Casting particulate and fibrous metal–matrix composites by vacuum infiltration of a liquid metal under an inert gas pressure

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A new method of making metal–matrix composites is reported. This method combines the essentials of three liquid–phase fabrication methods: (i) vacuum infiltration, (ii) infiltration under an inert gas pressure, and (iii) squeeze casting. In this method, the particulate or fibrous preform is placed in a mould and the matrix alloy is placed above the preform. The matrix alloy is heated to the liquidus temperature together with the mould and the preform under vacuum. Then an inert gas like argon is compressed on to the top surface of the matrix–alloy melt, forcing the melt to infiltrate the preform. The pressure is 1000 to 2500 psi. As the melt is just at liquidus temperature, it is much lower than that used in squeeze casting. Moreover, the pressure is an order of magnitude lower than that used in squeeze casting. The low temperature lessens the interfacial reaction between the matrix and the filler, while the low pressure essentially eliminates preform compression. This method has been successfully used to fabricate aluminium–matrix composites reinforced by short ceramic fibres, continuous ceramic fibres, SiC particles, Al_2O_3 particles, graphite flakes and SiC whiskers.

1. Introduction

Metals and alloys with low densities, such as aluminium, magnesium, titanium and their alloys, are usually chosen as the matrices of metal–matrix composites (MMCs). A wide variety of ceramic, boron and carbon materials are used as reinforcements. Unfortunately, these liquid metals and alloys do not wet the surfaces of most commercial reinforcements. In particular, much work has been done to develop efficient methods to improve the wettability between aluminium and the reinforcements [1–7], but no significant progress has yet been made. As conventional metallurgical methods cannot be used satisfactorily to produce MMCs, and some special procedures like vacuum infiltration, rheocasting and diffusion bonding were developed to solve the wetting problem. A controlled amount of reaction at the interface may be desirable for obtaining strong bonding between the matrix and the reinforcement, but if a deleterious reaction occurs, a very brittle intermetallic compound will be formed at the interface. As a result, it causes a decrease in the mechanical strength of the composites.

In 1983, the Toyota Motor Corporation announced its historic development in the manufacture of composite pistons used in automotive diesel engines [8]. The manufacture was essentially a squeeze casting operation. In this method, a porous ceramic fibre preform was inserted into a die, and the aluminium alloy melt was poured into the preheated die located on the bed of a hydraulic press. The applied pressure made the molten aluminium alloy penetrate the fibre preform and bond the fibres. The composite aluminium pistons were shown to be far superior to unreinforced pistons and the cost was low enough to be competitive with conventional pistons. Many piston manufacturers and research institutes are now interested in this fabrication method [9–15]. Squeeze casting processing is mainly suitable for preparing short fibre and whisker reinforced metals.

Powder metallurgy (PM) is another well known production technique. It is the simplest way to ensure good distribution of the reinforcements within the matrix. The SiC particulate composites prepared by the PM method have excellent properties [15–17], but the present price ranges from \$40 to \$150 per 1 lb (1 lb = 0.453 kg). The high price of this processing technique will hinder its commercial application on a large scale.

Rheocasting was successfully used to prepare particle or short fibre reinforced metals [18–20]. In this method, the matrix alloy is vigorously agitated to form a semi-solid slurry with a high viscosity, and particles or short fibres are introduced and retained in the partially solidified alloy slurry. The volume fraction of particulate material in the composites usually does not exceed 0.3. An excessive amount of particle addition makes the viscosity of the composite slurry increase significantly. It is difficult to cast a slurry with

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a very high viscosity into a mould. For the same reason, the volume fraction of short fibres (5 mm in length) generally cannot exceed 0.1. Another problem is associated with the fibre damage due to vigorous agitation. In spite of these problems, rheocasting represents the most effective of all manufacture processes for producing discontinuous fibre reinforced metals with small additions of reinforcements. For example, Al–matrix composites with 2% graphite particles can be prepared by this process for use as a piston material [21].

Dural Aluminium Composites Corporation developed a process to manufacture SiC particle reinforced aluminium by combining pretreated SiC particles with molten aluminium. It was reported that this method was cost effective [22], but no details on this process have been revealed.

The fabrication techniques for MMCs depend to a large extent upon the choice of reinforcements and matrices. Some methods like diffusion bonding and hot isostatic pressing are also employed, particularly in the case of continuous fibre reinforcements.

The principle of the process described in this paper is similar to that of the squeeze casting, but, instead of a ram, an inert gas is used to press the liquid aluminium alloy into the preform. To ensure that the aluminium melt remains in a liquid state, the melt temperature adopted may be as low as the liquidus temperature for alloys with a temperature range between the liquidus and solidus, or near to the liquidus temperature for pure aluminium and the eutectic alloys. The rate at which the pressure is increased is quite low.

