

Effects of microstructure on optimum heat treatment conditions in metal–matrix composites

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It is well known that the microstructure of metal–matrix composite materials is significantly different from that of the unreinforced matrix. Heat treatments which optimized strength values in the matrix therefore, no longer do so in the composite. It is thus beneficial to find these variations in the heat treatment process, and to pinpoint the microstructural mechanisms responsible. It is known that, in addition to a higher dislocation density, the composite also has a very fine grain size and many whisker interfacial nucleation sites. This study has found that the time required for solutionizing appears to be much shorter in the composite primarily due to the very small grain size. It was also observed that artificial ageing was not very effective in the composite since the precipitates nucleated and grew on the whisker interfaces, therefore, only natural ageing was necessary to achieve peak tensile and yield strengths.

1. Introduction

An area of fundamental importance in metal–matrix composite (MMC) research is the study of response of the composite to heat treatment conditions. Many investigations on whisker-reinforced MMCs have utilized heat treatment conditions which were devised for unreinforced matrix material [1–4]. It is well known, however, that in order to obtain optimum properties from the composite, variations in heat treatment conditions are required due to the microstructural differences between the composite and the unreinforced matrix. For example it had been found by Christman and Suresh [5] that the time for peak artificial ageing in their AA2124–SiC whisker composite was smaller than that in the matrix by a factor of 3. Likewise, Nieh and Karlak [6] had shown that reinforcing B_4C particles in AA6061 reduced the peak ageing time by a factor of 3. Similar results were also found in a theoretical investigation by Dutta and Bourell [7].

The more rapid artificial ageing kinetics in composites had been attributed to a higher diffusion rate resulting from a higher dislocation density. This high dislocation density was, in turn, due to the 5:1 difference in thermal expansion coefficients of Al and SiC, which was shown by Vogelsang *et al.* [8]. Christman and Suresh [5] have found dislocation densities as high as $5 \times 10^{14} \text{ m}^{-2}$ in an AA2124 13 vol % SiCw composite, as compared to $5 \times 10^{13} \text{ m}^{-2}$ found in unreinforced AA2124.

Other microstructural differences, however, also exist between the composite and the unreinforced matrix which affect the results of heat treatments. In order to fully utilize the potential of whisker reinforced composites it is necessary to develop an

understanding of the microstructural mechanisms that control the heat treatment response for these systems. It was therefore the objective of this study to investigate the effects of composite microstructure on the heat treatment conditions in a AA2124 15 vol % SiC whisker composite.

2. Experimental procedure

2.1. Material

A rolled sheet of AA2124 (SXA24E) reinforced with 15 vol % SiC whiskers. This sheet was hot rolled at a temperature of 438 °C to a thickness of 1.8 mm (0.071 inches); approximately an 80% reduction. The material was solutionized at 495 °C between passes to prevent fracturing. The last pass was performed at room temperature.

2.2. Tensile tests

Tensile bars of length 7.5 cm were cut from the material. The centre 5 cm of these bars were rounded on the lathe with 600 grit SiC paper. These samples were tested using an Instron tensile testing machine at an initial strain rate of 0.00067 s^{-1} .

2.3. Microstructural studies

The cross-section of metallographic samples were prepared using conventional polishing methods followed by a short period of ion milling (about 3 h) at a voltage of 4 keV and a current of 0.5 mA. With this method, a clean new surface is exposed in which whiskers, voids, precipitates, and in some instances even grain boundaries could be seen clearly on the SEM.

3. Results

3.1. Background

Earlier work had shown that hot rolling significantly reduced the tensile strength of the peak aged AA2124 SiCw composite by overaging the matrix [9]. Large Al_2Cu precipitates (3 to 5 μm in diameter) grew during the brief cooling period after hot rolling. It was also noted that after a T6 heat treatment (1 h at 500 °C, cold water quench, + 8 h artificial ageing at 150 °C, a practice suggested by Advanced Composite Materials Corporation) the tensile strength was regained. This recovery in tensile strength was determined to be due to solutionizing of the large precipitates which nucleated premature failure.

