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Slip homogenization in Al–Li alloys by thermomechanical treatment and its effect on mechanical behaviour

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The affects of a pre-ageing stretch and of duplex ageing on slip distribution have been examined in the Al–Li–Cu–Mg–Zr alloy 8090 peak aged at 170 °C. Stretching prior to ageing and duplex ageing were found to effectively homogenize the distribution of S′ in these alloys. In contrast, in unstretched materials that were not duplex aged, precipitation of S′ was intense on the grain and subgrain boundaries but scarce elsewhere.

Tensile, cyclic stress–strain, long fatigue crack growth and small fatigue crack growth data were gathered. These data showed that slip was planar in the unstretched and duplex-aged materials as compared to materials that were stretched prior to ageing. A model is developed to calculate the diameter where Orowan looping of the S′ precipitates was likely to give way to shearing of those precipitates. Based on this model, it was concluded that, although the distribution of S′ precipitates was homogeneous in the duplex-aged materials, the precipitates were too fine to effectively homogenize slip. It was also shown that if the artificial ageing temperature was increased to 190 °C the S′ precipitates were thicker, leading to a change in deformation behaviour.

1. Introduction

Commercial interest in aluminium–lithium (Al–Li) alloys is largely based on the increased stiffness and decreased density that they offer over conventional aluminium alloys. Often, slip tends to be localized in these alloys because δ′ (Al_3Li), the main strengthening precipitate, is shearable and, once sheared, is less able to resist further dislocations [1]. This localization of strain results in dislocation pile-ups, stress concentrations at the grain boundaries, eventual grain boundary fracture and low overall ductility. Many efforts to improve ductility have thus focused on the addition of other elements which form non-shearable precipitates and thus homogenize slip.

In 8090 the aluminium, copper and magnesium combine to form S′ (Al_2CuMg) which has been found to improve ductility and fracture toughness through homogenizing slip [1, 2]. S′ precipitates heterogeneously, thus the distribution of S′ may be changed by changing the distribution of nucleation sites. One method of obtaining a homogeneous distribution of S′, and thereby homogenize slip, is to stretch the material prior to artificial ageing [2]. Stretching produces a uniform distribution of dislocations to act as nucleation sites for the S′.

Duplex ageing, incorporating a natural ageing step, has been suggested as an alternative method of providing a homogeneous distribution of nucleation sites for the S′ [3]. During the natural ageing step, δ′ particles grow. As these precipitates grow, they incorporate lithium. The vacancies, which were orginally strongly bound to the lithium, are released thus allowing the formation of vacancy clusters, dislocation loops and dislocation helices, all of which act as nucleation sites for the S′. The objective of this work was to see how the size and distribution of the S′ precipitates, produced by duplex ageing or a pre-ageing stretch, affected mechanical behaviour in the Al–Li–Cu–Mg–Zr alloy 8090.

2. Experimental materials and methods

2.1. Materials

The materials used in this work were high copper and low copper variants of 8090 plate manufactured using standard commercial practice by Alcan International. The high copper content alloy will be referred to as 8090(H) and the low copper content alloy as 8090(L). These materials have been described in previous work [4]. The chemical compositions, however, are given in Table I.

Three thermo–mechanical treatments were used in this work to vary the S′ phase distribution. One heat treatment was to artificially age 6% stretched material at 170 °C for 32 h. A second heat treatment began with

TABLE I Chemical compositions in wt % of the 8090 alloys

Alloy	Li	Cu	Mg	Zr	Fe	Si	Al
8090 (L)	2.29	1.10	0.63	0.13	0.06	0.03	Bal.
8091 (H)	2.26	1.57	0.61	0.15	0.05	0.04	Bal.

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solution treatment at 540 °C for 30 min followed by a 24 h natural age. The material was then artificially aged at 170 °C for 48 h. The third heat treatment, expected to result in a non-uniform distribution of S′ precipitates, was a solution treatment at 540 °C for 30 min immediately followed by artificial ageing at 170 °C for 100 h. These thermomechanical treatments will be referred to in the remainder of this work as stretched, duplex aged, and unstretched, respectively. All materials were in the peak aged condition as indicated by measurements of hardness and yield strength as a function of ageing time [5].

