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Fabrication of cast particle-reinforced metals via pressure infiltration

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A new casting process for fabrication of particle-reinforced metals is presented whereby a composite of particulate reinforcing phase in metal is first produced by pressure infiltration. This composite is then diluted in additional molten metal to obtain the desired reinforcement volume fraction and metal composition. This process produces a pore-free as-cast particulate metal-matrix composite. This process is demonstrated for fabrication of magnesium-matrix composites containing SiC reinforcements of average diameter 30, 10 and 3 μm. It is compared with the compocasting process, which was investigated as well for similar SiC particles in Mg–10 wt % Al, and resulted in unacceptable levels of porosity in the as-cast composite.

1. Introduction

Several casting processes exist for the production of particle-reinforced composites of aluminium or magnesium, such as blending processes wherein particles are incorporated into fully molten metal [1–9], the "compocasting" process in which particles are added and stirred into partly solid metal [10–17], and co-spraying processes whereby metal and particles are splattered simultaneously on to a chill to build a composite of finite thickness [18–21]. Compared with alternative processes such as powder metallurgy, these casting processes are attractive from an economic standpoint, but present a recurrent problem in the presence of a relatively high level of porosity in the as-cast composites, often representing several volume per cent of the cast material. This porosity renders secondary processing such as hot working, squeeze-casting or hot isostatic pressing a necessity if acceptable mechanical properties are to be obtained in the final composite material.

In comparison, casting processes for fabrication of fibre-reinforced metal composites, usually one or another version of the pressure-infiltration process (such as squeeze-casting), are both equally economical and capable of producing virtually pore-free composites. Applying these processes for the fabrication of particle-reinforced metal is, however, precluded by the need for the reinforcement to stand mechanically on its own prior to infiltration, in the form of a silica-bonded preform, of a heap of touching ceramic particles, or of a bundle of close-packed parallel fibres. With particulate metal-matrix composites, therefore, the only reinforcement volume fraction that can be infiltrated without significant use of binder material is that of natural packing of the particles into a container, usually around or above 50 vol %.

The work presented here draws on these facts to propose a novel casting process for fabrication of particle-reinforced composites. The two-step process described here consists in (i) the production of a pore-free composite of particle-reinforced metal of high volume fraction by infiltration, followed by (ii) dilution of the resulting melted or remelted composite into more metal. This process is capable of producing economically a pore-free as-cast composite of particles distributed homogeneously in a metal such as aluminium or magnesium.

In what follows, we demonstrate the process on magnesium-matrix composites reinforced with silicon carbide particles. We also report here results of experiments on compocasting of similar composites, for comparison with the new process and to prove that pores in the compocast magnesium alloy matrix particulate composites result from the incorporation of the particles and not stirring of the melt.

2. Experimental procedure

2.1. Compocasting

The apparatus used in the semi-solid slurry processing investigations is drawn schematically in Fig. 1. It consists of a fused quartz tube (52 cm long, 10 cm o.d., 9.5 cm i.d.) held between brass platens which provide support and enclose the tube. The bottom plate contains feed-throughs for power, gas, connection to a vacuum pump, and a thermocouple. The top plate supports a smaller removable plate which contains feed-throughs for thermocouple, particle entry, and the stirrer shaft. The system was designed to permit processing under an inert gas to prevent oxidation of the melt while allowing observation of the melt surface.

The crucible is constructed of 416 stainless steel to reduce oxidation of the crucible and nickel contamination of the melt. It is firmly supported by two stainless steel dowels, which fit into slots in its base to prevent any movement during stirring while allowing rapid removal of the crucible for casting. A small

