Investigation on microstructure and selected properties of aluminum oxide–copper–nickel ceramic–metal composites

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Abstract
A new ternary system of ceramic–metal composites has been fabricated from alumina, copper, and nickel powders by the slip casting method. The research aimed to avoid copper leaking during the sintering process of the formation while processing the CuNi phase. Five different compositions were examined, differing in the ratio of nickel to copper. Stable slurries with good casting properties were prepared for all compositions. The final composites after the sintering process were characterized by a homogeneous distribution of the metallic phase, but they differed in the microstructure characteristics. The addition of nickel limits the outflow of the liquid phase (copper) during sintering but does not eliminate it. This resulted in a reduction in the hardness of the samples compared to the reference material.

Keywords Composites • Slip casting • Scanning electron microscopy • Hardness

Introduction
The history of composites formed by humans dates to the distance even up to 10,000 years BC. The composites as a separate group of materials were extracted in the twentieth century (Heinmann et al. 2010). They were created mainly to discover new or improved construction materials, e.g., due to their strength properties (Kendall 2004; Kassapoglou 2020; Batdorf 1994). As a result of the constant development of civilization, there was a necessity and need to discover novel materials and improve production methods. It was found that one of the fastest developing areas of life, which use composite materials, includes, among others: electronics and medicine. Compared to ceramic, metal, or polymer materials, composites make it possible to manufacture elements working in highly different conditions that traditional materials cannot work with.

The great advantage of composite materials is the ability to design the structure in such a way as to obtain a material with the desired properties. The literature data have shown that ceramic matrix composites are a promising and intensively developed group of composites mainly due to such properties as high hardness and strength, resistance to chemical substances, high melting point, or electrical, thermal, or optical properties (von Helden et al. 2019; Amateau 1998; Clegg 1998; de Portu and Guicciardi 2014; Glenny 1970; Zgalat-Lozynskyy et al. 2001; Stroe et al. 2020). A significant problem with these materials is their brittleness. This is why they are not used on elements working under high load. This problem may be the production of a ceramic matrix composite with a metallic phase (Guzanová et al. 2019; Restivo et al. 2019; Rebak 2020; Hufenbach and Gerhardy 1996). Various ceramic materials are currently used to form ceramic–metal composites, both in the monocrystalline and polycrystalline form (Pyzik et al. 1987). Based on the literature data, the most common materials used for forming the ceramic matrix in composites are alumina, silicon carbide, tungsten carbide, or zirconium oxide (Włosiński 1984; Wang et al. 2008; Gong et al. 2019; Bucevac 2018). While the most commonly used metallic powders added to the ceramic matrix include: copper (Liu et al. 2017; Yin...
et al. 2020), nickel (Zygmuntowicz et al. 2018), aluminum (Xie et al. 2020), iron (Goncharov et al. 2009), titanium (Klimov et al. 2020; Shengfang et al. 2020), and molybdenum (Matysiak et al. 2005).

A review of the literature relating to ceramic–metal composites revealed that special attention had been focused on the alumina-copper system in recent years. Based on the literature review, it was found that many publications point to the very poor wettability of Al$_2$O$_3$ ceramics on copper (Meier et al. 1995; Kuzmin 1974; Marks et al. 2001). For this reason, liquid copper is observed to flow out of the shaped body during the liquid phase, sintering the entire range of their compositions. Based on the phase equilibrium between copper and nickel, it was found that these elements form a solid solution continuously on the whole range of its composition. The melting point of this solution increases with the increase in nickel content, which can lead to the disappearance of the liquid phase during sintering and stopping the flow of copper in composites with the correct ratio of both elements. Therefore, it seems that retaining copper in the material by introducing an additional component can create a phase with a higher melting point.