2.2. Experimental methods

The small crack propagation experiments were conducted at ambient temperature and in laboratory air. An Avery plane bender was used to apply a cyclic bending moment to a sheet specimen. The stress level chosen was either 85% or 70% of the yield strength of the sample and the stress ratio was zero. After being milled to the approximate dimensions, the test specimens were heat treated. Both sides of the test specimens were then further milled to remove material depleted of lithium by the solution treatment and mechanically polished.

Crack growth was monitored using cellulose acetate replicates of the test specimen surface. This offered the advantage of an historical record of crack growth. Replicates were taken of the specimen surface every 5 000 to 10 000 cycles until nucleation of the crack was observed. Thereafter, replicates were taken every 1 000 cycles. The replicates were taken with the specimen under maximum load so that the crack was fully open. The replicates were examined and photographed optically.

The small fatigue cracks studied in this work were typically inclined and/or kinked both in the plane of the specimen surface and perpendicular to that plane. In this work ΔK was calculated based on the projection of the crack on to the plane normal to the applied stress. Sectioning of several cracks demonstrated that they could be modelled as semi-elliptical in shape with a depth to length ratio (a/c) of 0.4. The Newman–Raju equation [6] for a semi-elliptical crack under bending was used to calculate ΔK.

Tests to determine the cyclic stress–strain behaviour of these alloys were done in accordance with ASTM standard E 606 with the exception that the specimen diameter was 5 mm rather than the recommended 6.35 mm. The cyclic stress–strain behaviour was measured under stress control with $R = -1$.

Thin foils for TEM work were mechanically cut and abraded into discs about 0.18 mm thick. These discs were electrochemically polished and dished. Lineal roughness parameters for small fatigue cracks were measured from optical photographs at 140 ×. A MOP-AMOS 2 digitizer was used to measure crack lengths.

3. Results and discussion

3.1. The distribution and size of the S′ phase

Thin foils of 8090(L) and 8090(H) in the stretched, unstretched, and duplex-aged conditions were examined in the TEM. Representative photographs showing the distribution of S′ are given in Figs 1 and 2. The δ′ precipitates were small, spherical and uniform in distribution for all the alloys (see Fig. 3). No δ precipitation was observed. Due to the increased concentration of copper in 8090(H), very small amounts of T_1 were occasionally observed as thin trapezoidal plates. Since the amount of T_1 precipitation was very small and their overall mechanical effect would be similar to that from the S′ phase, T_1 would be unlikely to have a distinct effect on properties. When present, δ′ and T_1 were unaffected by thermo-mechanical treatment.

