Internal Friction and Young Modulus Behaviour of Hot-Rolled Cu-Al-Ni-Ti Shape Memory Alloys

E. Cesari, C. Seguí, J. Pons, F. Perelló

► To cite this version:

E. Cesari, C. Seguí, J. Pons, F. Perelló. Internal Friction and Young Modulus Behaviour of Hot-Rolled Cu-Al-Ni-Ti Shape Memory Alloys. Journal de Physique IV Proceedings, 1996, 06 (C8), pp.C8-413-C8-416. 10.1051/jp4:1996889 . jpa-00254700

HAL Id: jpa-00254700

<https://hal.science/jpa-00254700>

Submitted on 1 Jan 1996

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

Internal Friction and Young Modulus Behaviour of Hot-Rolled Cu-Al-Ni-Ti Shape Memory Alloys

E. Cesari, C. Seguí, J. Pons and F. Perelló

Dept. Física, Universitat Illes Balears, Campus, 07071 Palma de Mallorca, Spain

Abstract. The internal friction (IF) and modulus behaviour during thermoelastic martensitic transformation in grain refined Cu-Al-Ni-Ti shape memory alloys hot rolled at 650°C and 850°C has been studied using a three-point bending Dynamical Mechanical Analyser. In both alloys, Ti-rich X-phase particles, μm sized, are formed and inhibit grain growth. Lower IF peaks and modulus minima have been observed in the samples rolled at 650°C, which is related to the presence of micrometric γ_2 -phase particles. During ageing at 250°C, a decomposition process in the form of a fine dispersion of precipitates with sizes of a few nm after 50-80 hours ageing has been observed, together with a progressive reduction in the IF peaks and modulus minima of the martensitic transformation. These reductions are attributed to a loss of mobility of the parent/martensite interfaces in a oscillating stress cycle caused by the precipitates.

1. INTRODUCTION

Cu-Al-Ni alloys with the addition of Ti as grain refiner show thermoelastic martensitic transformation and concurrent shape memory effect at high temperatures (100-300°C) [1]. The applicability of the alloys depend on the stability of the parent (β , L2₁) and martensite (β' , 18R) phases, and, on the other hand, the effect of production processes such as hot rolling.

Long term ageing in the parent phase of the Cu-Al-Ni-Ti alloys leads to precipitation of γ_2 phase. These precipitates add to the X-phase precipitates which are present in Ti-bearing alloys [1], and both affect the martensitic transformation (MT) and specially the internal friction (IF) and modulus behaviour [2,3]. As a matter of fact, the internal friction maximum associated with the martensitic transformation has been found to decrease with increasing amount of X-phase [2], a small volume fraction of those precipitates being enough to suppress almost completely the IF peak. This effect has been explained in [2] by the decreasing mobility of the parent/martensite interfaces due to grain boundaries and fine dispersion of X particles. Less information is available concerning the effect of γ_2 -phase precipitates on the IF behaviour.

In this work the evolution with ageing in the parent phase of a hot rolled Cu-Al-Ni-Ti alloy has been studied. The relationship between microstructural changes and mechanical behaviour will be analyzed paying attention to the role of X-phase and γ_2 -phase precipitates on the IF and modulus results.

2. EXPERIMENTAL

A polycrystalline alloy with nominal composition Cu-11.9 Al-4.8 Ni-1.0 Ti wt % was hot rolled at 850°C (alloy 1) and 650°C (alloy 2) and water quenched to room temperature. Ageing in the parent phase was carried out in a salt bath at 250°C. The internal friction and modulus as a function of temperature were measured in a Dynamic Mechanical Analyzer Perkin-Elmer DMA-7 working in three point bending configuration. The phase difference ($\tan\delta$) between the sinusoidal applied stress and resulting strain gives the IF, and the elastic or storage modulus (E) is obtained as the real part of the complex modulus.

Prismatic samples with dimensions about $0.5 \times 3.5 \times 18 \text{ mm}^3$ were used for DMA measurements. DSC experiments were also carried out in a Perkin-Elmer DSC-7. Transmission Electron Microscopy (TEM) observation of samples in as received and aged conditions were performed using a Hitachi H600 100 kV. Thin foils were prepared by double jet electropolishing in 30 % nitric acid in methanol at 240 K.

