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Influence of Mg, Ag and Zn minor solute additions on the precipitation kinetics and strengthening of an Al-Cu-Li alloy

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ABSTRACT

The influence of minor solute additions of Mg, Ag and Zn and their combinations on the precipitation kinetics and strengthening of an Al-Cu-Li alloy has been systematically investigated. Combining differential scanning calorimetry (DSC) and small-angle X-ray scattering (SAXS) provides information on how alloy chemistry changes the precipitation sequence, the precipitation kinetics in non − isothermal and in isothermal conditions, and the related strengthening. When adding Mg, the precipitation kinetics are strongly accelerated, which is shown to be related to the dominant formation of T_1 precipitates. When adding Ag and Zn together with Mg, further enhancement of precipitation kinetics is observed, without change of the precipitation sequence, probably due to a more efficient nucleation of T_1. The intrinsic strengthening potential of the T_1 phase is shown to be unchanged by the addition of Ag and Zn, and the higher strength reached in the alloys containing these additions is shown to be due to a higher precipitate volume fraction.

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1. Introduction

Al-Cu-Li based alloys are currently the subject of intensive research for structural applications requiring a combination of high strength, low density, high fracture toughness and good corrosion resistance, these requirements being particularly relevant for aerospace applications [1–4]. Following the first generations of Al-Li alloys developed in the 1980s, the current developments concern alloys with lower Li content of the order of 1 wt%, which has helped improve some of the weak points of earlier developed alloys, such as limited long term microstructural stability [5–8].

The mechanical properties of Al-Cu-Li alloys are controlled by the fine scale distribution of precipitate phases. In the base Al-Cu-Li system, the main phases that can form in practice during conventional heat treatments belong to the two binary systems Al-Cu (GPI and GPII zones and θ' phase, towards Al_2Cu composition [9–12]), Al-Li (δ' phases – Al_3Li [12,13]), and to the ternary Al-Cu-Li system (T_1 phase Al_2CuLi [14–16]); other phases may form along higher temperature paths (θ, δ, T_2, σ, …). However, most Al-Cu-Li alloys have a more complex chemistry. Apart from solutes involved in dispersoids, which do not diffuse appreciably during precipitation heat treatments (Zr, Mn, …), combinations of Mg, Ag and/or Zn in fractions of % are frequently added, resulting in a potentially much wider variety of precipitating phases. Particularly, the importance of Mg-Cu phases (S phase and all its precursors such as clusters and GPB zones) has been clearly established [17–19].

The nucleation of the T_1 phase, usually considered to provide the highest strength in these alloys, forming thin plates of hexagonal structures lying on {111} matrix planes [20,21] is quite challenging, especially at the relatively low ageing temperatures required to retain a good compromise of mechanical properties. It can be promoted by the introduction of strain prior to the artificial ageing treatment [9,22]. Numerous studies have also shown that the precipitation of the T_1 phase could also be promoted by the addition of minor solute species, particularly Mg and Ag [13,17,23–27].

Ag addition alone does not alter the hardening potential to a great extent [23,25,27,28], but the effect of Mg addition is much more pronounced [23,26,28,29]. Mg addition promotes T_1 precipitation, to the expense of θ'' and θ' [19,26,28,30], and the resulting distribution of T_1 phase (in the absence of pre-strain) has been shown to be finer [23]. In the presence of Mg, the addition of Ag has been shown to further improve the strengthening of Al-Cu-Li alloys

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[23,28]. In parallel, Zn has been also used as a minor addition to Al-Cu-Li alloys, in combination with Mg [9,31,32].

In a recent article, we have studied the respective role of Mg, Ag and Zn additions on the precipitation sequence during different steps of artificial ageing at 155 °C following a pre-deformation of an Al-3.5 wt%Cu-0.9 wt%Li alloy [19], using atomic resolution high angle annular dark field scanning transmission electron microscopy (HAADF-STEM). The main conclusions drawn from this study were as follows:

- The presence or absence of Mg profoundly changes the precipitation path. In the absence of Mg, at early ageing times the microstructure contained GPI zones that later developed into θ' precipitates, curved along the dislocations where they nucleated. Only a limited number of T_1 precipitates were observed. In the presence of Mg, however, precursor Mg-Cu phases were found at dislocations very early along the heat treatment that further developed in GPB zones and S phase along with a high density of T_1 precipitates that dominated the microstructure. A threshold Mg concentration of the order of 0.1 at% was found necessary to activate this change of precipitation path [30].
- Ag was found at the interface between T_1 precipitates and the matrix as early during the heat treatment as the T_1 precipitates were observed, confirming the observations of [18]. However, its presence did not change qualitatively the precipitate microstructure after artificial ageing. Similarly, Zn was found to be present inside the precipitates, most probably substituting for Cu atoms. Like that of Ag, the addition of Zn did not change qualitatively the precipitate microstructure.