2. Manufacture equipment
The hot isostatic pressing (HIP) apparatus used in this work for preparing MMCs is schematically illustrated in Fig. 1. The chamber (steel) was evacuated using a mechanical vacuum pump. An inert gas (nitrogen or argon) bottle was connected to the chamber with tubes and a valve. If the pressure of the inert gas in the bottle is lower than 2500 psi, a compresser must be used to increase the chamber pressure when needed. The chamber wall must be strong enough to sustain simultaneously high pressures and high temperatures, at least 2500 psi at 665° C. The inner wall surface of the chamber was coated with a graphite paste for ease in demoulding. Thermocouples 1 and 2 were in contact with the outer wall of the chamber, near to the aluminium alloy in the upper part and the preform at the bottom respectively. Before the high pressure gas was introduced into the chamber the temperature of the aluminium melt was allowed to drop to its liquidus temperature at a very low cooling rate. We can consider that the temperature in any part of the chamber was approximately the same. The heating element was made from graphite.

3. Manufacture processing
The process used for the manufacture of MMCs is schematically illustrated in Fig. 2. The whole process may be divided into seven steps, which are described in detail below.

A_0–A_1: The aluminium ingot and the preform were put in the chamber. The chamber was then sealed and evacuated to a pressure of 50 to 200 $\times$ 10^{-3} torr (9.7 to 38.7 $\times$ 10^{-4} psi).

A_1–A_2: The chamber and its charges were superheated 50 to 100° C above the liquidus temperature of the alloy. In the mean time, evacuation continued.

A_2–A_3: The temperature was maintained for a period of time to ensure that the alloy melts completely and that the temperature of any part of the chamber was approximately equal.

A_3–A_4: The power input was gradually lowered until the temperature dropped to the liquidus or near to the liquidus temperature at the cooling rate of 0.5 to 3.0° C min^{-1}. Evacuation continued at the same time.

A_4–A_5: While the temperature was maintained, the evacuation was stopped. The valve connected to the inert gas bottle was opened. A pressure of 1000 to 2500 psi was applied to the surface of the melt to force the melt to penetrate the porous preform completely.
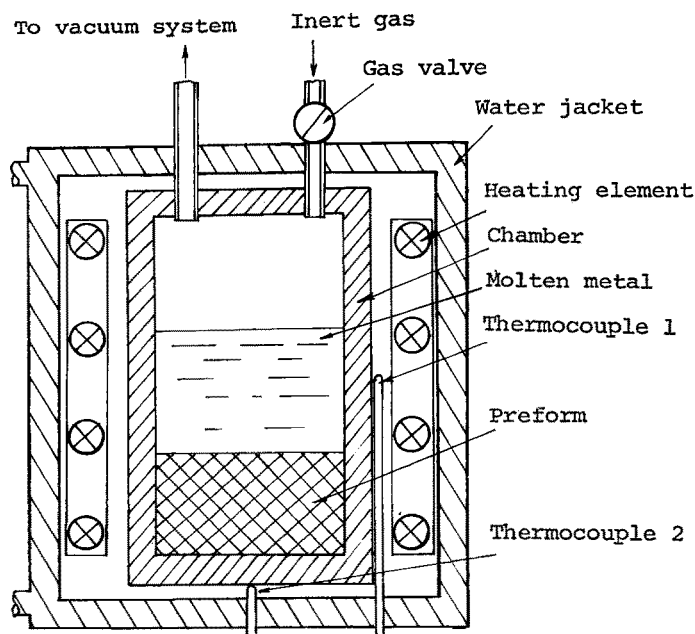
To vacuum system   Inert gas

Gas valve

Water jacket

Heating element

Chamber

Molten metal

Thermocouple 1

Preform

Thermocouple 2

Figure 1 Schematic of the apparatus for preparing aluminium–matrix composites.
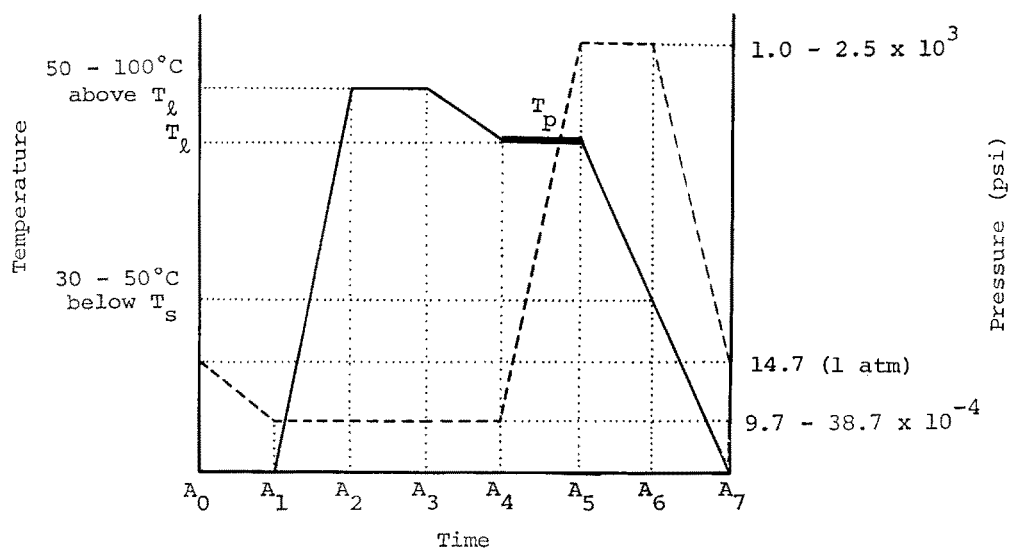
Figure 2 Schematic of the temperature (——) and pressure (‒ ‒ ‒) changes during the process used for fabricating aluminium–matrix composites. T_s = solidus temperature; T_f = liquidus temperature.

