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Effect of fibre–matrix–binder interactions on the matrix composition and age-hardening behaviour of 6061-based MMCs

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Reactions between magnesium, alumina fibre and silica binder, during the manufacture of 6061 metal matrix composite (MMC) by the pressure infiltration technique, have been investigated for their effect on the structure, composition and age-hardening response of the MMC with increasing infiltration distance. The structure and composition were examined using optical and scanning electron microscopy, and electron probe microanalysis. The age-hardening behaviour, of both the MMC and unreinforced alloy, was determined using hardness measurements. There was a progressive depletion of magnesium in the MMC with increasing infiltration distance, which was particularly marked when the silica binder content exceeded 1 wt % (in a 20% V_f preform). This has been explained in terms of a reaction which results in the formation of an oxide at the fibre/matrix interface and a release of silicon into the matrix. The depletion of magnesium was associated with a reduction in the age-hardening response of the MMC, consistent with predicted behaviour based on the Al–Mg_2Si pseudo-binary phase diagram. In spite of these effects, the overall ageing behaviour of the MMC was enhanced compared with the unreinforced alloy, showing both higher peak-aged hardnesses and enhanced ageing kinetics, particularly at lower ageing temperatures.

1. Introduction

When metal matrix composites (MMC) are fabricated with heat-treatable aluminium alloy matrices, similar ageing characteristics might be expected for both composite and unreinforced material, after allowing for the strengthening increment produced by the reinforcement employed in the composite. However, in practice these composites do not always realize their potential strengths and can even exhibit impaired strengthening when compared with the unreinforced alloy. The kinetics of ageing can also be different and both enhanced and retarded behaviour has been exhibited in different MMC systems.

Some aspects of the ageing behaviour of MMC can be explained by treating the reinforcement as an inert object of a specific shape, size and volume fraction but of different thermal expansion coefficient to the surrounding matrix [1]. In such models the large thermal strain produced in the matrix around the reinforcement is accommodated by either elastic or plastic deformation [2]. Either process can result in a modified response during ageing although the latter process appears to be the most important, in which the ageing behaviour is affected by the increased dislocation densities in the matrix around the reinforcement. It is also important to consider the chemical properties of the reinforcement and matrix and the possible reactions which can occur between these two components of the composite [3, 4]. Such reactions could effectively remove chemical species from the matrix by producing reaction products which do not dissolve during solution treatment. These reactions are most severe when the alloy is molten and consequently they are also likely to be influenced by the particular manufacturing process employed to produce the MMC [5].

In the pressure infiltration method for producing MMC a preform consisting of a fibre array is infiltrated by a superheated molten metal. The contact time between the melt and the reinforcement is relatively short due to the rapid solidification of the melt (typically about 1 min). Longer contact times are usual in other methods of MMC manufacture, for example contact times of about 15 min are used for the compocasting technique (at 600/650 °C) [4]. However, in the pressure infiltration technique the alloy may also be several hundred degrees above its upper melting temperature at the onset of infiltration increasing the possibility for reactions between the melt and reinforcement. In addition, the preforms usually contain a reagent such as colloidal or precipitated silica (at a concentration of approximately 2–5 wt % reinforcement) to mechanically bind the fibre array. There exists, therefore, the possibility of complex reactions between the metal matrix, binder and reinforcement. Evidence of matrix–reinforcement and matrix–reinforcement–binder reactions has been shown in several investigations on MMC in particular those based on aluminium alloys containing magnesium [4–6]. However, there is some inconsistency in these investigations as to the effect of such reactions on the ageing behaviour of MMCs when the matrix alloy contains magnesium as a constituent of an age-hardening phase. One investigation on 6061 MMC has shown an impaired hardening response, compared with the unreinforced alloy [7]. However, two other similar investigations have suggested that the depletion of matrix magnesium was not sufficient to affect the normal age-hardening behaviour of the composite [5, 8], provided the magnesium content of the alloy was sufficiently high [8].

The present work was undertaken to address these inconsistencies. The interaction effects present in a 6061 matrix MMC have been investigated with the aim of identifying their influence on the ageing behaviour.

2. Experimental procedure

2.1. Composite fabrication

The MMC employed in this work was based exclusively on a 6061 matrix (nominally Al–1% Mg–0.6%Si) reinforced with Saffil fibres, of approximate composition 95% δ-alumina and 5% silica. Two types of preform were used, one of which was commercially available and the other was manufactured in house by a standard technique [9]. Both types contained nominal fibre fractions of 20%, V_f. The preforms manufactured in-house contained variable silica binder contents which ranged from 0%–5% as shown in Table I. No details of silica content were available for the commercially supplied preforms but these were expected to lie in the range 0%–5%. The table also shows the preform thickness after infiltration and the resulting fibre fractions of the MMC.

The preforms were infiltrated under metallostatic pressure in a preheated cylindrical die as described previously [7, 10], using a melt temperature between 950 and 1000 °C, and a die temperature between 350 and 400 °C. All the MMCs were sound, with little evidence of porosity and there was little or no crushing of the preforms during infiltration except for the cast containing 0% silica (which was 20 mm thick prior to infiltration).

2.2. Ageing measurements

Hardness specimens (~ 10 mm × 10 mm × 30 mm) were cut parallel to the axis of the cylindrical casting, so that they contained both the reinforced and unreinforced alloy in a single specimen. The solution treatment employed for the specimens was 550 °C for 1 h. After solution treatment, the specimens were first water quenched and then transferred to liquid nitrogen prior to ageing. Isothermal ageing was performed at temperatures of 140, 160 and 178 °C.

Hardness was characterized by Vickers Hardness, H_v, using a 10 kg load. Approximately ten regularly spaced readings were taken over the full thickness of the MMC. Approximately four readings were taken in the unreinforced region commencing at the interface with the MMC. These two data sets were then averaged to construct the appropriate ageing curves. The specimens used to produce each ageing curve were all taken from the same cast and a separate specimen was used for each ageing time. Although this latter procedure produced some specimen to specimen scatter, an ageing curve could be produced which was characteristic of the whole cast. Selected specimens corresponding to the peak-aged condition were also measured by Rockwell Hardness, HR_B, for comparison of the present results with previously published data on the age-hardening behaviour of other 6061 MMC systems.

