Aluminium–matrix silicon carbide whisker composites fabricated by pressureless infiltration

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Al–matrix SiC whisker composites were fabricated by pressureless infiltration of liquid Al–Mg or Al–Si–Mg alloys at 830–950 °C in the presence of N_2 into a preform of nickel coated SiC whiskers. The nickel coating on the whiskers was obtained by electroless plating and made pressureless infiltration possible. The composite made by pressureless infiltration exhibited slightly lower tensile strength and modulus and slightly higher coefficient of thermal expansion than the corresponding composite made by pressure infiltration. However, the differences were small in spite of the lack of prior evacuation in the pressureless infiltration case. On the other hand, the hardness decreased with increasing distance from the preform–melt interface much more significantly in composites made by pressureless infiltration than those made by pressure infiltration. The hardness decrease, which was attributed to a porosity increase, was larger for composites made by pressureless infiltration without prior evacuation than those made by pressureless infiltration with prior evacuation. The Al–SiC reactivity was larger for composites made by pressureless infiltration than those made by pressure infiltration, because the infiltration time was longer in pressureless infiltration.

1. Introduction

Liquid metal infiltration is commonly used for the fabrication of metal–matrix composites. This method involves the infiltration of a liquid metal into a porous preform. The preform is an agglomerate or a compact of the reinforcement, which may be in the form of particles, whiskers or fibres. The attraction of this method is that the preform may be conveniently shaped or machined to the desired shape of the composite, resulting in a near net shape component after liquid metal infiltration. Furthermore, the method is fast compared to powder metallurgy.

Liquid metal infiltration typically involves the evacuation of the preform prior to infiltration, in order to avoid back pressure from the air in the preform, i.e. vacuum infiltration. The evacuation step means that a vacuum chamber is necessary for the preform. In most cases, the infiltration is carried out with the help of a gas (isostatic) pressure, i.e. pressure casting or pressure infiltration. The pressure application means that a compressor and a pressure chamber are necessary. The need for a vacuum–pressure chamber and a compressor makes the equipment for liquid metal infiltration quite expensive.

Pressureless infiltration refers to liquid metal infiltration without an applied pressure on the liquid metal. The removal of the pressure requirement greatly simplifies the equipment for the infiltration. Pressureless infiltration has been carried out without a vacuum for composites with Mg–containing Al alloy matrices (that wet ceramic fillers better than pure Al) and reinforcements in the form of particles or fibres [1–3]. It has also been reported that the addition of a metal fluoride, such as K_2ZrF_6, helps pressureless infiltration [4, 5]. Furthermore, it has been reported that coating the filler with Na_2O helps pressureless infiltration [3]. In this paper, the authors report that the coating of the filler with a metal helps pressureless infiltration. With the help of the metal coating, SiC whisker Al–matrix composites were fabricated by pressureless infiltration, even without a vacuum.

Although there have been numerous studies of the mechanical properties of metal–matrix composites, little attention has been given to the mechanical property distribution within a composite. This distribution is expected to be uniform in composites made by powder metallurgy, provided that the powder mixture prior to sintering is uniform. However, the mechanical property distribution may not be uniform in composites made by liquid metal infiltration, as the infiltration proceeds inward from the edge to the centre of the reinforcement preform. In this work, the mechanical property distribution of composites made by pressure infiltration is compared to that of composites made by pressureless infiltration.

The reaction between SiC and Al is known to result in Si and Al_4C_3 [6–15]. For composites made by liquid aluminium infiltration, the out-diffusion of the reaction product Si in the form of liquid Al–Si toward the excess liquid aluminium around the preform during infiltration results in a non-uniform Si concentration in the resulting composite, such that the Si concentration is lowest at the centre of the composite and highest at the edge of the composite next to the excess aluminium [16]. The presence of a nickel coating on the SiC whiskers does not alter the Al–SiC reactivity [17]. Since the time for pressureless infiltration is higher than that for pressure infiltration, the Al–SiC reactivity may be higher in pressureless infiltration. However, this possibility has not been previously investigated. In this work, it was found that pressureless infiltration resulted in higher Al–SiC reactivity, so that the Si concentration was higher and the Si concentration gradient was greater.