Consequently, it may be helpful in the technology of these materials. Furthermore, the choice of nickel as the second metal component also allows many of the results described in the literature to be used (Zygmuntowicz et al. 2020a, b). Therefore, the study aimed to determine the influence of the mutual ratio of metallic elements on the structure’s phase structure and the basic properties of composites in the Al$_2$O$_3$–Cu–Ni system. The research subject was the hybrid composites of the ceramic–metal system obtained by the slip casting method. To prepare the samples, five slurries were prepared, which contained 50 vol% of solid phase, including 15 vol% of metallic phase, with a different ratio of metallic components-Cu/Ni. Additionally, for comparison purposes, two reference series of samples were made: 100% Al$_2$O$_3$ and Al$_2$O$_3$ with the addition of 15 vol% of Cu to the total ceramic volume.

**Materials and methods**

**Materials**

The Al$_2$O$_3$ powder (Almatis) with an average particle size of 150 nm, the density of 3.95 g/cm$^3$, and specific surface area of 8.06 ± 0.06 m$^2$/g (measured by BET method on ASAP 2020, Micromeritics, USA) and with 99.99% purity was used as the ceramic powder. One of the metallic powders used in the experiment was nickel (BIMO) with an average particle size of 64.8 μm, a theoretical density of 8.9 g/cm$^3$, a specific surface area of 0.60 ± 0.01 m$^2$/g, and purity of 99.9%. However, the second metallic powder was used as copper (Sigma-Aldrich) with an average particle size between 14 and 25 μm, a theoretical density of 8.94 g/cm$^3$, a specific surface area of 0.29 ± 0.01 m$^2$/g, and purity of 99.9%. The morphology of the starting powders is shown in Fig. 1. While the results of X-ray diffraction

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**Fig. 1** The morphology of the starting powders
of the starting powders are shown in Fig. 2. The SEM images reveal that the Al₂O₃ particles are characterized by an irregular shape with the tendency to agglomeration. On the other hands, copper particles are characterized by spherical morphology. It was found that both metallic powders—copper and nickel, were not agglomerated. Moreover, based on SEM images, it has been observed that the Ni powder is characterized by an irregular, leaf-like shape.

As a dispersing agent for the suspensions, an ammonium salt of polyelectrolyte, commercially known as DURAMAX D3005, was used. The slurries included 1.5 wt% of the dispersant with respect to the total solid content weight. In addition, deionized water was used as a solvent.

**Manufacturing method**

In the presented research, composite samples from the ternary Al₂O₃–Cu–Ni system were fabricated using the slip casting technique in the multistage manufacturing process. The process begins with weighing the initial powders in specified proportions and mixing them with the proper amount of deflocculant and solvent to produce a slurry. Next, the fabricated slurry was mixed and degassed in the

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**Fig. 2** Diffractograms of powders used in the experiment

![Diffractograms of powders used in the experiment](image-url)
centrifugal mixer THINKY ARE-250. The mixing was carried out in a time of 8 min at the rate of 1000 rpm, while degassing was carried out at the speed of 2000 rpm for 2 min. The manufactured slurry in the next stage was cast into a gypsum mold. The application of the porous mold in the casting process enables the effective removal of the solvent from the casting slurry due to the capillary action forces. Afterward, the green bodies, removed from the gypsum mold, were dried for 24 h at 40 °C and then sintered in the reducing atmosphere consisting of 20% H₂ and 80% N₂. Finally, the sintering process was carried out at the temperature of 1400 °C with 2 h of dwell time.

In the investigation part, water-based ceramic slurries with 50 vol% solid content and 15 vol% metal powder with respect to the total solid volume were made. Five series of samples differing in the share of copper and nickel were prepared: Series I-contained 12 vol% Cu-3 vol% Ni, Series II-contained 10 vol% Cu-5 vol% Ni, Series III-contained 7.5 vol% Cu-7.5 vol% Ni, Series IV-contained 5 vol% Cu-10 vol% Ni, and Series V-contained 3 vol% Cu-12 vol% Ni. The volume of metal was calculated with respect to the total metal volume content in the slurries.

Research methods

In the investigation, several procedures were used to establish the properties of the slurries and microstructure and selected properties of the Al₂O₃–Cu–Ni composites.

The rheological properties of the suspensions were measured by using a Rheometer Anton Paar. The viscosity was measured as a function of shear rate when increased from 1.3 s⁻¹ to 260 s⁻¹ and back to 1.3 s⁻¹.

