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Al₂O₃-Ni COMPOSITES: IMPACT OF COOLING ON COMPOSITES IN CENTRIFUGAL GEL CASTING

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This study investigates the influence of polymerization kinetics and the processing sequence on the microstructure and mechanical performance of Al₂O₃-Ni composites fabricated via centrifugal gel casting. Two fabrication approaches were compared, differing in the sequence of component addition and the temperature of the casting mass. The optimized method, involving cooling the suspension to below 5°C prior to the addition of nickel powder and the polymerization initiator, significantly extended the gelation idle time from 225 s to 475 s, allowing improved control over polymerization and particle dispersion. As a result, the microstructure exhibited enhanced phase homogeneity and reduced porosity. Compression tests demonstrated substantial enhancement in mechanical performance: the compressive strength increased from 22 MPa in the non-optimized series to 185 MPa in the optimized series, representing more than an eightfold improvement. These findings highlight a practical strategy for tuning the polymerization behavior to engineer high-performance ceramic-metal composites with potential applications in structural and functional components.

Keywords: centrifugal gel casting, Al₂O₃-Ni, composites, polymerization, 2-2-carboxyethyl acrylate, CEA

INTRODUCTION

The development of ceramic-metal composites has gained significant interest due to their unique combination of mechanical strength [1–2], wear resistance [1–2], and thermal stability [3], making them highly suitable for applications in aerospace, biomedical, and structural materials [4–5]. Among the various processing techniques available for fabricating such composites, centrifugal slip casting has emerged as an effective method for producing gradient structures with a controlled microstructure [6]. This technique

takes advantage of centrifugal forces to influence the distribution of phases, enabling precise tailoring of material properties across the composite structure [6].

Further enhancement of this approach is achieved by employing gel casting, a shaping technique that utilizes in-situ polymerization to solidify the suspension into a stable green body [7–9]. Gel casting provides several advantages, including high green body strength, near-net shaping capability, and improved homogeneity of the

microstructure [9]. As highlighted in prior research [10], the polymerization process plays a critical role in determining the mechanical and structural parameters of the final sintered material. The structure and length of the polymer chains affect both the strength and flexibility of the green body, influencing its subsequent processing and operational performance [11].

Despite its advantages, one of the primary challenges in the gel casting process is ensuring homogeneous polymerization throughout the entire volume of the sample. Inhomogeneous polymerization can lead to internal stresses, which in turn reduce the overall mechanical integrity of the material [11]. This issue becomes particularly critical when working with metal-ceramic systems, where differences in material properties can further exacerbate stress concentrations. The chemical composition of the casting suspension, particularly the presence of metal ions such as Fe^{2+} , Al^{3+} , and Cu^{2+} , has been shown to significantly influence the polymerization reaction by altering the gelation kinetics and dead time [12].

When centrifugal casting is combined with gel casting, the presence of metal powders can act as catalysts, accelerating the polymerization reaction and thereby influencing the formation of the composite's gradient structure. However, an excessively short gelation time can introduce defects such as phase segregation, particle agglomeration, and increased porosity, which negatively impact the structural integrity and mechanical properties of the composite [13]. Consequently, optimizing both the composition of the casting suspension and the processing parameters is essential to achieve a defect-free and mechanically robust gradient composite structure.

The aim of this study is to investigate the effect of the polymerization parameters on the quality of the gradient structure in Al_2O_3 -Ni composites fabricated using the centrifugal gel casting method. The research focuses on controlling the gelation kinetics by modifying the chemical composition of the casting suspension and evaluating the impact of these modifications on the microstructure and mechanical properties of the final

material. By optimizing the interaction between the polymerization dynamics and centrifugal casting parameters, this study seeks to advance the fabrication of high-performance ceramic-metal composites with tailored properties for industrial applications. Therefore, the work focuses on developing a method for manufacturing composite elements with a reduced rate of the casting mass gelation process, namely extending the sterile gelation time and, as a result, facilitating the achievement of a homogeneous gradient structure of the composite, free from agglomerates of the employed components, and obtaining a homogeneous as well as non-porous composite material. For this purpose, an experiment was conducted in which the effect of lowering the temperature of the prepared mass on the obtained product was examined.

