# Fabrication of Al<sub>2</sub>O<sub>3</sub>-Ni Composites Using Ceramic Nanoparticles

## Thitirat Theerabornkul and Sukasem Kangwantrakool\*

School of Ceramic Engineering, Institute of Engineering, Suranaree University of Technology, Nakhon Ratchasima 30000, Thailand

\*Corresponding author. E-mail: <u>sukasem@yahoo.com</u>

#### ABSTRACT

In the present study,  $Al_2O_3$ -Ni composites were prepared by pressureless sintering. Nickel was used as a reinforcing material due to its high strength and toughness, both at room and elevated temperature. The purposes of this work are to improve the fracture toughness of the ordinary alumina with nickel additions and to investigate the effect of nickel on the characteristics and mechanical properties of  $Al_2O_3$ -Ni composites and also to study the proper sintering temperature for the optimum toughness of sintered composites. In this work, alumina nanoparticles were used as a matrix material and nickel as a particulate reinforcement material. The compositions were changed with different amount of nickel additions of 0, 10, 20 and 30 wt% in the composite. Then, the chemical composition, phase, microstructure and density of the sintered composites were characterized and also the flexural strength, hardness and fracture toughness were determined.

As the results, it was revealed that with the increase of nickel from 10 to 20 and 30 wt% in the sintered composite samples, the flexural strength was increased from 146.66 MPa to 178.08 MPa and 188.08 MPa, hardness was decreased from 8.59 GPa to 7.84 GPa and 4.59 GPa and fracture toughness was increased from 4.13 MPa.m<sup>1/2</sup> to 6.36 MPa.m<sup>1/2</sup> and 8.08 MPa.m<sup>1/2</sup>, respectively. The optimum density of composite was obtained with sintering temperature at 1,400°C.

Key words: Alumina, Nanoparticles, Composites, Pressureless sintering, Mechanical properties

#### **INTRODUCTION**

Alumina is most widely used in the ceramics industry because of its high hardness, high strength and good chemical stability, although weak in the fracture toughness. Many attempts have been made to improve the mechanical properties of alumina ceramics, mainly the fracture toughness, by using reinforcement additions such as ductile metal particles (Rodeghiero et al., 1995). For such purpose, Ni ductile metal has the potential to improve the fracture toughness of Al<sub>2</sub>O<sub>3</sub> ceramics because Ni possesses high toughness in a wide range of service temperature (Waterman and Ashby, 1991). However, Al<sub>2</sub>O<sub>3</sub>-Ni composites are currently the most important materials in a wide range of high strength and fracture toughness combinations and/with good oxidation and corrosion resistance especially at elevated temperature. (Fahrenholtz et al., 2000).

The purposes of this work are to develop/improve the fracture toughness of alumina with various amounts of Ni addition dispersed in alumina matrix, to study its influence on the relationship between compositions and mechanical properties and to determine their microstructure. Furthermore,  $Al_2O_3$ -Ni composites with different Ni contents were sintered by conventional low-cost pressure sintering method (Yang and Chen, 2000) which might be useful in practice for the production in ceramic industries.

# MATERIALS AND METHODS

As raw materials,  $Al_2O_3$  particle size of 100 nm is a matrix material and Ni as a particulate reinforcement. The composition employed was 0, 10, 20 and 30 wt% Ni inceptively. For comparison,  $Al_2O_3$  without Ni addition was also prepared by the same process and 2 wt% polyvinyl alcohol (PVA) was used as a binder for powder compaction. The powder samples were prepared by blending  $Al_2O_3$ , Ni and PVA at the same time with ethanol in a polypropylene (PP) pot, using a ball mill for 24h. The powder mixture was formed in a metallic die with an inside dimension of 35 mm X 35 mm under a pressure of 100 MPa by dry pressing machine. In order to increase the density of green body, all compacted samples were pressed by Cold Isostatic Pressing (CIP) at a pressure of 180 MPa. Then, CIPed samples were sintered under argon atmosphere for 120 min at 1,300, 1,400 and 1,500°C for  $Al_2O_3$  without Ni and 1,300, 1,400 and 1,450°C with Ni additions. The sintered samples obtained were 35 mm X 35 mm and 5 mm in thickness.

