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Al_2O_3-Ni composites produced with various rotational speed

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Abstract: - The recent years have seen the increasing practical interest in functionally graded materials (FGM), e.g. ceramic-metal composites, such as Al_2O_3-Ni. In the present study these composites were fabricated by the centrifugal slip casting method. The principal aim was to evaluate the influence of the centrifugal rotational speed on the microstructure and properties of the alumina-nickel composites. The slurries with the 55 vol.% solid content were consolidated using two rotational speeds (800 rpm and 1800 rpm). The composites obtained were characterized by macro and microstructural examination (XRD, SEM, EDS and stereological analysis). The hardness of the sinters was measured and compared. The graded distribution of the Ni particles in the Al_2O_3-Ni FGM ceramic matrix composites were only obtained at the centrifuge rotational speed of 1800 rpm. With the lower rotational speed of 800 rpm no gradient was achieved.

Keywords: - *Al_2O_3-Ni composites, FGMs, centrifugal slip casting*

I. INTRODUCTION

The increasing interest in ceramic-metal composites is chiefly associated with their specific properties. In view of the wide applicative possibilities of the Al_2O_3-Ni system many researchers undertake investigations of this compound [1-2]. Composites of this type combine the high wear resistance and high hardness of alumina with the better fracture toughness of the metal phase. Functionally graded materials (FGMs) have effectively been used in many applications since they offer the possibility of tailoring their properties gradually and avoiding the stress concentrations due to possible sharp changes of the properties between two neighboring regions [3]. The FGM composites are composed of two or more components whose concentrations are distributed in a gradient way [4-6]. Therefore, the tailored compositional gradient in the Al_2O_3-Ni composites permits adjusting their properties by altering the stress state.

According to literature reports [7-8] the FGM metal-ceramic composites can be fabricated by the centrifugal method. The centrifugal force is often applied to the mixture of the molten metal and the solid ceramic particles. The gradient can be obtained thanks to the difference in the densities between the molten metal and the particles. The ceramic-based materials can be produced using centrifugal forces by combining the action of the two processes: centrifugation and slip casting. In the centrifugal slip casting method (CSC): changes of the particle concentrations and the removal of the fluid through the porous mold take place at the same time. This method permits producing the composite in the form of a hollow cylinder. There are some literature publications concerning the preparation of ceramic materials such as Al_2O_3 [9-10] or ZrO_2 [11] by the CSC method. The advantages of this method lie in that we can fabricate green bodies with high relative density within a relatively short time. CSC also provides green and therefore sintered compacts with a small number of defects, because the air bubbles are removed by the centrifugal force [12]. Other investigators also report that the materials obtained by centrifugal casting are less defected compared to the materials formed by uniaxial or isostatic pressing [13-14]. Utilizing the difference in the mobility of the ceramic and metal particles under the centrifugal force, the CSC method can be used for producing functionally graded composites. Our own earlier researches have shown that the graded Al_2O_3-Ni composites can also be produced using the centrifugal slip casting method [15-16]. The formation of the gradient in these composites depends on many factors, such as e.g. the solid content in the slurry, the particle size of the powders, the amount of the metallic phase, and the rotational speed employed in the mixing. The present investigation was focused on the influence of the rotational speed on the microstructure of the graded Al_2O_3-10 vol.% Ni composite formed from a slurry with the 55 vol.% solid content. Two different rotational speeds were used in the experiments.

Composites were characterized by XRD, SEM, EDS and a stereological analysis. The hardness was measured using the Vickers method

II. MATERIALS AND METHODS

The starting materials were: an α-Al_2O_3 powder with 99,99% purity and an average particle size of 0.1 µm, and a Ni powder with 99.99% purity and an average particle size of 3 µm. The particle sizes were verified by laser diffraction measurements (Laser Particle Size Analyzer LA-960) conducted in a diluted well-dispersed suspension. The dispersing agents were the diammonium citrate and citric acid. Using the slurry with a proper pH (7.04), the surface of Al_2O_3 and Ni particles can absorb the negative charge sufficient to obtain a stable suspension. The SEM observations revealed that the α-Al_2O_3 and Ni particles show the tendency to create unstable agglomerates which disintegrate in an aqueous environment (Fig.1).

