MECHANICAL CHARACTERIZATIONS AND ELECTROMAGNETIC WAVE SHIELDING PROPERTIES OF METALLIC NI PARTICULATED Al_2O_3 CERAMIC MATRIX COMPOSITES

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Abstract

The present paper describes producing alumina ceramic matrix composites reinforced with nickel at ratios of 1 % to 7 % by weight and their resultant mechanical and electromagnetic properties. In this study powder metallurgy method was used and samples obtained at 1440 °C for 1 h in graphite powder. Argon atmosphere has widely used to produce metallic particulate reinforced ceramic based composites to prevent oxidation risk of metallic particulate. However, in this study using a graphite powder it can be easily sintered bulk materials in a ceramic pot. The firing shrinkage of sintered test materials ranged from 20.14 to 21.19 %. Uniform distribution of ductile nickel particles in the matrix was characterized by SEM microscopy and the presence of nickel and alumina phases were confirmed by EDS and XRD analysis. It was found that the fracture toughness of test materials increased with the nickel content from 3.0 to 7.05 $MPa \cdot m^{1/2}$. A slightly decreasing for hardness values was also detected. Hardness and fracture toughness was determined using Vickers indentation technique with applying, 0.98 and 98 N load respectively. Electromagnetic shielding effectiveness of nickel particle reinforced alumina composites were investigated in the in the wide range high frequencies of 12.4-18 GHz (Ku band). The experimental results indicate that in addition to increasing the mechanical properties electromagnetic characteristics of the composite has been preserved.

Keywords: Fracture toughness, Al_2O_3-Ni composite, sintering, powder metallurgy, electromagnetic shielding

1. INTRODUCTION

Ceramic matrix materials are very important materials and have wide applications, especially in some harsh environments, like high temperature, strong acid and base, etc. Alumina is an essential ceramic material with many prominent properties such as low density (3.95 g/cm3), excellent chemical inertness and high softening temperature. However, its application is restricted by the intrinsic brittleness [1-5]. To overcome the problem of brittleness ductile metal particles have often been incorporated as a second phase to improve the fracture toughness of ceramics. In this way mechanical properties of ceramics are improved. Dispersion of a ductile second phase enhances fracture toughness of ceramics through facilitating crack deflection, arresting and absorbing energy of microcrack, microcrack toughening, crack bridging and crack blunting [3-6]. The distribution of Ni in Al_2O_3 is very important for optimizing mechanical and functional properties of composites. This type of composites can be fabricated by sol-gel processing, pressureless sintering, hot pressing fine Al_2O_3 and nickel powder mixtures, and hot pressing the Al_2O_3 and NiO powders by reducing in hydrogen atmosphere [4-7]. Also to avoid explosive effect of hydrogen gas during sintering, graphite can be used safely to protect Ni particles from oxygen like as in our study.

Composite materials have gained more importance in recent years since they offer good electromagnetic shielding behavior in addition to their better mechanical resistance, strength, lightness, and isolation properties. However, in some applications, composite materials are preferred due to their improved properties, but it may be desired to maintain electromagnetic behavior of the material despite the metal addition [8-14]. The aims of this study are to process of Ni reinforced alumina ceramic composites and to search of these composites microstructural, physical, mechanical and electromagnetic properties.

2. METHOD AND PRODUCTION DETAILS

2.1. Powder processing

In this study, $Al_2(SO_4)_3$ was used as raw material to prepare pure alumina powders with an average particle size of 0.3 μm and purity of 99.9 %. Pure alumina was doped with metallic nickel powders with a particle size of 3-7 μm and 99.9% purity. Firstly, Al_2O_3 and Ni particles were mixed and ball milled for 48 h with alumina balls. Then the mixtures obtained were uniaxially pressed into round pellets of 15 mm x 3 mm under a pressure of 170 MPa. The compacts were then sintered at 1440 °C to avoid melting of Ni in an atmosphere controlled furnace at a heating rate of 5 °C/min for 1 h. After sintering they were cooled at a rate of 5 °C min^{-1} down to the room temperature. Schematic representation of production of alumina powder and the experimental sample preparation stages are given in **Figure 1**.

