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COMPOSITES THEORY AND PRACTICE

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FORMING GRADED MICROSTRUCTURE OF Al_2O_3-Ni COMPOSITE BY CENTRIFUGAL SLIP CASTING

The aim of this study is to examine the possibility of fabricating ceramic-metal composites with a gradient concentration of metal particles from the Al_2O_3-Ni system. As the method of composite fabrication, centrifugal slip casting (CSC) was chosen. This method is a technique for powder processing, that combines the effects of slip casting and centrifugal casting. In this work one variant of the centrifugal casting method was used. The horizontal rotation axis was applied. Aqueous based slurries (with 50 vol.% content of solid phase) consisting of alumina and nickel powder (10 vol.%) were tested. The macroscopic as well SEM observations confirmed the gradient concentration of nickel particles in the composites.

Keywords: centrifugal slip casting (CSC), functional graded material (FGM), ceramic-metal composites, Al_2O_3-Ni system

FORMOWANIE MIKROSTRUKTURY GRADIENTOWEJ KOMPOZYTU Z UKŁADU Al_2O_3-Ni METODĄ ODLEWANIA ODŚRODKOWEGO

Celem niniejszej pracy było zbadanie możliwości wytwarzania kompozytów ceramiczno-metalowych z gradientem stężenia cząstek metalu z układu Al_2O_3-Ni. Jako metodę wytwarzania kompozytów wybrano odlewanie odśrodkowe mas lejnych (CSC). Metoda ta jest techniką przetwarzania proszku, która łączy w sobie metody: odlewania z gęstwy oraz odlewanie odśrodkowe. W pracy zastosowano metodę odlewania odśrodkowego w układzie z poziomą osią obrotu. Wodną zawiesinę o zawartość fazy stałej 50% obj. przygotowano na bazie tlenku glinu i proszku niklu (10% obj.) Makroskopowe oraz mikroskopowe obserwacje potwierdziły uzyskanie materiału z gradientem stężenia cząstek niklu.

Słowa kluczowe: odlewanie odśrodkowe mas lejnych (CSC), funkcjonalne materiały z gradientem (FGM), kompozyty ceramika-metal, Al_2O_3-Ni

INTRODUCTION

In recent years, new composite materials have been intensively researched. Among of them are functional graded material (FGM). These materials when compared to others are characterized by a microstructure varying in some spatial direction [1-4], and their chemical and physical properties change gradually with position [1]. Experimental works proved that the gradient of properties in a composite is caused by a position-dependent microstructure, which is obtained by various processing parameters or the chemical composition [1, 5]. Several methods have been proposed to fabricate a graded structure in composite materials such as plasma spraying, chemical vapour deposition, self-propagating high temperature synthesis and ordinary ceramic powder processing [6]. Literature data has reported that the centrifugal method is one of the most effective methods to obtain a functionally graded composite material like: metal-ceramic, ceramic-metal or ceramic-ceramic [7-9]. This method is a technique for powder processing that combines the effects of centrifugation and slip casting [13-16]. During this process of centrifugation, the particles within the slip move through the slurry at a rate influenced by particle drag and G-loading [11, 13]. The particles when moving through the liquid medium (slurry), are oriented before reaching the mold wall. In the centrifugal method, the spinning of the mould enhances the particle distribution. In contrast to the slip casting method, consolidation takes place when the fluid liquid is removed by capillary action and gravity sedimentation of the particles. [11].

Literature data suggests that the advantage of the combined action of the two processes - slip casting and centrifugation, is that particle orientation and the fluid removal process can take place at the same time [13-16]. In addition, the centrifugal slip casting of an isometric enables the texturing of tube-shape components [17].

The aim of this study is to examine the influence of centrifugal slip casting on ceramic-metal composites with a gradient concentration of metal particles from the Al_2O_3-Ni system.