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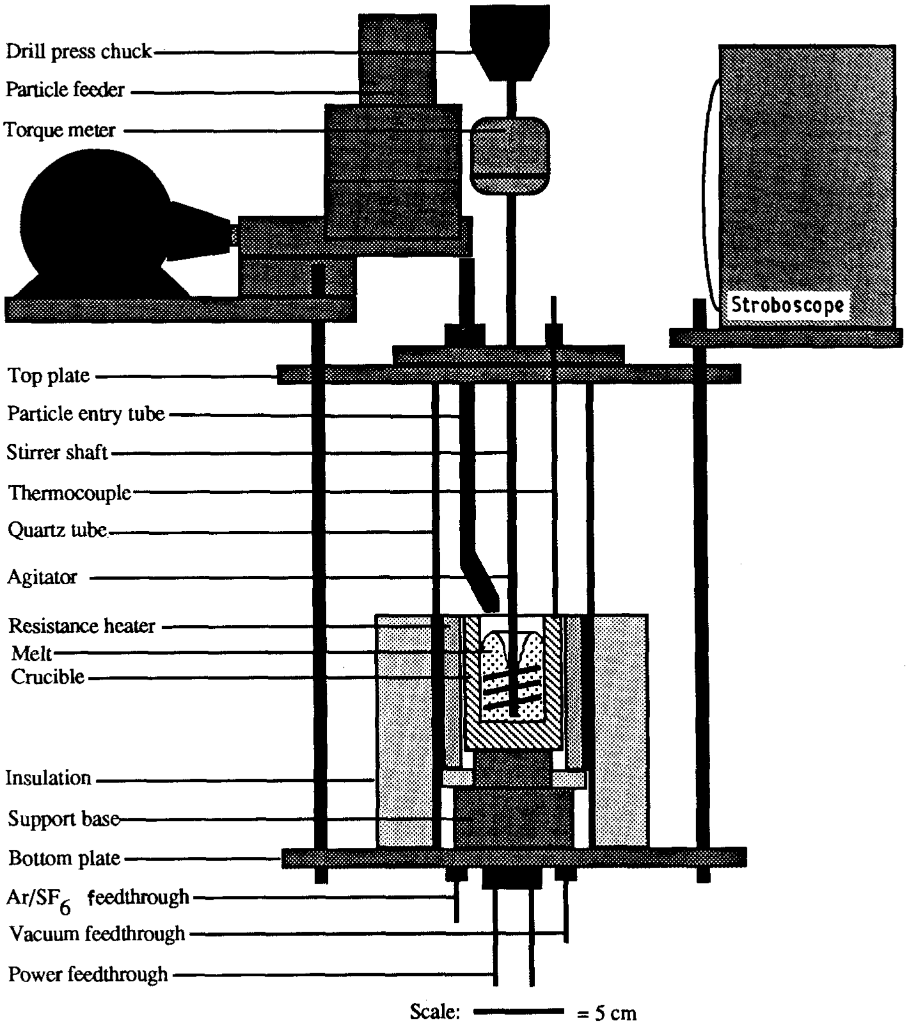

Figure 1 Schematic drawing of the experimental apparatus used for the compocasting process investigation.

resistance furnace (10.1 cm high, 9.1 cm o.d., 6.5 cm i.d.) located approximately 15.6 cm below the top plate surrounds the crucible. A thermocouple located within the wall of the crucible allows accurate temperature measurement and control while reducing possible thermocouple damage from the molten metal or the stirrer. The quartz tube assembly is mounted on a modified $\frac{3}{4}$ hp drill press which acts as a sturdy base while providing variable speed stirring and vertical movement of the stirrer.

The apparent viscosity of the melt was evaluated using a torque meter mounted in line between the drill press and the stirrer (which measures running torque through the use of a pre-calibrated torsion-spring). The running torque was determined by reading the deflection of the torque meter during stirring using a stroboscope. The storoboscope also provides measurement and control of the stirrer speed.

To determine the apparent viscosity of the melt from torque measurements, viscosity standard No. S600 was used. This viscosity standard exhibits a viscosity range of 2000 to 20 cP (mPa sec) as the temperature is varied from 293 to 373 K. The viscosity standard was stirred over a range of temperatures and the torque at several temperatures was determined. Using the equation

$$\mu_a = NT \qquad (1)$$

where μ_a is the apparent viscosity, N a geometrical constant for the stirrer and T the torque, the value of the geometrical constant N was determined to be 1.054 for torque measured in N m and the apparent viscosity in cP.

A review of mixing technology literature [22–34] showed that for non-Newtonian thixotropic fluids, the most rapid and efficient mixing is provided by helical ribbon mixers. According to the literature, their advantages include high shear at the walls even at low speed and excellent top-to-bottom movement of the fluid leading to creation of a vortex. Such a stirrer was constructed, consisting of two concentric sets of double helical impellers 180° apart, with one set on the outside and the other on the inside of the stirrer, of opposite pitch and fixed on a 6.5 mm diameter stirrer shaft. When rotated in a counter-clockwise direction the outer ribbons pump the fluid upward and the inner screws pump the fluid downward. This creates a vortex at the surface which entrains particles dropped from above into the melt and provides extensive top-to-bottom flow for adequate mixing of the slurry.

