# THE 4TH INTERNATIONAL CONFERENCE ON ALUMINUM ALLOYS

# LOW TEMPERATURE EMBRITTLEMENT OF 8090 IN THE DAMAGE TOLERANT CONDITION

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# Abstract

8090 and a binary Al-2.5Li alloy have been aged to a damage tolerant condition (24h at  $150 \,^{\circ}$ C) and subsequently exposed for up to 1000h at  $70 \,^{\circ}$ C. The 8090 underwent embrittlement but the Al-2.5Li alloy did not embrittle under these conditions. A further reversion treatment of 5 min. at 190  $^{\circ}$ C restored the toughness of 8090 to approximately that before exposure. These results are discussed in terms of the ageing processes taking place during low temperature exposure and during the reversion treatment.

# Introduction

Over the last few years a considerable amount of work has been carried out on the improvement of S-L toughness of 8090. Double ageing techniques have been developed and studied (1)(2)(3)(4) where a short second ageing treatment is given to the alloy at a temperature approximately 40 °C higher than the initial age. This treatment can double the fracture toughness with only a small decrease in strength relative to a conventional single age. Various explanations have been proposed for the improvement in properties promoted by the second age and these include desegregation of lithium atoms from the grain boundaries (1), reversion of small  $\delta'(Al_3Li)$  precipitates (2)(3) and disordering of ordered domains that are to be found in quenched or lightly aged alloys (4).

The double ageing techniques have also been applied to damage tolerant recrystallised 8090 sheet and here again the toughness is improved for sheet tested in both T-L and L-T orientations (2). However, a further problem arises with the 8090 damage tolerant sheet due in part to its lightly-aged condition (e.g. 24h 150°C). Such sheet if used at a slightly elevated temperature (e.g. 70°C) increases in strength and reduces in fracture toughness during service. A double age treatment, i.e. short second age at 190°C after exposure at 70°C, restores the toughness of the 8090 sheet to the value it had before exposure (2).

Although it is known that a double-age will remove embrittlement produced by low temperature exposure of damage tolerant 8090 sheet, less detail is available about the precise time and temperature required for the second ageing treatment. The present paper describes some preliminary data on this aspect. Additionally, results are given for the low temperature exposure of binary Al-2.5% Li alloy in an initial attempt to establish the metallurgical processes taking place during embrittlement.

# Experimental

Two alloys have been studied, 8090 and binary Al-2.5wt.%-0.10wt.%Zr, both in sheet form and processed to give a recrystallised grain structure. The damage tolerant ageing condition was 24h at 150 °C and all mechanical properties were measured for the L-T orientation. Standard servo-hydraulic tests were used for tensile properties and the Kahn tear test to measure fracture energy. Electrical resistivity measurements were by a four-probe potentiometric technique and were carried out at liquid nitrogen temperature (77 K).

# Results and Discussion

The embrittlement characteristics of both alloys was first established by holding the aged alloy (24h at  $150^{\circ}$ C) at  $70^{\circ}$ C, this being representative of conditions that 8090 may experience when in service. The study was followed by a detailed examination of the alleviation of embrittlement by a short term second age at  $190^{\circ}$ C.

#### Embrittlement

Fig. 1 shows the changes in strength that occur when aged alloys (24h at  $150^{\circ}$ C) are held for times up to 1000h at  $70^{\circ}$ C (subsequently referred to as exposure). The proof stress of 8090 increases by ~25 MPa during exposure, and the shape of the plot indicates that the precipitation reactions taking place during exposure are not complete after 1000h. The change in proof stress of Al-2.5Li is considerably smaller than in 8090, and at ~5 MPa is within experimental scatter of the data.





Assessment of brittleness was made by measuring the energy to fracture in a Kahn tear test (fig. 1). Significant reductions in energy to fracture occur in 8090 as a result of exposure and again the reaction taking place appears to be incomplete after 1000h at 70°C. In contrast, the energy to fracture Al-2.5Li alloy is unchanged by exposures of up to 1000h at 70°C.