2. Experimental procedure

Bare silicon carbide (SiC) whiskers were obtained from Advanced Refractory Technologies, Inc. (Buffalo, NY). The basic properties of the bare whiskers are listed in Table I. The whiskers (1.4 μm in diameter) were coated with nickel by electroless plating. The coating was not continuous. Roughly 60% of the surface area of the whiskers was coated. The average coating thickness was 0.02 μm. Fig. 1 shows scanning electron microscope (SEM) photographs of the bare and Ni-coated whiskers. The coated whiskers were made into a preform (40 mm diameter, 30–40 mm height) at a whisker content of 10 vol %. No binder was present in the preform. The preform was placed in a graphite mould, with an Al alloy ingot on its top surface. The alloys used were Al–5 Mg, Al–10 Mg and Al–5 Si–5 Mg. The mould was placed in a furnace with a nitrogen gas purge and then heated to different infiltration temperatures (800–950 °C) for 6 h. The effect of evacuation to 133.3 Pa (10^{-2} torr) prior to infiltration was also investigated.

TABLE I Basic properties of silicon carbide whiskers

Primary crystallographic phase	β
Average diameter, μm	1.4
Average length, μm	18.6
Average aspect ratio	13.2
Specific gravity	3.21
Dielectric constant,	40
Thermal conductivity, $Wm^{-1}\,°C^{-1}$	90
Coefficient of thermal expansion, $10^{-6}\,°C^{-1}$	4.6

TABLE II Infiltration distance obtained by pressureless infiltration

Matrix	Infiltration temperature (°C)	Distance (mm)	
		Without evacuation	With evacuation at 133.3 Pa
Al–5 Mg	800	0	
Al–10 Mg	800	0	
Al–10 Mg	830	5	
Al–10 Mg	900	16	
Al–10 Mg	950	17	⩾ 36
Al–5 Si–5 Mg	850		⩾ 25
Al–5 Si–5 Mg	900		⩾ 36
Al–5 Si–5 Mg	950		⩾ 40
Al–10 Mg	850		0
Al–10 Mg	900	0	
Al–10 Mg	950	0	