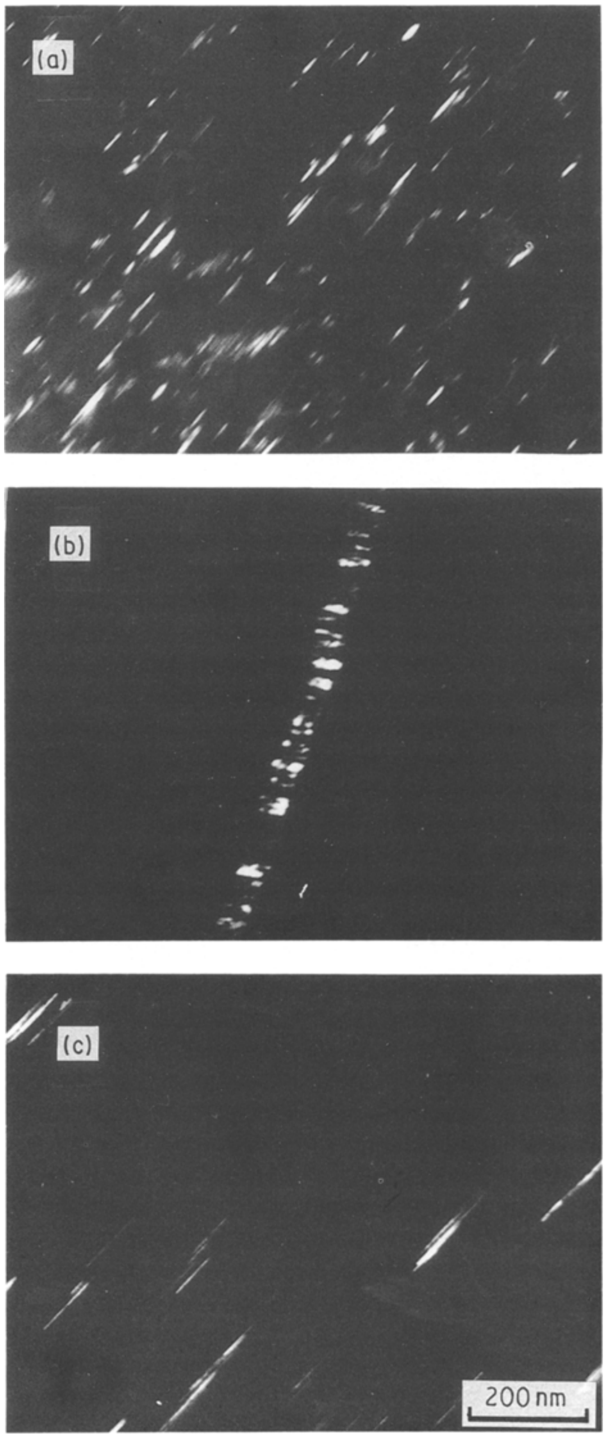

Figure 1 DF TEM photographs, $Z = \langle 100 \rangle$, showing distribution of S′ in 8090(L) (a) stretched, (b) unstretched and (c) duplex aged.

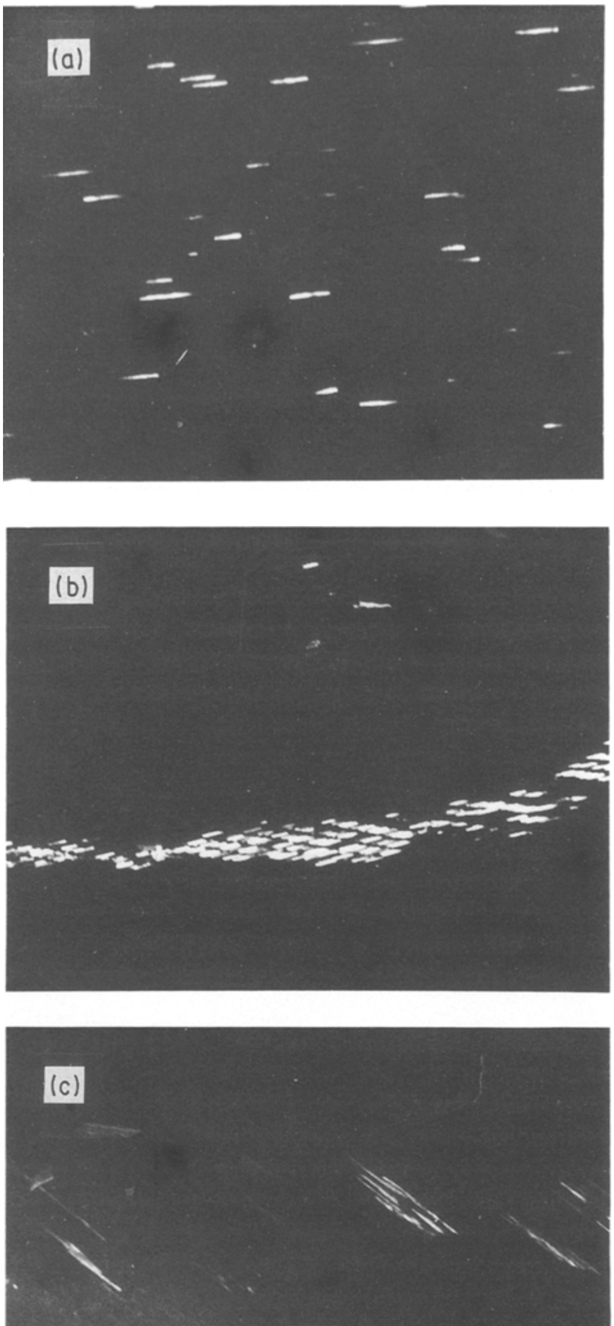

Figure 2 DF TEM photographs, $Z = \langle 100 \rangle$, showing distribution of S′ in 8090 (H) (a) stretched, (b) unstretched and (c) duplex aged.

