ISSN 1330–0008
CODEN FIZAE4

# EFFECT OF MECHANICAL EXCITATION ON THE OVER-RECOVERY OF INTERNAL FRICTION OF Al-16 Wt% Ag AND Al-16 Wt% Ag-0.28 Wt% Fe ALLOYS

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Received 3 July 2000; Accepted 6 November 2000

The phenomenon of internal friction (IF) of Al-16 wt% Ag and Al-16 wt% Ag-0.28 wt% Fe alloys aged at 428 and 523 K was studied at different testing temperatures using the free-decay method. The wire specimens were investigated for IF recovery under two different conditions of excitation. In the first, the specimens were continuously excited and in the other, they were subjected to normal condition of restrictive excitation. Under certain conditions of heat treatment and mode of excitation, an anomalous behaviour of IF recovery has been observed. TEM investigations confirmed that addition of Fe to Al-Ag alloy accelerates the precipitation of metastable phases. The mean values of the excitation energy of both alloys were found to be equal to that quoted for precipitate-dislocation interactions.

PACS numbers: 62.40.+1, 61.72.Ff UDC 539.67

Keywords: internal friction of alloys, Al-16 wt% Ag and Al-16 wt% Ag-0.28 wt% Fe alloys, aging at 428 and 523 K, free-decay method, anomalous internal friction recovery

## 1. *Introduction*

The basic sequence of decomposition in Al-Ag alloy has been found as $\alpha$ (fcc) $\rightarrow$ spherical Guinier-Preston (G. P.) zones (tetrahedral) $\rightarrow$ metastable $\gamma'$-phase (hcp) $\rightarrow$ equilibrium $\gamma$-phase (hcp) [1]. The experimental evidence for the formation of an ordered $\eta$-state of G. P. zones below 443 K and a disordered $\epsilon$-state above this temperature has been reported by many authors [2–4]. Positron trapping rate method showed that the coarsening of G. P. zones takes place simultaneously with the formation and growth of $\gamma'$-precipitates during isothermal aging above 473 K [5,6].

Internal friction (IF) measurements serve to supply much useful information concerning the internal structure of Al-Ag alloy, including mobile dislocations density and the precipitation phases which takes place during the aging process. The transient increase of internal friction (IF), $Q^{-1}$ ($Q^{-1}$ is defined as the tangent of the phase angle by which the strain lags behind the stress), 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 IF [7]. According to Granato and Lücke model [8], the increase of IF after plastic deformation is due to the creation of a large number of new, freely-moving dislocations. The subsequent disappearance of the effect with time (i.e., recovery of IF) is ascribed either to the pinning of dislocations by second-phase particles, or rearrangements of dislocation networks, resulting in an overall decrease in the dislocation damping.

The recovery of IF after deformation is usually determined by the free-decay method, i.e. by forcing the specimen to vibrate and measuring the decrement of amplitude of vibration as a function of time. Reviewing the literature on previous work on the recovery of IF using the torsion pendulum [9–12], one can recognize, as a matter of experimental procedure, that the time intervals during which no measurements of IF, $Q_1^{-1}$ take place, the specimen is normally kept steady. This mode of excitation is considered as a restrictive excitation (RE). The following question arises: if the test specimen is forced to vibrate, within the appropriate limits of strain amplitude, during the whole time of decay (i.e. during both intervals of measurement and between the measurements), how such a continuous excitation (CE) affects the recovery behaviour. The present work is aimed at studying the effect of such CE on the recovery of IF of Al–16 wt% Ag and Al–16 wt% Ag–0.28 wt% Fe alloys.