MATERIALS AND METHODS

In the experiment the following materials were used: α-Al_2O_3 TM-DAR Company (Japan) with an average particle size of 133 ± 30 nm and a density of 3.96 g/cm^3 and Ni powder produced by the Sigma Aldrich Company of an average particle size of 3 μm and density of 8.9 g/cm^3. Figure 1 shows the scanning electron microscopy (secondary electron) image of the α-Al_2O_3 and Ni powders.

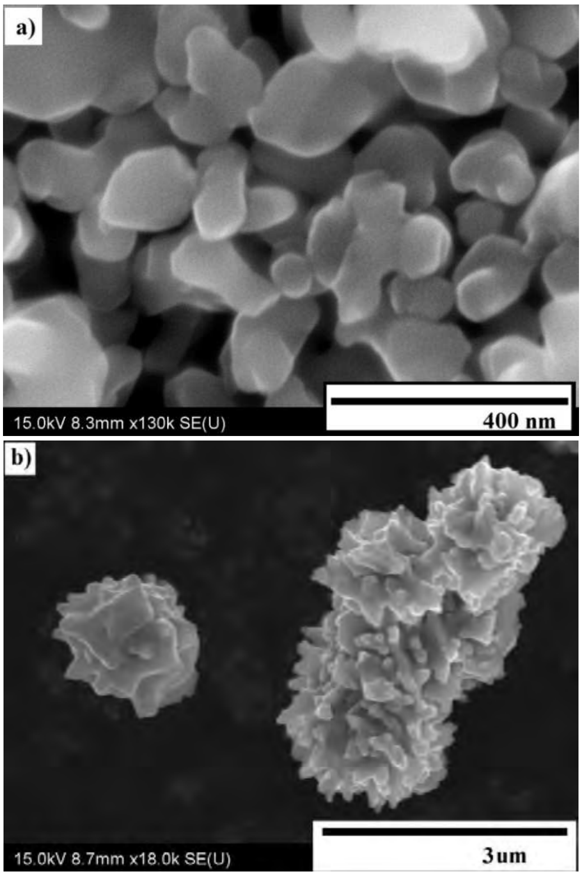

Fig. 1. SEM images of morphology of starting powders: a) α-Al_2O_3 powder, b) nickel powder

Rys. 1. Zdjęcie SEM wyjściowych proszków: a) proszek α-Al_2O_3, b) proszek niklu

Ceramic water-based slurries with a 50 vol.% solid content were prepared with 10 vol.% nickel powder with respect to the total solid volume. A composition of deflocculates i.e. diammonium citate (p.a., Aldrich) and citric acid (p.a., POCH Gliwice) was used. The ingredients were homogenized in a planetary mill at a rotating speed of 300 r.p.m. for 60 min. Afterwards, the air absorbed on the particle surface was removed by a THINKY ARE-250 planetary mixer. The mixtures were cast into thick-walled tubes using a plaster mold. A stirrer with a horizontal rotation axis was used in the centrifugal casting process. The process parameters were first chosen by a set of trials. The dimensions of the fabricated tubes are as follows: outer radius 20 mm, thickness 18 mm and length 40 mm. Figure 2 shows photos of the fabricated composites. Thereafter, the samples were dried and removed from the plaster mold. Final sintering was conducted on all the specimens at 1400°C in reducing atmosphere (N_2/H_2).

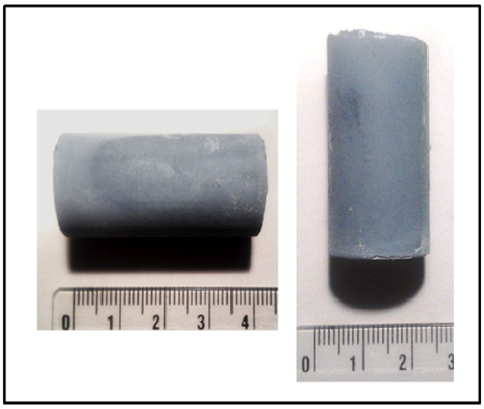

Fig. 2. Views of composites samples before sintering

Rys. 2. Widok próbek przed procesem spiekania

X-ray phase analysis was performed by a Rigaku MiniFlex X-ray diffractometer II (XRD) in the 2Θ mode with CuK$\alpha_{1.54}$ radiation.

