# **Ageing behaviour and toughness of silicon carbide particulate reinforced AI-Li-Cu-Mg-Zr metal matrix composites**

A. J. SHAKESHEFF

*Materials and Structures Department, Defence Research Agency, Farnborough, Hants, GU14 6TD, UK* 

The effects of lithium content on the ageing characteristic and notched tensile properties of particulate reinforced AI-Li-Cu-Mg-Zr based metal matrix composites (MMCs) have been investigated. MMC sheet containing 20 wt% silicon carbide particulate produced by a conventional powder metallurgy route aged at a similar rate as unreinforced sheet, and the highest strengths were achieved in samples containing 2-2.5 wt% Li. A proprietary processed 8090 AI-Li alloy MMC sheet aged more rapidly, however, and gave considerably higher strengths. The toughness of Al-Li-Cu-Mg-Zr MMC sheet, as indicated by the notched tensile behaviour, can be improved by reducing the lithium content albeit at the expense of strength.

# **1. Introduction**

Metal matrix composites (MMCs) consisting of aluminium alloys reinforced with silicon carbide particulate are being developed for aerospace applications owing to their higher strengths and stiffnesses compared to conventional aluminium alloys which can result in significant mass savings [1]. MMCs are currently being considered for various applications, including floor support beams, floor beam struts and instrument racks for civil and military transport aircraft, structural components for missiles, and mirrors and gimbals for satellites  $[2-4]$ . The main matrix alloy systems being evaluated are based on either A1-Cu-Mg (2124) alloy or  $Al-Li-Cu-Mg (8090)$  alloy. In some respects, 8090 alloy appears to be more attractive, since it has a 10% lower density  $(2.54 \text{ g cm}^{-3})$  and is up to 15% stiffer than 2124 alloy (density  $2.81 \text{ g cm}^{-3}$ ). Reinforcement of 8090 alloy with silicon carbide particulate offers the possibility of a further  $\approx 25\%$  increase in stiffness with only a small increase in density (e.g. 8090/20 wt % SiC has a density of  $\approx 2.66$  gcm<sup>-3</sup>). However, 8090 alloy is generally considered to exhibit lower ductility and toughness compared to conventional alloys  $\lceil 5 \rceil$  and the addition of reinforcement can have an adverse effect on these properties [6]. The current target properties for 8090 based MMC are a 0.2% proof stress of 380-420 MPa on ultimate tensile stress of 470-520 MPa, modulus of 110-120 GPa with a ductility of 6% and good toughness. 8090 MMC has also been reported to exhibit a rapid natural ageing response after solution heat treatment which may lead to fabrication difficulties [7]. Consequently improvements in A1-Li based MMCs must be

made if they are to be used in significant amounts for aerospace applications. The main objective of the work was to investigate whether optimization of the matrix alloy composition could lead to improvements in the ageing behaviour and toughness of silicon carbide particulate reinforced A1-Li-Cu-Mg-Zr alloys. To achieve this objective a range of alloy powders with lithium contents between 0 and 2.5 wt  $%$  were produced by inert gas atomization. The powders were blended with silicon carbide particulate and processed to sheet form using powder metallurgy techniques. The ageing, notched tensile behaviour and microstructures of these materials were then assessed.

# **2. Experimental procedure**

An unreinforced billet and MMC billets 115 mm long by 115 mm diameter were manufactured using powder metallurgy techniques by BP Metal Composites (now Aerospace Metal Composites). The unreinforced billet was made with aluminium alloy powder corresponding to the Al-Li-Cu-Mg-Zr  $(8090)$  alloy specification, while the MMC billets contained 20 wt % silicon carbide particulate\* (average particle size  $3 \mu m$ ) and aluminium alloy powders produced by Metalloys Ltd based on the composition of 8090 alloy, but containing varying amounts of lithium. The compositions of the alloy powders and their billet identities are given in Table I.

For the MMC billets the SiC particulate was dried overnight in a microwave oven prior to mixing with the alloy powder for 1 h in a Turbula blending machine. The powders were then processed to billet form

\* Black grade, F1200, silicon carbide particulate supplied by ESK, Germany.





