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## **MICROSTRUCTURE AND MECHANICAL PERFORMANCE OF NOVEL Al<sub>2</sub>O<sub>3</sub>-Cu-Cr COMPOSITES CONSOLIDATED BY CONVENTIONAL POWDER METALLURGY**

### **ABSTRACT**

This study investigates the fabrication, microstructural evolution, and mechanical properties of Al<sub>2</sub>O<sub>3</sub>-Cu-Cr ceramic-metal composites containing 2.5 vol.% metallic phase produced by uniaxial pressing followed by free sintering in a reducing atmosphere. The composites were sintered at temperatures ranging from 1200°C to 1400°C in order to evaluate the influence of thermal processing on densification behaviour, phase stability, and mechanical performance. The results demonstrated a strong dependence of physical properties on sintering temperature. Increasing the temperature from 1200°C to 1400°C improved the relative density from 82.85% to 97.99%, while open porosity decreased from 16.57% to 0.09%. Simultaneously, water absorption was reduced from 4.66% to 0.2%, indicating near-complete densification at 1400°C. XRD analysis confirmed the presence of stable  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>, Cu, and Cr phases without formation of undesirable secondary reaction products. SEM/EDS observations revealed a homogeneous distribution of discrete Cu-Cr metallic regions within the alumina matrix and satisfactory ceramic-metal interfacial bonding. The composites sintered at 1400°C exhibited a hardness of  $13.8 \pm 1.6$  GPa and fracture toughness of  $4.41 \pm 0.78$  MPa·m<sup>0.5</sup>. Digital image correlation analysis during compression testing demonstrated localized strain evolution preceding brittle fracture. The results indicate that Cr addition may contribute to improved microstructural stability and densification behaviour of Al<sub>2</sub>O<sub>3</sub>-Cu composites processed by conventional powder metallurgy routes.

**Keywords:** *Ceramic-metal composites; pressure-less sintering; Microstructure evolution; mechanical properties*

### **INTRODUCTION**

The development of advanced ceramic-metal composites has become an important direction in modern materials science due to the increasing demand for structural and functional materials capable of operating under extreme mechanical, thermal, and tribological conditions. Among ceramic materials, aluminium oxide is widely recognized for its high hardness, excellent wear resistance, chemical stability, and relatively low cost [1-3]. However, its intrinsic brittleness and limited fracture toughness significantly restrict its broader engineering applications, particularly in environments involving dynamic loading or thermal shock [4-7]. For this reason, numerous studies have focused on improving the

mechanical reliability of alumina-based systems through the incorporation of metallic phases, leading to the development of ceramic-metal composites [8-10].

The introduction of ductile metallic components into an alumina matrix can effectively enhance fracture toughness, crack deflection capability, thermal conductivity, and resistance to catastrophic failure [11-15]. Among the metallic additives investigated to date, copper has attracted considerable attention because of its high electrical and thermal conductivity, relatively low cost, and ability to improve energy dissipation during crack propagation [17-19]. Nevertheless, the direct incorporation of copper into alumina systems remains challenging due to poor wettability between Cu and  $\text{Al}_2\text{O}_3$ , the tendency of copper to segregate during thermal processing, and the mismatch in thermal expansion coefficients between ceramic and metallic constituents [20-22]. These factors may lead to interfacial decohesion, residual stresses, and microstructural instability after sintering.

To overcome these limitations, the addition of chromium represents a promising approach. Chromium may contribute to improved interfacial compatibility between Cu and  $\text{Al}_2\text{O}_3$ , possibly related to its higher oxygen affinity and possible localized interfacial interactions during sintering. [23-24]. Furthermore, chromium can influence diffusion processes during sintering, stabilize the microstructure, and potentially improve mechanical strength and wear resistance [25-28]. The simultaneous incorporation of Cu and Cr into an  $\text{Al}_2\text{O}_3$  matrix therefore creates the possibility of obtaining composites that combine the high hardness and chemical stability of ceramics with the enhanced toughness and functional properties associated with metallic phases.

Research on  $\text{Al}_2\text{O}_3$ -Cu-Cr composites is particularly important because relatively limited information is available regarding the synergistic interactions between copper and chromium in alumina-based systems processed by conventional powder metallurgy routes. Most previous investigations have focused primarily on binary  $\text{Al}_2\text{O}_3$ -Cu [29-33] or  $\text{Al}_2\text{O}_3$ -Cr systems [34-38], while ternary composites remain insufficiently explored, especially in the context of microstructure evolution, phase distribution, densification behavior, and interfacial phenomena during sintering. Consequently, there is still a need to understand better how the simultaneous presence of Cu and Cr affects the final composite architecture and resulting properties.

An additional scientific and technological motivation for this research is related to the processing method itself. Uniaxial pressing combined with free sintering constitutes one of the most industrially accessible and economically attractive fabrication routes for ceramic composites [39-40]. The method enables relatively simple shaping of green bodies, scalability for industrial production, and compatibility with conventional powder processing technologies. Despite these advantages, obtaining homogeneous  $\text{Al}_2\text{O}_3$ -Cu-Cr composites using this approach remains difficult because of differences in particle morphology, density, plasticity, and diffusion behaviour among the constituent powders. Therefore, investigations focused on optimizing processing conditions and understanding the relationship between powder characteristics, compaction behaviour, and sintering mechanisms are of considerable importance for both fundamental science and industrial implementation.

The novelty of the present research lies in the fabrication and characterization of ternary  $\text{Al}_2\text{O}_3$ -Cu-Cr composites produced by uniaxial pressing and free sintering, with particular emphasis on the role of chromium in modifying the microstructure and interfacial interactions within the composite system. The study contributes to the field of materials science by providing insight into the mechanisms governing densification, metallic phase distribution, and ceramic-metal bonding in complex multiphase composites. Furthermore, the work expands current knowledge concerning the design of alumina-based materials with simultaneously improved mechanical and functional properties.

Despite previous investigations of binary Al<sub>2</sub>O<sub>3</sub>-Cu and Al<sub>2</sub>O<sub>3</sub>-Cr systems, the influence of chromium on the densification behaviour, metallic phase distribution, and ceramic-metal interactions in ternary Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composites remains insufficiently understood. Therefore, the objective of the present study was to fabricate Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composites by uniaxial pressing followed by pressureless sintering and to evaluate the influence of processing conditions on phase composition, microstructural evolution, densification behaviour, and mechanical performance. Particular attention was devoted to assessing the distribution of the Cu-Cr metallic phase within the alumina matrix and to identifying microstructural factors governing the mechanical response of the composites.

