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EFFECT OF LOADING ON RECOVERY OF INTERNAL FRICTION OF Al-16 wt % Ag AND Al-16 wt % Ag – 0.28 wt % Fe ALLOYS

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The phenomenon of internal-friction recovery of Al-16 wt % Ag and Al-16 wt % Ag – 0.28 wt % Fe alloys is studied after cold-working the samples in uniaxial tension using the free decay method. The wire samples are investigated for internal friction recovery while they were loaded, within the elastic limit, by different loads at different testing temperatures. It is found that by increasing the temporary loading, the rate of recovery decreases. TEM investigations confirmed that the addition of Fe to Al-Ag alloy accelerates the formation and coarsening of Guinier-Preston zones. The results are explained on the basis of dislocation damping adopting the Granato-Lücke model for recovery of internal friction.

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1. *Introduction*

The basic sequence of decomposition in Al-Ag alloy has been found as: α (f.c.c) $\rightarrow$ spherical Guinier-Preston (G. P.) zones (tetrahedral) $\rightarrow$ metastable γ'-phase $\rightarrow$ equilibrium γ-phase (hcp) [1]. Dubey et al. [2], using small angle X-ray scattering (SAXS) studies, concluded that the composition of G. P. zones ($\sim Ag_2Al$) remained constant over a wide temperature range from 20 to 250 °C. The experimental evidence for the formation of an ordered η-state of G. P. zones below 170 °C and a disordered ϵ-state above this temperature was obtained by many authors [3,4].

Internal friction measurements serve to supply much useful information concerning the internal structure of Al-Ag alloy, including mobile-dislocations density and the precipitations of G. P. zones which takes place during the aging process. The transient increase of internal friction, Q^{-1}, observed in metals after plastic deformation, is known to decrease more or less rapidly as a function of time. Such a phenomenon is known as the recovery of internal friction [5]. According to Koehler [6] and Granato and Lücke [7] models, the increase of internal friction (IF) after plastic deformation is interpreted as being caused by the creation of a large number of new, freely moving dislocations. The subsequent disappearance of the effect (i.e. recovery of IF) is ascribed either to the pinning of diclocations by point defects and second-phase particles or rearrangements of dislocation networks resulting in an overall decrease in dislocation damping.

Reviewing the literature of previous work on the recovery of IF using torsion pendulum, one can recognize, as a matter of experimental procedure, that the stress acting on the test sample is virtually zero. The question arises: if the test sample is loaded, within the elastic limit, for certain periods of time just before measuring its IF, how such loading will affect the recovery behaviour. No studies were made on the effect of loading on the recovery of IF as affected by second phase particles. Hence, the present work is devoted to investigate the effect of loading on the recovery behaviour of IF in samples of Al-16 wt % Ag and Al-16 wt % Ag – 0.28 wt % Fe alloys strained plastically in tension immediately before measurements. Here an ordered η-state of G. P. zones formed by aging in the appropriate temperature range represents the second-phase particles to be considered.

2. *Experimental procedure and technique*

Two alloys each containing Al-16 wt % Ag were made by melting silver of purity 99.999 % with either pure Al (99.999 %) (alloy A) or with Al containing 0.28 wt % Fe (alloy B). Each alloy was melted under vacuum in a high purity graphite crucible. After annealing for homogenization at 823 K for 4 days, the ingots were awaged and cold drawn into wires 0.35 mm in diameter for IF measurements, or rolled into strips 0.2 mm in thickness for electron microscopy investigation. Chemical analysis revealed that the composition of samples is very close to the weighed-in compositions.