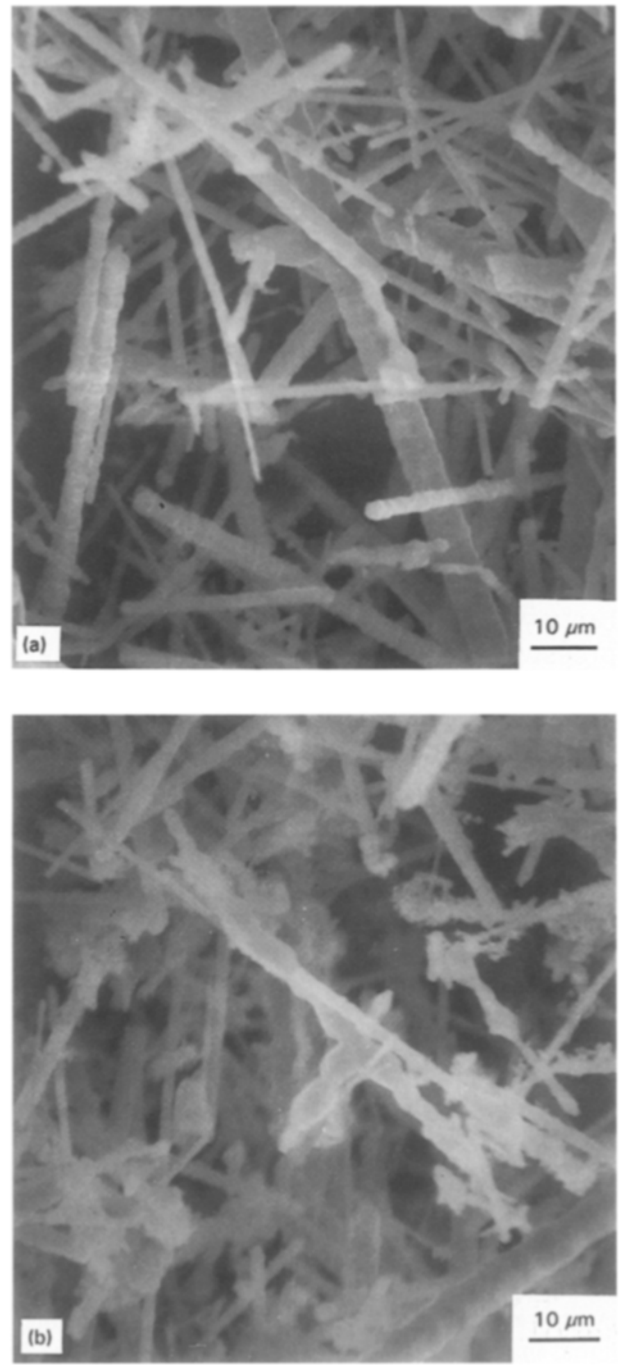

Figure 1 SEM photographs of (a) bare and (b) Ni-coated SiC whiskers prior to preform fabrication.

Table II shows the infiltration distances corresponding to the different infiltration temperatures and matrix alloys. The infiltration distance increased significantly with increasing infiltration temperature for a given alloy. At 800 °C, no infiltration occurred. The use of bare SiC whiskers (without the metal coating) and the Al–10 Mg matrix led to no infiltration up to 950 °C. In contrast, the use of coated SiC whiskers and the same matrix led to infiltration at just 830 °C, even without prior evacuation. This means that the coating aided the infiltration. Evacuation greatly increased the infiltration distance, as shown for the Al–10 Mg matrix and an infiltration temperature of 950 °C.

