QUANTIFICATION OF δ' PRECIPITATION OCCURRING DURING LOW TEMPERATURE EXPOSURE OF BINARY AL-LI ALLOYS

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ABSTRACT

Binary Al-Li alloys containing 1.7 and 2.6wt%Li have been aged 24h at 150°C and subsequently exposed for up to 1000h at 70-130°C. After exposure, δ' volume fractions have been calculated from electrical resistivity measurements and δ' sizes determined by transmission electron microscopy. These data have been analysed in terms of order strengthening and compared with data available in the literature.

Keywords: Aluminium-Lithium Alloys, δ' Precipitation, Exposure, Order-Strengthening.

1. INTRODUCTION

Aged Al-Li alloys when exposed in service to temperatures of 70-100°C, undergo continued-ageing which results in an increase of strength and a reduction in ductility and toughness [1]. This thermal instability is a problem to the aerospace industry where potential candidate materials need to withstand service exposure to temperatures up to 80-90°C. One possible cause of the exposure embrittlement in binary Al-Li alloys is the formation of additional fine δ' at low exposure temperatures and the growth of existing δ' at higher exposure temperatures [2] [3]. However, very little information is available as to the quantity of δ' being produced during service exposure. This has been the objective of the present work. The size, r, and volume fraction, f, of δ' produced by exposure has been measured by transmission electron microscopy (TEM) and electrical resistivity respectively. The resulting data have been analysed in terms of order strengthening, the magnitude of which is proportional to $(rf)^{1/2}$.

2. EXPERIMENTAL

Two Al-Li alloys containing 1.74 and 2.63wt% Li were cast under argon and processed to 1.6mm strip by hot and cold rolling. Both alloys contained 0.08wt%Zr to control the grain size, and both alloys recrystallised during solution treatment to produce an equiaxed grain structure of approximate size 100μm.

A standard damage-tolerant heat treatment of 24h at 150°C was given to both alloys after solution treatment. To simulate exposure conditions, the alloys were then further heat treated in silicone oil baths for times up to 1000h at temperatures of 70, 100 and 130°C. The electrical resistivity was monitored during the exposure period. Samples were also removed from the oil baths after various exposure times and subjected to standard tensile testing and TEM examination.

3. RESISTIVITY CHANGES DURING EXPOSURE

The presence of lithium in aluminium solid solution significantly increases the electrical resistivity [4].Since the formation of δ' removes lithium from solid solution, the precipitation of δ' can be followed by electrical resistivity measurements. Fig.1 shows the changes in resistivity of the 1.7Li alloy during the standard pre age at 150°C and for subsequent exposure at 70, 100 and 130°C. The 24h age at 150°C produces only a small change in resistivity, ie very little δ' precipitation, and this was confirmed by TEM observations. Subsequent exposure at 70°C produces very fine δ' precipitation ($r \sim 1$nm) and this results in an increase of resistivity which is still increasing after a 1000h exposure. Exposure at 100°C produces a significant decrease in resistivity and TEM showed

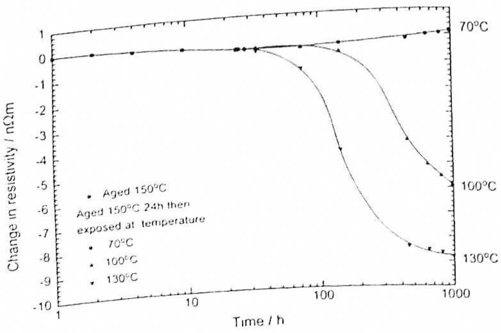

Fig. 1 Change in resistivity during the 150°C pre-age and during exposure at 70, 100 and 130°C. Alloy composition 1.7%Li.

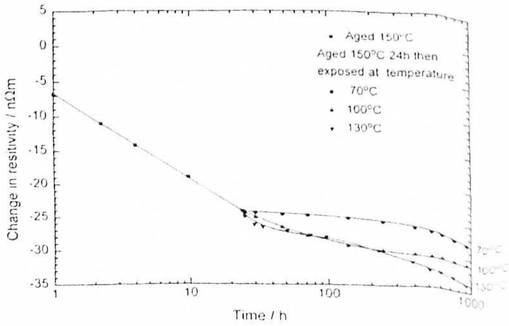

Fig. 2 Change in resistivity during the 150°C pre-age and during exposure at 70, 100 and 130°C. Alloy composition 2.6%Li.

the size of the resulting δ' to be 3.0nm after 1000h. Exposure at 130°C caused an ever larg decrease in resistivity and in this case the δ' had grown to a size of 14.5nm during exposure.