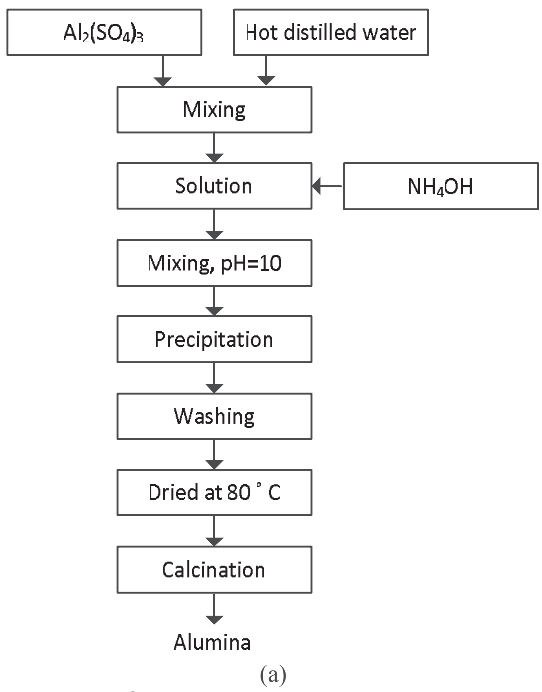

Figure 1 The flowchart showing experimental stages of preparation of test samples, a) alumina powder production, b) preparation of test samples

2.2. Characterization

The presence of phases within the sintered samples was determined by x-ray diffraction using CuKα radiation with a wavelength of 0.15418 nm over a 2θ range of 10 to 80°. The morphology of sintered alumina-nickel ceramic composites was revealed by means of scanning electron microscope (SEM). In order to verify the presence of Al_2O_3 and Ni powders energy-dispersive x-ray spectrometric analysis (EDS) was used. The relative densities of sintered composites were determined by using Archimedes' method in distilled water. Firing shrinkages of the samples were identified by measuring dimension of both green and sintered compacts. The hardness of samples was determined by using Vickers indentation technique. Fracture toughness of samples was measured via indentation fracture technique using Vickers diamond indenter under 98 N load.

The equation used for calculating fracture toughness was:

$$K_c = XP / c^{3/2} \quad (1)$$

where X is the residual-indentation coefficient [15, 16] which depends on hardness-to- modulus ratio (E/H) of ceramic composites. The constant X is 0.016 $(E/H)^{1/2}$, where H and E hardness and Young's modulus of test materials respectively. P is the applied load and c is the indentation half crack length.

3. EXPERIMENTAL RESULTS

3.1. XRD characterization

X-ray diffraction analysis has revealed that α alumina phase and metallic nickel has formed in the sintered samples. As it can be seen in **Figure 2**, oxidation-free nickel has obtained and the more Ni content has provided the higher intensity. A slightly oxidation has been determined during SEM-EDS observation (**Figure 3**) but this oxidation amount has not detected in XRD analyses. In addition it can be easily claimed that from $Al_2(SO_4)_3$ to Al_2O_3 powder production obtained successfully.

3.2. Microstructure

Figure 3 reveals SEM images with EDS spectrum of the alumina nickel powder mixtures sintered at 1440 °C for 1 h. Grey areas are Al_2O_3 and white ones are Ni. The distribution of Ni is homogenous in composite. SEM examinations revealed that Ni particles have a close spherical morphology with 3-4 micrometer particle size scale and do not present significant agglomeration. In order to verify the presence of Al_2O_3 and Ni powders energy-dispersive x-ray diffraction analysis (EDS) was used and it was found that the dominants constituents are alumina and nickel. It is clear that Ni particles were well-dispersed in the alumina matrix. As a result it is possible to claim that Al_2O_3-Ni composites can be obtained via powder metallurgy method without using argon atmosphere cheaply.

3.3. Mechanical properties

The relative density of sintered test materials was measured according to Archimedes' method and firing shrinkage was estimated by measuring diameter of the samples. As it can be seen in **Table** 1, the relative density and firing shrinkage of test materials increased as the amount of ductile second phase increased. When it comes to hardness values a slightly reducing also seen. The fracture toughness of alumina-nickel ceramic composites calculated by Vickers indentation technique increased with the addition of nickel particles.

Table 1 Codes, composition, firing shrinkage, relative density and mechanical properties of test materials

# of Sample	Samples	Composition (wt.%)		Firing shrinkage (%)	Relative density (%)	Hardness, HVN	Fracture toughness (MPa·m$^{1/2}$)
		Al_2O_3	Ni				
1	Al_2O_3	100	0	-	96.7	1570 ±13	3.0
2	Al_2O_3-1 wt. % Ni	99	1	20.14	96.60	1559 ±5	5.39
3	Al_2O_3-3 wt. % Ni	97	3	20.51	96.69	1506 ±11	5.70
4	Al_2O_3-5 wt. % Ni	95	5	20.86	97.38	1467 ±6	6.11
5	Al_2O_3-7 wt. % Ni	93	7	21.19	97.89	1393 ±8	7.05

The variation of fracture toughness and relative density of Al_2O_3-Ni composites as a function of nickel amount showed that due to ductile Ni Particles, the fracture toughness of test materials increased and their relative density also slightly enhanced as shown in **Figure 4**.