TABLE I Details of Preforms

Cast	Preform thickness (mm)	Nominal binder content (wt %)	V_f (%)
1	15	–	20
2	15	–	20
3	17	0	23
4	16	1	19
5	19	2	16
6	24	5	17
7	24	2	17

All preforms were nominally 100 mm diameter. Those used for Casts 1 and 2 were supplied commercially.

2.3. Microstructural characterization

Optical microscopy was carried out on selected MMC specimens to examine both qualitatively and quantitatively the distribution of the fibre reinforcement. The distribution and type of second phases within the MMC was also examined. Scanning electron microscopy with energy dispersive X-ray analysis was employed to characterize the second phases identified by optical microscopy. More detailed examination of the magnesium and silicon contents in the region of fibre interfaces, and the distribution of these elements within the MMC was performed by wavelength dispersive X-ray analysis in an electron probe microanalyser. For the latter examination a probe diameter of approximately 5 μm was located in fibre-free regions in the top, centre and bottom of the MMC. Three composition measurements were conducted at each position.

3. Results

3.1. Optical microscopy

The structure of all the MMCs was typical of composites fabricated by the pressure infiltration of fibre preforms. During preforming the fibres are laid down in a mat-like structure and this results in a "two-dimensional planar random" morphology of fibres which is altered little during melt infiltration. In addition there was some clumping of fibres which gave micro variations in fibre density ranging from high to low or even zero fibre regions. This structure and the local variation in fibre distribution are illustrated in Fig. 1 which shows that the fibre-free regions were approximately 50 μm in size. In addition to these effects, Cast 2 also showed some fine planar delaminations within the composite which were parallel to the top surface (i.e. normal to the axis of the cylindrical

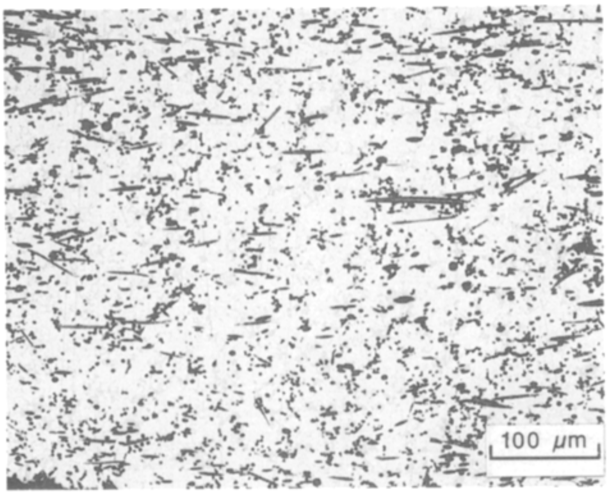

Figure 1 Optical micrograph showing the fibre distribution in a 6061 MMC (from Cast 6).

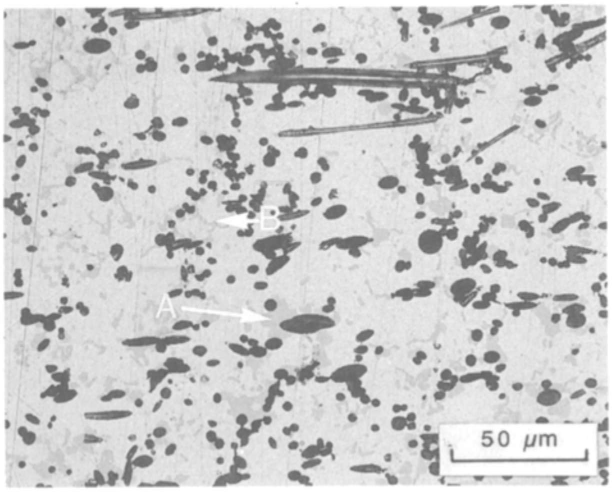

Figure 2 Optical micrograph showing the phases present in a 6061 MMC (from Cast 6). Phase A, Mg_2Si (globular). Phase B, AlFeSi (script-like).

casting). Care was taken, therefore, to avoid these regions when making hardness measurements in this cast.

All the castings exhibited intermetallic "Chinese script-like" phases both in the MMC and in the unreinforced alloy. These consisted of two distinct phases which optically appeared as dark-grey and light-grey phases. The MMC also contained a further phase which appeared more globular or blocky. This third phase is shown in Fig. 2. The amount of this phase appeared to increase towards the base of the casting, i.e. with increasing depth from the initial point of preform infiltration. In a previous paper it was assumed that such phases were associated with the dissolution and washing through of the preform's silica binder. However, in the present work, even the cast manufactured without silica binder contained this phase with the same distribution as in the other, silica-containing casts.

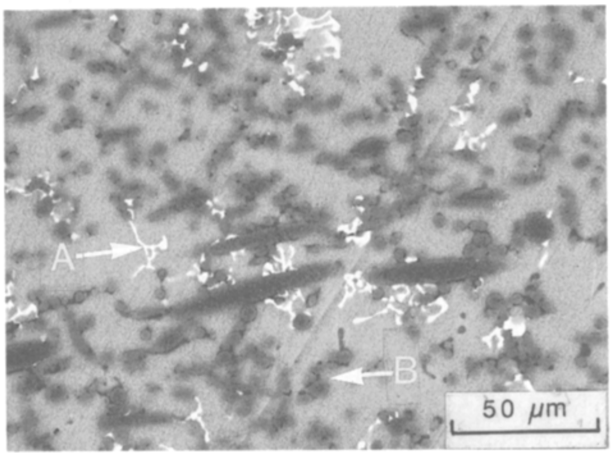

Figure 3 SEM image of a 6061 MMC (from Cast 2) Phase A, AlFeSi (light). Phase B, SiO_2 binder (dark).

3.2. Scanning electron microscopy

A backscattered electron (BE) image of the MMC produced by scanning electron microscopy (SEM) is shown in Fig. 3. In the BE image, the Chinese script-like phase appeared white and energy dispersive analysis gave an approximate composition of 70%Al, 10%Fe, 5%Si, 10%Ni, suggesting the phase was intermetallic AlFeSi. Iron is present in the alloys as an impurity element. In all cases the phase was relatively small compared with the probe size which resulted in a high aluminium count from the background. No explanation can be given for the presence of nickel unless this had arisen from an addition made to the preform which, in this case, was a commercial product. A dark-grey/black phase was also present in the BE images as shown in Fig. 3. This was situated at the fibre surfaces and also as an interconnecting phase between fibres in high fibre-packed regions. Analysis of this phase showed only a peak for silicon which suggests that it was residual silica binder. The microstructure shown in Fig. 3 is very similar to one previously published for an Al 2.3% Mg alloy [5]. However, in the latter case the dark phase was described as Mg_2Si. In the present work an indistinct phase with an analysis indicative of Mg_2Si was found to occur closely associated with fibres. This was revealed by using low penetrating voltages to improve contrast and which produced images of the type shown in Fig. 4a. The X-ray spectrum of the phase marked in Fig. 4a is shown in Fig. 4b. It is likely that this is the phase which was identified in optical micrographs as the globular type (Fig. 2).