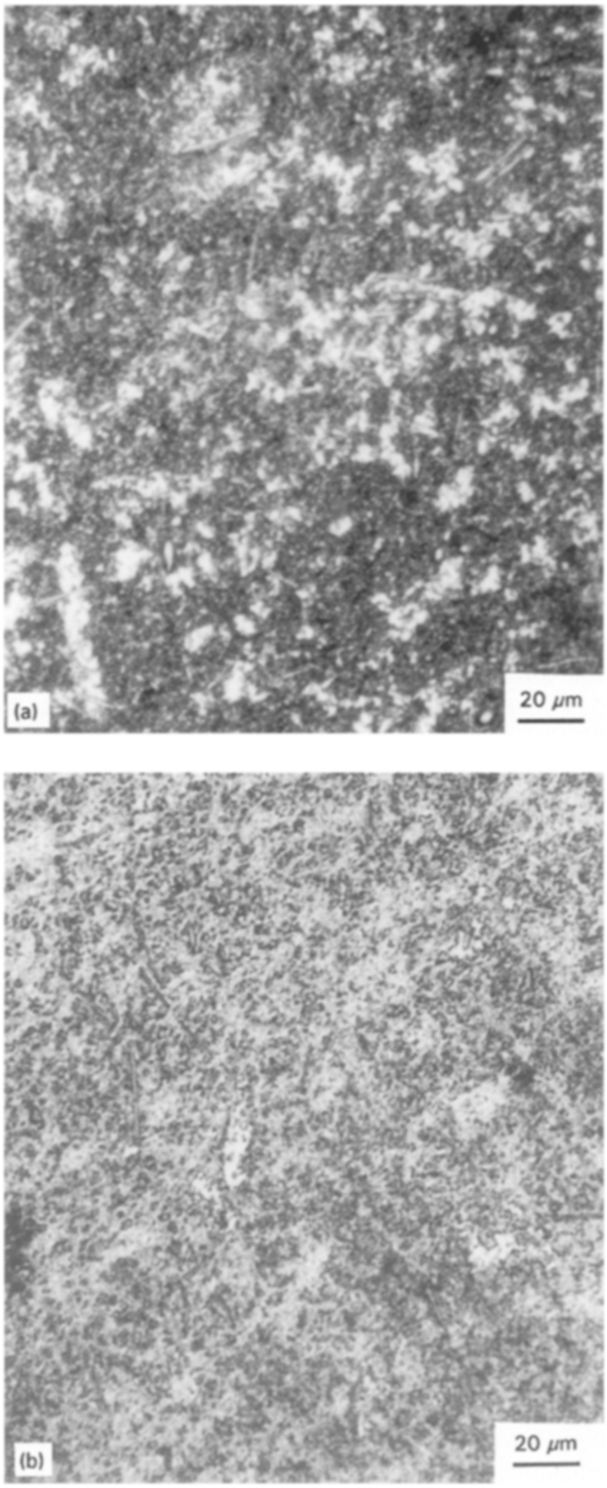

Figure 2 Optical micrographs of polished sections of SiC whisker composites made by pressureless infiltration at 900 °C: (a) Al–5 Si–5 Mg as the matrix and with prior evacuation, and (b) Al–10 Mg as the matrix and without prior evacuation.

Optical and scanning electron micrographs of polished sections of the 10 vol % SiC whisker composites made by pressureless infiltration at 900 °C are shown in Figs 2 and 3, respectively. In each figure, the composite with Al–5 Si–5 Mg as the matrix was made with prior evacuation, whereas that with Al–10 Mg as the matrix was made without prior evacuation. The higher magnification of Fig. 3 reveals the whiskers more clearly. The whisker distribution was quite uniform. The whisker orientations were three-dimensional.

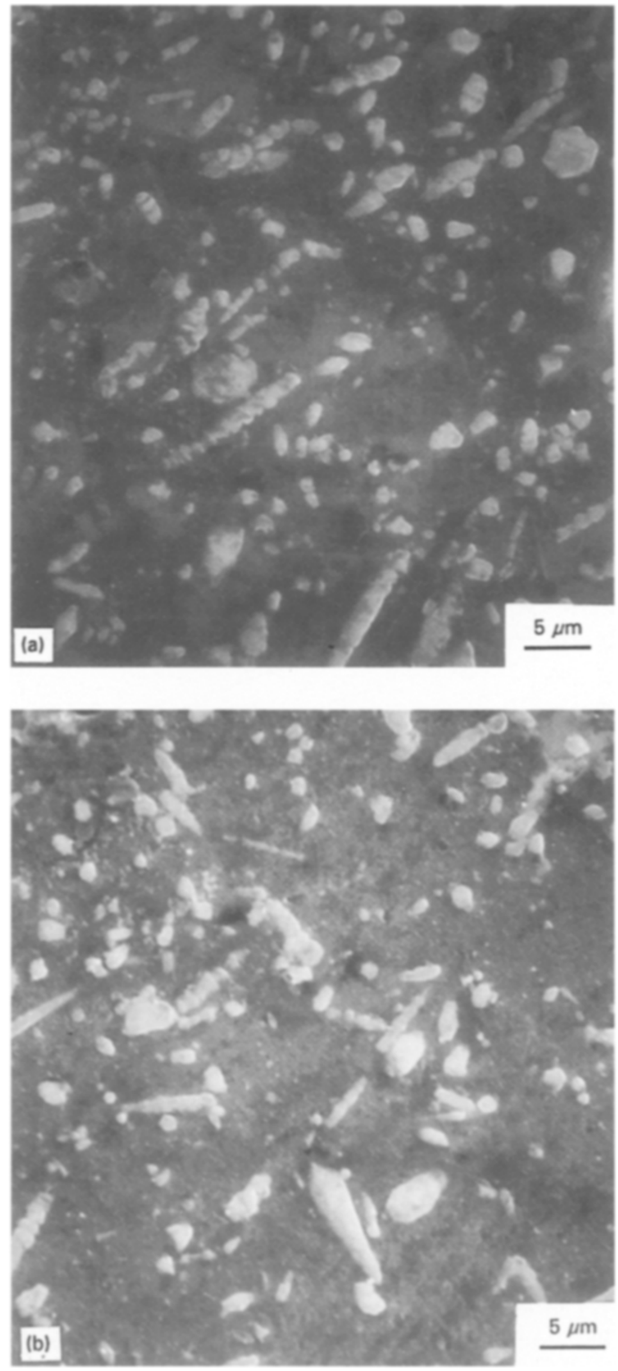

Figure 3 SEM photographs of polished sections of SiC whisker composites made by pressureless infiltration at 900 °C: (a) Al–5 Si–5 Mg as the matrix and with prior evacuation, and (b) Al–10 Mg as the matrix and without prior evacuation.