3.2. Solutionizing results

Solutionizing experiments were performed to find the minimum time necessary to dissolve the large precipitates. As-rolled material was solutionized at 500 °C for different periods of time, room temperature aged, and then tensile tested. The solutionizing time was taken to be the time between placing the samples in the heated air furnace and the time of removal and immediate cold water quenching. Fig. 1 shows the graph of tensile and 0.2% yield strength plotted against solutionizing time for the rolled AA2124 15 vol % SiCw composite. It can be seen that the precipitates were dissolved and the strength regained in less than 1.5 min of solutionizing time, as was also confirmed by microscopic examination. This appears to be somewhat faster than in unreinforced aluminium since the "Metals Handbook" [10] suggested that an AA2024 sheet of thickness 1.8 mm should be solutionized at a temperature of 495 °C for a time of 35 to 45 min in an air furnace or for a time of 25 to 35 min in a salt bath to fully solutionize the precipitates.

3.3. Microstructural observations

Fig. 2 is an SEM micrograph oriented parallel to the whiskers showing the precipitates, whisker ends, and the grains in the as-received composite. The grain size was measured using the mean intercept length method [11]. In this composite the mean intercept length was

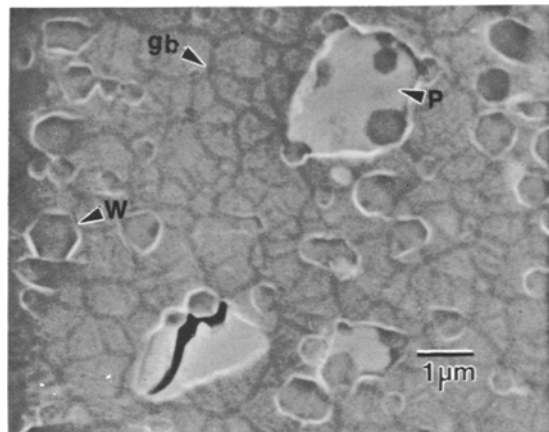


Figure 2 SEM micrograph parallel to whiskers, showing the grain size, the whisker ends and the precipitates.

measured to be 0.5 μm . These grain sizes were also confirmed with the TEM.

3.4. Ageing of the composite

Experiments were also carried out to investigate the ageing kinetics of the composite. Natural ageing and artificial ageing were studied. Fig. 3 shows 0.2% yield and tensile strength plotted against room temperature ageing time in the AA2124–SiCw system. This can be compared to Fig. 4, a graph of yield and tensile strength plotted against artificial ageing time in the same material. It was observed that natural ageing for over 24 h gave yield and tensile strength values very similar to those of material which had been artificially aged at 150 °C followed by 24 h of room temperature ageing. These results are in contrast to those of unreinforced AA2014, where peak artificial ageing results in yield [12] and tensile [13] strengths quite a bit higher than those obtained from natural ageing, as can be seen in Table I.

Although natural ageing and artificial ageing resulted in similar strength values in the composite, the ductility values were slightly different. For three samples natural ageing resulted in an average strain to

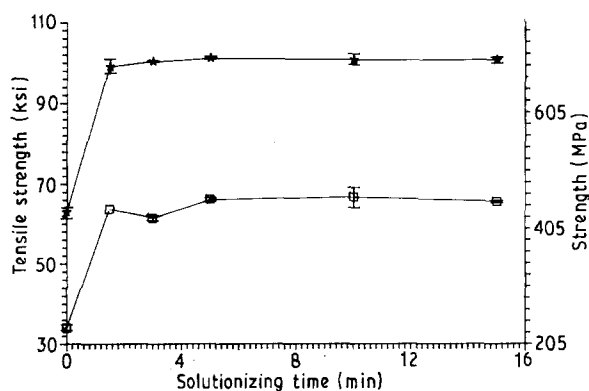


Figure 1 Tensile (★) and 0.2% yield strength (□) plotted against solutionizing time for hot rolled AA2124 15 vol % SiCw composite. Samples were solutionized at 500 °C, CWQ, followed by 24 h natural ageing.

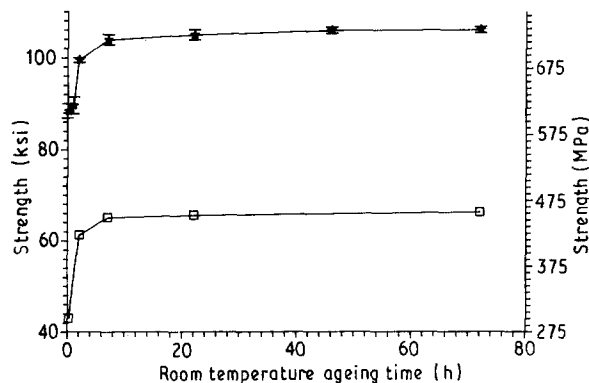


Figure 3 Tensile (★) and 0.2% yield strength (□) plotted against room temperature ageing time for the AA2124 15 vol % SiCw composite.