<sup>a</sup> UR unreinforced alloy.

TABLE II Summary of process routes

Process route	Sheet identity	Description of process route
Unreinforced	UR 8	Powder was canned, vacuum degassed and consolidated by hot isostatic pressing
Blend and HIP	$MMC 1-5$	Blended powder was canned, vacuum degassed and consolidated by hot isostatic pressing
Blend and HP	MMC 6	Blended powder was canned, vacuum degassed and consolidated by hot pressing
Proprietary processed	MMC 7	Blended powder was proprietary processed, canned, vacuum degassed and consolidated by hot isostatic pressing

by different routes as summarized in Table II. The powders were vibratory compacted into aluminium cans, which were afterwards fitted with welded lids. The canned powders were then vacuum degassed for  $\approx$  24 h at 545 °C and sealed. MMC billets 1-5 and the unreinforced billet UR8 were consolidated by hot isostatic pressing (HIP) at  $530^{\circ}$ C using a pressure of 104 MPa. Billet MMC 6 was pre heated to  $475^{\circ}$ C and consolidated by hot pressing using a 5 MN capacity Instron press at DRA Farnborough. Pressing was done lengthwise within a closed die of diameter 125 mm to a thickness of  $\approx$  50 mm using a rate of  $2 \text{ mm s}^{-1}$ . After consolidation and removing the cans by machining the billets were forged. This was done by preheating the billets for 1 h at 510 $^{\circ}$ C, forging diametrically to 60 mm, upsetting and reforging to 60 mm with a reheat for 40 min prior to forging to a thickness of 25 mm. The forged plates were trimmed by abrasive water jet cutting to remove edge cracks and hot rolled at  $510^{\circ}$ C to a sheet thickness of 2 mm using reductions of 10-15% per pass.

Solution heat treatments (SHT) were carried out for 30 min at  $530^{\circ}$ C in an air circulating furnace followed by cold water quenching (CWQ). Samples were artificially aged at either 150 or  $170^{\circ}$ C.

Plain tensile and notched tensile [8] testpieces were machined parallel to the sheet rolling direction. Tensile testing was carried out to EN2002/1 (formerly BS18 cat2) using a transducer extensometer of gauge length 20 mm. The notch-yield ratio, i.e. the ratio of the notched tensile strength to the 0.2% proof stress,

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was used as an indicator of the toughness of the materials (see Section 3.2).

Metallographic specimens were prepared by grinding with carborundum papers, polishing to  $1/4 \mu m$ diamond using napless cloths and finally polishing with a proprietary alumina based suspension.

Thin foils for transmission electron microscopy were prepared from 5 mm discs punched out from sheet specimens which had been ground to within 0.2 mm of the sheet mid-thickness using carborundum papers. The discs were electropolished in a solution containing 30 vol % nitric acid and 70 vol % methanol at  $-30^{\circ}$ C using a twin jet polishing machine. The foils were examined in a Jeol 200CX transmission electron microscope.

# **3. Results**

### 3.1. Effects of ageing time and lithium content on tensile properties

The effects of ageing temperature on the tensile properties of unreinforced 8090 sheet (UR 8) and MMC 6 are shown in Fig. 1. The 0.2% proof stresses and the tensile strengths of the MMC sheet after ageing at 150 and  $170^{\circ}$ C were similar and increased during ageing reaching values of  $\approx 340$  and  $\approx 470$  MPa, respectively, after 24–48 h, while the ductility decreased from 6 to 4%. The unreinforced sheet exhibited a similar ageing behaviour, but in the peak aged condition the 0.2% proof stress and the tensile strength were lower by 20 and 45-60 MPa, respectively, with the modulus reduced from  $\approx 100$  to  $\approx 80$  GPa. The addition of