MATERIALS AND METHODS

Commercially available nanometric aluminum oxide (Al_2O_3 , TM-DAR, Taimei Chemicals) and micrometric nickel (Ni, Sigma Aldrich) powders were used as the starting materials to prepare the composite system. Demineralized water served as the solvent for suspension preparation. To facilitate dispersion and stability of the suspension, diammonium hydrogen citrate (DAC) was employed as a liquefier in an amount of 0.3 wt% relative to the total solid content. The polymerization process was initiated using a free-radical polymerization mechanism. Specifically, 2-2-carboxyethyl acrylate (CEA) was introduced as the monomer, while N,N,N',N'-tetramethylethylenediamine (TEMED) was utilized as the activator at a concentration of 1 wt% relative to the monomer mass. The polymerization reaction was initiated by ammonium persulfate (APS), added in a proportion of 2 wt% relative to the monomer mass. These components were systematically selected to ensure the stability and homogeneity of the suspension, facilitating the subsequent processing steps in the formation of the Al_2O_3 -Ni composite.

Two series of composites - A and B - were produced, differing in the method of preparing the slip used for further forming of the composites. Slip A

was prepared by the preliminary mixing of: water in the amount of 13.59 g, diammonium hydrocitrate (DAC) liquefier (0.3 wt% in relation to the sum of the powder masses, i.e. 0.1336 g), N,N,N',N'-tetramethylethylenediamine (TEMED) polymerization activator in the amount of 0.5 wt% in relation to the monomer, in the form of a 10% solution, i.e. 134 μ l, Al₂O₃ powder in the amount of 35.64 g, Ni powder in the amount of 10 vol.% in relation to Al₂O₃ powder, i.e. 8.9 g, and an organic monomer in the form of 2-carboxyethyl acrylate (CEA) in the amount of 6 wt% in relation to the total mass of powders, i.e. 2.67 g. Mixing was carried out in a Thinky ARE-250 device using the following parameters: mixing: 2 min, 500 rpm; deaeration: 2 min, 1800 rpm; mixing 2 min, 800 rpm; deaeration: 2 min, 1800 rpm. After homogenization, an initiator (ammonium persulfate) was added to the casting mass in the form of a 5% aqueous solution in the amount of 2% by weight in relation to the monomer mass, i.e. 0.05 g. Then, Slurry A with the initiator was subjected to mixing and deaeration under the following conditions: mixing: 30 s, 1200 rpm and deaeration: 15 s, 1800 rpm. Slurry B was prepared by the preliminary mixing of water in the amount of 10.0 g, diammonium hydrocitrate (DAC) liquefier (in the

amount of 0.3 wt% relative to the sum of the powder masses, i.e. 0.1336 g), N,N,N',N'-tetramethylethylenediamine (TEMED) polymerization activator in the amount of 0.5 wt% relative to the monomer, in the form of a 10% solution, i.e. 134 μ l, Al₂O₃ powder in the amount of 35.64 g, and an organic monomer in the form of 2-carboxyethyl acrylate (CEA) in the amount of 6 wt% relative to the sum of the powder masses, i.e. 2.67 g. Mixing was carried out in a Thinky ARE-250 device using the following parameters: mixing: 2 min, 500 rpm; deaeration: 2 min, 1800 rpm; mixing 2 min, 800 rpm. After homogenization, Slip B was cooled to a temperature not exceeding 5°C, then Ni powder was added to the slip in an amount of 10% by volume in relation to the Al₂O₃ powder, i.e. 8.9 g, and an initiator (ammonium persulfate) was added in the form of a 5% aqueous solution in an amount of 2% by weight, i.e. 0.05 g. In the next step, Slip B with the initiator was subjected to mixing and deaeration under the following conditions: mixing: 30 s, 1200 rpm and deaeration: 15 s, 1800 rpm. The above description of the preparation of mixtures for the production of composites is presented in Figure 1.