Any change in slip behaviour as a function of thermo-mechanical treatment must be related to the distribution–size of the S′ precipitates.

The distribution of S′ was significantly affected by thermo–mechanical treatment. In the unstretched materials, precipitation of S′ was very heavy on the grain and subgrain boundaries but light elsewhere. This is the expected distribution because there were few S′ nucleation sites in the grain interiors. In contrast, the distribution of the S′ in the stretched materials was much more homogeneous because of the homogeneous distribution of dislocations which acted as nucleation sites. In the duplex-aged materials, where the vacancies had been liberated from the lithium and allowed to coalesce, the S′ was distributed throughout the grain in clumps. On the basis of the S′ distribution, the distribution of slip would be expected to be relatively homogeneous in the stretched and duplex-aged alloys as compared with the unstretched alloys.

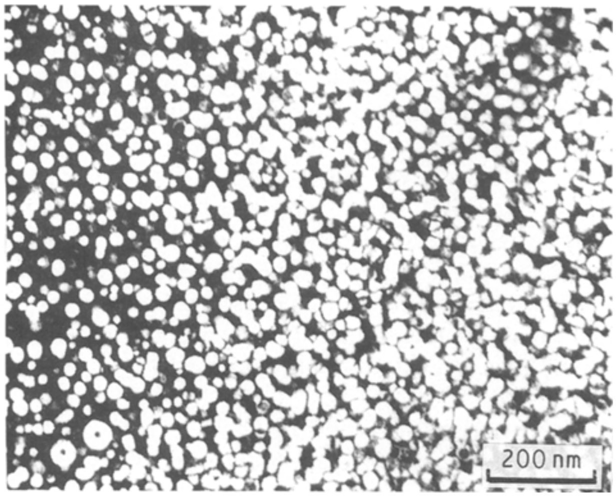

Figure 3 DF TEM photograph, $Z = \langle 100 \rangle$, showing distribution of δ′.

As shown in Table II, however, the size of the S′ precipitates was also affected by the thermo-mechanical treatment. Notably, the S′ precipitates resulting from the duplex age treatment were significantly smaller than in the unstretched and stretched alloys. The diameter of the S′ precipitates determines whether they are sheared or looped by dislocations. Precipitates that are looped would be effective slip homogenizers with successive dislocations being hindered by the passage of the first dislocation. Shearable precipitates would be less able to homogenize slip regardless of the distribution. In the following section the diameter of S′ particles required for Orowan looping is estimated.

3.2. Calculation of S′ diameter required for Orowan looping

Based on the literature [7], the precipitates were assumed to be circular in cross-section. In order to calculate the precipitate diameter where shearing gives way to Orowan looping, the geometry shown in Fig. 4 was used. From equilibrium of forces, $F = 2T\cos\theta$ where F is the force on the precipitate and T the

TABLE II S′ rod diameter as a function of material and thermo-mechanical treatment

Material	S′ rod diameter (nm)
8090 (L) stretched 6%	6
8090 (L) unstretched	4
8090 (L) duplex aged at 170 °C	2
8090 (H) stretched 6%	6
8090 (H) unstretched	5
8090 (H) duplex aged at 170 °C	3

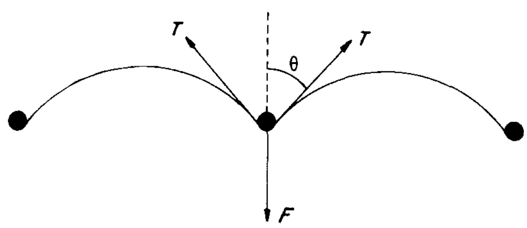

Figure 4 Schematic of dislocation held up at precipitate particles.

dislocation line tension. At the transition from particle cutting to Orowan looping θ equals 0. The dislocations that control plastic flow in aged Al–Li alloys are initially screw in character. When the dislocations are bowed around particles, however, they become edge dislocations in character [8]. So, taking T as approximately given by $T = Gb^2/2$, the force required for Orowan looping becomes $F = Gb^2$. G is the shear modulus of the matrix and b the Burger's vector.

