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Eutectic $\text{Al}_2\text{O}_3$–$\text{GdAlO}_3$ composite consolidated by combined rapid quenching and spark plasma sintering technique

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INTRODUCTION

Directionally solidified oxide materials have drawn strong research interest because of their electrical, magnetic and mechanical properties. The microstructures of eutectic materials are quite different from those of ordinary materials. The binary $\text{Al}_2\text{O}_3$–$\text{Gd}_2\text{O}_3$ system has a characteristic microstructure and superior mechanical properties. Its most remarkable property is its high strength at characteristic microstructure and superior mechanical properties.1 The microstructures of eutectic material are in the Graduate School of Science and Technology, Chiba University, 1-33 Yayoi-cho, Inage-ku, Chiba 263–8522, Japan. Manuscript received 1 June 2004; accepted in revised form 12 August 2004. © 2004 Institute of Materials, Minerals and Mining. Published by Maney on behalf of the Institute.

EXPERIMENTAL PROCEDURES

The raw materials were powders of $\text{Al}_2\text{O}_3$ (99.99%, Sumitomo Chemical, Japan) and $\text{Gd}_2\text{O}_3$ (99.99%, Santoku Metal Industry, Japan) with unknown particle sizes. The mixture was melted, so particle sizes were not measured.

For the binary $\text{Gd}_2\text{O}_3$–$\text{Al}_2\text{O}_3$ system (Fig. 1), grey lines indicate equilibrium. When the liquid of the eutectic composition was cooled, a product of $\text{GdAlO}_3$ and $\text{Al}_2\text{O}_3$ resulted in an ultrafine eutectic $\text{Al}_2\text{O}_3$–$\text{GdAlO}_3$ structure. Thus the actual behaviour of the system is described by the solid line. For traditional eutectoid formation, a rod shaped powder compact formed from a mixture of $\text{Gd}_2\text{O}_3$ and $\text{Al}_2\text{O}_3$ was calcined at 1000°C for 60 min, and melted by arc discharge to produce a solid eutectic composite. The top of the rod was melted by arc discharge, and the discharge was turned off to produce ‘slow cooled’ samples. Rapid cooling was performed by quenching the droplet between rotating aluminium rollers to form an amorphous film. This rapid quenching technique is shown schematically in Fig. 2.

Microstructures of cross-sections of slow cooled and rapid cooled samples are shown in Fig. 3. The rapid cooled microstructure has much smaller crystal size than after slow cooling. The translucent amorphous film formed in the rapid quenching technique was ground before sintering.

For complete sintering, conventional isostatic sintering was performed above 1600°C for 24 h. Using SPS, however, sintering conditions ranged from 1300 to 1500°C with no soaking time, i.e. sintering was much quicker and at a lower temperature than with conventional sintering. Using the SPS technique (Fig. 4), with its advantages of low sintering temperature and short sintering time, the production of a sintered body with a very fine eutectic structure was expected from the rapid quenched amorphous material.

RESULTS AND DISCUSSION

Cross-sectional micrographs showed microstructures with features several micrometres in size in both slow and rapid cooled samples. The XRD pattern of the film formed by rapid quenching was amorphous (Fig. 5). SEM observation of film cross-sections, indicated by the line, which was moulded with epoxy resin, revealed no eutectic structure.
Further heat treatment might result in eutectic precipitation.

XRD patterns of rapid quenched materials after heating at 1200 and 1300 °C for 30 min are shown in Fig. 6. The heat treated materials showed crystalline phase peaks. At 1200 °C, Al₂O₃ phase was not detected probably because the peak strength of Al₂O₃ is weak. The diffraction peak for Al₂O₃ is so weak in the sample at 1300 °C that the atomic scattering factor of Al is much smaller than that of Gd.

Figure 7 compares slow cooled material and material precipitated from the amorphous phase. The two SEM images on the right show structures from amorphous phase produced by rapid quenching. With the same magnification as the slow cooled material, eutectic structure was not observed, but higher magnification revealed ultrafine eutectic structure.

An amorphous powder with a particle size of 2–20 μm, ground from the rapid quenched material, was used to fabricate a bulk specimen by conventional sintering for 24 h at a heating rate of 10 K min⁻¹. The specimen was 12 mm in diameter and 1-5–2-0 mm thick. Below 1500 °C, a sintered body was not available for measurement of sintered density.

In Fig. 8, the microstructure of material conventionally sintered at 1600 °C for 24 h from rapid quenched powder is shown. Grain growth was significant, with a final size of about 50 to 100 μm, and the material could not be fully sintered.

Figure 9 presents the relationship between density and sintering temperature for samples consolidated by SPS at...
1300, 1400 and 1500°C with no soaking time. The specimens were 15 mm in diameter and 1.5–2.0 mm thick. Highly sintered materials with 99% relative density could be obtained at 1500°C, a significantly lower temperature than with the conventional sintering method.

The SEM micrographs in Fig. 10 show significant microstructural change with increasing sintering temperature from 1300 to 1500°C. An ultrafine microstructure is established after sintering at 1500°C, which looks like a very typical microstructure of binary Al₂O₃–GdAlO₃. Comparing conventional sintering at 1600°C for 24 h (Fig. 8) and SPS at 1500°C with no soaking time (Fig. 10), a typical ultrafine eutectic Al₂O₃–GdAlO₃ microstructure may be observed only in the SPS sample.

It is evident that a proper combination of rapid quenching and SPS can synthesise a sintered material with ultrafine and dense eutectic structure. An Al₂O₃–GdAlO₃ sintered body with eutectic microstructure can be
CONCLUSIONS

After melting and rapid quenching a mixture of Al2O3–GdAlO3, the result is an amorphous film. Using spark plasma sintering, this ground amorphous powder was consolidated into a dense sintered body with a typical ultrafine Al2O3–GdAlO3 eutectic structure. An ultrafine eutectic structure can be obtained by heat treatment of amorphous material produced by rapid quenching. Sintered material with ultrafine and dense eutectic structure was obtained by an appropriate combination of rapid quenching and SPS at lower temperature and more quickly than by conventional sintering.

REFERENCES