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A Study on Surface Morphology of Ni-Al_2O_3 Composite Prepared by Agglomeration Process

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Aluminium(III) oxide (Al_2O_3) particles (mean diameter: 250 μm) were coated with fine Ni particles (mean diameter: 5 μm) by an agglomeration coating technique. The Ni content was 40-50 vol. %. The compact of coated particles was sintered at temperature of 1200-1500 °C. The use of coated particles resulted in uniform dispersion of Al_2O_3 particles in Ni matrix. The pre-coating of Al_2O_3 particles with fine W particles (mean diameter: 1 μm) was effective to improve the adhesion and wettability of Ni to Al_2O_3. Although the relative density of the sintered body was 70-80 %, the dense composite was produced by the infiltration of molten Ni into the porous sintered body.

Keywords: Ni-Al_2O_3 composite, Agglomeration coating, Adhesion, Wettability.

INTRODUCTION

The fracture strength and toughness of ceramics can be increased by introducing ductile metal inclusions. The common factors that influence the mechanical properties of ceramic-metal composites are density, quantity of the metal component and the metal-ceramic bonding interface [1-3]. The reinforcement mechanism has been attributed variously to strengthening by metal inclusions and to bridging/stretching of the ductile particles with the generation of microcracks within the ceramic [4,5]. Bridging/stretching of the ductile particles plays the main role in protecting against crack propagation [5]. When cracks and a stress field are generated in the composite, they lose energy as the ductile metal particles plastically deform, thereby enhancing the toughness of the composite.

Bridging reinforcement becomes especially important when the shape of the dispersed particles is fibrous rather than spherical. Numerous studies of fibers in composites have explored fiber properties such as type, length, volume fraction and orientation [6-8].

However, the microstructure of fibrous particles is difficult to control during fabrication. In general, the fibers are produced separately, surface treated and finally added to the molten matrix. However, this process is confined to cases in which the melting point of the matrix is lower than that of the fibers. Furthermore, expensive and complex processes such as hot isostatic pressing, infiltration, reactive forming, or fibrous monolith methods are required to add fibers to high-temperature ceramics [9].

The uniform dispersion of ceramics particles in metal matrix and the high bonding strength between metal and ceramics are important to improve the mechanical property of metal-ceramics particulate composites. The use of metal-coated ceramics particles must be the most useful method to control the microstructure in the production of these composite materials. Matsui *et al*. [10] have developed an agglomeration coating technique to prepare metal-coated ceramics particles, in which ceramics particles were coated with fine metal particles.

In the present work, Ni-Al_2O_3 composite was produced by the sintering of Ni-coated Al_2O_3 particles prepared by the agglomeration process. The microstructure of sintered body and the effect of W intermediate layer on Ni-Al_2O_3 bonding were investigated. Furthermore, the infiltration of molten Ni into the porous Ni-Al_2O_3 sintered body was carried out to produce the dense composite.

EXPERIMENTAL

Electrofused Al_2O_3 particles (Nikkei Kako Co., Ltd) having the average size of 250 μm were coated with Ni particles having the average size of 5 μm by an agglomeration coating technique using polyethylene glycol as a binder, aluminium stearate as dispersant of Ni particles and a rotational tilted cylinder of acrylic resin as agglomeration apparatus. The Ni coating was repeated to increase Ni content after the heat treatment of the initial Ni-coated Al_2O_3 particles at 1000 °C in

H_2. The Al_2O_3 particles were also coated with W particles having the average size of 1 μm. After W-coated Al_2O_3 particles were heat-treated at 1500 °C in H_2, they were coated with Ni particles. The mechanical mixture of Al_2O_3 and Ni particles was prepared using rotational tilted cylinder including alumina bowls to compare the microstructure of sintered body with that of metal coated Al_2O_3 particles.

Powders were pressed into the pellets of 10 mm in diameter and 2-3 mm thick using a steel die at 3 t/cm^2. The green density was about 70 %. The pellets were set in alumina container and heated for 4 h at 1200-1500 °C in H_2. A pellet of Ni particles was set on a porous sintered body and heated above the melting point of Ni in H_2 to infiltrate molten Ni into the pores. The bulk density of the composite was determined from the size and weight. The microstructure was observed with a field emission scanning electron microscope (FE-SEM, JSM-890S JEOL). X-ray diffraction (XRD, JDX-35HS JEOL) was conducted to check for the presence of a second phase in the Ni-Al_2O_3 composite and the distribution of W element was observed with an X-ray microanalyzer (XMA, JXA-8230 JEOL).

RESULTS AND DISCUSSION

Ni-coated Al_2O_3 particles and their sintered body: SEM photographs of original Al_2O_3 particles and Ni-coated Al_2O_3 particles are shown in Fig. l. The Ni content was 44 vol % and the whole surface of Al_2O_3 particles was covered with fine Ni particles.