A solution treatment was carried out by heating the samples at a temperature above solvus (about 823 K) for 2 h and samples were then quenched rapidly in cold water and immediately aged at 428 K for 2 h. Internal friction was determined by the free decay method by means of a low-frequency (about 1 Hz) torsion pendulum with electromagnetic excitation and optical read out. The uniaxial tensional deformation was carried out "in situ" only at room temperature. The tensional deformation $\epsilon = \Delta\ell/\ell$ (where $\Delta\ell$ is the increase of the length of the samples and ℓ is its original length) was 0.04 for all samples. The IF, Q^{-1}, was first measured before exerting tensional deformation upon samples, this is denoted by Q_u^{-1} (i.e. Q^{-1} corresponding to the undeformed state). Immediately after deformation ($\epsilon = 0.04$), the IF corresponding to a time $t = 0$ (Q_0^{-1}) is determined. Then the sample wire is set at a certain testing temperature, and a certain load is applied for a certain time ranging from 0.5 at the beginning of the recovery process to 3 min at its end. The load is then removed, and IF (Q^{-1}), is measured. The above step is repeated by loading the sample for an additional period of time, then the load is removed and Q_t^{-1} is measured. In following steps Q_t^{-1} is determined where t is the time measured immediately after the tensional deformation. The short intervals during which the samples were unloaded for IF measurement are actually accounted for in t, thus a negligibly small error in the value of Q_t^{-1} remained. Calculations which took such an error into consideration proved that it has no appreciable effect on the value of Q^{-1} as a function of t. The recovery behaviour under different loads of 0.45, 0.95, 1.45 and 1.95 N was studied at testing temperatures of 293, 323, 343 and 363 K.

The sheet samples for electron microscopy investigation were prepared by electropolishing using a twin jet machine. The electrolyte was 10 % perchloric acid and 90 % ethanol at 273 K. The voltage and current values were around 15 to 25 V and 10 to 20 mA, respectively. Microstructures were examined using a JOEL-100 transmission electron microscope operating at 100 kV.

3. *Experimental results*

The recovery behaviour of IF is presented as a relation between the normalized IF $S(t) = (Q_t^{-1} - Q_u^{-1})/(Q_0^{-1} - Q_u^{-1})$ and the time t (S is a fractional quantity which represents the freely moving dislocations, i.e., those dislocations contributing

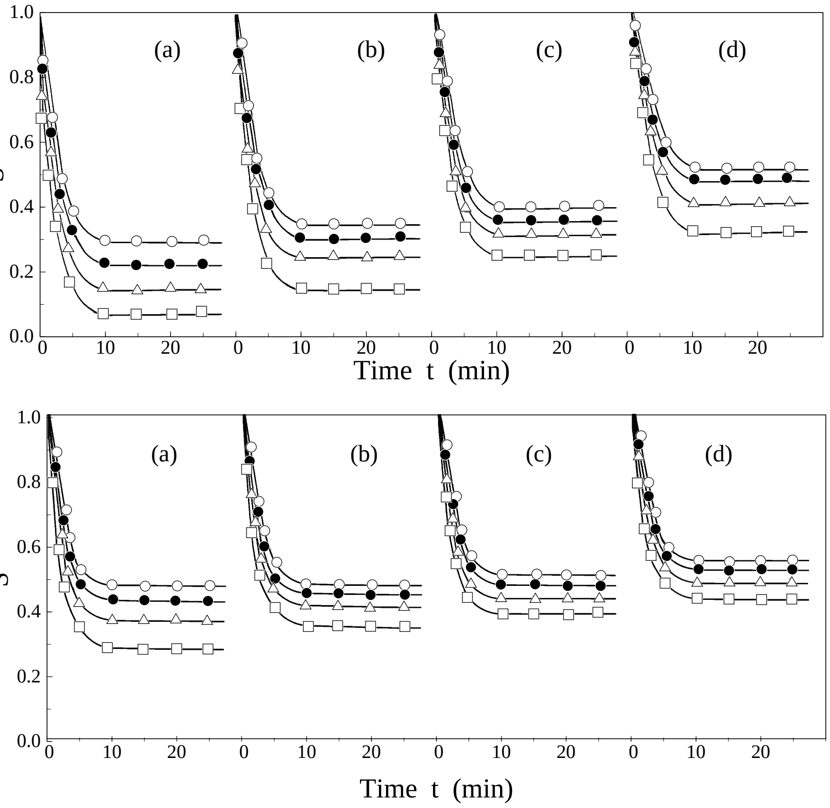
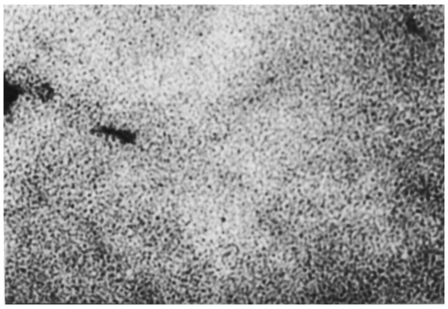


