



Alumina matrix ceramic-nickel composites formed by centrifugal slip casting[☆]

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Abstract

The paper is focused on the possibility of fabricating the alumina matrix ceramic-nickel composites with gradient concentration of metal particles. Centrifugal slip casting method was chosen for the composite fabrication. This method allows fabrication of the graded distribution of nickel particles in the hollow cylinder composites. The horizontal rotation axis was applied. The samples were characterized by XRD, SEM and quantitative description of the microstructure. The macroscopic as well as SEM observations of the prepared composites confirmed the gradient concentration of Ni particles in the composite materials. The application of the centrifugal slip casting method allows for the graded distribution of metal particles in the samples.

Keywords: composites, centrifugal slip casting, SEM, structural characterization

I. Introduction

Functionally graded materials (FGM) are new variation of composites materials. These materials consist of two or more components and it is characterized by a compositional gradient from one component to the other [1]. In FGM composites chemical and physical properties vary gradually depending on position [1–4]. The gradual change of properties of components allows obtaining new materials with a wider range of usage as compared to traditional composites. Several techniques have been proposed to obtain the gradient structure in composites materials. These methods include: centrifugal solid-particle method [5], centrifugal method [6,7], tape casting [8,9], pulsed laser deposition (PLD) [10], self-propagating high-temperature synthesis – SHS [9,11], dry powder compaction [12]. It is reported in literature that among the methods of preparing functionally graded materials, the centrifugal casting technique is one of the most economical and popular method for producing continuous change in com-

position through the radius of sample [13]. An example of FGM can be ceramic-metal composites with gradient concentration of the metal particles. Such composites can be used as structural material, and also as functional materials for example in an aerospace industry.

This work primarily concerns with possibility of the fabricating the FGM using centrifugal slip casting. This method is a technique for powder processing that combines the effects of slip casting and centrifugation [3,14]. In this work, nickel and alumina powders were taken as the starting materials. The centrifugal slip casting allows fabrication of the graded distribution of nickel particles in the hollow cylinder composites.

II. Materials and methods

The following powders were used: α -Al₂O₃ TM-DAR from Taimei Chemicals (Japan) with average particle size 133±30 nm and density 3.96 g/cm³, Ni powder from Sigma Aldrich with average particle size 3 µm and density 8.9 g/cm³. The purity of the powders was as follows: α -Al₂O₃ - 99.99%, nickel - 99.99%. Because of the difference in densities of metal and ceramic, it is convenient to fabricate the alumina matrix ceramic-nickel composites with gradient concentration of metal particles by the compositional gradient in radial direc-

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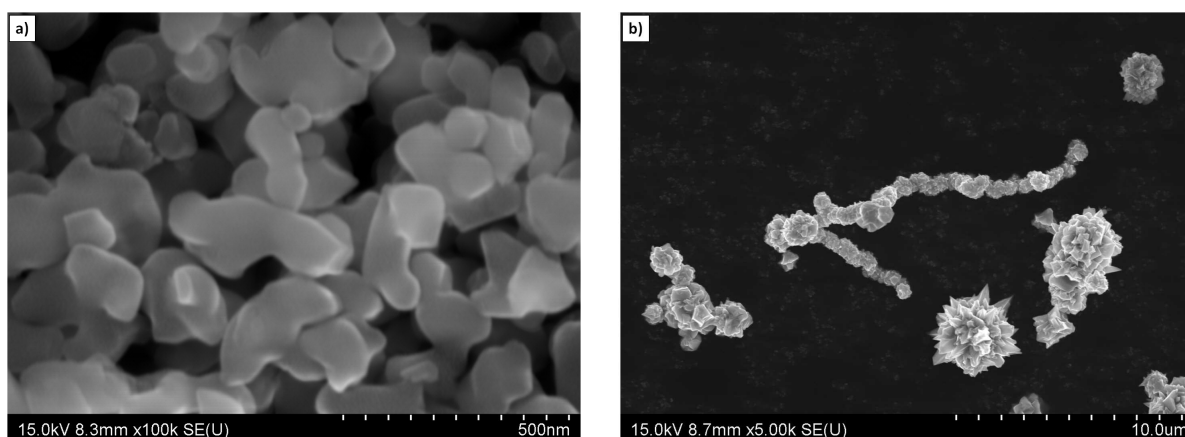


Figure 1. Scanning electron micrograph of the starting powders: a) α - Al_2O_3 powder, b) the Ni powder