## **MATERIALS AND METHODS**

Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composites were fabricated via uniaxial pressing. As a binder, a 10 wt.% aqueous solution of polyvinyl alcohol (PVA) was introduced in an amount corresponding to 10 wt.% of the total powder mixture. The selection of both the binder type and its content was based on earlier investigations performed by the research group [39, 44]. Green bodies were formed using a hydraulic press equipped with a 20 mm diameter cemented carbide die. In all cases, the applied compaction pressure was 100 MPa. The obtained specimens contained 2.5 vol.% of the metallic phase, with an equal Cu-to-Cr ratio (1.25:1.25). This choice was intentional, as it reflects a design strategy aimed at maintaining the structural integrity of the ceramic matrix while systematically investigating the influence of chromium additions at the interface level. Subsequently, the green compacts were subjected to pressureless sintering in a Nabertherm RHTC 80-230/16 tube furnace under a reducing atmosphere consisting of 95% Ar and 5% H<sub>2</sub>. Sintering experiments were conducted at four different temperatures: 1200°C (Series I), 1250°C (Series II), 1300°C (Series III), and 1400°C (Series IV). The maximum temperature was selected based on literature data reporting its suitability for obtaining highly densified Al<sub>2</sub>O<sub>3</sub>-based ceramic-metal composites [45]. Lower temperatures were additionally investigated to evaluate the feasibility of reducing the overall processing temperature while maintaining satisfactory material consolidation. For all tested variants, the thermal cycle remained unchanged. The heating schedule consisted of three stages: heating from 20°C to 120°C at a rate of 5°C/min, followed by heating from 120°C to 750°C at 1°C/min, and finally heating from 750°C to the target sintering temperature at 2°C/min. Each sample was held at the final temperature for 2 h and subsequently cooled to room temperature at a rate of 4°C/min.

The physical properties of the Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composites were determined using the Archimedes immersion technique. Measurements were performed according to standard procedures commonly applied to sintered materials and were based on mass measurements carried out in air and in a liquid of known density, namely distilled water. Before the analysis, all specimens were dried until a constant mass was achieved in order to eliminate the effect of residual moisture on the measurement results. Each sample was first weighed in air, then immersed in distilled water to determine the suspended mass, and finally weighed in the water-saturated condition after careful removal of excess surface liquid. The Archimedes method provides an effective approach for evaluating the densification behaviour and porosity characteristics of ceramic-metal composites. In the case of Al<sub>2</sub>O<sub>3</sub>-Cu-Cr materials, the obtained density values indirectly reflect the efficiency of powder compaction during uniaxial pressing as well as the degree of consolidation achieved during sintering. Furthermore, the apparent porosity values indicate the presence and extent of residual open pores within the

material structure, which may significantly affect properties such as mechanical performance, thermal transport, and the overall functional behaviour of the composites.

Linear and volumetric shrinkage measurements were performed to evaluate the dimensional changes occurring in the  $\text{Al}_2\text{O}_3\text{-Cu-Cr}$  composites during the sintering process. The dimensions of the green and sintered specimens were determined using a digital caliper with an accuracy of  $\pm 0.01$  mm. For each sample, the diameter and thickness were measured at several locations, and the average values were used for further calculations in order to minimize measurement uncertainty associated with possible geometric irregularities. Linear shrinkage was calculated based on the change in the characteristic dimensions of the specimens before and after sintering (regarding changes occurring along the sample diameter and along the sample height). The volumetric shrinkage was determined from the calculated sample volumes before and after thermal treatment. The shrinkage measurements provide important information regarding the densification behaviour of the ceramic-metal composites and enable assessment of the influence of sintering temperature on the consolidation efficiency of the  $\text{Al}_2\text{O}_3\text{-Cu-Cr}$  system.

Phase composition and crystallographic analysis of the  $\text{Al}_2\text{O}_3\text{-Cu-Cr}$  composites were carried out using X-ray diffraction (XRD) techniques with  $\text{Cu K}\alpha$  radiation ( $\lambda = 1.5406 \text{ \AA}$ ). Diffraction measurements were performed over a selected  $2\theta$  angular range using a constant scanning step and fixed counting time in order to obtain diffraction patterns with adequate peak intensity and resolution. The XRD measurements were performed using an operating voltage of 30 kV and a tube current of 15 mA. Diffraction data were acquired within a  $2\theta$  angular range of  $20^\circ\text{--}100^\circ$ , which enabled the identification of the principal crystalline phases present in the investigated composites. All scans were recorded using a step increment of  $0.05^\circ$ , while the counting time for each measurement point was set to 5 s in order to obtain diffraction patterns with satisfactory signal intensity and peak resolution. The measurement conditions were chosen to ensure accurate detection of both the ceramic matrix and metallic or oxide phases that could form during the sintering process. Prior to analysis, the surfaces of the sintered samples were carefully prepared to minimize the influence of surface irregularities and contamination on the recorded diffraction signals. The collected diffractograms were subsequently processed and interpreted through comparison with reference crystallographic data available in standard diffraction databases. Particular attention was paid to the identification of phases associated with the  $\text{Al}_2\text{O}_3$  matrix, metallic Cu and Cr constituents, as well as possible reaction products and oxide phases generated during thermal treatment. The XRD analysis enabled the evaluation of phase stability in the composite system after sintering under a reducing atmosphere and provided information regarding potential phase transformations occurring at elevated temperatures. In addition, the diffraction data were used to assess whether undesired secondary phases, including spinel-type structures or oxidation products, were formed during processing.

Detailed microstructural investigations of the  $\text{Al}_2\text{O}_3\text{-Cu-Cr}$  composites were carried out using scanning electron microscopy (SEM). The analyses included observations of the starting powders as well as the microstructure of the sintered materials in both polished cross-sections and fracture surfaces. Examinations were performed using a JEOL JSM-6610 scanning electron microscope operated at an accelerating voltage of 15 kV. Secondary electron (SE) imaging was employed primarily to evaluate the surface topography, particle morphology, and fracture characteristics of the investigated materials. In addition, backscattered electron (BSE) observations were conducted to improve the contrast between phases with different average atomic numbers, thereby enabling a more precise distinction between the  $\text{Al}_2\text{O}_3$  ceramic matrix and the metallic Cu-Cr regions. The use of BSE imaging also facilitated the assessment of metallic phase distribution, agglomeration phenomena, and the presence of

microstructural heterogeneities formed during the sintering process. Prior to SEM examination, all specimens were prepared carefully to ensure high-quality imaging conditions. Polished samples were mechanically ground and polished to obtain smooth surfaces suitable for microstructural analysis, while fracture surfaces were analysed in the as-fractured state to investigate fracture mechanisms and interfacial behaviour between the ceramic and metallic constituents. To minimize charging effects associated with the non-conductive ceramic matrix, the samples were coated with a thin conductive carbon layer using a JEOL Fine Coat Ion Sputter JFC-1100 system before microscopic observations. This conductive coating improved signal stability and image quality during SEM analysis.

