INFLUENCE OF THE THERMO-MECHANICAL TREATMENTS ON THE RELAXATION PHENOMENA OBSERVED ON A SINGLE CRYSTAL ALLOY (CU – AT.11% AL) IN INTERNAL FRICITION AT HIGH TEMPERATURE.

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Abstract

The aim of this study is to highlight the thermo-mechanical treatments influence on internal friction behavior at high temperature of a copper single crystal alloy Cu-Al (11% at.). To do this; tests are performed, using the Isothermal Mechanical Spectroscopy technique (IMS), at different stabilized levels of temperatures, on two initial states of a specimen: quenched and cold worked at 1% by torsion after quenched.

In the two cases: the specimen have been progressively heated to approximately 1160 K and then cooled down to room temperature. During heating the annealing temperature ($T_{ANN}$) is equal to the temperature of measurement ($T_{MEAS}$) and during cooling after annealing $T_{MEAS}$ is lower than $T_{ANN}$.

The results obtained, on the two initial states, show globally the same behavior. They reveal the existence of three (3) independent relaxation peaks we call respectively: $P_Z$, $P_{MT}$ and $P_{HT}$. During heating the three peaks exist whereas during cooling after annealing remain only two (2) peaks: $P_Z$ and $P_{HT}$.

The $P_Z$ peak was identified to a Zener relaxation peak. Its origin being due to the reorientation, under constraint, of the pairs of aluminium atoms. The existence of the other peaks $P_{MT}$ and $P_{HT}$ appear to be associated with thermo-mechanical treatments. Indeed; $P_{MT}$ appears at average temperature and $P_{HT}$ at high temperature during heating but after cooling only $P_{HT}$ persists.

It is clear that the strain hardening is responsible for the up growth of $P_{MT}$ and is therefore linked to fresh dislocations introduced by cold working. The Arrhenius plots confirm this sensitivity to the annealing. Thus; these curves present two distinct slopes during heating and only one slope during slow cooling.

This behavior can be explained by an evolution of the dislocations microstructure. During heating this microstructure evolving continuously and becomes stable only after a high temperature annealing. Otherwise, these results are compatible with the predictions of Darynskyi's model.

Keywords: Isothermal mechanical spectroscopy, Relaxation peak, single crystal, strain hardening, dislocations.

1. INTRODUCTION.

The study of the high temperature internal friction is manifested, in general, by the presence of a relaxation peak which has long been attributed to grain boundaries. Later these same peaks were also observed in single crystals of pure metals [1-12].

Recent studies [13-15] on single crystals of pure aluminium, and copper have identified the mechanism responsible for the appearance of relaxation peaks observed on this materials type at the motion of dislocation segments inside cell walls or inside dislocation networks created by a small cold work or by handling. This microstructure is intimately linked to the stacking fault energy. Thus, for elevated values of the latter, the microstructure evolves quickly at relatively low temperature and, concurrently, the corresponding relaxation peak disappears [13]. On the contrary; for lower values, the evolution of the microstructure is slow
and the relaxation peak is relatively stable [14]. For sufficiently high cold working, a second relaxation peak can appear at higher temperature corresponding to a new sample microstructure. The influence of the stacking fault energy on the relaxation behavior can be investigated by experiments the single crystals of Cu-Al alloys. The high sensitivity of the stacking fault energy to the concentration of solute atoms (Al) for contents in the range 0-19% at. makes these alloys interesting for the study of this parameter's influence.

This study follows an anterior work [16] and reports the results of experiments performed on a single crystal of Cu-Al alloy with 11 at.% Al, on two states. During heating after quenching and during heating after 1% cold work followed by an in-situ cooling after annealing at high temperature: approximately 1160 K.

2. EXPERIMENTAL PROCEDURE.

Isothermal internal friction experiments were carried out with a forced torsional pendulum under a vacuum of 10-8 Pa. The pendulum and the experimental technique have previously been described [17]. In the case of the forced vibrations, Q-1 is equal to \( \tan \phi \) where \( \phi \) is the phase lag between the applied stress and the resulting strain. The measurement frequency ranged between 10 Hz and 10-4 Hz and five frequencies per decade were used. Experiments were performed with a maximum strain amplitude of 10-5.

The Cu-Al single crystal sample was cut by spark machining in bars of dimensions: 40 mm \( \times \) 4 mm \( \times \) 1 mm. Then the bar was chemically polished to remove the volume affected by machining and 1% cold worked in torsion. X ray diagrams made before and after internal friction experiments showed that no recrystallization occurred in the sample during the high temperature tests.

3. EXPERIMENTAL RESULTS

The Fig. 1 and 2, show the internal friction spectra obtained during the first heating after, respectively, quench and the 1% cold work. They exhibit a relaxation peak, called here \( P_{MT} \) that decreases with increasing the temperature of the experiments. This peak is very sensitive to annealing temperature as shown in Fig. 3. The increase of damping for temperatures higher than approximately 850K indicates that a new peak which we call \( P_{HT} \) grows progressively (Fig. 4 and 5), whose height increases with increasing the temperature of the measurement.

![Fig.1](image1.png)
![Fig.2](image2.png)
![Fig.3](image3.png)

Fig.1: \( Q^{-1} = f(\text{freq./Hz}) \)
First heating after quench. (\( P_{MT} \) peak)

Fig.2: \( Q^{-1} = f(\text{freq./Hz}) \)
First heating after 1% cold work. (\( P_{MT} \) peak)

Fig.3: Influence of the annealing on \( P_{MT} \) peak at \( T_{MEAS.} = 757K \)
After annealing approximately at 1160K, the internal friction spectra shown only $P_{HT}$.