Delivery of particles to the surface of the melt is provided by a particle feeder consisting of a feeder screw rotated by a variable-speed motor. Particles are held in a hopper above the screw and an electronic vibrator mounted above the hopper aids the flow of the particles. The particle feeder is thus able to provide a consistent rate of particle delivery to the melt surface over a range of feed rates.

An Mg–10 wt % Al alloy was chosen as the matrix material. The high-purity green silicon carbide (SiC) particulate reinforcements used in this investigation were provided by the Norton Company.

Initial experiments on the rheological characterization of the matrix alloy involved melting an appropriate volume ($\sim 86\,\text{cm}^3$) of the matrix alloy. The stirrer was inserted and the temperature lowered. Agitation of the melt was begun when the temperature was just above the melting point of the alloy and was continued as the temperature of the melt was continuously dropped. The apparent viscosity of the slurry was then measured as a function of shear rate (i.e. stirrer speed) for a constant cooling rate and as a function of cooling rate at a constant shear rate.

Specimens of the stirred materials were sucked into a copper chill mould to allow investigation of the microstructure. In this manner, the microstructure and rheology of the rheocast or compocast material could be evaluated during experiments. The incorporation of SiC particles into the semi-solid slurry was studied by stirring the alloy taken into its semi-solid temperature region and by dropping particles on the surface of the melt via the particle feeder. The feed rate of the particles was varied in order to characterize the rate at which the particles could be incorporated into the melt as a function of shear rate and volume fraction of solid phase present. The composites produced in this manner were cast after heating to a temperature above the alloy liquidus.

2.2. Infiltration/dispersion process

2.2.1. *Infiltration*

The pressure-casting apparatus permits application of a vacuum to the particles before infiltration, and is drawn schematically in Fig. 2. It consists of an 83.2 cm long, 2.9 cm i.d. stainless steel pressure vessel. The bottom section of the vessel lies within a tube furnace. A cap, placed on top, contains a thermocouple feed-through to provide temperature measurement and support for movement of the specimen. Two other feed-throughs to the pressure vessel allow application of the vacuum and pressurization via nitrogen gas.

The specimen is contained within a stainless steel crucible (28.6 cm high, 2.6 cm o.d., 2.3 cm i.d.) in which the particles are packed to a height of about 6.5 cm. A slug of commercial-purity magnesium, 1.6 cm diameter by 13 cm long with Dow No. 230 flux on top, is placed on top of the powder and the entire crucible is inserted into the pressure vessel which is preheated to melt the metal. The thermocouple lies approximately