The chemical composition and elemental distribution within the Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composites were investigated using energy-dispersive X-ray spectroscopy (EDS) coupled with scanning electron microscopy (SEM). The analyses were carried out at an accelerating voltage of 15 kV, which provided sufficient excitation energy for the characteristic X-ray emissions of both the ceramic and metallic constituents while preserving satisfactory spatial resolution for microstructural observations. Elemental distribution maps and point analyses were acquired in a semi-quantitative mode without the application of external calibration standards. The obtained results were therefore interpreted primarily on a qualitative basis in order to determine the spatial distribution of the principal elements and to evaluate the morphology of the interfaces between the ceramic matrix and the metallic phase. For each analysed region, the acquisition time was set to approximately 120 s, ensuring an adequate signal-to-noise ratio and reliable detection of the major constituent elements. Particular attention was paid to the identification of the distribution of aluminium, oxygen, copper, and chromium throughout the composite microstructure, as well as to the possible occurrence of secondary phases or reaction products formed during sintering. The EDS investigations were additionally used to assess the homogeneity of the metallic phase and the presence of local agglomerations or compositional gradients within the material. It should be emphasized that EDS analysis is subject to several intrinsic limitations. In particular, the detection and quantification of light elements, especially oxygen, are associated with reduced accuracy, possibly related to factors such as X-ray absorption, detector sensitivity, and the relatively large electron interaction volume generated within the sample. Moreover, at the applied accelerating voltage, the interaction volume may exceed the dimensions of fine microstructural features, which can result in partial overlap of signals originating from neighbouring phases, especially near phase boundaries and interfaces. Consequently, the EDS results presented in this work should be regarded mainly as qualitative confirmation of elemental distribution and phase arrangement on the micrometer scale. Although no additional reaction products or secondary phases were identified within the detection limits of the applied method, the existence of extremely thin interfacial layers or nanoscale reaction zones cannot be entirely excluded.

The mechanical properties of the Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composites were evaluated by means of Vickers hardness measurements. The tests were carried out on flat, polished surfaces prepared according to conventional metallographic procedures in order to ensure adequate surface quality and minimize the influence of roughness or geometric irregularities on the indentation results. Prior to testing, the specimens were sequentially ground and polished to obtain parallel surfaces suitable for reliable hardness determination. Hardness measurements were performed using an HVS-30T Vickers hardness tester. A constant load of 98 N was applied with a dwell time of 15 s for each indentation. Identical testing parameters were maintained throughout the entire experimental series to ensure consistency, reproducibility, and direct comparability of the obtained results between individual samples and sintering conditions. For each specimen, multiple indentations were introduced at different locations on the polished surface in order to reduce the influence of local microstructural heterogeneities, such as pores,

metallic phase agglomerations, or grain size variations. The diagonal lengths of the indentation marks were measured optically, and the corresponding hardness values were calculated automatically by the testing system. The final hardness value reported for each material variant was determined as the arithmetic mean of the individual measurements. The Vickers hardness analysis provided information regarding the influence of sintering temperature, densification degree, and microstructural evolution on the mechanical response of the investigated composites. In ceramic–metal systems such as  $\text{Al}_2\text{O}_3$ -Cu-Cr, hardness is strongly affected by factors including residual porosity, phase distribution, and the quality of interfacial bonding between the ceramic matrix and metallic constituents.

The fracture resistance of the  $\text{Al}_2\text{O}_3$ -Cu-Cr composites was assessed using the indentation fracture method based on Vickers indentation testing. Fracture toughness values were estimated using the empirical relationship proposed by Lankford [46], which is commonly applied to ceramic materials exhibiting different crack morphologies generated around Vickers indentations. This approach enables an indirect evaluation of the material's resistance to crack initiation and propagation under localized mechanical loading. The measurements were carried out on polished specimen surfaces prepared according to standard metallographic procedures. Indentations were introduced using a load of 98 N, identical to that applied during hardness testing, in order to ensure consistency between the mechanical characterization methods. After indentation, the radial cracks extending from the corners of the Vickers imprints were examined by optical microscopy, and their lengths were measured for subsequent calculations of fracture toughness. For each material variant, including samples sintered at different temperatures, ten independent indentations were performed to improve the statistical reliability and reproducibility of the obtained results. The individual crack length measurements were averaged and used to determine the representative fracture toughness values for each composite composition and processing condition. The indentation fracture method provides comparative information regarding the influence of microstructural features such as porosity, grain bonding, metallic phase distribution, and interfacial cohesion on the crack resistance of ceramic–metal composites. Nevertheless, it should be emphasized that this technique yields approximate fracture toughness values and is sensitive to factors including crack geometry, residual stresses, and the presence of local microstructural heterogeneities. Therefore, the obtained results were interpreted primarily in a comparative manner to evaluate trends associated with changes in sintering temperature and composite microstructure.

The mechanical behaviour of the sintered  $\text{Al}_2\text{O}_3$ -Cu-Cr composites was evaluated under monotonic uniaxial compression to determine their compressive strength and to characterize deformation and failure mechanisms using digital image correlation (DIC). Cylindrical specimens ( $\text{Ø}20 \text{ mm} \times \sim 10 \text{ mm}$ ;  $H/D \approx 0.5$ ) were machined to minimize buckling and ensure stable stress conditions typical for brittle ceramic-based materials. Prior to testing, both loading faces were ground and finely polished to ensure planarity and parallelism, thereby reducing bending effects and ensuring uniform stress distribution. Final surface finishing removed residual irregularities to limit local stress concentrations at the platen–specimen interface. Careful alignment was applied during mounting, and friction at the contacts was minimized using a thin lubricant or compliant interlayer when required.

Compression tests were performed on a servo-hydraulic testing machine under displacement control at  $0.5 \text{ mm/min}$ , corresponding to a nominal strain rate of  $\sim 10^{-3} \text{ s}^{-1}$ . Load and displacement were continuously recorded up to catastrophic failure. All tests were conducted at  $22 \pm 2^\circ\text{C}$ . At least eight specimens per composition were tested to ensure statistical significance, and the averaged results are reported. Full-field strain measurements were obtained using a Dantec Q-400 DIC system equipped with two 8-megapixel cameras.

The system provided a displacement resolution of  $\sim 0.01$  pixel and a strain resolution of  $10^{-4}$ . Data acquisition and processing were performed using ISTR 4D software, enabling quantitative analysis of strain localization and qualitative visualization of deformation evolution. Force displacement curves were derived from the recorded mechanical response, while representative DIC strain maps were used to elucidate local deformation and failure processes in the investigated composites.

The grain size of the Al<sub>2</sub>O<sub>3</sub> ceramic matrix was determined through stereological evaluation of fracture surfaces observed by scanning electron microscopy (SEM). Individual alumina grains were identified and manually segmented, with particular attention paid to their irregular three-dimensional morphology as reflected in the two-dimensional fracture surface projections. Image-based analysis was performed using digital processing techniques to delineate grain boundaries and convert the micrographs into binary representations suitable for quantitative measurements. Quantitative stereological analysis was carried out using Micrometer software [46-49], which enabled the determination of the equivalent circle diameter for each grain. The equivalent grain size parameter, denoted as  $d_2$ , was defined as the diameter of a circle having the same projected area as the analysed grain [46-49]. This approach allows complex and non-equiaxed grain geometries to be represented by a single, statistically meaningful size descriptor. On the basis of the measured population, grain size distributions were constructed for all investigated composite variants, expressed as percentage frequency histograms of equivalent diameters. To ensure statistical robustness and minimize sampling bias, SEM images were selected randomly from different regions of each specimen, and at least 1000 grains were evaluated for each material condition, including samples tested under different temperature regimes. This extensive dataset provided a reliable representation of the microstructural state of the alumina matrix. The resulting grain size distributions were subsequently used to support microstructure property correlations, enabling a more detailed interpretation of the relationship between ceramic matrix morphology and the observed mechanical response of the composites.

