

A comparative study of Al_2O_3 /metal composites processed by melt infiltration and directed metal oxidation

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The reactive melt infiltration (RMI) process and the directed metal oxidation (DIMOXTM) process were used to fabricate Al_2O_3 /metal composites. Pure Al powder was infiltrated into Al_2O_3 preforms with the addition of borosilicate powder for the production of Al_2O_3 /metal composites in the RMI process. In the case of the DIMOXTM process, Al 7039 alloy has been infiltrated into Al_2O_3 preforms to produce Al_2O_3 /metal composites involving the directed oxidation of the Al 7039 alloy. A lower temperature and shorter processing time were required to fabricate Al_2O_3 /metal composites in the RMI process than in the DIMOXTM process.

Key words: Reactive melt infiltration, Directed metal oxidation, Fracture toughness, Fractography.

Introduction

Ceramic/metal composites have been known to be made by infiltrating molten metal into a ceramic preform containing open porosity. Necessary conditions with respect to chemical reactivity, melt viscosity, and wetting must be satisfied in this process. Pressureless infiltration has the advantage of the simplicity of the processing equipment. However, wetting between metal and nonmetal is often poorer than between metals. In some cases, chemical reactions between liquids and solid surfaces were reported to induce good wetting and enhance the infiltration process [1, 2, 3]. Newkirk and coworkers reported that they produced Al_2O_3 /metal composites by directed metal oxidation (DIMOXTM) of a molten Al alloy with suitable alloying elements under the appropriate gaseous atmosphere [4]. However, the growth rate of the composite was very low and the additional alloying process such as the addition of Si and Mg must be conducted to facilitate oxidation of the molten Al alloy. Tai and coworkers [5] reported that they fabricated Al_2O_3 /metal composites by infiltrating pure Al powder melt into Al_2O_3 preforms whilst applying borosilicate powder as an infiltration initiator in an oxygen atmosphere without applying external pressure [5]. In the present study, two different processes of RMI and DIMOXTM were used to fabricate Al_2O_3 /metal composites. The purpose of the present work is to compare the mechanical properties of Al_2O_3 /metal

composites produced by the two different methods and characterize the physical and micro-structural properties.

Experimental Procedures

The fabrication of composites by the DIMOXTM process

Al 7039 alloy was cut to a rectangular bar (12 mm × 6 mm × 60 mm) and polished by abrasive paper (#1500). Al_2O_3 powder (10 mm, 99.99%) was isostatically pressed at 300 MPa to a rectangular ceramic perform (12 mm × 6 mm × 60 mm) for the infiltration. SiO_2 powder was spread onto the interface between the Al 7039 alloy and the Al_2O_3 preform (0.03 g/cm²) as an infiltration initiator that facilitated the directed oxidation infiltration of the Al 7039 alloy into the Al_2O_3 powder perform. The chemical composition of the Al7039 alloy is shown in Table 1. PVA was used as a binder for homogeneous packing. The Al 7039 alloy was used as a source material for the oxidative infiltration process which was conducted at 1100°C for 24 hrs. in a tubular furnace under 1 atmosphere of oxygen.

The fabrication of composites by the RMI process

A pure Al powder (0.25 mm, 99.8%) compact and Al powder compacts containing 4, 8, and 15 wt.% Cu powder were pressed at 50 MPa to the shape of

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Table 1. Chemical composition of the Al7039 alloy employed

Element	Zn	Mg	Mn	Cr	Fe	Si	Cu	Ti
wt.%	3.88	2.50	0.15	0.20	0.24	0.25	0.03	0.04

rectangular bars ($12 \text{ mm} \times 6 \text{ mm} \times 60 \text{ mm}$). Al_2O_3 powder ($10 \mu\text{m}$, 99.99%) was isostatically pressed at 300 MPa to form a rectangular Al_2O_3 ceramic perform for the infiltration of molten Al. Boro-silicate powder (0.03 g/cm^2) was spread on the interface between the Al_2O_3 preform and the metal compact to facilitate melt infiltration. The melt infiltration process was conducted at 964°C in a tubular furnace under 1atmosphere oxygen. The metal-ceramic preform combination was heated in a crucible assembly with a heating rate of 5°C/minute to the processing temperature. After the infiltration, the sample was cooled in the furnace at 3°C/minute .

