AGING OF QUENCHED ALUMINUM AFTER MINOR DEFORMATION AT 77°K

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The low-temperature aging of quenched aluminum deformed at various rates to a minor degree was investigated in this study. Using the theory of Coulomb–Friedel, it was possible to estimate the coefficients of vacancy diffusion along the dislocation center and from the bulk towards dislocations.

A number of papers [1-3] have indicated the possibility of a substantial increase of the yield point of pure metals with h.c.p. and f.c.c. lattice, after their rapid quenching from premelting temperatures. This phenomenon, which was named "the effect of quench hardening," was described in the survey of Kimura and Maddin [4]. Similarly as radiation hardening, quench hardening is the result of adsorption of the excess of point defects (vacancies) on dislocations and on secondary defects which originated with condensation of vacancies [4]. The character of changes with time in the yield point of metals after quenching, and in internal friction depending on the rate of quenching [3], indicates that the basic cause of hardening of quenched metals is not the presence of thermal stresses, but the locking of dislocations by point defects. In the purer state, the kinetics of interaction between point defects and dislocations can be studied at low temperatures, but such at which the processes of redistribution of point defects already proceed with adequate intensity. Thus, Birnbaum [5, 6] has studied low temperature aging (at 77°K) of highly deformed copper, and, from the curves of changes in the yield point depending on the time and temperature of aging, has determined the basic parameters characterizing the nature and mobility of point defects originated at deformation. Gindin and Neklyudov with co-workers [7, 8] have studied the aging of quenched specimens of alloy D-1 and high purity aluminum, after minor deformation at 77°K. It has been shown that from the change of the yield point with time of aging of aluminum deformed after quenching, the coefficient of vacancy diffusion in the bulk close to the dislocation center and along the line of dislocation can be evaluated.

Data from the studies on low temperature aging of quenched aluminum, after high and low rates of loading proceeding to various degrees of deformation at 77°K, are reported in the present paper.

METHOD

Flat specimens 2 × 1 mm in section, with 16 mm long working part made from polycrystalline aluminum of 99.996% and 99.8% purity, were used for the study. First, the specimens were annealed at 500°C for one hour. Nonequilibrium concentration of vacancies was produced by quenching the specimens from 550°C in water. According to calculated evaluation, the concentration of vacancies in the specimens had reached in this case from 10^{-5} to 10^{-6}.

Some of the high purity aluminum specimens were quenched from the temperatures of 500, 600, and 650°C. After quenching and cooling to 77°K, the specimens were at once deformed to a predetermined degree, the load was removed, and, after a certain period of aging time at the deforming temperature, they were loaded again. Elongation of specimens was carried out on a small installation which, due to the speed variator, made it possible to carry out loading in a wide range of rates, from 10 to 10^6 gf/mm^2 per hour at temperatures from 77 to 1000°K.

Recording of stress–strain diagrams was conducted on a light-sensitive film using an optical sensor. Furthermore, the possibility of recording the stress–strain curves on a chart strip of the recorder, using

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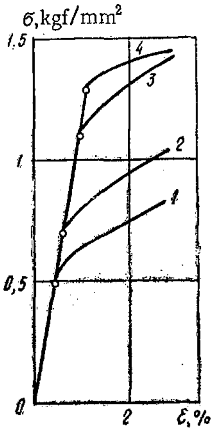

Fig. 1

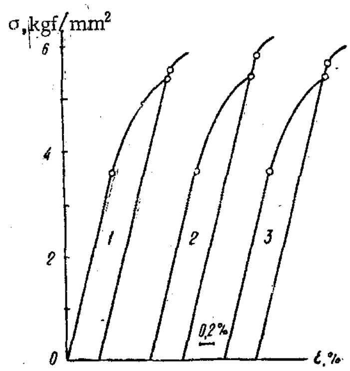

Fig. 2

Fig. 1. Initial sections of load–elongation curves of pure aluminum specimens at room temperature; original (1) and after quenching from 500°C (2), 600°C (3), and 650°C (4).

Fig. 2. Load elongation curves for quenched specimens of aluminum with intermediate stress relief and annealed at 77°K for 10 min (1), 50 min (2), and 80 min (3).

strain gauges cemented to the thrust ring of the dynamometer, has been provided in the installation. The use of sensitive transducers made it possible to measure the changes in the load with an accuracy of ±5 gf/mm^2. The flow stress before and after deformation of quenched specimens was determined from the starting point of the deflection of the load–elongation curve from the straight line which virtually corresponded to the elastic area of strain.

RESULTS

To illustrate the effect of quench-hardening, Fig. 1 presents the load–elongation curves taken at room temperature for high purity aluminum samples after annealing and quenching. When the specimens were quenched from premelting temperature, the yield point of aluminum increased by a factor of 2.5. At larger strains, the differences between the elongation curves of all specimens were small. When the temperature of quenching was lowered, the effect of hardening and the degree of its time and temperature stability was reduced. To study the process of interaction between vacancies and dislocations, samples quenched from 550°C were used. From the curves of re-straining at 77°K the increase of flow stress was determined, and graphs of its dependence on the aging period were plotted. The main results on the interaction of vacancies with dislocations in quenched high purity aluminum were previously described by the authors [8].