## RESULTS AND DISCUSSION

Figure 1 presents SEM micrographs of the metallic powders (Cu and Cr) used for the fabrication of Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composites consolidated by uniaxial pressing followed by free sintering. The morphology and particle size distribution of the powders are expected to significantly influence powder packing, densification behaviour, and the final microstructure of the composites. The Cu powder (Figure 1a) consisted predominantly of irregular agglomerates with sizes ranging from approximately 10 to 80  $\mu\text{m}$ , with the majority of particles observed in the 20-50  $\mu\text{m}$  range. Higher magnification analysis showed that these agglomerates were composed of fine, rounded primary particles with diameters of approximately 1-5  $\mu\text{m}$ . The copper powder (Figure 1a) is characterized by strongly agglomerated particles with an irregular, dendritic-like morphology. At higher magnification, the agglomerates are seen to consist of numerous rounded primary particles fused into porous clusters. The particle surfaces appear relatively smooth, while the overall morphology indicates a high specific surface area and a strong tendency toward agglomeration. Such morphology may promote enhanced interparticle contact during sintering, possibly related to the increased surface energy of the powder. However, the presence of large agglomerates can also reduce powder flowability and lead to local heterogeneities during compaction. The characteristic size of the Cu agglomerates ranges from several micrometers to approximately several tens of micrometers.

In contrast, the chromium powder (Figure 1b) exhibits a distinctly different morphology. The particle size, measured along the largest dimension, ranged from approximately 10  $\mu\text{m}$  to 120  $\mu\text{m}$ , with most particles between 20  $\mu\text{m}$  and 60  $\mu\text{m}$ . Compared with the Cu powder, the Cr particles appeared denser and less agglomerated, with relatively smooth surfaces and irregular, sharp edges characteristic of mechanically fragmented powders. The particles are predominantly flake-like and angular, with irregular, sharp-edged geometries. The powder particles show a relatively dense and compact structure with smoother surfaces compared with the copper agglomerates. Chromium particles possess a broader size distribution and are generally larger in lateral dimensions than the primary Cu particles. The lamellar morphology of the Cr powder is typical for mechanically crushed metallic powders and may contribute to anisotropic packing behaviour during uniaxial pressing. Additionally, the angular morphology can increase mechanical interlocking between particles, influencing green body strength and densification kinetics during free sintering.

The substantial differences in morphology between Cu and Cr powders are expected to affect the processing behaviour of the  $\text{Al}_2\text{O}_3$ -Cu-Cr composites. The presence of Cu may enhance local mass transport during sintering. Consequently, the combined presence of both metallic phases can strongly influence the microstructural evolution, porosity distribution, and mechanical properties of the sintered composites.

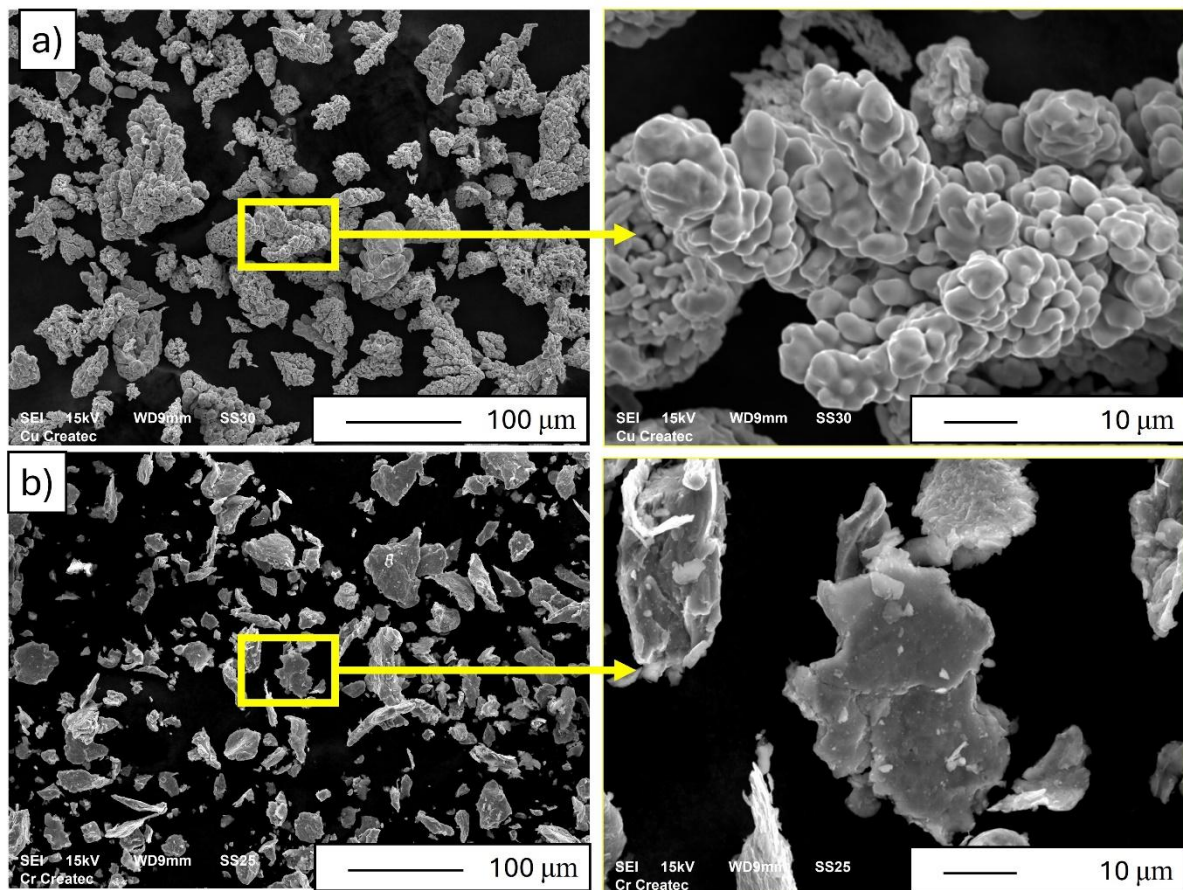


**Fig. 1.** SEM micrographs of the metallic powders used for fabrication of  $\text{Al}_2\text{O}_3$ -Cu-Cr composites by uniaxial pressing and free sintering: a) Cu powder with agglomerated morphology composed of fine, rounded primary particles, and b) Cr powder with irregular flake-like morphology. The estimated particle/agglomerate sizes were approximately 10-80  $\mu\text{m}$  for Cu and 10-120  $\mu\text{m}$  for Cr

The SEM micrograph of the TM-DAR alumina powder (Figure 2) reveals a fine-grained morphology composed of nearly spherical to slightly irregular primary particles forming soft agglomerates. The powder exhibits a relatively homogeneous microstructure, with particles densely packed and interconnected, which is characteristic of nanosized transition alumina powders with high specific surface area. Based on the scale bar analysis, the primary Al<sub>2</sub>O<sub>3</sub> particles are estimated to be in the nanometric range, typically about 50-150 nm in diameter. In several regions, the particles appear fused into larger agglomerates with sizes reaching approximately 0.2-0.5 μm. The observed agglomeration is most likely associated with the high surface energy of the fine alumina particles, promoting particle-particle interactions during powder processing and drying. The particle size distribution appears relatively narrow, and no large faceted grains or abnormal grain growth can be observed in the analysed area. The Al<sub>2</sub>O<sub>3</sub> powder is characterized by a fine and uniform morphology suitable for preparing highly stable ceramic suspensions and obtaining dense sintered Al<sub>2</sub>O<sub>3</sub>-based composites.