3.3. Electron probe microanalysis

Electron probe microanalysis (EPMA) was used to determine the variation in the matrix magnesium and silicon content with increasing infiltration distance (i.e. the distance from the initial point of infiltration of the preform) for a number of the casts. The results are shown in Table II. There was a general trend for the matrix magnesium content to be high near the top of the composite (the initial infiltration surface) with lower, roughly equal contents in the middle and bottom positions of the composite. When this occurred

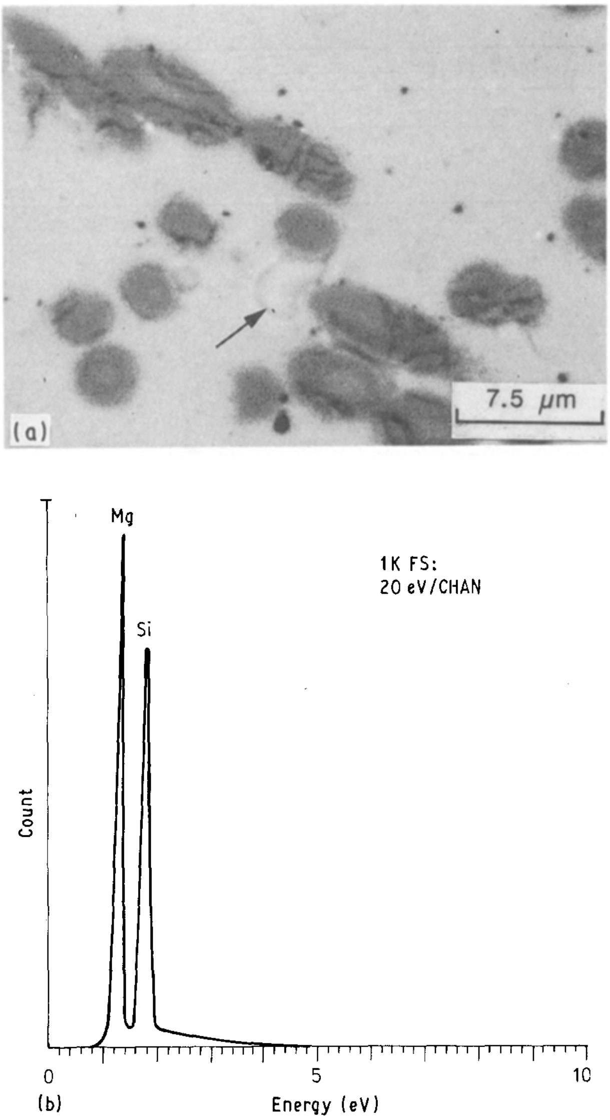

Figure 4 Mg_2Si phase in a 6061 MMC (from Cast 3). (a) SEM image (at 10 kV accelerating voltage); (b) energy dispersive spectrum of Mg_2Si phase, indicated by arrow in (a).

the reduction in magnesium concentration was typically 20%. However, specimens from Casts 1 and 5 exhibited much larger reductions of 70% and 50%, respectively. There was no obvious variation in silicon content with infiltration distance if all the samples were taken into account. However, there did appear to be an effect arising from the silica binder on the general silicon content of some of the analysed casts. Silicon contents substantially higher than expected are highlighted in Table II. It should be noted that the magnesium and silicon contents shown in Table II are uncorrected values and so whilst the true magnesium content is probably close to its measured concentration, silicon requires an upward correction (probably in the region of 50%). In effect this means that the silicon content frequently exceeded the requirements for stoichiometry, i.e. Si > Mg/1.73.

The distribution of magnesium and silicon at a position in the MMC of low matrix magnesium content were investigated by X-ray digimapping of the structure. The X-ray map shown in Fig. 5 reveals the regions of high magnesium concentration. As might be expected there was some association of magnesium and silicon, presumably as Mg_2Si. However, in general, magnesium exhibited significant segregation around fibres but there appeared to be no corresponding variation in silicon content and, in fact, high silicon concentrations in the MMC were shown to be coincident with fibres. These observations were confirmed by a spot analysis on a region of high magnesium concentration which gave uncorrected compositions of 77% magnesium and 2.5% silicon. Therefore, it appears that the segregation of magnesium to the fibres was not generally associated with Mg_2Si formation. The most likely explanation for the high magnesium analysis is that magnesium was present as a complex oxide around the reinforcement.

3.4. Age-hardening results

In all the specimens examined there was a variation in hardness from the top to the bottom of the MMC, irrespective of the ageing treatment employed. For some specimens this variation consisted of a particularly high hardness in the first few millimetres of infiltration with a nearly constant value over the remainder of the infiltration distance. This was particularly true for the casts with binder contents of 0% and 1% and for one of the casts containing 2% binder.

TABLE II Variation in magnesium and silicon contents with infiltration distance

Cast	SiO_2 binder (%)	Magnesium (wt %)	Silicon (wt %)	Infiltration distance (mm)[a]
1	–	1.01	0.71[b]	1
		0.28	0.87[b]	7
2	–	0.98	0.39	1
		0.84	0.42	7.5
		0.78	0.40	13.5
4	1	1.01	0.48	1
		0.81	0.38	7
		0.77	0.31	14
5	2	0.74	0.85[b]	2
		0.36	0.61[b]	8
6	5	0.83	0.63[b]	2
		0.55	0.54[b]	12
		0.54	0.56[b]	22

[a] Infiltration distance measured from the initial infiltrated surface of the preform.
[b] Silicon content higher than expected.