The Vickers hardness distribution within the composite cylinder was determined by measuring the hardness along a radius in the plane half way up the cylinder and along the cylindrical axis at the centre of the cylinder, as shown in Figs 4 and 5. Each figure contains data for Al–10 Mg matrix 10 vol % nickel coated SiC whisker composites made by pressureless infiltration without prior evacuation, pressureless infiltration with prior evacuation and by pressure infiltration (at 13.8 MPa, with prior evacuation). The infiltration temperature was 900 °C in all three cases. The hardness distribution was much more uniform in the composite made by pressure infiltration than those

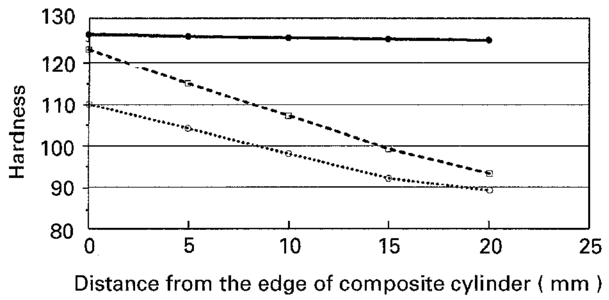

Figure 4 Hardness distribution along a radius in the mid-plane of the composite cylinder: (—) composite made by pressure infiltration at 13.8 MPa, (- - -) composite made by pressureless infiltration with prior evacuation, (......) composite made by pressureless infiltration without prior evacuation.

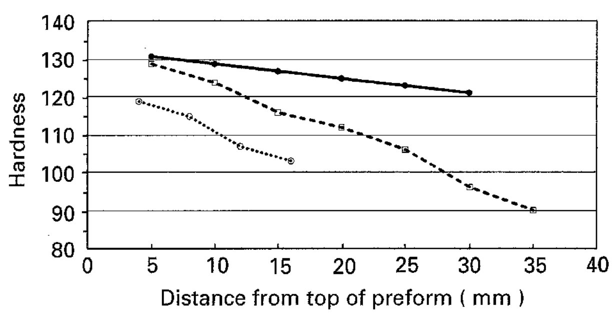

Figure 5 Hardness distribution along the axis of the composite cylinder: (—) composite made by pressure infiltration at 13.8 MPa, (- - -) composite made by pressureless infiltration with prior evacuation, (......) composite made by pressureless infiltration without prior evacuation.

made by pressureless infiltration. Among the composites made by pressureless infiltration, prior evacuation resulted in greater hardness at any point in the composite. These differences in hardness are attributed to the differences in porosity; a higher hardness was associated with a lower porosity, as shown by metallography.

The composites were subjected to tensile testing. The dog-bone sample configuration used for tensile testing did not allow measurement of the tensile strength distribution, but just the average tensile strength near the centre of the composite cylinder. Table III indicates that the Al–10 Mg matrix composite made by pressureless infiltration was similar in tensile properties to that made by pressure (13.8 MPa) infiltration at the same infiltration temperature of 900 °C. Moreover, the coefficient of thermal expansion

TABLE IV Coefficient of thermal expansion (CTE) of composites with Al–10 Mg as the matrix and containing 10 vol % nickel coated SiC whiskers

		CTE (10^{-6} °C^{-1})		
Infiltration method	Infiltration temperature (°C)	50–100 °C	50–200 °C	50–300 °C
[a] Pressureless	900	17.71	18.55	18.80
[b] Pressure	900	16.24	16.40	17.92
[b] Pressure	800	17.52	17.81	18.53

[a] No evacuation.
[b] Evacuated to 133.3 Pa prior infiltration.

