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#### COPRECIPITATION HARDENING IN Al-Li-Cu-Mg ALLOYS

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#### ABSTRACT

The structural features of the matrix precipitates families governing the strength of AI-Li-Cu-Mg(-Zr) alloys within a wide composition range are discussed. The specific effects of alloying and heat treating on matrix coprecipitation of  $\delta$ ', GPB zones, S', S, T' or  $\theta$ ', T1, and T2 and the dependence of the solid solution decomposition sequence on cold working between quenching and ageing are illustrated by transmission electron microscopy results. Conventional artificial ageing treatments and selective reversion heat treatments, performed on both laboratory and commercial alloys, allow to evaluate the intrinsic contributions of matrix precipitates on complex coprecipitation hardening in industrial alloys.

#### INTRODUCTION

The need for high strength and damage tolerant low density aluminium-lithium alloys for the aerospace industry has raised a considerable research activity devoted to processing and heat treating optimization of a number of Al-Li-Cu-(Mg)-Zr alloys since the 80's [1-4]. In the meantime, many basic studies [1-5], and the recent review paper by Flower and Gregson [6], have focussed on the identification of matrix and grain boundary precipitates and on the understanding of structural hardening mechanisms on experimental and commercial alloys leading to the establishment of typical coprecipitation sequences. A number of new designations, some of them still controversial, were also proposed to account for new metastable precipitates.

The aim of this paper is to highlight the influence of alloying, cold working and artificial ageing on nucleation, coprecipitation sequences and structural hardening effects in Al-Li-Cu-(-Mg)(-Zr) alloys belonging to a wide composition range (including commercial alloys). Following the identification of the structural features of ternary or quaternary phases (T' or  $\theta$ ', T1, S' or S, T2) coprecipitating with  $\delta$ '-Al3Li, the specific strengthening effects of each family of matrix precipitates will be discussed or revlewed in order to bring a qualitative understanding of coprecipitation hardening in commercial alloys heat treated to the industrial tempers.

#### **EXPERIMENTAL**

The composition ranges of the commercial or industrial alloys  $(1.7wt\% \le Li \le 2.6wt\%)$  used for Transmission Electron Microscopy (TEM) and Selected Area Electron Diffraction (SAED) studies are given on Fig.1. An extensive TEM analysis of matrix precipitation was also performed on a series of experimental AI-Li-Cu-(Mg) alloys  $(2.0wt\% \le Li \le 3.3wt\%; Cu \le 3.5wt\%;$ Mg  $\le 3.5wt\%$ ) that were simply cast into small ingots (18 mm in diameter), homogenized, cold water quenched and artificially aged for 24 h at 190°C to the T6 temper. In addition, high temperature reversion heat treatments followed by rapid cooling, allowing  $\delta'$  dissolution and selective precipitation of only one type of ternary phase were performed on a number of alloys. At last, comparative Vickers hardness measurements were carried out on selected cast alloys as well as on sheets of alloys 2091 and of a Li free AI-Cu-Mg alloy (both of them with similar Cu and Mg contents) that were cold worked by 2 % prior to ageing treatments.

#### EFFECT OF ALLOYING ON COPRECIPITATION IN T6 TEMPER

The results of matrix precipitates identification on laboratory cast samples heat treated to peak hardness (24 h 190°C) without prior stretching are given in Fig.1. The latter shows the extent of phase fields for metastable precipitates in Al-Li-Cu-Mg alloys (1.7wt% $\leq$ Li $\leq$ 3.3wt %).  $\delta$ '-Al3Li is found to coprecipitate in all alloys with either one or several types of the phases designated T'-(Al,Cu,Li) or  $\theta$ '-Al2Cu, T1-Al2CuLi and S' or S-Al2CuMg.