The particle will be cut when the shear stress equals the shear strength of the particle (τ_c). The area on which the shear stress acts is $A = 1.732\pi d^2/4$ where the factor of 1.732 reflects the increase in cross-sectional area due to the angle at which the S′ precipitates are cut by the $\{111\}$ planes. The shear stress applied to the particle is $\tau = F/A$ which, when rearranged to solve for the force required to cut the particle, becomes $F = 1.36\tau_c d^2$. At the transition from looping to cutting the force required for looping must equal the force required for cutting. Rearrangement of that equality to solve for the critical precipitate diameter results in

$$d_c = \left[\frac{1.36Gb^2}{\tau_c}\right]^{1/2}$$

There are three quantities required for the evaluation of this expression, namely G, b and τ_c. G is the shear modulus of the matrix solid solution which would be very similar to the shear modulus of the overall alloy [9]. The Young's modulus of the overall alloy was 78.9 GPa so G (taken as approximately $0.4E$) is approximately 32 GPa. The Burger's vector is given by the average distance between atoms in the solid solution α, i.e. 0.286 nm [10]. Unfortunately, the shear strength τ_c of the S′ precipitates is not known. Its magnitude can, however, be bounded.

When magnesium is added to a 2.5% stretched Al–Li–Cu–Zr alloy of similar composition to the alloys studied in this work, the yield strength rises [11]. The addition of magnesium strengthens the alloys through several mechanisms including the dispersion hardening effect of the S′ phase. The lower bound of the strength of the S′ phase was, therefore, taken as the yield strength of an alloy containing 0% magnesium. From the literature [11] the tensile yield strength of such an alloy is 380 MPa. This value must be divided by two since τ_c is the yield strength of the S′ phase in shear. This results in a lower bound estimate for τ_c of 190 MPa.

The theoretical yield strength can be used as an upper bound. This is commonly considered to be $\tau_{theor} = G/10$. The modulus of intermetallic compounds can be approximately estimated by the rule of mixtures [12]. This results in an estimate of 26.8 GPa for the shear modulus of S′. The upper bound to the shear strength of S′ is, therefore, 2.68 GPa. With these bounds to the shear strength of S′, the diameter at which dislocations shear S′ precipitates rather than loop them must be between 0.8 and 3.1 nm.

This calculation of the transition diameter is, of course, only an estimate. The strength of S′ was estimated and it is assumed that precipitates only interact with single dislocations. In reality, the build up of dislocations in the vicinity of a precipitate will cause a local stress concentration that may cause shearing of particles with larger diameters than calculated above [13], nevertheless, quite conservative bounds were used for the strength of S′. The tendency of single dislocations to loop precipitates larger than 3.1 nm must influence slip distribution even if the precipitates are ultimately sheared when the stress concentration is sufficiently high. The estimate in the preceding paragraph, therefore, for the critical diameter is felt to be relevant to predicting whether, based on their diameters, the S′ precipitates are likely to homogenize slip.

Referring to Table II, it is apparent that looping is likely for the stretched and unstretched materials and that the S′ precipitates resulting from the duplex heat treatments are likely to be sheared. Thus, despite the homogeneous distribution of S′ in the duplex-aged alloys, the individual precipitates will be easily sheared and the S′ may be expected to be less effective at homogenizing slip.

3.3. Mechanical behaviour as a function of S′ distribution

In this section, the microstructural observations made in the preceding section will be related to the mechanical behaviour of these alloys. Monotonic tensile data are plotted in Fig. 5. The stretched materials strained the least for a given stress. The duplex-aged materials strained the most for a given stress in spite of the more uniform distribution of S′ described. The unstretched materials were intermediate in response between the stretched and the duplex-aged materials. On the whole, the 8090(H) materials were less affected by thermo–mechanical treatment than the 8090(L) materials – probably due to the enhancement of S′ precipitation by the higher copper content. The strain hardening exponent was 0.05 for all of these heat treatments and both alloys.

