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$_2$O$_3$-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$_2$O$_3$-Ni system

**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$_2$O$_3$-Ni system.

**MATERIALS AND METHODS**

In the experiment the following materials were used: α-Al$_2$O$_3$ 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$_2$O$_3$ and Ni powders.

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 THINKKY 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$_2$).

**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$_2$O$_3$ 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).

![Image: XRD pattern of Al<sub>2</sub>O<sub>3</sub>-Ni composites](image1)

**Fig. 3.** XRD pattern of Al<sub>2</sub>O<sub>3</sub>-Ni composites

Rys. 3. Wynik rentgenowskiej analizy fazowej kompozytu Al<sub>2</sub>O<sub>3</sub>-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<sub>2</sub>O<sub>3</sub>-Ni composites.

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

![Image: Microstructures of specimens fabricated by centrifugal slip casting](image2)

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

Rys. 5. Mikrostruktura próbek wykonanych poprzez odlewanie ośrodkowe 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.

![Image: Changes in sample metallic phase content from outer zone to inner zone](image3)

**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 Al₂O₃ were present when sintering was carried out at 1400°C in reducing atmosphere (N₂/H₂).

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.

Acknowledgements

The results presented in this paper were obtained within the project from The Polish National Science Centre (NCN) No. 2013/11/B/ST8/0029.

REFERENCES