FIG.1. Matrix precipitates domains in Al-Li-Cu-Mg-(Zr) alloys aged 24h at  $190^{\circ}$ C without preliminary stretching (from results on experimental (  $\bullet$  ) or industrial alloys in T6 temper)

Evidence of homogeneous and coarse matrix precipitation of the T2-Al6Cu(Li,Mg)3 phase is found in a number of alloys with intermediate Mg content, as sketched on Fig.1. Outside this field, the T2 phase precipitates more discretly as coarse rods with very low volume fraction in quaternary alloys belonging to a wider composition range (Cu≥1wt%, Mg≥0.4wt%) after quenching and ageing 24h 190°C.

#### PRECIPITATION FEATURES

Fig.2 gives typical TEM precipitate micrographs and SAED patterns with a <100> matrix zone axis.

<u>T' phase</u> : A homogeneous precipitation of T' (Fig.2.a) is obtained simultaneously with  $\delta'$  in a Al-2.6%Li-3.1%Cu alloy artificially aged 24 h at 150°C. This phase precipitates in alloys with high Cu/Li ratios. Its designation (T', T'B or  $\theta'$ ) is still under controversy since it could not be discriminated from  $\theta'$  except by the electron diffraction intensities [7]. In the underaged alloy CP276, moderate magnesium addition induces a refinement and a stabilization of T' [4,5]. This effect has already been observed on  $\theta'$  in Al-Cu-Mg alloys [8] in which the Mg content tends to refine the precipitates in the matrix and to accelerate their formation.

<u>T1 phase</u>: In alloys with copper content below 3%, overageing or stretching before ageing favours the nucleation and growth of T1 which otherwise has a very large domain of precipitation (Fig.1). Silcock [9], Noble and Thompson [10] and more recently other authors [11,12] have analyzed the nucleation-growth and the structural features of this phase precipitating in Al-Li-Cu-(Zr) solid solutions with high copper content. Fig.2.b shows this precipitation in a 2090 alloy heat treated 30 min. at 265°C and water cooled.

<u>S and S' phases</u>: The 2091 alloy, stretched 2% and reverted for 1 hour at 230°C, exhibits a dense precipitation of S' and S (Fig.2.c). The nature of these precipitates has been extensively studied in previous studies of AI-Cu-Mg alloys. Different precipitation sequences have been proposed including the successive precipitations of GPB zones, S", S' and S. Whereas the S" formation has been debated [13], the S' precipitate is now assumed to have approximately the same structure as the S phase [14] and to be simply in a slightly strained state induced by the surrounding matrix. In alloy 2091 and, to a lesser extent, in alloy 8090 aged to peak strength, the S and S' phases are found to precipitate along the <100> matrix directions, either homogeneously as fine rods or heterogeneously on dislocations as coarse laths with (210) matrix habit planes. As a consequence, the relative proportion of these phases is strongly modified by stretching before





Fig 2a-d : Typical TEM micrographs and SAED patterns of (100) zone axis for precipitate types in Al-Li-Cu-Mg (Zr) alloys.

a) Alloy Al-2,6 % Li-3,1 % Cu- Underaged (24 h 150°C)  $\Rightarrow \delta' + T'$ b) Alloy 2090 = reverted (T 4 + 30 min 265°C)  $\Rightarrow$  T1 c) Alloy 2091 = reverted (T4 + 1 h 230°C)  $\Rightarrow$  S'+ S (+ Al<sub>3</sub>Zr) d) Alloy Al-2,1 % Li-2,1 % Cu-1,0 % Mg-Aged 4 h 30 250°C  $\Rightarrow$  T2

Fig 2e-g : SAED patterns typical of quaternary coprecipitation of  $\delta'$ , T1, S (or S') and T2 in an overaged Al-Cu-Li-Mg alloy. zone axis (100) (e); (211) (f) (111) (g) C3-410

ageing (figures 5.a and 5.b). In the present study, rod shaped precipitates are considered to consist of S' phase close to the GPB zones, whereas laths are close to a more defined S structure. The coarsening of the latter induces GPB or S' free zones formation. Continuous rod free zones are also observed along the grain boundaries.

