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## GRAIN BOUNDARY PRECIPITATION OF THE I-PHASE IN AN Al-Li-Cu-Mg-(Zr) ALLOY (8090)

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### Abstract

Grain boundary precipitation of the I-phase having icosahedral symmetry has a detrimental effect on the mechanical properties of 8090. Transmission Electron Microscopy (TEM) was used to study I-phase formation in three alloys of 8090, differing only in their zirconium content. Specimens were examined at different stages in the production route of 8090 plate and sheet to ascertain the effects of thermomechanical processing (TMP) and alloy composition on the formation of the I-phase. It will be shown that I-phase forms during the early stages of TMP and is not removed completely by a standard solution heat treatment practice; furthermore I-phase formation is influenced by both the bulk and "local" Zr content of the alloy.

### Introduction

Grain boundary precipitation of I-phase in 8090 has a detrimental effect on mechanical properties most notably in thick product forms e.g., plate and forgings (1). The I-phase exhibits icosahedral crystallographic symmetry and has been a subject of intensive experimental (2-5) and theoretical (5) research activity. In Al-Li-Cu alloys with stoichiometric compositions close to that of the I-phase it may form during solidification, but in alloys of commercial interest its presence is attributed to formation during thermomechanical processing, and is influenced by local variation in chemical composition (6). The formation of I-phase in 8090 during extended annealing at 300-350°C has been studied (7), but its structure and composition (proposed as  $Al_6Cu(Li,Mg)_3$ ) remain ambiguous (3). There is evidence that it forms during quenching from the solution heat treatment temperature in thick product forms, but its formation can be suppressed by high quench rates in alloy sheet forms (1). Other workers have observed these particles to nucleate and grow during isothermal heat treatment in the range 170-520°C (8) and a recent study (9) showed a duplex distribution of I-phase particles, the coarse particles being formed prior to ageing, with fine particles being formed during a range of ageing treatments. The present work sets out to ascertain the factors controlling the formation of coarse grain boundary I-phase in 8090, these particles being considered to influence significantly the mechanical properties of this alloy, toughness in particular.

### Experimental

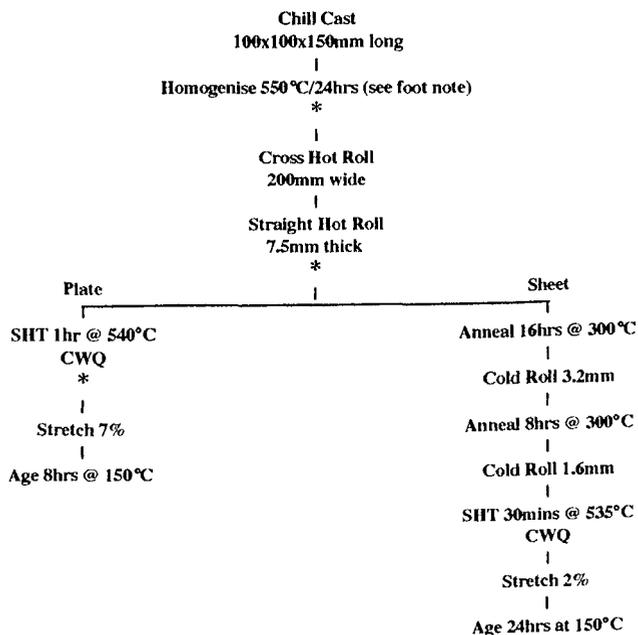
Three alloy variants of the 8090 alloy having different Zr content were studied, the compositions being given in Table 1.

Table 1. Chemical composition (%wt) of alloys.

alloy	Al	Li	Cu	Mg	Zr	Mn	Ti	Fe	Si	B
LV346	bal.	2.45	1.20	0.82	<b>0.00</b>	0.003	0.001	0.05	0.04	<0.001
LV326	bal.	2.45	1.20	0.82	<b>0.05</b>	0.003	0.001	0.05	0.04	<0.001
LV329	bal.	2.45	1.19	0.84	<b>0.10</b>	0.003	0.018	0.05	0.04	<0.001

The alloys were chill cast and thermomechanically processed at Alcan International Limited: Banbury Laboratories according to the schedule summarised in Figure 1. Specimens for TEM examination were taken at various steps in the processing route (annotated by \* in Figure 1). Sections for grain structure evolution were polished and anodised in Barkers solution before being viewed under polarised light.

Specimens for investigation by Transmission Electron Microscope were ground to 300µm prior to electropolishing in 30% Nital at -20°C. Thin foils were examined in a JEOL 2000 FX TEM operating at 200kV. TEM investigation of grain boundary precipitation required observation of a consistent number of grain boundary regions.



§ Homogenised specimens were studied after slow cooling in air and after rapid quenching.

