JOURNAL OF MATERIALS SCIENCE 33 (1998) 4707–4709

Low-volume-fraction particulate preforms for making metal-matrix composites by liquid metal infiltration

YUNSHENG XU, D. D. L. CHUNG
Composite Materials Research Laboratory, State University of New York at Buffalo, Buffalo, NY 14260-4400, USA

A preform technology for making particulate metal-matrix composites with a low particulate volume fraction (as low as 18%) by liquid metal infiltration is provided. This technology used a non-combustible reinforcement (SiC) as the primary particulate and combustible particles (carbon) as the secondary particulate in the preform. The secondary particulate was removed from the preform by oxidation prior to liquid metal infiltration.

1. Introduction

Metal-matrix composites are attractive for their high modulus, high strength and low thermal expansion. They are most successfully made by either liquid metal infiltration or powder metallurgy; stir casting tends to suffer from non-uniformity in the reinforcement distribution after solidification [1–22]. In liquid metal infiltration, liquid metal is allowed to infiltrate a porous preform consisting of the reinforcement (particles, whiskers or fibres) and a small amount of a binder (e.g. silica). Upon subsequent solidification of the liquid metal, a metal-matrix composite is obtained. In powder metallurgy, the metal matrix in powder form is mixed with the reinforcement and the mixture is compacted and subsequently sintered to form the composite. Due to the very higher pressure required for powder metallurgy compared with liquid metal infiltration, liquid metal infiltration is economically more viable. In the case of a particulate reinforcement, liquid metal infiltration suffers from being restricted to composites with a high volume fraction (>50%) of reinforcement. Due to the decrease in ductility of the composite with increasing reinforcement volume fraction, a low volume fraction is desirable unless a very high modulus or a very low thermal expansion is required. This restriction stems from the difficulty of making particulate preforms with a low particulate volume fraction, since the particles naturally want to touch one another in the preform. On the other hand, powder metallurgy is restricted to composites with a low volume fraction, since the volume fraction of the metal matrix must be sufficient for the matrix powder to spread out and bind the composite together. Stir casting is also restricted to composites with a low volume fraction.

This paper provides a preform technology for making particulate metal-matrix composites with a low particulate volume fraction (as low as 18%) by liquid metalinfiltration. This technology uses a non-combustible reinforcement (i.e. SiC particles) as the primary particulate in the preform and a combustible (removable by oxidation) particulate (i.e. carbon particles) as the secondary particulate in the preform. A preform is made using a mixture of the primary and secondary particulates, together with a small amount of binder. Then, the secondary particulate in the preform is removed by oxidation. Subsequently, the preform is infiltrated with a liquid metal to form a metal-matrix composite with a low volume fraction of reinforcement.

2. Experimental procedure

An acid phosphate binder [13–15, 17, 18] was used for fabricating preforms comprising SiC particles and carbon particles. The binder solution was prepared by mixing one part of aluminum hydroxide, $Al(OH)_3$, with phosphoric acid, H_3PO_4 (85%), so that the solution had a P : Al molar ratio of 23. The mixture, consisting aluminum hydroxide and phosphoric acid, was stirred and heated to 140 °C. It was held at 140 °C until all the aluminum hydroxide was dissolved and a clear solution was obtained.

SiC particles (Green 1200, obtained from Electro Abrasives Corp., Buffalo, NY, size 3–5 μm) and carbon particles (C-30, activated carbon, 20 μm mean particle size, equiaxed, obtained from Osaka Gas Chemicals Co., Ltd, Osaka, Japan) were mixed in a predetermined ratio in a ball mill for 12 h, using acetone as the medium for mixing. Afterwards, the mixture was dried at room temperature for one day and then at 110 °C for 12 h. The weight ratio of C : SiC was 2.1 : 15, 1.5 : 15 and 1.0 : 15 for preforms 1, 2, and 3, respectively.