Fig. 1. Normalized internal friction (S) of deformed and then loaded samples as a function of time t for alloys A *(upper diagrams) and* B *(lower diagrams). Different loads applied were 0.45 (□), 1.45 (△), 1.45 (•) and 1.95* N *(○). The testing temperatures applied were: a) 293, b) 323, c) 343 and d) 363* K.

to the damping effect). Figure 1 shows the recovery of IF for both alloys A and B, respectively, as a relation between the normalized IF, S, and time t under the effect of elastic stressing by different loads at different testing temperatures mentioned before. From this figure, it is seen that, for both alloys, the recovery behaviour can be divided into three stages, namely, a rapid initial stage, an intermediate stage and finally a slow saturation stage. We will be only interested in the first and second stages. Generally, it is clear that S takes larger values by increasing both testing temperatures and the loads. It is worth noting that under the same conditions, corresponding variations of S are appreciably higher in alloy B than in alloy A.

4. *Discussion*

The Granato-Lücke [7,8] break-away mechanism for recovery of IF has been adopted here to explain the results of the present work. It is well known that IF and its recovery are both influenced by the presence of solute atoms and second-phase particles [9–15]. In the present work, the G. P. zones are considered as the second phase-particles that affect the recovery behaviour (see Fig. 2) where they act as pinners for dislocations [16–19]. Before cold-working the heat treated samples, the interaction between the pre-existing dislocations and G. P. zones (as well as other IF sources, like damping of dislocations due to the point-defect diffusion, grain boundary relaxation, thermoelasticity, etc.) gives a value of IF equal to Q_u^{-1} (Q^{-1} corresponding to the undeformed state). Immediately after deformation, the freshly created dislocations are vibrating with full network loop length, causing the highest value of IF, Q_0^{-1} (Q^{-1} corresponding to a time $t = 0$). The time dependence of Q_t^{-1} (and consequently of S) after straining, as shown in Fig. 1, can be explained in view of the rate of interaction of G. P. zones with dislocations. According to Cottrell and



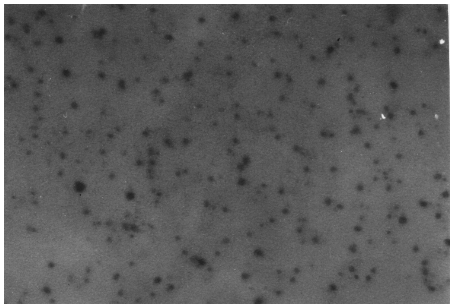


Fig. 2. Thin foil micrograph from Al-*16 wt %* Ag *samples heated at 428* K *for 2* h*, showing G. P. zones.*

Fig. 3 (right). Thin foil micrograph from Al-*16 wt %* Ag *– 0.28 wt %* Fe *samples heated at 428* K *for 2* h*, showing G. P. zones of large sizes.*

Bilby [20], the number of second- phase particles (G. P. zones here) per unit volume, which interacts with dislocation, proceeds by a relatively higher rate immediately after deformation (rapid stage of recovery). As a result, the average dislocation loop length L_p (L_p is the average distance between zone pins and is less than the full network loop length) and consequently S are both reduced by a higher rate. In the intermediate stage of recovery, due to the relatively lower rate of interaction between zones and dislocations, a relatively lower rate of reduction of L_p results in a lower rate of recovery. Finally, a slow saturation stage is reached corresponding to a minimum interaction of G. P. zones with dislocations, i.e. minimum value of L_p.