Figure 1. Production route for 8090 sheet and plate.

## Results

The grain structures for each alloy in the hot rolled condition are included in Figure 2; LV329 (0.1% Zr) exhibited the finest grain structure consistent with increased concentration of Zr grain refining additions.

All alloys contained an inhomogeneous distribution of Fe-containing intermetallic particles which form interdendritically during solidification.

The presence, average size (length of particle) and aspect ratio of coarse grain boundary particles of the I-phase at various stages in the TMP route is summarised in Table 2. I-phase particles were measured using bright field imaging and were identified by standard diffraction analysis, an example of which is shown in Figure 3.

Some small I-phase particles ( $<0.5\mu\text{m}$  long,  $0.2\mu\text{m}$  thick) were observed in alloy LV329 (0.1%Zr) after slow cooling from the homogenisation temperature, the absence of any such particles in alloys homogenised and rapidly quenched has confirmed that the I-phase present in alloy LV329 after homogenisation originated during slow cooling from  $550^\circ\text{C}$  rather than through precipitation at the homogenisation temperature.

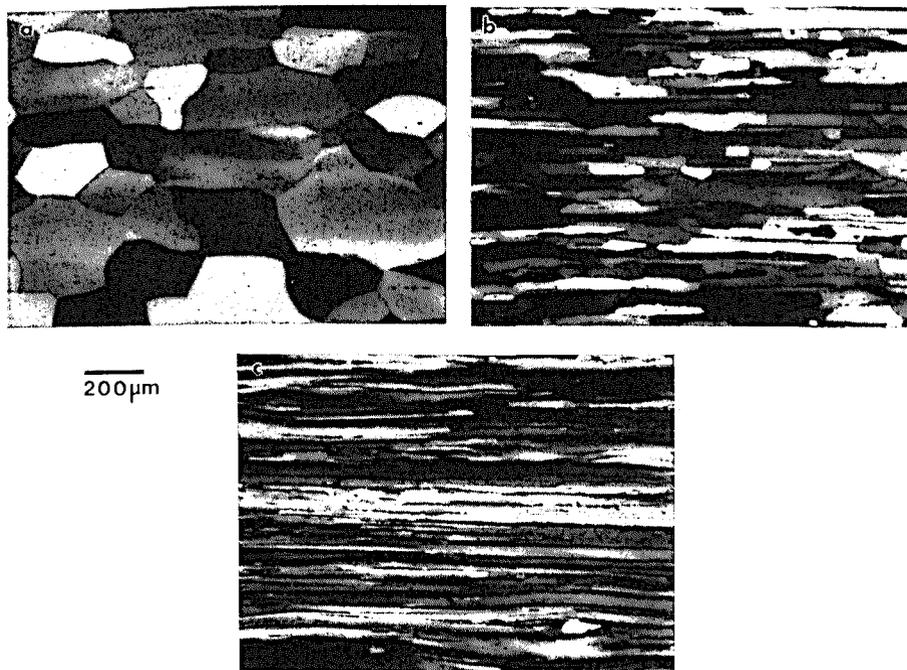


Figure 2. Grain structures of 7.5mm plate following solution heat treatment at  $540^\circ\text{C}$ ; (a) LV346, (b) LV326, (c) LV329.

Table 2. Abundance, particle sizes and aspect ratio in alloys at various stages of processing.

TMP condition	Alloy	Abundance	Size	Aspect Ratio
Homogenised C	All	---	---	---
Homogenised slow cool	LV346	---	---	---
	LV326	---	---	---
	LV329	√	$<0.3\mu\text{m}$	1:1-2:1
Hot Rolled	LV346	---	---	---
	LV326	√√	$0.2-0.3\mu\text{m}$	1:1-2:1
	LV329	√√√	$0.1-2\mu\text{m}$	1:1-2:1
SHT 1hr @ $540^\circ\text{C}$	LV346	---	---	---
	LV326	---	---	---
	LV329	√	$0.5\mu\text{m}$	2:1-4:1

√ traces      √√ small amounts      √√√ large amounts      --- absent

An increased density of I-phase particles, having a wide size range (0.1-2.0 $\mu\text{m}$ ), was observed in the high Zr containing alloy (LV329) after hot rolling (Figure 4a); in the same condition the alloy LV326, containing 0.05% Zr, exhibited smaller particles (0.2-0.3 $\mu\text{m}$ ) which were not present in the homogenised condition (Figure 4b). The particles present in LV 329 after solution treatment were of different morphology in that they had a relatively high aspect ratio and were elongated along the grain boundaries (Figure 5).