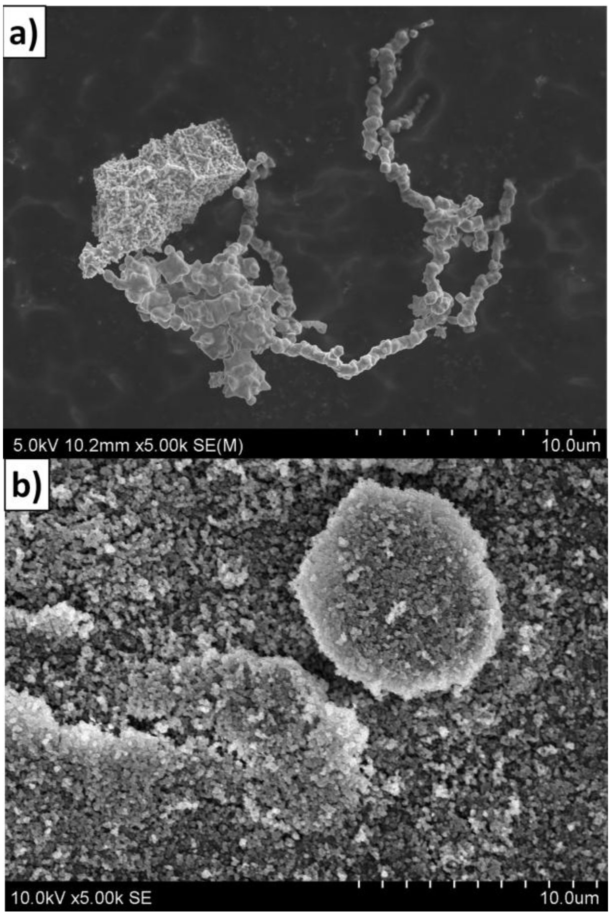

Fig.1. Scanning electron micrographs of the Ni powder (a) and the α-Al_2O_3 powder (b)

The aqueous ceramic slurries contained 55 vol.% of the solid phase and 10 vol.% of the nickel particles (with respect to the total solid volume). The slurries were prepared by adding the alumina and nickel powders to the water solution and milling the mixture in a planetary ball mill with rotational speed of 300 rpm for 1 hour. The aqueous suspensions were poured into a gypsum mold with the inner diameter of 0.02 m. Then, the tubular mold was centrifuged in the radial direction with the specified speed for 4 hours. After the centrifugation, the sample together with a gypsum mold was removed from the metal mold and the gypsum mold with the sample inside was dried in the vertical position in a vacuum chamber at 25°C for 24 hours. The sample, dried and shrunk, can be easily removed from the gypsum mold. Then, the sample was sintered at 1400°C in a H_2/N_2 atmosphere (N_2 of 80 vol.% and balance H_2). During the sintering, the heating and cooling rate were 5°C/min. The flow chart of the centrifugal slip casting process is shown in Fig.2.

