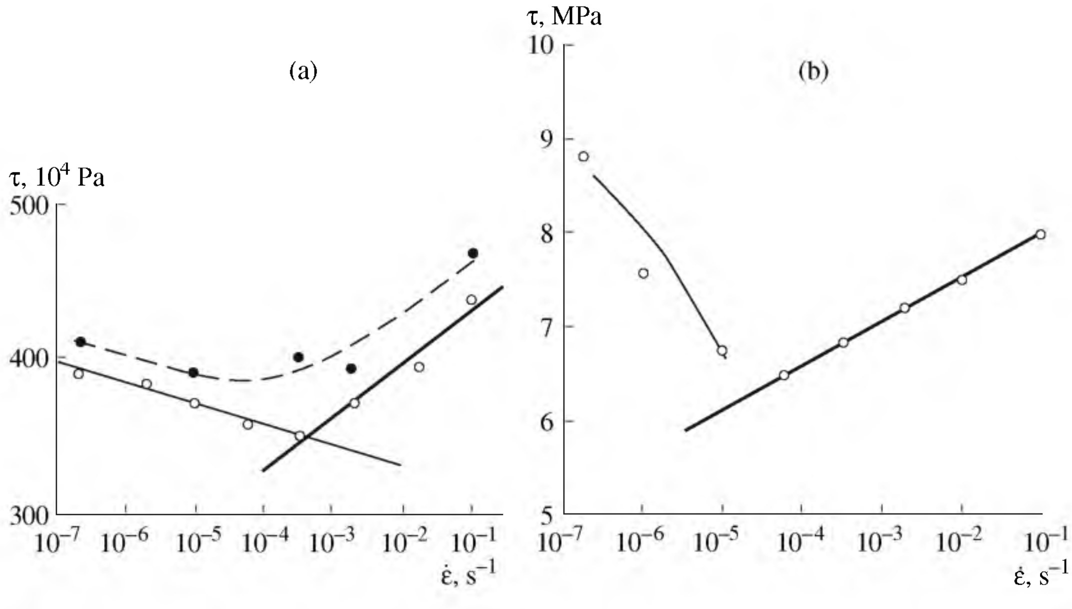

Fig. 5. (a) Dependence of critical shear stresses on the strain rate of aluminum single crystals at temperatures of (○) 300 and (●) 77 K. Line (—) corresponds to the dependence $\tau = 0.404 \times 10^7 – 0.384 \times 10^7 \sqrt{\dot{\varepsilon}}$; line (—), to the dependence $\tau = 0.464 + 0.34 \times 10^{-9} \ln \dot{\varepsilon}$. The numerical values of the coefficients in the equations are obtained by the least squares method. The dashed line corresponds to the envelope curve that was drawn through experimental points (●) for 77 K. (b) Dependence of critical shear stresses on the strain rate of copper at a temperature of (○) 300 K. Line (—) corresponds to the dependence $\tau = 0.992 \times 10^7 – 0.401 \times 10^8 \sqrt{\dot{\varepsilon}}$; line (—), to the dependence $\tau = 0.845 \times 10^7 + 0.467 \times 10^6 \ln \dot{\varepsilon}$. Numerical values of the coefficients in the equations are obtained by the least squares method.

a strain-rate range of 10^{-4}–10^{-1} s^{-1}, the observed dependence of τ_0 on $\ln \dot{\varepsilon}$ is linear. A further reduction in the strain rate is accompanied by a deviation from this dependence.

If we assume that the flow stress is mainly determined by the mechanism of thermally activated cutting of obstacles by dislocations in the slip plane in the range of high strain rates ($\dot{\varepsilon} \geq 10^{-4}$ c^{-1}), then the $\tau(\dot{\varepsilon})$ dependence can be described by the following equation [14]:

$$\tau = \frac{1}{V}\left(E_0 + kT \ln \frac{\dot{\varepsilon}}{\dot{\varepsilon}_0}\right) + \tau_G, \qquad (2)$$

where V is the activation volume, E_0 is the activation energy for the processes of cutting, τ_G is the internal long-range stress, $\dot{\varepsilon}$ is the strain rate; $\dot{\varepsilon}_0$ is the frequency factor, and k is the Boltzmann constant.

In order to explain the observed deviation of $\tau(\dot{\varepsilon})$ at $\dot{\varepsilon} \leq 10^{-4}$ s^{-1} from the dependences consistent with classical concepts, it is necessary to introduce an additional term into expression (2), which could take into account the appearance of new centers of dislocation pinning at small loading rates at the expense of migration of point defects from the surrounding volume. The change in the concentration of impurities c_{imp} that have penetrated, owing to diffusion, from the crystal volume with a concentration c_0 for the time Δt that is smaller than the saturation time is determined by the formula [14]

$$\Delta c_{imp} = \frac{\pi c_0}{b^2}\left[\frac{n(n+2)DWb^n \Delta t}{kT}\right]^{\frac{2}{n+2}},$$

where c_0 is the equilibrium value of the concentration of point defects; D is the diffusion coefficient of point defects near the dislocation; W is the energy of interaction of the dislocation with the defect; n is the number equal to 1 or 2 ($n = 1$ when the size effect prevails; and $n = 2$ when the elastic constants in the energy of interaction of the impurity with the dislocation are dominant); and b is the Burgers vector.

