O U R N A L O F

Ceramic Processing Research

Characteristics of aluminum - nickel aluminate composite

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A composite consisting of nickel aluminate ceramic - aluminum metal was fabricated by melting the metal in the ceramic skeleton. The porous nickel aluminate skeleton was fabricated by a polyurethane foam process with synthesized nickel aluminate powder. In the fabrication of the composite, powder-type aluminum alloys were melted in the porous nickel aluminate. The aluminum alloy containing Mg and Mn was used and melted in nitrogen atmosphere at 1000°C. The mechanical properties of the composites, which have a unique microstructure consisting of continuous phases of ceramic and metal, were examined according to the pore size of the nickel aluminate skeleton. A flexural strength of 370 MPa was obtained in the composite prepared from the nickel aluminate skeleton having a pore size of 300 μ m, showing a non-brittle fracture behavior. A numerical study for the prediction of the mechanical failure behavior of the composite was also performed under the modification of finite - element code in DIANA.

Key words: Nickel aluminate, Aluminum, Composite, Strength, Computer simulation.

Introduction

Nickel aluminate (NiAl₂O₄) has excellent strength and good wet ability with metals at high temperatures, in addition to the general merits of spinel materials [1, 2]. In particular, a nickel aluminate having a skeleton structure can be applied to a new fabrication method for metal - ceramic composites. A nickel aluminate ceramic - metal composite can be fabricated by the infiltration of metals into the skeleton. The wet ability between ceramic and metal has been studied over the last decades [3, 4], and the Lanxide process [5] has been known as a representative method for making ceramic-metal composite. The wetting behavior of molten aluminum is improved by the addition of wetting agents, such as Mg or Zn. In the study of the surface chemistry of molten aluminum, spinel phases, such as MgAl₂O₄ on the surface of molten aluminum led to infiltration of molten aluminum into the micro channels of oxide ceramics [6, 7]. Unlike the Lanxide process for aluminum and alumina composites, there has been little investigation of aluminum and nickel aluminate composites; studies have only been carried out in the limited fields of the fabrication process of nickel aluminate by solid state reaction or sol-gel synthesis [8, 9]. Also, the fabrication process for the nickel aluminate - aluminum composite using a skeletontype nickel aluminate has not been studied at all.

In this study, powder-type aluminum alloys including

several wetting agents were melted in a porous nickel aluminate under various atmospheres. The porous nickel aluminate skeleton was fabricated by a polyurethane foam process [10] using a nickel aluminate powder synthesized by a new powder synthesis method, the polymer solution technique [11]. The effects of the wetting agents in the metal alloy and heating atmosphere on the infiltration behavior were investigated. The mechanical properties of the composites were examined according to the pore size of the nickel aluminate skeleton. A numerical study for the prediction of mechanical failure behavior of the composite was also performed under the modification of finite element code in DIANA [12].

Experimental procedure

Nickel nitrate and aluminum nitrate were used for the polymer solution synthesis technique [11]. Once the nitrates were completely dissolved, 5 wt% PVA solution was added. The precursor sol was dried at 150 °C and the resulting gel-type precursor was calcined and crystallized at 900 °C in an air atmosphere. To make the nickel aluminate porous body, a nickel aluminate slurry was fabricated using the synthesized and ballmilled nickel aluminate powder and water solvent. And then, polyurethane foams, which had three different PPIs (pore per inch), were dipped in the nickel aluminate slurry and were squeezed to remove the slurry from the sponge pores. The wet polyurethane foams were dried and calcined in an air atmosphere at a slow heating rate of 0.5 °C/min up to 700 °C for burn-out of the polyurethane skeleton. Finally, the calcined, porous nickel aluminate bodies were sintered

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Table 1. Composition of Aluminum Alloys (wt%).

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	Si	Fe	Cu	Mn	Mg	Ti	V	Al
a1	0.149	0.575	0.191	0.003	0.001	0.013	0.013	99.04
a2	3.778	0.828	0.104	0.007	0.001	0.019	0.015	95.23
a3	0.206	0.593	0.165	1.211	4.200	0.017	0.012	93.58

at 1600 °C for 1 h in an air atmosphere. Powder-type aluminum alloys were filled and melted in the porous nickel aluminates in a vacuum or nitrogen atmosphere at 1000°C. In this study, three kinds of alloys $(a1 \sim a3)$ were used and the composition of the each alloy is listed in Table 1. The microstructures of the porous nickel aluminate and the composite interfaces were examined by scanning electron microscopy (SEM, Hitachi, S-3500N). The flexural strengths of the nickel aluminate - aluminum composites were examined using a universal testing machine (model 4502, Instron Corp., Canton, MA). The bend bars with dimensions of $30 \text{ mm} \times 3.0 \text{ mm} \times 4.0 \text{ mm}$ were polished to a #1200 finish with SiC abrasive paper. Four-point flexural testing was performed using a 9.0 ± 0.1 mm inner span and a 20.0 ± 0.1 mm outer span, at a crosshead speed of 0.05 mm/min. The compressive strengths of the nickel aluminate skeletons were also examined with cubicshaped samples.