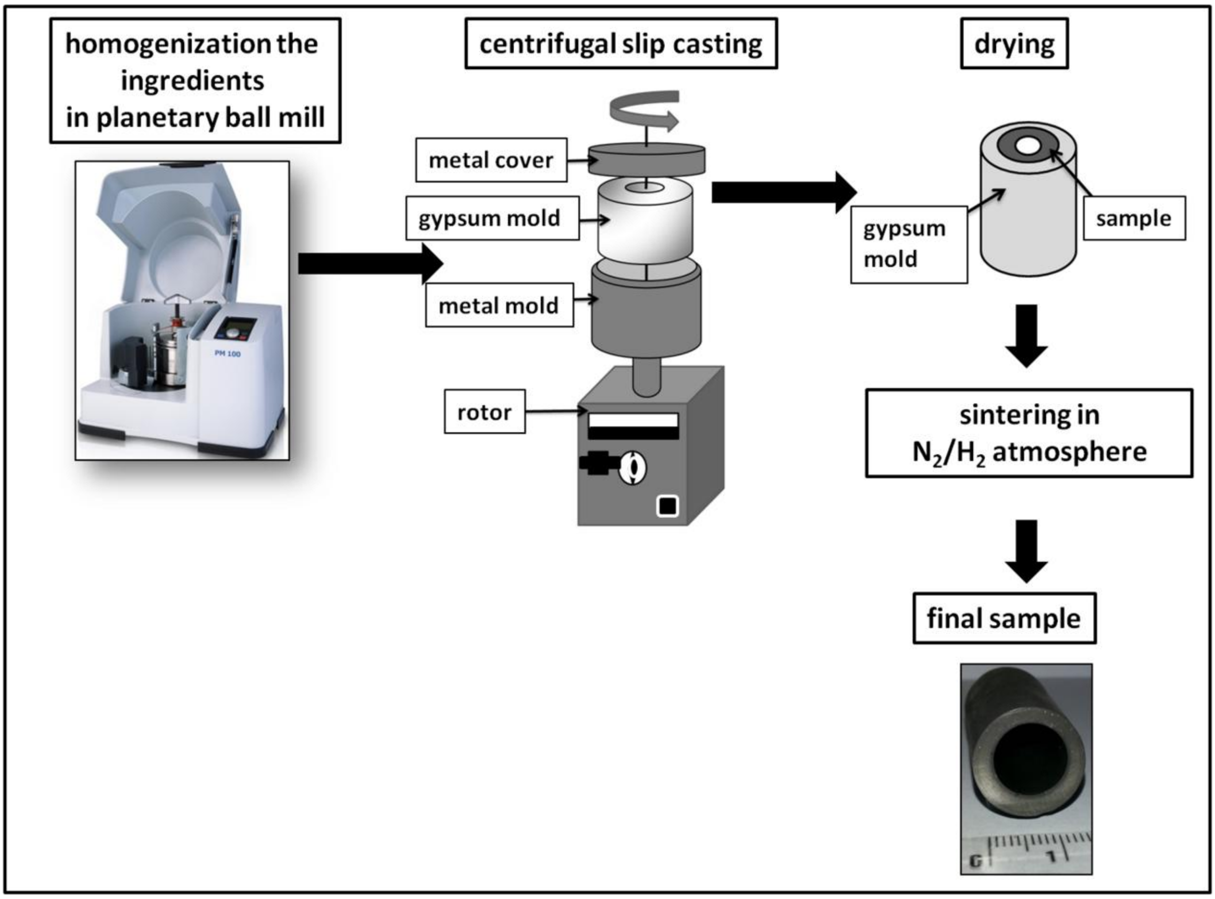

Fig.2. Flow chart of the centrifugal slip casting process

Two series of the samples were prepared using two different centrifugation rotational speeds: series I - 800 rpm, series II - 1800 rpm.

The X-ray diffraction measurements were performed in a Rigaku MiniFlex II diffractometer with $CuK_{\alpha 1.54}$ (λ = 1.54178 Å radiation). The results were obtained in the form of plots of the diffracted intensities as a function of 2θ on the surface of both the series.

Certain selected physical properties of the composites obtained were measured by the Archimedes method according to the Standard PN-76/E-06307. The theoretical density was calculated according to the rule of mixture to be: 3.96 g/cm^3 for Al_2O_3 and 8.9 g/cm^3 for Ni.

The microstructure of the samples was analyzed on a polished sample cross-section using scanning electron microscopy (HITACHI SU-70). The changes of the size distribution of the Al_2O_3 particles in the graded composites were determined using a stereological analysis. Observations were made in the samples after their thermal etching.

The chemical composition was examined in microareas on a cross-section of the sample by EDS (HITACHI S-3500N).

The hardness of the composites was measured by the Vickers method on the polished sample surface along the radial direction under a load of 9.8 N. The measurements were made at equal distance intervals (300 μm) to detect the compositional changes.

III. RESULTS

Figure 3 shows typical diffracted intensities obtained for Ni and Al_2O_3 in the samples series I and series II. The diffraction patterns obtained for series II appeared to be similar to those obtained for series I. Application of the reductive atmosphere (H_2/N_2) during the sintering permitted avoiding the formation of the alumina nickel spinel phase ($NiAl_2O_4$) which frequently during this system (Al_2O_3-Ni) [17-18].

