Precipitation Processes in a Cu-18 at%Li Alloy Produced by Electrodeposition

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The occurrence of precipitation processes in Cu-Li alloys, produced by electrodeposition, with lithium concentrations close to 18 at%, has been studied. Analytical methods employed include the following techniques: thermogravimetry, differential scanning calorimetry, thermo-electric power, X-ray diffraction, amplitude-dependent damping and modulus measurements.

A precipitation process at low temperatures occurs in the range 325 to 425 K. Subsequently, these precipitates disappear at temperatures between 425 and 525 K. Furthermore, another precipitation process at higher temperatures around 525 K was observed.

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I. Introduction

In the field of light metal alloys, there has been great interest in the aluminium-lithium based alloys. These alloys have been widely studied because of their potential applications in the aerospace industry, due to the lithium addition that produces a simultaneous reduction in the density and an increase in the elastic modulus of the resultant alloy⁽¹⁾⁻⁽⁴⁾. Beryllium additions have a similar effect to lithium on the mechanical properties of aluminium, but the cost and toxicity of beryllium complicate its industrial application. The copper-beryllium alloys have also been widely studied in the literature due to their excellent mechanical properties, high electrical conductivity and high corrosion resistance. Cu-Be alloy is employed widely as a traditional alloy for the fabrication of springs in electronic devices⁽⁵⁾⁻⁽⁹⁾.

In contrast, very scarce information has been reported on the copper-lithium alloys. In fact, the only information about this alloy available in the literature is related to the phase diagram, production from an electrodeposition process, diffusion coefficient and dynamical behaviour⁽¹⁰⁾⁻⁽¹⁶⁾. Recently, the possible existence of precipitation processes in Cu-Li alloys was reported based on a qualitative study of the damping behaviour⁽¹⁵⁾.

In the current work, the occurrence of precipitation processes in Cu-Li alloys have been carefully and system-

atically checked employing the following techniques: thermogravimetry (TGA), differential scanning calorimetry (DSC), thermo-electric power (TEP), X-ray diffraction (XRD), amplitude-dependent damping (ADD) and modulus measurements.

The occurrence of a precipitation process within the temperature range 325 to 425 K was found. The reversion of these precipitates appears at temperatures between 425 and 525 K. Furthermore, another precipitation process at higher temperatures around 525 K was also observed.

II. Experimental Procedure

1. Samples

Copper-lithium alloys were obtained by electrodeposition using a sheet of high-purity copper (99.99%) as the cathode and a similar size graphite sheet as the anode⁽¹⁴⁾. Electrodeposition was done in a bath of fused salts of lithium at constant temperature of about 753 K with both electrodes vertically positioned inside the fused salts and parallel to each other. A constant direct current, $I=1.5$ A, was allowed to circulate during a given period of time. The size of each electrode was about $(50 \times 25 \times 0.1)$ mm³ and the distance between the electrodes was 10 mm. Samples were electrodeposited for different periods of time. When the electrodeposition was finished, the samples were washed in water at room temperature to remove the excess of lithium and subsequently they were stored in air at room temperature for times longer than 10^6 s. Three samples, labeled (a), (b) and (c), were analyzed in the

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Table 1 Sample conditions.

Sample denomination	Electrodeposition time (min)	Lithium content (at%)
(a)	5	1.07
(b)	10	1.50
(c)	15	2.01

present work. The status of the studied samples is summarised in **Table 1**.

Preceding each kind of test, the external layers of Cu-Li crystals of the cathode were removed by mechanical polishing. **Figure 1** shows a scanning electron micrograph of the sample after mechanical polishing, with the remnant Cu-Li crystals on the surface.

Cu-Li crystals formed on the surface of the cathode have a composition of 18 at%Li, as determined by both emission chemical analysis and atomic absorption analysis. In Table 1, we quote the lithium content of the electrodeposited samples determined by atomic absorption analysis. However, although the lithium concentrations listed in Table 1 are referred to the total lithium content of the sample, the samples may have non-uniform variations of the lithium concentration. In fact, as can be seen from the scanning micrograph of **Fig. 2**, inclusions of

Fig. 1 Scanning electron micrograph of the remnant Cu-Li crystals on the surface of the sample, after mechanical polishing.