The scanning electron microscope (SEM) model JEOL JSM-6610 was used to investigate the morphology of the base powders and the microstructure of the obtained ceramic–metal composites. The SEM observations were carried out using 15 kV acceleration voltage. To analyze the microstructure of the obtained composites, observations were made using a backscattered electron detector (BSE) available in the SEM. BSE mode enabled the separation of the distribution of the metallic phases and matrix on the cross section of the composite. As part of the metallographic sample preparation, all samples used for the experiments were mounted in resin, ground with abrasive paper of 80–4000 gradations, and polished using 3 μm and 1 μm diamond suspensions. Additionally, a microanalysis using energy dispersion spectroscopy (EDS) was carried out to identify the chemical composition of the investigated series of composites. The EDS maps the collected distributions of the elements on the surface of the samples.

The X-ray diffraction examination was performed using a Rigaku MiniFlex II diffractometer with a Cu-Kα radiation source (wavelength λ = 0.154178 nm) with current 15 mA, voltage 30 kV, angular range 20°: 20°–100°, counting time 1 s and step 0.02°.

The hardness of the samples was measured by the Vickers method on a polished sample surface of the cross section at a load of 196 N with 15 s holding time. For each series of sintered composites, a minimum of 17 indentations was made. The fracture toughness was calculated based on the Niihara equation for 0.25 < l/a < 2.5 (Niihara et al. 1982; Niihara 1983).

Results and discussion

In the investigation, several procedures were used to establish the properties of the slurries and microstructure and selected properties obtained from the Al₂O₃–Cu–Ni composites.

The scores of rheological experiments are collected in Fig. 3. Based on the curve of viscosity as a function of shear rate (Fig. 3a), it can be established that the viscosity decreases with increasing shear rate for all tested suspensions. These results prove that the produced suspensions are non-Newtonian shear thinned fluids. The obtained slurries
do not show constant viscosity as a function of shear rate. This is also related to the phenomenon of thixotropy, as evidenced by the hysteresis loops produced by the viscosity curves made first to increase and decrease the shear rate (Fig. 3b). The phenomenon of thixotropy is the result of the aggregation of slurry particles. Particle aggregation is related to the formation of the internal structure of the suspension, and the reduction in viscosity results from its partial or complete disintegration. Therefore, reducing the shear rate can lead to reconstructing the damaged structure of particles in the suspension. Analyzing the results, it can be seen that the lowest viscosity was characteristic for the slurry with the same content of both metallic components (Cu: Ni = 7.5 vol%: 7.5 vol%), which allows claiming that it will be the most advantageous in terms of casting. The values of viscosity of the prepared suspensions as a function of shear rate and shear stress of prepared suspension as a function of the shear rate were had shown in Table 1. Moreover, based on the flow curves, it was found that the slip masses produced were characterized by the presence of the suspension flow limit (Fig. 3b). The flow limit is defined as the value of the minimum tangential stress needed to cause the fluid to flow.

Figure 4 presents the SEM pictures of all obtained series of the investigated composites. In BSE mode, the metal phases were light gray, while the matrix was dark gray areas. The examination of the microstructure confirmed that the composites are well densified. It was found that the general distribution of metal phases in the ceramic matrix for all samples is relatively homogeneous. Still, the observation revealed a difference in the concentration of metal phases. The difference was not so high but visible in the SEM images. The lowest metal phase concentration was observed in Series I, which contained 12 vol% Cu-3 vol% Ni, while the highest concentration was revealed in Series III (7.5 vol% Cu-7.5 vol% Ni).