The precipitation reaction taking place during exposure of Al-2.5Li alloy is  $\delta'$  precipitation due to the increased lithium supersaturation resulting from the change in ageing temperature from 150°C to 70°C. TEM examination gave values of ~7 nm for the radius of  $\delta'$  after ageing 24h at 150°C and this was substantially unchanged after further exposure at 70°C. Since the change in lithium supersaturation must cause further lithium to come out of solid solution, further very fine  $\delta'$  precipitation must be taking place in the matrix between existing  $\delta'$  particles. This fine  $\delta'$  precipitation could not be detected in the present study due to the masking effect from the small size and high volume fraction of the pre-existing  $\delta'$  from the 150°C age. However, it is probably of a size 1-2 nm radius and with a volume fraction of 0.03 (calculated from the difference in  $\delta'$  solvus at 150 and 70°C). Bimodal distributions of  $\delta'$  in 8090 have been successfully imaged by Pitcher et al (2) by employing initial ageing temperatures higher than 150°C, e.g. alloys aged 24h at 190°C followed by 100h at 170°C produced fine background  $\delta'$  precipitates of ~2 nm radius.

In the 8090 alloy the likely reactions taking place during exposure are a combination of  $\delta$ ' and GPB zone (or precursor to S') precipitation. The  $\delta$ ' formation is again due to the change in lithium supersaturation as the ageing temperature is changed from 150 to 70°C. GPB zones have not been directly observed in the present work as a result of exposure, again due to the high volume fraction of  $\delta$ ' from the 150°C age, but their presence is likely for two reasons. Firstly, many previous investigators have produced evidence for the presence of GPB zones in Al-Li-Cu-Mg alloys aged at low temperatures, e.g. Miller et al (5) after 1000h at 20°C, Ozbilen et al (6) after 5000h at 20°C, Welpmann et al (7) after 100h at 150°C, and Gomiero et al (8) after 48h at 150°C. Secondly, TEM observation in the present work revealed fine needle-shaped S' in alloys given long term exposure at 70°C and therefore it is possible that GPB zones may also be present at an earlier stage of exposure at this temperature.

Since further precipitation of fine  $\delta'$  during exposure of Al-2.5Li at 70°C does not appear to embrittle the alloy, it follows that further  $\delta'$  precipitation may not be the major cause of embrittlement of 8090 exposed to 70°C. It also follows, of course, that segregation of lithium to grain boundaries or order/disorder changes also may not be the major embrittling process in 8090. This leaves the possibility that GPB zones or other precursor to S' may have an important role in embrittlement of 8090.

Precipitation of S' particles at high ageing temperatures is known to reduce brittleness because S' is not sheared by dislocations and therefore helps to disperse slip (9). However, GPB zones formed at low ageing temperatures will be sheared by dislocations and will therefore probably intensify slip localisation and cause reduced toughness. There is little direct information of the effect of GPB zones on fracture toughness of 8090 but their formation at 20°C is known to reduce elongation to fracture. Also, Pitcher et al (2) have shown that 8090 naturally aged at 20°C for 2400 h (a significant fraction of GPB will be present in the microstructure) has a lower fracture toughness than 8090 aged 32h at 170°C (which will have a  $\delta$ ' microstructure).

#### Second Ageing Treatment at Higher Temperature

To alleviate the embrittlement produced by long term exposure of 8090 at 70°C a second age at 190°C has been given to the alloy (hereafter referred to as a reversion-age). 190°C is the first of a series of reversion temperatures to be studied and was selected as being a temperature well below the  $\delta$ ' solvus (which is approximately 290°C for Al-2.5Li) and therefore large  $\delta$ ' precipitates produced by the initial 150°C age should be unaffected. However, according to the capillarity calculations of Baumann et al (10) this temperature is high enough to dissolve the very small (~1 nm radius)  $\delta$ ' particles produced by the low temperature exposure. For the 8090 alloy, the temperature is also sufficient to dissolve a significant fraction of GPB zones which have a lower solvus temperature than  $\delta$ '.