The load–elongation curves for commercial aluminum specimens after quenching and intermediate 0.5% deformation, with aging for 10, 50, and 80 min, are presented in Fig. 2. In all cases, repeated deformation was accompanied by the increase of flow stress which, in a complex way, depends on the duration of aging (Fig. 3a). The curves $\Delta\sigma(t)$ are characterized by the presence of a maximum whose magnitude and time of attainment are determined by temperature of deformation [8], purity of the metal, and rate and magnitude of preliminary deformation (Figs. 3, 4). With the increase of the degree of deformation of the quench-hardened specimens, the magnitude of maximum increase in flow stress and the time of its attainment are reduced (Fig. 4). After 0.5% deformation of quenched high purity aluminum, and aging at 77°K, $\Delta\sigma_m$ amounts to 90 gf/mm^2, and is attained after 30 min. In specimens from commercial aluminum, treated in the same manner, $\Delta\sigma_m \approx 500$ gf/mm^2, and $t_{max} = 50$ min.

The rate of loading the specimens after quenching exerts a substantial influence on the type of the dependence $\Delta\sigma(t)$. With small rates of loading ($\approx 10^3$ gf/mm$^2 \cdot$h), resistance to deformation is noticeably increased in the quenched samples (Fig. 5). While, with the loading rate of 10^6 gf/mm^2, the stress $\sigma_{0.5}$ of quenched aluminum equals 5 kg/mm^2, at slow deformation it amounts to $\approx$7 kg/mm^2. Aging of samples at 77°K after slow loading is virtually not accompanied by the change of flow stress on repeated deformation

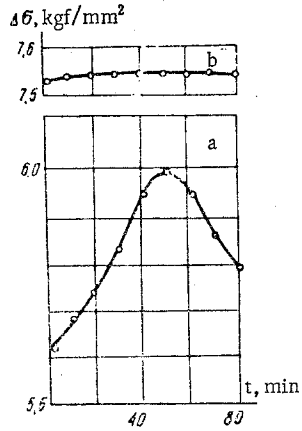

Fig. 3

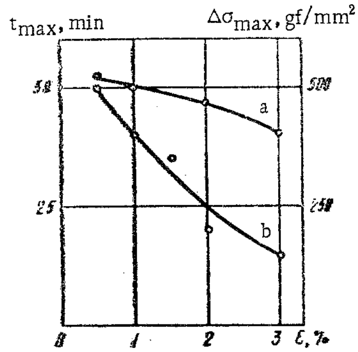

Fig. 4

Fig. 3. Dependence of the increase in flow stress ($\Delta\sigma$) of quenched aluminum on the duration of aging at 77°K after rapid (a), and slow (b) 0.5% loading.

Fig. 4. Dependence of the magnitude of maximum increases $\Delta\sigma_{max}$ (a), and of time t of attainment of maximum value in the increase of stress (b) at low temperature aging, on the magnitude of preliminary deformation.

(Fig. 3, curve b). Hardening of quenched aluminum as a result of low-temperature aging under smoothly increasing load is more stable at subsequent reheating to room temperature (Fig. 5, curve 2b). The flow stress in commercial aluminum specimens, quenched in liquid nitrogen and slowly deformed to 0.5%, equals 6.2 kg/mm^2 after reheating, but in rapidly deformed specimens it equals approximately 3.8 kg/mm^2.

DISCUSSION OF RESULTS

According to the classical theory of Coulomb–Friedel [9], the most likely hardening mechanism of quenched metals is the elastic interaction of dislocations and vacancies. The latter, similarly as the atoms of impurities, form complexes of the type of Cottrell atmospheres around the dislocations. In contrast to the impurity atmospheres, the vacancy atmospheres can disintegrate due to the annihilation of vacancies on the jogs of dislocations and other sinks. Stable quench-hardening is possible only in such a case when the vacancies are condensed into small cavities which efficiently arrest dislocations. Furthermore, in the quenched metals there is a large number of dislocation loops and micropores which exert strong resistance to the motion of dislocations. Apparently, the quenching of high purity aluminum from premelting temperature facilitates both the formation of cavities on dislocations and the occurrence of dislocation loops. Maddin and Cottrell [2], however, consider that adsorption of vacancies on the already existing dislocations is more important in hardening than the condensation of vacancies accompanied by the formation of pores and dislocation loops.

From the load–elongation curves for aluminum specimens (Fig. 1) it is evident that quenching increases resistance to the start of plastic deformation, and the modulus of strain hardening at the subsequent deformation becomes very small. This shape of curves of hardening of the specimens after quenching indicates also that preference should be given to the mechanism of immobilizing the dislocations by vacancy atmospheres. Recalling the basic propositions of the theory of vacancy atmospheres formation around the dislocations, and of their disintegration [9], it is possible to explain the observed dependence of the flow stress on the time of aging of samples deformed after quenching. The increase of flow stress at the initial stages of aging of aluminum deformed after quenching ($t < t_{max}$) is the consequence of the occurrence of atmospheres on dislocations because the rate of approach of vacancies towards dislocations exceeds the rate of their annihilation on the jogs and other sinks.

