



# The effect of Mn and Zr dispersoid-forming additions on recrystallization resistance in Al–Cu–Li AA2198 sheet

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## Abstract

The influence of sole and joint Zr and Mn additions on the recrystallization behaviour of an AA2198-base alloy has been compared during extended annealing at 535 °C, following hot rolling to sheet. With a constant Zr level recrystallization resistance was seen to diminish with the addition of Mn and became progressively worse with a decrease in Zr content, as more Mn was added. It has been found that this behaviour arises because the additional pinning pressure of Al<sub>20</sub>Cu<sub>2</sub>Mn<sub>3</sub> dispersoids does not adequately compensate for the expansion in width of the Al<sub>3</sub>Zr-free bands that occurs on the addition of Mn to the base alloy, even with the same Zr level. The lower potency of Mn, relative to Zr, in inhibiting recrystallization has been attributed to the poorer coherency and higher aspect ratio of the Al<sub>20</sub>Cu<sub>2</sub>Mn<sub>3</sub> dispersoids, which reduces their Zener pinning pressure by a factor of four relative to that of the Al<sub>3</sub>Zr phase. In addition, the presence of the coarser Al<sub>20</sub>Cu<sub>2</sub>Mn<sub>3</sub> dispersoids was found to increase the stored energy after hot rolling. The recrystallization mechanism was dependent on the dispersoid type. The addition of Zr led to the dominance of broad front strain-induced boundary migration (SIBM), whereas the addition of Mn-containing dispersoids favoured particle-stimulated nucleation (PSN). Texture measurements have verified this observation, with SIBM favouring the growth of recrystallized grains of orientations typical of the deformation texture and PSN promoting the growth of randomly orientated grains during recrystallization.

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## 1. Introduction

The demand for better damage tolerance in advanced Al–Cu–Li alloys has revived interest in obtaining a more complete understanding of the role that dispersoid-forming alloy additions perform in these alloys [1]. This enhanced interest stems from the fact that dispersoids play an important role in preventing recrystallization during solution treatment and because partially recrystallized microstructures exhibit inferior fracture toughness, compared to fully fibrous grain structures [2–9]. The joint addition of Zr and Mn dispersoid-forming elements has been introduced

relatively recently to plate products, such as AA2050 [10–12]. The rationale behind the combined addition of Mn and Zr is based on the opposite microsegregation patterns these elements form during casting [13,14]. In theory, this should increase recrystallization resistance by leading to a greater uniformity of dispersoid coverage [15–19], consequently improving fracture toughness [20–22]. However, in recent work on AA2198 alloy sheet by the present authors [14,23,24], a subtle interaction was observed between Zr and Mn when the latter element was added to the baseline alloy, which resulted in a reduction in recrystallization resistance.

It is well known that in commercial Al–Cu–Li alloys the addition of Zr leads to the precipitation of the metastable β′-Al<sub>3</sub>Zr phase during homogenization treatments [25].

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This phase typically forms as fine spherical dispersoids that are coherent with the Al matrix [26]. In comparison, Mn forms larger, semicoherent, lath-shaped  $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$  dispersoids [27]. The size, number density, distribution and morphology of these dispersoids depends strongly on the homogenization treatment and local supersaturation in the cast microstructure [28,29]. Because Zr and Mn have opposing microsegregation patterns in aluminium (arising from partition coefficients of  $>1$  and  $<1$ , respectively [30,31]),  $\beta'$  is typically found to precipitate within the dendrite cores and the  $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$  dispersoids are concentrated towards the dendrite/grain boundaries. These two families of dispersoids are consequently found to have a banded distribution (in the normal direction (ND)) in rolled products, with layers between the bands containing a low dispersoid density of the two respective dispersoid types [23].

In a recent previous study [23] it was found that addition of Mn to an AA2198 alloy containing 0.11 wt.% Zr resulted in the average number density of the  $\text{Al}_3\text{Zr}$  dispersoids being reduced and their size slightly increased. However, a more important influence was found on the width of the low  $\text{Al}_3\text{Zr}$  particle density bands seen in sheet material. This behaviour was shown to be caused by a small amount of Zr being dissolved into the Mn-bearing phases, which had a disproportionate effect on  $\text{Al}_3\text{Zr}$  precipitation in the interdendritic regions in the cast billet, where there was already a low Zr supersaturation. However, in this work the dominant recrystallization mechanisms operating in the materials and the effect of the two different dispersoid types on their texture development were not investigated.