For the reversion treatment at 190 °C to be successful in restoring toughness to 8090, a balance has to be achieved between allowing sufficient time to reverse the precipitation reaction that has taken place during exposure, and insufficient time to precipitate a significant fraction of coarse  $\delta$ ' which would reduce strength to a level below that achieved by the initial age at 150 °C. To assess the optimum time required at 190 °C two types of measurement were carried out on 8090; tensile tests and resistivity determinations.

Tensile tests were carried out on 8090 aged 24h at  $150^{\circ}$ C followed by a short second age at  $190^{\circ}$ C for 5-120 min., i.e. with no low temperature exposure. Results are given in fig. 2 and show a reduction in proof stress (relative to 24h at  $150^{\circ}$ C) of 25 MPa after 120 min. at  $190^{\circ}$ C and an associated increase in fracture energy of 10 kJ/m<sup>2</sup>.



Fig. 2. Effect of second age at  $190^{\circ}$ C on the tensile and fracture properties of 8090 with no prior exposure at  $70^{\circ}$ C.



Fig. 3. Effect of a reversion-age at 190°C on the resistivity of 8090 with prior exposure of 1000 hours at 70°C.

Resistivity measurements were carried out on 8090 aged 24h at 150 °C, exposed 1000h at 70 °C, and followed by a reversion-age at 190 °C for 5-50 min. Fig. 3 shows results normalised to the resistivity value measured after 24h at 150 °C. After low temperature exposure the resistivity has fallen approximately 8% due to precipitation during exposure. A reversion-age of a few minutes at 190 °C is sufficient to restore the resistivity back to that of 24h at 150 °C, i.e. to redissolve those precipitates produced during exposure. Longer reversion times at 190 °C cause the resistivity to increase further as the smaller size fraction of the  $\delta$ ' distribution, produced by the 150 °C age, go back into solid solution.

From these tests a standard reversion treatment of 5 min. at 190°C was chosen on the basis that all the low temperature exposure reaction would be negated and that the treatment would not cause a reduction in proof stress (relative to 24h at 150°C) greater than 10 MPa.

#### Alleviation of Embrittlement

Figs. 4 and 5 show the effect of the reversion-age of 5 min. at  $190^{\circ}$ C on 8090 that has been exposed for up to 1000h at 70°C. The reversion treatment has reduced the proof stress almost back to the level attained in 8090 after reversion without low temperature exposure. More importantly, the energy to fracture shows significant improvements due to the reversion treatment. Also included on fig. 5 is data by Pitcher et al (2) where toughness measurements have been made on larger samples than those used in the present study. The trend of improvement of toughness by reversion is the same for both the small and larger samples.



Fig. 4. Effect of reversion-age of 5 min. at 190°C on the tensile properties of 8090 exposed for different times at 70°C.

Fig. 5. Effect of a reversion-age of 5 min. at 190°C on the fracture properties of 8090 exposed at different times at 70°C. Previous data from Pitcher et al (2).

TEM examination of alloys not exposed but given the second ageing treatment at 190°C showed little change in the size of the coarse  $\delta$ ' precipitates, but it should be noted that small changes in  $\delta$ ' caused by double ageing are difficult to measure by TEM and conflicting results have been reported in the literature. Perhaps the most reliable data is that produced by small angle neutron scattering (4) which showed a second age of 15 min. at 200°C after an initial age of 32h at 170°C produced a reduction in volume fraction of  $\delta$ ' from 0.13 to 0.11 and a small decrease in  $\delta$ ' size. In the present work the effect of reversion on the fine  $\delta$ ' and GPB from the exposure at 70°C could not be assessed by TEM due to the high volume fraction of  $\delta$ ' present from the 150°C age. However, for reasons outlined in the previous section, it is likely that the reversion treatment will have dissolved most of the GPB zones along with fine (1-2 nm radius)  $\delta$ ' particles, thus leading to the alleviation of embrittlement produced by the exposure of 8090 for long periods at 70°C.