Microstructural analysis and mechanical properties of composites

Microstructural characterization and fractography of the fabricated composites were carried out by optical microscopy, SEM and TEM. Phase analysis was conducted by X-ray diffractometry and the density of the composites was measured by the Archimedes method. Vickers hardness indentation technique [6] were used to measure fracture toughness. Three-point bending tests (JIS-R1601) were conducted using $3 \text{ mm} \times 4 \text{ mm} \times 36 \text{ mm}$ rectangular bar (20 mm span) type specimens to measure strength.

Results and Discussion

Figure 1 shows the X-ray diffraction patterns of Al_2O_3 /metal composites which were produced by the RMI process and DIMOXTM process. Fig. 1(a) and (b) show the results of the composites infiltrated by pure Al and Al-4 wt.% Cu powder melt respectively in the RMI process. Al_2O_3 and Al were observed to be the main components of the composites and a trace of Si was detected. It was thought that Si dissolved from the interface into the pure Al infiltrated into the perform at the processing temperature and then precipitated in the composites during cooling to room temperature. A CuAl_2 phase which was newly formed in the composite containing Cu addition was observed in Fig. 1(b). It

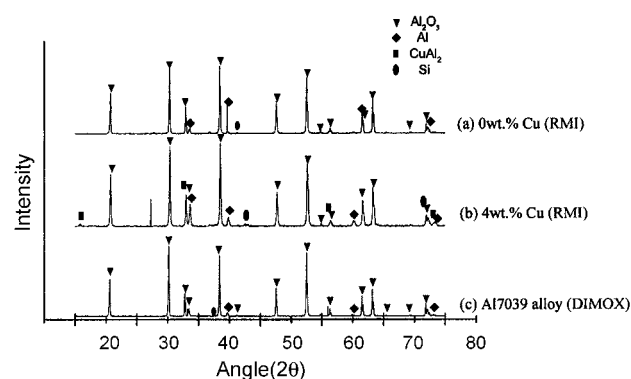
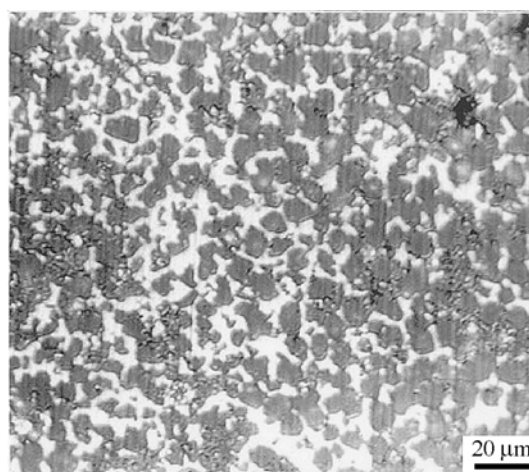
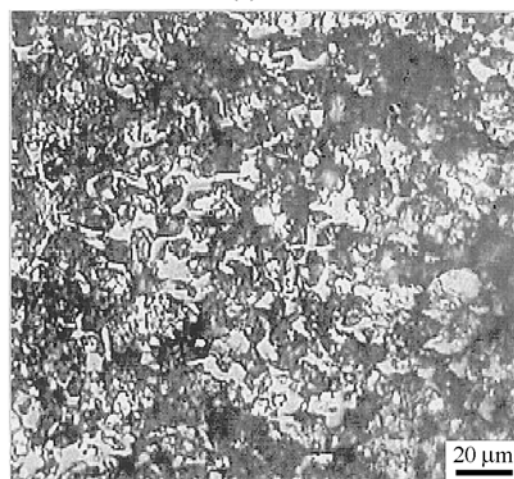


Fig. 1. XRD patterns of the composites fabricated by the RMI and DIMOXTM processes.



(a) RMI



(b) DIMOXTM

Fig. 2. Optical micrographs of Al_2O_3 /metal composite fabricated by the RMI and DIMOXTM processes.

was believed that the formation of the CuAl_2 phase was due to the precipitation from a supersaturated solid solution of Cu in Al at the processing temperature during cooling. The X-ray diffraction pattern of the specimen produced by DIMOXTM process also represented the distribution of phases similar to that in the RMI process and is shown in Fig. 1(c).

Figure 2(a) and (b) show the microstructures of the composites produced by the RMI and DIMOXTM processes, respectively. Typical examples of the channel structure are shown in Fig. 2. The bright channels in their micrograph are the three-dimensionally interconnected channels where the molten metal infiltrated to the reaction front by capillary forces. The formation of metal channels in the composite is known to be affected by various factors such as the filler material, particle size, shape of the ceramic perform, wetting between the molten metal and ceramic perform. Higher growth rate of the composites was observed in the RMI process than in the DIMOXTM process.