A time of 30 sec was needed to reach the pressure of 2000 psi used in this work.

A_5–A_6: Once the predetermined pressure was reached, the electricity supply to the heating element was cut off. In order to increase the cooling rate of the chamber, a cooling water jacket outside the chamber was used. The pressure must be maintained during the solidification period and the cooling period afterward.

A_6–A_7: When the temperature was 30 to 50° C below the solidus, it was supposed that the composite had solidified completely. The outlet valve was then opened to release the inert gas. Premature release would result in shrinkages, cavities and cracks in the composite. The temperature continued to drop until it was below 300° C. The lid of the chamber was then opened. The composite material was then demoulded from the chamber.

The HIP apparatus may be subjected to a temperature of 2200° C and a pressure of 30 000 psi. In case of preparing aluminium-matrix composites, the maximum temperature and pressure needed are separately 720° C and 2500 psi, respectively — much below the capacity of the HIP equipment. Therefore, the processing can be achieved with a rather simple apparatus in place of the HIP.

4. Results

Different kinds of aluminium-matrix composites were prepared using the processing method described above. Three commercial aluminium alloys were used as matrix alloys:

(a) Pure aluminium (170.1), chemical composition: Al(99.77%), Fe(0.16%), Si(0.07%).

(b) A132 aluminium alloy, chemical composition: Al–1.43Cu–12.13Si–1.3Mg–2.53Ni.

(c) 413.1 aluminium alloy (Al–12Si), chemical composition: Al(85.98%), Si(11.85%), Fe(0.92%), Cu(0.56%).

Reinforcements were short ceramic fibres, continuous ceramic fibres, SiC whiskers and particulate materials.

4.1. Short ceramic fibre reinforced Al alloys

Fibrefrax HSA Fibre manufactured by Sohio Engineered Materials Company was used as reinforcement. The fibre properties are as follows

fibre diameter: $1.2\,\mu m$ (mean),

fibre length: 3 mm,

specific gravity: $2.7\,g\,cm^{-3}$,

chemical compositions: Al_2O_3 (43.4%), SiO_2 (53.9%), Fe_2O_3 (2.8%), TiO_2 (1.6%), K_2O (0.1%), Na_2O (0.1%).

The reinforcement was commercial grade, readily available as a furnace refractory. It was similar to that used in composite pistons except for the small diameter of $1.2\,\mu m$. The fibre diameter in composite pistons is 3 to $5\,\mu m$. The fibres were first suspended in water and separated thoroughly. The preform was made by a combination of suction filtration and pressing. Sometimes a binder was required to improve the stability of the preform. In the present work a silica binder was found to be quite satisfactory. The preform manufacture method was discussed in detail elsewhere by other authors [9, 12].

Fig. 3 shows the microstructure of the composite. The fibre volume fraction (V_f) of the composite was 7.1 vol. %. The binder (silica colloid) content was 5 wt % of the preform.
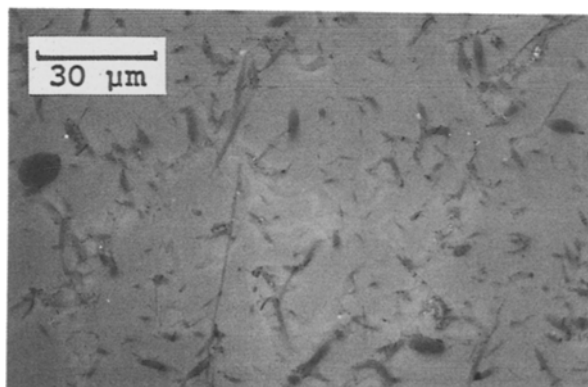

Figure 3 SEM photograph of short ceramic fibre reinforced Al–12Si alloy.

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An Al–12Si commercial ingot was used as the matrix alloy. The melt temperature (T_p, defined in Fig. 2), was 580.0° C as the inert gas began to apply pressure on the surface of the melt. The cooling rate in the A_3–A_4 period was 3° C min^{-1}. It took 35 sec for nitrogen gas to increase its pressure in the chamber from 0 to 2000 psi.

The original thickness of the preform along the pressing orientation was 25.5 mm. The final thickness of the fibre reinforced area in the composite decreased to 25.3 mm. The compression deformation due to the pressing operation was less than 0.8%.

