MECHANICAL SOFTENING KINETICS AT HIGH TEMPERATURES IN AN AlMgZnCu ALLOY : EXPERIMENTAL CHARACTERIZATION AND MICROSTRUCTURAL INTERPRETATION[†]

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ABSTRACT The high temperature precipitation developed at low cooling rates from the solutionizing temperature drags out solutes from the solid solution and lowers the solid solution hardening. This effect has been studied using an interrupted quenching procedure for high temperature isothermal holdings. The yield stress decreases as the time of holding increases, and in-situ resistivity measurements allow to relate the softening to the loss of solute content from the solid solution. The relation between can be expressed with the classical power law (n=2/3) for low solid solution depletions.

Keywords : *7000 alloys; mechanical softening; heterogeneous precipitation; solid solution hardening*

1. INTRODUCTION

To obtain optimal mechanical properties, AlMgZnCu alloys undergo a complex thermomechanical sequence : hot rolling, solutionizing, quenching and aging. Quenching is a critical step in this sequence when manufacturing 7xxx thick products. Precipitation can indeed occur during the cooling itself when cooling rates are too low. This precipitation is non-hardening when developed at high temperatures but can induce a significant hardening when homogeneously nucleated coherent precipitates are formed in a low temperature range. In both cases the supersaturation of the solid solution is decreased, thus lowering the hardening potential of the alloy for subsequent aging [1,2,3]. Optimal thermal treatments have thus to be performed in such a way that the loss in hardening potential remains limited as well as the amount of internal stresses [4]. To precise these optimal treatments, numerical simulations are performed where the temperature variations, the microstructure evolution (precipitation kinetics) and the internal stresses are calculated (see for example [5] in the case of steels).

Our work deals with the study and the modeling of the precipitation kinetics as well as the mechanical behaviour during the quench from the solution treatment temperature. In this paper, we focus on the mechanical softening induced by the depletion of the solid solution associated with the heterogeneous precipitation during quenching.

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2. EXPERIMENTAL PROCEDURE

The alloy investigated is an industrial 7010 alloy (Al-5.99Zn-2.33Mg-1.73Cu in wt%) which was cast and rolled to 100 mm thick plate by Pechiney Rhenalu Issoire. Tensile samples (4 mm in diameter cylinders with a gauge length of 22 mm) and "electrical resistivity" samples (3 mm in diameter cylinders) were all taken from quarter-plane along the rolling direction.

Thermal and thermomechanical treatments were performed on a laboratory thermomechanical testing apparatus (DITHEM) where the thermal variations as well as the mechanical testing are computer controlled. Electrical resistance variations were continuously measured during the thermal treatments with a "four points" method developed in the laboratory in order to reach the precipitation kinetics. Independently, thermomechanical treatments were performed to measure the evolution of the yield stress. Figure 1 is a sketch of the thermal and mechanical set point curves for the thermomechanical tests. After solutionizing (30 min at 475°C) and in order to avoid any precipitation during the cooling, the samples were cooled at a 50°C/s controlled cooling rate between 475°C and the isothermal holding temperature. To obtain the mechanical behaviour of the alloy for different precipitation amounts, the specimens were tensile tested after different holding times (one specimen per test). Each test was performed with a deformation rate of 10^{-3} s^{-1} up to a 1% deformation. The variations in electrical resistivity were measured for similar thermal treatments. Results are given for isothermal transformation at 400, 350 and 300°C.

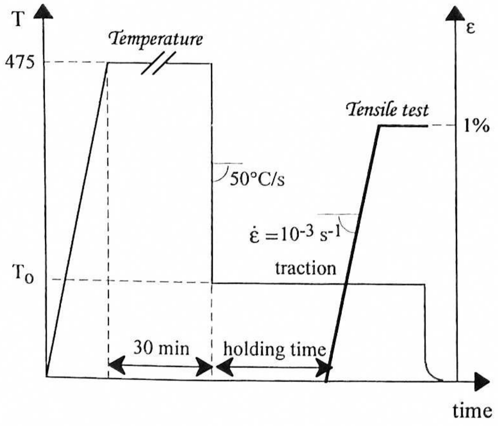


Fig. 1 : schematic variations of temperature and strain during the thermomecanical test

3. RESULTS AND DISCUSSION

3.1 Mechanical properties variations

A set of experimental stress-strain curves obtained for different holding times at 300°C is presented on figure 2. A decrease in the yield stress is observed as the holding time increases. For the holding time of 20 s, the yield stress decreases slowly as deformation increases. This dynamic softening is not evidenced for the other holding times. From these curves we obtain the variations in 0.2% yield stress which are plotted in figure 3 for the three test temperatures versus holding time. The values are obtained with an uncertainty of ±5 MPa. Two main points can be outlined :

1. The values for 20 s vary considerably with the test temperature (28 MPa at 400°C, 72 MPa at 350°C and 118 MPa at 300°C).
2. A softening is observed on the three curves. It is followed by either an asymptotic plateau at 400 and 350°C reached after 100 s or a smoother second decrease at 300°C after 300 s.