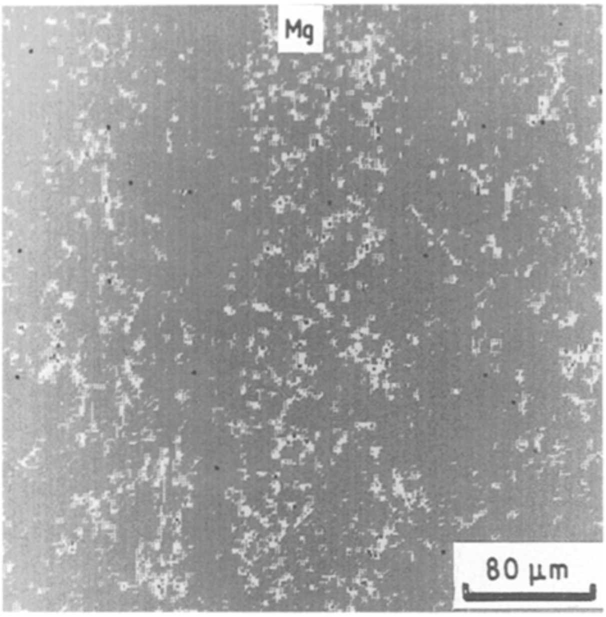

Figure 5 Magnesium X-ray map of 6061 MMC manufactured with a commercial preform (from Cast 1) showing a high magnesium concentration (white) adjacent to fibres.

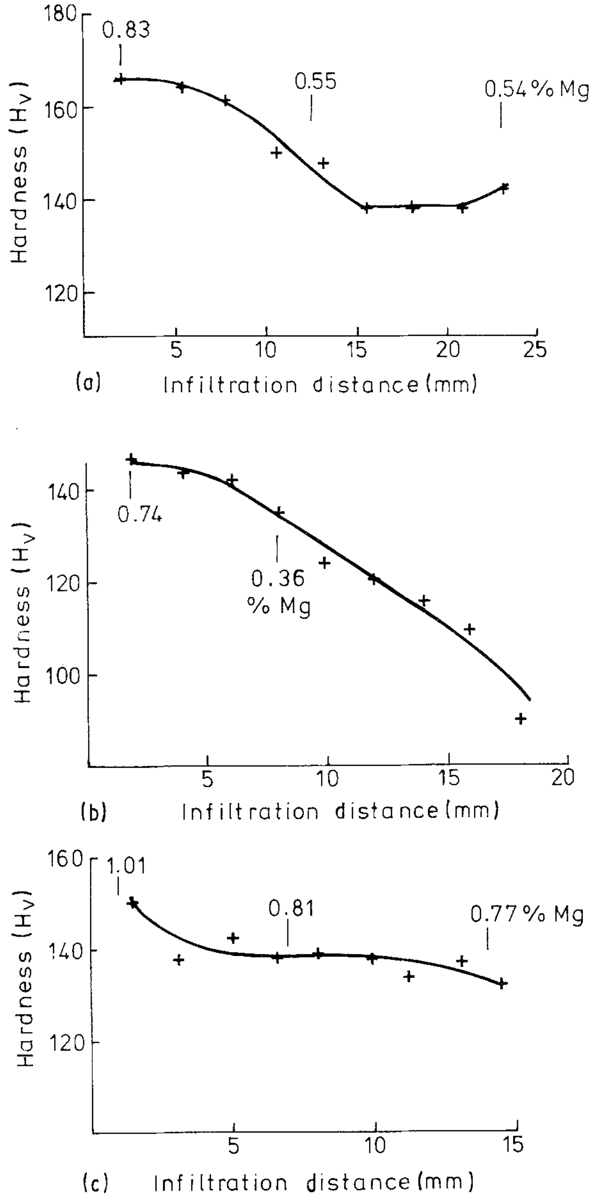

Figure 6 Examples of hardness profiles for 6061 MMC containing different silica binder contents of (a) 5%, (b) 2%, and (c) 1%. The magnesium content at different infiltration depths is also shown.

However, for the majority of the composite casts there was an overall reduction in hardness with increasing infiltration distance. The extremes of behaviour observed are illustrated in Fig. 6 and these are very similar to the variations in magnesium content which were observed. Within casts there was some variation in the hardness gradient for different specimens, although this could not be associated with any systematic sampling effect.

The hardness results were averaged to obtain representative ageing curves for both reinforced and unreinforced material aged under the same conditions, see Table III for details. Results from Cast 1 have not been included in this table because the gradient in hardness was so severe as to preclude the determination of a representative average hardness value for this MMC. Typical ageing curves at 178 °C for reinforced and unreinforced 6061 are shown in Fig. 7. For both materials there was a progressive increase in hardness with ageing time from the onset of ageing, although the peak-aged hardness was significantly higher in the reinforced material, achieving ~ 150 Hv (80 HR_B).

The ageing curves at 160 °C were similar to those at 178 °C. However, at 140 °C slightly different ageing curves were obtained for the different MMC casts and these varied between the two examples shown in Figs 8 and 9. In the first type of ageing behaviour, Fig. 8, exhibited by Cast 7, there was relatively little hardening during the initial period of ageing, followed by a rapid rise in hardness with increasing ageing time. In the second type of ageing response, Fig. 9, exhibited by Casts 2, 3 and 6, there was a more gradual increase in hardness with ageing time. The unreinforced material also exhibited a gradual increase in hardness with time. As before, the peak-aged hardness was significantly higher in the reinforced material and also slightly higher than at 178 °C, achieving ~ 160 Hv (85HR_B).

The detailed results from the ageing curves are contained in Table III. In this table the ageing kinetics have been summarized in terms of the ageing time to peak hardness. There were some problems in determining this time exactly because of specimen-to-specimen scatter and the frequent absence of a sharp maximum at peak hardness as shown by Fig. 8. However to a first approximation the MMCs appeared to age up to twice as fast as the reinforced alloy at 178 °C, and up to ten times faster at 140 °C. The increase in hardness during ageing was ~ 50/60H_v for the reinforced 6061 and ~ 70/80H_v for the unreinforced 6061 alloy. In summary, the reinforced material exhibited both enhanced ageing kinetics and higher peak-aged hardnesses than the unreinforced alloy.