All the resulting sintered samples were ground with a SiC paper with several grits and polished with diamond suspension of particle size 3, 1 and 0.25 (m in this order. The polished samples were cut into bars of 4 mm x 30 mm x 3.25 mm for strength and hardness measurements and microstructure observations. The microstructure and fracture surface were observed by means of scanning electron microscopy (SEM). The phase compositions of the sintered samples were analyzed by X-ray diffraction (XRD) using CuK $\alpha$  radiation. Densities of sintered samples were measured, using an ultra-pycnometer based on weight-volume basis. The flexural strength was measured by the 3-point bending technique with a span length of 20 mm and a cross-head speed of 0.2 mm/min. The hardness was measured using Vickers indentation under the load of 98 N. The fracture toughness (K<sub>IC</sub>) was evaluated by the modified indentation method, using a diamond Vickers indenter under a load of 98 N and a loading duration of 15 s. The results were averaged with ten indentations per specimen. The K<sub>IC</sub> value was calculated from Eq. (1) as below:

KIC (MPa.m<sup>1/2</sup>) = 
$$\zeta$$
 (E/H)<sup>1/2</sup> P/C<sup>3/2</sup>

where  $K_{IC}$  is the toughness of the composite,  $\zeta$  is the constant value of 0.02, E is the Young's modulus (GPa), H is Vickers hardness (N/mm<sup>2</sup>), P is a load (N) and C is a half of the crack length (mm).

### **RESULTS AND DISCUSSION**

Figure 1 shows the XRD patterns of  $Al_2O_3$  and  $Al_2O_3$ -Ni composites sintered at 1,400°C for 2 hr. The XRD analysis reveals only  $Al_2O_3$  and Ni phases in the sintered composites.

Figure 2 shows the relative density of  $Al_2O_3$ -Ni composites against the Ni content. The density of all composites increased with the sintering temperature from 1,300°C up to 1,400°C. As for the optimum sintering temperature of 1,400°C with 30Ni content, the highest relative density was obtained. This is because, at high temperature, Ni viscous phase could flow and fill up through the void between  $Al_2O_3$  particles and resulted in the higher density than other compositions. Nevertheless, the lowest density was obtained with higher sintering temperature at 1,450°C. As known well, this temperature is too high for the lower melting point of Ni component. Then, the volatile of Ni could occur in the system to produce gas products and pore residue in the composite, resulting in lower density.



**Figure 1.** XRD patterns of sintered materials of (a) Al<sub>2</sub>O<sub>3</sub>, (b) Al<sub>2</sub>O<sub>3</sub>-10Ni, (c) Al<sub>2</sub>O<sub>3</sub>-20Ni and (d) Al<sub>2</sub>O<sub>3</sub>-30Ni composites.



Figure 2. Relative density of Al<sub>2</sub>O<sub>3</sub>-Ni composites with various sintering temperature.



Figure 3. Vickers hardness of Al<sub>2</sub>O<sub>3</sub>-Ni composite.

Figure 3 shows Vickers hardness of  $Al_2O_3$ -Ni composite. The hardness decreased with higher amount of Ni, i.e., the Vickers hardness decreased from 8.59 to 4.59 GPa when Ni was increased from 10 to 30 wt%. However, the highest hardness was obtained from  $Al_2O_3$  sample without Ni addition due to the harder ceramic, in other word, with the smaller amount of Ni ductile metal phase.



Figure 4. Fracture toughness as a function of Ni addition.