The volume fraction of δ' that forms during exposure is difficult to measure by TEM due precipitate overlap effects. However, an accurate volume fraction can be obtained from resistivity measurements using the expression

$$f = \frac{C_o - C_m}{C_p - C_s} \qquad (1)$$

Where C_o, C_m, C_p and C_s are respectively the concentrations of lithium in the alloy, in the matrix temperature T, in the δ' precipitate at temperature T, and at the δ' solvus at temperature T. Th resistivity changes were converted to concentrations using an expression determined by Noble a Thompson [4], and a recent measurement of the δ' solvus [5] was used to obtain C_s. The to volume fraction of δ' in the alloy after 1000h exposure at 100°C was calculated at 0.039 and th increased to 0.053 after 1000h at 130°C. It should be noted that this means that only 40% of t total possible δ' has precipitated during a 1000h exposure at 100°C, and only 60% at 130 °C.

Fig. 2 shows the change in resistivity of the 2.6Li alloy during the standard pre-age at 150°C and subsequent exposure at 70, 100 and 130°C. Here, the 24h at 150°C has precipitated a signific amount of δ' and the subsequent exposure at lower temperatures has continued this process. T volume fractions that precipitate can again be calculated from the resistivity data, but in the case the 2.6Li alloy it should be noted that it is essential to allow for the fact that δ' precipitation dur exposure can take place only in that part of the matrix that has not transformed during the pre-ag After a 1000h exposure at 100°C the total volume fraction of δ' was calculated to be 0.197 and th represents 79% of the total possible δ' that is able to form at this temperature. It can be conclu that in both the 1.7Li and 2.6Li alloys after 1000h exposure time, substantial amounts of δ' are st able to precipitate if the exposure heat treatment is continued beyond 1000h.

4. MECHANICAL PROPERTY CHANGES DURING EXPOSURE

The formation of δ' during exposure, as described in the last section, is accompanied by an increasing strength and reduced ductility. Figs. 3 and 4 show typical tensile data for the 1.7Li and 2.6Li alloys exposed for up to 1000h at 130°C. Similar but smaller changes were observed for 100°C exposure. It is these changes in mechanical properties that occur in Al-Li alloys during exposure that are posing a real problem to the aerospace industry.

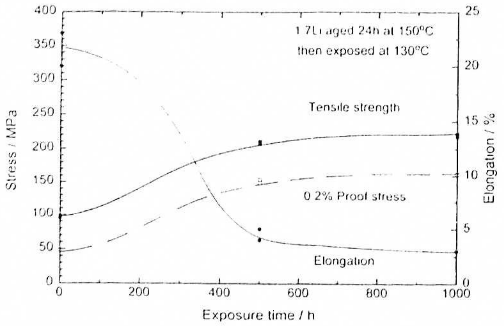

Fig. 3 Tensile mechanical properties after exposure at 130°C. Alloy composition 1.7%Li.

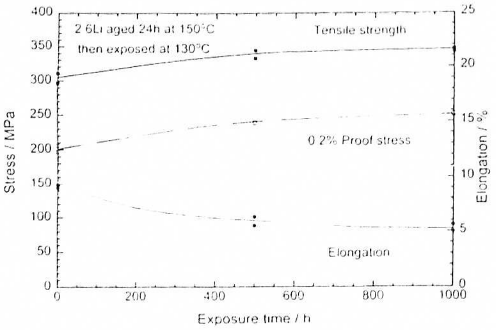

Fig. 4 Tensile mechanical properties after exposure at 130°C. Alloy composition 2.6%Li.

The changes in proof stress that occur during exposure at 130°C can be correlated to the size and volume fraction of δ' precipitated during exposure by assuming that the dominant dislocation precipitate interaction in the alloys is shearing of the δ' precipitate to produce order strengthening [6] [7]. Several recent papers are in the literature that give experimental data on the dependence of the critical resolved shear stress of under aged Al-Li single crystals on the radius, r, and volume fraction, f, of δ' precipitates [8] [9] [10]. The groups of researchers analyse their results in terms of order strengthening but two different approaches are used. Schlesier and Nembach [9] show that the increment of critical resolved shear stress, τ_p is given by the expression:

$$\frac{2b\tau_p}{f} = A_1\gamma^{\frac{3}{2}}\left(\frac{r}{fS}\right)^{1/2} + \gamma\left(A_2\xi - \propto\right) \qquad (2)$$

γ is the antiphase boundary energy on the {111} planes of the δ' phase, and b and S are respectively the Burgers vector and line tension of dislocations. The range of the interaction force between the leading dislocation of the gliding pair and the δ' particles is given by ($\xi\omega r$), where ξ is an adjustable parameter. The terms A_1, A_2 and ω are statistical factors and since the δ' particles were found to follow a Lifshitz-Slyozov-Wagner distribution, the values of these terms are 0.91, 0.44 and 0.82, respectively [9]. The expression used for line tension of the dislocation is:

$$S = Kb^2 In\left(\frac{L_s}{b}\right) \qquad (3)$$

K is the geometric mean of the elastic prefactors for edge and screw dislocations, and for Al-Li solutions is equal to 2.3 GPa. L_s is the square lattice spacing of the precipitates in the glide plane.