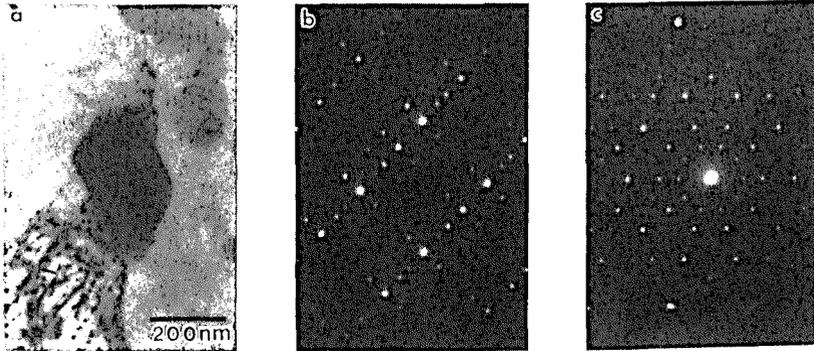


Figure 3. I-phase in LV329; (a) Bright Field image, (b) 2-fold diffraction pattern, (c) 5-fold diffraction pattern.

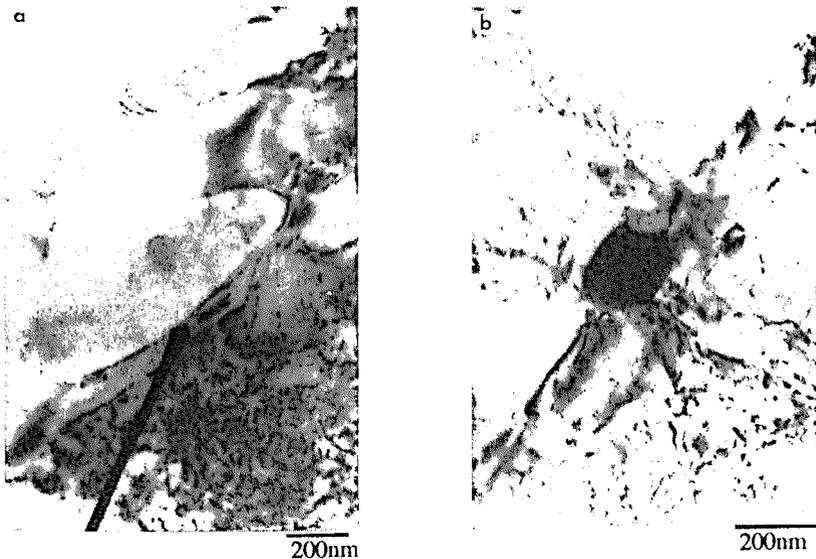


Figure 4. I-phase particles after hot rolling; (a) LV326, (b) LV329.

It is evident that there are significant amounts of grain boundary I-phase particles in alloys with Zr additions typical of commercial 8090 (LV326 and LV329). Furthermore, the I-phase particles were found to be distributed heterogeneously and were observed in regions having a high

concentration of  $\beta'$  ( $\text{Al}_3\text{Zr}$ ) particles (Figure 6). The I-phase is less prevalent in alloys of lower Zr content and has not been observed in the Zr-free alloy.



Figure 5. High aspect ratio I-phase particles in LV329 after SHT.

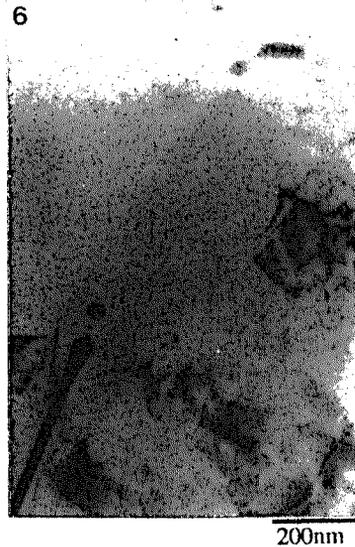


Figure 6. Coincident I-phase and  $\beta'$  ( $\text{Al}_3\text{Zr}$ ) particles in LV329.

### Discussion

The observed formation of I-phase particles in alloy LV329 during slow cooling from the homogenisation temperature ( $550^\circ\text{C}$ ) and during subsequent hot rolling, is consistent with previous work in which, in 8090, such particles are shown to form in the temperature range  $300\text{--}350^\circ\text{C}$  (7) and  $300\text{--}400^\circ\text{C}$  (10). The large size range of these particles after hot rolling may reflect continuous nucleation and growth during cooling from the homogenation temperature and during the extended time at elevated temperature during hot rolling. The change in both size and morphology of the grain boundary particles after solution treatment suggests a dissolution and reprecipitation reaction. This is consistent with previous work (9) in which suppression of the grain boundary precipitation by very rapid quenching after solution heat treatment has been reported. Quantification of the dissolution and reprecipitation kinetics, with a view to defining solution treatment practices capable of suppressing I-phase formation, is the subject of ongoing research.