*Figure 1* Effect of ageing time and temperature on the tensile properties of unreinforced 8090 alloy and 8090 alloy reinforced with 20 wt % SiC<sub>p</sub>: for MMC 6: at 150 °C ageing temperature, ( $\circ$ ) 0.2% proof stress and  $(\bullet)$  tensile stress; at 170 °C ageing temperature, ( $\Box$ ) 0.2% proof stress and  $(\blacksquare)$  tensile stress. For unenforced alloy MMC 8: at 170 °C ageing temperature, ( $\triangle$ ) 0.2% proof stress and ( $\triangle$ ) tensile stress.

silicon carbide did not result in significant changes in ductility. 600

The effects of ageing at  $170^{\circ}$ C on the tensile properties of MMC sheet with lithium contents in the range 500 0-2.4 wt% (MMC 1-5) are shown in Fig. 2. The  $\frac{1}{2}$  400 moduli values were generally unaffected by lithium content and were in the range 100-110 GPa. However, the lithium additions had a marked effect  $\frac{1}{2}$  300 moduli values were generally unaffected by lithium content and were in the range  $100-110$  GPa. How-  $\frac{90}{60}$  300 ever, the lithium additions had a marked effect  $\frac{1}{6}$  200 on strength. The lowest strengths were measured in the lithium free sheet  $(MMC 1)$  with maximum values 100 of 0.2% proof stress and tensile strength of 271 and 354 MPa, respectively, after 168 h ageing. Increasing the lithium content to  $1-1.5$  wt % (MMC 2 and 3) resulted in similar ageing behaviour, with the 0.2% proof stress continually increasing while the tensile strength reached a plateau of 385-400 MPa after 24 h. Increasing the lithium content further to  $> 2\%$ (MMC 4 and 5) resulted in significantly higher strengths with values of  $0.2\%$  proof stress of  $500$ 375-420 MPa and tensile strengths of 490-530 MPa in the peak aged condition. However, in this condition  $\frac{1}{\pi}$  400 ductilities were significantly reduced from about 10 to 4% elongation.<br>MMC 7, produced by the proprietary process, ex- $\frac{30}{8}$  300

hibited a more rapid ageing response with the peak strength reached after 3 h, Fig. 3. In this condition  $\frac{5}{8}$  200 the tensile strength was almost 80 MPa higher than the peak strength reached by MMC produced by the  $\frac{100}{6}$  100 blend only route (MMC 5).

#### 3.2. Effects of lithium content on notched tensile properties

Comparison of the notched tensile data for the MMCs 1–5 containing  $0-2.5$  wt % lithium revealed a sigmoidal relationship between 0.2% proof stress and notch yield ratio, Fig. 4. An approximately linear portion occurred over the central 0.2% proof stress range 200-345 MPa, but outside this range the notch yield ratios levelled out. In general high lithium contents tended to be associated with low notch yield ratios.



*Figure 2* Effect of lithium content and ageing time at 170 °C on the tensile properties of sheet containing 20 wt %  $\rm{SiC_p}$  produced by the blend and HIP route. MMC 1,0 wt % Li:  $(\frac{1}{2})$  0.2% proof stress, ( $\star$ ) tensile stress. For MMC 2, 1 wt % Li: ( $\square$ ) 0.2% proof stress, ( $\blacksquare$ ) tensile stress. For MMC 3, 1.5 wt % Li: ( $\triangle$ ) 0.2% proof stress, ( $\blacktriangle$ ) tensile stress. For MMC 4, 2 wt % Li: ( $\bigcirc$ ) 0.2% proof stress, ( $\blacklozenge$ ) tensile stress. For MMC 5, 2.4 wt % Li:  $\circlearrowright$  0.2% proof stress,  $\blacklozenge$ tensile stress.



*Figure 3* Effect of ageing time at 170 °C on the tensile properties of proprietary processed MMC 17 sheet: ( $\circlearrowright$ ) 0.2% proof stress, ( $\bullet$ ) tensile stress.