Particularly for the alloy B, the higher value of S and lower rate of IF recovery, as compared with corresponding values for the alloy A, may be explained as follows. For alloy A, immediately after a minimum time of aging, the number of G. P. zones is small. As the time of aging proceeds, new zones are formed leading eventually to a large number of small-sized zones. In alloy B (Al-Ag-Fe), due to the binding between vacancies and Fe solutes, vacancy-impurity clusters are likely to be formed [21]. These clusters might act as nucleation sites for a large number of small zones. Thus, a large number of zones is expected to be formed immediately after a minimum time of aging. In the course of aging time, growth of the zones on the expense of their number occurred leading to existence of a smaller number of large-sized zones (compare Fig. 2 with Fig. 3). These large-sized zones of smaller number should be less effective as pinners for dislocation [19] and hence produce less recovery.

The recovery of IF which occurs during the sequence of time after deformation could be controlled by an Avrami-type relation [22] of the form

$$S(t) = \exp\left(-Kt^n\right) .$$

n represents the power of time dependence of damping and K is the decay rate.

Figures 4a and b are plots of $\ln\left[\ln\left(S^{-1}\right)\right]$ versus $\ln t$ for samples loaded to different values at different testing temperatures for both alloys A and B, respectively. At each testing temperature, the intercept of $\ln\left[\ln\left(S^{-1}\right)\right]$ axis in Fig. 4. represents $\ln K$, from which the decay rate K can be obtained. From this figure, it is clear that two linear parts with different slopes n are distinguishable. They are considered to represent two stages.

4.1. Rapid stage of recovery

The power n of time dependence of damping, as calculated from the slopes of the parallel straight lines in Fig. 4, has an average value of unity, for both alloys, indicating that the operating mechanism for the decay of IF in this stage (stage I) is the same, irrespective of the conditions of loading and testing temperatures applied. However, this value is high when compared with the ordinarily known limits [8] which range between 0.33 and 0.7. It is clear that the relatively high value of n obtained here indicates that the responsible mechanism is highly time-dependent. In view of Granato-Lücke model, this may be attributed to the relatively large numbers of G. P. zones which act as pinners.