The alloy leaner in Zr (LV326) appeared to have a lower driving force for formation of the I-phase, with finer particles ( $0.2\text{--}0.3\mu\text{m}$ ) being present after hot rolling. The present research indicates that both the bulk Zr content and localised Zr concentration affect the development of the I-phase. This local influence of Zr may be derived either from the presence of fine  $\beta'$  ( $\text{Al}_3\text{Zr}$ ) particles which are distributed inhomogeneously following homogenisation; this distribution resulting from the peritectic solidification, or from the Zr solute atoms, the concentration of which may also vary locally.

The presence of  $\beta'$  ( $\text{Al}_3\text{Zr}$ ) particles have previously been shown to act as preferential sites for the nucleation of  $\delta'$  ( $\text{Al}_3\text{Li}$ ) (12,13),  $\theta$  ( $\text{CuAl}_2$ ) (14) and  $\text{T}_1$  ( $\text{Al}_2\text{CuLi}$ ) (15) during ageing heat treatment. Heterogeneous precipitation of the I-phase on these coherent  $\beta'$  particles at high temperatures seems unlikely, although the present study and previous work (15) often shows I-phase particles and  $\beta'$  particles to be coincident. The Zr solute atoms may additionally influence chemical microsegregation and the kinetics of formation and dissolution of I-phase particles, and this is the subject of further investigation. Nevertheless, alloys LV326 and LV329 would be expected to differ only in the  $\beta'$  particle content since the solid solubility of Zr in Al, being 0.05% wt at 500°C (16,17), is well below the bulk Zr concentration in the alloys. Thus, the substantial differences in I-phase formation observed in the present work may be influenced indirectly by  $\beta'$  particles, but further work is required to develop satisfactory mechanism for such an effect.

### Conclusions

In 8090 type alloys of commercial composition the I-phase forms during the early stages of thermomechanical processing. There is evidence of dissolution and reprecipitation of this phase during a standard solution heat treatment practice. The present work has also shown that I-phase formation is influenced by Zr content: low Zr additions reduce the incidence of grain boundary I-phase particles.

### Acknowledgements

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### References

1. J. White and W. S. Miller, (1987), Proc.4th Intl. Aluminium-Lithium Conf., Paris (France), Eds. G. Champier, B. Dubost, D. Miannay and L. Sabetay, C3-425-431.
2. C. Degand, K. Wang and P. Garoche, (1993), *J.Non-Crys. Sol.*, **153 & 154**, 478-481.
3. P. Sainford and B. Dubost, (1986) *J.Phys. Coll. C3*, **7**, 321-330.
4. N. Larguet, A. Chenoufi, J. Dixmier, B. Bouchet-Fabre, M. Harmelin, (1991), *Phase Transitions*, **B32 (1-4)**, 103-111.
5. X. Nie, R. Wang and Y. Yiyang, (1993), *Phys.Stat.Sol.B*, **177 (2)**, 261-268.
6. P. Donnadiou and C. Degand, (1993) *Phil.Mag.B*, **68 (3)**, 317-328.
7. M. Ball and H. Lagace, (1985), Aluminium-Lithium Alloys III, Proc. 3rd Intl. Aluminium-Lithium Conf., Oxford (England), Eds. C. Baker, P. J. Gregson, S. J. Harris and C. J. Peel, 555-564.
8. W. A. Cassada, G. J. Shiflet and S. J. Spoon, (1986), *Phys.Rev.Lett.*, **56**, Number 21, 2276-2279.
9. P. J. Gregson and S. A. Court, (1994), *Scripta Metall. Mater.*, **30**, Number 10, 1358-1363.

10. A. K. Mukhopadhyay, D. S. Zhou and Q. B. Yang, (1992), *Scripta Metall. Mater.*, **26**, 237-242.
11. H. U. Nissen and C. Beeli, (1993) *J.Non-Crys. Sol.* **153 &154**, 68-71.
12. P. J. Gregson and H. M. Flower, (1984) *J.Mat. Sci.Lett.*, **3**, 829-834.
13. H. M. Flower and P. J. Gregson, (1987), *Mat.Sci. Techn.*, **3**, 81-90
14. C. N. J. Tite, P. J. Gregson and P. D. Pitcher, (1988) *Scripta Metall.*, **22**, Number 7,1005-1010.
15. K. Satya Prasad, A. K. Mukhopadhyay, A. A. Gokhale, D. Banerjee and D. B. Goel, (1994), *Scripta Metall. Mater.*, **30**, Number 10, 1299-1304.
16. L. F. Mondolfo. Aluminium Alloys- Structures and Properties, pp. 413-414, Butterworth and Co (Publishers) Ltd, London (1976).
17. H. W. L. Phillips. Equilibrium Diagrams of Aluminium Alloy Systems, p. 40 Aluminium Development Association, London (1961).