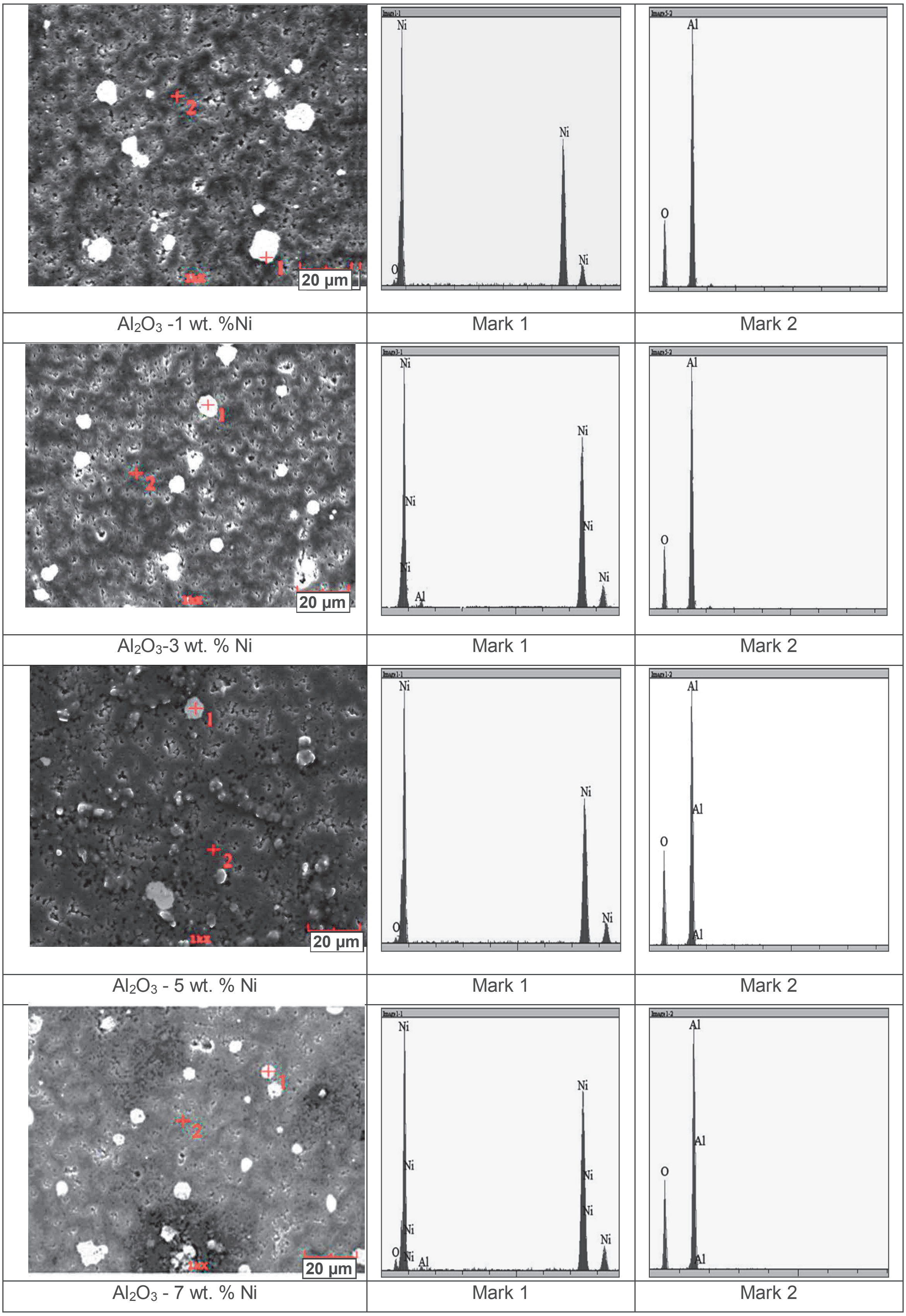

Figure 3 SEM images of Al_2O_3-Ni composites including EDS spectra

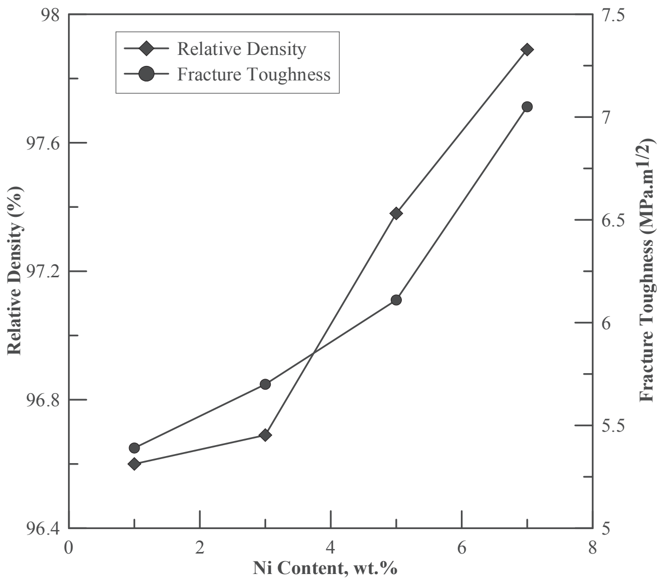

Figure 4 The variation of relative density and fracture toughness of Al_2O_3-Ni composites as a function of nickel amount