## 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 molten under vacuum in a high-purity graphite crucible. After annealing at 823 K for four days (for homogenization), the ingots were swagged and cold drawn into wires 0.35 mm in diameter for IF measurements, and rolled into strips 0.25 mm thich for electron-microscopy investigation. Chemical analysis revealed that the specimen composition is very close to the weighted-in composition. A solution treatment was carried out by heating the specimens at 823 K for two hours, followed by rapid quenching in cold water at 273 K and immediately aged at 428 and 523 K for one hour. The rate of quenching was about $10^4$ K/s. The values $Q^{-1}$ of IF were determined using a torsion pendulum of low frequency (about 1 Hz) with electromagnetic excitation and optical readout. The uniaxial tensional deformation $\epsilon = \Delta\ell/\ell$ (where $\Delta\ell$ is the increase of length of the specimen and $\ell$ is its original length) was carried out "in situ" at room temperature for all specimens and was equal 0.04. After deformation, the specimens were set at various testing temperatures of 298, 323, 343, 363 and 383 K, and the recovery behaviour under CE and RE was studied. All values of $Q^{-1}$ were taken under constant strain amplitude equal to $2.1 \times 10^{-4}$. Preliminary experiments have been done which proved that IF doesn't depend on the strain amplitude within the value applied. The accuracy of temperature measurements was about $\pm 1$ K. The sheet specimens for electron microscopy measurements 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 13 to 19 V and 8 to 14 mA, respectively. Microstructure was examined using a JOEL-100 transmission electron microscope operating at 100 kV.

## 3. *Results and observations*

Before applying the tensile deformation to the heat-treated specimens, the value of IF of each specimen was measured first. That value is denoted by $Q_u^{-1}$ (i.e. $Q^{-1}$ corresponding to the undeformed state). Immediately after deformation, a large increase in IF value was observed. This value is denoted by $Q_0^{-1}$ ($Q^{-1}$ corresponding to the time $t = 0$). The recovery behaviour was indicated by the value $Q_t^{-1}$ that was determined at time $t$. It was influenced by the mode of excitation (RE or CE) and by the heat treatment.

Figure 1 shows the recovery of IF as a relation between the normalized IF

$$S = \frac{Q_t^{-1} - Q_u^{-1}}{Q_0^{-1} - Q_u^{-1}}$$

and time. From the figure, it is clear that:

1) The recovery behaviour of both alloys can be divided into three stages: (i) an initial short stage (stage I) which predominates within the first 4 minutes, (ii) an intermediate stage (stage II) which starts after stage I and extends to about 9 minutes, and finally (iii) a slow saturation stage which takes place after more than 10 minutes.

2) Aging the specimens at 428 or 523 K for one hour resulted in a decrease in $Q_1^{-1}$ to the extent that the value of $S$ became negative, which means that under certain conditions of heat treatment and mode of excitation, the test specimen can be brought to a condition that its IF during a stage of recovery period is less than its IF in the undeformed state. This anomalous behaviour of IF recovery is considered as an "over-recovery effect".

3) For the same alloy and the same heat treatment, the rate of decrease of $S$ for the CE specimens is higher compared with that of RE specimens.

4) Under the same condition of heat treatment and mode of excitation, the rate of decrease of $S$ is appreciably lower for alloy B than in alloy A.

5) Under the same conditions of testing temperature and mode of excitation, $S$ value for alloys aged at 523 K is higher than for those aged at 428 K.

We are interested only in the first and second stages of recovery.