3.2 Microstructure evolution

The resistivity variations obtained for the three test temperatures are shown figure 4. The initial decrease is mainly due to the thermal component (cooling at 50°C/s before holding). This is followed by a smoother variation and in each case the electrical resistivity tends to an asymptotic value after about 1 h. In order to relate the electrical resistivity to the depletion of the solid solution [6], we have measured the resistivity of pure aluminum for the same thermal conditions. We thus define the differential electrical resistivity [7] as the difference between the electrical resistivity of the investigated alloy and of pure aluminum. At these temperatures, this quantity is directly related to the amount of solutes in solid solution via the Mathiessen's rule [8] because the contribution of precipitates to the resistivity is negligible in this temperature range [9]. Moreover, microstructural observations by TEM permitted to relate the observed macroscopic precipitation kinetics to microscopic precipitation characteristics [10].

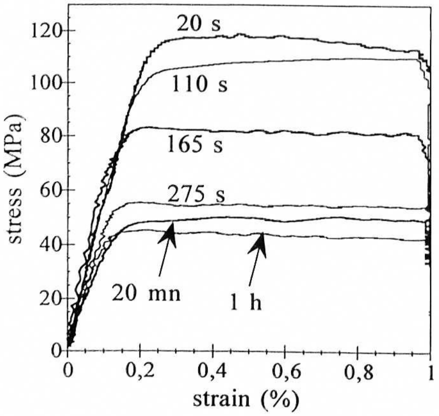


Fig. 2 : stress/strain curves obtained after different holding times at 300°C

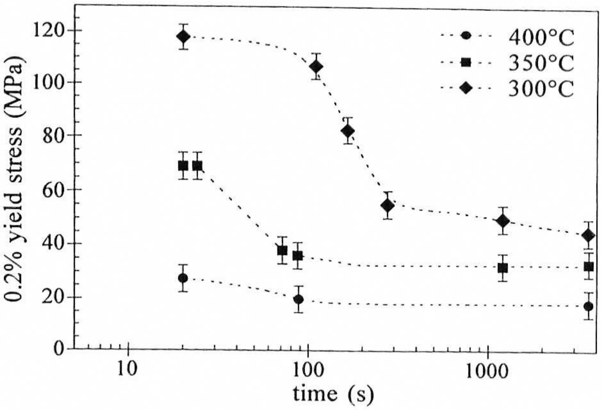


Fig. 3: yield stress evolution during high temperature holding

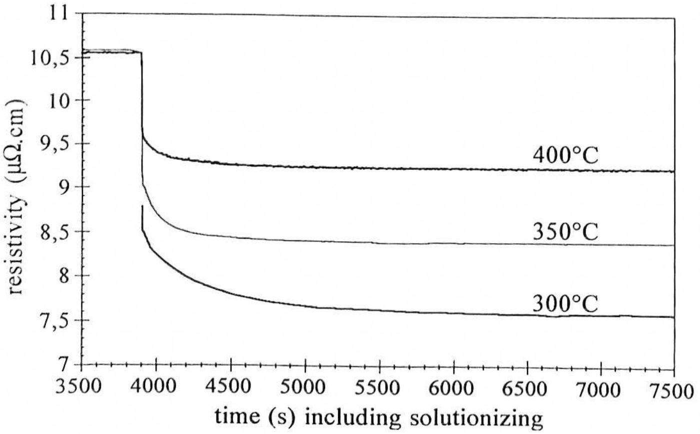
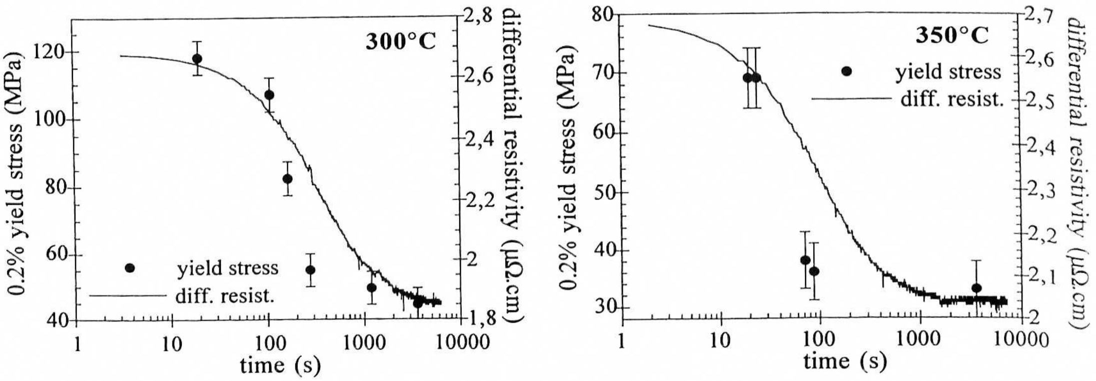