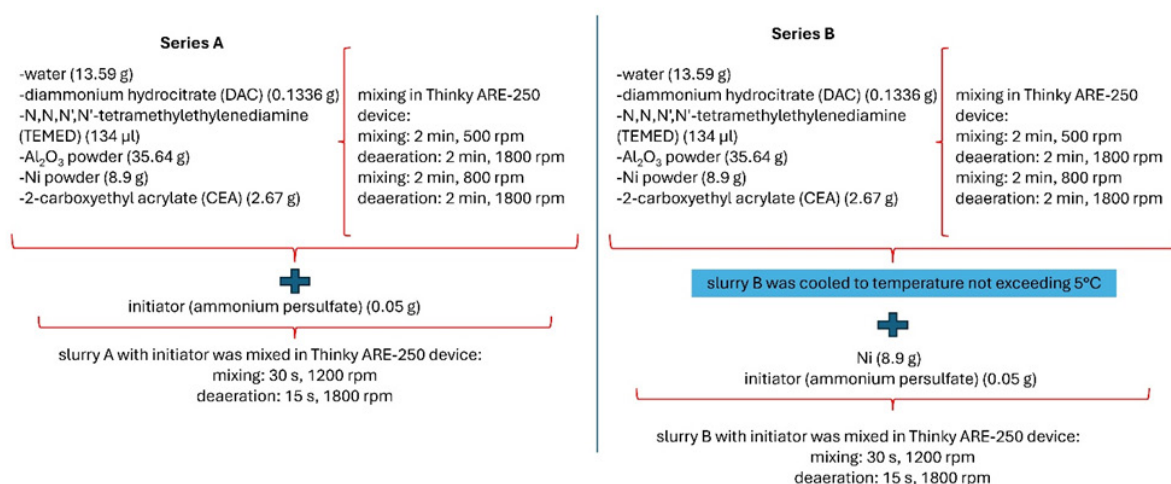


Fig. 1.

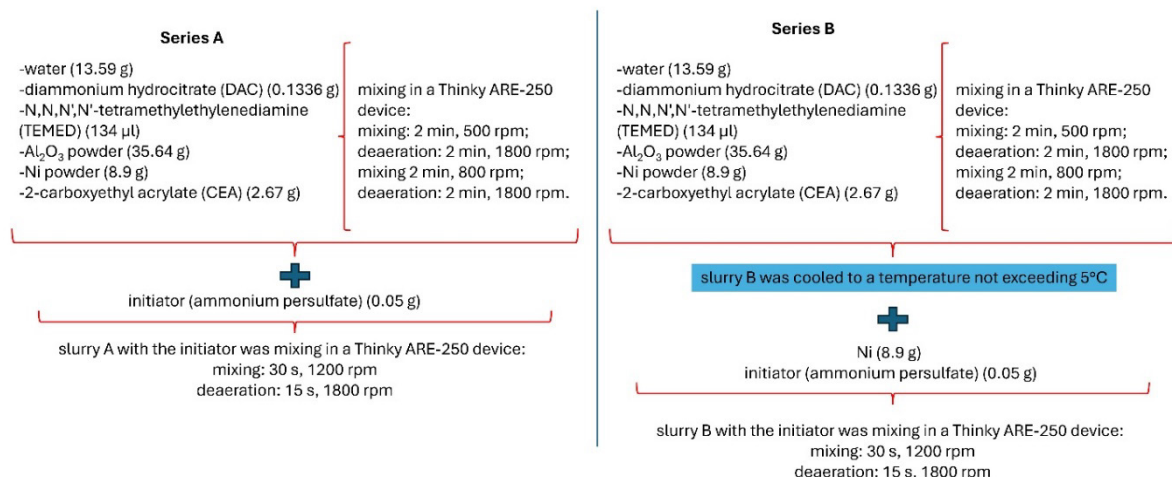


Fig. 1. Scheme for preparing mixtures for production of composites

Then the homogeneous masses were poured into the mold and subjected to centrifugal casting at 1800 rpm for 180 minutes, after which the obtained shapes were removed from the mold and subjected to the process of burning out the organic additives and sintering the produced shape at the temperature of 1400°C in a reducing atmosphere. Previous studies on the forming of composites by centrifugal slip casting or centrifugal gel casting were characterized by a stage of a simultaneous addition of metal and ceramic powder [6, 14–16]. In this work, a change was introduced to this stage consisting in dividing it into two, namely, the ceramic powder was added before homogenization of the mixture, and the metal powder was added after homogenization of the mixture and after cooling it to a temperature not exceeding 5°C. Literature data and the authors' own works [14–16] indicate that at present there are no available studies on this subject.