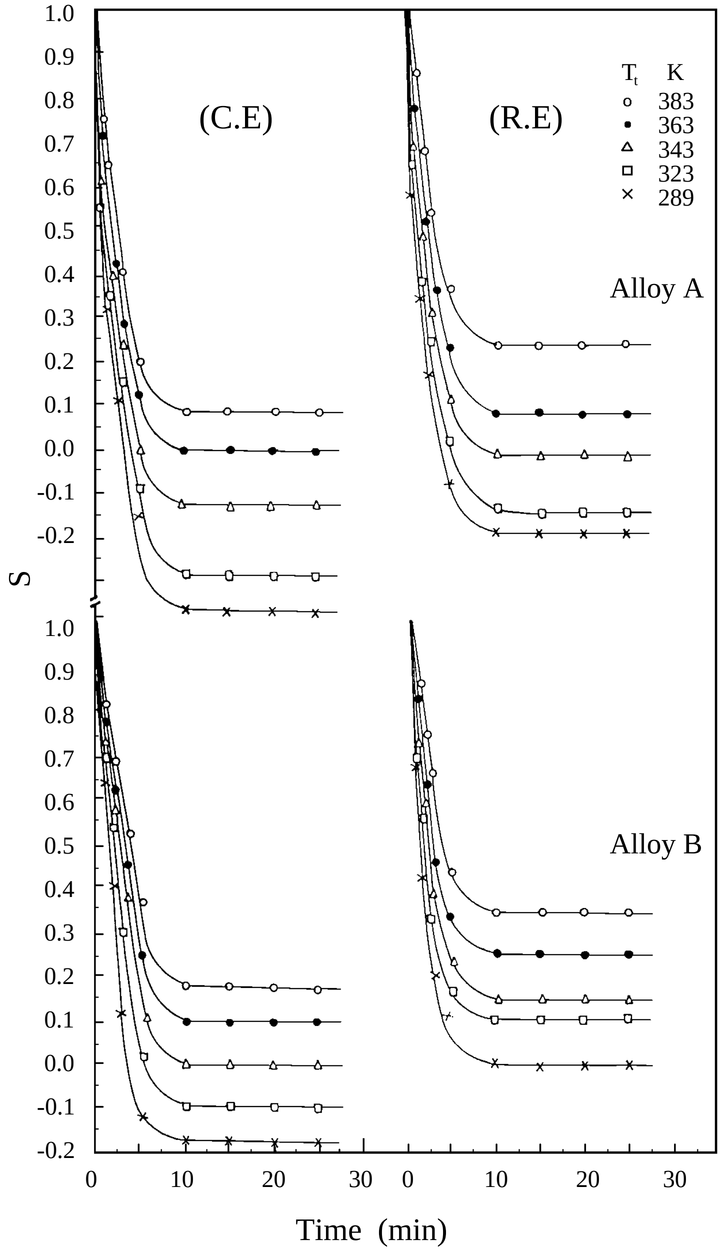



*Fig. 1a. Normalized internal friction ($S$) as a function of time of deformed specimens aged at 428* K. *The applied testing temperatures, type of alloys and mode of excitation are indicated.*

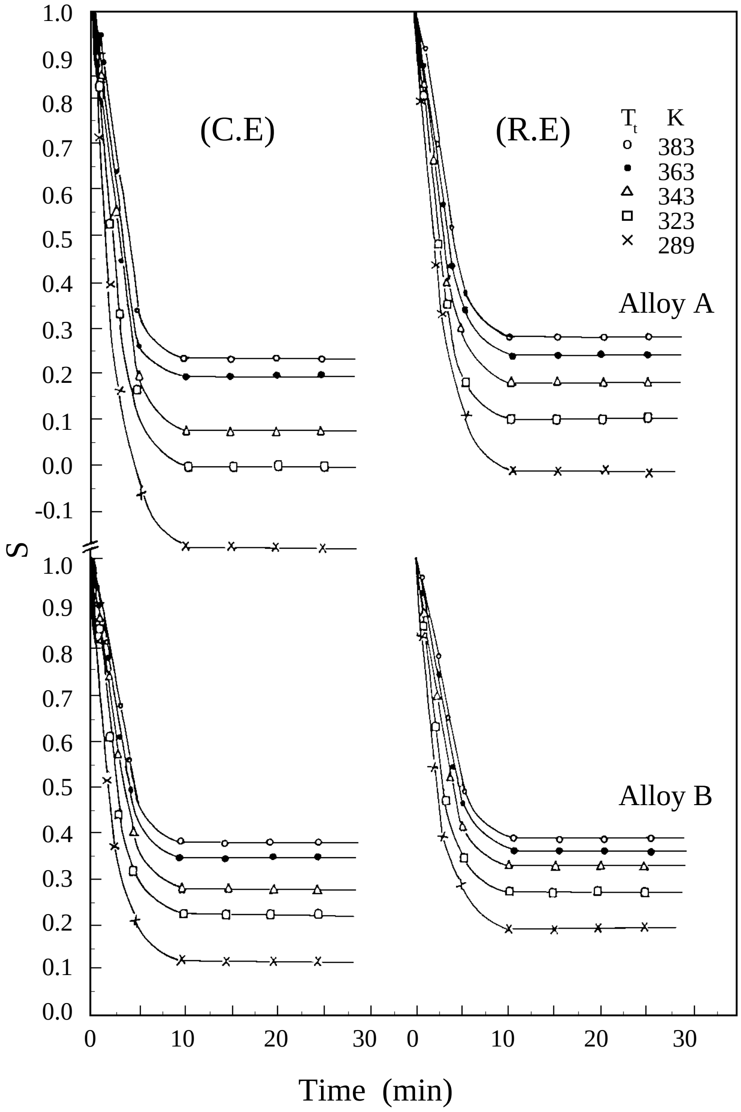
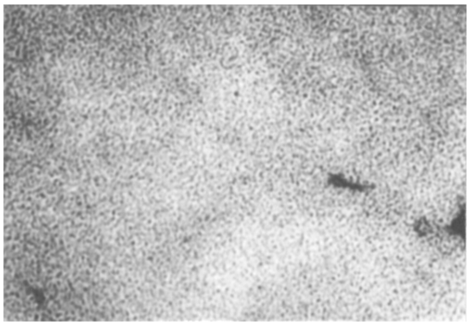