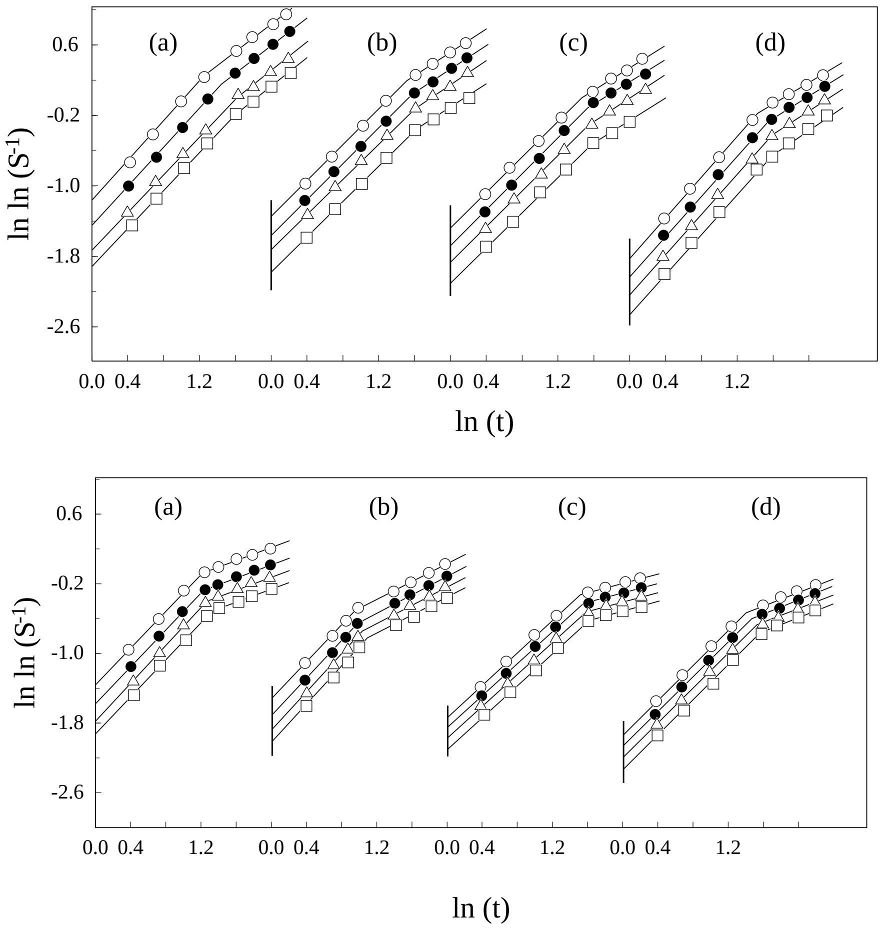


Fig. 4. Plot of $\ln\left[\ln\left(S^{-1}\right)\right]$ *and* $\ln t$ *as a function of time* t *for alloys* A *(upper diagrams)and* B *(lower diagrams) for differently loaded samples a) 0.45, b) 0.95, c) 1.45 and d) 1.95* N. *The testing temperatures applied were: 293 (○), 323 (●), 342 (△) and 363* K *(□).*

Under the effect of loading, the crystal is elastically strained due to the tensional forces acting on it. During loading with a load P, the elastic energy (E) stored in the crystal is proportional to P^2 [23]. Figures 5a and b show the dependence of the decay rate K on the applied load, P, as a relation between K and P^2. From Fig. 5, it is clear that, at a certain testing temperature, the value of K decreases linearly with the elastically stored energy E. This energy, which is stored in the crystal during loading, is assumed to allow moving dislocations to surmount the G. P. zones which act as pinning points for the moving dislocations, i.e. reducing the rate

