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Characterization of Al_2O_3/Ni composites manufactured via CSC technique in magnetic field

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ABSTRACT

In the case of this article, the research were involved an innovative concept of forming composites materials. The goal of this study was to determine the possibility of forming microstructure of Al_2O_3/Ni composites with a gradient structure by new method combining centrifugal casting with the action of magnetic force. By using this innovative method, it was possible to obtain a composite material with a zone structure. The use of the magnetic field resulted in the characteristic distribution of the metallic phase in the ceramic matrix. The material was characterized by negligible porosity due to the application of centrifugal force. Gradient structure was confirmed by SEM observation, EDS measurements and XRD analysis. In addition, selected mechanical properties (hardness, compression strength) were characterized.

1. Introduction

Due to the constantly increasing demand for materials with unique and new properties, there is a need to seek technological solutions allowing to obtain such materials. The existing solutions do not meet the required standard. In particular, it refers to a production of composite materials. The increasing interest in ceramic-metal composites is mainly associated with their specific properties. Since the 80's of the XX century, the ceramic-metal composites aroused considerable interest primarily due to their properties [1]. Ceramic-metal composites are a group of materials that could combine the following properties: high hardness, heat resistance and corrosion resistance [2,3]. The metal is mainly introduced into the ceramic matrix in the form of particles. With the addition of the metallic phase we can obtain a noticeable improvement in fracture toughness, which allows a wider range of application than in case of monolithic ceramic materials [3–7]. The increase of fracture toughness is dependent on several factors. Due to the presence of ductile particle the cracks could be deflected by a slowdown or bridging which prevented their further propagation. Moreover, nickel particles can cause crack relaxation via local plastic flow. These processes and their dominance in the ceramic-metal system depend, inter alia, on factors such as: amount, size, shape of the ductile phase and on the uniformity of metallic phase distribution [7,8]. According to the literature and also the own research it is reported that the size of the metal phase has a significant effect on the crack propagation in the composites [1,2,8–10]. In addition, the metallic phase distributed throughout the ceramic matrix can modify its magnetic, electric and thermal properties. Ceramic matrix composites with functionally gradient (FGM) are a particular group of composites in which another phase is distributed in a specific way in a volume of material. These materials are characterized by the variation in composition and structure gradually over volume, resulting in corresponding changes in the chemical and physical properties of the composite [11,12].

The current state of knowledge indicates that graded materials can be prepared by a variety of methods differ fundamentally. Currently, one of the most popular technique for producing FGM are powder technology method, inter alia: dry powder compaction [13,14], tape casting [11,13,15–18], self-propagating high - temperature synthesis - SHS [18,19], slip casting and filtration [7,20–24]. However, techniques such as in – situ methods: spray forming [25,26], centrifugal casting [27,28] or deposition methods: Electrophoretic Deposition - EDP [29–31], Pulsed Laser Deposition - PLD [32,33] or centrifugal slip casting technique [34] gained broad attention. Ceramic-metal composites with gradient concentration of the metal particles are an example of FGM materials. The ceramic-metal composites may be produced by various methods such as infiltration of porous ceramics by liquid metal [35], slip casting method [7] and others. It is also possible to create a specific gradient structure by applying a magnetic field during the

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process of samples forming [7]. A very important and useful tool in the production of composites is the use of analytical models. Literature data show that Barrett and his team were conducted an interesting research on contributions on analytical modelling of functionally gradient materials [36–39]. These studies allows the prediction of the properties of composites with a gradient microstructure is a complicated issue. Nevertheless in the case of such complex systems is still a big problem to conscious control the microstructure of the final composite. As a result, are still being developed a new technology to create a new methods to produce gradient materials.

Own research shown that centrifugal slip casting technique allows the production of ceramic-metal composite with or without gradient of the metallic phase [34,40,41]. Centrifugal slip casting method (CSC) combines the classic slip casting with the centrifugal force. As a result of using of the porous gypsum molds we can gained removal of the liquid medium from the slurry, which in turn leads to compaction of the material. Additionally, the use of centrifugal force causes the simultaneous variable deployment of metal particles in a ceramic matrix. Previous studies have shown that this method allows the use of smaller value of centrifugal acceleration than the classical method of centrifugal casting composites with metallic matrix, which allows us to shorten the process time. The process consumes less energy than classical centrifugal casting. The centrifugal slip casting technique allows the achieving of a finished product in the shape of a hollow cylinder. In addition, the finished product obtained by this method has a high relative density after sintering.

Therefore, it would be useful to extend the research carried out so far to formation of gradient of metallic phase via the centrifugal slip casting with magnetic field. The novelty of this article is the use of a magnetic field, which will allow the influence of change of concentration of Ni content in the sample. The article had applied the original concept of formation of materials as gradient composites. Formation by centrifugal slip casting with magnetic field was used as a the innovative technology for fabricate the composites. This was allowed to create a gradient structure with properties that vary across the samples. Simultaneously, the classic idea of materials science engineering would be used to determine the possibility of forming microstructure of Al_2O_3/Ni composite in an appropriately modified process. Obtained results of experiments would be used to develop the basic of technological aspects of shaping of Al_2O_3/Ni with a gradient of metallic phase using a centrifugal slip casting using a magnetic field. Thanks to that, it should be possible to tailor the microstructural and mechanical properties of FGM ceramic-metal composites.