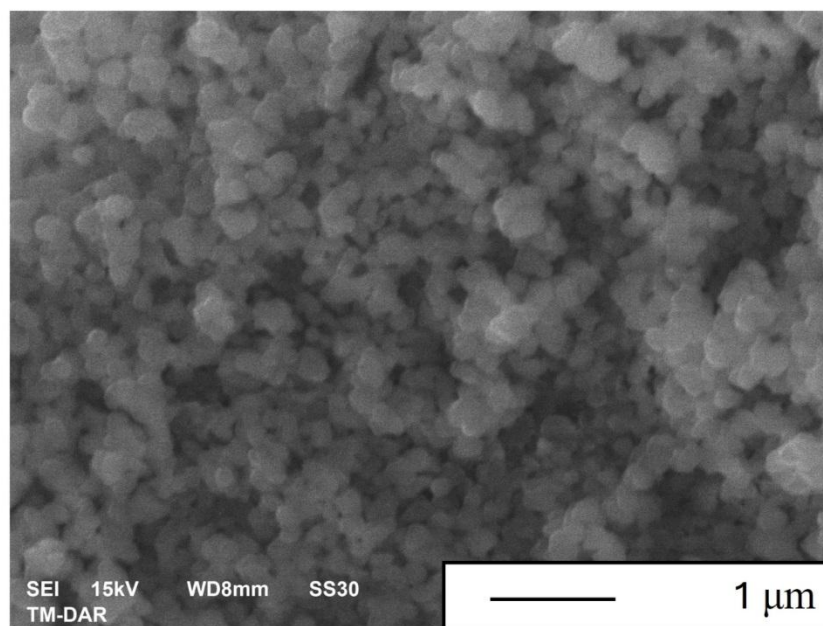


**Fig. 2.** Morphology of the starting alumina powder used for the fabrication of Al<sub>2</sub>O<sub>3</sub>-Cu-Cr

Table 1 presents the selected physical properties of Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composites containing 2.5 vol.% metallic phase, fabricated by uniaxial pressing followed by free sintering at temperatures ranging from 1200°C to 1400°C. The obtained results clearly demonstrate that the sintering temperature had a significant influence on the densification behaviour and pore structure evolution of the composites. At the lowest sintering temperature of 1200°C, the composites exhibited the lowest relative density, equal to  $82.85 \pm 0.55\%$ , accompanied by a high open porosity of  $16.57 \pm 0.54\%$  and water absorption of  $4.66 \pm 0.18\%$ . These results indicate insufficient densification of the ceramic-metal system at this temperature. The limited diffusion processes occurring during sintering were not sufficient to effectively eliminate interparticle voids, resulting in a highly porous microstructure. In ceramic composites, such high porosity is typically associated with weak particle bonding and incomplete neck formation between alumina grains and metallic particles. Increasing the sintering temperature to 1250°C resulted in a substantial improvement in densification. The relative density increased to  $91.08 \pm 0.50\%$ , while the open porosity and water absorption decreased to  $8.06 \pm 0.52\%$  and  $2.11 \pm 0.15\%$ , respectively. This behavior is related to the intensification of diffusion mechanisms and enhanced mass transport during sintering. At higher temperatures, particle rearrangement and grain boundary diffusion become more effective, promoting pore

shrinkage and consolidation of the composite structure. A further increase in temperature to 1300°C led to a moderate additional improvement in physical properties. The relative density reached  $92.43 \pm 0.10\%$ , whereas the open porosity and water absorption decreased to  $5.33 \pm 0.13\%$  and  $1.39 \pm 0.04\%$ , respectively. Although the improvement between 1250°C and 1300°C was less pronounced than that observed between 1200°C and 1250°C, the results indicate the continuation of densification processes and progressive elimination of residual open pores. The reduced rate of density increase may suggest that the system approached an intermediate stage of sintering where pore closure became increasingly difficult. The most significant improvement in composite properties was observed after sintering at 1400°C.

The relative density increased to  $97.99 \pm 0.15\%$ , approaching theoretical densification, while the open porosity decreased dramatically to only  $0.09 \pm 0.02\%$ . Simultaneously, water absorption reached a very low value of  $0.2 \pm 0.01\%$ , confirming the formation of a nearly fully dense microstructure. Such behaviour indicates highly effective sintering kinetics at 1400°C, enabling extensive diffusion and efficient pore elimination. The near-complete reduction of open porosity suggests that most pores became closed or were entirely removed during densification. The observed relationship between sintering temperature and composite properties is strongly associated with the behaviour of the metallic Cu-Cr phase within the alumina matrix. As the temperature increases, the metallic particles can promote localized diffusion processes and improve particle packing efficiency. Chromium may additionally enhance interfacial bonding between copper and alumina due to its higher chemical affinity for oxygen and improved wettability characteristics compared with pure copper systems. Improved interfacial interactions contribute to stronger bonding at the ceramic-metal interface and facilitate densification. The decrease in open porosity directly influenced the reduction in water absorption. Since water penetration occurs mainly through interconnected pores, the progressive closure and elimination of porosity at higher temperatures significantly limited liquid uptake. This correlation confirms the strong dependence of water absorption on the pore structure of the composites. The results demonstrate that increasing the sintering temperature substantially improves the physical properties of  $\text{Al}_2\text{O}_3\text{-Cu-Cr}$  composites containing 2.5 vol.% metallic phase. Higher temperatures promote enhanced densification, reduction of porosity, and formation of a more compact microstructure. Among the investigated conditions, the best overall properties were obtained for composites sintered at 1400°C, indicating that this temperature provides the most favourable conditions for effective consolidation of the  $\text{Al}_2\text{O}_3\text{-Cu-Cr}$  system.

The shrinkage behaviour of the  $\text{Al}_2\text{O}_3\text{-Cu-Cr}$  composites presented in Table 1 demonstrates a strong dependence on the applied sintering temperature and provides important insight into the densification mechanisms occurring during free sintering. Both linear and volumetric shrinkage are directly associated with the elimination of pores, particle rearrangement, and mass transport processes activated during thermal treatment. The obtained results indicate that increasing the sintering temperature significantly influenced the dimensional changes of the composites, reflecting the progressive evolution of the microstructure and densification kinetics. The highest values of linear and volumetric shrinkage were observed for the samples sintered at 1200°C. The linear shrinkage measured relative to the sample diameter reached  $16.43 \pm 0.07\%$ , while the shrinkage measured relative to the sample height was equal to  $14.73 \pm 0.28\%$ . Simultaneously, the volumetric shrinkage attained the maximum value of  $40.45 \pm 0.29\%$ . Such high shrinkage values indicate intensive dimensional contraction during the initial and intermediate stages of sintering. At this temperature, the composite still exhibited relatively high open porosity and low relative density, suggesting that the material underwent substantial geometric contraction associated with the rearrangement of particles and formation of initial sintering necks. The large