Cyclic stress–strain curves for the stretched, unstretched and duplex aged 8090(L) and 8090(H) alloys (all generated at R = − 1) are shown in Fig. 6. The trends for the cyclic stress–strain data are exactly similar to those observed in the tensile tests with the slightly higher average work hardening exponent of 0.08. These data indicate that the effectiveness of S′ in interfering with dislocation motion is greatest in the stretched alloys, intermediate in the unstretched alloys and the least in the duplex-aged alloys.

The same sequence of materials is also found in long fatigue crack data on the same alloys [14]. Long fatigue crack threshold values of ΔK were lowest in

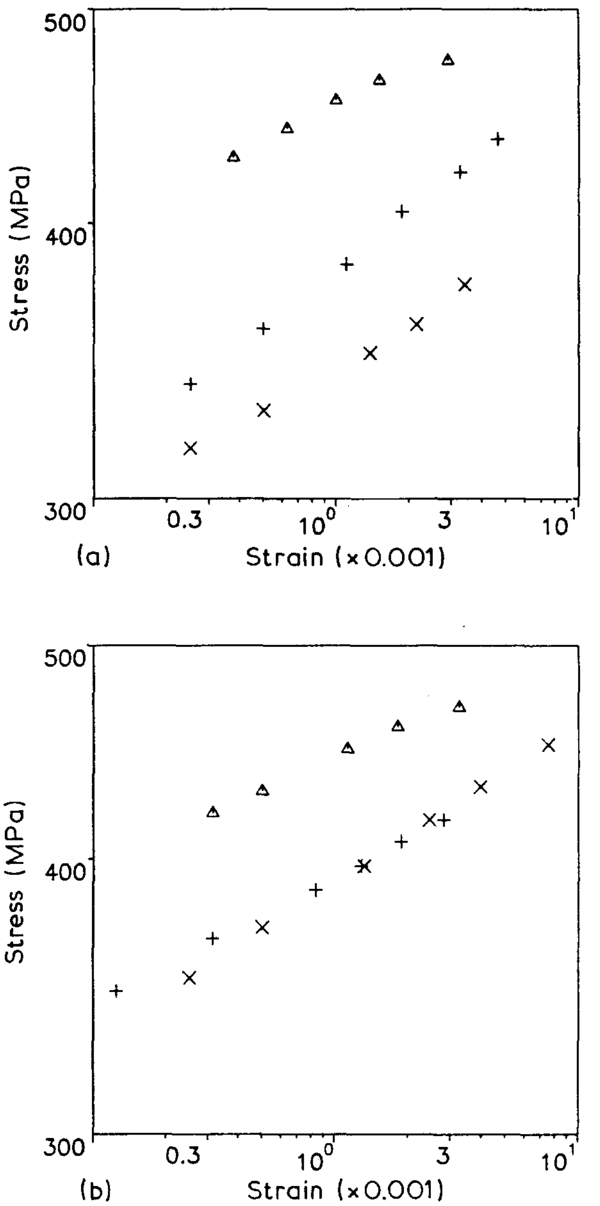

Figure 5 Tensile data for peak aged (a) 8090 (L) and (b) 8090(H). (Δ stretched, + unstretched, × duplex).