<u>T2 phase</u> : The T2-Al6Cu(Li,Mg)3 compound has been reported to precipitate along grain boundaries in several alloys in peak-ageing and over ageing tempers [4,5] and has raised a considerable interest since the discovery of its "quasi-crystalline" structure [15]. According to Fig.1, the homogeneous nucleation and coarsening of T2 is also observed in a domain extending from the composition range of the alloy CP276 [16] to that of the alloy 2091 in T6 temper.

In an alloy Al-2.1%Li-2.1%Cu-1%Mg reverted for 1h30 at 250°C (Fig.2.d) the T2 phase precipitates alone with high volume fraction. In that case, its pencil-like morphology exhibits a preferential growth axis (two-fold symmetry axis of the icosahedron structure) lying along the [110] matrix directions [17]. On matrix SAED patterns of [111] zone axis, three two-fold symmetry patterns of T2 are superimposed with a rotation of 60°. A five-fold axis and therefore the (100000) T2 intense diffracted spots are nearly parallel to a [112] matrix direction with a misorientation of nearly 1.7°. Double diffraction leads to complex triangular patterns typical of the T2 precipitation (Fig. 2.g). In this study the homogeneous nucleation of T2 was found to be inhibited to the benefit of S nucleation by stretching before ageing to the T651 or T8 tempers.

#### SPECIFIC HARDENING CONTRIBUTIONS of PRECIPITATES

The T1 precipitates contribution in strengthening has been analyzed in different Al-Li-Cu-(Zr) alloys [9,10,18] and has been reported to be 2 to 10 times greater than that of  $\delta'$  [18]. The lack of similar work on T' (due to the unavoidable  $\delta'/T'$  interactions) does not allow to point out its specific contribution on tensile properties. The latter is nevertheless expected to be similar to that of the  $\theta'$  phase in Al-Cu or Al-Cu-Mg alloys. In those alloys,  $\theta$  precipitation homogenizes multiple slidings and leads to a strong work hardening in the early deformation states [19]. Besides coherency stress hardening, an Orowan mechanism has been set up to quantitatively explain the experimental strengthening observed after ageing treatments in Li-free alloys [20].

The S' contribution on hardness has been analyzed by Silcock on Al-Cu-Mg alloys [21]. From this work, GPB zones are demonstrated to have no major effect on the age-hardening. On the other hand, the increase in the yield strength is correlated with the (GPB $\rightarrow$ S') phase transformation. This effect is weakly observable on the 8090 alloy [22], but it is particulary effective in alloy 2091 which exhibits a more dense and homogeneous S' precipitation in peak aged temper (Fig. 5.a and 5.b). A conclusive comparison between the hardness of alloy 2091 and of an Al-Cu-Mg alloy with similar Cu and Mg contents is shown in Fig.3. The strong increase in hardness corresponds effectively to the beginning of the (GPB $\rightarrow$ S') phase transformation in alloy 2091. As for alloy 8090, the maximum hardness of alloy 2091 is achieved when the (GPB $\rightarrow$ S') phase transformation is completed. In that case, both  $\delta'$  and S' (or S) contribute to the strengthening at a similar level. As shown in Fig.3, the difference of hardening between the Li-free and the 2091 alloys arises qualitatively from extensive  $\delta'$  precipitation.

The intrinsic strengthening effect of T2 is very low as shown in Fig.4 on a 2.1%Li-2.1%Cu-1%Mg alloy heat treated at 250°C following  $\delta$ ' and S' dissolution. The poor increase in hardness observed in that case is likely to result from the rather coarse mean size of the homogeneous dispersion of T2. Therefore this phase behaves as the Al2LiMg phase, which causes no hardening increment in overaged Al-Li-Mg alloys. It has to be reminded that such ageing heat treatments also lead to coarse grain boundary precipitation of T2.

#### COPRECIPITATION AND HARDENING IN COMMERCIAL ALLOYS

Keeping in mind the complex precipitation phenomena involved in Al-Li-Cu-Mg alloys and the prominent effects of  $\delta$ ', T', T1 and S' (or S) precipitates on hardening, the major coprecipitation strengthening contributions can be identified in commercial aluminium-lithium alloys.