volumetric shrinkage may also result from the collapse of loosely packed powder agglomerates formed during uniaxial pressing. As the sintering temperature increased to 1250°C and 1300°C, both linear and volumetric shrinkage systematically decreased. At 1250°C, the linear shrinkage decreased to  $14.84 \pm 0.04\%$  in diameter and  $11.70 \pm 2.59\%$  in height, while the volumetric shrinkage dropped to  $35.97 \pm 1.93\%$ . Further increasing the temperature to 1300°C reduced these values to  $13.11 \pm 0.05\%$ ,  $11.56 \pm 0.39\%$ , and  $33.23 \pm 0.26\%$ , respectively. This gradual reduction in shrinkage despite improved densification indicates that the composites entered a more advanced sintering stage. In this regime, the pore volume was progressively reduced, and the microstructure became increasingly compact, limiting the extent of further dimensional contraction. The reduction in shrinkage can therefore be associated with the transition from rapid particle rearrangement toward grain-boundary diffusion and pore closure mechanisms. The lowest shrinkage values were obtained for the composites sintered at 1400°C, where the linear shrinkage measured relative to the sample diameter and height reached  $10.11 \pm 0.11\%$  and  $9.62 \pm 0.52\%$ , respectively, while the volumetric shrinkage decreased to  $26.98 \pm 0.32\%$ . These results correspond closely with the highest relative density and the nearly complete elimination of open porosity observed at this temperature. The reduced shrinkage at 1400°C suggests that most densification processes had already been completed and the microstructure approached a near-equilibrium dense state. At this stage, the remaining isolated pores were significantly smaller and less capable of contributing to further dimensional contraction. The lower shrinkage values combined with high densification indicate highly efficient sintering kinetics and stabilization of the composite structure. The observed decrease in shrinkage with increasing temperature may initially appear counterintuitive, as higher temperatures generally intensify diffusion processes. However, in the investigated system, the reduction in shrinkage is associated with the improved packing and consolidation efficiency achieved at elevated temperatures. At lower temperatures, the structure contains a large amount of interconnected porosity and undergoes substantial contraction during densification. In contrast, at higher temperatures, densification becomes more effective, resulting in earlier pore elimination and stabilization of the dimensions of the sintered body. Additionally, the presence of the Cu-Cr metallic phase may contribute to enhanced local diffusion and improved particle rearrangement during sintering, facilitating more homogeneous densification and reducing anisotropic shrinkage effects. An important observation is the relatively small difference between shrinkage measured along the diameter and height directions, particularly at 1400°C. Slightly higher shrinkage values measured in the radial direction may indicate residual anisotropy introduced during pressing, where particle orientation and packing density can differ between axial and radial directions. The shrinkage results confirm that the sintering temperature strongly affects the densification behaviour and dimensional stability of Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composites. Increasing the temperature from 1200°C to 1400°C led to progressive stabilization of the microstructure, improved densification efficiency, and reduction of total shrinkage. The combination of low shrinkage and near-theoretical density obtained at 1400°C indicates that this temperature provides optimal conditions for producing dense and dimensionally stable Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composites by uniaxial pressing and free sintering.

Figure 3 presents the X-ray diffraction (XRD) patterns of the Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composites containing 2.5 vol.% metallic phase fabricated by uniaxial pressing followed by free sintering at temperatures of 1200°C, 1250°C, 1300°C, and 1400°C. The diffraction patterns reveal that the dominant crystalline phase in all investigated samples is  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>, identified by the characteristic intense reflections located approximately at  $2\theta = 25.6^\circ$ ,  $35.1^\circ$ ,  $43.3^\circ$ ,  $52.5^\circ$ ,  $57.5^\circ$ ,  $66.5^\circ$ , and  $68.2^\circ$ . The high intensity of alumina peaks results from the significantly larger fraction of the ceramic matrix relative to the metallic additives. In addition to the

alumina phase, diffraction peaks corresponding to metallic Cu and Cr were detected in all samples. Copper reflections are visible mainly near  $2\theta \approx 43.3^\circ$ ,  $50.4^\circ$ , and  $74.1^\circ$ , while chromium reflections appear at approximately  $2\theta \approx 44.5^\circ$ ,  $64.7^\circ$ ,  $81.7^\circ$ , and  $98.9^\circ$ .

**Table 1.** Selected physical properties of  $\text{Al}_2\text{O}_3$ -Cu-Cr composites with a 2.5% metallic phase content formed by uniaxial pressing with free sintering

| Physical properties |                      |                   |                      |                                                                      |                                                             |                          |
|---------------------|----------------------|-------------------|----------------------|----------------------------------------------------------------------|-------------------------------------------------------------|--------------------------|
| Temperature [°C]    | Relative density [%] | Open porosity [%] | Water absorption [%] | Linear shrinkage measured relative to the sample diameter linear [%] | Linear shrinkage measured relative to the sample height [%] | Volumetric shrinkage [%] |
| 1200                | $82.85 \pm 0.55$     | $16.57 \pm 0.54$  | $4.66 \pm 0.18$      | $16.43 \pm 0.07$                                                     | $14.73 \pm 0.28$                                            | $40.45 \pm 0.29$         |
| 1250                | $91.08 \pm 0.50$     | $8.06 \pm 0.52$   | $2.11 \pm 0.15$      | $14.84 \pm 0.04$                                                     | $11.70 \pm 2.59$                                            | $35.97 \pm 1.93$         |
| 1300                | $92.43 \pm 0.10$     | $5.33 \pm 0.13$   | $1.39 \pm 0.04$      | $13.11 \pm 0.05$                                                     | $11.56 \pm 0.39$                                            | $33.23 \pm 0.26$         |
| 1400                | $97.99 \pm 0.15$     | $0.09 \pm 0.02$   | $0.2 \pm 0.01$       | $10.11 \pm 0.11$                                                     | $9.62 \pm 0.52$                                             | $26.98 \pm 0.32$         |

The presence of Cu and Cr peaks confirms that the metallic components remained in the crystalline state after the sintering process, and no complete dissolution of the metallic phase into the alumina matrix occurred. The comparison of diffraction patterns obtained at different sintering temperatures indicates that increasing the sintering temperature from  $1200^\circ\text{C}$  to  $1400^\circ\text{C}$  does not lead to the formation of additional crystalline phases. In particular, no peaks associated with copper oxides, chromium oxides, spinel phases, or other reaction products were identified within the detection limit of the XRD method. This observation suggests relatively high phase stability of the  $\text{Al}_2\text{O}_3$ -Cu-Cr system during free sintering in the investigated temperature range. At lower sintering temperatures ( $1200^\circ\text{C}$ - $1250^\circ\text{C}$ ), the diffraction peaks assigned to Cu and Cr are relatively weak and partially overlap with intense alumina reflections due to the low metallic phase content. As the sintering temperature increases to  $1300^\circ\text{C}$ , the metallic reflections become slightly more pronounced, which may indicate improved crystallinity of the metallic particles and enhanced densification of the composite material. The sample sintered at  $1300^\circ\text{C}$  exhibits the highest overall peak intensities and the sharpest diffraction maxima, suggesting improved structural ordering and reduced microstructural defects. For the sample sintered at  $1400^\circ\text{C}$ , the diffraction peaks remain sharp and well-defined; however, a slight decrease in relative peak intensity can be observed compared with the specimen sintered at  $1300^\circ\text{C}$ . This effect may be associated with microstructural changes occurring at elevated temperatures, such as partial redistribution of the metallic phase, local diffusion phenomena, or residual stress relaxation.