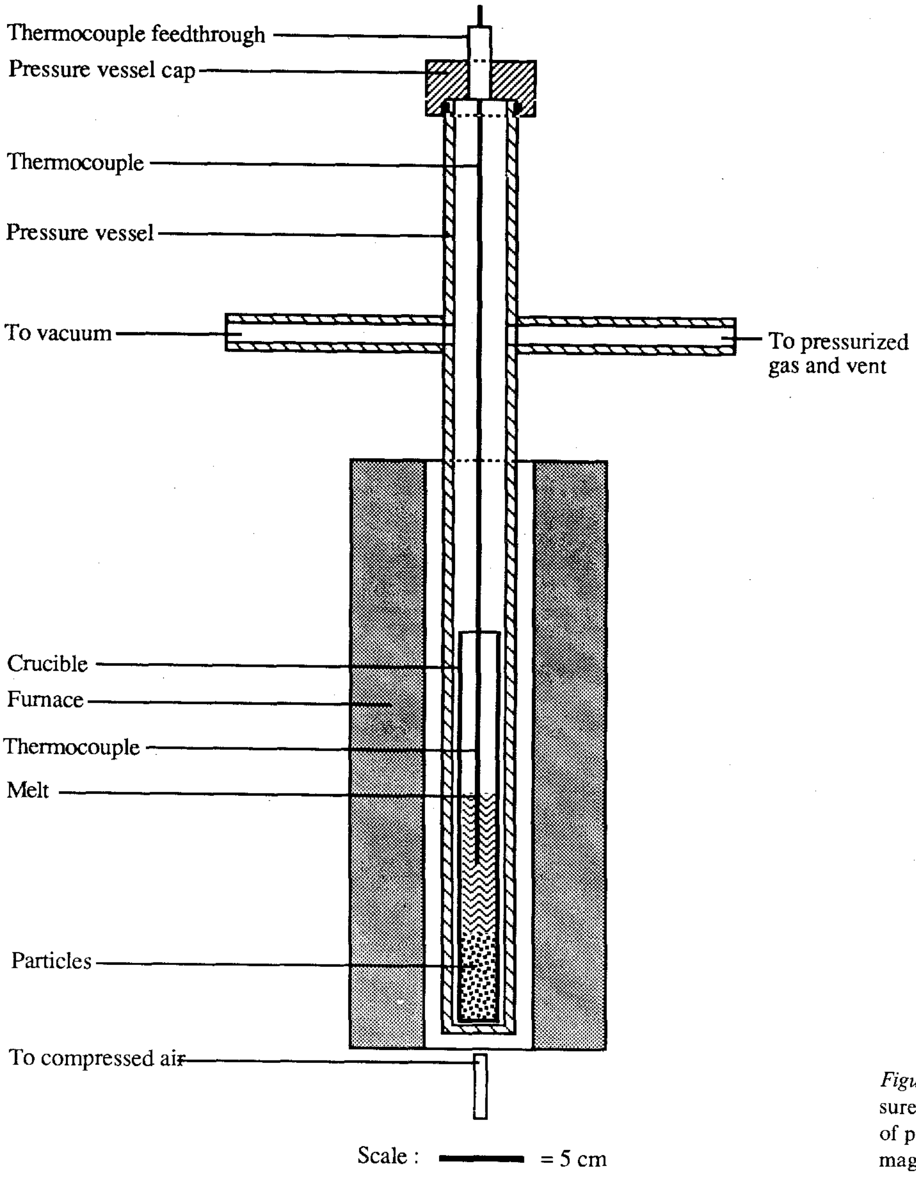

Figure 2 Schematic drawing of the pressure-casting apparatus used for infiltration of packed beds of SiC particles by molten magnesium.

1.3 cm above the top of the packed powders. A vacuum is applied after insertion of the crucible. After about 30 min the melt and particle temperature are high enough to permit infiltration, which is accomplished around 973 K. The vacuum is then closed off and a solenoid valve is opened which allows pressurization of the tube to about 7 MPa in less than 5 sec. Pressure is maintained at temperature for 5 min, then air cooling is applied to the bottom of the pressure vessel to permit directional solidification of the infiltrated composite. After about 20 min the composite is solidified. The pressure is then released and the specimen removed.

2.2.2. Dispersion of high volume-fraction composite

The apparatus used to disperse the high volume-fraction composite into liquid metal to obtain a lower volume fraction of reinforcement is shown schematically in Fig. 3. It consists of a small resistance furnace in which the 416 stainless steel crucible used in the semi-solid slurry studies is placed. This assembly is supported in the modified $\frac{3}{4}$ hp drill press which provides variable-speed stirring and vertical movement of the stirrer. Dispersion is provided by a high-shear agitator consisting of a small turbine which, when rotated at high speed (2000 to 3000 r.p.m.), produces a high shear stress in the agitated fluid. A system of baffles is also included to eliminate significant vortexing and the resulting gas entrapment in the melt.

The dispersion procedure consists of preheating a sand-blasted graphite-coated crucible to 1023 K. A slug of commercial-purity magnesium, about 75 g, is placed in the furnace under a high-purity argon cover gas. When molten, the appropriate amount of high volume-fraction infiltrated composite material for the final volume fraction desired, usually 10 to 15%, is placed in the molten metal. The charge is allowed to heat up for about 5 min. At this time the preheated stirrer and baffle system, supplied with argon cover gas, is placed on the crucible. The melt, now maintained at about 973 K, is stirred for 5 min. The stirrer is then immediately removed and the melt cast on to a copper chill mould (1 cm deep × 5.2 cm wide × 10.4 cm long).

2.3. Metallography

Cast specimens were sectioned on a low-speed saw with a diamond wheel, mounted and ground flat with 600 grit SiC paper. Polishing was with diamond paste using polishing oil as a lubricant on a hard, perforated synthetic cloth. Each polishing step was followed by ultrasonic cleaning in pentane to prevent corrosion of the matrix. The porosity volume fraction was assessed by measuring pore surface area on micrographs using automated image analysis equipment.