Microstructure observations of the sample cross-sections were carried out using a high resolution scanning electron microscope - HITACHI SU-70. The cross-sections were prepared by cutting the samples across at 1/2 the height in the axial direction with a diamond saw. The polished cross-sections were prepared by grinding and polishing with 1 μm and 0.05 μm diamond paste. Quantitative description of the microstructure of the graded region in the composites was made on the basis of SEM images using computer image analysis equipped with the Micrometer program [18]. The parameters presented in this article were measured for individual cross-sections of the graded samples, and then the values of the studied parameters were averaged.

RESULTS AND DISCUSSION

Preliminary macroscopic observations of the samples before the sintering process (green body) showed that the composites specimen surfaces have a pale blue color, suggesting the presence of Ni content phases. The result of X-ray phase analysis from the cross-section of the sintered composites confirmed the presence of two phases: Al_2O_3 and Ni. Figure 3 presents an exemplary diffraction pattern. No cracks and pores on the surface of any of the samples were visible. In the macroscopic observation of the samples before sintering, a gradient concentration of Ni particles was not visible (Fig. 2).

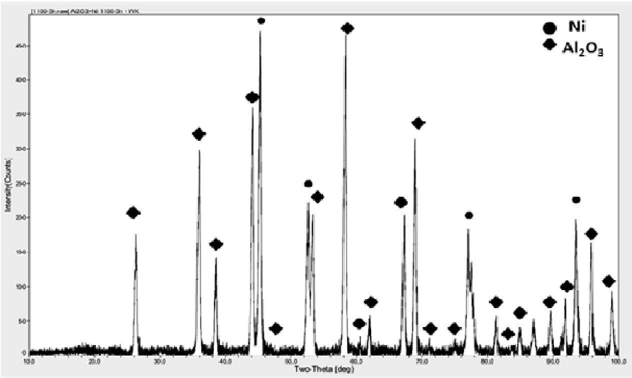

Fig. 3. XRD pattern of Al_2O_3-Ni composites

Rys. 3. Wynik rentgenowskiej analizy fazowej kompozytu Al_2O_3-Ni

The view of the sintered composite cross-section is presented in Figure 4. Three zones of different contrast (semi-dark grey, bright grey, dark grey) can be noticed. It suggests that the microstructure of the sample cross-section is not homogeneous, and it can be concluded the CSC process has given a gradient concentration of metal particles in the Al_2O_3-Ni composites.

A series of electron micrographs across the graded regions (masked in Fig. 4) are show in Figure 5.

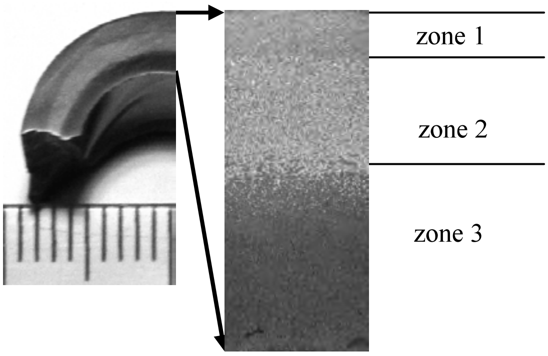

Fig. 4. Fragment of sintered composite sample cross-section fabricated by centrifugal slip casting in horizontal rotation axis, additionally marked zones I, II, III

Rys. 4. Fragment przekroju poprzecznego próbki kompozytowej wykonanej poprzez odlewanie odśrodkowe mas lejnych w poziomej osi obrotu, dodatkowo zaznaczono strefy I, II, III

In these micrographs, the nickel particles appear as bright spots. The changes in microstructure locations are represented by three zones in the sample, from the outer surface towards inner side of the graded hollow cylinder (Fig. 4). The first part of the graded region was a result of removing fluid through capillary action in the plaster mold. In the second part of the sample the micrographs reveal that Ni is relatively distributed and gradation was present from the outer side of the hollow cylinder towards the inner direction. This part was produced as a result of centrifugal acceleration. The third zone is composed almost entirely of Al_2O_3. The whole volume of the metallic phase is located in the first and second zones as a result of the centrifugal process.