The preforms were prepared by wet forming, which involved compressing in a steel die a slurry containing the powder mixture, a liquid carrier (water) and the phosphate binder. The die allowed excess liquid to be squeezed out from the slurry, so that a wet cake was formed. The binder and carrier (water) were in the ratio 1 : 40 and the total amount of binder in the preform was less than 0.16 wt %. The compressive pressure during wet forming was 1.8 MPa for all preforms. The preforms were cylindrical in shape, 4.0 cm in diameter, with a height-to-diameter ratio of 0.3–0.5. After removal from the die, the wet cake was dried in a fume hood at room temperature for 72 h and then the preform was dried and oxidized by

1. Placing the preform in a furnace at room temperature.
2. Heating to 120 °C at a controlled rate of 1 °C min^{-1}.
3. Holding at 120 °C for 100 min.
4. Heating to 510 °C at the rate of 1 °C min^{-1} and holding at 510 °C for 120 min.
5. Heating to 750 °C in an oxygen gas flow (50 ml min^{-1}) at 1 °C min^{-1}.
6. Holding at 750 °C in the oxygen flow for 120 min.
7. Cooling in the closed furnace at a controlled rate of 1 °C min^{-1} to room temperature.

Steps 1–4 are for drying and binder treatment [13–15], whereas steps 5–7 are for oxidation (i.e. burning off the carbon in the preform).

Liquid metal infiltration was performed by (i) evacuation, (ii) melting the aluminum (pure, No. 170.1) ingot placed above the preform, and (iii) using argon gas to push the liquid aluminum into the preform. The detailed method is described below. The preform was placed at the bottom of a steel mould. Above the preform was placed an aluminum ingot. The mould chamber was then sealed and evacuated to a pressure of 30–40 Pa. Then evacuation was stopped and the chamber was filled with argon until a pressure of 1.4 MPa was reached. After this, the outlet valve was opened to release the argon and then the chamber was evacuated to a pressure of 20–30 Pa. Then the chamber was again filled with argon to a pressure of 1.4 MPa. In this way, three evacuation cycles were conducted in order to minimize the amount of residual air in the chamber. The final evacuation was to a pressure of less than 13.4 Pa. The chamber was then heated at a rate of 20 °C min^{-1} to 630 °C, maintained at 630 °C for 30 min, and then heated to 805 °C at a rate of 10 °C min^{-1}, while evacuation continued all the time. The temperature was maintained at 805 °C for 40 min. After 30 min within the 40-min period, evacuation was stopped and argon was introduced to pressurize the mould chamber from 10 Pa to 48 MPa; the pressurization took about 5–6 min. At the end of the 40-min period, the temperature was allowed to drop. When the temperature had dropped to 670 °C, both temperature and pressure were maintained for 20 min. When the temperature had dropped to 350 °C, the outlet valve was opened to release argon from the chamber. Then, the mould was taken out of the chamber and immersed in water to cool.

Tensile testing was performed by using a hydraulic mechanical testing system (MTS 810). The displacement rate was 0.5 mm min^{-1}. Dogbone-shaped specimens were obtained by cutting along two parallel planes, which were then double-side grooved to the required dimension. The thickness of the sample was 1.25–1.5 mm. The narrow part of the dogbone shape was 19–20 mm long and 5–6 mm wide. The thick part of the dogbone shape was 9–10 mm wide. A strain gauge (Measurements Group, EA-06-120LZ-120) was used to measure the strain (hence the modulus) of each sample. Four samples were tested for each composite.

3. Results

Fig. 1 shows scanning electron microscope (SEM) photographs to preform 2 before and after burning off the carbon. The larger particles observed before burning are carbon, while the smaller particles are SiC. After burning, the larger particles have vanished. Fig. 2 shows SEM photographs of the composites made from preforms 1, 2 and 3 (all with carbon burnt off), corresponding to SiC volume fractions of 18.0, 31.2 and 38.6%, respectively. The SiC particle distribution is quite uniform in all the composites.

Table I shows the tensile properties of the composites. The strength and modulus increased with increasing SiC volume fraction, while the ductility decreased, as expected. The tensile property values are quite comparable with those of similar composites fabricated by other methods, though comparison is difficult due to differences in both matrix alloy and SiC volume fraction [1–22].

