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VARIATION IN STRUCTURE AND PROPERTIES IN AN Al-Li-Cu-Mg-Zr ALLOY PRODUCED BY EXTRUSION PROCESSING

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Synopsis

The effects of extrusion processing variables on the structure and properties of an Al-Li-Cu-Mg-Zr alloy (8090 type) have been investigated. A combination of light and transmission electron microscopy have been used to characterise the as extruded microstructures and the precipitation reactions which take place on subsequent heat treatment. The corresponding mechanical properties have been determined by hardness, tensile and fracture toughness test methods. As extruded tensile properties are affected by the processing variables whilst within heat treated material the precipitation processes control the mechanical properties of the alloy. The effects of variation in heat treatment involving natural ageing and stretching on the fracture toughness are discussed in relation to the microstructural changes produced. By suitable process and heat treatment control, good combinations of strength, toughness and ductility can be obtained.

Introduction

The widespread use of aluminium alloys containing lithium has, until quite recently, been hindered because of the inadequate fracture properties which are typical of superlattice alloys in which co-planar slip and subsequent strain localisation leads to premature grain boundary failure. The recent widespread development of quaternary Al-Li-Cu-Mg alloys appears to have, at least partially, overcome this problem by introducing precipitation reactions involving the S (Al2CuMg) and T (Al2CuLi) phases in addition to the δ' (Al3Li) strengthening precipitate. The formation of the S phase concurrent with δ' precipitation has been shown to promote ductility and toughness by dispersing co-planar slip and hence producing a commercially acceptable balance of properties. In the United Kingdom LITAL A and LITAL B were developed whilst in the United States the quaternary alloys 8090, 8091, 2090 and 2091 with modified compositions have reached production.

These alloys are likely to find application in extruded product form because of the complex shapes which can readily be produced by the process. Moreover, in conventional aluminium alloys' optimisation of the process leads to the properties being significantly modified, usually resulting in improved strength and toughness. A recent study by Parsons and Sheppard indicated the extent to which processing would significantly vary the properties in an Al-Mg-Li-Cu alloy. However, the alloy had a composition quite different to those currently being pursued commercially. The work reported in this communication concerns the effect of extrusion processing on the structure and properties of an alloy encompassed by the AA 8090 specification.
Experimental Procedure

Material was supplied by the Aluminum Company of America in the form of 75mm diameter billets machined out of DC cast ingots. The composition of the alloy is given in Table I.

<table>
<thead>
<tr>
<th>Li</th>
<th>Cu</th>
<th>Mg</th>
<th>Zr</th>
<th>Fe</th>
<th>Si</th>
<th>Ti</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.50</td>
<td>1.04</td>
<td>1.11</td>
<td>0.12</td>
<td>0.04</td>
<td>0.04</td>
<td>0.01</td>
<td>bal</td>
</tr>
</tbody>
</table>

Billets were homogenised in an air circulating furnace, the lithium depleted layer being machined off before extrusion. Extrusion was performed in the direct mode in a 5MN press with a 75mm diameter container. Ram speeds utilised were in the range of 3-15 mm s\(^{-1}\) and the round bar extrusion ratio varied from 20:1 to 80:1. Initial billet temperatures were varied from 400°C to 500°C. The material could not be extruded below 350°C using a 20:1 reduction ratio and the lowest ram speed whilst the highest processing temperature was restricted to 500°C to avoid the risk of incipient melting. Heating for extrusion was by induction giving a heat-up rate of 125°C/min and the electrically heated press container was maintained at 30°C below the initial billet temperature. Unless otherwise stated, all extrusions were press quenched.

All high temperature solution treatments and low temperature ageing treatments were carried out in the air circulating furnaces. A standard solution treatment temperature of 530°C was used throughout. Extrudes made at 500°C were solutionised for 20 minutes while 40 minutes was employed for the products of lower temperature extrusion. Ageing was performed at 190°C. Heat treatments were established from age-hardening curves to give three tempers, underaged (UA), peak aged (PA) and overaged (OA). The hardness data were obtained using a Vickers diamond indenter with a 10kg load. A combination of light microscopy, using anodised surfaces and polarised light, scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were used to characterise the microstructures and the precipitation reactions which took place on subsequent heat treatment. TEM foils were electropolished by standard methods using a 30% nitric acid/70% methanol solution.

Tensile testing was performed on standard Hounsfield-13 and Hounsfied-12 test pieces using a cross head speed of 0.2mm/minute. A modified Terratek\(^4\) short rod fracture toughness test was used to assess fracture properties. This method produces excellent correlation with $K_{IC}$ values determined in accordance with ASTM method E399.