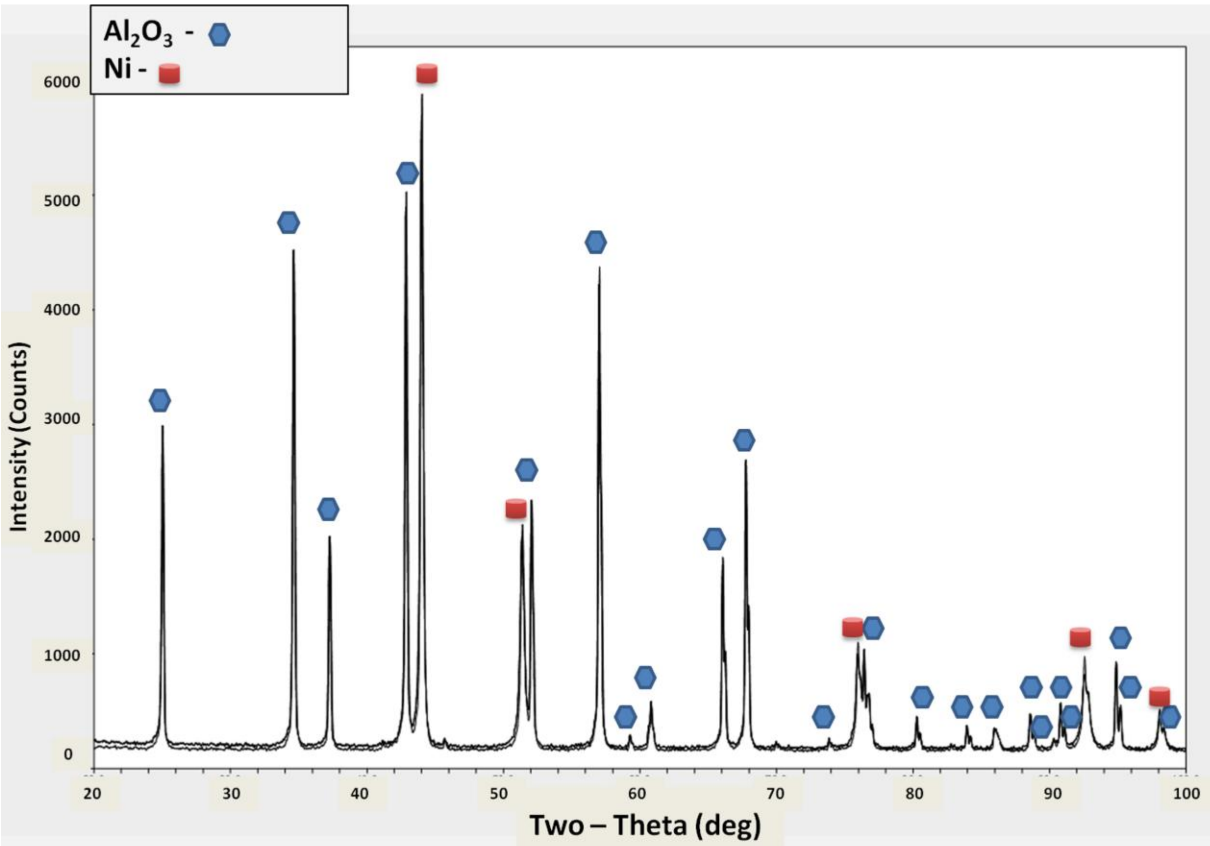

Fig.3. X-ray diffraction analysis of the ceramic-metal composite (series I and series II)

The composites made by centrifugal slip casting have the shape of hollow cylinders. In series I, the relative density appeared to be lower than that in series II. After the sintering the relative density was 94.8 ± 0.5% in series I and 99.0 ± 0.6% in series II. The low relative density of the series I samples can be explained by the presence of pores throughout the sample volume. This is so since in the case of series II, the greater centrifugal force favored better packing of the particles in the green body state, which resulted in the higher density after the sintering process. Moreover, series I contains a greater number of open pores (3.37 ± 1.39 %) than series II (0.67 ± 0.24 %).