Hardness profiles such as those shown in Fig. 6 have been used for a detailed investigation of the ageing behaviour at different infiltration distances (of 2, 5 and 10 mm from the initial infiltration surface) within the MMC from Cast 2. The results for isothermal ageing at 178 and 140 °C are shown in Fig. 10a

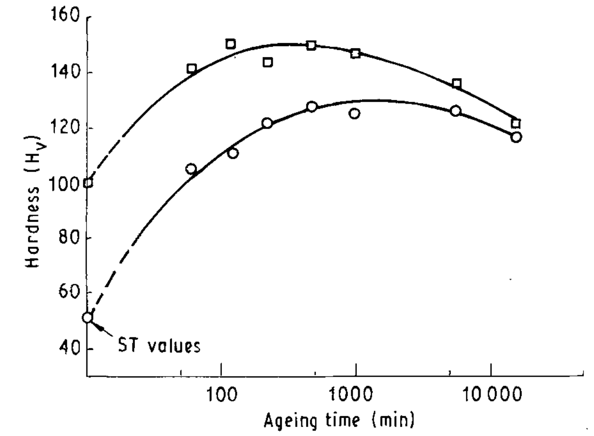

Figure 7 Isothermal ageing characteristics at 178 °C for Cast 6. (□) MMC, (○) unreinforced alloy.

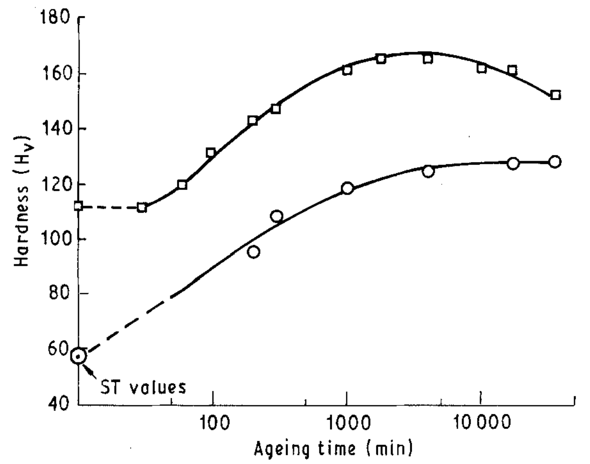

Figure 9 Isothermal ageing characteristics at 140 °C for Cast 2. (□) MMC, (○) unreinforced alloy.

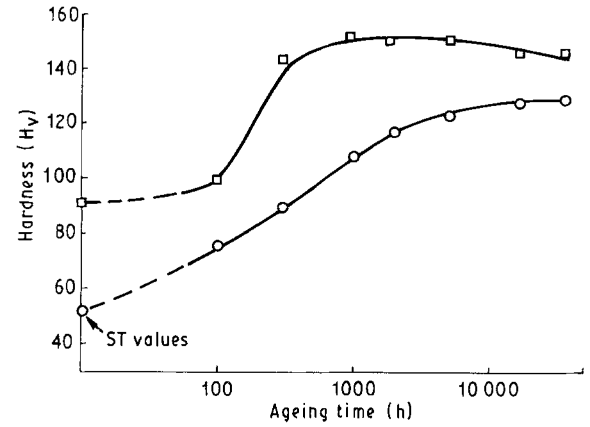

Figure 8 Isothermal ageing characteristics at 140 °C for Cast 7. (□) MMC, (○) unreinforced alloy.

and b. At both temperatures the peak hardness occurred at ageing times which increased with increasing infiltration distance although this trend was greatest at 140 °C (Fig. 10b). The larger differences in kinetics at 140 °C occurred because of a combination of increasing nucleation time and decreasing rate of ageing with increasing infiltration distance. Surprisingly the enhanced ageing kinetics to peak hardness near the initial infiltration surface at 140 °C were not followed by rapid overageing. The peak hardness values at both temperatures decreased with increasing infiltration distance, although this effect was greatest at 178 °C.

Some of the effects of infiltration distance on the ageing response of the MMC can be related to the through thickness variation in magnesium content. This is demonstrated in Fig. 6 which also includes the magnesium contents at different points along these gradients. The effect of magnesium content on the hardness of the MMC developed during ageing is shown more clearly by Fig. 11. With the exception of Cast 2, the results for each MMC produce a set of parallel lines, demonstrating a similar relationship between decreasing magnesium content and decreasing hardness. There was no obvious explanation for the markedly different results for Cast 2, although this MMC was manufactured using a commercial preform which, in this study and in a previous investigation, produced very steep hardness gradients during solution treatment and subsequent ageing.

3.5. Summary of results

1. In 6061 MMC produced by the pressure infiltration technique the matrix magnesium content was altered by reaction with the Saffil fibre preform. The magnesium content decreased with increasing infiltration distance (for unidirectional infiltration).

2. The matrix silicon content of these composites remained relatively constant with increasing infiltration distance. However, relatively high silicon con-

TABLE III Ageing results for 6061 MMC and unreinforced 6061

Cast	V_f (%)	Ageing temperature (°C)	6061 - MMC H_v(ST)[a]	6061 - MMC H_v(PA)[b]	6061 - MMC Time to PA[b] (min)	6061 alloy H_v(ST)[a]	6061 alloy H_v(PA)[b]	6061 alloy Time to (PA)[b] (min)
2	20	178	112	150	350	57	127	700
5	19	178	94	150	300	54	120	–
6	17	178	100	150	400	54	130	1 000
4	19	160	81	145	–	52	125	> 10 000
6	17	160	100	158	600	53	130	~ 10 000
2	20	140	113	167	4000	57	128	> 20 000
3	23	140	98	163	8000	–	–	–
6	17	140	100	163	4000	54		–
7	17	140	90	153	2000	52	130	~ 40 000

[a] ST = solution-treated condition; [b] PA = peak-aged condition.