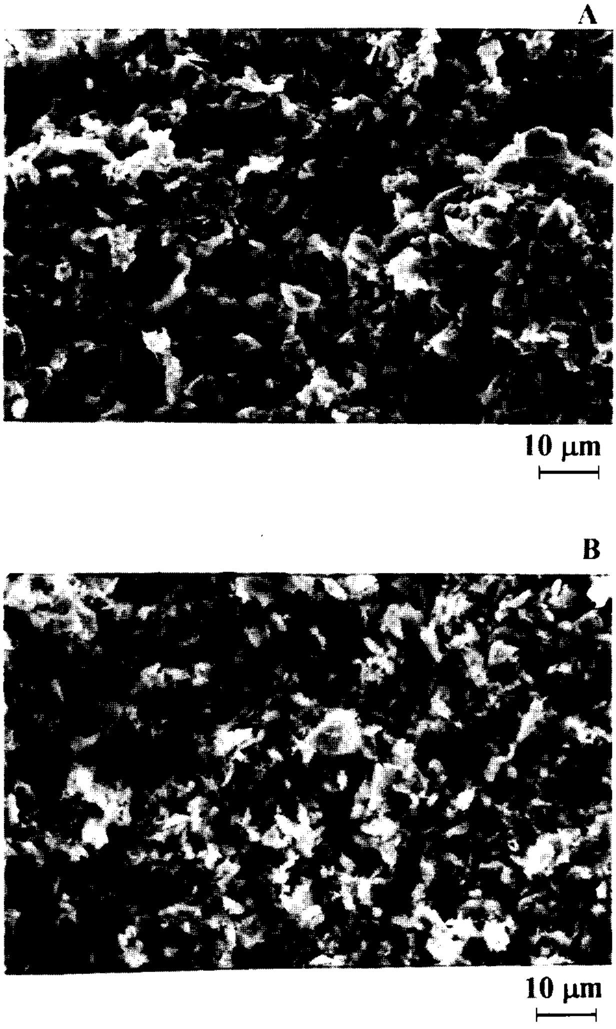

Figure 1 SEM photographs of preform 2 before (a) and after (b) burning off the carbon.

TABLE I Properties of composites

Preform No.	SiC (vol %)	Density ($g\,cm^{-3}$)	Tensile strength (MPa)	Tensile modulus (GPa)	Tensile ductility (%)
1	18.0	2.79	114 ± 11	87 ± 3.7	2.45 ± 0.35
2	31.2	2.86	209 ± 10	121 ± 4.3	1.56 ± 0.27
3	38.6	2.89	241 ± 8	136 ± 5.5	1.32 ± 0.19