TABLE I Peak yield and tensile strengths for unreinforced AA2014 and AA2124 15% SiCw composites after natural ageing and artificial ageing

Material	Ageing condition	Peak 0.2% yield strength		Peak tensile strength	
		(ksi)	(MPa)	(ksi)	(MPa)
AA2014 (12, 13)	Natural aged	42	290	60.9	420
AA2014 (12, 13)	Aged at 150 °C	63	435	68.2	470
AA2124 15% SiCw	Natural aged	66.2	456	106	733
AA2124 15% SiCw	Aged at 150 °C	72.8	502	105	724

failure of 2.6% compared to 3.4% from artificial ageing. On the other hand, the ductility of unreinforced matrix material reacted in the opposite manner. The strain to failure of natural aged material was 22% compared to 10% in the artificially aged material [13].

4. Discussion

The solutionizing experiments appeared to show that the solutionizing rate was more rapid in the AA2124 15 vol % SiCw composite than in the unreinforced matrix. This effect seemed to be due to the very small grain size observed. Generally the recrystallized grain size in aluminium sheet products is greater than 25 μm . Jarry *et al.* [14] have, however, found that the introduction of ceramic whiskers or particles induces a much smaller grain size of about 1 μm , an effect attributed to the mean spacing of reinforcements. The fine grain size observed in the composite results in more grain boundary area, thus the amount of material allowed to diffuse rapidly along the grain boundaries would be increased, hence, the time necessary to solutionize the material would be decreased.

In order to better show the affect of grain size on diffusivity, a more quantitative analysis is performed. It is well known that the total diffusion in a material can be written as [15]

$$D = D_1 + g_d D_d + g_{gb} D_{gb} \quad (1)$$

where D_1 is the diffusion rate in the lattice, g_d is the fraction of atoms in the dislocation core, D_d is the diffusion rate in the core, g_{gb} is the fraction of atoms in the grain boundary, and D_{gb} is the diffusion rate in the grain boundary.

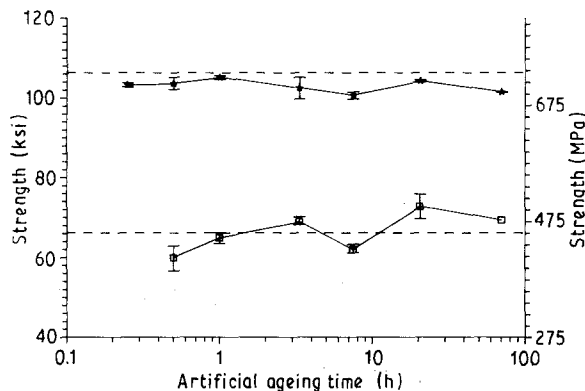


Figure 4 Tensile (\star) and 0.2% yield strength (\square) plotted against ageing time at 150 °C. Artificial ageing was followed by 24 h at room temperature (--- natural aged only).

The values of g_d and g_{gb} obviously depend on the dislocation density and the grain size in the material. As stated earlier the dislocation density in a similar composite was found to be of the order of $5 \times 10^{14} \text{ m}^{-2}$ [15]. This dislocation density gave a value for g_d of 0.0005, assuming the dislocation core has a cross-sectional area of 1 nm^2 [16]. A model using cubic grains 0.5 μm on edge with a grain boundary thickness of 3 atoms [17], gave a value for g_{gb} of 0.007.

The diffusion constants were found to be the following

lattice diffusion [18],

$$Q = 32.3 \text{ kcal mol}^{-1}$$

$$D_0 = 0.647 \text{ cm}^2 \text{ s}^{-1}$$

dislocation diffusion [19],

$$Q = 19.6 \text{ kcal mol}^{-1}$$

$$D_0 = 0.028 \text{ cm}^2 \text{ s}^{-1}$$

grain boundary diffusion (estimate) [16]

$$Q \approx 16 \text{ kcal mol}^{-1}$$

$$D_0 \approx 0.03 \text{ cm}^2 \text{ s}^{-1}$$

Fig. 5 shows the diffusivity (gD) plotted against temperature results for these values. It is apparent that the very small grain size in the composite leads to grain boundary controlled diffusion even at high temperatures.