Results and Discussion

As-Cast Material and Homogenisation

In the as-cast material the phases observed within the matrix and at the grain boundaries were copper-rich. The precipitates within the matrix were found to have an average composition of 15-19%Cu, 2.5%Mg (balance Al) and some contained a fine periodic stacking fault structure indexed as cubic with a = 2.09nm resembling the Li bearing C phase recently reported\(^5\). Analysis for Li could not be made and hence these precipitates remained unspecified. Grain boundaries were decorated with an Al-Cu-Mg eutectic of average composition 22%Cu, 5.5%Mg (balance Al) and an unidentified iron rich phase (9%Fe, 5%Cu, 4%Mg, <1%Si [balance Al]) was also present in small volumes. Differential scanning calorimetry revealed a eutectic melting temperature of 547°C and almost complete dissolution of these phases was achieved by homogenising at 537°C for 24 hours.
Effect of Processing Variables on the Product Structure

For all the extrusion conditions investigated the surface region was fully recrystallised, the core essentially unrecrystallised and the mid-radius location partially recrystallised (except for material extruded below 450°C). An increase in extrusion temperature increased the volume fraction recrystallised, the grains becoming finer and more uniformly distributed. At the lower temperatures the recrystallised grains were observed to be coarser. Increasing the ram speed or the reduction ratio increased the depth of the recrystallised annulus and the core increasingly exhibited partial recrystallisation. Thus as the temperature compensated strain rate is increased so is the volume fraction recrystallised. Figure 1 indicates the structural variations observed under varying process conditions. The structures illustrated in Figure 1 were extremely resistant to further static recrystallisation remaining essentially unchanged after the solution treatment at 530°C. The only modification observed in morphology was the growth of the existing small recrystallised grains. Even when the extrudates were allowed to air cool the structures remained unchanged. This inhibition of static recrystallisation in Zr containing Al-Li alloys has previously been observed by Makin and Stobbs who attributed the phenomenon to the solute drag effect of Li on grain boundaries together with boundary pinning by Al$_3$Zr precipitates.

The present observations were a clear indication that the recrystallisation was occurring dynamically under the influence of strain during the deformation process. A consistent feature of the structures shown in Figure 1 is the banded nature of the recrystallised grains and the proximity of the nucleation points to original grain boundaries. Inspection by TEM revealed that under all extrusion conditions, notably at lower reduction ratios and ram speeds, recrystallisation was nucleated by local migration of original grain boundaries. Figure 2 is one illustration of this mechanism in which the portions of the high angle boundary limiting the bulge are pinned by the second phase particles. Selected area diffraction patterns covering the periphery of the bulge region (from A,B,C) confirm the high misorientation of its interface with the surrounding matrix. Recrystallisation by this mechanism could occur either during or immediately after deformation without any incubation period. This mechanism is responsible for most of the recrystallised grains observed within the press quenched structures. Since increasing ram speed, reduction ratio and temperature all increase the volume fraction of new grains, it seems likely that the process is dynamic; this is further supported by the fact that extrusions performed at lower temperatures do not subsequently recrystallise during the solution soak. TEM also revealed that this bulging mechanism was supplemented by a second process at higher deformation rates. TEM indicated the presence of highly misoriented subgrains adjacent to original high angle grain boundaries indicating that rotation recrystallisation may also be taking place. In this mechanism these subgrains must rotate to accommodate the inhomogeneous strain at the original boundary thus forming a new high angle boundary with both original grains. These new grains were frequently observed to contain substructures indicating the process to be dynamic and similar to that recently shown to be responsible for recrystallisation in Al-Mg alloys.

Extrudates produced at 500°C remained single phase during extrusion. However, at lower temperatures coarse (<1μm) intragranular Cu,Mg rich particles were produced as a result of the thermal exposure (Figure 3).
The Effect of Process and Heat Treatment Parameters on Tensile Properties

As-Extruded Properties

Tensile testing was performed on extrudates processed at varying temperatures and reduction ratios and a ram speed of 3mm/sec. The results are presented in Figure 4 which indicates that the tensile strength is increased by both increasing temperature and reduction ratio but ductility is relatively insensitive to the processing parameters but is least where the volume fraction of Cu,Mg rich particles is greatest and the degree of recrystallisation is least after extrusion at 400°C. The variables had a similar effect on proof stress except at the highest extrusion temperature where the properties were observed to decrease with increasing extrusion ratio. This is consistent with the increased volume fraction recrystallised observed in the microstructures. The general increase in strength with temperature and extrusion ratio is most probably due to the solutionising effect as the temperature of the emergent extrude is increased due to either increased initial thermal energy or conversion of work to heat during the process.

Heat Treated Properties

The age hardening curves for the alloy solutionised for 20 minutes at 530°C indicated that extrusion at either 500°C or 400°C resulted in the same peak hardness but the 400°C extrudate had inferior properties prior to peak. TEM investigation revealed incomplete solutionising of the Cu,Mg rich particles produced by 400°C extrusion. Increasing the solution time to 40 minutes resulted in identical behaviour in both the 400°C and 500°C extrudates; it was, therefore, adopted as the standard solution treatment for extrudes produced below 500°C.

The variation in tensile properties in the fully solutionised and aged condition (T6) is shown in Figures 5 and 6. The properties in the underaged, peak aged and overaged conditions are presented as functions of the reduction ratio and two extrusion temperatures. The properties do not appear to be extremely sensitive to the processing variables although differences can be detected; both increasing extrusion ratio and increasing temperature lead to improved elongations and small differences in tensile strength. The property differences at least for round rod extrusion, are eliminated by the ageing sequence.