*Figure 4* Effect of process route and lithium content on the notched tensile behaviour of MMC sheet: MMC:  $(\frac{\pi}{4})$  1, 0 wt % Li; ( $\Box$ ) 2, 1 wt % Li; ( $\triangle$ ) 3, 1.5 wt % Li; ( $\bigcirc$ ) 4, 2 wt % Li; ( $\bigcirc$ ) 5, 2.4 wt % Li; (◆) 7, 2.4 wt % Li.

It was only possible to measure the notch yield ratios for the proprietary MMC 7 over a limited strength range, with the values ranging from 0.86 to 1.21. However, these ratios were associated with higher 0.2% proof stresses indicating that the material may be tougher than the blend only route.

## 3.3. Optical and transmission electron microscopy

The microstructures of the unreinforced sheet and MMC sheets produced by the blended only and proprietary process routes after SHT and ageing for 24 h at  $170^{\circ}$ C are compared in Fig. 5a–c. The microstructure of the unreinforced sheet consisted of large grains  $\approx 100-200$  µm in size, whereas the grain structures of the MMC sheets could not be distinguished by optical microscopy. However, transmission electron microscopy revealed that the MMC sheets contained sub grains typically  $0.5-1.5 \mu m$  in size, although complete characterization of the grain structure was not achieved. The SiC distributions in MMCs 1-6 were similar and fairly uniformly distributed in the *L-ST*  and *T-ST* planes; while in the *L=T* plane, areas devoid of SiC were apparent. In the proprietary processed sheet, MMC 7, the SiC generally appeared finer in size and more uniformly distributed.

After ageing for 120 h at 170 $^{\circ}$ C the unreinforced sheet (UR 8) contained well developed homogeneous precipitation of  $\delta'(Al_3Li)$ , but only sparse amounts of  $S'(A)$ <sub>2</sub>CuMg) precipitation. The lithium content of the MMC sheet had a marked effect on precipitation when aged at  $170\,^{\circ}\text{C}$  to the peak strength condition, i.e. 168 h. Well developed homogeneous 6' precipitation and sparse amounts of heterogeneous S' phase were evident in sheet containing 2.4 wt  $\%$  Li (MMC 5) produced by the blend only and proprietary processed MMC sheet (MMC 7), Fig. 6a–b. In a sheet containing 0 to 1.5 wt % lithium (MMCs  $1-3$ ), dense mainly homogeneous S' precipitation was observed, but  $\delta'$ precipitation was absent, Fig. 6c-d.

# **4. Discussion**

The MMC sheet produced by the blend only routes aged at a similar rate to the unreinforced 8090 sheet, but with higher strengths and moduli values. The proprietary processed sheet (MMC 7), gave the highest strengths and aged more rapidly reaching the peak strength condition after 3 h, which is consistent with earlier work by Hunt et al.  $[7]$  and Miller et al.  $[9]$ . Hunt *et al.* [7] reported that  $\delta'$  precipitation was finer in proprietary processed 8090 MMC than in unreinforced 8090 alloy sheet produced by either ingot or powder metallurgy routes. The strengths and ductilities of the MMC sheet were influenced by lithium content. Sheet containing 2-2.5 wt % lithium produced the highest strengths as a result of homogeneous  $\delta'$ precipitation. In these alloys S' precipitation was relatively sparse and heterogeneous which may be due to the reduced number of nucleation sites available (such as dislocation loops and helixes) owing to the strong bonding between the lithium atoms and vacancies inhibiting vacancy condensation during quenching  $[10]$ . The lower strengths obtained when the lithium content was reduced to  $\langle 1.5 \text{ wt\%} \rangle$  were due to the absence of  $\delta'$  precipitation and in spite of dense homogeneous precipitation of S' produced as a result of the increased free vacancy concentration. Studies on proprietary processed 8090 MMC sheet [11] also showed that the ageing behaviour and strength was







*Figure 5* Microstructure of 8090 alloy and 8090 MMC sheet after solution heat treatment at 530 °C, CWQ and ageing for 24 h at  $170 \degree C$ : (a) grain structure of unreinforced sheet, (b) SiC distribution in blend and HIP MMC sheet, (c) SiC distribution in proprietary processed sheet.