In order to determine the idle time, both slips (Series A and B) were tested by means of a Brookfield RVDV-II+PRO viscometer, using a 34 spindle, at the rotation speed of 3.5 rpm. The measurements were carried out until the moment of an intensive increase in viscosity, which indicates the actual initiation of the polymerization reaction in the slip and the formation of polymer chains. The time that elapses until the intensive increase in viscosity is called the idle time of polymerization. The test was performed at room temperature (23°C).

Volumetric and linear shrinkage were determined by measuring the dimensions of the green bodies and the sintered samples. Linear shrinkage was calculated along both the axial (length) and radial (outer diameter) directions using a digital caliper with ± 0.01 mm precision. Volumetric shrinkage was estimated based on the dimensional measurements, assuming cylindrical geometry. Six samples per series were analyzed to ensure statistical reliability, and the results are presented as mean values with standard deviations.

The Al₂O₃-Ni composite samples were analyzed utilizing a JEOL JSM-6610 SEM to examine their microstructure and nickel particle distribution. The cross-sections of the sintered composites were polished to obtain a smooth surface for imaging. A thin conductive layer (carbon) was applied to prevent charging effects during SEM analysis. Imaging was performed in high-vacuum mode to ensure high-resolution observations. Secondary electron (SE) and backscattered electron (BSE) modes were used to distinguish the ceramic (Al₂O₃) and metallic (Ni) phases based on contrast differences. A range of magnifications was used to analyze both the overall structure and detailed features of Ni particle distribution. Moreover, energy-dispersive X-ray spectroscopy (EDX) was employed to confirm the elemental composition of the phases.

The mechanical behavior of the Al₂O₃-Ni composites was evaluated by means of a monotonic compression test to determine their compressive strength and deformation characteristics. The tests were conducted in accordance with ISO 2739:2010 (EN). The test was performed on an Instron 8802 MT hydraulic pulsator equipped with specialized software enabling continuous recording of the load as a function of the displacement of the plate compressing the specimen.

The average grain size of the alumina (Al₂O₃) phase in the sintered composites was ascertained by stereological analysis based on the SEM micrographs. The analysis was conducted utilizing MicroMeter software, which enables precise grain size measurements using image processing techniques [17–20]. At least 850 grains per sample were analyzed to obtain a representative grain size distribution. The results were expressed as the mean grain size \pm standard deviation, and a histogram of the grain size distribution was generated. The obtained grain size data were further correlated with the processing parameters to assess their influence on the microstructure evolution of the Al₂O₃-Ni composites.

RESULTS AND DISCUSSION

The idle time graphs for the investigated masses are shown in Figure 2. The measurements show that the idle time for Slurry A was about 225 s, and the idle time for Slurry B was about 475 s. The idle time obtained for Slurry B is long enough to allow effective performance of further operations in the centrifugal gel casting process. The experimental studies confirmed that the key stage of the process is adding Ni powder after homogenization of the ceramic mass, i.e. as the last component, and before adding the Ni powder the casting mass should be cooled to a temperature not exceeding 5°C. The proposed method of preparing casting masses for forming composites using the centrifugal gel casting method allows the idle gelation time to be extended by lowering the casting mass temperature in a critical phase of the manu-

facturing process, namely immediately before introducing the metallic phase and initiator into the casting mass. The extension of the sterile gelation time is also achieved by introducing metal powder (before homogenization) and ceramic powder (after homogenization), which leads to a decrease in the rate of monomer polymerization as well as stabilizes the viscosity of the casting mass over a longer period of time.