(CTE) of the former composite was slightly higher than that of the latter, as shown in Table IV. In addition, comparison of the first two of the three lines in Table III indicates that Al–5 Si–5 Mg is far less attractive than Al–10 Mg for serving as the matrix for producing composites of high strength. This is because the whisker–matrix bonding was stronger for Al–10 Mg than Al–5 Si–5 Mg, as shown by SEM photographs of the tensile fracture surfaces (Figs 6 and 7). A gap was observed at the whisker–matrix interface when the matrix was Al–5 Si–Mg (Fig. 6), but not when the matrix was Al–10 Mg (Fig. 7).

In order to investigate the effect of pressureless infiltration versus pressure infiltration on the Al–SiC reactivity, the Si concentration distribution was determined by measuring the liquidus–eutectic invariant temperature difference, ΔT, by differential scanning calorimetry (DSC) and, through the Al–Si–Mg phase diagram, relating ΔT to the Si concentration. The procedure is essentially the same as in [16] and [17]. Table V and Fig. 8 show the Si distributions along the radius of Al–5 Si–5 Mg matrix composite cylinders containing 10 vol % SiC whiskers and made by infiltration with and without pressure (13.8 MPa). The composite made by pressureless infiltration was made at 900 °C with prior evacuation, whereas those made by pressure infiltration were made at 800 or 900 °C with prior evacuation. At the same infiltration temperature of 900 °C, the silicon concentration, as well as the slope of its variation with spatial position, were higher when the infiltration was pressureless than when the infiltration involved pressure, whatever was the location within the composite cylinder. The difference in Si concentration between the cases with and without pressure increased from the edge to the centre of the composite cylinder. The higher Al–SiC reactivity

TABLE III Tensile properties of composites

Infiltration method	Infiltration temperature (°C)	Matrix	Vol % whiskers	Strength (MPa)	Modulus (GPa)	Ductility (%)
[a] Pressureless	950	Al–5 Si–5 Mg	10	178 ± 3	94 ± 1.6	5.7 ± 0.3
[b] Pressureless	900	Al–10 Mg	10	226 ± 6	81 ± 1.0	6.8 ± 0.3
[a] Pressure	900	Al–10 Mg	10	228 ± 1	91 ± 2	5.5 ± 0.5
[a] Pressure	800	Al–10 Mg	10	232 ± 1	92 ± 1	5.8 ± 0.3

[a] Evacuated to 133.3 Pa prior to infiltration.
[b] No evacuation.