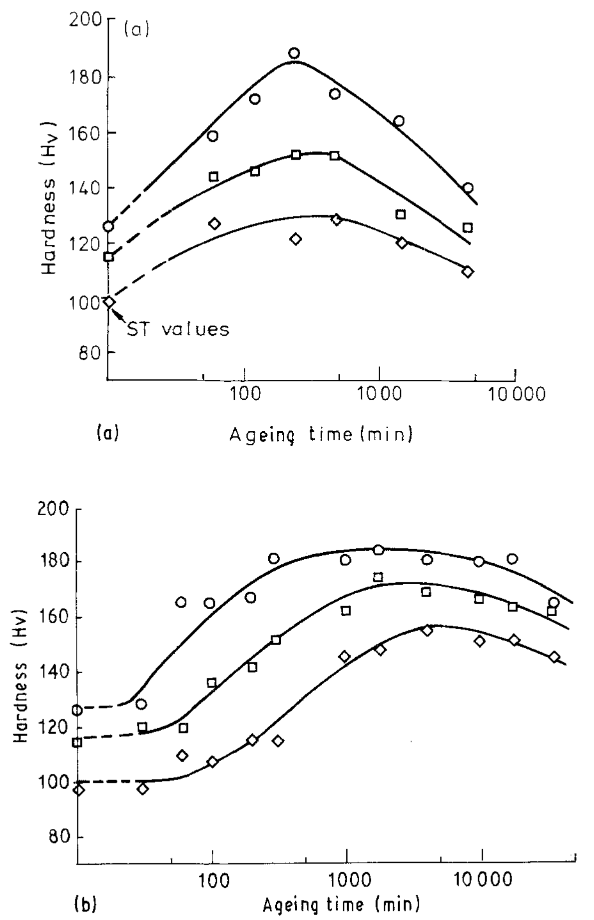

Figure 10 Dependence of isothermal ageing characteristics on infiltration distance within MMC from Cast 2. (a) at 178 °C, (b) at 140 °C. Infiltration distance: (○) 2 mm, (□) 5 mm, (◇) 10 mm.

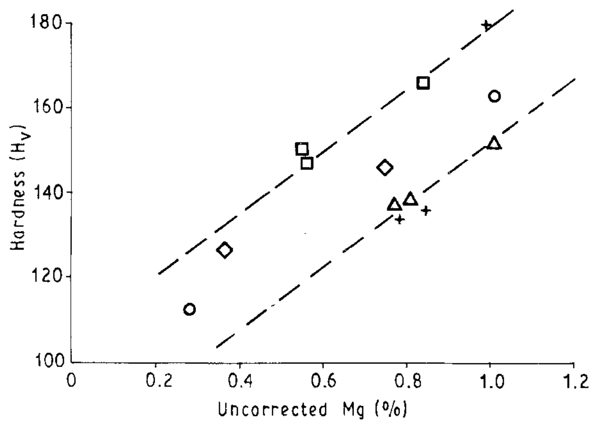

Figure 11 The effect of magnesium content on the hardness of the MMC developed during ageing. Cast: (○) 1, (+) 2, (△) 4, (◇) 5, (□) 6.

tents were observed in some of the MMC casts compared with the silicon content of the feed alloy.

3. The steepest magnesium concentration gradients and the highest silicon contents occurred in casts with a high silica binder content, i.e. 2 wt % or more in the preform.

4. In general, the 6061 MMC achieved peak hardness more rapidly than the unreinforced 6061 alloy during ageing and the difference in kinetics between the two increased with decreasing ageing temperature. The 6061 MMC was harder than the unreinforced alloy both in the solution-treated and peak-aged conditions.

5. Within the MMC the peak hardness decreased, and the ageing time to peak hardness increased, with increasing infiltration distance. The magnitude of these effects depended on the ageing temperature employed. The effect of infiltration distance on the peak hardness was greatest at 178 °C whilst its effect on the ageing time to peak hardness was greatest at 140 °C.

4. Discussion

The discussion of these results centres on the magnesium and silicon concentration profiles developed during infiltration and the effect these have in controlling the ageing characteristics of the MMC.As described above, magnesium was progressively depleted from the matrix of the MMC during infiltration of the liquid alloy into the preform. A corresponding depletion in silicon did not occur and the final matrix silicon content of the MMC was often higher than that of the feedstock alloy.

A possible explanation for the existence of the magnesium concentration gradient is that it is due to macrosegregation occurring during solidification of the MMC. Mortensen and Michand [11] have developed an analytical model which predicts composition profiles developed during infiltration of a binary hypoeutectic alloy into a fibre preform for a variety of combinations of processing parameters. In general, their model is consistent with some of the effects observed in the present investigation for casts with low silica binder contents. For example, the model predicts a marked rise in solute content towards the base of the infiltrated MMC and this is consistent with the present observation of enhanced Mg_2Si precipitate in this position in the 6061 MMC. However, the model cannot explain the large magnesium gradients which were observed in the present investigation for casts with higher silica binder contents, or the difference in concentration profiles observed for magnesium and silicon.

A more likely explanation for the observed effects is that they are due to a complex interaction between the molten alloy, the alumina fibres and the silica binder. A possible reaction has been proposed by a number of authors [4, 6] to explain the presence of an $MgAl_2O_4$ spinel at the surface of both alumina and silicon carbide reinforcements in MMC. This is

$$2SiO_2 + 2Al + Mg \rightarrow MgAl_2O_4 + 2Si \quad (1)$$

where the silica originates from the binder or from the silica-rich surface layer which is often present naturally on Saffil and silicon carbide reinforcements. Such a reaction can account for all the composition effects observed within the MMC, namely the gradients in magnesium content, the magnesium enrichment around fibres, and the high silicon contents.

Although the observation of the spinel phase is fairly common there are relatively few investigations where this has been clearly linked with depletion of magnesium from an MMC matrix. In fact there appears to be only two investigations apart from the present one in which there is direct chemical evidence for depletion, and again both of these relate to MMC produced by pressure infiltration of Saffil fibre preforms. In one of these investigations, Warren and Li [5] have shown that there was a general reduction in magnesium in the matrix of an Al–Mg–Si MMC, accompanied by an enhanced silicon content, compared with the unreinforced alloy and in the second Henriksen [6] observed a progressive reduction in magnesium during infiltration with an Al–Cu–Mg alloy. Both observations are very similar to those made in the present work.

Other evidence of magnesium depletion comes indirectly from ageing investigations on Al–Mg–Si type MMC by measuring the hardness developed during ageing. In a previous investigation on ageing of 6061 MMC [10] the hardening response of the matrix was significantly reduced in MMC containing higher volume fractions of reinforcement. In a subsequent investigation [7], a reduction in hardness with increasing infiltration distance was also highlighted and chemical mapping showed enhanced magnesium around the Saffil fibre reinforcement. The more extensive work carried out in the present investigation again demonstrates these effects and, in particular, shows that the reduction in the hardening response depends on the amount of silica binder used in the preform. A substantial reduction in age hardening occurs when the silica binder is $\geqslant 2$ wt % of the preform (or $\geqslant$ 0.6 wt % of the infiltrated matrix alloy, for a 20% V_f preform).