Fig. 4 : electrical resistivity variations on isothermal holdings

The experimental softening kinetics is compared to the differential resistivity variation for each test temperature on figure 5. Different scales have been used so that direct comparisons are made possible between both kinetics. These figures reveal a clear correlation between the yield stress and the resistivity variations. However, the mechanical softening seems faster than the resistivity decrease for the end of the precipitation.

Assuming an effective correlation between the two properties, extrapolation of the 0.2% yield stress to the short times (no precipitation with $\Delta\rho$=2.67 μΩ.cm) has been graphically performed. This allows estimating the 0.2% yield stress of the supersaturated solid solution and for the longest times, the softening due to the complete transformation. The results are presented table 1.



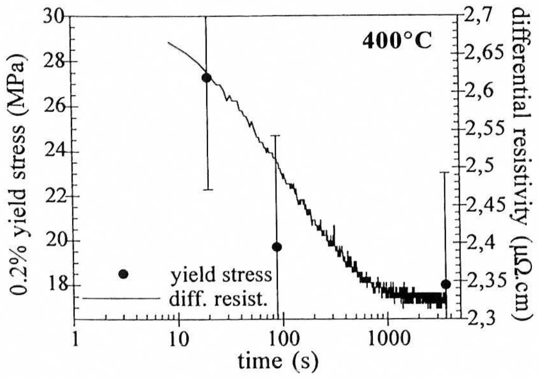


Fig. 5 : comparisons between the yield stress and and the electrical resistivity fort the three isothermal holding temperatures

Temperature (°C)	Extrapolated 0.2% yield stress of the supersaturated solid solution (MPa)	Maximum softening amplitude (MPa)	Δρ at the end of precipitation (µΩ.cm)
300	122	85	1.85
350	78	46	2.04
400	29.5	11.5	2.32

Table 1 : Extrapolated 0.2% yield stress and maximum softening amplitude associated with the precipitation for the three temperatures.

3.3 Correlation between mechanical softening and solid solution depletion

The yield stress of the solid solution is strongly dependent on the solutes concentration. For the high temperature range investigated, the incoherent precipitates are considered as non hardening. The softening observed during the isothermal holdings can then be understood as a consequence of the heterogeneous precipitation which drags out solute atoms from the solid solution.

From our experimental results, the softening amplitude is correlated to the Mg content out of the solid solution. This is calculated from the experimental differential resistivity using the Mathiessen's rule and assuming that :

1. precipitates belong to the η Mg(Zn_2, AlCu) phase. This assumption has been verified by TEM, see [10].
2. η precipitates have the same composition : 0.75 wt% $MgZn_2$ [11].

Results of this correlation for the 300°C isothermal holding are shown on figure 6 where the 0.2% yield stress softening Δσ (difference between the extrapolated yield stress for the supersaturated solid solution and the current value) is plotted against the Mg loss from the solid solution ΔX_{Mg} (difference between the Mg concentration in the supersaturated solid solution and in the current solid solution). Results for the 350 and 400°C isothermal holdings are presented on figure 7.

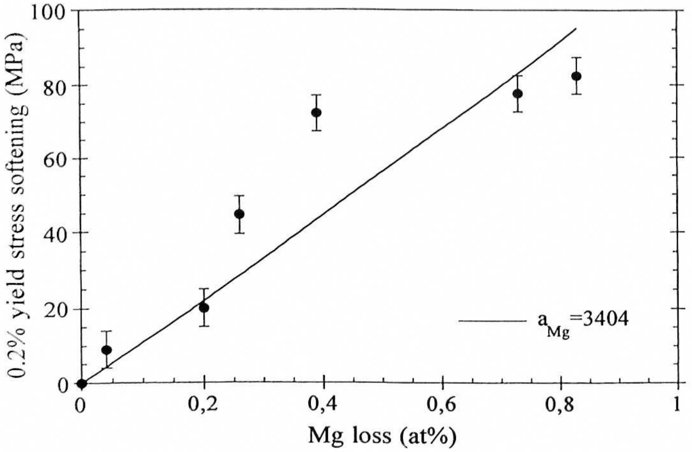


Fig. 6 : correlation between the experimental mechanical softening and the loss of Mg content in the solid solution deduced from the resistivivty measurements (300°C)