3. Results

3.1. Compocasting

3.1.1. Matrix alloy rheology

The effect of cooling rate and stirrer speed on apparent viscosity at a given temperature was found to be relatively small. Variations in apparent viscosity of 50% were found for cooling rates of 0.1 to 4 K min^{-1} at a stirrer speed of 400 r.p.m. Likewise, stirrer speeds from 400 to 600 r.p.m. resulted in apparent viscosity variations around 30% at a cooling rate of 0.5 K min^{-1}. The effect of decreasing temperature was much more pronounced, and apparent viscosities as high as 350 cP were observed at 850 K. The unreinforced matrix microstructures were typical of rheocast alloys, consisting of large "nodules" of the magnesium-rich primary phase within a fine degener-

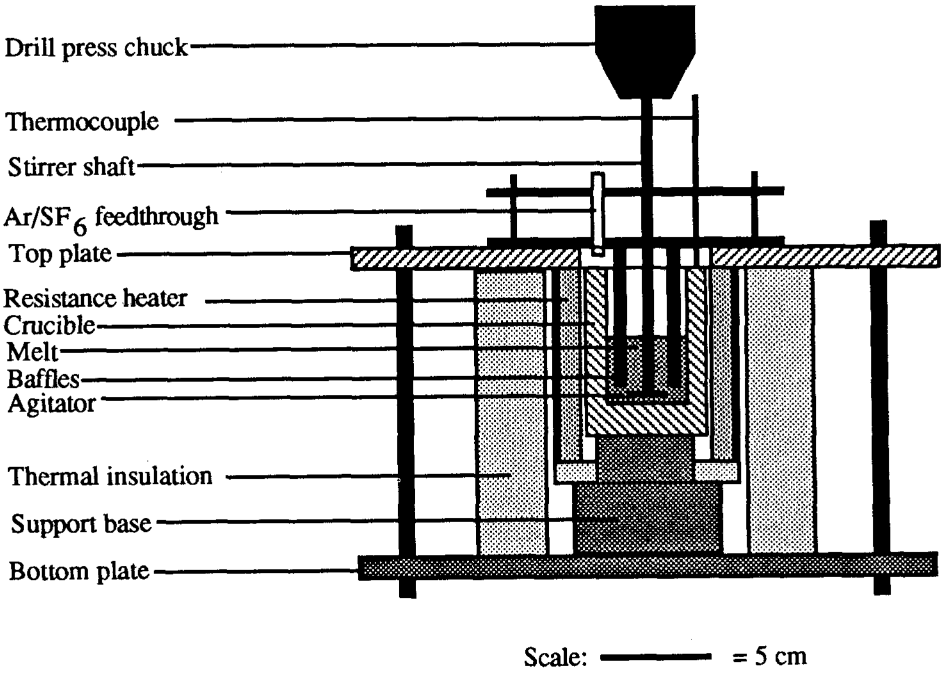

Figure 3 Schematic drawing of the apparatus used for dilution and dispersion of infiltrated high volume-fraction composites into additional molten metal for production of a lower volume-fraction particulate composite of SiC in magnesium.

ate dendritic structure which was liquid at the time when the specimen was quenched. The rheocast alloy did not exhibit porosity at normal stirrer speeds despite the formation of a vortex.

3.1.2. Composite fabrication

A processing window was found to exist for optimized particle incorporation in the compocasting process. One limit of this window was determined by the need for a sufficiently high viscosity of the slurry and a sufficiently pronounced vortex to break up the oxide layer on the surface of the melt. At that time the surface of the melt became shiny and particles dropped on to the melt were readily incorporated. This occurred at stirrer speeds in excess of 250 r.p.m. and volume fractions of solid phase (primary magnesium and SiC) of about 20 vol %. The other limits of the processing window were defined by (i) stirrer speeds at which melt splashing and very significant gas entrapment resulted, above 650 r.p.m., and (ii) the attainment of too high a melt viscosity, above 300 cP, above which the melt clung to the stirrer and stopped flowing. It was found that as the particle feed was increased above 0.2 g sec^{-1}, with all other processing parameters constant and within the processing window, the feed rate became greater than the rate of particle incorporation, resulting in the formation of large particle clusters on the melt surface and often resulting in oxidation of the metal. This maximum rate of successful particle addition was rather insensitive to the value of the processing parameters within the processing window defined above.

Within this processing window, the material produced still showed significant porosity. The volume fraction of pores was found to increase as the volume fraction of particulate reinforcement increased (Fig. 4). The porosity was significant, and pores were usually surrounded by particles (Fig. 5). Particle clusters were also present, and were usually associated with pores or entrapped oxide films. Such clustering was much worse when smaller particles were incorporated.

The viscosity of the resulting composites was measured after remelting the characterized compocast material. It was found that the resulting curves of viscosity against temperature showed a positive deviation over the corresponding curve for the unreinforced matrix, by an amount on a par with, but no higher than, that observed by varying cooling rate or shear rate on the rheocast matrix alone.