With the squeeze casting method, the corresponding deformation in general is higher than 10%, even as high as 25%. The fibre V_f in the Toyota pistons was 5 to 7% [8, 12, 15]. A porous preform with low V_f is very weak and can be easily deformed or broken in the pressing operation. The compression deformation was affected by many variables in the processing, such as the temperatures of the aluminium alloy, the die and the preform, the retention time between preform placement and alloy pouring, and between alloy pouring and ram pressing. It was difficult to keep all the variables constant, so the deformation amounts distributed over a wide range. The usual way to solve the problem is to increase the amount of the binder in the preform, but excessive binder addition is harmful to the composite. Fig. 4 shows large flake-like binder remaining in the composite. The remaining binder was mechanically weak and brittle, and it severed the matrix. Therefore the amount of binder addition had to be limited.

The processing method described here offers a way to solve the afore-mentioned problem. Even with a small binder content of 5 wt %, no significant deformation of the preform was observed.

The high hardness of the ceramic fibres means that the composites are very difficult to machine, even though diamond or nitride edge tools can be used. The problem of producing near-net-shape components has received much attention recently. Obviously, due to the small deformation (less than 1%) the method
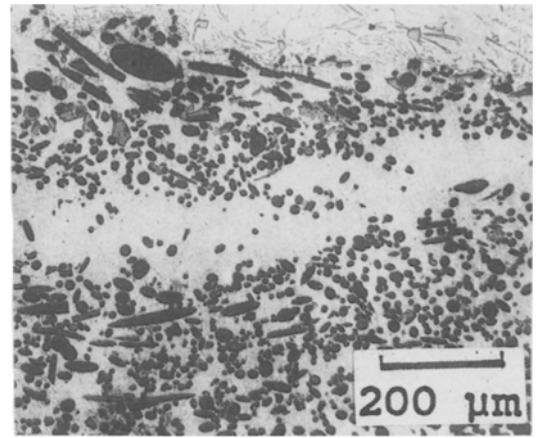
Figure 5 Photomicrograph of a short ceramic fibre reinforced Al–Si alloy prepared by squeeze casting, showing a crack present in the original preform.

described here can be a new precision technique for eliminating the machining step.

In the squeeze casting method, the ram speed used in general is about 10 mm sec^{-1}. At this rate, the infiltration of a 20 mm thick preform appears to take only 2 sec. The unwetting between the preform and the melt means that, the melt cannot penetrate the preform by its own weight. The infiltration process depends completely upon the huge pressure of 7000 to 28000 psi. The pressure which is applied on the melt front of a preform increases so rapidly (within 2 sec) that cracks of the preform can be observed in the composite occasionally (Fig. 5). The maximum pressure in the new method of this paper was about 2500 psi, far less than that of squeeze casting. It takes more than 30 sec to increase the pressure from 0 to 2500 psi — much longer than the corresponding time in squeeze casting. We propose that the melt can penetrate a preform completely before the pressure reaches 2000 psi. The low rate of pressure increase to avoid preform cracks and deformation is also an advantage.

4.2. Continuous fibre reinforced aluminium alloys

Continuous Nextel 440 ceramic fibre manufactured by 3M Center was used as a reinforcement. The fibre properties are:

fibre diameter, 10 to 12 μm,
density 3.05 g cm^{-3},
chemical composition Al_2O_3 (70%), SiO_2 (28%), B_2O_3 (2%).

Nextel fibres are continuous polycrystalline metal oxide fibres which are woven into a fabric. The fibres had been coated with an organic polymer, so the fibre cloth was heated to 800° C in an oven in air to remove the polymer finish. The cloth was then cut to the size of the preform and laid up in the chamber. A total of 40 to 60 layers was used to form a fibre preform. The matrices used were Al–12Si alloy and pure aluminium.

Fig. 6 shows the microstructure of a composite with Al–12Si as the matrix, prepared at 583.0° C (T_p) and a pressure of 2500 psi. Four bundles of fibres with different orientations are shown in the photograph. No evidence of fibre breakage in the composite is
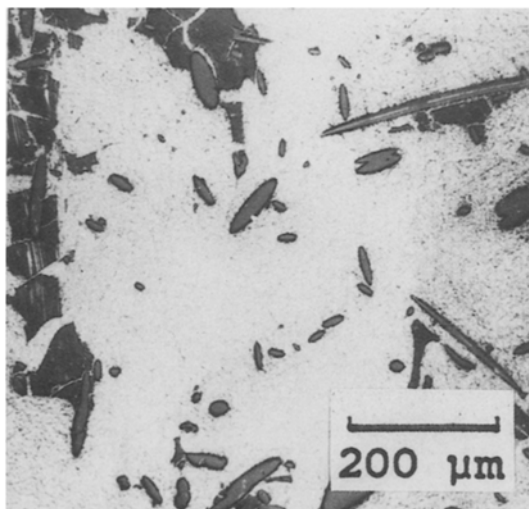