Thus, a better level of understanding of the precipitation sequence has been reached with respect to the effect of minor solute additions of Mg, Ag and Zn. However, little is known about the influence of these profound changes of precipitation sequence with minor alloying additions, on the precipitation kinetics and on the associated evolution of strength. Moreover, Mg, Ag and Zn may be used together or in various combinations, and the role of the combination of these has not yet been established. In Al-Cu-Li based alloys, maximizing the intragranular precipitation kinetics is of paramount importance because at long ageing times, grain boundary precipitation contributes to a reduction in ductility and toughness [33]. As a consequence, the T8 condition of these alloys is generally chosen in a slightly underaged state at the beginning of the hardness plateau where a good compromise between intra-granular strength and minimal grain boundary precipitation can be achieved.

In this context, this paper will present a thorough evaluation of precipitation kinetics, in both isothermal and non-isothermal conditions, as well as the related strengthening kinetics, in a series of alloys with systematic combinations of minor solute additions (Mg, Ag, Zn). The precipitation kinetics will be investigated by combining small-angle X-ray scattering (SAXS) and differential scanning calorimetry (DSC), supported by transmission electron microscopy (TEM).

2. Materials and methods

The alloys of the study all share the same concentration of 3.5% Cu and 0.9%Li (all compositions are in wt%) as major alloying elements. Based on this solute content (named subsequently the base alloy), a number of alloys containing different combination of Mg, Ag and Zn in small proportions were prepared. The alloy compositions are given in Table 1. The heat treatment consisted of a solution heat treatment at 505 °C for 30 min, followed by a water quench. A plastic pre-deformation of 4% was added after water quench followed by 3 days of natural aging. Artificial aging started with a ramp heating from room temperature (RT) up to 155 °C with 20 °C/hour and isothermal aging was then performed at 155 °C in an oil bath.

Table 1
Alloy compositions (all in wt%).

Alloys	Cu	Li	Mg	Ag	Zn
Base	3.5	0.9			
Base +0.3Ag	3.5	0.9		0.35	
Base + Mg	3.5	0.9	0.35		
Base + Mg0.1Ag	3.5	0.9	0.35	0.11	
Base + Mg0.3Ag	3.5	0.9	0.35	0.35	
Base + MgZn	3.5	0.9	0.35		0.6
Base + Mg0.1AgZn	3.5	0.9	0.35	0.11	0.6

For hardness measurements, the samples were water quenched from 155 °C for different aging times and tested on a fully automatic Buehler TukonTM1102 instrument. The applied load was 500gr and indentation time 10 s. For each ageing time and alloy, the average value is calculated on 60 measurements.

Small-Angle X-ray Scattering (SAXS) measurements were performed both on a laboratory rotating anode (Rigaku MicroMax-007 HF, Cu K_α source) and at the European Synchrotron Radiation Facility (ESRF). For aluminium alloys the optimal sample thickness at the energy of the Cu Kα the energy (8.048 keV) is around 75 μm. The data were acquired by a DECTRIS Pilatus 300K X-ray Detector, which performs single-photon counting on each pixel. The same energy was provided at the ESRF. Due to a higher flux, it is possible to measure with shorter acquisition times compared to the laboratory setup. This was taken advantage of for the continuous heating experiments. A CCD camera was used for detection in this case. The precipitates present in this system are strongly anisotropic, which results in a signal on the detector which is the combination of streaks representative of the precipitates lying close to an edge-on geometry. However, the particular grain texture present in our alloys did some averaging of this intensity, which prevented the analysis of individual streaks such as performed in Refs. [34,35] to extract the morphology of the precipitates. Instead, a radial averaging of the data was made, which is equivalent to making the hypothesis that grains of all orientations are within the illuminated volume. This hypothesis was sufficiently robust to extract quantitatively morphological parameters such as the precipitate thickness (see Appendix), but the integrated intensity, which is usually related to the precipitate volume fraction in a rather simple way [36], is very sensitive to the detailed texture of the zone covered by the X-ray beam. As a consequence, although the relative variation of intensity could be safely interpreted during an in-situ heating experiment because the local texture does not change, converting this relative intensity to an absolute volume fraction measurement required the use of an independent measurement, which was made using Differential Scanning Calorimetry.

DSC measurements were performed with a Perkin Elmer Pyris Diamond apparatus. DSC samples had a weight between 30 and 50 mg. The ramp heating was either 50 °C/min or 200 °C/min. The scanned temperature interval goes from 25 to 550 °C. Baseline corrections were done, by adjusting temperature intervals between 50 and 100 °C and above 500 °C to zero heat flow by using a polynomial function of 3rd order. These temperature intervals were chosen, because precipitation is expected to occur above this lower temperature boundary and all present phases should have been dissolved above 500 °C [37].

Conventional TEM was performed at the SIMaP laboratory on a JEOL 3010 instrument, operated at 300 kV. With the aperture diameter available on this instrument, the $\langle 112 \rangle_{Al}$ orientation was favourable to form good dark field images (with the aperture centred around the $\{110\}_{Al}$ forbidden reflexion) given the larger

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