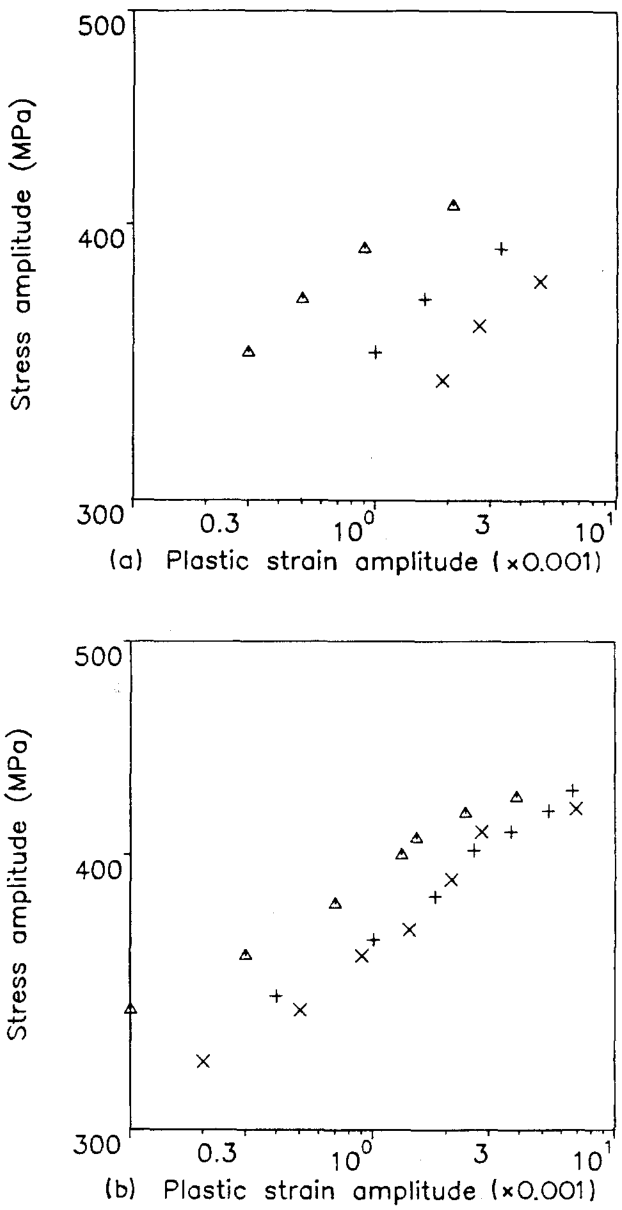

Figure 6 Cyclic stress–strain data for peak aged (a) 8090(L) and (b) 8090(H). (Δ stretched, + unstretched, × duplex).

the stretched materials, intermediate in the unstretched materials and highest in the duplex-aged materials. These differences were attributed to increased planarity of slip in the unstretched and duplex aged alloys, which again indicates that the S′ is less effective at homogenizing slip in the unstretched and duplex aged alloys.

In this work, the amount of slip planarity associated with small fatigue crack growth was quantified using measurements of the lineal roughness parameter (R_L). R_L is the ratio of the actual length of the crack to the length of the crack projected normal to the load. Increasing slip planarity results in higher values of R_L. As shown in Table III, the R_L values for the unstretched and duplex-aged materials are higher than those for the stretched materials, further demonstrating that the S′ is relatively ineffective at homogenizing slip in the unstretched and duplex-aged alloys.

The planarity of slip observed in the unstretched alloys appears to be associated with the inhomogeneous distribution of S′. In the duplex-aged materials, however, the distribution of S′ was relatively homogeneous. This suggests that the lack of the ability of S′ to homogenize slip in these alloys is associated with the small diameter of the S′ precipitates.

This conclusion is confirmed by data generated on duplex-aged materials where the artificial ageing temperature was increased by 20 to 190 °C. As shown in Table IV, the increase in artificial ageing temperature

TABLE III Lineal roughness parameter as a function of material and thermo-mechanical treatment

Material	Average R_L
8090 (L) stretched	1.19
8090 (L) unstretched	1.40
8090 (L) duplex aged	1.38
8090 (H) stretched	1.19
8090 (H) unstretched	1.28
8090 (H) duplex aged	1.36

TABLE IV S′ rod diameter as a function of artificial ageing temperature

Alloy and heat treatment	S′ rod diameter (mm)
8090 (L) duplex aged at 170 °C	2
8090 (L) duplex aged at 190 °C	4
8090 (H) duplex aged at 170 °C	3
8090 (H) duplex aged at 190 °C	4

significantly increased the diameter of the S′ precipitates at peak hardness. The tensile and cyclic stress–strain data in Figs 7 and 8 indicate that dislocation motion is more difficult in the alloys aged at the higher temperature. This suggests that the dislocations are more effectively impeded by the thicker S′ precipitates in the alloys aged at 190 °C.

4. Conclusions

The conclusions are as follows.