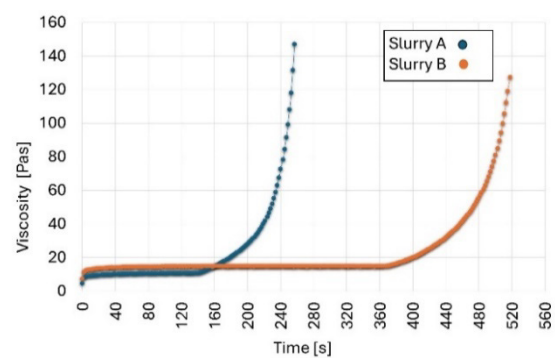


Fig. 2. Idle time measurements

Figure 3 presents a comparative analysis of the composite samples from Series A and Series B, before and after the sintering process. In Figure 3 a) Series A: the image on the left shows the composite tube before sintering, with a visible inner core. After sintering (image on the right), the sample exhibits a more compact structure, with reduced porosity and a darker shade, indicating successful densification. In Figure 3 b) Series B: similar to Series A, the image on the left displays the composite sample before sintering. Post-sintering (image on the right – Figure 3 b), the sample shows structural compaction and a darker appearance, highlighting the impact of high-temperature treatment in a reducing atmosphere.

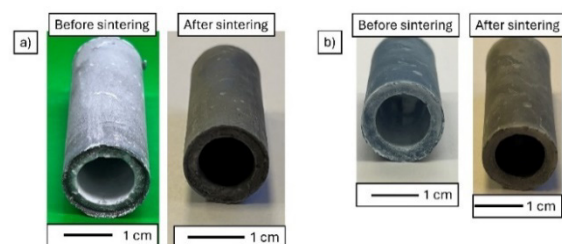


Fig. 3. Comparison of exemplary composite samples from Series A – a) and Series B – b) before and after sintering

The volumetric shrinkage for Series A was $36.60 \pm 0.36\%$, while for Series B it was equal to $21.61 \pm 0.58\%$. Linear shrinkage measured along the length of the sample was determined at the level of $14.40 \pm 0.75\%$ for Series A, whereas for Series B it was a value of $13.36 \pm 0.46\%$. Linear shrinkage measured along the outer diameter of the samples for Series A was equal to $13.56 \pm 0.68\%$, and for Series B $13.96 \pm 0.63\%$.

The micrographs in Figure 4 present the microstructural differences between the two series of Al_2O_3 -Ni composites fabricated using centrifugal slip casting. The key distinction between Series A and Series B lies in the preparation method of the casting slip, specifically the sequence of nickel (Ni) addition. This procedural variation influences the distribution of Ni particles and the width of distinct microstructural zones observed after casting and sintering.

Based on the observations, it can be assumed that the micrograph of samples from Series A (simultaneous addition of Ni and Al_2O_3) reveals a more gradual transition between the zones, resulting in broader intermediate regions (Zone II) where Ni is dispersed within the alumina matrix. The centrifugal force during casting led to a relatively uniform gradient in particle distribution, causing the Ni-enriched zone to exhibit a moderate and continuous thickness (Zone III). The ceramic-dominant region (Zone I) appears to be well-integrated with the metallic phase, with fewer sharp boundaries between the Ni-rich and Al_2O_3 -rich areas. For series I (Figure 4 a), the following zones can be conventionally distinguished on the cross-section. Zone I is an Al_2O_3 -rich zone, well-distributed, with Ni particles still present but more dispersed. Zone II is the transition zone, which is wide and continuous, indicating effective mixing. Zone III is an Ni-rich zone, moderately broad, exhibiting a smooth gradient transition.

In contrast, it can be seen that the Series B samples (sequential addition of Ni after cooling) have sharper boundaries between the Ni-rich and Al_2O_3 -rich zones (Figure 4 b). The delayed introduction of Ni after cooling appears to have influ-

enced its sedimentation behavior during centrifugal casting, leading to more distinct and concentrated Ni layers. This results in narrower transition zones and more pronounced segregation of the Ni particles within specific regions. Zone I in Series B (Figure 4 b) is the Al_2O_3 -rich zone, broader, with fewer Ni particles compared to Series A, while Zone II, the transition zone, is noticeably thinner, indicating a sharper phase separation. Zone III is the Ni-rich zone, more concentrated and slightly narrower than in Series A, with a higher Ni content.