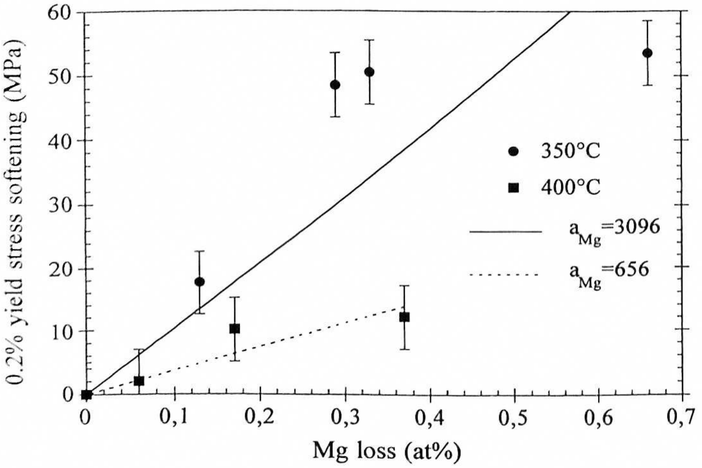


Fig. 7 : correlation between the experimental softening and the Mg loss from the solid solution deduced from resistivity experiments for 350 and 400°C

The hardening potential of solute atoms in an aluminum matrix is usually expressed as [12] :

$$\sigma(T) = \sigma_0(T) + \left(\sum_i a_i(T).X_i \right)^n \qquad (1)$$

where σ_0 represents the contribution of the matrix, a_i are temperature-dependent coefficients and X_i is the concentration of element i in the matrix. Numerical values of a_i can be found in [13]. Copper has the strongest hardening effect, followed by magnesium. Zinc has a little hardening effect. The value of n is given by [14] as 2/3.

Copper is present in η precipitates at a low volume fraction (see assumption 2 above) and its variation in solid solution can therefore be neglected. Zinc in solid solution has a little hardening effect and its influence is also neglected. The softening is then expressed as a function of the magnesium concentration only and eq. (1) links the mechanical softening of the matrix $\Delta\sigma$ to the loss in Mg content ΔX_{Mg} (X^{ss0} is the concentration for the supersatured solid solution) :

$$\Delta\sigma = \left[a_{Mg} . X_{Mg}^{ss0}\right]^{2/3} . \left\{1 - \left[1 - \frac{\Delta X_{Mg}}{X_{Mg}^{ss0}}\right]^{2/3}\right\} \qquad (2)$$

Eq. (2) is plotted in figure 6 and 7. In the concentration range investigated ($\Delta X << X^{ss0}$), it gives a linear relationship between $\Delta\sigma$ and ΔX_{Mg}. We found that the coefficients a_{Mg} decrease with temperature : 3404 at 300°C, 3096 at 350°C and 656 at 400°C (concentrations in at%).

Eq. (2) gives a satisfactory representation of experimental results for a low depletion of the solid solution (ΔX_{Mg} < 0.2 at%). Discrepencies are observed for higher depletions. To explain these differences, we should outline that the demonstration of eq. (1) is done at low temperatures [15]. In our case, the change of deformation mechanism can influence the behaviour of the solid solution. Indeed, when the temperature is increased, the deformation mechanism changes from dislocation slip to (intra- and intergranular) dislocation creep (dislocation climb) and to diffusion creep. According to Ashby's deformation maps [16] both dislocation slip and creep can be observed in pure aluminium at 300°C (T/T_f=0.6). For the higher temperatures, the creep contribution will be larger.

Also some differences may be induced by the assumption of constant composition for η precipitates. If η composition changes, the kinetics of the Mg loss will be modified as their precipitation progress whereas a significant fraction of copper can be dragged out from the solid solution.

4. CONCLUSION

The variations of mechanical properties have been determined on isothermal holdings i.e. for different precipitation contents in the 300-400°C temperature range. The resistivity variations monitored during similar thermal treatments allowed to study the precipitation kinetics. Both mechanical properties and precipitation kinetics were found related. The mechanical softening is due to the solute depletion of the solid solution. A reasonable quantitative link between the mechanical softening and the Mg content out of the solid solution has been found using a power law type (n of 2/3) in the low solid solution depletion range.

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