#### Effect of a Second Age Prior to Exposure

Data from fig. 2 demonstrates that 8090 aged 24h at  $150^{\circ}$ C and then given a second age at  $190^{\circ}$ C (without low temperature exposure) produces an improvement in fracture energy. Similar effects have been reported in detail by Lynch (1) for alloys aged at  $170^{\circ}$ C followed by a second age at 200-230°C, by Pitcher et al (2) for alloys aged at  $150-170^{\circ}$ C followed by a second age at  $190-210^{\circ}$ C, and by Blankenship et al (3) for alloys aged at  $170^{\circ}$ C followed by a second age at  $230-300^{\circ}$ C.



Fig. 6. Effect of exposure at 70°C on the tensile and fracture properties of 8090 previously given a second ageing treatment of 5 minutes at 190°C.

A series of tests were undertaken to see if this improvement in toughness was maintained after subsequent low temperature exposure at  $70 \,^{\circ}$ C. Fig. 6 shows this to be the case; fracture energy is approximately 10% higher at all exposure times and the increase appears to be independent of the time given for the second age. As would be expected, the downward trend of fracture energy with increasing exposure time remains unchanged. Unfortunately, for the ageing conditions used in the present work, the decrease in toughness with exposure far outweighs the improvement achieved by the second age applied before exposure.

# **Conclusions**

- (a) For 8090 aged 24h at 150°C an exposure of 1000h at 70°C produces a 28% decrease in fracture energy and a 6% increase in proof stress. A binary Al-2.5Li alloy given a similar treatment results in no change of fracture energy and <3% increase in proof stress.
- (b) The embrittlement of 8090 during exposure at 70 °C is probably being caused by further ageing of the alloy which produces a mixture of fine  $\delta$ ' particles and GPB zones. Further work is under way to establish the relative importance of  $\delta$ ' and GPB zones in this embrittlement process.
- (c) For 8090 exposed 1000h at 70°C a reversion-age of 5 min. at 190°C restores the proof stress and fracture energy to levels approximately the same as those before exposure.
- (d) The alleviation of embrittlement due to reversion-ageing is probably the result of dissolution of fine  $\delta$ ' and small GPB zones. Again, further work is under way to determine the relative importance of these two processes.

# Acknowledgements

The authors thank B Evans and P D Pitcher of the RAE for many helpful discussions, and the Defence Research Agency, Farnborough, for financial assistance.

# References

- 1. S P Lynch, Mat. Sci. and Eng., A136, 1991, 25.
- 2. P D Pitcher, D S McDarmaid, C J Peel and G Hall, <u>Proc. Sixth Int. Al-Li Conf.</u>, DGM, Oberursel, Germany, 1992, p.235.
- 3. C P Blankenship and E A Starke Jr., Met. Trans. A, 24A, 1993, 833.
- 4. P D Pitcher, R J Stewart and S Gupta, Scripta Metall. Mater., 26, 1992, 511.
- 5. W S Miller, J White and D J Lloyd, Int. Conf. <u>"Aluminium Alloys their Physical and Mechanical Properties"</u>, University of Virginia, Ed. E A Starke Jr. and T H Sanders Jr., EMAS, Cradley Heath, UK, 1986, p.1799.
- 6. S Özbilen and H M Flower, Proc. Fifth Int. Al-Li Conf., MCEP, Warley, UK, 1989, p.651.
- 7. K Welpmann, M Peters and T H Sanders Jr., <u>Proc. Third Int. Al-Li Conf.</u>, Inst. of Metals, London, 1986, p.524.
- 8. P Gomiero, F Livet, O Lyon and J P Simon, <u>Acta Metall. Mater.</u>, <u>39</u>, 1991, 3007.
- 9. P J Gregson and H M Flower, Acta Metall., 33, 1985, 527.
- 10. S F Baumann and D B Williams, Acta Metall., 33, 1985, 1069.