The analysis of the zone widths suggests that Series A exhibits a more gradual Ni distribution, resulting in a wider transition zone and more uniform material integration. In contrast, Series B demonstrates a more defined layering effect, where the Ni-rich and Al_2O_3 -rich zones are more distinct and separate. These structural differences may influence the mechanical performance, thermal stability, and fracture behavior of the composites, necessitating further investigation.

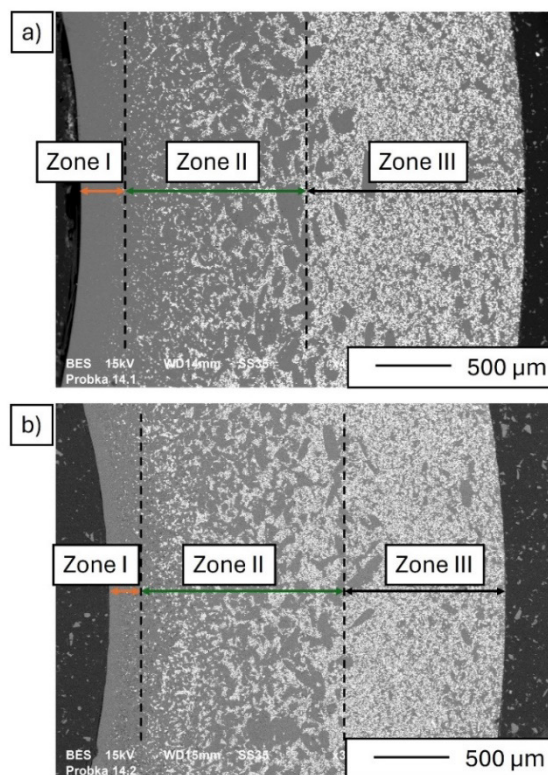


Figure 4. Microstructural comparison of Al_2O_3 -Ni composites from Series A and Series B, highlighting differences in width of individual zones

Figure 5 shows the microstructural and elemental distribution analysis of the Al₂O₃-Ni composites. The first column in Figure 5 presents the backscattered electron (BSE) micrographs, highlighting the contrast between alumina (gray matrix) and nickel (bright phase). The second column displays the energy-dispersive X-ray spectroscopy (EDS) elemental mapping, where nickel is shown in cyan, aluminum in green, and oxygen in red. The third to fifth columns provide individual element maps for aluminum, oxygen, and nickel, respectively, confirming the distribution of these phases within the composite structure. The micrographs reveal the clustering behavior of the nickel particles within the alumina matrix and the potential differences in phase distribution between the two series.

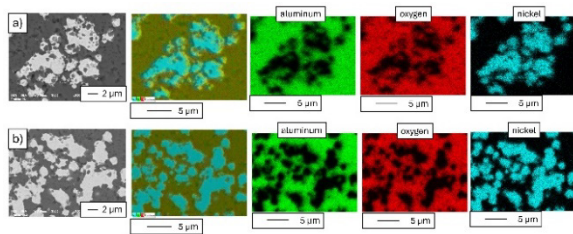


Fig. 5. Microstructural and elemental distribution analysis of Al₂O₃-Ni composites: a) Series A, b) Series B

In the next stage of the study, the produced series were subjected to monotonic compression and digital image correlation analysis. Figure 6 (a, b) presents the strain distribution obtained using the digital image correlation method for a representative sample from the investigated series. The compression strength-displacement curve for the composites is shown in Figure 7. The presented graph is characterized by a linear trend, which exhibits typical brittle fracture behaviour. The observed linear increment in load for the studied material indicates the absence of artifacts in the composite structure. The monotonic test results revealed that the Series A samples transferred a maximum load of 22 MPa at a deformation of 0.9%. The cracking process shows a chaotic course. The example crack shown in Figure 6 a) was dynamic and occurred in several places. These places are random and do not coincide with the classic cracking course for samples of this type. Classically, cracks should occur in places where the greatest bending moments occur. The pressure level of 20 MPa may