*Fig. 1b. Normalized internal friction ($S$) as a function of time of deformed specimens aged at 523* K. *The applied testing temperatures, type of alloys and mode of excitation are indicated.*

Figures 2 and 3 show the precipitation of G. P. zones in specimens aged at 428 K for one hour for alloys A and B, respectively. It is clear that the sizes of G. P. zones in alloy B (with Fe addition) are coarser than in alloy A. Figure 4 shows the formation of G. P. zones and some plates of $\gamma'$-precipitates in alloy A aged at 523 K for one hour.



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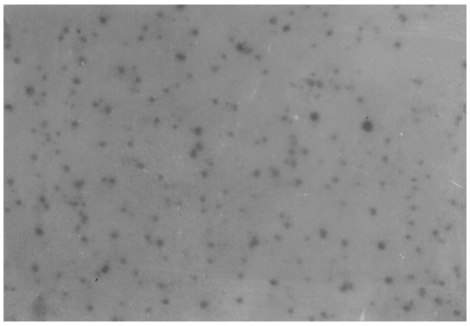

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*Fig. 2. Thin-film micrograph of* Al*–16 wt%* Ag *specimen heated at 428* K *for 1 hour, showing G. P. zones.*

*Fig. 3 (right). Thin-film micrograph of* Al*–16 wt%* Ag*–0.28 wt%* Fe *specimen heated at 428* K *for 1 hour, showing G. P. of zones of larger sizes.*

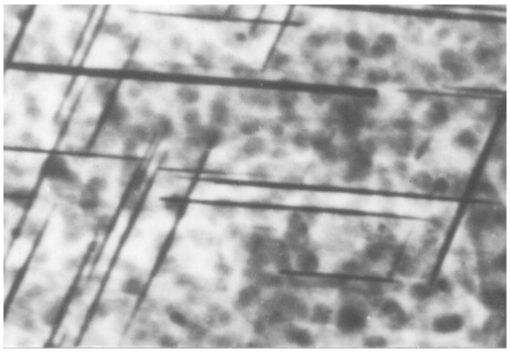

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*Fig. 4. Thin-film micrograph of Al–16 wt% Ag specimen heated at 523* K *for 1 hour, showing G. P. zones and some plates of $\gamma'$-precipitates.*

## 4. Discussion

It is well known that IF and its recovery are influenced by the presence of solute atoms and second-phase particles [8–12]. In the present work, the metastable phases, the G. P. zones and $\gamma'$-precipitates (see Figs. 2, 3 and 4) are considered as second-phase particles, whereas they can act as major barriers or pinners for moving dislocations. Before applying the tensile deformation to the heat-treated specimen, the interaction between the pre-existing dislocations and metastable phases (as well as other IF sources, like damping of dislocations due to the point-defect diffusion, grain-boundary relaxation, thermoeleasticity 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 the full network loop length, causing the highest value of IF, $Q_0^{-1}$ ($Q^{-1}$ corresponding to the time $t = 0$). The time dependence of $Q_t^{-1}$ (and consequently $S$) after straining, as shown in Fig. 1, can be explained by the rate of interaction of metastable phases with dislocations. According to Chambers and Smoluchowski [13], the number of second-phase particles per unit volume, which act as major barriers of dislocation motion, proceeds by a relatively higher rate immediately after deformation (rapid stage of recovery). As a result, the average dislocation loop length, $L_p$, 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 metastable phases 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 value of $L_p$.