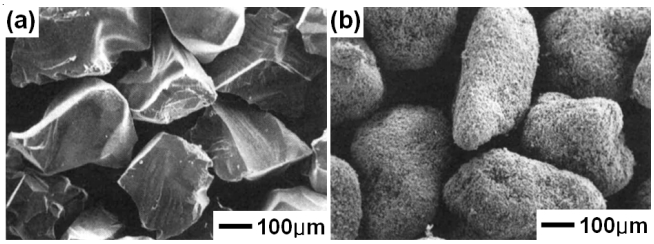

Fig. 1. SEM of (a) Al_2O_3 particles and (b) Ni-coated Al_2O_3 particles (Ni: 44 vol. %)

The microstructure of the sintered body obtained at 1400 °C from Ni-coated Al_2O_3 particles is compared with that obtained from the mechanical mixture of Al_2O_3 and Ni particles, as shown in Fig. 2.

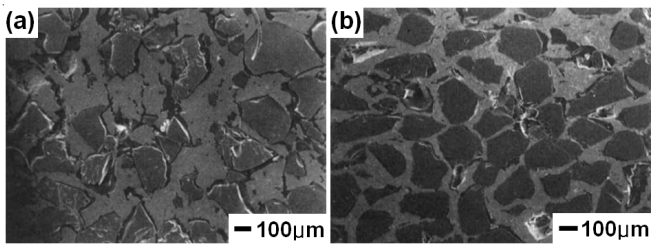

Fig. 2. Section of sintered bodies of (a) mechanically-mixed powder (Ni: Al_2O_3 = 45:55 vol. %) and (b) Ni-coated Al_2O_3 particles (Ni:Al_2O_3 = 40:60 vol. %). Sintering temperature is 1400 °C

When mechanically-mixed powder was used, Al_2O_3 particles were concentrated in parts and Ni phase was localized. On the other hand, Ni-coated Al_2O_3 particles gave more uniform microstructure, in which Al_2O_3 particles were dispersed in Ni matrix. The relative density of these sintered bodies was about 80 % and large voids were observed at Ni/Al_2O_3 interface. The void enlarged with an increase in sintering temperature from 1200 to 1400 °C. At 1500 °C molten Ni was eliminated from the powder compact. Ni has a poor wettability and low bonding strength to Al_2O_3 [11,12].

The XRD pattern (Fig. 3) shows that the Ni-Al_2O_3 composite was successfully fabricated without formation of a second phase or NiO, which can be detrimental to the formation of the composite.

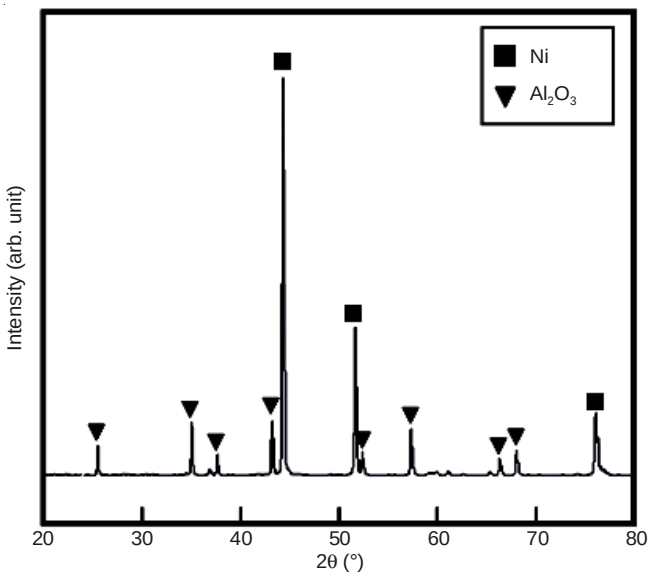

Fig. 3. XRD pattern of sintered body of Ni-coated Al_2O_3 particles

Effect of W intermediate layer between Ni and Al_2O_3: The microstructure of the sintered body obtained at 1400 °C from Ni/W-coated Al_2O_3 particles is compared with that obtained from Ni-coated Al_2O_3 particles in Fig. 4. When Al_2O_3 particles were pre-coated with W particles, Ni phase was intimately bound to Al_2O_3 particles. Even above the melting point of Ni, molten Ni stayed in the sintered body.

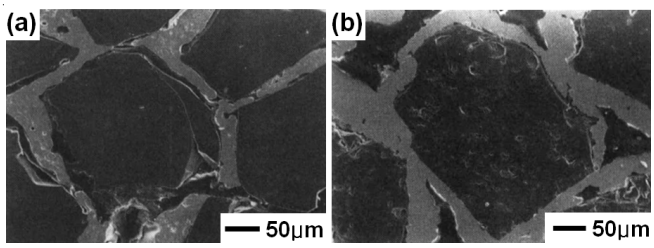

Fig. 4. Section of sintered bodies of (a) Ni-coated Al_2O_3 particles (Ni:Al_2O_3 = 40:60 vol. %) and (b) Ni/W-coated Al_2O_3 particles (Ni:W:Al_2O_3 = 39:11:50 vol. %). Sintering temperature is 1400 °C

The distribution of W element is shown in Fig. 5. It is found that W layer exists at Ni/Al_2O_3 interface and a part of W dissolves in Ni matrix. Tungeston is known to be available for the metallization of alumina ceramics [13].