3. RESULTS AND DISCUSSION

The results obtained from the DMA measurements performed on alloys 1 and 2 after different ageing time are shown in figures 1 and 2, and are summarized as follows:

Figure 1.- Evolution with ageing time of the transformation temperatures and hysteresis, IF peak values and corresponding E modulus minima for the studied alloys.

For alloy 1, rolled at 850°C , the peak temperatures (M_p and A_p , representative of the forward and reverse transformations respectively), remain almost constant up to 1h ageing at 250°C , increasing afterwards in a sustained way up to 65h. Both A_p and M_p increase, but M_p does by less than A_p , producing an increase of the transformation hysteresis ($\Delta T = A_p - M_p$) which becomes noticeable after 6h ageing. As for alloy 2, rolled at 650°C , the transformation temperatures and hysteresis are higher than those of alloy 1 in as-rolled and aged condition; both M_p and A_p increase after about 1h ageing, although to a lesser extent than alloy 1, while ΔT strongly rises after about 6h ageing. After about 40h ageing, M_p starts to decrease. DMA and DSC experiments gave out the same behaviour for the transformation temperatures and hysteresis.

The maximum value of the internal friction peak during the forward and reverse transformations decreases with increasing ageing time for both alloys 1 and 2, alloy 2 showing lower $\tan\delta$ peaks for all ageing times. Nevertheless, the martensitic transformation is spread over a larger temperature range in the case of alloy 2, and as a consequence, the integrated internal friction peak shows comparable values for alloys 1 and 2, decreasing in both cases with increasing ageing time. It has to be emphasized that for the used stress amplitude (4 MPa), the intrinsic damping in martensite (and in β -phase) is below the resolution of the DMA, which facilitates integration of the IF peak.

The storage (E) modulus values in β and martensite phases do not show remarkable differences for alloys 1 and 2, and no evolution with ageing time has been observed, being $E_\beta > E_m$ in all cases. On the

contrary, the modulus minimum which accompanies the forward and reverse martensitic transformations progressively diminishes with increasing ageing time until it disappears. Indeed, "complete" disappearance of the modulus minimum is only seemingly occurring, since the modulus level with respect to which the minimum has to be evaluated is unknown. The relative value of the modulus minimum ($\Delta E/E = (E_m - E_{min})/E_m$) is lower for alloy 2 in as-rolled as well as in aged condition, and "complete" disappearance occurs at a shorter ageing time (about 20h) than in the case of alloy 1, where disappearance occurs after 60h. The transformation heat was also measured as a function of ageing time for alloys 1 and 2. No changes were observed for alloy 1 even after 65h ageing at 250°C. On the contrary, the transformation heat started to decrease after about 20h ageing of alloy 2.

Figure 2.- IF and E modulus vs temperature spectra obtained during heating after 5 min and 65h ageing at 250°C (stress amplitude 4 Mpa, frequency 1 Hz, temperature rate 5 K/min).

For the as-rolled samples, optical and electron microscopy observations performed at room temperature show the presence of intra and intergranular particles with sizes of the order of μm in a martensitic matrix (fig. 3). In the case of alloy 1, rolled at 850°C, all particles identified from the selected area electron diffraction patterns were Ti-rich X phase (designated as X_L in [4]), whereas X and γ_2 phase particles were found in as-received alloy 2, rolled at 650°C. In this sense, it is worth to notice that for the studied composition 650°C corresponds to the $\gamma + \beta$ region in the equilibrium phase diagram, while 850°C lies inside the single β phase region. The X and γ_2 particles look very similar to each other, since they have the same size and undefined shapes; both kind of particles are incoherent with the matrix. The grain size is around 15-20 μm for alloy 1 and 5-10 μm for alloy 2.

Figure 3.- TEM image of the as-rolled alloy 2, showing the X and γ_2 particles in martensite.

Figure 4.- Small precipitates observed after 85h at 250°C in a sample of alloy 1.