Due to the tensional deformation, a large number of introduced dislocations move amongst the metastable precipitates that are known to block the dislocation movement very effectively through the pinning process [14–17]. These pinning dislocations "per se" may act to hinder the motion of other, unpinned dislocations to the extent that $Q_t^{-1}$ of a lower value than $Q_u^{-1}$ could be obtained. This might account for the observed negative value of $S$ (over-recovery effect). The disappearance of this effect is explained in the following discussion.

The lower values of $S$ of the CE specimens, as compared to the corresponding values of the RE specimens, may be related to the fact that an additional elastic energy, associated with the continuous vibration, would be stored in the CE specimens [18]. Such energy is expected to increase the rate of interactions between metastable phases and moving dislocations, i.e. enhancing the process of rearrangement of dislocations to form a more stable configuration of networks through a redistribution of a large number of pinning agents (G. P. zones and $\gamma'$-precipitates) on the dislocation segment. Thus the redistribution of dislocation loop lengths will be in favour of diminishing the effective loop lengths, $L_p$. By increasing the testing temperature, which leads to a decrease of $S$ values, provided an explanation that the unpinning process is a thermally activated mechanism.

The lower values of $S$ for the specimens aged at 428 K, as compared with the specimens aged at 523 K, is attributed to an intense interaction between the $\eta$-states of G. P. zones and dislocations. Aging specimens at 523 K for one hour leads to partial dissolution and coalescence of G. P. zones and a formation of a smaller number of coherent $\gamma'$-precipitates (see Fig. 4). G. P. zones of larger sizes and $\gamma'$-precipitates are known to be less effective as bariers for moving dislocations [16,17]. As a result, a lower rate of recovery (i.e. a higher value of $S$) is expected.

A close inspection of many electron micrographs aged at 428 K revealed that G. P. zones in alloy B were present in smaller numbers and had a larger size as compared to alloy A (compare Fig. 2 with Fig. 3). The reason is that in alloy B (Al-Ag-Fe), due to the binding between vacancies and Fe solutes, the vacancy-impurity clusters are likely to be formed [19]. These clusters might act as nucleation sites for a large number of small zones. Thus a large number of zones would be obtained immediately after a minimum time of aging. In the course of aging time, the growth of zones at the expense of their number occured, leading to the existence of a smaller number of large-sized zones and $\gamma'$-precipitates (see Fig. 3). Such an effect reflected itself in higher values of $S$ for alloy B as compared to the corresponding values for alloy A, since coarse G. P. zones and $\gamma'$-precipitates are less effective as barriers for motion of dislocations, as mentioned above. So, one can conclude that the presence of Fe plays a significant role in coarsening of G. P. zones and acceleration of formation of $\gamma'$-precipitates.

To elucidate the mechanism governing the recovery process, one can assume that it is controlled by the reaction-rate equation

$$t \ \exp(-\frac{E}{kT_1}) = const.\,,$$

where $E$ is the activation energy per mol, $T_1$ the testing temperature and $k$ the Boltzmann constant. By making cuts at different values of $S$ in Fig. 1 and obtaining the corresponding values of $\ln t$, a relation between these values and $1000/T_1$ could be drawn as shown in Fig. 5. The energy activating the process was calculated from the slopes of the straight lines obtained. The results are shown in Table 1.

*Table 1. Values of activation energy in* kJ/mol. *The accuracy of the values is approximately* ±0.2 kJ/mol.

| Stage type | Mode of excitation | $T_a = 428$ K | | $T_a = 523$ K | |
|---|---|---|---|---|---|
| | | Alloy A | Alloy B | Alloy A | Alloy B |
| First stage | (CE) | 12 | 10 | 10 | 10 |
| | (RE) | 12 | 15 | 11 | 10 |
| Second stage | (CE) | 12 | 15 | 11 | 10 |
| | (RE) | 18 | 19 | 15 | 14 |

From Table 1, it is observed that:

1) The slight differences in the values of activation energies between the continuous and restricted modes of excitation may be due to the additional elastic energy associated with the continuous vibration, as mentioned above.