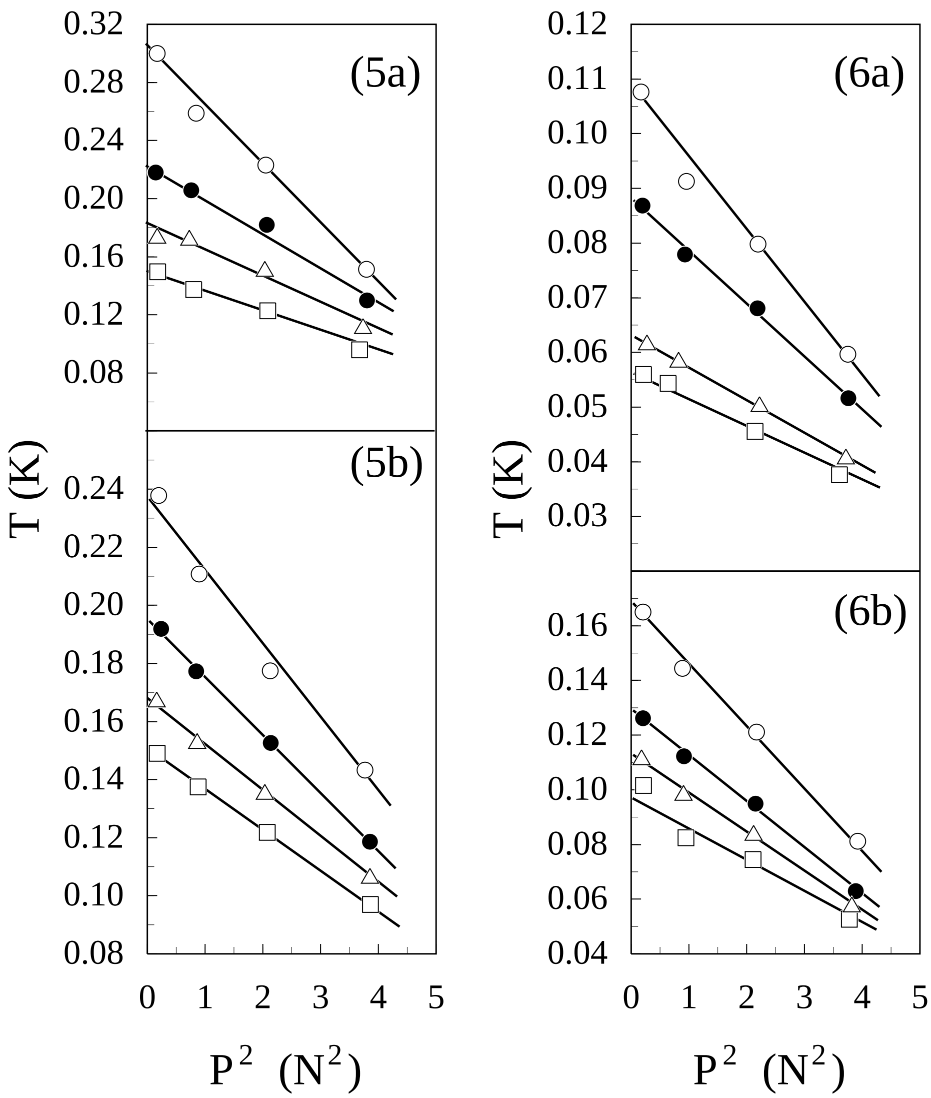


Fig. 5. Relation between the decay rate (K) for stage I and the square of the applied load P^2 for alloys A *(a) and* B *(b). The testing temperatures applied were: 293 (○), 323 (●), 342 (△) and 363* K *(□).*

Fig. 6 (right). Relation between the decay rate (K) for stage II and the square of the applied load P^2 for alloys A *(a) and* B *(b). The testing temperatures applied were: 293 (○), 323 (●), 342 (△) and 363* K *(□).*

of pinning exerted by G. P. zones on dislocations. Thus redistribution of dislocation loop-lengths will be in favour of magnification of the effective average loop length L_p. Such a magnification proceeds at a higher rate as the load applied is larger, and, accordingly, the value of K decreases. Increasing the testing temperature, which leads to a deceasing of the value of K, explained that the unpinnig process is a thermally-activated mechanism. dovde

4.2. *Intermediate stage of recovery*

The power n of time dependence in this stage (stage II) was found to have an average value of 0.7 for the alloy A and 0.4 for the alloy B. These values are in good agreement with the ordinarily known limits mentioned before. Figures 6a and b show the relation between K and P^2, where straight lines are obtained as in the previous stage (stage I), but with a lower value in K as compared with those having the same testing temperatures in stage I.

Since the two groups of curves in both stages are similar, one can conclude that the operating mechanism for the recovery behaviour, for both stages, is the same. The difference is that the rate with which the mechanism proceeds in stage II is lower. This is acceptable in view of the fact that the recovery is basically a time-dependent process, i.e. during the stage II, the "residual" fraction of dislocations that contribute to damping, being less numerous, should correspond to a lower rate of recovery as compared to that in stage I.

4.3. *Activation energy calculation*

To calculate the activation energy of the operating mechanism for the recovery behaviour, the procedure used by Graiss et al. [23] is followed. Figures 7a and b show a relation between the slopes $(\partial K/\partial P^2)$ of the straight lines of Fig. 5 and 6 and $1000/T$ for both alloys A and B, respectively, where T is the testing temperature in K. For both stages, the activation energy yielded a mean value of about (14 ± 0.7) kJ/mol for alloy A and a mean value of nearly (18 ± 0.9) kJ/mol for alloy B. In view of such a low values for the activation energy we assume the recovery mechanism not to be diffusion. A similar conclusion was reposted by Kenawy et al. [13] as a result of their early work on Al-Fe alloy. Adopting the Granato-Lücke model [7,8] for the recovery of IF as being the loop length redistribution due to the change in concentration of pinning points, we may assume that the mechanism responsible for the recovery process is the pinning imposed to vibrating dislocations by G. P. zones. When the dislocation line acquires an energy higher than 14 to 18 kJ/mol, it can surmount the G. P. zones which act as pinning points for the moving dislocations. Depending on the amount of energy supplied to the sample either thermally and/or by loading, we may except different concentrations of pinning points on the dislocation line leading to different network lengthes, L_p. At higher testing temperatures and/or applying larger loads, large values of L_p are progressively obtained, corresponding to lower values of decay rate K. In view of this, the values of the slopes $(\partial K/\partial P^2)$ at different testing temperatures in Figs.