The electromagnetic shielding effectiveness (SE) behavior of a material can be defined by measuring transmitted power (or field) and incident power (or field) [13, 14, 17, 18]. The SE is defined as the logarithmic ratio of these two parameters in decibels as follows:

$$SE_{dB} = 10 \log (P_T/P_I) \quad (2)$$

$$SE_{dB} = 20 \log (E_T/E_I) \quad (3)$$

P_T and E_T are transmitted power and electric field values measured. P_I and E_I are incident values of same quantities. For the positive quantities, it can also be thought of as having a negative sign in the equation. Measurements of test materials have been carried on an Agilent E5071C ENA Series Network Analyzer and wave guide based setup [18-20] in the frequency ranges of 12.4-18 GHz (Ku band). The samples measured in this study are given in **Table 1** according to composition. Measurement results are shown in **Figure 5**.

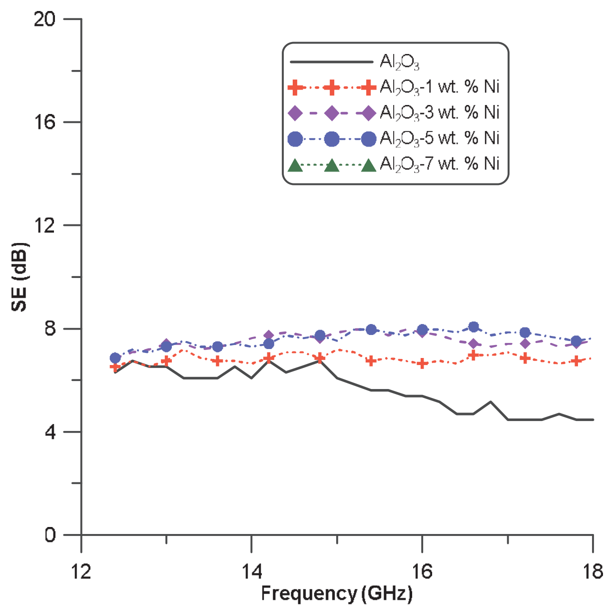

Figure 5 Electromagnetic shielding effectiveness measurement results for Al_2O_3 and sample composites

CONCLUSIONS

The authors developed the ceramic matrix composites with the metallic nickel reinforced material via powder metallurgical technique in graphite atmosphere. While using metallic particulate addictive it is aimed to increase not only mechanical properties such as fracture toughness but also preserve electromagnetic interference behavior of alumina type ceramic.

It is well known that the main problem in producing ductile phase-reinforced ceramic matrix composites is the oxidation of ductile phase. In order to overcome this problem in present study, sintering process of the test materials was carried out in graphite powder as a result of which oxidation was successfully prevented.

The presence of alumina and nickel were confirmed by x-ray diffraction (XRD) analysis and no oxidation of nickel was observed. SEM examinations on the cross-sections of test materials showed that Ni particles are uniformly distributed in the alumina matrix. The relative densities of the test materials, measured in accordance with Archimedes' principle, were 96.6 %, 96.7 %, 97.4 % and 97.9 %, respectively. The fracture toughness of test materials was 5.4, 5.7, 6.1 and 7.1 $MPa \cdot m^{1/2}$ and the corresponding values of hardness measured by Vickers indenter were 1560, 1506, 1467 and 1394 HVN. When the addition of nickel from 1% to 7 %, hardness decreased from 1560 HVN to 1394 HVN and indentation fracture toughness increased from 5.4 to 7.1 $MPa \cdot m^{1/2}$, respectively. Hence the results indicate that, the toughness of Al_2O_3-Ni composites has been improved 50 % compared to that of pure alumina which has a fracture toughness of 3.9 $MPa.m^{1/2}$. This increment in toughness can be attributed to crack deflection ability and absorbing energy of crack by ductile nickel phase. Also firing shrinkage values are increased from 20.14 % to 21.19 % depends on the Ni content. The electromagnetic shielding characteristics of composites have been investigated by measurements and results have been compared with the pure alumina. It is found that, despite increasing the Ni ratio in composition, within specified ratios in the study, electromagnetic wave shielding rates have remained almost same as it desired.

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