Nevertheless, the absence of new diffraction peaks indicates that no significant chemical interaction between  $\text{Al}_2\text{O}_3$ , Cu, and Cr occurred even at the highest applied sintering temperature. The XRD analysis demonstrates that the applied sintering temperatures in the range of  $1200^\circ\text{C}$ - $1400^\circ\text{C}$  primarily influence the crystallinity and structural ordering of the composite rather than its phase composition. The  $\text{Al}_2\text{O}_3$ -Cu-Cr composites preserved a stable multiphase structure composed exclusively of  $\alpha$ - $\text{Al}_2\text{O}_3$ , Cu, and Cr throughout the entire sintering process.

Based on the results presented in Table 1, it was concluded that the sintering temperature had a decisive influence on the densification effectiveness of the  $\text{Al}_2\text{O}_3$ -Cu-Cr composites. The materials sintered at temperatures between  $1200^\circ\text{C}$  and  $1300^\circ\text{C}$  were characterized by relatively low relative density values, ranging from  $82.85 \pm 0.55\%$  to  $92.43 \pm 0.10\%$ , accompanied by considerable open porosity and elevated water absorption. Such results indicate incomplete consolidation of the composite structure and insufficient elimination of

pores during the sintering process. In ceramic-metal composites, residual porosity strongly affects mechanical performance, structural integrity, and reliability of the material, particularly under loading conditions. Therefore, the relatively low densification obtained at these temperatures suggests that the composites may not exhibit representative functional and mechanical properties required for further detailed characterization.

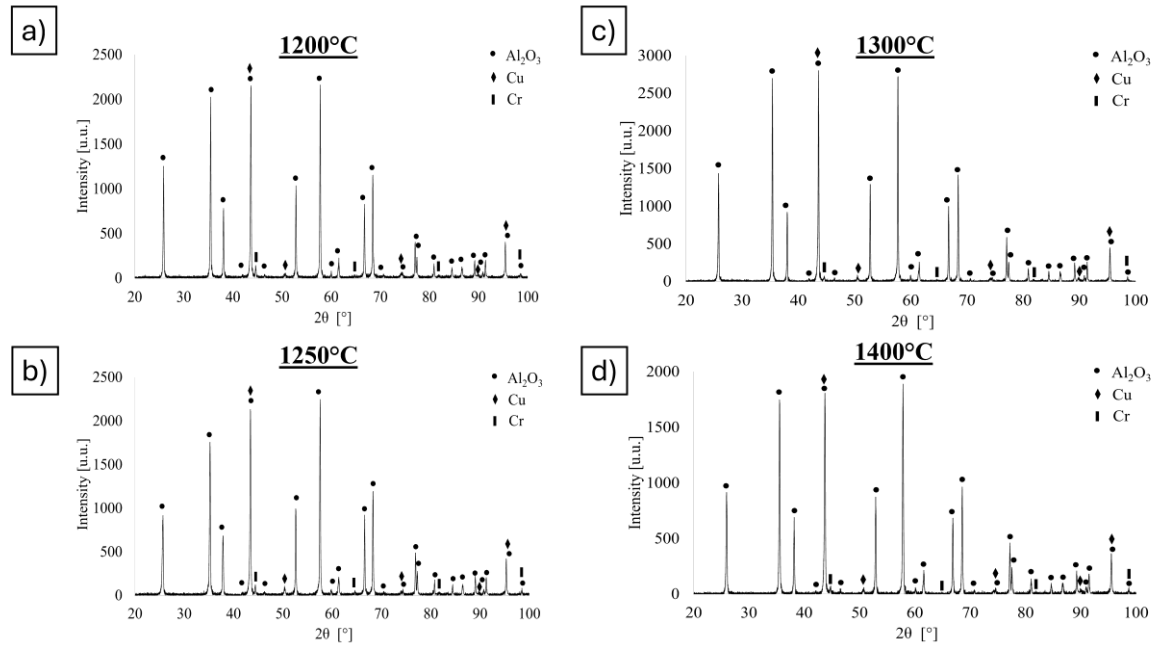


**Fig. 3.** The X-ray diffraction patterns of the Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composites containing 2.5 vol.% metallic phase were fabricated by uniaxial pressing followed by free sintering at temperatures of 1200°C, 1250°C, 1300°C, and 1400°C

A substantial improvement in densification was achieved only after sintering at 1400°C. The composites fabricated at this temperature reached a relative density of  $97.99 \pm 0.15\%$ , while the open porosity decreased to  $0.09 \pm 0.02\%$ , indicating the formation of a nearly fully dense microstructure. Simultaneously, the water absorption was reduced to a negligible level of  $0.2 \pm 0.01\%$ , confirming the effective elimination of interconnected pores. The absence of extensive interfacial decohesion suggests relatively good mechanical compatibility between phases. Considering the large differences in densification behaviour between the investigated sintering temperatures, further investigations were limited exclusively to the composites sintered at 1400°C. This decision was made to ensure that subsequent analyses, including microstructural observations, phase characterization, and mechanical testing, would be performed on materials with sufficiently high density and minimal porosity. Restricting further research to the fully densified series allows the obtained results to reflect the intrinsic properties of the Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composites rather than the effects associated with excessive residual porosity or incomplete sintering.

Figure 4 presents representative SEM micrographs of the Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composites containing 2.5 vol.% metallic phase after free sintering at 1400°C. The obtained microstructure is characterized by a dense alumina matrix with a relatively homogeneous distribution of metallic Cu-Cr regions embedded within the ceramic phase. The substantial reduction of residual porosity observed previously in the physical property analysis is confirmed by the microstructural observations, as only isolated pores of limited size are visible in the analysed areas. The microstructure appears well consolidated, indicating that the applied sintering temperature provided sufficient activation of diffusion-driven densification