Fig. 2 Inclusions of Cu-18 at%Li on the Cu surface, under the shell of crystals.

Cu-Li crystals can be observed on the sample surface, under the crystal shells. These inclusions have a similar lithium content to the crystals in the external layers, as determined by back scattered-electrons analysis. Therefore, the zones around these inclusions should have a variation in the lithium concentration.

The electrodepositions were done with as-received copper *i.e.* copper in the thermomechanical state as commercially provided.

2. Measurements

TGA and DSC measurements were performed by using a Perkin-Elmer 7000 Series Thermal Analysis System, under blowing nitrogen with a heating rate of 10 K per minute.

As a function of time, *in situ* TEP measurements were carried out at different ageing temperatures, employing pure aluminium blocks. The equipment operates under high vacuum at temperatures between room temperature up to about 800 K. This experimental device is very similar to that described in Ref. 17. Employing a Wheatstone bridge and a data acquisition system, measurements of electromotive force were performed with an error less than 4%. The ageing temperature, T_a , was defined as $T_a = T + \Delta T/2$, where T is the temperature of the block at lower temperature and ΔT is the temperature difference between the two blocks of the TEP device. T_a was kept constant with a stability of ± 0.05 K.

Samples for TEP measurements were cut from the polished cathode in sheets of $(0.1 \times 2 \times 40)$ mm³. A small sample is required to avoid temperature gradients between the sample and the equipment blocks.

XRD measurements were carried out in a powder diffractometer, manufactured by STOE, working in the transmission mode at room temperature, employing a monochromator and a rotational sample device to eliminate directional texture effects. The measurement conditions were 40 kV and 20 mA using the K_α of Cu as incident radiation. Prior to XRD studies, the samples were treated as follows: they were put in a high vacuum atmosphere for 0.5 h subsequently heated at 1 K/min until reaching pre-determined different temperatures with argon flush, and quenched in water at room temperature.

The damping, D , was measured employing a traditional torsion pendulum of the inverted type which works both at low and high temperatures, under vacuum of the order to 10^{-5} Pa in the sample environment. The recording of the oscillations was made with the usual optical system, including a mirror and a photodiode. This device is driven by an automatic digital data acquisition system.

Damping measurements were carried out in free decay at frequencies close to 1 Hz at a ramping speed of 1 K/min, employing a maximum shear strain on the sample of less than 5×10^{-5} . The damping was measured with an error less than 1%. The samples employed were sheets of $(2 \times 0.1 \times 20)$ mm³.

The amplitude dependent damping (ADD) effects were carefully checked as a function of temperature⁽¹⁸⁾⁽¹⁹⁾. The freely decaying torsional amplitudes A_n , $n=0, 1, 2, \dots$,

N were measured at a constant temperature by means of a data acquisition system. The temperature during the decay was kept constant with a stability of ± 0.2 K. As a first step, polynomials were fitted to the data $\ln(A_n)$ vs. n representing the decaying oscillations. The damping as a function of amplitude was determined by the first derivative of the polynomial $\ln(A_n)$ against n . The results were depicted as D against the maximum strain on the sample, $\varepsilon_m^{(20)}$. Best fits were obtained with polynomials of degree ≤ 3 . It should be emphasised that the linearity of the oscillating amplified signal response during the decaying oscillations was accurately checked. The linearity can be assured up to the ratios of $A_0/A_n = 10^4$. This point is crucial for a check of ADD effects.

III. Results and Discussion

As a first step, the stability of the Cu-Li alloy within the temperature range 300–800 K was checked by means of TGA measurements. The relative weight, ΔW , as a function of temperature for the sample of type (c) (see Table 1) during heating is shown in Fig. 3. ΔW is defined as $(W_i - W(T))/W_i$, where W_i and $W(T)$ are the initial weight and the weight at temperature T , respectively. Similar behaviour was found in the thermogravimetry test of all three types of samples. Therefore the loss of mass can be neglected within the temperature range studied, indicating that the alloy is stable, without lithium loss.