Figure 4 Photomicrograph showing the flake-like binder remnants dispersed in the composite due to excessive binder addition in the preform. (The composite sample was prepared by squeeze casting.)
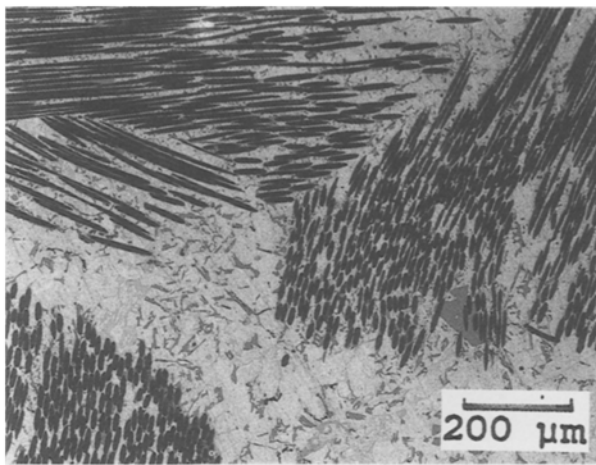
Figure 7 SEM photograph of the same composite as Fig. 6. No fibre breakage was observed.

Figure 6 Photomicrograph of a Nextel fibre reinforced Al–12Si alloy showing four bundles of fibres.

found in the SEM photograph (Fig. 7). SEM examination of the interface between fibres and the matrix also showed that no significant reaction occurred.

Fig. 8 shows the microstructure of a composite with Al–12Si as the matrix, prepared at 770°C (T_p) and a pressure of 15 000 psi. Both temperature and pressure were higher than those of the composite in Fig. 7. Fibre breakage and dark (black) patches dispersed in the matrix can be observed in the photomicrograph (Fig. 8b). The SEM test showed that these dark patches contained the element boron, which must be from boron oxide in the fibres. It was also confirmed that the patches were not the remnants of broken fibres because they contained much more silicon than aluminium and no oxygen was found (Fig. 9). Therefore it must be caused by some interface reaction.

High temperatures and high pressures may promote interface reaction. A severe reaction is not desirable. The interface reaction of the composites would be alleviated by using a temperature near the liquidus and a low pressure.

After solidification, the cooling rate of the composite made by this method was quite low. It means that the fibre reinforcements were exposed to high temperatures for a long time, which may cause excessive reaction at the interface and growth of coarse grains in the matrix.
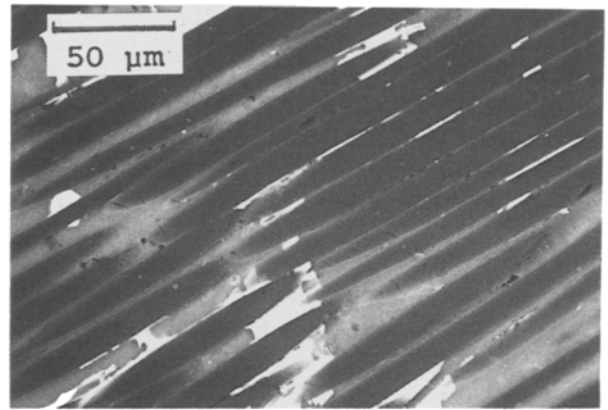
In the case of pure aluminium (170.1) used as the matrix, a significant improvement in mechanical strength was noted. The tensile strength of the Nextel fibre cloth reinforced aluminium ($V_f = 0.3$) was 143.5 MPa, as opposed to 60 MPa for pure aluminium.

4.3. Particulate composites

The particulate materials used as reinforcements were SiC particles in the 240, 320, 400 and 600 mesh size, alumina particles in the 0.05, 0.3, 1, 5 and 60 μm size, graphite flakes and graphite powder. The particles were put into the chamber and then infiltrated. The matrices were pure aluminium and Al–12Si alloy.

Fig. 10 shows the microstructure of alumina particle reinforced aluminium alloy. The alumina particles 1 μm in size were dispersed in the Al–12Si alloy homogeneously. The V_f value was about 0.4. It took 60 sec to reach the pressure of 1230 psi at 578.8°C (T_p)

Fig. 11 shows the microstructure of alumina particle reinforced aluminium. The particles were about 60 μm in size. The matrix was pure aluminium (170.1). It took 150 sec to reach the pressure of 2508 psi at 660.3°C (T_p).

Fig. 12 shows the microstructure of SiC particles (400 mesh) reinforced pure aluminium (170.1). It took 30 sec to reach the pressure of 1910 psi at 579.2°C (T_p).