2. Experimental

The powder materials in this work were alumina powder from Taimei Chemicals (Japan) of average particle size of 0.1 μm and purity of 99.99% and nickel powder from Sigma Aldrich (Poland) of average particle size of 27 μm and purity of 99.99%. The density given by the manufacturer of powders was as follows: α-Al_2O_3 – 3.96 g/cm^3, Ni – 8.9 g/cm^3. The density of powders was also measured with the use of a helium pycnometer AccuPycII 1340 Pycnometer (Micromeritics, USA), according to Boyle's Law. The powders before measurement were kept in a desiccator at the room temperature (25 °C). Afterwards, the sample mass was recorded as m_s and limited by a volume of a sample cup (2.76 cm^3). The cup filled with the powder was placed in the pycnometer sample compartment and locked firmly. The compartment was purged with the gas under pressure not exceeding 13.8 kPa (2.0 psi). The pressure upon filling the sample chamber and then discharging into a second empty chamber permitted the computation of the sample solid phase volume. Hundred gas purges of each sample and hundred measurements were carried out. The specific gravity was determined by dividing m_s by the determined sample volume v_s. The specific surface area of the powders was measured by BET method (ASAP, 2020, Micromeritics, USA).

The magnetic properties of nickel powder were examined in a Lake Shore 7010 vibrating sample magnetometer (VSM) in which the sample vibrates perpendicularly to the applied magnetic field. The sample was placed at the end of the non-magnetic rod and was suspended between the pole pieces of the electromagnet. The sample was vibrated. The movement of sample in the magnetic field induces a variable voltage signal in the measurement coil system. This signal was proportional to the magnetic permeability of the medium, amplitude and frequency of vibrations. The measurement allowed to determine the magnetic susceptibility of the material and magnetization changes as a function of the external magnetic field.

For materials molded by slip casting method, a suspension with highest solid phase concentration (equal 50 vol%), high homogeneity and stability over time is required. The production of such a slurry is only possible with the use of appropriate selected system of deflocculates – compounds capable of changing the double electrical layer around the particles in suspension producing a steric barrier of appropriate thickness to prevent the particles from approaching and coagulation, or acting by electrodeposition [42]. The effect of metallic powders on the electrokinetic stability of the suspension was determined. The stability of the of the powders in the slurries were measured as a function of pH through zeta potential, which were performed using Zetasizer Nano ZS (Malvern Instruments). The ionic strength was fixed with 10^{-3} M NaCl. The pH adjustments were made by adding appropriate amounts of NaOH and HCl. The pH of suspension was measured on LAB 850 Schott pH-meter. The pH values of the suspensions were measured immediately after the suspensions were made. Prior to measurements, all suspensions were homogenized with an ultrasounds probe for 1 min. The measured pH value was read 2 min after the immersion of the electrode in the suspension. This work constituted the technological preparation for conducting basic experimental.

During the first stage of composites preparation the aqueous slurry containing 50 vol% of the solid phase and including 10 vol% of the nickel particles was prepared by adding the alumina and metal powders to the solvent with the dispersants. Dispersion was achieved by adding 0.3 wt% of diammonium hydrocitrate, DAC (puriss, POCh, Poland) and 0.1 wt% citric acid, CA (≥99.5% Sigma-Aldrich, Poland) referred to total solid weight, in accordance with our previously study [40,41]. For ecological and economic reasons, water was used as a solvent. The slurries were place in an alumina containers and mixed using a planetary ball mill (PM100, Retsch) for 60 min with a speed of 300 rpm. Slurries were degassed in a THINKY ARE-250 Mixer and Degassing Machine for 5 min at a speed of 1800 rpm. The equipment allows to release bubbles > 1 μm from the slurry, whereby it reduce the probability of defects in the composite. The optimum parameters of the process were first established by a series of trials. After mixing, the slurries were cast into thick-walled tubes contained a gypsum mold which was rotated around its vertical axis. Around the plaster mold the magnets were arranged to generate a magnetic field during the process. Scheme of location of magnets was shown in Fig. 1. The composites were fabricated at a rotational speed of 1500 rpm for 90 min. The strength of the magnetic field to fabricate the ceramic metal composites during the experiment was 0.5 T was measured by Teslametr HGS-10 A. The prepared samples after the casting process were subjected to drying and then sintered. The heating process during sintering was divided into three stages. The heating rate to the temperature 120 °C was 5 °C/min and then increased to 1 °C/min to the temperature 750 °C and during the last part of sintered decreased to 2 °C/min to the temperature 1400 °C. The dwell time was 2 h. The sintering temperature was chosen below the melting point of the Ni (1453 °C). Sintering was carried out in a reducing atmosphere (N_2 of 80 vol% and balance H_2). The six hollow shape composites samples were manufacturing for the study.

The phase analysis of the prepared composites was made by using X-ray analysis on the Rigaku MiniFlex II diffractometer using CuK_α of the