Table 1  The values of the viscosity of the prepared suspensions as a function of shear rate and the shear stress of the prepared suspensions as a function of the shear rate

| Series | Viscosity [Pa·s] | Shear stress [Pa] |
|--------|----------------|------------------|
|        | Shear rate = 1.3 [1/s] | Shear rate = 260 [1/s] | Shear rate = 1.3 [1/s] | Shear rate = 260 [1/s] |
| Series I-12 vol% Cu-3 vol% Ni | 0.614 | 0.0231 | 0.798 | 6 |
| Series II-10 vol% Cu-5 vol% Ni | 0.0973 | 0.00393 | 0.126 | 1.02 |
| Series III-7.5 vol% Cu-7.5 vol% Ni | 0.0964 | 0.00358 | 0.125 | 0.93 |
| Series IV-5 vol% Cu-10 vol% Ni | 0.255 | 0.00622 | 0.331 | 1.62 |
| Series V-3 vol% Cu-12 vol% Ni | 0.247 | 0.00595 | 0.321 | 1.54 |

Fig. 4  The microstructure of composites
Figure 5 shows the microstructures of sintered composites from all five series. The darker area corresponds to the Al₂O₃ phase, while the bright areas are the metal phase. It was found that metallic particles tend to form clusters. However, depending on the volume of metal used to obtain the composite, some differences are visible. In Series I, the revealed metallic phases are more porous than in the other series. This effect can be related to the effect of the outflow of the liquid copper during the sintering process. Series I samples contain the most copper in comparison to other series. Such copper content of the composite may result in an insignificant porosity due to the leakage of the liquid phase during the sintering process.

Figure 6 presents the results of the chemical composition microanalysis made by an energy dispersive spectroscopy (EDS) technique of samples sintered with different metal volumes. In all series, the presence of aluminum, oxygen, nickel, and copper was detected. The darker area in the SEM images (on the left) corresponds to the Al₂O₃ phase, while the bright regions correspond to the CuNi phase. A characteristic is that the copper atoms and nickel atoms (blue and yellow color, respectively) occupy the same area. It must be remembered that they were added and mixed with alumina as a separate phase, so the distribution of phases should be homogenous, so it was expected to be observed as two separate metallic phases in the final material. This result suggested that the nickel and copper particles combined and probably reacted with each other during consolidation. To examine the phase composition and XRD, an analysis was conducted.

Phase composition results are shown in Fig. 7. revealed the presence of three phases in all examined composite series: Al₂O₃, Ni, and CuNi solid solution. Direct measurements allowed us to observe characteristic patterns for composites with differential metal phase composition. In the case of composite samples obtained with 12 vol% of copper and 3 vol% of nickel in metal phase (Series I), the reflexes at 2θ value equal 43.26°, 50.28°, 74.54°, 91.06°, 95.42° correspond to the (111), (200), (220), (311), (222) Ni atomic planes, respectively (#04-006-6387). For the samples with 10 vol% of copper and 5 vol% of nickel in metal phase (Series II), reflexes with identical Ni plane indices were observed with 2θ values and equal to 44.12°, 51.30°, 75.34°, 91.60°, and 95.76° in sequence (#04-006-6387), while for the samples with an equal amount of both metallic components in metal phase (Series III), XRD analysis indicates that picks at 2θ value equal 44.22°, 51.44°, 75.67°, 91.88°, and 95.74° also corresponds to (111), (200), (220), (311), and (222) of the Ni atomic planes, respectively (#04-006-6387). In samples with the predominance of nickel in the metal phase, reflexes from (111), (200), (220), (311), and (222) Ni plane indices were observed at 2θ values equal to 44.18°, 51.44°, 75.90°, 92.18°, and 95.52°, respectively, for samples with 5 vol% of copper and 10 vol% of nickel in metal phase and 44.42°, 51.72°, 76.31°, 92.56°, and 95.68°, respectively, for samples with metallic phase composed in 3 vol% of copper and 12 vol% of nickel. During the analysis, it was found that in all examined samples, the reflexes of the CuNi phase corresponded to (111), (200), (220), (311), and (222) in a sequence of CuNi atomic planes. For samples
fabricated with 12 vol% of copper and 3 vol% of nickel in metal phase (Series I), the reflexes at 2θ value equal 43.26°, 50.56°, 74.44°, 90.46°, and 95.14°, corresponding to (111), (200), (220), (311), and (222) CuNi atomic planes, respectively (#04-003-7260). In comparison, reflexes from the same sequence of atomic planes for the sample with metallic phase composed with 10 vol% of copper and 5 vol% of nickel (Series II) were observed at 2θ values equal to 44.12°, 51.30°, 75.28°, 91.40°, and 95.44° in sequence. For the samples obtained with 7.5 vol% of both copper and nickel...
in metallic phase (Series III), XRD pattern analysis indicates that the peaks observed at 2θ = 44.22°, 51.44°, 75.56°, 91.74°, and 95.44° correspond to the mentioned above (111), (200), (220), (311) and (222) CuNi atomic plane, respectively (#04-003-7260). In the case of samples obtained with a predominance of nickel in the metallic phase, the reflexes at 2θ values equal to 44.18°, 51.44°, 75.70°, 92.00°, and 95.32° for samples with 5 vol% of copper and 10 vol% of nickel in the metallic phase and 44.42°, 51.72°, 76.10°, 92.46°, and 95.38° for samples with 3 vol% of copper and 12 vol% of nickel both correspond to the (111), (200), (220), (311), and (222) in sequence CuNi atomic planes, respectively. XRD analysis revealed that Ni and CuNi solid solutions in all analyzed composite series could be characterized by cubic structure (Fm-3m).