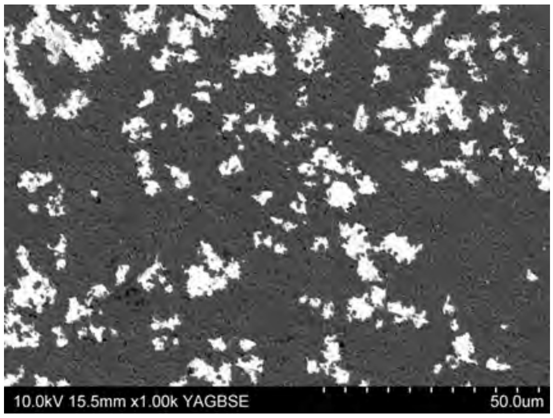

Fig. 5. Microstructures of specimens fabricated by centrifugal slip casting

Rys. 5. Mikrostruktura próbek wykonanych poprzez odlewanie odśrodkowe mas lejnych

Quantitative description of the microstructure of the graded region in the composites was made on the basis of BSE-SEM images using computer image analysis equipped in the Micrometer program [18]. The variations in the compositional profile across the graded specimens are shown in Figure 6.

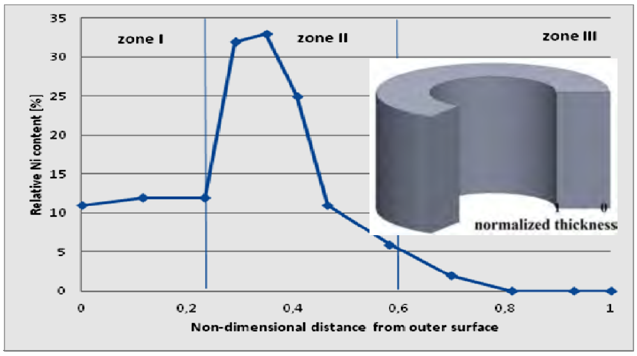

Fig. 6. Changes in sample metallic phase content from outer zone to inner zone

Rys. 6. Zmiany zawartości fazy metalicznej od zewnątrz próbki do wewnątrz

The quantitative description has shown that in the first zone, the nickel particle content was equal to 12 vol.% per 1 mm width. In the second zone there was a maximum of nickel particles equal to about 33 vol.% per 1.5 mm width. Then there was a sharp decline in nickel particles. However, in the third zone there was a mild decrease in the percentage of nickel particles down to 0 vol.%.

Additionally, the microstructures of the samples reveal that the methodology of slurry preparation has a vital influence on the gradation of Ni along the radial direction. In addition to the production of centrifugal slip casting, the driving force for particle consolidation is highest at the mold wall or largest radius and decreases linearly toward the center of rotation.

SUMMARY AND CONCLUSIONS

No additional phases in the composites except Ni and Al_2O_3 were present when sintering was carried out at 1400°C in reducing atmosphere (N_2/H_2).

Macroscopic observations as well as SEM revealed the graded microstructures of the composite. Three zones were distinguished, from the outer surface towards the inner side of the graded hollow cylinder. Application of the centrifugal casting technique allows for the graded distribution of particles in the composite material. The Ni particles were graded located in three zones. Starting from outer zone where 12 vol.% of Ni was found to 33 vol.% in the middle zone and finally 0 vol.% in the third central zone.

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