result from material defects in the sample as well as the small width of the wall. The Series B samples transferred a maximum load of 185 MPa at a strain of 0.14%. The sample presented in Figure 6 b) did not show any changes in the strain fields during loading until the maximum pressure was achieved. The crack occurred dynamically along the vertical axis of the sample. The nature of the crack indicates that the load was transferred by the vertical core of the sample, i.e. there was no bending of the sample. The results indicate high cohesion and hardness of the material, free from defects, since several parallel cracks occurred. It was found that the obtained compressive strength values are higher than those reported in previous studies by the authors [21] concerning Al₂O₃/Ni composites. In the case of the previous study, it was shown that the compressive strength of Al₂O₃/Ni composites containing 50 vol.% of the solid phase, including 10 vol.% of the metallic phase, fabricated using the centrifugal slip casting method, was determined to be 42.45 MPa. It was also found that the centrifugal gel casting method enables the fabrication of composites with higher compressive strength than the centrifugal slip casting method in the case of ceramic-metal composites.

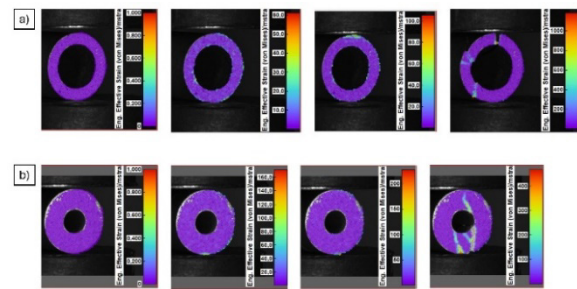


Fig. 6. Strain distribution for composites using digital image correlation (DIC) method: a) Series A, b) Series B

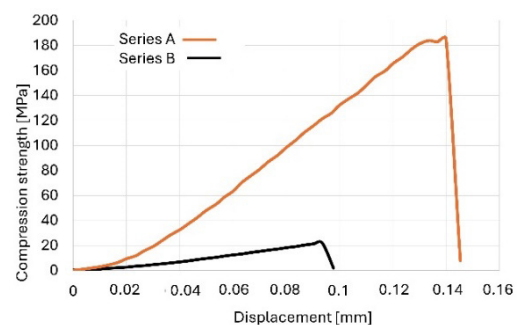


Fig. 7. Deformation as a result of monotonic compression for samples fabricated via centrifugal gel casting

The SEM micrographs (Figure 8 a, b) present the fracture surface for the Series A and B composites. The micrographs reveal that individual grains of Al_2O_3 are clearly discernible as roughly equiaxed polyhedral in shape. The boundaries are well-defined, outlining the original grain shapes. This suggests that the crack propagated along the grain boundaries, i.e. an intergranular fracture mode. The grains appear largely intact (not split apart), indicating that the fracture path followed the interfaces between the grains rather than cutting through them. Evidence of this intergranular mode includes contiguous grain-boundary traces of the fracture surface. In such intergranular fractures, the weakest links are the grain boundaries, hence the crack traverses along those interfaces. Any ductile Ni phase located at the grain boundaries may have been pulled out during fracture, potentially leaving behind small voids or impressions on some grains. The exposed grains in Figure 8 a – Series A – are fairly uniform in size, in the order of 0.4–1 μm across. Most of the grains fall in a similar size range, indicating a narrow distribution (fine, equiaxed microstructure). A few larger grains are visible, but they are rare. The effective grain size for the Series B composites (Figure 8 b) appears to be similar to the grain sizes in the Series A composites.

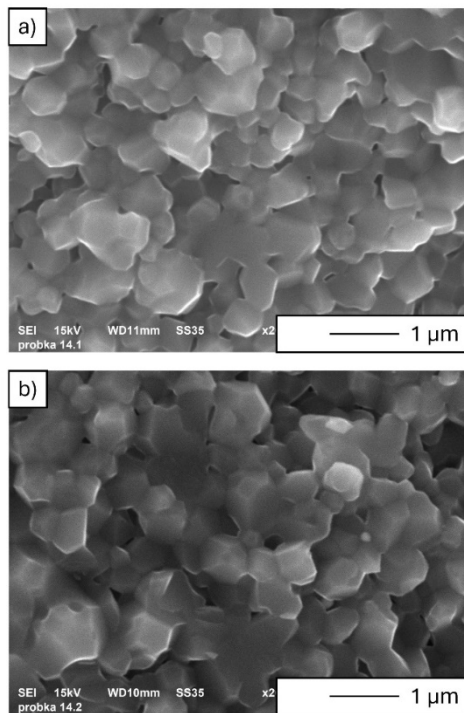


Fig. 8. SEM micrographs of fracture surfaces of Al_2O_3 -Ni composites: (a) Series A and (b) Series B