5 and 6 are considered to reflect the rate of pinning of dislocations by G. P. zones. Since the values of the activation energy in the two stages (I and II) for both alloys are very close, so the same pinning mechanism for the recovery process is applied.

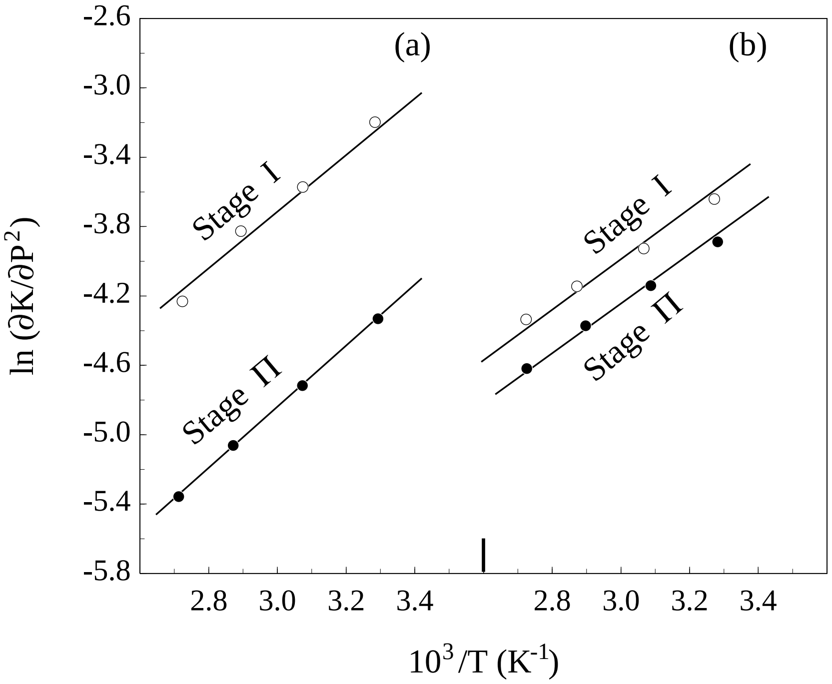


Fig. 7. Arhenius plot of $\ln(\partial K/\partial p^2)$ *as a function of* $1000/T$ *for alloys A (a) and B (b). Stage type is indicated.*

5. Conclusion

Increasing the temporary loading in Al-Ag alloys was found to decrease the rate of internal recovery. The presence of Fe in Al-Ag alloys accelerates the precipitation of G. P. zones. The activation energy of recovery process was found to correspond to the pinning imposed to vibrating dislocations by G. P. zones.

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UTJECAJ OPTEREĆENJA NA OBNAVLJANJE UNUTARNJEG TRENJA LEGURA Al-16 wt % Ag I Al-16 wt % Ag – 0.28 wt % Fe

Proučava se pojava oporavka unutarnjeg trenja legura Al-16 wt % Ag i Al-16 wt % Ag – 0.28 wt % Fe nakon hladne obrade jednoosnim naprezanjem primjenom metode slobodnog opadanja. Ispitivali smo žičane uzorke pod opterećenjem unutar elastične granice za više opterećenja i na više ispitnih temperatura. Našli smo da se s povećanjem opterećenja smanjuje brzina oporavka. Ispitivanja TEM-om su potvrdila da dodavanje Fe leguri Al-Ag ubrzava stvaranje i okrupnjivanje G. P. zona. Ishodi se tumače Granat-Lücke-ovim modelom dislokacijskog gušenja za oporavak unutarnjeg trenja.