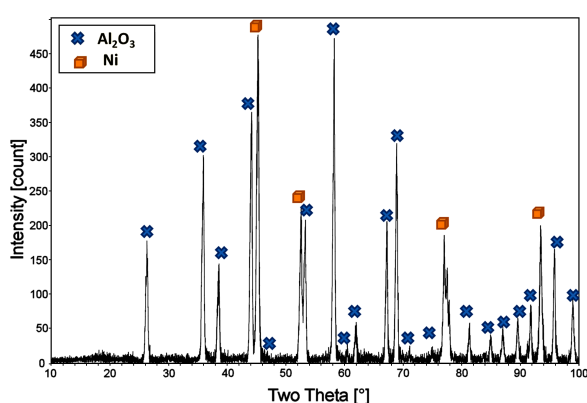


Figure 2. XRD pattern of sintered FGM composite

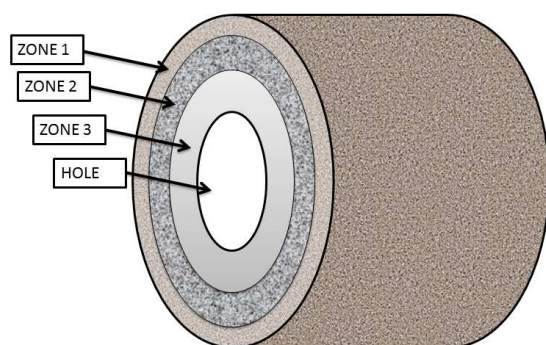


Figure 3. Schematic illustration of functionally graded tube samples

tion. Figure 1 shows the scanning electron micrographs of the α - Al_2O_3 and Ni powders. It was found that they were firmly agglomerated.

Ceramic water-based slurries with 50 vol.% solid content were prepared with 10 vol.% Ni powder with respect to the total solid volume. Diammonium hydrocitrate (puriss, POCh, Poland) and citric acid ($\geq 99.5\%$ Sigma-Aldrich) were used as dispersants in the ceramic slurries. Ceramic water-based slurries contained: diammonium hydrocitrate with 0.3 wt.% and citric acid with 0.1 wt.%, respectively, with respect to the total solid volume. Ceramic suspensions were prepared with

deionized water. Dispersants were added to water followed by alumina and nickel powders. The slurries were mixed in alumina containers in a planetary ball mill PM100 (Retsch) for 90 minutes with a speed of 300 rpm. Afterwards the slurries were degassed in a THINKY ARE-250 Mixer and Degassing Machine for 10 minutes with a speed of 800 rpm. The equipment allows to remove the air absorbed on the particles surface. The mixtures were cast into thick-walled tubes using a gypsum mold. The stirrer with vertical rotation axis was used in centrifugal slip casting process. The parameters of process were chosen by set of trials. Dimensions of the fabricated tubes were the following: length was 40 mm, thickness was 18 mm and outer radius was 20 mm. Afterwards, the samples were dried and removed from the gypsum mold. The last step was sintering at 1400 °C in reducing atmosphere (N_2/H_2).

X-ray diffraction (XRD) was used to identify the phases present in the samples. The structural studies were carried out by using a Rigaku MiniFlex II for 2θ values ranging from 10° to 80° with $\text{CuK}\alpha$ radiation and $\lambda = 1.54178 \text{ \AA}$. The analyses were done at the cross-sections of samples.

The microstructures of the sintered samples were observed on the cross-sectioned surfaces, cut perpendicular to the longitudinal axis of the hollow cylinders using scanning electron microscope – HITACHI SU-70. The samples were polished up with the diamond paste of 1 μm and 0.05 μm .

Quantitative description of the microstructure of the composites was made on the basis of SEM images of randomly selected areas on the cross - sections using computer image analyzer [15]. The objective of the study was to use the stereological analysis to determine the volume fraction of metal particles in graded region in the composites.

III. Results and discussion

The XRD patterns of the FGM composites (Fig. 2.) show that the composites sample consisted of two phases: Al_2O_3 and Ni. There was no presence of spinel phase (NiAl_2O_4) in the studied composites.

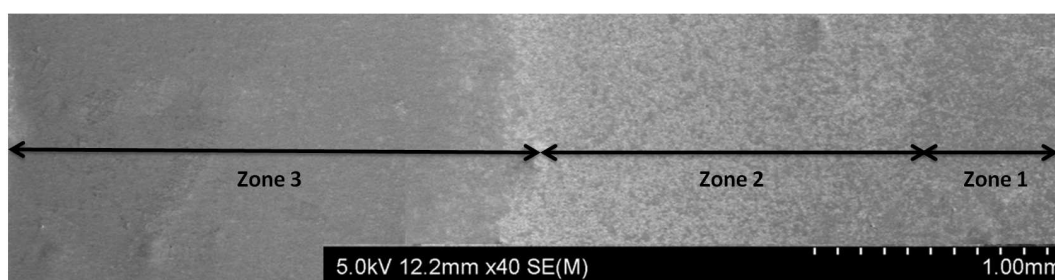


Figure 4. Typical microstructure of the composites were fabricated with speed of 1000 rotations per minute during 4 hours

Figure 3 shows the schematic illustration of the obtained composites with gradient concentration of metal particles. The functionally graded tube was radially divided into three regions. The sintered samples had only alumina regions at the inner side (zone 3) and nickel particles rich regions in the center region (zone 2) of hollow cylinder composites, while in the outer side (zone 1) the contents of the metal particles is less than in the zone 2.