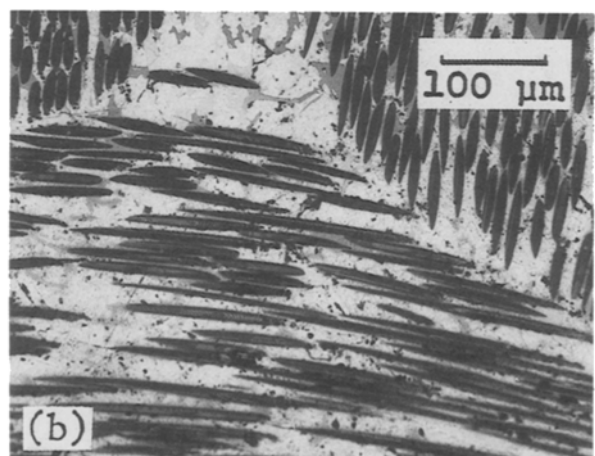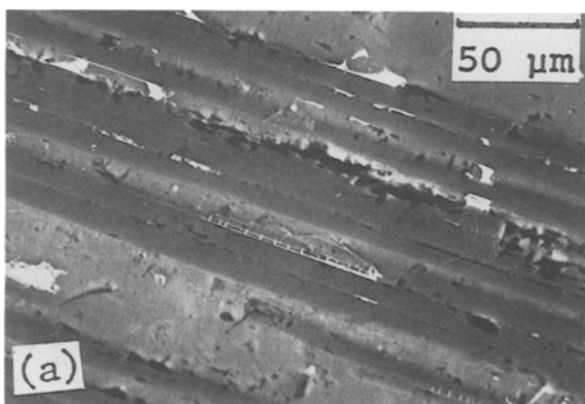Tests were performed to investigate the influence of the processing pressure. For this purpose, alumina

Figure 8 (a) SEM photograph of Nextel fibre reinforced Al–12Si alloy prepared at 770°C and 15 000 psi. Fibre damage was observed. (b) Photomicrograph of the same composite as (a). Dark (black) patches dispersed in the matrix were observed.
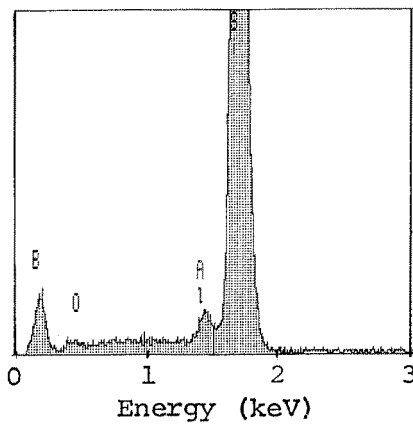
Figure 9 X-ray spectroscopic elemental analysis result for the dark (black) patches dispersed in the matrix shown in Fig. 8.

Figure 11 Photomicrograph of Al_2O_3 particle (60 μm size) reinforced aluminium.

particles 5, 1, 0.3 and 0.05 μm in size were spread in the chamber layer upon layer. Ceramic fibre cloth was used to keep one layer separate from another. The thickness of a layer was about 6 mm, with a total thickness of 25 mm. The chamber with charges was heated to the liquidus temperature. Pressure of 1230 and 2000 psi were applied separately. Under the pressure of 2000 psi and aluminium melt infiltrated all four layers completely. No porosities or cracks were found in the final composite. The melt also infiltrated four layers of alumina particles under a pressure of 1230 psi. However at 1230 psi in the 5 μm particle layer, porosities were observed due to the failure of penetration by the aluminium melt. Obviously the pressure should be an important factor in processing. The pressure must be high enough to force the aluminium melt to penetrate the gaps between the particles. The width of the gap between particles is also a very important factor. The width of the gap depends upon the particle size, particle strength (the formability of particles) and particle size distribution. The smaller the particle size, the smaller the gap. In this work the preform of alumina particles 0.05 μm in size was infiltrated by aluminium melt under a pressure of 1230 psi, which can be easily attained by rather simple equipment.

The particle size distribution plays an important role in the gap width. We noted that any particulate material has its own size distribution. Some distribu-
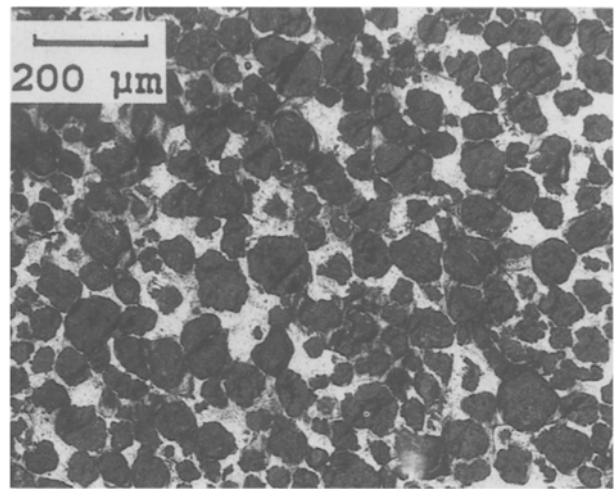
tions scatter over a narrow range near the nominal size, while others scatter over a wide range. Two different particulate materials with the same nominal particle size may have different size distributions. The particles with a wide distribution range tend to have a narrow gap width between adjacent particles because the gap between large particles may be filled by small particles. A particulate material with a narrow size distribution has particles of nearly the same size. Hence, the gaps will not be easily filled because of the lack of small particles. For convenience, the term "close-packed density" (D_{cp}) is defined as the density of an agglomerate of particles that are closely packed by ramming and vibration. It was confirmed that the particulate material with a high D_{cp} value had a wide size distribution. The D_{cp} value may be measured by putting a certain amount of particulate material into a cylindrical container. After ramming and vibrating, the particles are packed closely. The volume of the particles remain constant. The D_{cp} value can be calculated in terms of the weight and the volume of the agglomerate [23].