Typical microstructures observed on cross-sections of the samples are shown in Fig. 4. It can be seen that the samples of series I have a homogeneous porous microstructure contrary to the samples of series II. The rotational speed used for series I was too low to remove the air bubbles present in the slurry. Moreover the concentration of the nickel particles did not vary. In series II the inner zone did not contain the metal particles. Because of the high solid particle content in the suspension (55 vol. %) the distances between the particles were quite short there. Moreover, the particles could interfere with each other which also reduced their mobility. It was only with the use of a greater centrifugal force (series II) when the gradient structure could be obtained

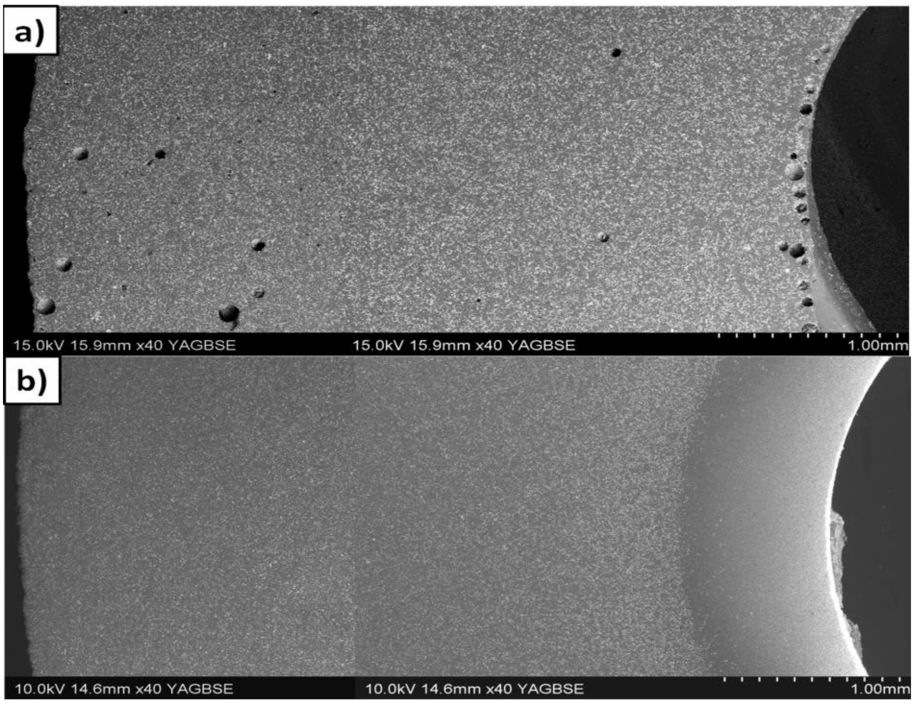

Fig.4. Microstructures of the Al_2O_3-Ni FGM hollow cylinder fabricated by centrifugal slip casting: a) series I b) series II

To prepare the samples to stereological examinations (determination of the Al_2O_3) grain size) they were subjected to thermal etching. The particle size of Al_2O_3 in series I and II are presented in the form of the violin plots. The series I samples had a uniform grain size throughout the sample volume, whereas in series II the Al_2O_3 particle size was varied with the greatest particles being observed in the outer and central parts of the sample and the smallest in its inner part. In series II the action of the centrifugal force could in addition result in the gradation of the alumina particles concentration.