The temperature dependence of the microstructure was examined by DSC thermograms, where the peaks of heat flow, HF, indicate the precipitation sequence in the alloy⁽²¹⁾. Figure 3 shows the average thermogram over three heating runs performed on different samples of type (c). Four small peaks A, B, C and D appear sequentially during the increase of temperature.

A straightforward interpretation led to the conclusion that the exothermic A-peak is related to a precipitation process, while the endothermic B-peak indicates the reversion of the precipitates. The C-peak could be related to another precipitation process, and the D-peak is related to the melting of Cu-18 at%Li crystals. In fact, the temperature of D peak, 620 K, is in agreement with the solidus-solidus+liquidus transition temperature of the Cu-18 at%Li alloy reported in the literature⁽¹⁰⁾.

On the other hand, DSC thermograms do not show clear changes in their shape for the different kind of samples of the Table 1. This lack of resolution in the thermograms for the samples with different lithium content could be related to the existence of small zones in the sample around the inclusions where the precipitation process should be occurring. The heat flow signal related to the precipitation process is very small relative to the background signal due to the whole matrix of the cathode⁽²¹⁾. The above explanation is also in agreement with the small peak related to the melting of crystals.

The physical mechanism involved in the onset of precipitation processes could be the migration of lithium from crystals to copper matrix. In fact, the sample con-

Fig. 3 TGA and DSC behaviour for an electrodeposited sample of type (c).

Fig. 4 TEP evolution for a sample of type (c) for several ageing temperatures.

sists of two parts: Cu-18 at%Li and copper⁽¹⁵⁾, and therefore the lithium content of sample is not homogeneous. The lithium concentration gradient from the alloy crystals to the copper matrix could produce the lithium migration, giving rise to the thermodynamic configuration that allows the occurrence of the precipitation processes, as indicated by the DSC peaks. The above assumptions will be checked in the following paragraphs using the other experimental results.

In order to check the assumption of a precipitation process related to the A peak in the thermogram of Fig. 3, isothermal TEP measurements were carried out at different temperatures within the temperature range of this DSC peak, as shown in Fig. 4. The results have been plotted as ΔTEP vs. ageing time. We define $\Delta TEP = TEP_i - TEP(t)$, where TEP_i and $TEP(t)$ are the initial TEP value and the TEP at time t , respectively. TEP curves show the appearance of a peak in the plot ΔTEP

against time, in the positive side of the Δ TEP axis, which shifts towards shorter times on increasing the ageing temperature. As can be seen from the figure, in the sample aged at 323 K the peak in Δ TEP cannot be reached for ageing times less than 10^5 s.

It should be pointed out that a similar Δ TEP behaviour was already reported by Pelletier *et al.*⁽²²⁾ in Cu-Be alloys, when the formation and growth of Guinier Preston (GP) zones take place. Following the work of Pelletier *et al.*, the behaviour of the Δ TEP measurements against ageing time could be considered as the overlapping of two co-operative mechanisms: (i) an increase of Δ TEP related to depletion of Li atoms from Cu matrix and (ii) a decrease in Δ TEP related to the development of internal stresses induced by the growth of precipitates.

On increasing the ageing temperature, the Δ TEP peak shifts towards shorter times since the lithium diffusion is accelerated at higher temperatures. The precipitate growth also becomes faster, leading to larger Δ TEP decreases at shorter times.

However, a difference appears between Pelletier's results and our measurements. In the present measurements the peak height does not decrease on increasing the ageing temperature, as it does in Pelletier's results. This decrease in the TEP peak height was attributed to the solubility increase of Be with an increase in temperature limiting the Δ TEP variation related to the solid solution effect. Nevertheless, to relate the decrease in the Δ TEP peak height with an increase in the solubility of solute atoms is very difficult and must be viewed with considerable caution, owing to the overlapping of the two effects which contribute to the total measured Δ TEP value. In fact, the lack of change in the Δ TEP peak height, in our TEP measurements, could be simply the result of the mathematical compensation of the functions related to each effect. Therefore, the difference between the Pelletier's results and the current ones does not obstruct the analysis carried out in the present work.

