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PRECIPITATION STAGES IN Al-Ag ALLOYS DETERMINED BY THE EVOLUTION OF THE INTERNAL FRICTION SPECTRUM

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Abstract.- The evolution of the internal friction spectrum during precipitation in Al-Ag alloys is characterized by the appearance of three atomic relaxation peaks and an evolution of the background due to the GP-zones evolution and to the precipitation hardening phenomena. Two of these peaks are Zener peaks due to the quenched solid solution and to the partially reverted one respectively, when the GP-zones are destabilized by the appearance of new metastable precipitates, the γ" precipitates. The third peak is due to atomic relaxations inside the γ phase precipitates (γ' and γ). Its thermodynamical characteristics show that the γ" precipitates are different from the γ' precipitates.

Introduction.- Numerous studies (1 to 14) devoted to the precipitation in the Al-Ag alloys have led to define the following sequence: α_{ss} → GP-zones → γ' → γ, where α_{ss} is the quenched supersaturated solid solution, the GP-zones spherical clusters of high silver concentration and the γ' metastable semicoherent precipitates of the equilibrium γ phase (Ag_{2}Al - hcp).

During this precipitation, hardness shows two maxima attributed respectively to the GP-zones (cold hardening stage) and to the γ' precipitates (warm hardening stage) (1,2). The cold hardening stage developed by isochronal ageings, is interrupted at ~450K by a sudden hardness drop attributed to the GP-zones reversion.

Yet this reversion stage cannot be supported by the small angles X-ray scattering results which show that the GP-zones are growing continuously when the hardness goes down (3-7). On the other hand, calorimetric measurements (10) present a heat absorption giving an account for a zones dissolution at this stage. But a total reversion has not been observed by transmission electron microscopy (8,9).

The apparent disagreement of the results obtained by the classical technics concerning the transformation stage "GP-zones → γ' precipitates" can give an account of the difficulty to explain the internal friction evolution during precipitation in the Al-Ag system.

As in the case of classical technics, the internal friction results obtained by different authors (11-14) are only in agreement among each other after the appearance of the γ' precipitates, which give rise to a stable relaxation peak. But, during the first precipitation stages, the results seem to be contradictory.
So depending on the experimental conditions it is possible to obtain one peak (11, 14), two peaks (12) or no peak (13) during the first increase of temperature after quenching. This complex evolution of the spectrum is due to the great sensitivity of internal friction to all changes of the microstructure.

In the present work the discovery of the origin of the relaxation peak associated with the γ' precipitates has led us to a new interpretation of the evolution of the internal friction spectrum during precipitation. This interpretation has been supported by results obtained by hardness and electrical resistivity measurements, and by electron microscopy observations (15, 16, 17).

Results and interpretation: During the first increase of temperature after quenching, the internal friction spectrum shows two peaks (P₁ and P₂) in concentrated Al-Ag alloys (more than 10 at % Ag) (fig. 1 a). For a lower amount of Ag, these peaks do not appear. (fig. 1 b). One can see only a saturation of the internal friction increase at ~450 K.

After annealing at ~520 K, the internal friction has decreased and a third peak P₃ appears associated with the γ'-precipitates. Once it has appeared, this peak remains stable during all treatments of overageing. This means that P₃ peak is insensitive to the shape modifications of the γ' precipitates. It is easy to understand the stability of P₃, when it is known that the anelastic relaxation takes place in the γ phase. P₃ peak appears in all its magnitude in a γ monophase alloy i.e. Al - 85 wt % Ag (fig. 2). In the two-phase alloys, the height of P₃ is directly proportional to the volume fraction of the precipitated γ phase. At the equilibrium, it is possible to predict the height of P₃ by the linear relation.
\[ Q_{\text{max}}^{-1}(c) = \frac{c}{c_{\gamma}} \] where \( c \) and \( c_{\gamma} \) are respectively the concentrations of silver in the alloy and in the precipitated \( \gamma \) phase. 

\( c_{\gamma} \approx 59.5 \text{ at } \% \text{ Ag} \).

In addition, the anelastic characteristics of the \( P_3 \)-peak are those of an atomic relaxation. Therefore, one can imagine that this relaxation is due to the stress induced orientation of elastic dipoles in the hexagonal \( \gamma \) phase.

In the Al-Ag alloys, the \( \gamma \) phase precipitates present an equilibrium concentration, given by the phase diagram (18), of 59.5 at \% Ag (\( \approx \) 85 wt \% Ag).

This value is lower than the stoichiometric composition \( \text{Ag}_2\text{Al} \) (66.6 at \% Ag).

Some Al atoms are in excess with regard to the stoichiometry.