The fig. 6 and 7, show only $P_{HT}$ peak. Down to low temperature, its height decreases with the temperature of measurement.

We note that the same behavior is observed on the two initial states (quenched and cold worked at 1% by torsion after quenched). For this reason, we will consider that the same mechanism is responsible for the appearance of relaxation peaks observed on the two states of this alloy. The internal friction evolution versus temperature at $10^{-2}$ Hz on the specimen, that cold worked at 1% by torsion after quenched, is represented on fig.8. It confirms the presence of $P_{MT}$ and $P_{HT}$ peaks during the first heating and the only presence of $P_{HT}$ during cooling. The annealing contributes to develop $P_{HT}$.

Finally, we can conclude that the height of $P_{MT}$ peak decreases with the annealing temperature and above 910K approximately, it is totally removed. The second peak $P_{HT}$ appears whose its height increases with the annealing temperature. It is clear that an existence of $P_{MT}$ is linked to the strain hardening caused by 1% cold work or by handling.

Consequently at this behavior, the Arrhenius plots for $P_{MT}$ and $P_{HT}$ peaks are influenced as shown in Fig. 9. It turns out that for the two peaks, their straights corresponding to the first heating haven’t the same slope.
On the contrary, after annealing at very high temperature: around 1160K, the plots are straight lines and the corresponding apparent activation parameters are: limit relaxation time $\tau_0 = 8 \times 10^{-11}$ s and activation energy $H \approx 2$ eV.

Until now, we have focused only on $P_{MT}$ and $P_{HT}$ peaks. It should be noted that at relatively low temperature, we also found the presence of one peak that disappears completely at 717K (fig.10). It moves towards higher frequencies while, at low frequency; the $P_{MT}$ peak begins to appear (right side).

This peak we called $P_Z$ is a relaxation peak and its presence does not depend on thermo - mechanical treatments. The relaxation parameters, deduced from the Arrhenius plot (fig.11), give the following values:

- $\tau_0 = 1.15 \times 10^{-16}$ s and $H$ (activation energy) $\approx 2$ eV.
- The pre exponential term indicates that it's the atomic jumps, what allows us to identify $P_Z$ at Zener peak whose existence is due to the reorientation of pairs of solute atoms (Al) under constraint.

4. Discussion.

This study has clearly evidenced the influence of the strain hardening on the internal friction behavior of the single alloy Cu-Al at 11%. This influence is reflected by the appearance of a $P_{MT}$ peak which disappears after a high annealing temperature (around 1160K).

Its existence is therefore closely linked to an introduction of fresh dislocations by the 1% cold work in torsion. During the first heating, its amplitude decreases progressively while a new peak $P_{HT}$ developed. Above 950 K approximately, the $P_{MT}$ peak is totally removed. After annealing at 1160K the measurements performed at various temperatures during cooling reveals only the $P_{HT}$ peak. The relaxation peaks observed could be explained by the presence of dislocation segments inside cell walls or network according [13]. Thus, $P_{MT}$ peak is attributed to the dislocation microstructure created by the 1% cold work or by handling.

This microstructure progressively changes during the first heating and above about 950K, a new dislocation arrangement spreads out giving rise to the appearance of $P_{HT}$ peak. This new microstructure is also progressively modified during the first heating and it becomes stable at high annealing temperature (1160K).
Thus the relaxation parameters have been obtained only for $P_{HT}$ peak after high temperature annealing and stabilization of the microstructure. The value of the limit relaxation time is compatible with a relaxation mechanism due to dislocations and the value of the activation energy with the model proposed by Caillard [19] involving a cross-slip mechanism between different nature planes of dislocations arranged inside dislocation wall [13].

However, our results may very well be explained by the Darinsky model based only on the dislocations density, their length and the distance between partials.

This model provides for two peaks: one at average temperature ($Q_{MT}^{-1}$) and the other at high temperature ($Q_{HT}^{-1}$). Thus:

$$Q_{MT}^{-1} = \frac{2\pi(1-v)}{3(2+v)} \rho d_0^2$$

Where predominant term is the distance between the partials and:

$$Q_{HT}^{-1} = \frac{8\pi(1-v)}{9(4-5v)} \frac{\rho L^2}{\log(L/r_0)}$$

Where predominant term is the length of dislocations loops.

Although this model treats only the dislocations nature and not the type of dislocations microstructure (walls, cells...), it explains very good our results. Indeed, the $Q_{MT}^{-1}$ is due to presence of dipoles introduced by cold work or by handling. When the temperature increases, the energy of stacking fault increases [20-23] so the distance between the partials dislocations decreases. This facilitates the recombination of dislocations partials (the destruction of dipoles inducing the decreasing of $Q_{MT}^{-1}$) giving the long dislocations segments and dislocations loops (increasing of $Q_{HT}^{-1}$).

This phenomenon explains the disappearance of $P_{MT}$ and the appearance of $P_{HT}$. So we can conclude that the Darinsky model is compatible with our results.

4. CONCLUSION.

This study has clearly evidenced the relaxation phenomena responsible of the internal friction observed on single crystal Cu-Al (11% at.). So three (3) relaxation peaks have been found:

$\bullet$ $P_Z$

This peak appears at relatively low temperature and it has been identified to the Zener peak whose existence is due to the reorientation of pairs of solute atoms (Al) under constraint.

$\bullet$ $P_{MT}$

This peak appears at average temperature and disappears with the annealing. It has been attributed to an introduction of fresh dislocations by cold work or by handling.

$\bullet$ $P_{HT}$

This peak appears at high temperature and persists during cooling after the high annealing temperature. It has been attributed to the motion of long dislocations segments in a stable microstructure.

LITERATURE