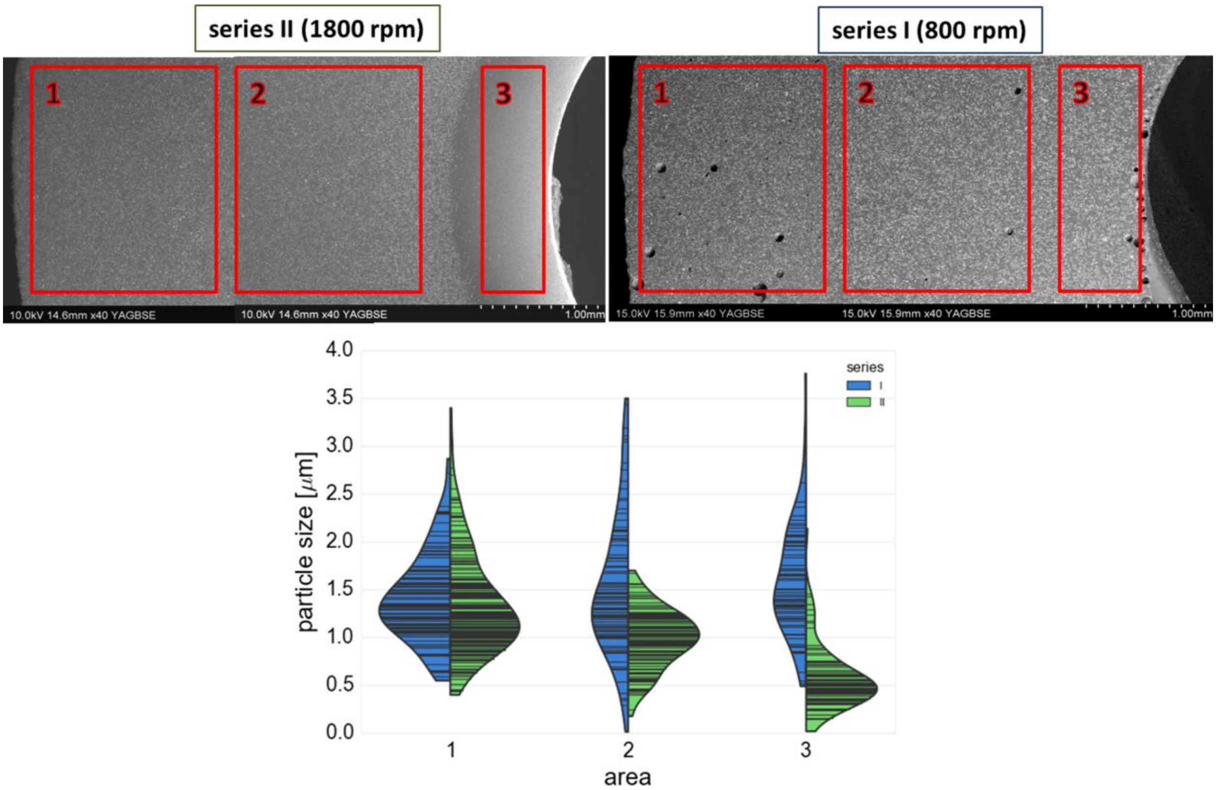

Fig.5. Violin plots of the Al_2O_3 particle size after thermal etching (series I and II)

That the gradient structure had been obtained was confirmed by the EDS examinations (Fig.6). The EDS analysis shows that all the samples contained only Al, Ni, O. In series I, the elements (Al, O, Ni) were uniformly distributed throughout the sample. The chemical composition was assessed at Al - 53.8 ± 1.98 wt.%, Ni – 20.13 ± 1.11 wt.%, O – 25.6 ± 1.68 wt.% in the whole sample volume. whereas in series II the chemical composition gradually changed from alumina through oxygen to nickel. The Ni content in the outer and central regions of the sample series II was estimated at 23.12 ± 1.93 wt.%, and it decreased to 0 wt.% in the inner part of the sample, whereas the oxygen and aluminum contents were similar (oxygen - 27.00 ± 5.12 wt.%, and aluminum - 55.09 ± 5.52 wt.%). The absence of the Ni particles in the innermost part of the sample in series II can be attributed to the fact that they all are gathered in the outer and central parts of the composite. This is so since from the outer part they were removed together with the fluid by the capillary forces active in the gypsum mold and in the central and inner parts of the composite they were distributed by the centrifugal acceleration.