So on the compact planes, these Al atoms can create elastic dipoles around them (fig. 3).

Fig. 2: \( P_3 \) peak, after background substraction, as it appears in the \( \gamma \) phase (Al - 85 wt \% Ag) and in the two-phase alloys (Al - 10, 20, 30 wt \% Ag).

Fig. 3: Two possible configurations of dipoles \( D_1, D_2, D_3 \) created by an excess Al atom in the \( \gamma \) phase. 
a) without stress  b) with an applied stress.
Without external stresses, the dipoles $D_1$, $D_2$, $D_3$ (fig. 3a) create an isotropic deformation of the lattice. But the deformation can become anisotropic by a stress induced orientation of one dipole (fig. 3b).

The intensity of such a relaxation gives a reasonable account for the height of the $P_3$ peak (15).

The new interpretation of the $P_3$ peak, which refutes the models based on the interfaces relaxation (13), allows one to claim that the Al-Ag alloys exhibit more than one peak during precipitation: the $P_1$ peak cannot be the $P_3$ peak because the $\gamma$ phase is absent in the quenched specimen.

So the evolution of the internal friction spectrum can be followed during a programme of isochronal ageings by a series of measurements made during heatings ($M_i$) and coolings ($D_i$) (fig. 4).

One can observe three main stages in the evolution of the curves: the disappearance of the $P_1$ peak in the temperature range where the GP-zones are growing ("cold hardening stage"), the appearance of an hysteresis in the internal friction curves associated with the $P_2$ peak (hardness reversion stage) and finally the disappearance of the hysteresis with the creation of the $P_3$ peak by the precipitation of the $\gamma$ phase ($\gamma'$ precipitates).

The characteristics of the $P_1$-peak which vanishes when the concentration in the solid solution is lowered, are those of an atomic relaxation. In addition, the height of this peak depends linearly on the square of the atomic concentration of
the solid solution. So the $P_1$ peak is a Zener (19) peak due to the quenched solid solution. Its disappearance allows one to follow the GP-zones evolution as in the case of residual electrical resistivity measurements (15,16).

The transition GP-zones + $\gamma'$ precipitates gives rise to internal friction curves which present an hysteresis phenomenon: one peak is present on the curves obtained during cooling and this peak is absent on the curves measured during heating. This means that there are two states of metastable thermodynamical equilibrium: one state connected with the creation of the peak and another one with its destruction.

This internal friction hysteresis is associated with an hysteresis of the electrical resistivity, which increases during the creation of the internal friction peak and decreases during its destruction (16).

In addition, transmission electron microscopy shows that new small precipitates appear at the temperature of creation of the peak, i.e. at $\sim 450\,K$ (fig.5). In the neighbourhood of these precipitates, which we can call $\gamma''$ precipitates, the GP-zones are smaller than in the matrix. This would mean that at 450K the alloy is composed of GP-zones, new $\gamma''$ precipitates and a solid solution enriched by a partial dissolution of the GP-zones.

The internal friction curves obtained during coolings show that $P_2$ peak is formed by two contributions (fig.6): an evolution of the internal friction which could be connected with the evolution of the GP-zones, and one relaxation peak which is only observed during cooling after a partial reversion of the GP-zones. This peak is not due to the $\gamma''$ precipitates because these are still present during the increases in temperature when the peak does not appear. On the other hand, the electrical resistivity measurements support the interpretation of this peak as a Zener peak due to the reverted solid solution in the neighbourhood of the $\gamma''$ precipitates.
The growing and fusion of the $\gamma''$ precipitates lead to the appearance of the $\gamma'$ precipitates which are responsible for the stable $P_3$ peak.

**Conclusions.** - The internal friction spectrum associated with precipitation in the Al-Ag alloys is composed of three relaxation peaks: $P_1$ and $P_2$ due to the quenched and reverted solid solutions respectively, and $P_3$ due to the $\gamma$ phase precipitates.

The evolution of this spectrum allows one to define the following sequence:

$$\alpha_{ss} \rightarrow \text{GP-zones} \rightarrow \gamma'' \rightarrow \gamma' \rightarrow \gamma$$

with the following phenomena:

- $\alpha_{ss} \rightarrow \text{GP}$: disappearance of $P_1$ peak
- $\gamma'' \rightarrow \gamma'$: hysteresis phenomenon, with creation of $P_2$ peak due to reversion
- $\gamma' \rightarrow \gamma$: diminution of $P_3$ peak, increase of background

The $\gamma''$ precipitates are not small $\gamma'$ precipitates, because they do not give rise to a $P_3$ peak which is characteristic of the $\gamma$ phase. On the other hand, they are not GP-zones, because they serve as nuclei for the $\gamma'$ formation.

**References**