Table I shows the D_{cp} values of the alumina and SiC particles, together with the volume fractions under a close-packed condition.
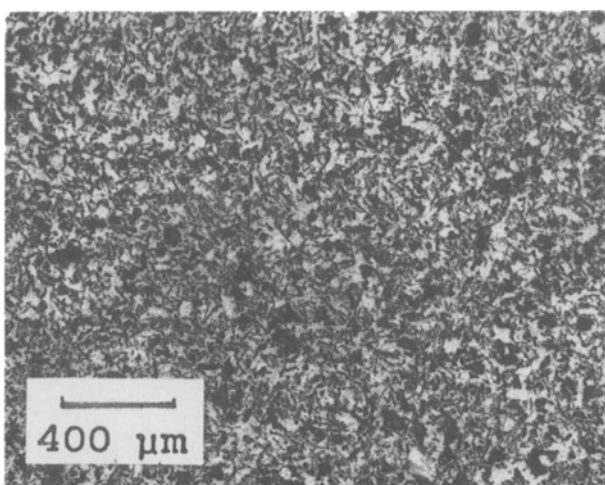

Figure 10 Photomicrograph of Al_2O_3 particle (1 μm size) reinforced Al–12Si alloy.
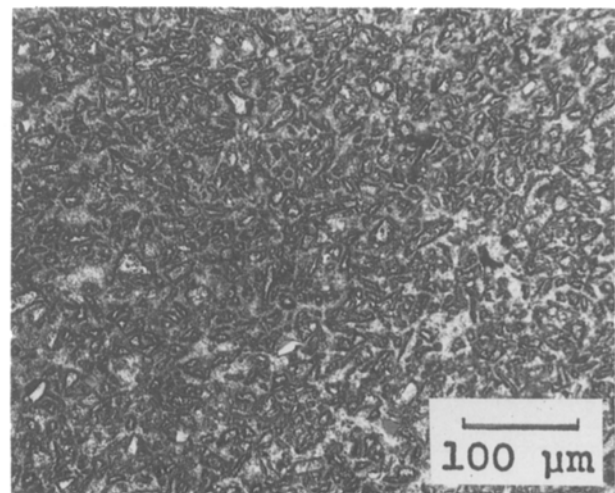

Figure 12 Photomicrograph of SiC particle (400 mesh) reinforced pure aluminium.

TABLE I The D_{cp} and V_f values of alumina and silicon carbide particulate materials with different nominal particle sizes

Particulate materials	Al_2O_3				SiC			
Particle size (μm)	5	1	0.3	0.05	23.4	29.4	19.4	11.3
mesh	–	–	–	–	240	320	400	600
D_{cp}(g cm^{-3})	2.22	0.95	0.88	0.68	2.10	2.38	2.11	1.86
V_f*	0.57	0.24	0.23	0.18	0.65	0.74	0.65	0.58

* V_f under a closely packed condition.

The mean diameter of the 5 μm alumina particles is larger than the three others, but it has a wide size distribution — from large particles 125 μm in size to small ones less than 0.9 μm in size. The D_{cp} value is twice as high as the others (Table I). As a result, the width of the gaps between particles are very narrow, as confirmed by the photograph in Fig. 13. The pressure of 1230 psi was not enough to make a sound composite in this case.

We repeated the test with four SiC particulate materials with different sizes. It is interesting that all the V_f values are higher than those of the alumina particles, but no porosity was observed in SiC reinforced aluminium. It can be explained that SiC particles can wet the aluminium melt more easily than alumina particles can. As a result, a SiC particulate preform with a high V_f value can be completely infiltrated by the aluminium melt under a low pressure.

The strength and deformability of the particles are also important in processing. The particulate preform made of soft graphite powder (120 mesh or smaller) lacked stiffness, so when the aluminium melt applied a force on it, the soft particles were squeezed together and all the gaps disappeared and the porous preform could not be infiltrated and became a solid lump. However, if graphite flakes of a size of less than 500 μm were used to make a preform, the aluminium melt can infiltrate the preform completely (Fig. 14).

4.4. SiC whisker reinforced aluminium
The whiskers are β-SiC (cubic) and are stoichiometric, with impurities less than 1000 p.p.m. The whisker diameter is 1 to 3 μm; the whisker length is 30 to 200 μm. The technique to prepare a whisker reinforced metal is almost the same as for a short fibre reinforced metal.

Fig. 15 shows the SiC whisker preform ($V_f = 0.15$). Fig. 16 shows the microstructure of SiC whisker reinforced pure aluminium. The composite was made under 1778 psi at 660.9° C (T_p). No change in the preform thickness along the pressing direction was observed.