In the ageing treatments carried out at temperatures higher than 338 K, a Δ TEP peak appears on the negative side of the Δ TEP axis at short times (around 60 s). This peak, on increasing ageing temperatures, shifts towards shorter times, decreasing in its height. This signal corresponds to a reversion process of the precipitates. After quenching in the electrodeposition process, small precipitates could nucleate and subsequently grow during the storage of the sample at room temperature^{(23)–(26)}. Therefore, during the ageing treatments carried out at temperatures above 338 K, some precipitates would dissolve to reduce their interface energy. Moreover, at higher ageing temperatures the driving force for reversion is larger, leading to the decrease and shift towards shorter times of the Δ TEP valley as shown in Fig. 4. Subsequently, during the high temperature ageing the precipitate mass fraction increases again with a precipitate morphology which should be different from that developed during the room temperature ageing^{(23)–(26)}.

Summarising our experimental TEP results shown in Fig. 4, the increase in Δ TEP behaviour could be related

to the lithium loss from solid solution, and the subsequent Δ TEP decrease could be related to the growth of precipitates. The shifting of the positive Δ TEP peak towards shorter ageing times is consistent with a larger diffusivity of lithium at higher temperatures, which accelerates the process.

The decrease in Δ TEP curves for samples aged at temperatures higher than 338 K could be related to the reversion of precipitates which grew during the room temperature ageing (storage), which increased the lithium content in the solid solution. Therefore, the TEP results clearly indicate that a precipitation process appears within the temperature range 325–450 K.

On the other hand, in the above discussion we proposed that the precipitation process related to each one of the DSC peak, could be located in the neighbouring zone to the Cu-Li crystals.

The lithium migration as a function of temperature from the Li-rich zones, *i.e.* from the crystals of Cu-18 at%Li towards the Cu matrix, was studied employing the X-ray diffraction, as shown in Fig. 5, in samples with the thermal treatment described in Section II.2. Figure 5 shows a zoom of the more intense diffraction peak of copper at approximately $2\theta = 43.35^\circ$ related to the (111) reflection. The other peak at around $2\theta = 43^\circ$, which has an intensity about 6% of the former peak of copper, is also the intense diffraction peak of the Cu-18 at%Li crystals related to the (111) reflection. As can be seen, these crystals have the same fcc structure with a slightly larger lattice parameter⁽¹⁵⁾.

The reduction in intensity of the diffraction peak related to the Cu-18 at%Li crystals for increasing temperatures indicates that the volume fraction of Cu-Li crystals has decreased. Therefore, taking into account the TGA results which report no lithium loss, it could be established that the lithium has migrated in a down-hill process towards the copper matrix giving rise to the precipitation processes. Moreover, as can be seen also from Fig. 5

Fig. 5 X-Ray diffraction patterns for a sample of type (c) measured after quenching at room temperature from different temperatures.

at temperatures closer to the transition temperature, the diffraction peak decreases strongly.

The effects of the precipitation process on the mechanical behaviour within the temperature range of the A peak in the DSC thermogram of Fig. 3 were also checked by means of modulus measurements.

The isothermal modulus evolution during the ageing time at different temperatures for samples of type (c), have been plotted in Fig. 6. As can be seen from the figure, an age-hardening appears clearly. As would be expected, the kinetics is faster for the ageing treatments performed at higher temperatures. Therefore, this hardening during the ageing treatment at temperatures within the range of the A-peak in the DSC curve could also indicate the occurrence of a precipitation process. The different degrees of strength obtained after the ageing treatment at different temperatures depend on both the contribution of the dislocations arrays and the solid solution hardening⁽²⁴⁾. The degree of strengthening at different temperatures is a complex process and will be discussed in future work.

The appearance of precipitation processes were also studied through ADD measurements in the whole temperature range of the DSC study. ADD measurements are well suited for studying interactions processes between dislocations and weak pinning points (WPP). The occurrence of ADD could be related to the appearance on the dislocation line of WPP owing to the precipitation process. Note that contrary to macroscopic flow stress measurements, in ADD measurements the dislocations are only forced to overcome the weak obstacles and not the strong ones. Thus, while the flow stress has contributions from solution hardening and strain hardening, ADD measures only the effects of the WPP⁽²⁷⁾⁽²⁸⁾. Although, a relationship between the macroscopic and microscopic yield stress can be determined under some conditions⁽²⁹⁾⁽³⁰⁾, in this work only micro-yield stress behaviour will be studied.