It is possible that magnesium reaction with the reinforcement or binder is more severe in MMC produced by pressure infiltration because of the high melt temperatures employed. In stir casting, which is performed at much lower processing temperatures, spinel has also been shown to form on the surface of SiC particulates [8]. This diminished the age hardening of a matrix of low magnesium content but did not appear to affect the behaviour of a 6061 matrix alloy. However, a relatively coarse particulate size ($\sim$ 13 μm) was used in these studies compared with Saffil fibre ($\sim$ 3 μm diameter) and this may also be an important variable because of the different surface areas available for reaction. An additional problem in general with MMC produced by processes other than pressure infiltration is how to establish if the age-hardening behaviour of the MMC has been affected by reactions between the reinforcement and the matrix. In effect this may only be possible when the reaction has been so severe that the peak-aged hardness of the MMC is comparable with, or even less than, that of the unreinforced alloy. A feature of the present type of investigation is that the existence of the hardness gradient indicates a reaction between the matrix and reinforcement has occurred and has had a significant effect on magnesium depletion.

The microanalysis results in the present work clearly demonstrate that the reaction between magnesium and the fibre or fibre/binder surface results in large differences in the magnesium content, and hence in the Mg/Si ratio, in the matrix of the MMC, both within and between casts. This has produced a significant effect on the ageing behaviour of the MMC. A simplified mechanism for the precipitation of Mg_2Si during ageing of Al–Mg–Si alloys can be described as follows [12]:

Solid solution $\rightarrow$ coherent GP zone needles (β^{11})
$\rightarrow$ semicoherent rods (β^1) $\rightarrow$ incoherent platelets or discs (β)

Hardening has been associated with both β^{11} formation and β^1 precipitation by different investigators but whichever hardening "precipitate" is most important, the amount of hardening produced will depend primarily on the amounts of magnesium and silicon available for Mg_2Si formation. The size and spacing of the precipitates or zones will also be important.

The amount of Mg_2Si precipitation can be predicted qualitatively from the Al–Mg_2Si pseudo binary phase diagram. This is shown in Fig. 12, and includes the β^1 and β^{11} solvus curves after Dorward [12]. The β^{11} solvus delineates a critical temperature, T_c, above which GP zones not achieving a critical size dissolve. The magnesium and silicon contents in the MMC matrix of selected casts made in the present investigation are shown in Table II. In the present case it is primarily the magnesium contents which determine the amount of Mg_2Si formed because silicon is usually present in excess. This was confirmed indirectly by Fig. 11 which demonstrated that as the magnesium content decreased (with increasing infiltration distance) there was a corresponding decrease in hardness. On the basis of Fig. 12, a point corresponding to an ageing temperature of $\sim$ 178 °C and a matrix containing about 1% Mg (corresponding to $\sim$ 1.6 wt % Mg_2Si) falls well below both solvus curves and substantial "precipitation" would be expected. In comparison for a lower matrix magnesium content of about 0.5 wt % ($\sim$ 0.9 wt % Mg_2Si), 178 °C is close to the β^{11} solvus curve and little or no precipitation would be expected. These predictions are qualitatively

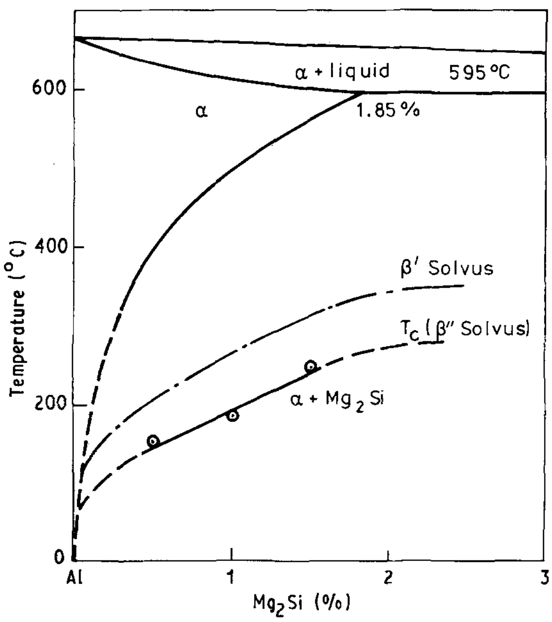

Figure 12 Pseudo-binary phase diagram for Al-Mg_2Si, including the β^1 and β^{11} solvus curves after Dorward [12].

in agreement with the peak-aged hardness results obtained at 178 °C for casts showing a marked variation in magnesium content in the present study. Low hardnesses corresponded to regions of low magnesium content, i.e. very little Mg_2Si precipitation.

The ageing profiles shown in Fig. 10a and b for Cast 2 clearly illustrate that reducing the temperature from 178 °C to 140 °C results in a higher hardness during ageing, particularly at lower (predicted) Mg_2Si contents. This is again in qualitative agreement with Fig. 12 because lower ageing temperatures favour precipitation in alloys containing lower Mg_2Si contents by decreasing the solubility of the precipitate in the matrix. A similar result was obtained by Dorward [12] for unreinforced alloys.

These observations on the effect of magnesium on the age-hardening behaviour of the MMC matrix are complicated because as the magnesium content decreases during infiltration the silicon excess (i.e. over the amount required for Mg_2Si formation) increases. Ceresara *et al.* [13] have shown that even a small amount of silicon excess results in a significant increase in the peak-aged hardness during ageing. This may explain why Casts 2 and 6 exhibit similar peak hardnesses even though their average magnesium contents differ significantly (see Table II). Unfortunately, there are insufficient data in the present investigation to allow the effect of silicon on the ageing behaviour to be separated from the effect of magnesium.

The mapping of magnesium and silicon contents in the present study was confined to the fibre-free regions. The matrix close to the fibres, and in areas of high fibre density (i.e. clumped regions) was not analysed; however, these regions must also affect the hardness of the MMC. This may explain an apparent inconsistency in the results for Casts 2 and 4 containing high and low binder contents, respectively. These exhibit very similar compositional variations with infiltration depth but significantly different hardness profiles as shown in Fig. 6. The effect of the near fibre region is, therefore, also important and requires further investigation.

In addition to its effect on the peak hardness, the Mg_2Si content also has a marked effect on the ageing kinetics. In the present work the rate of age hardening (particularly at 178 °C) decreased and the incubation time for ageing (particularly at 140 °C) increased as the Mg_2Si content decreased as shown in Fig. 10a and b. The former observation is very similar to a trend in the results presented by Dorward [12] and both observations are consistent with those made by Ceresara *et al.* [13] of increasing incubation times and decreasing age-hardening rates with a reduction in the silicon excess of the matrix. In the latter case, a reduction in the silicon excess was associated with a reduction in the Mg_2Si precipitate content. Therefore, in 6061 MMC in which the matrix magnesium is significantly depleted due to reaction with the fibre or binder, both the age-hardening and the ageing kinetics are also affected.