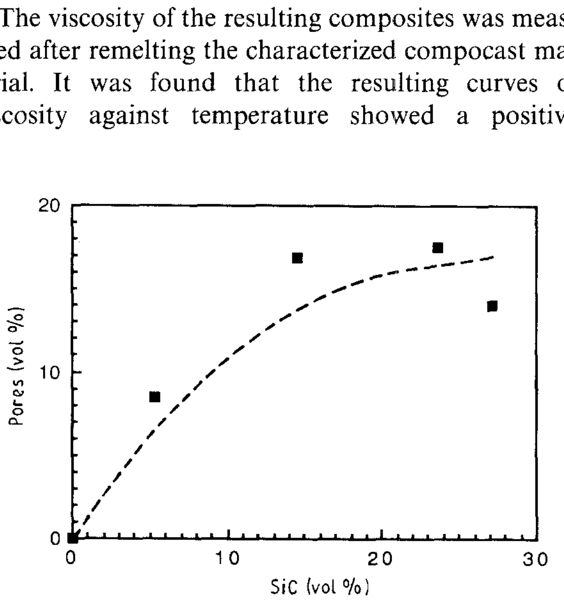

Figure 4 Porosity in compocast particulate composites of 30 µm SiC in Mg–10 wt % Al as a function of final content of SiC.

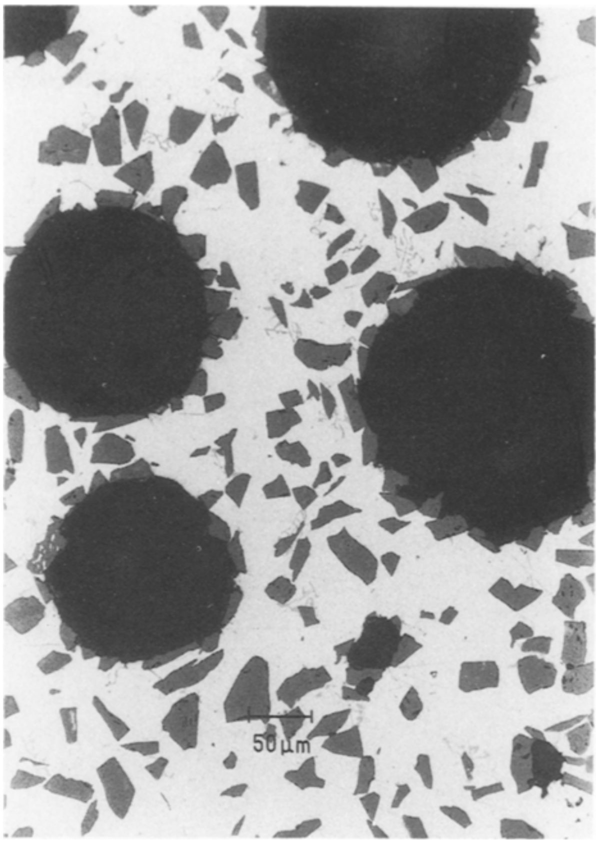

Figure 5 Typical pores in compocast particulate composite of 30 µm SiC in Mg–10 wt % Al.

3.2. The infiltration/dispersion process

Close-packed beds of 30, 10 and 3 µm average diameter SiC particles were successfully infiltrated with commercial-purity magnesium. On occasion, small regions of macroscopically observable porosity were found near the top of the specimens. Otherwise, the composites were sound, devoid even of small discrete pores. A second phase was found within the matrix, usually at the centre of the composite and in greater quantities with finer particles. A typical microstructure of the infiltrated composite is given in Fig. 6 for SiC particles of 10 µm average diameter.

When heated above the melting point of the matrix material, the infiltrated composites became mushy and were found to break down easily under a small pressure applied manually with a stainless steel rod. In dispersing the infiltrated composites into more metal, stirrer speeds of 2500 r.p.m. (corresponding to a stirrer tip tangential speed of 2.5 m sec^{-1}) appeared adequate to eliminate clusters in the resulting lower volume-fraction composites with the larger particles, 30 µm in average diameter. Cluster elimination was more difficult with finer particle sizes. The intermetallic present in the initial infiltrated composite produced an aligned structure in the dispersed composite, resembling second phases found between columnar dendrites. With