In underaged tempers, the main contributors to coprecipitation hardening are  $\delta$ ' and T' in

high strength alloys with high Cu and nil or low Mg (e.g. 2090, CP276 - Fig.5.c) whereas  $\delta'$  precipitation and solid solution hardening govern matrix strengthening in damage tolerant alloys with higher Mg content (2091, 8090). In alloys aged to peak strength, the coprecipitation of  $\delta'$  and T1 is mainly responsible for the maximum strength achievements in Al-Li-Cu-Mg (-Zr) alloys with high Cu content (e.g. 2090, CP276 - Fig.5.d) whereas coprecipitation strengthening mainly by S' (and/or S) and  $\delta'$  occurs in alloys with higher Mg contents (e.g. 2091, 8090 - Fig.5.a, 5.b).

Cold working before ageing favours a dense heterogeneous precipitation of T1 and S to the detriment of respectively T' and S' in peak aged (T651, T8) and over-aged tempers (Fig 5.a-d). However, dense homogeneous coprecipitation of either T' (alloy CP276) or S' (alloy 2091) with  $\delta'$ 



FIG.3. Hardness evolution of sheets of alloys AI-2.3%Cu-1.3Mg and 2091 after artificial ageing at 135°C, 150°C and 190°C (both alloys with similar Cu and Mg content and initial T351 condition).



ageing time at 250°C

FIG.4. Extensive matrix precipitation and low hardening contribution of T2 in alloy Al-2.1%Li-2.1%Cu-1.0%Mg aged at 250°C after quenching.



- Fig 5 : TEM microcraphs at low (left) and high (right) magnification and SAED patterns of (100) zone axis. Showing the effects of stretching (2 %) between quenching and ageing on coprecipitation in commercial aluminium-lithium alloys.
  - a)  $2091 T6 = \delta' + S' (+S, T2 rare)$ b)  $2091 - T651 = \delta' + S (+S')$ c)  $CP276 - T6 = \delta' + T' + T1 (+ S')$ d)  $CP276 - T651 = \delta' + T1 (+ S)$

contributes to the achievement of high strength for these alloys in slighly underaged or peak aged condition even in the absence of prior cold work. The solid solution decomposition may therefore be summarized as follows :

$\alpha \rightarrow$	α <sub>1</sub> +δ'+GPB	$\rightarrow \alpha_2 + \delta'(+S',S)$	$\rightarrow \alpha_3 + \delta' + S', S(+T2)$	$\rightarrow \alpha_4$ +S+T2	(2091)
$\alpha \rightarrow$	α <sub>1</sub> +δ'(+GP?)	$\rightarrow \alpha_2 + \delta' + T'$	$\rightarrow \alpha_3 + \delta' + T1(+S+T2)$	$\rightarrow \alpha_4$ +T1	(CP276)

The additional matrix precipitation of T2 which can be observed in alloys with intermediate Mg content ( $0.5 \le Mg \le 1.5 wt\%$ ) heat treated to the T6 temper or overaged exerts no positive effect on matrix hardening.

#### CONCLUSION

Depending on alloy compositions and ageing conditions, most of the intermetallic phases in stable equilibrum at high temperature with the  $\alpha$ -AI matrix in AI-Cu-Li and AI-Cu-Mg systems are found to coprecipitate with  $\delta'$  in the matrix of AI-Li-Cu-Mg(-Zr) alloys, either as compounds of same structure (T1, T2, S) or as precursors (T' or  $\theta'$ , S').

A better understanding of matrix coprecipitation hardening in Al-Li-Cu-Mg(-Zr) alloys is expected to result from this work and the considerable literature data already published on this topic. However, a comprehensive study of the mechanical behaviours of aluminium-lithium alloys (fracture, ductility), should also take into account the effects of preprecipitation hardening (e.g. in underaged alloys) and intergranular precipitation in industrial semi-products.

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