The bending strength and fracture toughness of com-

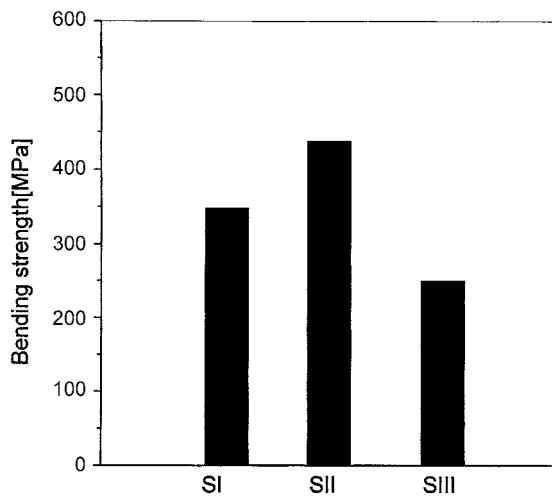


Fig. 3. Bending strength of composites produced by the RMI and DIMOXTM processes: SI: Al₂O₃/Al (RMI) SII: Al₂O₃/Al-4 wt.% Cu (RMI) SIII: Al₂O₃/Al (DIMOX).

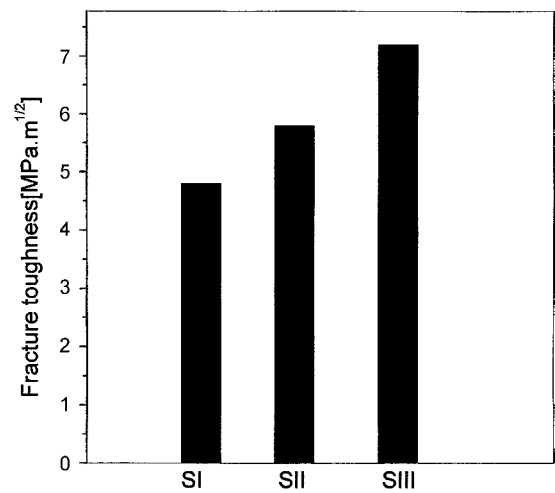
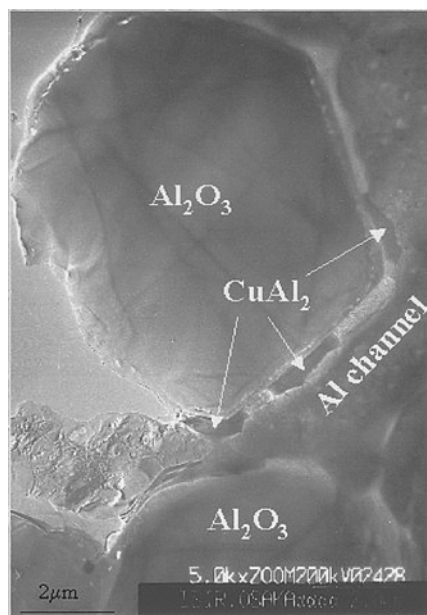


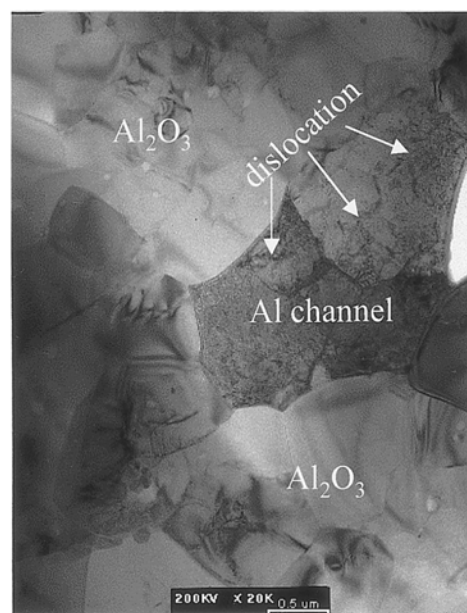
Fig. 4. Fracture toughness of composites produced by the RMI and DIMOXTM processes: SI: Al₂O₃/Al (RMI) SII: Al₂O₃/Al-4 wt.% Cu (RMI) SIII: Al₂O₃/Al (DIMOX).