2) The values of activation energy for both alloys are about the same. This indicates that the IF recovery is due to the same mechanism. The small values of the activation energies indicate that the recovery process is not due to diffusion [7]. It is more likely due to the precipitate-dislocation interactions [7,10,17].

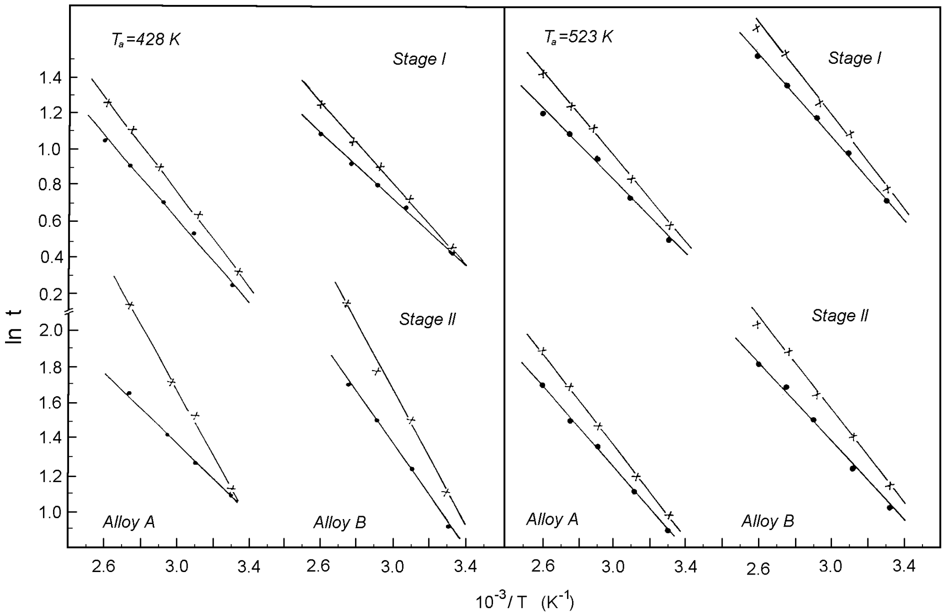


*Fig. 5. Arrhenius plot of* $\ln t$ *as a function of* $10^3/T$ *for restrictively* ($\times$) *and continuously* ($\bullet$) *excited specimens. Aging temperatures, type of alloys and stage are indicated.*

## 5. *Conclusion*

An anomalous behaviour of IF recovery has been observed in Al-Ag alloy that had been aged at temperatures of 428 and 523 K to form G. P. zones and $\gamma'$-precipitates. Under certain conditions of heat treatment and mode of excitation, the test specimen can be brought to a condition that its IF during a stage of recovery period is less than its IF in the undeformed state. This anomalous behaviour is considered as an "over-recovery" effect.

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## UČINAK MEHANIČKE UZBUDE NA PREKO-OPORAVAK UNUTARNJEG TRENJA LEGURA Al-16 Wt% Ag I Al-16 Wt% Ag-0.28 Wt% Fe

Proučavali smo pojavu unutarnjeg trenja (UT) u slitinama Al-16 wt% Ag i Al-16 wt% Ag-0.28 wt% Fe starenim na 428 i 523 K, metodom slobodnih oscilacija na nizu mjernih temperatura. UT uzoraka žice ispitivali smo dvama načinima uzbude. U prvom s neprekidnom uzbudom, a u drugom smo uzorke podvrgavali normalnim uvjetima ograničene uzbude. U nekim uvjetima toplinske obrade i načina uzbude, opazili smo anomalan oporavak UT. Ispitivanja pomoću TEM potvrdila su da dodavanje Fe slitini Al-Ag ubrzava precipitaciju metastabilnih faza. Našli smo da je srednja energija uzbudne energije obje slitine jednaka onoj koja se navodi za interakcije dislokacija s precipitatima.