The fracture toughness describes the ability of a material to withstand crack propagation. Figure 4 shows changes in the fracture toughness of Al<sub>2</sub>O<sub>3</sub>-Ni composite as a function of Ni addition. The values in the figure indicate the average values of ten indentations per specimen being tested. The fracture toughness of composites increased with the higher amount of Ni content. With an increase of Ni content from 10 to 30 wt%, the fracture toughness of the composite increased from 4.69 to 8.23 MPa.m<sup>1/2</sup>. Nevertheless, the highest fracture toughness was obtained with the composite of 30Ni due to higher amount of Ni ductile metal. However, the 90Al<sub>2</sub>O<sub>3</sub>-10Ni composite possessed a higher hardness with lower fracture toughness than the 70Al<sub>2</sub>O<sub>3</sub>-30Ni composite. This can be explained as follow: The fracture toughness can be obtained from the dispersion of Ni ductile phase in Al<sub>2</sub>O<sub>3</sub> matrix phase which can generate the toughening property and result in mechanical interlocking among different phases to increase fracture toughness and to create extra resistance to the propagating crack front. When the crack intersected the Ni phase, the crack can not propagate directly through the Ni phase. It is mainly due to the better ductility of Ni than that of Al<sub>2</sub>O<sub>3</sub>. That is to say, the crack would be deflected by the Ni phase and pinned through Al<sub>2</sub>O<sub>3</sub> matrix and then the crack can not further propagate and close due to reduction of stress energy. Here, it can be concluded that the optimum mechanical properties, both of Vicker's hardness and fracture toughness, can be obtained from the composition of ~21.616Ni by weight percentage as shown in Figure 5.



Ni content (wt%)

Figure 5. Effect of Ni content in  $Al_2O_3$ -30Ni composite on mechanical properties.



Figure 6. Flexural strength of Al<sub>2</sub>O<sub>3</sub>-Ni composite with Ni content.

Figure 6 shows the flexural strength of  $Al_2O_3$ -Ni composites against the Ni content. Error bars show the maximum and minimum values measured. With the  $Al_2O_3$  sintered sample without Ni addition, the highest flexural strength was obtained and then decreased significantly with the Ni addition due to, in general, the strength of Ni metallic which is lower than  $Al_2O_3$  ceramic. Therefore, the addition of Ni metallic in  $Al_2O_3$  could improve the fracture toughness but weaken the strength. However, the flexural strength of composites increases again from 10%Ni up to 30%Ni by weight percentage due to higher amount of the Ni reinforcing dispersions phase which is formed similarly to the ligament shape to generate the dispersive strengthening effect, as shown in Figure 7. Nevertheless, the flexural strength of  $Al_2O_3$ -Ni composites was lower than monolithic  $Al_2O_3$ .



Figure 7. SEM photographs of (a) 90Al<sub>2</sub>O<sub>3</sub>-10Ni, (b) 80Al<sub>2</sub>O<sub>3</sub>-20Ni, (c) 70Al<sub>2</sub>O<sub>3</sub>-30Ni.

Figure 7 shows the microstructures of the  $Al_2O_3$ -Ni composite prepared by pressureless sintering, sintered at 1,400°C for 2 hrs. In the figure, the gray particles are Ni phases and the black matrix is  $Al_2O_3$ . As can be seen, the distribution of the Ni phases in the  $Al_2O_3$ -Ni composite specimen shows a Ni ligament shape. The increase in Ni content should determine an increase of fracture toughness. Finally, the morphology of the Ni dispersing phase can be accounted for the fracture toughness increase, as a dispersing phase of 30Ni with higher aspect ratio can represent bridging sites across crack faces. This Ni metal acts as a crack inhibitor to generate the toughening effect of the metal dispersions which shows the variation of Ni shape as a function of wt% Ni content. As the results, the higher amount of Ni could lead to the higher fracture toughness.

## CONCLUSION

From the results, it was revealed that by increasing nickel from 10, 20 and 30 wt% in the sintered composite samples, the flexural strength was found to increase. This was due to Ni reinforcing dispersions phase which is formed similarly to the ligament shape to generate the dispersive strengthening effect. Hardness was decreased with the higher amount of Ni ductile metal phase, while the fracture toughness increased due to the dispersion of Ni ductile phase in  $Al_2O_3$  matrix phase which can generate the toughening effect. Nevertheless, the optimum mechanical properties, both of Vicker's hardness and fracture toughness, can be obtained from the composition of 20Ni by weight percentage. Finally, the optimum relative density of composite was obtained with sintering temperature at 1,400°C for 2 hrs.

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