(1) Both the incorporation of a pre-ageing stretch and a 24 h natural age prior to artificial ageing were successful in homogenizing the distribution of S′ precipitates. The pre-ageing stretch provided dislocations for the S′ to precipitate upon and the natural age provided time for the formation of vacancy clusters upon which the S′ could precipitate.

(2) The precipitate sizes that formed as a result of the duplex heat treatment were dependent upon the artificial ageing temperature.

(3) A model was developed that predicted the critical diameter at which the S′ precipitates in the 8090 alloy studied would be sheared by dislocations rather than looped in the Orowan mechanism to be between 0.8 and 3.1 nm.

(4) Tensile tests and cyclic stress–strain behaviour showed that the S′ in the duplex-aged and unstretched alloys was not as effective at impeding dislocation motion as the S′ in the stretched alloys. Similarly, long and small fatigue crack behaviour showed that slip was non-homogeneous in the duplex and unstretched alloys but homogeneous in the stretched alloys. These observations are attributed to non-homogeneous distribution of S′ in the unstretched alloys and easily sheared, fine S′ precipitates in the duplex-aged materials.

(5) Artificial ageing temperature is identified as a key parameter in determining the effectiveness of duplex-ageing treatments in Al–Li alloys.

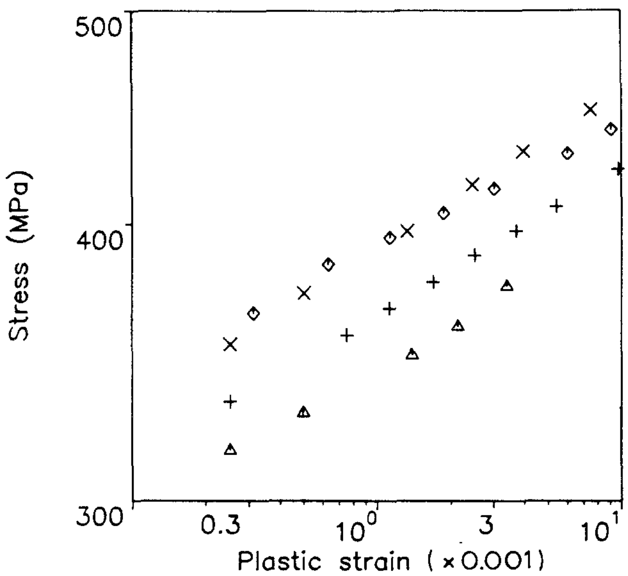

Figure 7 Comparison of monotonic tensile data for alloys duplex aged at 190 °C with those aged at 170 °C. (Δ 8090, 170 °C, + 8090 190 °C, × 8091 170 °C, ◇8091, 190 °C).

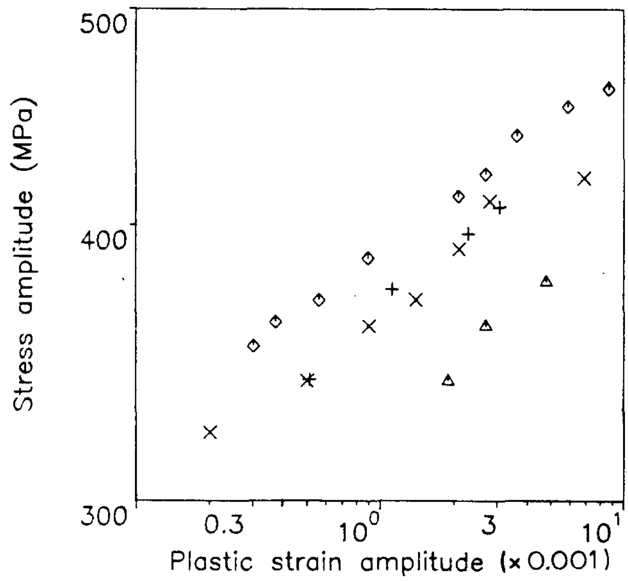

Figure 8 Comparison of cyclic stress–strain data for alloys duplex aged at 190 °C with those aged at 170 °C. (Δ8090 170 °C, + 8090 190 °C, × 8091 170 °C, ◇ 8091, 190 °C).

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