With an estimate of the spacing of the precipitates, calculations could be made in order to get an approximation of the time it should take the precipitates to be dissolved in the material. The precipitates in the over-aged material could be seen using backscattered electron imaging in the compositional mode. Fig. 6 shows these precipitates in as-rolled composite material. The precipitates are approximately 3 to 5 μm in diameter

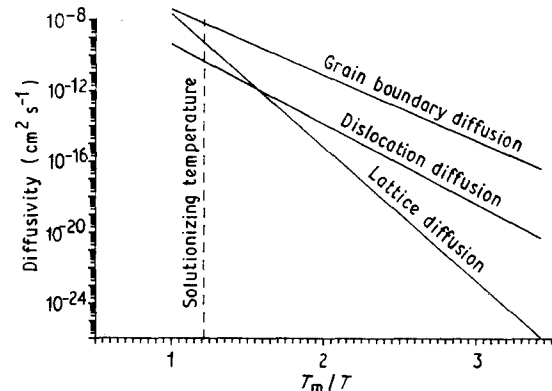


Figure 5 Diffusivity of copper in aluminium at various temperatures, assuming a grain size of 0.5 μm and a dislocation density of $5 \times 10^{14} \text{ m}^{-2}$.

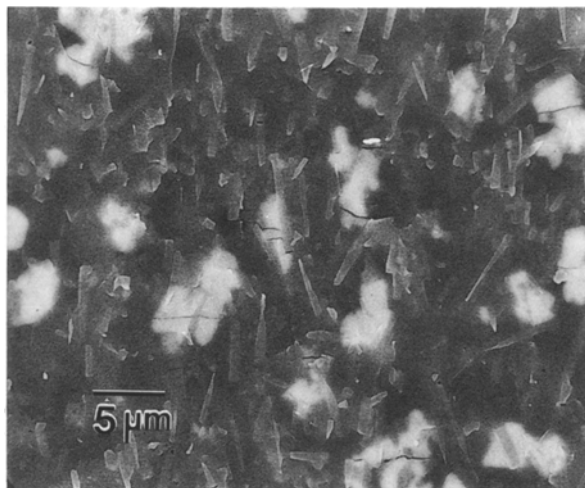


Figure 6 SEM backscattered micrograph showing the size and spacing of the large precipitates.

with a spacing of about 10 μm . The precipitates should, therefore, be dispersed in roughly the amount of time it takes for copper to diffuse a mean distance of about 5 μm at the solutionizing temperature. This distance can be estimated with the equation [20]

$$d \approx (Dt)^{1/2} \quad (2)$$

where d is the mean diffusion distance, D the diffusivity, and t the time allowed for diffusion. With grain boundary controlled diffusion it should therefore theoretically take only about 1 min to dissolve the precipitates at 500 $^{\circ}\text{C}$, which agrees well with the earlier observations. With self diffusion it would theoretically take about 7.5 min, although this could not be checked since unreinforced SXA24E alloy was not available. It should also be noticed that since the grain size (0.5 μm) was much smaller than the precipitate size (3 to 5 μm), many grain boundaries intersected the large precipitates (as can be seen in Fig. 2). Grain boundary diffusion would thus indeed have a significant effect on the solutionizing rate of the precipitates.

The ageing experiments (Table I) showed that peak natural ageing and peak artificial ageing gave virtually identical tensile and yield strength values in the composite material. Whereas in unreinforced aluminium alloys, peak artificial ageing was found to give much higher strength values than natural ageing [12, 13]. Artificial ageing thus did not appear to be as effective in the composite material. This effect appeared to be a result of precipitate nucleation on the whiskers. In unreinforced aluminium-copper alloys, ageing at 150 $^{\circ}\text{C}$ results in the growth of θ'' precipitates, which are similar in shape to GP zones only larger [21]. GP zones are described as disc-like copper-rich regions about 9 nm in diameter that homogeneously nucleate and grow parallel to the $\{100\}$ planes [22]. In the composite material aged at 150 $^{\circ}\text{C}$, however, precipitates appeared to heterogeneously nucleate and grow at the whisker interfaces, as can be seen in Fig. 7. This interfacial nucleation may have had an adverse effect on strength by embrittling the interfacial region. In addition, the depletion of copper in solution would prevent the formation of many θ'' precipitates, resulting in reduced precipitate strengthening. This reduced precipitate strengthening in the T6 material may have also been responsible for the slightly higher ductility found in this material, as compared to the naturally aged material.