A numerical study was performed by computer simulation to estimate the actual failure behavior of the composite on the basis of continuum materials, and the results were compared with the results obtained by mechanical testing. The material properties required in the analysis were obtained from additional experimental data and reference data. Young's moduli of the nickel aluminate and aluminum were taken as 180 GPa and 60 GPa, and Poisson's ratios were 0.2 and 0.3, respectively. The estimated ceramic volume fraction was taken as 40 vol%. Numerical simulation was conducted using a commercial finite - element code, DIANA [12], which was modified to reflect the effects of the filling rate of pores and the combined bond strength of various materials in the sense of micro-mechanics.

Results and Discussion

The ball-milled, fine nickel aluminate powder showed an average particle size of 0.28 μ m with a specific surface area of 23.0 m²/g. The precursor gel was aerated and the synthesized powder was also soft and porous for easy grinding. Porous nickel aluminate bodies were successfully fabricated by the polyurethane foam method. The sintered nickel aluminate skeleton samples are shown in Fig. 1 with a representative microstructure obtained from the sample showing a 600 μ m pore size. The channel-type, open pores were distributed evenly over the whole sample and the skeleton thickness was about 100 μ m. Fig. 2 shows the



Fig. 1. Photographs of porous nickel aluminate samples with a skeleton microstructure.



Fig. 2. Compressive strengths of porous nickel aluminate skeletons according to pore size.

compressive strength of the porous nickel aluminate skeletons at various pore sizes. The compressive strengths were dependent on the pore size. As expected on normal porous ceramics, the highest compressive strength of 8.1 MPa was obtained in the sample with the smallest pore size of $300 \,\mu\text{m}$.

In the melting behavior of the aluminum alloys, all kinds of powders $(a1 \sim a3)$ were partially wetted to the ceramic skeletons at 1000 °C in a vacuum. The wetting of the molten aluminum was not complete and the interface was not dense. A representative microstructure of a debonding interface resulting from poor wetting is shown in Fig. 3. In the case of nitrogen atmosphere, the aluminum alloys a1 and a2 did not completely melt in the nickel aluminate skeleton, while the alloy a3 melted extensively through the whole skeleton sample. According to a previous study [13], it has been known that the oxidizing surface of molten aluminum prevents wetting reaction with ceramics. However, Mg in molten aluminum inhibits the formation



Fig. 3. Interface microstructure of nickel aluminate - aluminum alloy composite heat treated at $1000 \,^{\circ}$ C in vacuum.



Fig. 4. Interface microstructure of nickel aluminate - aluminum alloy composite heat treated at $1000 \,^{\circ}$ C in nitrogen atmosphere.



Fig. 5. Flexural strengths of nickel aluminate - aluminum composites according to pore size of ceramic skeleton.



Fig. 6. Displacement and load curve for numerical simulation in nickel aluminate ceramic - aluminum metal composite.

of an oxidation film. In particular, it has been shown that Mg reacted with nitrogen gas forms Mg-N compounds and they improve the wetting between molten aluminum and alumina ceramics. In this experiment, the nickel aluminate spinel was used instead of alumina, and the aluminum alloy (a3) containing 4.2 wt% Mg was employed. Fig. 4 shows the interface microstructure of the nickel aluminate - aluminum (a3) composite heated at 1000 °C in nitrogen atmosphere. The composite revealed a dense and pore-free interface microstructure. In the a3 aluminum alloy, Mn and Mg worked as wetting agents for good infiltration into the spinel ceramics [5, 13].

The measured flexural strengths of the composites prepared with the a3 aluminum alloy in nitrogen atmosphere ranged between 370 MPa to 310 MPa according to the pore size of the nickel aluminate skeleton as shown in Fig. 5. The composite strength was much higher than the strength of the porous nickel aluminate skeleton. In the composite consisting of continuous phases of ceramic and metal, the strength was certainly affected by the occupied volume of the ceramic skeleton which was dependent on pore size. As shown in Fig. 5, the strength almost linearly decreased as the ceramic volume decreased.

The mechanical behavior of the composite was examined by computational simulation. The results of the numerical failure analysis showed non-brittle fracture behavior as shown in Fig. 6. It was estimated with the assumption of 40 vol% nickel aluminate. The calculated strength was 410 MPa, and the data was higher than the measured strength of the sample prepared with the skeleton having a 300 μ m pore size (Fig. 5). It is thought that the difference of strength is due to the pores in the composite. Some pores were locally observed in the microstructure of composite.

Conclusions

A nickel aluminate - aluminum composite was successfully fabricated by the control of processing factors. The composite showed a unique microstructure consisting of continuous phases of ceramic and metal. A powder-type aluminum alloy containing Mg and Mn was melted in a nickel aluminate skeleton resulting in a good wetting behavior to the nickel aluminate in nitrogen atmosphere at 1000 °C. The flexural strength of the composite was dependent on the pore size of the nickel aluminate skeleton. A flexural strength of 370 MPa was obtained in the composite prepared with the nickel aluminate skeleton having a pore size of 300 μ m, showing a non-brittle fracture behavior.

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