The critical strain corresponding to the beginning of ADD, which is related to the micro-yield stress, was called ε_0 ⁽³¹⁾⁽³²⁾. The ε_0 behaviour as a function of temperature for the samples of the Table 1 is shown in Fig. 7.

ε_0 frequently decreases with increase of temperature owing to the increase in the thermal agitation energy of the dislocation line^{(31)–(33)}. However, in the current work ε_0 practically does not decrease for samples (a) and (b) and even increases slowly for samples (c), within the temperature range of the DSC's A peak. This behaviour in ε_0 could be explained by the onset of a precipitation process. In fact, during the evolution of the precipitate distribution, new pinning points could appear on the dislocation line, which reduces the free dislocation length, producing an increase in the stress for the break-away of dislocations, even if the thermal agitation of the dislocation line has increased with increasing temperature.

Subsequently, within the temperature range of the assumed dissolution of precipitates (range of the DSC's B peak in Fig. 3), ε_0 decreases, as could be expected owing to the disappearance of the obstacles on the dislocation

Fig. 6 Elastic shear modulus behaviour during different ageing treatments at different temperatures for a sample of type (c).

Fig. 7 Micro-yield stress behaviours as a function of temperature for the samples of Table 1.

line. In fact, the dissolution of precipitates decreases the stress for the break-away of dislocations owing to the increase in the free dislocation length.

At temperatures higher than 500 K, even if ε_0 decreases with increase of temperature, the ε_0 decrease is slower for the samples with a larger lithium concentration. This behaviour could be related to a larger quantity of precipitates on the dislocation line, indicating that another precipitation process has appeared.

Summarising, the ADD results for the ε_0 behaviour versus temperature show the onset of an interaction process between dislocations and weak obstacles. This precipitation process appears at temperatures ranging between room temperature and 450 K. Furthermore, a dissolution range for the obstacles was detected between 450 and 500 K. Another interaction range between dislocations and obstacles at higher temperatures around 500 K was also

found. It should be pointed out that the temperatures where the dislocation interaction processes appear (precipitation) or disappear (dissolution) are in good agreement with the temperature intervals where the precipitation and dissolution was determined through DSC thermograms. Besides, the temperature ranges of precipitation and dissolution are close to those previously proposed in an early work⁽¹⁵⁾, as determined from the behaviour of the slope of damping against strain $\Delta D/\Delta \varepsilon_m$ as a function of temperature.

The whole of the experimental results of the different techniques employed in the current work strongly suggest precipitation processes in the Cu-Li system, in the concentration range close to 18 at%Li. The observed variation of the thermo-electric power was very similar to the behaviour due to the appearance of GP zones, but transmission electron microscopy and selected area diffraction studies have not been able to resolve the existence of GP zones in Cu-Li alloy.

In contrast, Pelton's equilibrium phase diagram of Cu-Li⁽¹⁰⁾ shows only one solid phase with up to 20 at% Li. Nevertheless, only eight experimental points describe the solidus line in the zone of lithium concentrations up to 20 at%Li, which corresponds to the temperature range between 573 and 1300 K. The scatter of these few points obstructs the accurate determination of solidus line and moreover, the determination of another phases in such plot. Therefore, as it is also pointed out by Pelton, further examination of the phase diagram should be made for a definitive assessment. This new revision of the phase diagram could allow to determine other phases which are related to the precipitation processes mentioned above. Furthermore, more effort should be made for observing GP zones in Cu-Li alloys directly.

IV. Conclusions

The onset of precipitation processes in copper-lithium alloys produced by electrodeposition at lithium concentrations close to 18 at% was observed. The first precipitation process appears at low temperatures in the range 325–425 K. The subsequent reversion of these precipitates was found to occur at temperatures between 425 and 525 K. Furthermore, another precipitation process at higher temperatures around 525 K was also observed.

The experimental results obtained from thermo-electric power and dynamical techniques suggest that the precipitation process at lower temperatures should be the formation of GP zones. Unfortunately, GP zones could not be resolved by transmission electron microscopy or selected area diffraction.

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