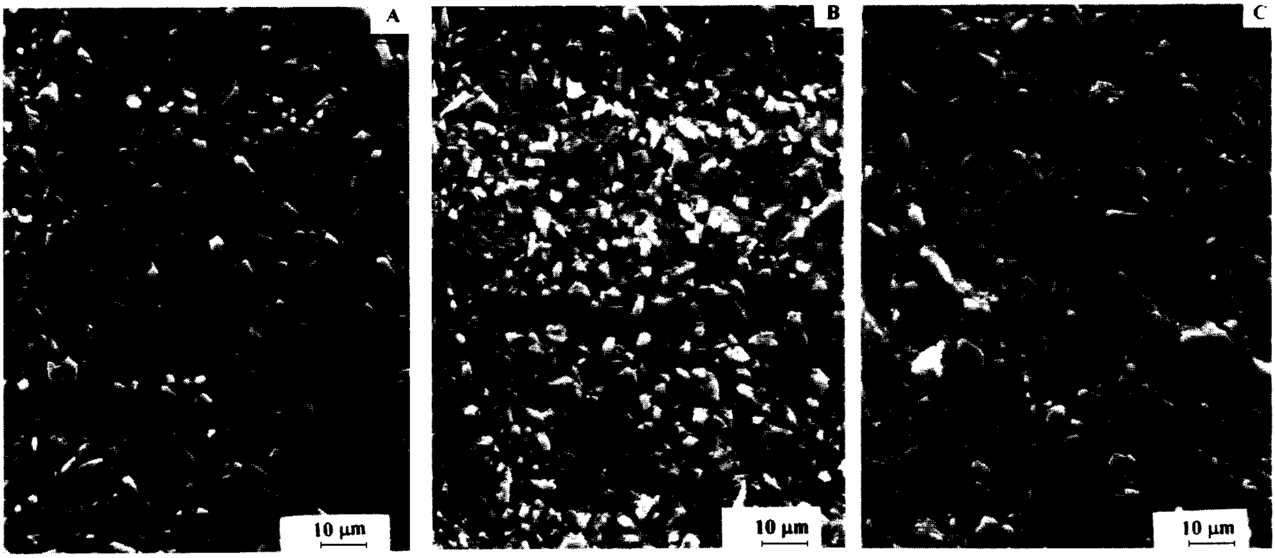

Figure 2 SEM photographs of composites made from preforms (a) 1, (b) 2 and (c) 3.

4. Conclusions

A preform technology for making particulate metal-matrix composites with a low particulate volume fraction (as low as 18%) by liquid metal infiltration is provided. This technology used a non-combustible reinforcement (SiC) as the primary particulate and combustible particles (carbon) as the secondary particulate in the preform. The secondary particulate was removed from the preform by oxidation prior to liquid metal infiltration. The oxidation was conducted by heating the preform at 750 °C in an oxygen gas flow. The weight fraction of carbon particles in the preform prior to oxidation was 6–12% for attaining composites containing 18–38 vol % SiC particles.

References

1. O. T. MIDLING and O. GRONG, *Key Engng Mater.* **104–107** (1995) 329.
2. BOQ-KONG HWU, SU-JIEN LIN and MIN-TEN JAHN, *Mater. Sci. Engng* **A207** (1996) 135.
3. M. GUPTA, L. LU, M. O. LAI and S. E. ANG, *Mater. Design* **16** (1995) 75.
4. H. XU and E. J. PALMIERE, *Mater. Sci. Forum* **217–222** (1996) 1091.
5. T. CHOH, M. KOBASHI, H. NAKATA and H. KANEDA, *ibid.* **217–222** (1996) 353.
6. W. CHUNG and S. LIN, *Mater. Res. Bull.* **31** (1996) 1437.
7. G. S. HANUMANTH and G. A. IRONS, *Met. Mater. Trans. B.* **27B** (1996) 663.
8. X. M. XI, L. M. XIAO and X. F. YANG, *J. Mater. Res.* **11** (1996) 1037.
9. F. M. YARANDI, P. K. ROHATGI and S. RAY, *J. Mater. Eng. Performance* **2** (1993) 359.
10. M. SUÉRY, G. L'ESPÉRANCE, B. D. HONG, L. NGUYEN THANH and F. BORDEAUX, *ibid.* **2** (1993) 365.
11. P. K. ROHATGI, K. PASCIAK, C. S. NARENDRANATH, S. RAY and A. SACHDEV, *J. Mater. Sci.* **29** (1994) 5357.
12. M. VEDANI, E. GARIBOLDI, G. SILVA and C. DI GREGORIO, *Mater. Sci. Technol.* **10** (1994) 132.
13. J. CHIOU and D. D. L. CHUNG, *J. Mater. Sci.* **28** (1993) 1435.
14. *Idem, ibid.* **28** (1993) 1447.
15. *Idem, ibid.* **28** (1993) 1471.
16. J. CHIOU, B.-Y. WEI and C.-M. CHEN, *J. Mater. Eng. Performance* **2** (1993) 383.
17. S. LAI and D. D. L. CHUNG, *J. Mater. Sci.* **29** (1994) 3128.
18. *Idem, ibid.* **29** (1994) 2998.
19. *Idem, J. Mater. Chem.* **6** (1996) 469.
20. *Idem, J. Mater. Sci.* **29** (1994) 6181.
21. Y. CHEN and D. D. L. CHUNG, *ibid.* **31** (1996) 407.
22. P. YIH and D. D. L. CHUNG, *ibid.* **32** (1997) 2873.

Received 17 April
and accepted 18 June 1998