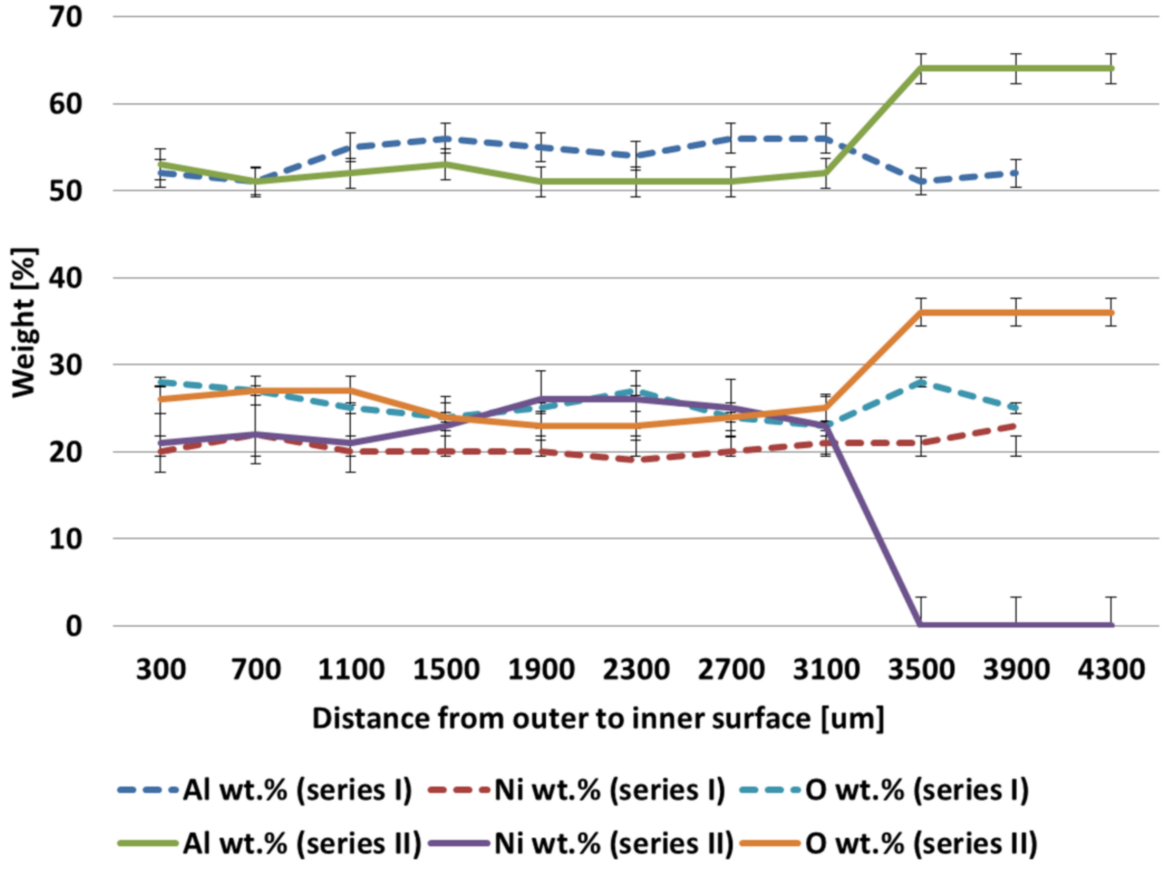

Fig.6. Compositional analysis (wt.%) of the Al, Ni, O content in series I and II

Figure 7 shows the hardness of the samples (series I, series II, and the sample with Al_2O_3 alone) as a function of the distance from the outer to the inner surface (Fig.7) of the sample. It can be seen that in series I the hardness ranges from 1200 to 1800 HV which can be attributed to the defects generated in the sample. In series II, the hardness increases in the Al_2O_3-rich regions and decreases in Ni-rich regions. The regions with the maximum amount of alumina shows the highest hardness similar to that of the sample prepared from Al_2O_3 alone. The resulting hardness can also depend on the differentiation of the grain size of the composite matrix