During ageing at 250°C, a slow decomposition process has been observed for both alloys. At the beginning (*i.e.* after 10h at 250°C), the TEM images show a very finely mottled contrast, but after a more prolonged ageing (*i.e.* observations performed after 50 and 85h), small individual precipitates with sizes of few nm can be distinguished forming a dense dispersion throughout the sample (fig. 4). The precipitates

are more densely accumulated at the B2 and L₂₁ antiphase boundaries, making them visible even in bright field images. Due to their very small sizes, no extra diffraction spots could be observed, but despite the lack of an accurate identification, they are believed to be most probably γ phase, coherent with the parent β phase. For alloy 2, in addition to the above fine precipitation, growth of the big γ_2 particles has been observed during ageing at 250°C. This leads to an increase of the volume fraction of precipitated particles, estimated to be about 7% in as-rolled condition and about 15% after 85h at 250°C. It has to be noticed that the X particles are not affected by ageing, while the fine precipitates do not constitute an appreciable fraction of material.

According to TEM observations, it can be stated that the fine γ_2 precipitation observed for both alloys 1 and 2 upon ageing is responsible for the observed evolution of A_p , M_p and ΔT , while the presence of the big γ_2 particles in alloy 2 causes the transformation temperatures and hysteresis for this alloy to be higher than for alloy 1 both in as-rolled and aged conditions. Improvement of nearest-neighbours short range order, strain fields around the precipitates and Al depletion of the matrix are the more feasible causes for this behaviour as it occurs in other Cu-based alloys [5,6].

As for the DMA results, the lower IF peaks (and less pronounced modulus minimum) and larger temperature spread of the IF peaks in alloy 2 are thought to be a consequence of the big γ_2 particles present in this sample, while the finely dispersed precipitates are held responsible for the observed progressive decrease during ageing of both alloys at 250°C. A decrease in $\tan\delta$ values and integrated IF peaks could arise from a decrease in transformed fraction, since, as a first approach, the IF values depend on the amount of transformed fraction in an oscillation cycle performed at each temperature. Nevertheless, the fact that the transformation heat (which is considered to be proportional to the amount of transformed material) remains constant except for the longest ageing time in alloy 2, allows to rule out this possibility. Being the IF peak during the MT essentially due to the hysteretic movement of parent/martensite interfaces [7], the decrease in IF values is attributed to the decreasing mobility of these interfaces caused by the precipitated particles: the big ones (X and γ_2) cannot be overcome by the growing parent-martensite interfaces, causing a considerable reduction of the martensite plates size, while the small and coherent precipitates are actually absorbed by the martensite plates when they grow [6], a process which is energetically expensive (due to the accommodation of precipitates in the martensite phase [8]) and, then, limits the displacement of the interfaces under the oscillating stress. The enlargement of the transformation temperature range caused by precipitation of γ_2 and the decrease in strain per oscillating cycle caused by the poor mobility of the interfaces are responsible for the progressive disappearance of the E-modulus minimum.

Acknowledgments

A.M.T. company (Belgium) is gratefully acknowledged for providing the studied alloys. Partial financial support has been received from the Spanish CICYT (project MAT93-0188) and DGICYT (PB94-1173).

References

- [1] Sugimoto K., Kamei K., Komatsu S., Akamatsu K. and Sugimoto T.; *J. de Phys.*, **12** (1982) C4-761.
- [2] Nakaniwa M., Sugimoto K., Kamei K., Nakamura Y. and Sugimoto T.; *J. Japan Inst. Metals*, **56** no. 1 (1992) 30.
- [3] Hurtado I., "Order, defects, precipitates and the Martensitic Transformation in β -phase Cu-Al-Ni base alloys". Doctoral Thesis, Dept. MTM, Katholieke Universiteit Leuven, 1994.
- [4] Adachi K., Hamada Y. and Tagawa Y., *Scripta Metall.* **21** (1986) 453.
- [5] Rodríguez P. and Guénin G.; *Mat. Sci. and Eng. A*, **129** (1990) 273.
- [6] Pons J., Cesari E.; *Acta Met. Mat* **41** (1993) 2547; Lovey F.C., Cesari E., Auguet C., Mañosa Ll., Rapacioli R.; *Mat. Sci. Forum*, vol **56-58** (1990) 493.
- [7] Van Humbeeck J., Stoiber J., Delaey L. and Gotthard R.; *Z. Metallkde.*, **86** (1995) 176.
- [8] Lovey F.C., Torra V., Isalgué A., Roqueta D., Sade M.; *Acta Met. Mat.* **42** (1994) 453.