In the peak aged condition, TEM indicated that both homogeneously nucleated δ' and S phases were present together with sparse heterogeneously nucleated S and T1 phase particles on sub-boundaries (Figure 7). The homogeneously distributed δ' and S gave rise to high strength (UTS = 580MPa) and reasonable ductility (= 7% el.) in the peak aged material. The observation of homogeneously nucleated S phase is consistent with previous work which indicates that as the upper end of the Lital A specification range the Cu + Mg concentration are sufficient to cause such precipitation δ. Natural ageing further promoted homogeneous S precipitation and delayed the onset of overaging in the artificially aged condition; on the other hand, stretching prior to ageing promoted heterogeneous formation of S at the expense of homogeneous precipitation (Figure 8).

Effect of Heat Treatment on the Fracture Toughness

The effect of varying heat treatment cycle on the fracture toughness of the alloy has so far only been investigated for an extrusion performed at 500°C, 20:1 and ram speed of 3mm/sec. In all cases load was applied at 90° to the extrusion direction. The variations in heat treatment involved
natural ageing and stretching and combinations of these factors summarised in the legend to Figure 9. Figure 9 also indicates the SR4 values obtained for alloys AA 2014 and AA 2024 which were tested as controls. The alloy was tested in the UA, PA and OA conditions as indicated in the figure. For all conditions the fracture toughness decreased with ageing time and was improved by the insertion of natural ageing before the 190°C treatment; the exception being the combination of natural ageing and stretching. Poor toughness values were in fact associated with any cycle which included the 2% stretch.

These results indicate the critical role of S phase precipitation in determining the mechanical properties. Property improvements in this alloy are associated with the production and refinement of a homogeneous distribution of this phase. Thus natural ageing is beneficial while stretching is detrimental. This may appear to conflict with the conclusion of Gregson and Flower that stretching is beneficial in a dilute Lital A composition. However, in that work, the only mode of S nucleation achievable in practical heat treatment times, and in the absence of natural ageing, was heterogeneous. Hence, stretching refined the distribution of S and improved the properties. In the present case, stretching encourages heterogeneous S formation at the expense of homogeneous precipitation and thus coarsens the particle dispersion. The conclusion in both cases is the same: refinement of the S dispersion results in improved strength and toughness.

Conclusions

1. An optimum homogenisation schedule was established for DC cast Al-2.5Li-1.0Cu-1.1Mg-0.12 wt%Zr alloy. Annealing for 24 hours at 537°C results in complete dissolution of the as cast phases with no sign of incipient melting.

2. Extrusion produces microstructures which vary through the extrude section: the surface was fully recrystallised whilst the core was essentially unrecrystallised. The volume fraction recrystallised increases with increasing temperature, extrusion ratio and ram speed. It is proposed that recrystallisation is dynamic and occurs by grain boundary bulging and by rotation recrystallisation.

3. The extruded structure is extremely resistant to static recrystallisation during subsequent heat treatment.

4. The as extruded tensile properties are affected by the processing variables; whilst, within the heat treated material, the mechanical properties are dominated by the precipitation processes which occur. The production of homogeneously distributed $\delta'$ and S precipitate phases results in high strength ($= 580$ MPa) and reasonable ductility ($= 7$% el.) in peak aged material.

5. The fracture toughness of the material is improved when it is subjected to natural ageing prior to artificial ageing. The improvement in properties results from the refinement of the homogeneous distribution of S phase within the microstructure.

Acknowledgements

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References


![Extrudate Grain Structure](image)

**FIGURE 1.** Extrudate Grain Structure

(a) 500°C, 20:1, extrudate surface;  (b) 400°C, 20:1, extrudate surface
(c) 500°C, 20:1, extrudate core,  (d) 500°C, 80:1, extrudate core
(e) 450°C, 20:1, extrudate core when annealed for 5 hours at 530°C under argon atmosphere.
FIGURE 2. TEM micrograph showing recrystallisation by grain boundary bulging mechanism.

FIGURE 3. Precipitation of second phase particles formed due to an initial billet temperature of 400°C; TEM micrograph of longitudinal section.
Fig. 4

AS EXTRUDED TENSILE PROPERTIES

Fig. 5

T6 TENSILE PROPERTIES
INITIAL BILLET TEMPERATURE – 400°C
Fig. 6

T6 TENSILE PROPERTIES
INITIAL BILLET TEMPERATURE - 500°C

Fig. 7. Homogeneous S precipitation within the material when solution-treated, water quenched and aged for 24 hours at 190°C; <100> Al orientation.
FIGURE 8. S precipitation within the material when solution treated, water quenched and (a) 2% stretched, (b) NA 1 month and 2% stretched, (c) NA 1 month and artificially aged for 24 hours at 190°C.

(a) and (c) (<110> Al orientation), (b) (<112> Al orientation).

SHORT ROD FRACTURE TOUGHNESS INDICATOR TEST

Fig.9