Although others have found that artificial ageing was more rapid in composites than in unreinforced material [5-7], this does not imply that room temperature ageing would be faster. In fact, it can be seen in Fig. 8 that the rate of room temperature strengthening was nearly identical for the composite and unreinforced AA2024 [23]. Ageing done at temperatures near or below 100 $^{\circ}\text{C}$ strengthens the alloy through the formation of GP zones [24]. Since these GP zones nucleate homogeneously and are very small and close together, their rate of growth will not depend on the grain size or the dislocation density of the composite. One would thus expect the rate of room temperature

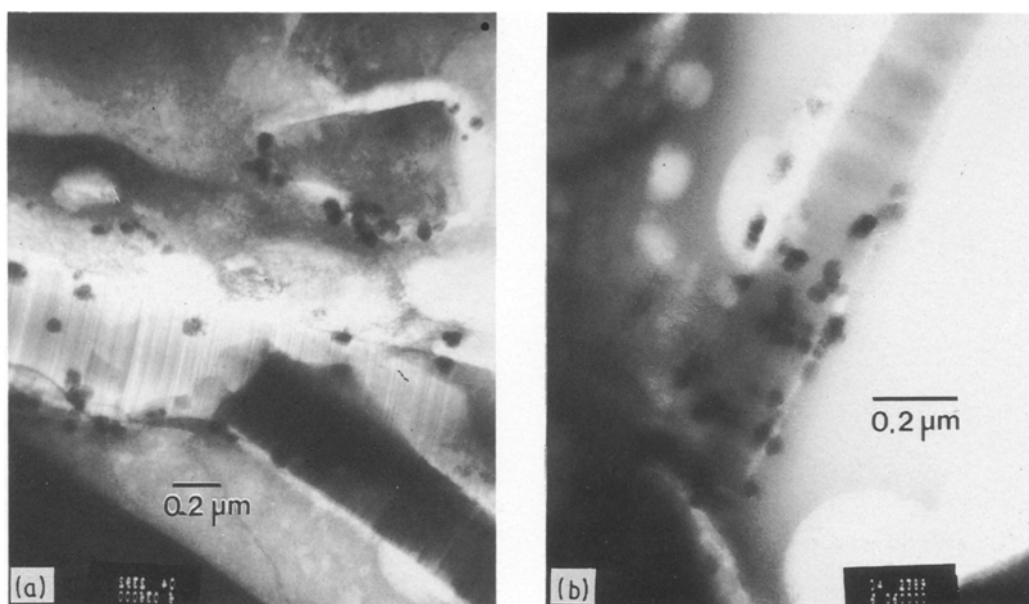


Figure 7 Small spherical precipitates at the whisker interfaces in the material aged at 150 $^{\circ}\text{C}$ for 8 h.

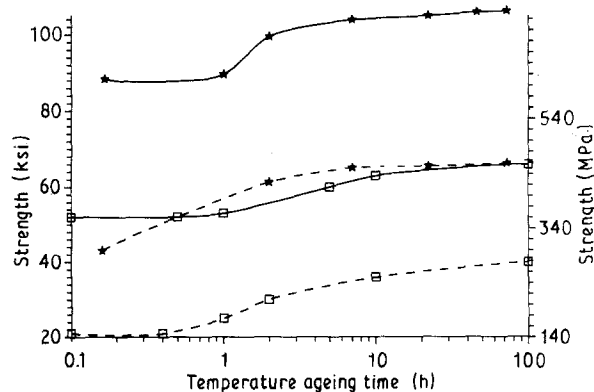


Figure 8 Rate of room temperature strengthening in the reinforced and the unreinforced matrix material. (★—★ 2124 15% SiCw tensile strength, ★---★ 2124 15% SiCw 0.2% yield strength, □—□ 2024 unreinforced tensile strength, □---□ 2024 unreinforced yield strength).

strengthening to be very similar in both the reinforced and unreinforced aluminium alloy, as was shown.

5. Conclusions

Heat treatment conditions appear to be different in metal-matrix composites than in the matrix alloys alone. This study has found that the time necessary for solutionizing appeared to be much shorter in the composite primarily due to a very small grain size. The precipitates were completely dissolved in less than 1.5 min at 500 °C. The normally applied T6 solutionizing time of 1 h is not, therefore, necessary and much time and energy can be saved.

It was also observed that artificial ageing was not very effective in the AA2124 15 vol % SiCw composite since the precipitates nucleated and grew on the whisker interfaces. Only natural ageing was, therefore, necessary to get peak tensile and yield strength values, although a slight decrease in ductility was noticed. The rate of room temperature ageing, however, was observed to be nearly identical in both the composite and the unreinforced alloy.

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