5. Discussion
The new method described in this paper for the fabrication of metal–matrix composites combines the essentials of three liquid-phase fabrication methods: (i) vacuum infiltration, (ii) infiltration under an inert gas pressure, and (iii) squeeze casting.

A major advantage of the new method over squeeze casting is the much smaller preform compression associated with the new method. This stems from the much lower rate of pressure increase used in the new method. A second advantage of the new method over squeeze casting is the lower melt temperature that the new method can provide. A third advantage is that the new method is more versatile, being applicable to particulate, short fibre, continuous fibre and whisker reinforcements, whereas, to our knowledge, squeeze casting can be applied to short fibre and whisker reinforcements only. The third advantage of the new method over squeeze casting stems from the higher temperature for the chamber in the new method; this allows the liquid metal to penetrate the small pores in the preform more effectively.

The main disadvantage of the new method over squeeze casting is the lower cooling rate which enhances grain growth in the metal and might even
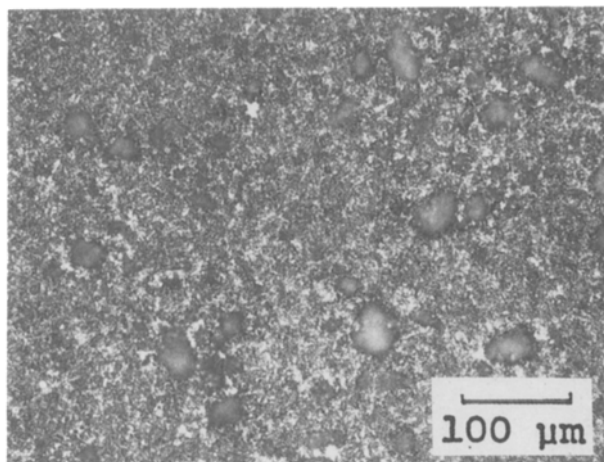

Figure 13 Photomicrograph of Al_2O_3 particle (5 μm size) reinforced aluminium. It was difficult to find passages for the aluminium melt to penetrate due to the wide particle size distribution of the reinforcement.

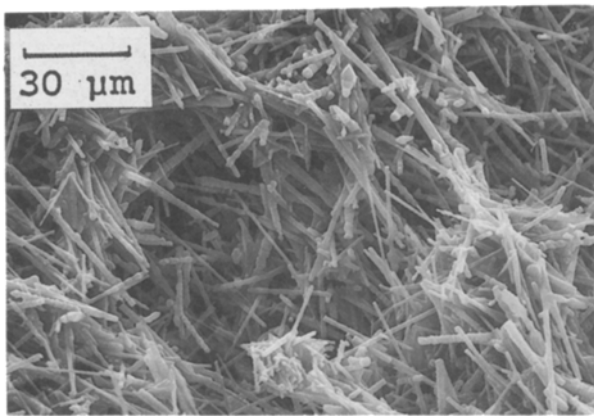Figure 14 Photomicrograph of a graphite flake particulate composite.

Figure 15 SEM photograph of a SiC whisker preform.

enhance the interfacial reaction between the metal and the reinforcement. Another disadvantage is the slower rate of production, but this problem can be solved by producing a large composite ingot.

Compared to powder metallurgy, the new method has three advantages. Firstly, the new method can use a metal ingot as the raw material whereas powder metallurgy must use metal powder as the raw material; ingots are cheaper and purer than powders. Secondly, the new method is less expensive because of the much lower pressure that it uses. Thirdly, the new method is more versatile, being applicable to particulate and fibrous reinforcements, whereas powder metallurgy is limited to particulate reinforcements since powder metallurgy requires mechanical mixing of the metal particles and the reinforcement, and the mixing can cause fibre damage.

6. Conclusions

The conclusions are as follows.

(1) Short fibre, whisker, particle and continuous fibre reinforced aluminium alloy can be produced by the new casting process described in this paper.
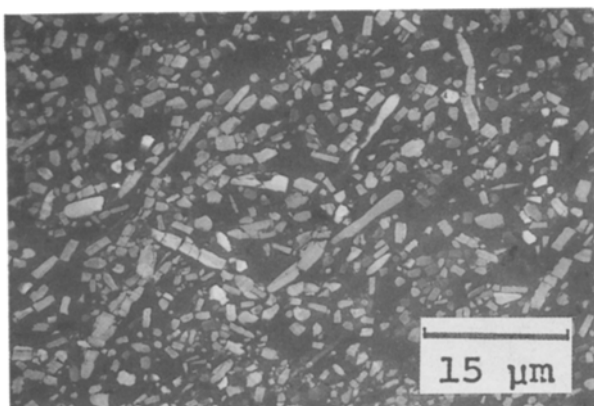

Figure 16 SEM photograph of a SiC whisker reinforced aluminium ($V_f = 0.15$).

(2) Sound composite parts were produced under low temperatures (liquidus or near the liquidus) and low pressures (lower than 2500 psi).

(3) Interface reactions between matrices and reinforcements may be alleviated due to the low temperature and low pressure.

(4) The process can be used as a near-net-shape method to produce composite parts.

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