A plot of ($2b\ \tau_p/f$) against (r/fS) is given in Fig. 5 using various single crystal and polycrystalline literature data as assembled by Schlesier and Nembach [9]. Also on this plot are data from the present work. Data only from the standard 150°C age and 130°C exposure are shown since these treatments were the only ones to give a unimodal precipitate distribution. It should be noted that the present work has used polycrystalline material and to convert tensile proof stress to critical resolved shear stress a Taylor factor of 3.0 has been used. Fig. 5 shows that the data from the present work fits the order strengthening analysis of Schlesier and Nembach extremely well.

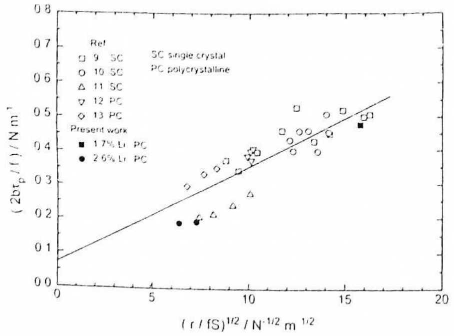

Fig. 5 Order strengthening plotted according to the relationship of Schlesier and Nembach [9].

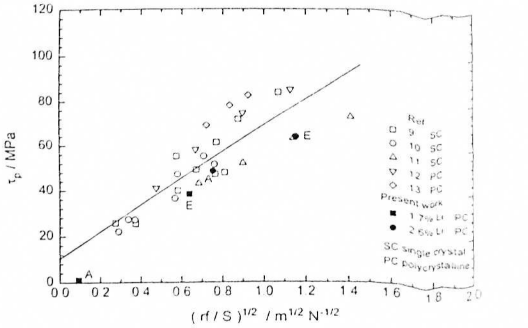

Fig. 6 Order strengthening plotted according to the relationship of Huang and Ardell [8] and Jeon and Park [10]. A is the aged Condition (24h 150°C) E is the exposed condition (1000h 130°C)

The alternative analysis of Huang and Ardell [8] and Jeon and Park [10] uses the relation:

$$\tau_p = \frac{\gamma}{2b}\left(\frac{3\pi^2\gamma}{32}\right)^{1/2}\left(\frac{rf}{S}\right)^{1/2} \tag{4}$$

In this case the favoured expression for the line tension of the dislocations is:

$$S = \frac{Gb^2}{4\pi}\left(\frac{1+\nu-\nu\beta^2}{1-\nu}\right)\ln\left(\frac{L_s}{2b\beta^{1/2}}\right) \tag{5}$$

G is the shear modulus, ν is poissons ratio and β is a measure of the strength of the δ' particles equal to πrγ/4S. Since S is dependent on r, f, and γ, an iterative procedure was required when plotting the data; values of γ and S were assumed and iteration undertaken until γ converged to a constant value [8] [10]. Fig. 6 shows literature data and data from the present work plotted according to this relation. Again, the present data fits well with existing order strengthening data of δ' precipitates. It should be noted that the line of best fit through the data on Fig. 6 does not pass through the origin, as would be demanded by equation (4). However, what is clearly evident from Fig. 6 is that exposure of both the 1.7Li and 2.6Li alloys at 130°C produces an increase in r and f that results in an increase of order strengthening that closely follows that observed by a variety of workers using higher ageing temperatures and conventional ageing practice.

5. CONCLUSIONS

Electrical resistivity measurements have been used to measure the volume fractions of δ' that precipitate during the long term exposure of Al-Li alloys containing 1.7 and 2.6%Li. For a standard pre-age of 24h at 150°C followed by 1000h at 100°C these volume fractions amounted to 0.039 and 0.197 for the 1.7Li and 2.6Li alloys respectively. This means that for both alloys a significant amount of δ' has yet to precipitate at times beyond a 1000h exposure. Mechanical property measurements have also been made after 1000h exposure and it has been shown that the increase in volume fraction and size of δ' that takes place during exposure produces a level of order strengthening that is consistent with literature data obtained at higher ageing temperatures using conventional ageing practices.

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