The effect of changes in the matrix chemistry on the ageing kinetics of MMC has received relatively little attention in the literature and the reason most commonly proposed for different ageing kinetics in MMC is that they contain high dislocation densities compared with unreinforced alloys, generated to relieve the thermal stresses produced during cooling. In some cases such high dislocation densities may reduce the age-hardening rates of the MMC by effectively removing the "quenched in" vacancies which are an important constituent in the early stages of GP zone formation [7]. However this seems unlikely in the present investigation because it is difficult to envisage how this mechanism could be affected by infiltration distance. It is more likely that higher dislocation densities in the MMC contribute positively to ageing rates by either enhancing the diffusion rates of elements within the matrix or by providing preferential sites for precipitate nucleation.

The effect of different types of reinforcement on the dislocation densities, and hence the ageing kinetics, of 6061 MMC has been modelled by Dutta and Bourell [1]. They predict that the dislocation densities and ageing kinetics increase as the reinforcement changes from particulate to fibre to whisker, for given volume fraction. There are three other investigations [8, 14, 15] on isothermal ageing of 6061 MMC in addition to the present one which can be used to assess these predictions. It has been assumed that the compositions of the 6061 used in these investigations were similar. This seems reasonable for at least three of the investigations, because in these the peak-aged hardnesses of the 6061 MMCs were similar, i.e. 80–85 HR_B or 150–160 H_v, indicating similar amounts of Mg_2Si precipitate (these investigations are the present one and those from the other references [14, 15]). The results of these investigations are summarized in Table IV by using the time to peak hardness as an indicator of the ageing kinetics. This table also includes results for unreinforced 6061 manufactured in exactly the same way as the MMC, as well as the method of manufacture in each case.

The data at the ageing temperatures of 175–180 °C have been ranked in the table in order of ageing kinetics. To a first approximation the ranking is similar for both the unreinforced and reinforced alloys, with a minimum difference in ageing rates between the investigations (for the unreinforced alloy) of about four times. In general, the 6061 MMCs age between 2.5 and 3.5 times faster than the unreinforced alloys, except for the 6061–10% $SiC_{(p)}$ MMC [8] which exhibits a much smaller difference.

The general behaviour predicted by Dutta and Bourell is consistent with the enhancement of ageing observed in practice. However, the data in Table IV at 175–180 °C suggest that the effect of different reinforcement types and volume fractions on the enhancement of ageing kinetics appears to be relatively small. This possibly should be expected because practical observations on whisker- and particulate-reinforced MMC have shown that above a relatively low reinforcement volume fraction, changing the reinforcement type or volume fraction has little effect on either the dislocation density [16, 17] or the ageing kinetics of the MMC [17]. An interesting observation which arises from the data at 175–180 °C is that the

TABLE IV Comparison of ageing times to peak hardness for 6061 and 6061 MMC from different investigations.

Ageing temp. (°C)	Time to PA (min)		Reinforcement employed in the MMC
	Unreinforced	MMC	
175	240	85	20% SiC_w [1]
177	600	180	23% B_4C_p [2]
178	700–1000	300–400	20% Saffil [3]
175	840	600	10% SiC_p [4]
140	> 20 000	2000–8000	20% Saffil [3]
140	~ 10 500	2550	23% B_4C_p [2]

Method of manufacture of MMC: [1] powder metallurgy [15]; [2] powder metallurgy [14]; [3] pressure in filtration [present work]; [4] compocasting [8].

ageing kinetics of both the reinforced and unreinforced material appear to be strongly dependent on the processing route employed. A general enhancement of ageing kinetics when materials have been fabricated by the powder metallurgy route has been noted before [18] and this effect is most apparent in the work of Rack [15]. The ranking of the ageing kinetics does not appear to result from compositional differences between the alloys used in the different investigations for reasons already described.

As the ageing temperature is reduced, both the results produced by Nieh and Karlack [14] and those produced in the present investigation exhibit a large increase in the ageing kinetics of the MMC compared with the unreinforced material. The effect of lower isothermal ageing temperatures has not been theoretically modelled, although Nieh and Karlack [14] suggest the relatively rapid ageing of the MMC at lower temperatures is due to an enhanced contribution from dislocation pipe diffusion. The present investigation has shown that the enhancement of ageing kinetics in MMC at lower ageing temperatures is also strongly dependent on the magnesium content of the MMC matrix.

Although the contribution of increased dislocation densities to the ageing behaviour of MMC is reasonably well established their presence cannot account for all the effects observed in the present investigation. The effect of the reinforcement on the matrix chemistry of the MMC has received relatively little attention in the literature and yet, as shown in the present work, magnesium interaction with the reinforcement or binder depletes the matrix magnesium content, increases the silicon content and can affect both the ageing kinetics and the peak hardness. Therefore, for a complete understanding of the ageing behaviour of MMC, both the physical and chemical effects of the reinforcement on the matrix should be taken into account.

4. Conclusions

1. In Saffil-reinforced 6061 MMC manufactured by the pressure infiltration method, there is a reaction between the alloy's magnesium addition and the preform. This depletes the matrix of the MMC of this alloying element but increases its silicon content.

2. The amount of magnesium depletion appears to be associated with the amount of silica binder in the preform and when this exceeds 1 wt % (for a 20% V_f preform) the MMC can exhibit a progressive and significant depletion of magnesium with increasing infiltration distance.

3. Depletion of magnesium in the MMC results in a reduction of the hardening produced during ageing. This results from a reduction in the amount of Mg_2Si phase produced during heat treatment, in accordance with predicted behaviour based on the Al–Mg_2Si pseudo-binary phase diagram.

4. If the ageing is performed at lower temperatures (e.g. 140 °C rather than 178 °C) some of this magnesium depletion can be offset by enhanced precipitation of Mg_2Si due to the decreased solubility of this phase in the matrix. In general, a lowering of ageing temperature produces slightly higher peak hardnesses in the MMC, and ageing kinetics which increase with the magnesium content of the matrix.

5. MMC based on 6061 ages more rapidly than the unreinforced alloy and this difference in ageing kinetics increases with decreasing ageing temperature.

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