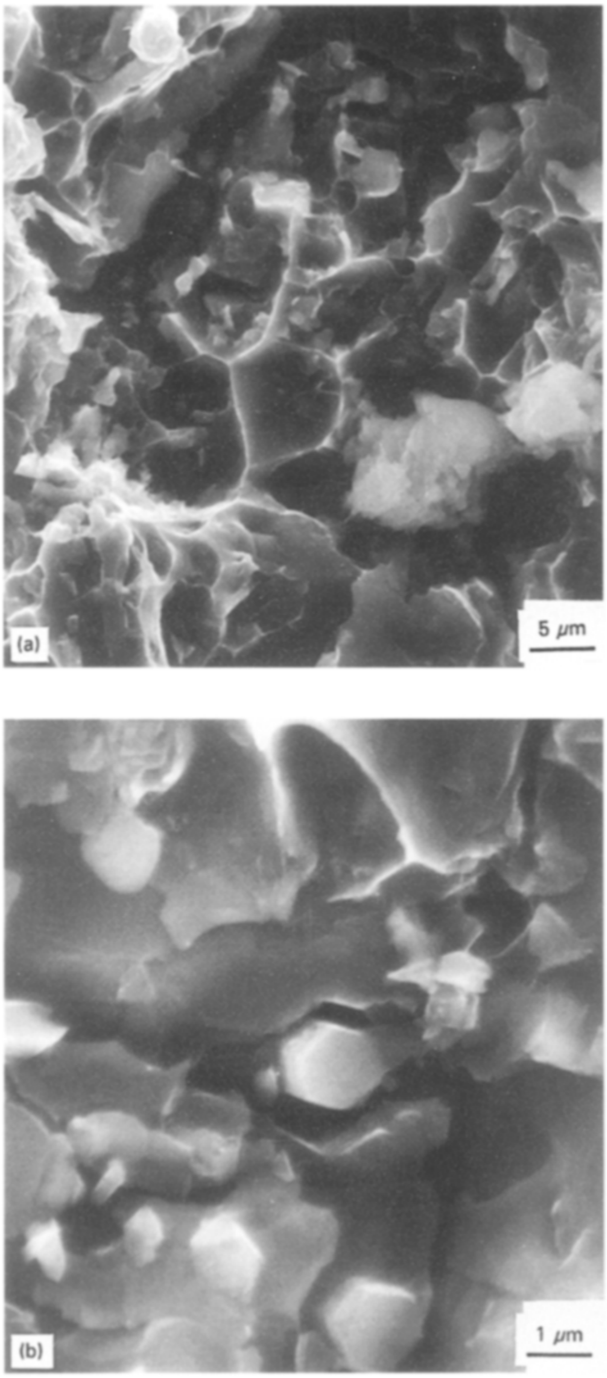

Figure 6 SEM photographs at two magnifications of the tensile fracture surface of an SiC whisker Al–5 Si–5 Mg matrix composite made by pressureless infiltration (with prior evacuation) at 900 °C.

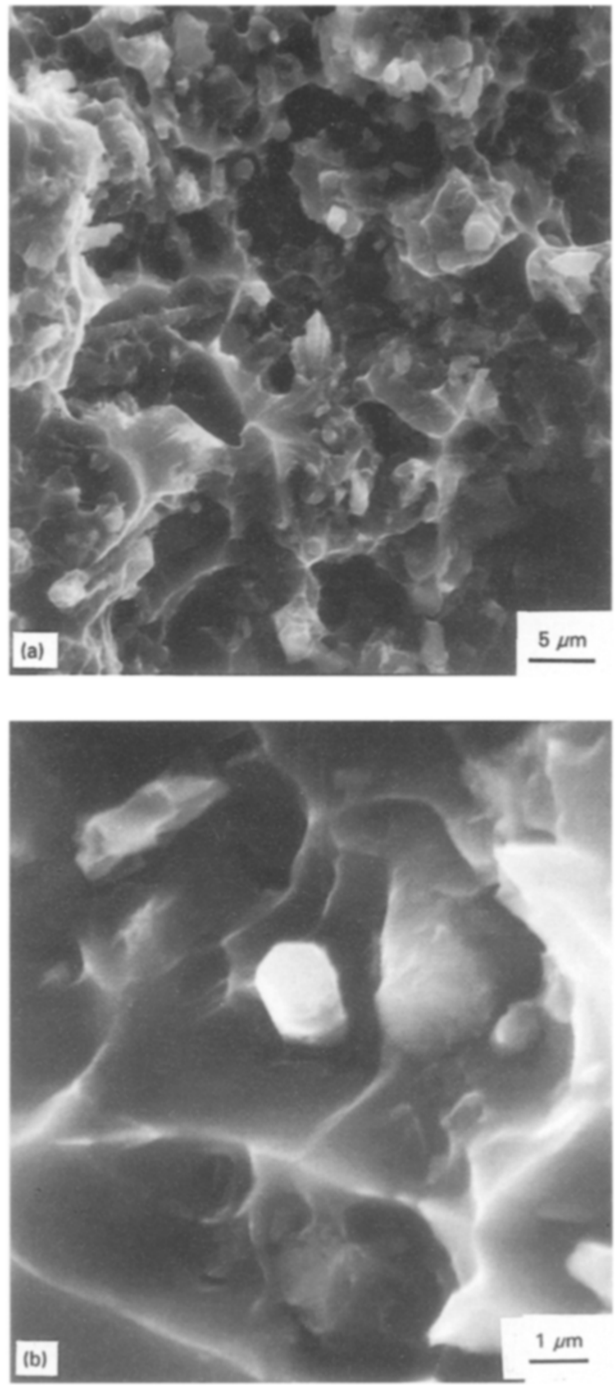

Figure 7 SEM photographs at two magnifications of the tensile fracture surface of an SiC whisker Al–10 Mg matrix composite made by pressureless infiltration (without prior infiltration) at 900 °C.