posites are shown in Fig. 3 and Fig. 4. In the RMI process, the bending strength of the composite without Cu was 348 MPa whilst the bending strength of the composite infiltrated by Al powder containing 4 wt.% Cu was 438 MPa. The bending strength of the composite made using the DIMOXTM process was 250 MPa, which is lower than the bending strength achieved with the RMI process. However, a higher fracture toughness was observed samples made by the DIMOXTM process than made by the RMI process. The fracture toughness of the composite containing 0 wt.% and 4 wt.% Cu made by the RMI process were 4.8 MPa^{1/2} and 5.8 MPa^{1/2}, respectively. On the other hand, the fracture

toughness of the composite made by the DIMOXTM was 7.2 MPa^{1/2}. The reason for the increased bending strength with a Cu addition in the RMI process could be explained by precipitating hardening of the metal phase in the Al-Cu system. Cu dissolved in Al at the processing temperature formed the precipitates (CuAl₂) within the Al channels during cooling as shown in Fig. 5(a). The CuAl₂ phase was found to be flake type formed along the interface between the Al₂O₃ and Al channels. Heterogeneous precipitation of CuAl₂ on the Al₂O₃ particles depicts a platelet shape. Figure 5(b) shows a TEM micrograph of a Al₂O₃/metal composite produced by the DIMOXTM process. Dislocations were

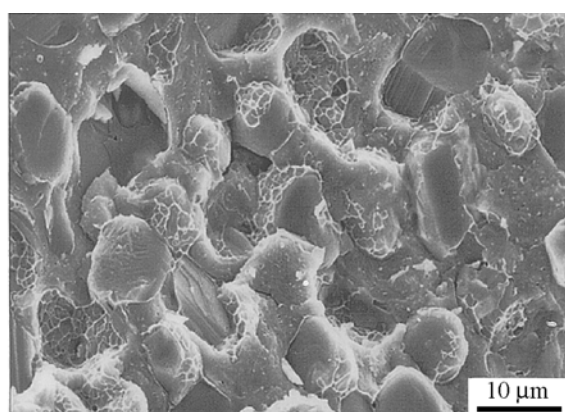


(a) RMI



(b) DIMOXTM

Fig. 5. TEM micrographs of Al₂O₃/metal composites fabricated by the RMI and DIMOXTM processes.



(a) RMI

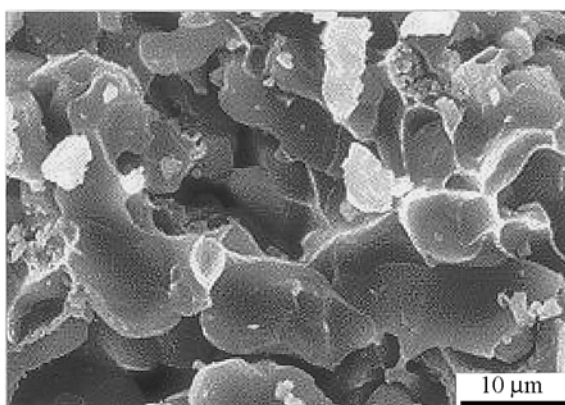
(b) DIMOXTM

Fig. 6. Fractography of Al_2O_3 /metal composites fabricated by the RMI and DIMOXTM processes.

observed near the interface between the metal channels and Al_2O_3 particles. Figure 6 shows the fractography of

the composites made by the RMI and DIMOXTM processes after bending tests. The fractography depicts the mixture of trans- and inter-granular fracture and a small amount of plastic deformation in the metal channels was observed.

Conclusions

Al_2O_3 /metal composites were fabricated by the reactive melt infiltration (RMI) process and the directed metal oxidation (DIMOXTM) process in an oxygen atmosphere. Al_2O_3 /metal composites were made at a lower temperature in the RMI process than in DIMOXTM process and required shorter processing time. The bending strength of composites were higher from the samples made by the RMI process than by the DIMOXTM process, but the fracture toughness was higher for samples made by the DIMOXTM process.

References

1. C. Toy and W. D. Scott, J. Am. Ceram. Soc. 73[1] (1990) 97-101.
2. D. C. Halverson, A. J. Pyzik, and I. A. Aksay, Ceram. Eng. Sci. Proc. 6[7-8] (1985) 736-44.
3. I. A. Aksay, C. E. Hoge, and J. A. Pask, J. Phys. Chem. 78 (1974) 1178-83.
4. M. S. Newkirk, A. W. Urquhart, and H. R. Zwicker, J. Mat. Res. 1[1] (1986) 81-89.
5. W. P. Tai, T. Watari, and T. Torikai, J. Am. Ceram. Soc. Bulletin 7[4] (1997) 3-6.
6. B. R. Lawn and E. R. Fuller, J. Mat. Sci. 10 (1975) 2016-2024.