Since W layer was heat-treated before Ni coating, W layer seemed to form a strong bonding to Al_2O_3. When the mechanical mixture of Ni, W and Al_2O_3 particles was sintered at 1400 °C large voids were observed at metal/Al_2O_3 interface. It is important that W is localized between Ni and Al_2O_3.

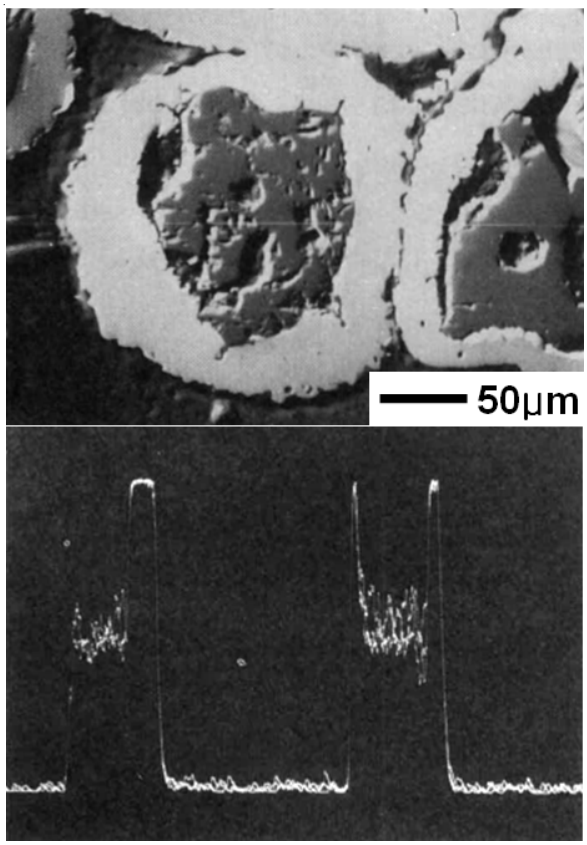

Fig. 5. X-ray line profile of W in the sintered body of Ni/W-coated Al_2O_3 particles. Sintering temperature is 1400 °C

The relative density of these sintered bodies was still low and large voids were observed in Ni matrix. The reason for the restricted densification may be that large Al_2O_3 particles are hardly rearranged during the sintering of Ni particles.

Infiltration of molten Ni into the porous sintered body: Molten Ni was infiltrated into the porous sintered body obtained from Ni/W-coated Al_2O_3 particles. The microstructure of the composite is shown in Fig. 6. Large voids in the sintered body were filled up with molten Ni. The reason for the easy penetration of molten Ni into pores may be that metal layer on Al_2O_3 particles acts as a guide for molten Ni. The relative density of the composite was 97 %. The residual porosity may be assigned to closed pores included in the electrofused Al_2O_3 particles.

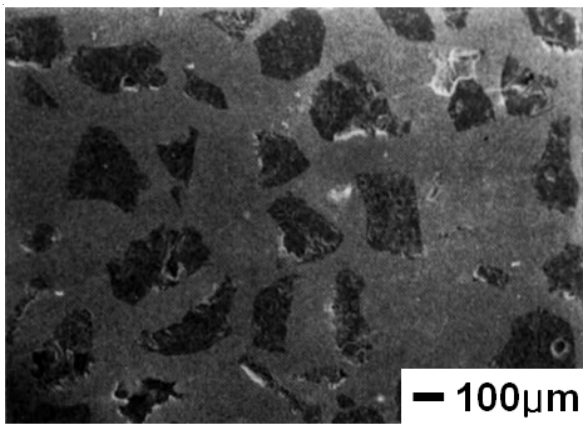

Fig. 6. Ni-infiltration into the sintered body of Ni/W-coated Al_2O_3 particles. Infiltration temperature is 1460 °C. Ni:W:Al_2O_3 = 56:8:36 vol. %

Conclusion

Ni-Al_2O_3 particulate composite was produced by the sintering of the metal-coated particles prepared by an agglomeration coating technique. Ni-coated Al_2O_3 particles gave a uniform microstructure, in which Al_2O_3 particles were dispersed in Ni matrix. The wettability and bonding strength of Ni to Al_2O_3 were significantly improved with the inclusion of W intermediate layer. Although the sintered body was porous, the dense composite was obtained by the infiltration of molten Ni into the porous body.

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