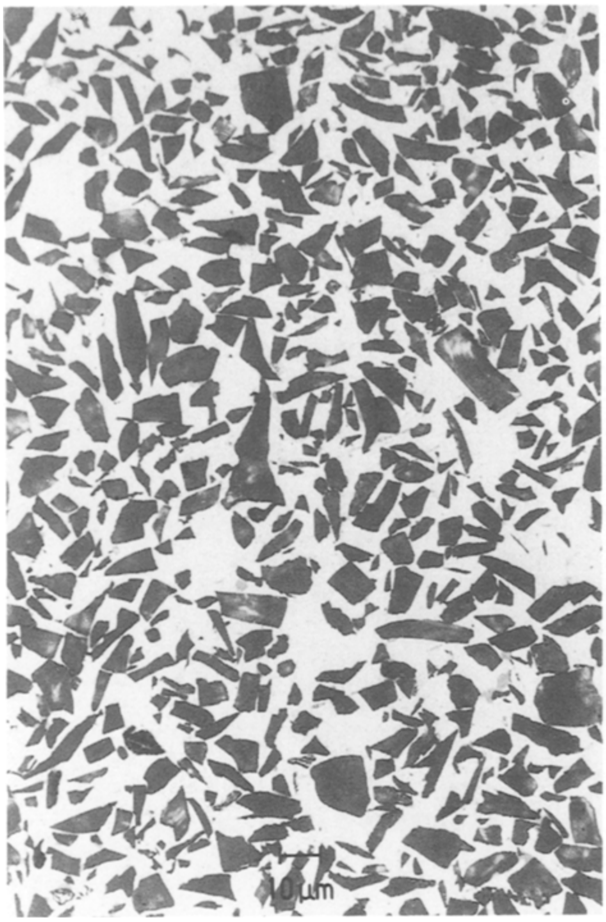

Figure 6 Typical microstructure of a 10 μm SiC particle–commerical-purity magnesium composite produced by pressure infiltration. The volume fraction of SiC is 48%.

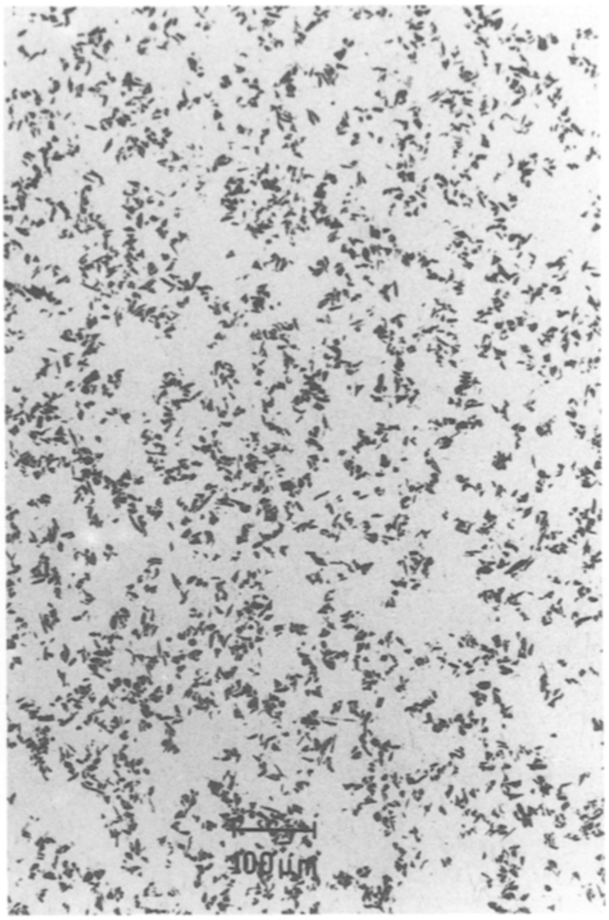

Figure 7 Typical microstructure of a 10 μm SiC particle–commercial-purity magnesium composite produced by pressure infiltration and dispersion into more metal. The volume fraction of SiC is 11%.

proper foundry practice (including sand-blasting the crucible before each dispersion experiment and preheating the high-shear agitator and baffle system), porosity was virtually absent from the cast, dispersed composites. The particles were reasonably well distributed throughout the matrix (Fig. 7) although some small regions with no particles did exist. The quality of dispersion of the particles varied within the samples, and in some regions was remarkably good (Fig. 8). Finally, it was found that the 30 μm SiC particles settled very rapidly, resulting in a significant particle volume-fraction variation across the roughly 1 cm thick chill-cast composite. Settling of the 10 μm or 3 μm SiC particles was not observed under similar casting conditions.