The presence of two dispersoid families that form alternating bands in the ND in a rolled sheet is likely to interact in a complex manner with any recrystallization front that forms on annealing. In hot-rolled aerospace alloys the main mechanisms of recrystallization are generally found to be particle-stimulated nucleation (PSN), when new grains grow from the deformation zones around coarse constituent particles located at prior grain boundaries that are associated with a low dispersoid density [32–39]; and strain-induced boundary migration (SIBM), which takes place by the migration of a pre-existing high-angle grain boundary (HAGB) into a region of higher stored energy [32–34,40–44]. Both of these mechanisms are strongly dependent on the presence of pinning particles. The effect of pinning particles on PSN is to increase the critical size of the nucleus [32] and on SIBM to retard grain boundary mobility, which can favour broad front SIBM [45,46]. The recrystallization mechanism can also affect the recrystallization texture, through influencing the growth or consumption of specific texture components [32,33,45,47–54].

Although the effect of dispersoid particles on the recrystallization resistance of aluminium alloys has been extensively investigated [32,47–49,55–57], currently no work in the literature has investigated in detail the mechanisms of recrystallization operating when dual  $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$  and

$\text{Al}_3\text{Zr}$  dispersoid phases are present in commercial Al–Cu–Li alloys. The scope of the present work was to address this issue by systematically comparing the recrystallization behaviour of an AA2198 base alloy, containing individual and joint  $\text{Al}_3\text{Zr}$  and  $\text{Al}_{20}\text{Cu}_2\text{Mn}_3$  dispersoid phases. The effect of the dispersoid additions on the texture evolution is also discussed.

## 2. Experimental

The materials used in this study were supplied by Constellium, Centre de Recherches de Voreppe, France, in the form of 6 mm thick sheet, with two different temper conditions; hot-rolled (F temper), and solution-treated, stretched and naturally aged (T351 temper). The production route involved direct chill (DC) casting, homogenization, and hot rolling down to the final gauge, for which the total true strain was  $\sim 4$ . The experimental alloys were based on the composition of AA2198, and were produced with both individual and joint combinations of the dispersoid-forming elements Zr and Mn. The alloy compositions and designations are presented in Table 1. Although the exact composition has not been given for the Cu, Li, Mg, and Ag levels, great care was taken in ensuring tight composition control, with variability between castings kept to  $<3\%$  for each element. However, the Zr and Mn levels provided in Table 1 are actual values measured from chemical analysis. The experimental alloys underwent a standard single-temperature homogenization treatment. Unfortunately, at the time of the research the 2198–0.1Zr alloy was not available in the F temper with the same homogenization treatment and an otherwise identical sample that had been subjected to a two-stage homogenization was consequently included in the study. This alloy has been labelled 2198–0.1Zr-D, so that it can be distinguished from the alloy with the standard treatment.

Isothermal annealing treatments were carried out on the samples, in both the F and T351 tempers, at the solution treatment temperature of 535 °C followed by quenching in water. Microstructural analysis was carried out by means of scanning electron microscopy plus electron backscatter diffraction (SEM/EBSD) and transmission electron microscopy (TEM) on samples cut from rolling direction (RD)–ND cross-sections after different annealing times. An FEI Sirion FEG-SEM with an EBSD system was used to characterize the grain structures, textures and the volume fraction of recrystallization on samples that were metallographically prepared followed by light electropolishing. Several hundred grains were measured in each condition. An FEI Tecnai FEG-TEM operating at 300 kV was used to analyze the microstructures in more detail and measure the dispersoid and subgrain sizes more accurately. Subgrain sizes were measured by the linear intercept method in accordance with ASTM E 562-02 [58]. To quantify the dispersoid number densities and volume fractions, the thickness of the TEM foils was measured with the aid of electron energy loss spectroscopy (EELS)

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