Vickers’s hardness analysis revealed the dependence between the analyzed results and the metal phase composition in the fabricated specimens. The obtained hardness results are shown in Fig. 8. It was observed that a reduction of the disparities between the proportion of particular metal phase components — Cu and Ni — results in a deterioration of the composite hardness. Following the observed correlation, the lowest Vickers hardness value was measured in Series III, which contained an equal percentage of both metal components in the metal phase. Hardness for Series III was equal to 224.8 ± 36.9 HV. The highest hardness values characterized specimens with the predominance of one of the components in the metal phase. The highest hardness composites from the ternary Al₂O₃–Cu–Ni system indicated samples from Series I consisting of 12 vol% of copper and 3 vol% of nickel in the metal phase. In Series V, composed of 12 vol% of Ni and 3 vol% of Cu in the metal phase, the measured hardness was slightly reduced and equal to 450.2 ± 22.4 HV. A comparison of the obtained results with the reference Al₂O₃ and Al₂O₃–Cu samples revealed that the introduction of the second metallic component to the metal phase of the composite resulted in hardness deterioration. The hardness of the ternary Al₂O₃–Cu–Ni composites was lower than the values measured for the samples of pure alumina and composites consisting of only copper in the metal phase. Hardness values measured for reference specimens of Al₂O₃ and Al₂O₃–Cu were equal to 869.4 ± 30.3 HV and 619.2 ± 6.5 HV, respectively. However, it should be noted that the hardness values were obtained for reference. Al₂O₃ samples were lower than the available literature data. In the work of Broniszewski et al. (2015) hardness of the samples from pure alumina obtained with the SPS, the technique was equal to 1664 HV [36]. Many factors influence the hardness values of the composite samples including both the manufacturing methodology and the initial materials used in the process.

In this study, manufactured the Al₂O₃–Cu–Ni composites obtained by the slip casting method, and the characterized microstructure and selected properties were investigated. Based on the conducted research, the following conclusions were obtained:

**Conclusion**

In this study, manufactured the Al₂O₃–Cu–Ni composites obtained by the slip casting method, and the characterized microstructure and selected properties were investigated. Based on the conducted research, the following conclusions were obtained:
• It was found that the addition of the nickel phenomenon limits the outflow of the liquid phase (Cu) during sintering but does not eliminate it.
• It has been observed an even distribution of the metallic phase in the ceramic matrix.
• Phase composition results revealed three phases in all obtained composites: Al₂O₃, Ni, and CuNi solid solution.

The trials presented in the manuscript are preliminary studies. The investigations on this problem are in progress, and the results will be published in the succeeding papers.

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Declarations

Conflict of interest The authors declare that has no any conflict of interest.

Ethical approval This article does not contain any studies with human participants or animals performed by any of the authors.

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