The reduction of $\Delta\sigma$ with the time of low temperature aging at $t > t_{max}$ indicates that the density of arresting centers on dislocations is decreased. In this case, the total concentration of vacancies in the volume of grains is decreased and the diffusion stream of vacancies towards the dislocations becomes smaller than the stream along the dislocations towards the sinks. The maximum of the increase of flow stress in the process of low temperature aging corresponds apparently to the condition of equilibrium in the rates of formation and disintegration of fixing nuclei on dislocations. It has been proved that such a model of low temperature aging of quenched aluminum after its minor deformation could be applied to estimate the diffusion coefficients of vacancies in the vicinity of the dislocation center D and along dislocations D'. Comparing the velocities and magnitudes of diffusion streams of vacancies towards dislocations and along dislocation lines towards sinks at $t = t_{max}$, we obtain [8] the following expressions for D' and D:

$$D' = \frac{2}{3} l^2 t_{max}^{-1}; \quad D = \left(\frac{2}{3} l\right)^{3/2} \left(\frac{U_0}{\kappa T}\right)^{1/2} b^{1/2} t_{max}^{-1},$$

where U_0 is the energy of the bond between dislocations and vacancies, l is the distance between jogs on dislocations, b is Burgers vector, k is Boltzmann constant, and T is temperature.

Using experimental values of t_{max} and the literature data for $U_0 = 0.02$ eV [10], and $l = 10^{-5}$ cm [11], we obtain for T = 77°K, $D' = 2 \cdot 10^{-4}$ cm²/sec, and $D = 10^{-15}$ cm²/sec. The large values of D' and D are due to the fact that the quenched samples contained high concentration of vacancies. Moreover, the method used for estimation of diffusion coefficients does not give values of these quantities for the whole volume, but only for the most distorted local sites in which the values of diffusion coefficients differ substantially from those for the volume [12]. According to the Coulomb–Friedel theoretical calculations, for the attainment within 1 h of the maximum concentration of vacancies in the atmospheres, with the concentration of vacancies $c \approx 10^{-5}$, it is necessary for the diffusion coefficient to be of the order of 10^{-14} to 10^{-12} cm²/sec at room temperature. The magnitudes of D and D' obtained from the model for aluminum vacancy aging at 77°K are close to these values.

The concentration of point defects in metal, necessary for the creation of dislocation fixing centers, is proportional to the density of dislocations [9]. Therefore, with the increase of the degree of deformation of aluminum after quenching, the magnitude and the time of attainment of the maximum increase of flow stress in the process of low temperature aging are reduced (Fig. 4). With the increase of dislocations density, the number of sinks for vacancies increases, and the length of the path from the vacancy to the dislocation becomes shorter.

The general character of the change in flow stress with low temperature aging of high purity aluminum and of commercial aluminum indicates that contaminants do not play a substantial role in this phenomenon. However, the presence of contaminants can retard the process of vacancy pile-up formation and can facilitate the creation of small tetrahedrons of stacking faults [13].

Increase of flow stress in quenched specimens under smoothly increasing load is apparently associated with the creation of a stable dislocation structure with a large number of dislocation loops and vacancy complexes [14]. With low rates of loading, a substantial role is played by diffusion processes of interaction between point defects and dislocations [15]. Increase of the concentration of vacancies facilitates in this case the more vigorous formation of dislocation loops, vacancy atmospheres, and jogs on dislocations which reduce the mobility of dislocations. For that reason, the noticeable change of flow stress in slowly deformed samples is not observed at low-temperature aging (Fig. 3, curve b), and the effect of hardening is preserved after reheating to room temperature.

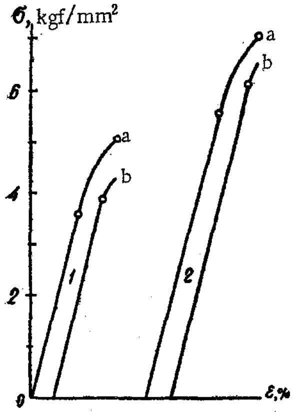

Fig. 5. Ordinary (1a) and slow (2a) load–elongation curves of quenched specimens of aluminum at 77°K, and their initial sections (1b and 2b, respectively) under ordinary strain after reheating to 300°K.

Therefore, the data obtained on the change of flow stress in low temperature aging of aluminum deformed after quenching is indicative of the processes of diffusion redistribution of vacancies, and formation and disintegration of arresting centers of dislocations. Proceeding from the Coulomb–Friedel model of emergence and disintegration of vacancy atmospheres around dislocations, it proved to be possible to estimate the values of vacancy diffusion coefficients in the vicinity of the dislocation nucleus, and along the dislocation line in aluminum specimens slightly deformed after quenching. The low temperature aging under smoothly increasing load is accompanied by the increase of aluminum yield point, which indicates the possibility of using quenching defects to organize structures with increased resistance to the movement of dislocations.

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