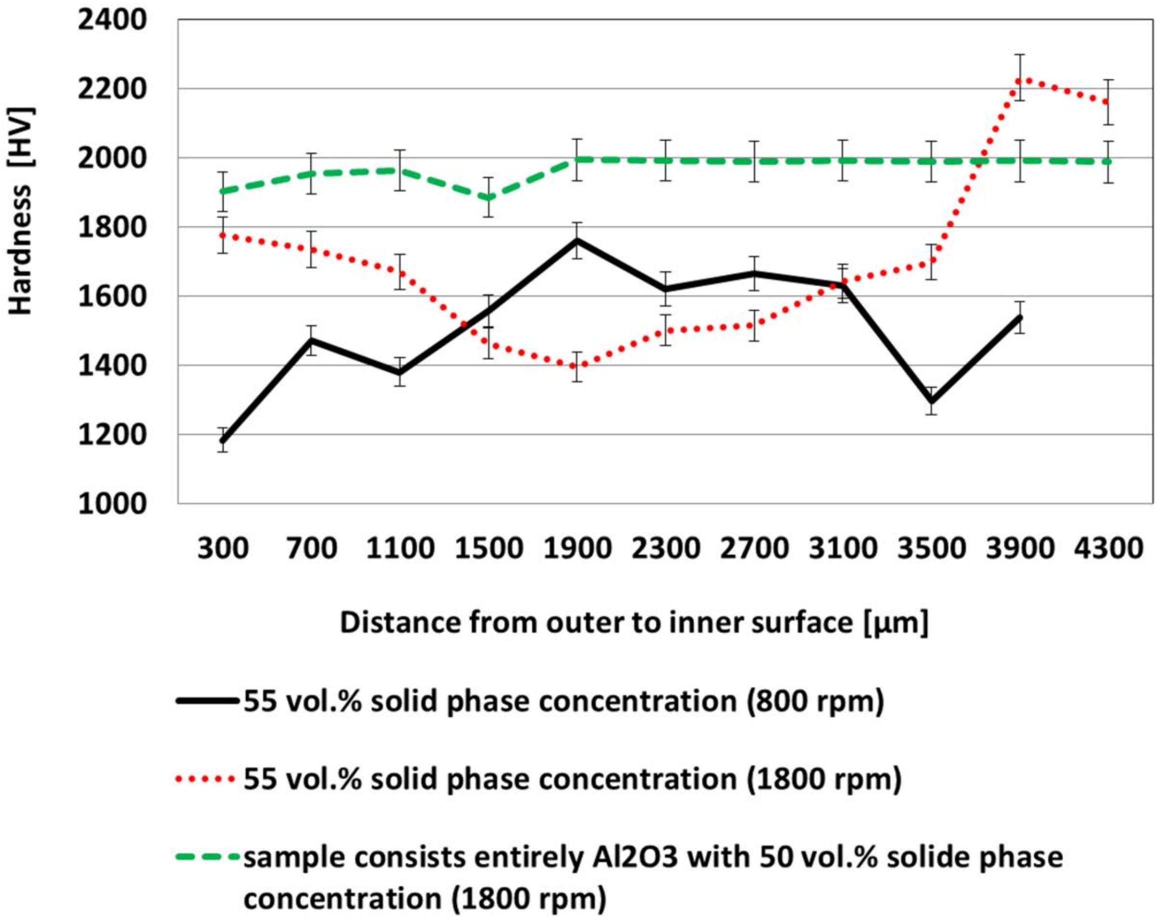

Fig.7. Vickers hardness profile from the outer edge to the inner edge of the sample

Figure 8 shows the crack path visible on the polished cross section of the composite series II after the Vickers indentation. It can be seen that the crack proceeds along the nickel/alumina interface. The crack is deflected by a metal inclusion.

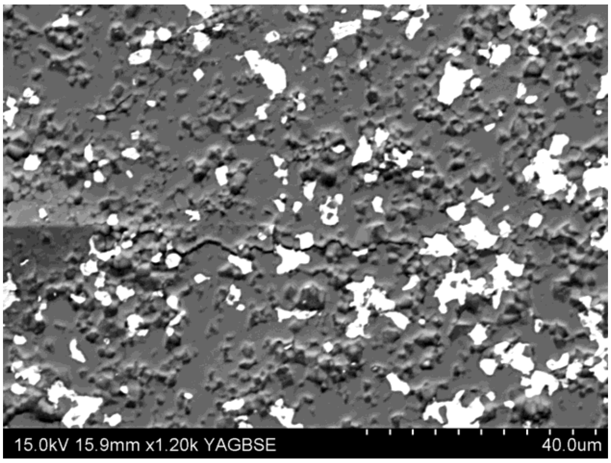

Fig.8. SEM micrograph showing an indentation crack in the Al_2O_3-Ni composite

IV. CONCLUSIONS

The microstructure and properties of the composite materials obtained by centrifugal slip casting depends on the two competing phenomena: the movement of the particles in the slurry under the action of the centrifugal force (which takes place in the entire volume of the sample) and the decrease in the liquidity of the slurry due to the removal of the fluid from the gypsum mould by the capillary forces (when the slurry is in contact with the mould). The proportion between the effects of these two mechanisms chiefly depends on the speed of casting and the properties of the slurry. If the rotational speed was too low (series I), the centrifugal force cannot activate the metallic particles to move intensively so that the graded structure cannot be formed (series I). With the higher centrifugal force (series II), the movement of the nickel particles is intensified and we obtain the required graded structure.

The present study has also shown that using a sufficiently high centrifugal force we can eliminate de-aeration of the slurries, which shortens the time of the process and facilitates its procedures

When producing a material with the graded structure we can easily control the distribution of hardness on its cross-section.

Understanding the mechanism of formation of the FGM microstructures produced by centrifugal slip casting allows us to design ceramic-metal composites with the desired characteristics.

V. ACKNOWLEDGEMENTS

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