TABLE V The liquidus–eutectic invariant temperature difference ΔT and Si content (wt%) in the matrix of Al–5 Si–5 Mg SiC composites

Infiltration		Distance from the edge of the composite cylinder (mm)				
method		0	5	10	15	20
[a] Pressureless	ΔT, °C	58.00	56.80	52.20	43.70	41.20
	Si, wt%	5.62	5.77	6.37	7.48	7.82
[b] Pressure	ΔT, °C	59.10	57.50	55.10	52.50	52.20
	Si, wt%	5.47	5.68	6.00	6.34	6.38
[c] Pressure	ΔT, °C	59.60	58.20	56.90	54.80	52.70
	Si, wt%	5.41	5.59	5.76	6.03	6.31

[a] 900 °C, with prior evacuation.
[b] 900 °C, 13.8 MPa, with prior evacuation.
[c] 800 °C, 13.8 MPa, with prior evacuation.

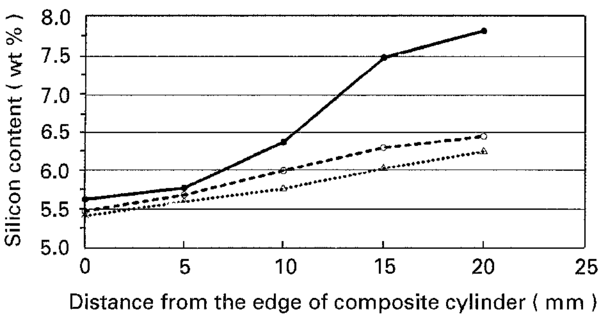

Figure 8 Silicon concentration variation along a radius in the mid-plane of the Al–5 Si–5 Mg matrix composite cylinder. (———) composite made by pressureless infiltration at 900 °C with prior evacuation, composites made by pressure infiltration at 13.8 MPa and (......) 800 °C or (- - -) 900 °C with prior evacuation.

of pressureless infiltration compared to pressure infiltration is attributed to the longer infiltration time in pressureless infiltration. As a non-uniform Si concentration itself is not associated with a non-uniform mechanical property distribution [16], the more severe Si concentration variation in the composite made by pressureless infiltration should not cause a more non-uniform mechanical property distribution in this composite. The observed greater non-uniformity in hardness distribution in this composite (Figs 4 and 5) is attributed to the porosity non-uniformity rather than the Si concentration non-uniformity. Comparison of the curves in Fig. 8 for the two composites made by pressure infiltration shows that the Al–SiC reactivity increased with increasing infiltration temperature, as expected.

3. Conclusions

Pressureless infiltration, even without evacuation, was achieved at 830–950 °C in a nitrogen purge, using Al–Mg or Al–Si–Mg as the matrix alloy and nickel coated SiC whiskers as the reinforcement. The coating, though not continuous, helped the infiltration due to the improved wetting of the reinforcement. Without the coating, no infiltration took place. The infiltration distance increased if the preform was evacuated prior to infiltration. The resulting composite was slightly lower in tensile strength and modulus and slightly higher in CTE than the corresponding composite made by pressure infiltration. However, the differences were small in spite of the lack of prior evacuation in the pressureless infiltration case. The hardness distribution was almost uniform in composites made by pressure infiltration, but was not uniform in composites made by pressureless infiltration. The hardness decreased with increasing distance from the preform–melt interface to the centre of the composite much more significantly in the composite made by pressureless infiltration than that made by pressure infiltration. This hardness non-uniformity in the composite made by pressureless infiltration was still significant, but was reduced when evacuation had been undertaken prior to pressureless infiltration. The hardness non-uniformity is attributed to the greater porosity at the centre of the composite. The Al–SiC reactivity was found to be greater in pressureless infiltration than pressure infiltration, due to the longer infiltration time required for pressureless infiltration.

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