*Figure 6* Effect of lithium content on fine precipitation in MMC sheet: (a) and (b)  $\delta'$  and S' precipitation in sheet containing 2.4 wt % lithium, (c) S' precipitation in sheet containing 1.5 wt % lithium (d) S' precipitation in sheet containing 0 wt % lithium.

influenced by lithium content; the highest strengths were obtained in sheet containing 2.5 wt % lithium with a marked reduction in strength when the lithium content was reduced to 2 wt %. Both of these alloys aged faster and gave substantially higher proof stresses and tensile strengths than alloys containing between 0 and 1.5 wt % lithium.

In Al–Cu based MMCs the accelerated ageing behaviour has been attributed to dislocation enhanced precipitation as a result of thermal mismatch between the reinforcement and the matrix alloy on quenching [12]. However, in the present study the rate of ageing in 8090 MMC containing  $0-1.5$  wt % lithium was no faster than the unreinforced sheet, even though dense homogeneous S' precipitation occurred. This suggests accelerated ageing behaviour cannot be explained by enhanced precipitation. It is more likely to be due to thermal mismatch strains enhancing either the diffusion rate of solute atoms  $\lceil 13 \rceil$  or the amount of solution strengthening [7].

The notch-yield ratio has been reported to obey an inverse linear relationship when plotted against the 0.2% proof stress, giving an indication of the relative toughness of high strength aluminium alloys [14]. However, in the present study the lithium content of blended only MMC sheet had a marked effect on the notch-yield ratio, resulting in an almost sigmoidal relationship between the 0.2% proof stress and notch yield ratio. At high proof stresses it was noticeable that the proprietary processed sheet (MMC 7) gave higher ratios indicating improved toughness.

Similar behaviour was also observed for A1-Cu and A1-Cu-Mg based MMCs [15] produced by the blend and HIP route, although they gave similar notch-yield ratios in the range  $0.8-1.2$  at higher  $0.2\%$ proof stress values indicating better toughness, Fig. 7. The notch-yield ratio and hence toughness of MMCs is probably limited by either void nucleation associated with matrix grain boundary oxides, matrix-SiC particle decohesion or SiC particle cracking [16]. It is



*Figure 7* Comparison of the notch-tensile behaviour of  $(\triangle)$  Al-Cu and Al-Cu-Mg  $[15]$  and  $(\square)$  Al-Li-Cu-Mg based MMCs.

clear that there is some scope for modifying the toughness of 8090 alloy based MMCs by changes in alloy composition, albeit at the expense of strength. However, the use of the proprietary process route (which generally gives a higher notch-yield ratio for a given strength) in conjunction with a reduced lithium content could lead to an MMC with an attractive balance between toughness and strength.

#### **5. Conclusions**

1. MMC sheets processed by the blend only route containing  $2-2.5$  wt % lithium aged at a similar rate as unreinforced 8090 alloy sheet, but exhibited higher strengths and moduli values in the peak aged condition.

2. Proprietary processed MMC sheet (MMC 7) containing 2.4 wt % lithium aged more rapidly than unreinforced sheet and gave the highest strengths.

3. MMC sheet containing between  $2-2.5$  wt % lithium exhibited the highest strengths owing to the presence of homogeneous  $\delta'$  precipitation. Precipitation of S' was sparse owing to the high lithium-to-vacancy binding energy which suppresses vacancy condensation and hence reduces the number of sites on which S'nucleates. The reduced strengths obtained when the lithium content was in the range  $1-1.5$  wt % was due to the absence of  $\delta'$  precipitation in spite of the fact that homogeneous precipitation of S' phase is increased.

4. The notched tensile properties obtained for 8090 based MMCs containing different amounts of lithium exhibited a sigmoidal relation between 0.2% proof stress and notch-yield ratio, indicating that toughness can be improved by reducing the lithium content although this would be at the expense of strength.

5. The notch-yield ratios measured for the proprietary processed MMC 7 sheet were higher at  $0.2\%$  proof stresses  $> 350$  MPa, indicating increased toughness in comparison with sheet made by the blend only route.

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