Then, using the Orowan formula for the elastic limit $\tau \approx \frac{2Gb}{l}$, we obtain the expression

$$\tau = 2Gb\left\{\frac{1}{\lambda_0} + \frac{\pi c_0}{b^2}\left[\frac{n(n+2)DWb^n t}{kT}\right]^{\frac{2}{n+2}}\right\}, \qquad (3)$$

where λ_0 is the initial spacing between the obstacles and D is the diffusion coefficient of point defects near the dislocation.

With taking into account this term in the dependence of the stress for motion of dislocations on the strain rate (assuming that the time of "standing" of a dislocation at obstacles is $t = \frac{\alpha}{\dot{\varepsilon}}$, where α = const), we obtain

$$\tau = C + A\ln\dot{\varepsilon} + B\dot{\varepsilon}^{-\frac{n}{n+2}}, \tag{4}$$

where $C = \frac{E_0 - kT\ln\dot{\varepsilon}_0}{V} + \tau_G + \frac{Gb}{\lambda_0}$, $A = \frac{kT}{V}$, $B = \frac{\pi G c_0}{b^2} \times \left(\frac{n(n+2)DWb^n\alpha}{kT}\right)$.

According to Eq. (4), at high strain rates it is the second term that is determining. This is supported by the linear dependence of τ on $\ln\dot{\varepsilon}$ in Figs. 5a and 5b at high strain rates. The numerical values of the coefficients in these equations are obtained by the least squares method. For aluminum, the equation obtained has the form of $\tau = 0.464 + 0.34 \times 10^{-9} \ln\dot{\varepsilon}$; for copper, $\tau = 0.845 \times 10^7 + 0.467 \times 10^6 \ln\dot{\varepsilon}$. The coefficient at $\ln\dot{\varepsilon}$ is equal to $A = \frac{kT}{V}$. The numerical values obtained make it possible to calculate the activation volume. For aluminum this activation volume is 0.10×10^{-23} m^3; for copper, 0.88×10^{-26} m^3.

At small strain rates, when diffusion processes of the formation of centers of pinning (and obstacles) of dislocations play a significant role, the $\tau(\dot{\varepsilon})$ dependence is mainly determined by the third term, according to which the critical shear stress τ_0 should grow with decreasing strain rate. In single crystals of aluminum, at $\dot{\varepsilon} \le 10^{-6}$ s^{-1}, the observed τ_0 value tends to saturation (Fig. 4), which appears to be a consequence of a «viscous» motion of dislocations (microcreep) [15].

The magnitude of the critical strain rate, below which diffusion strengthening manifests itself markedly, is determined by the minimum condition $\frac{d\tau}{d\dot{\varepsilon}} = 0$

$$\dot{\varepsilon} = \left(\frac{2}{n+2}\frac{B}{A}\right)^{\frac{n+2}{2}}. \tag{5}$$

The values of $\dot{\varepsilon}$ calculated by the formula (5) are in good agreement with those determined experimentally [16].

The thus-obtained expression (5) can be used for choosing the rate of programmed loading of metals. Since the programmed loading is effected in the range of the applicability of Hookes's law (the residual deformation upon programmed loading is smaller than 0.1–0.2%), the rate of programmed loading $\dot{\sigma}_n$ must be

$$\dot{\sigma}_n < E\dot{\varepsilon} = E\left(\frac{2}{n+2}\frac{B}{A}\right)^{\frac{n+2}{2}}. \tag{6}$$

According to the $\tau(\dot{\varepsilon})$ dependence (Fig. 5b), a considerable contribution to diffusion strengthening of the copper single crystals under study begins at $\dot{\varepsilon} < 2 \times 10^{-4}$ s^{-1}, which corresponds to the loading rate $\dot{\sigma} < 3 \times 10^6$ Pa/h. With decreasing temperature, the magnitude of the loading rate at which diffusion strengthening starts manifesting itself is reduced.

CONCLUSIONS

The effectiveness of the method of programmed strengthening for enhancement of the yield strength of copper and aluminum has been shown. The enhancement in the strength characteristics is accounted for by the processes of relaxation of internal stresses and by the formation of stable complexes of point defects at the expense of their diffusion. The loading rate upon programmed strengthening has been estimated.

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