Figure 4 shows the graded distribution of metal particles in Al_2O_3 -Ni composites. The grey area is Al_2O_3 and the bright area is Ni. The changes in microstructure locations are represented by three zones, from outer surface towards inner side of the graded cylindrical composite.

Quantitative description of the microstructure of the graded region in the composites was made on the basis of SEM images using computer image analyzer [15]. The objective of the study was to use the stereological analysis to determine the volume fraction of nickel particles in the composites. The image analysis results show that the outer periphery of the cylinder composites (the zone 1) contains 13 vol.% of Ni particles followed by gradient and in the zone 2 increases to 20 vol.%. Afterwards, Ni volume fraction in the zone 3 reduces from 8 vol.% to 0 vol.%. The volume fraction of Ni particles decreases below 8 vol.% to zero over the entire width of the zone 3 at 1.95 mm. The location of particles in the zone 1 is the result of removing fluid through capillary action in the gypsum mold. In contrast, the zone 2 was produced as a result of centrifugal acceleration. The zone 3 is composed entirely of Al_2O_3 , because the whole volume of Ni particles is located in the zones 1 and 2 as a result of the centrifugal process.

The analysis of histograms showed that in each zone the average Ni particle size is $3\ \mu\text{m}$ (Fig. 5). Moreover, it was observed that in the case of the zones 1, 2 and 3, the particles size distribution of Ni was similar.

The SEM images show the more uniform distribution of Ni particles in the zone 2 than in the zone 1 which confirmed result of location of Ni particles in this zone by capillary forces. In the zone 3 near the edge with the zone 2 absence of the nickel particles were observed.

IV. Conclusions

The application of centrifugal slip casting is a very good method for the fabrication graded materials such as alumina matrix ceramic-nickel composites in partic-

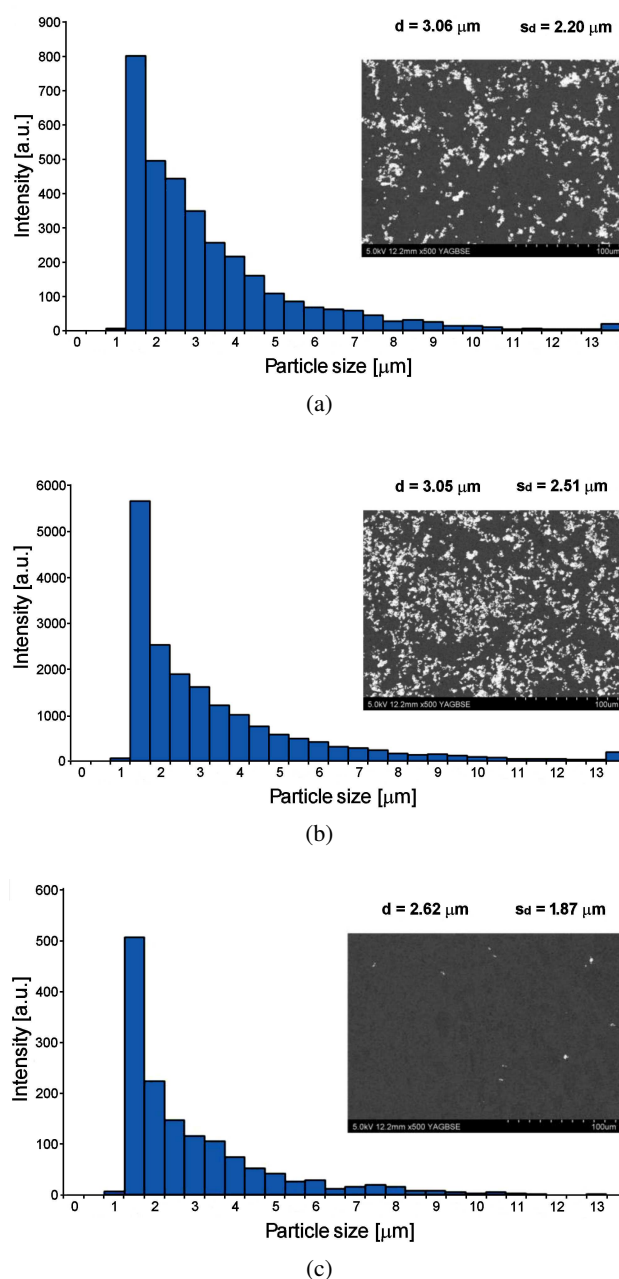


Figure 5. Distribution of size of Ni particles of the graded region in the composites: a) zone 1 b) zone 2 c) zone 3

ular with the hollow cylindrical shape. The gradient distribution of Ni particles is represented by three zones, from outer surface towards inner side of the composite. Maximum of 20 vol.% Ni particles are obtained in the

center region (zone 2), while minimum is observed near the inner periphery (zone 3) of the composite.

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