4. Discussion

4.1. Semi-solid slurry processing

Over the range of temperatures at which the Mg–10% Al slurries could be stirred, the effect of shear rate and cooling rate was rather small, but did, along with the corresponding microstructures, follow the expected trends [35, 36].

Observations on the inclusion of particulates clearly revealed a window, within the range of shear rates and temperatures investigated, where particles dropped on the surface were incorporated into the slurry. Variations in the window as a function of shear rate and cooling rate were not observed, presumably because the rheology of the system is relatively insensitive to these parameters. Although the connection between volume fraction porosity and volume fraction particles incorporated was not perfectly monotonous (Fig. 4), there is, in general, an increase in porosity as more particles are introduced, in agreement with the data of Ray [10].

Since there was no porosity in the unreinforced rheocast alloy processed using the same apparatus under identical conditions and with similar viscosity as that of the composite, the observed porosity is clearly a consequence of particle incorporation and not of stirring in the compocasting experiments. This is corroborated by the fact that pores were typically surrounded by particles (Fig. 5). This porosity may be inherent in the incorporation of non-wettable particles, or may result in part from gases adsorbed on the particle surface as shown by Yamada *et al.* [17] for aluminium-matrix composites. A simple calculation shows that gas adsorbed on the particle surface can easily account for the high porosity found in the composites: with the coarse 30 μm diameter particles of specific surface 0.18 m^2 (measured by the BET technique), a monolayer of water at room temperature represents 24 l of H_2 liberated after reduction by magnesium per litre of 20 vol % molten SiC–Mg composite.

Figure 8 Region of well-distributed particles in a 30 µm SiC particle–commercial-purity magnesium composite produced by pressure infiltration and dispersion into more metal. The volume fraction of SiC is 16%.

A second limitation found with this processing route is the low rate of particle incorporation: at a maximum particle incorporation rate of 0.2 $g\,sec^{-1}$, the particle feeding time needed to produce a batch of composite with the small apparatus built for this work was around 10 min. This process time would increase with a larger apparatus.

4.2. The infiltration/dispersion process

Since sound high volume-fraction composites were produced in a reliable and consistent manner, pressure infiltration after evacuation of the particles is a viable approach to overcome the poor wettability of the particles. It was found in experiments reported elsewhere that evacuation of gas initially present between the particles is a necessary step for production of sound infiltrated composites [37]. The second phase within the matrix of the infiltrated composites was determined by energy-dispersive X-ray analysis on a scanning electron microscope to contain magnesium and silicon and is therefore most likely Mg_2Si. This second phase thus appears to result from silicon in the SiC reinforcement, which is present both as SiO_2 at the particle surface and as free silicon within the SiC, and which is known to be readily reduced by molten magnesium. This interpretation is corroborated by the observation that the amount of second phase within the matrix increased as the particle size decreased, and hence as the specific surface of the reinforcement increased. The microscopic morphology of this second phase was, further, found to agree with that of Mg_2Si in magnesium. This second phase was not found to be deleterious either to the infiltration or to the dilution steps of the process.

The dispersion experiments indicate that dilution of the infiltrated composites can be conducted without great difficulty to obtain a material with minimal porosity. However, the technique used in this work was clearly not optimal, since the average microscopic quality of the dispersion left much to be desired and pores were, on a few occasions, found in the diluted composites. The fact that in some regions of the composite a microstructure of the quality shown in Fig. 8 can be obtained proves, however, that with more sophisticated apparatus, preferably diluting the infiltrated composite in a continuous fashion rather than in a batch process, a pore-free dispersed composite with excellent microscopic particle distribution can be cast.

In closing, it is noted that a limitation of this process exists in that the infiltrated composites may be impossible to dilute if the particles react with the matrix metal to the extent that they are bonded together by the reaction product formed after infiltration. This was found to be the case for boron carbide in magnesium and SiC in aluminium melts unalloyed with silicon, but not for SiC in Mg–10 wt % Al (in this last case, aluminium carbide forms at the particle–matrix interface) [37].

5. Conclusions

1. Studies of the compocasting process for SiC particle-reinforced Mg–10 wt % Al revealed the presence of a window for optimized processing. Within that window, however, all samples contained unacceptable levels of porosity which was shown to result from particle incorporation and not stirring of the semi-solid material.

2. An alternative process based on infiltration of a packed bed of SiC particles with magnesium, followed by dispersion into more metal, was shown to produce a virtually pore-free as-cast composite while allowing control of the volume fraction of reinforcement within the metal.

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