The histograms in Figure 9 display the alumina (Al_2O_3) grain size distribution in the Al_2O_3 -Ni composites for (a) Series A and (b) Series B. The x-axis represents the alumina grain size in micrometers (μm), while the y-axis shows the percentage of grains in each size range. The results indicate that the grain size distribution is centered around 0.4–0.6 μm , with the majority of grains in both series falling within this range. In Series A, 55.96% and 30.46% of the grains are in this dominant range, whereas in Series B, the respective fractions are 55.18% and 31.77%. The average grain size was measured as $0.42 \pm 0.18 \mu\text{m}$ for Series A and $0.41 \pm 0.16 \mu\text{m}$ for Series B, demonstrating that increasing the solid phase content from 45% to 50% does not significantly impact grain growth. The fraction of larger grains ($>1.0 \mu\text{m}$) remains minimal in both series, indicating a uniform microstructure. These results align with the microstructural observations discussed earlier, confirming that the cooling of the casting slip before adding the Ni powder influences the phase distribution without significantly altering grain growth. The relatively uniform grain size distribution in both series suggests that the casting process and polymerization kinetics were well-controlled, minimizing undesirable grain coarsening effects.

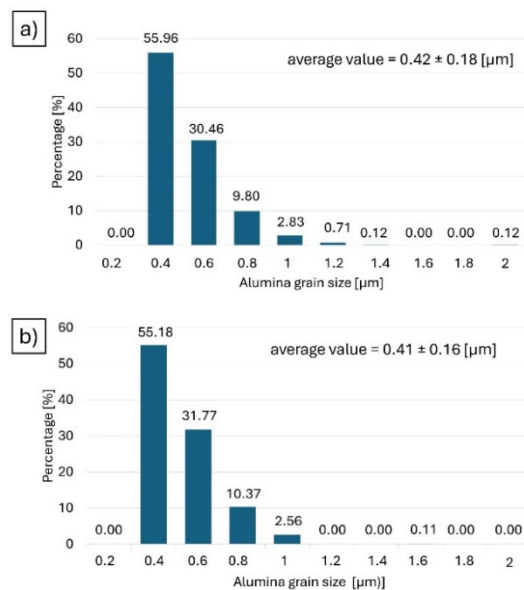


Fig. 9. Alumina grain size distribution in Al_2O_3 -Ni composites for (a) Series A and (b) Series B

CONCLUSIONS

This study investigated the effect of polymerization kinetics on the microstructural and mechanical properties of Al₂O₃-Ni composites fabricated using centrifugal gel casting. A novel approach is introduced by modifying the casting slip preparation process, specifically by lowering the temperature of the suspension before adding the metallic phase and polymerization initiator. This method significantly extends the gelation idle time, allowing improved process control and preventing premature polymerization.

The key findings include:

- **Extended idle time for polymerization:** Cooling the slip before introducing the Ni powder slows the polymerization reaction, enabling better phase distribution and reducing agglomeration.
- **Improved microstructural homogeneity:** Sequential addition of the materials enhances the gradient structure, minimizing porosity and ensuring uniform nickel particle dispersion.
- **Enhanced mechanical properties:** The compression tests reveal that the optimized process leads to higher material strength and more predictable fracture behavior, making the composite more structurally robust.

This study introduced a temperature-controlled polymerization approach to regulate gelation kinetics and improve microstructural integrity. Additionally, it demonstrates the effectiveness of a modified sequence of material addition (delayed Ni introduction) as a strategy to reduce defects and refine phase distribution. This is the first time an evaluation of cooling effects on centrifugal gel casting has been performed, providing insights into the relationship between the processing parameters and composite performance. These findings contribute to the development of more reliable and defect-free ceramic-metal composites with potential applications as structural materials.

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