mechanisms and effective elimination of interparticle voids. In the BSE micrographs, the metallic regions are visible as bright contrast areas, possibly related to the significantly higher average atomic number of Cu and Cr compared with the  $\text{Al}_2\text{O}_3$  matrix. The ceramic matrix forms a continuous dark-grey phase surrounding the metallic inclusions. Most metallic particles exhibit irregular morphologies and are distributed individually or in small localized agglomerates throughout the composite. The observed metallic inclusions generally range from several micrometers up to approximately several tens of micrometers in size, which is consistent with the morphology and particle size distribution of the starting metallic powders. The micrographs indicate that the metallic phase remained discrete during sintering and did not form an interconnected continuous network within the ceramic matrix. Higher magnification observations reveal that the bright metallic regions are characterized by complex internal morphology and irregular interfaces with the surrounding alumina matrix. In several regions, the metallic particles appear partially embedded within locally densified zones surrounding the inclusions. A characteristic feature visible around some metallic regions is the occurrence of a lighter grey transition zone between the bright metallic core and the darker alumina matrix. This contrast variation may indicate localized compositional gradients, diffusion effects, or subtle differences in polishing relief associated with the ceramic-metal interface. However, no distinct continuous reaction layer of significant thickness can be identified in the analysed regions. The interfaces between the metallic particles and the alumina matrix appear relatively well bonded, with no evidence of extensive interfacial decohesion or large interfacial cracks. This observation suggests satisfactory interfacial compatibility between the ceramic and metallic constituents during high-temperature sintering. The addition of chromium likely contributed to improved interfacial behaviour within the  $\text{Al}_2\text{O}_3$ -Cu system. Chromium may influence interfacial behaviour and metallic phase distribution during sintering. The absence of large interfacial gaps additionally indicates the high densification level previously identified by Archimedes measurements. At higher magnifications, the metallic regions exhibit locally heterogeneous contrast, suggesting the coexistence of Cu-rich and Cr-rich areas within individual metallic agglomerates. This behaviour is expected considering the limited mutual solubility and distinct diffusion characteristics of copper and chromium under the applied processing conditions. Copper, possibly related to its lower melting point and enhanced diffusivity at elevated temperatures, may locally redistribute during sintering and partially fill neighbouring pore regions. In contrast, chromium particles appear more angular and structurally stable, retaining morphologies closer to those of the starting powder. Consequently, the metallic inclusions visible in the microstructure likely consist of complex Cu-Cr regions formed through partial rearrangement and local consolidation during sintering rather than complete alloy homogenization. The SEM observations further indicate that the alumina matrix surrounding the metallic inclusions is relatively dense and fine-grained. No abnormal grain growth of the ceramic matrix was observed in the analysed regions, suggesting that the presence of the metallic phase did not induce excessive grain coarsening during sintering at  $1400^\circ\text{C}$ . The relatively uniform ceramic morphology indicates stable sintering behaviour and controlled microstructural evolution of the alumina phase. The absence of extensive grain growth is beneficial for maintaining high hardness and structural stability of the composite. An important observation is the very limited amount of residual porosity present near the metallic inclusions. In many ceramic-metal systems, thermal expansion mismatch between the metallic and ceramic phases can lead to pore formation or interfacial cracking during cooling. However, in the investigated composites, such defects appear significantly reduced. This suggests that the selected metallic phase content (2.5 vol.%) was sufficiently low to minimize

excessive thermal mismatch stresses while still enabling local toughening effects associated with the metallic inclusions.

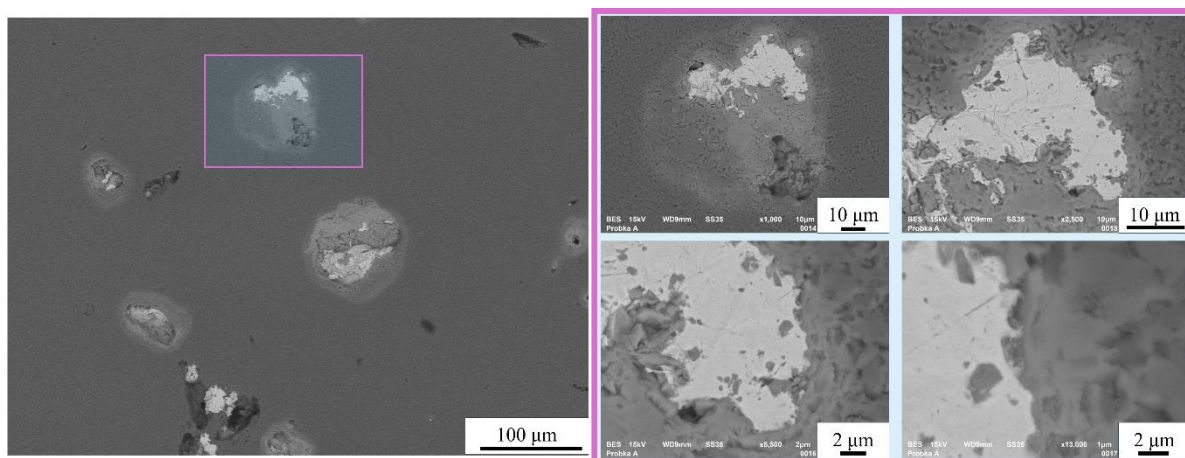


**Fig. 4.** SEM micrographs of the  $\text{Al}_2\text{O}_3$ -Cu-Cr composites containing 2.5 vol.% metallic phase after free sintering at  $1400^\circ\text{C}$

Figure 5 presents representative SEM/EDS elemental distribution maps obtained for the  $\text{Al}_2\text{O}_3$ -Cu-Cr composites containing 2.5 vol.% metallic phase after free sintering at  $1400^\circ\text{C}$ . The analysis was performed to evaluate the spatial distribution of the principal constituent elements and to assess the microstructural homogeneity of the composite after high-temperature consolidation. The obtained elemental maps confirm the presence of a multiphase microstructure composed of an  $\text{Al}_2\text{O}_3$  ceramic matrix and dispersed Cu-Cr metallic regions. The backscattered electron (BSE) micrograph reveals the presence of a bright, irregular metallic inclusion embedded within the darker alumina matrix. The strong contrast difference observed in the BSE image results from the significantly higher average atomic number of the metallic phases compared with the ceramic matrix. The metallic region exhibits a complex morphology with irregular boundaries and localized internal heterogeneity, suggesting partial redistribution of the metallic constituents during sintering. The elemental distribution map corresponding to aluminium demonstrates that Al is distributed homogeneously throughout the entire matrix region surrounding the metallic inclusion. A clear decrease in Al signal intensity is observed within the metallic particle itself, confirming that the bright inclusion does not belong to the alumina phase. Similarly, the oxygen map shows strong and uniform distribution across the ceramic matrix, while the metallic regions appear depleted in oxygen intensity. The combined Al and O distributions confirm that the continuous matrix phase consists predominantly of  $\text{Al}_2\text{O}_3$  and that no major disruption of the ceramic continuity occurred during sintering. The copper distribution map reveals that Cu is concentrated primarily within the upper bright region of the metallic inclusion. The Cu-rich area appears relatively compact and locally agglomerated, indicating limited diffusion of copper into the surrounding alumina matrix during thermal treatment. The high local intensity of the Cu signal suggests that copper remained predominantly in metallic form after sintering at  $1400^\circ\text{C}$ . The localized nature of the Cu distribution additionally indicates that the metallic phase did not form a continuous interconnected network within the composite. In contrast, the chromium map demonstrates a broader and more diffuse elemental distribution compared with copper. Chromium is detected not only within the metallic inclusion itself but also in the surrounding interfacial region adjacent to the ceramic matrix. This behaviour may indicate partial interfacial diffusion of chromium during high-temperature sintering. Possibly related

to its higher chemical affinity for oxygen and greater thermodynamic reactivity compared with copper, chromium may preferentially segregate near the ceramic–metal interface and contribute to improved interfacial bonding between the metallic and ceramic phases. The overlap of Cu and Cr rich regions observed in the combined elemental map confirms the coexistence of both metallic constituents within the same microstructural feature. However, the distributions are not completely homogeneous, indicating that complete alloy homogenization between Cu and Cr did not occur during sintering. This observation is consistent with the limited mutual solubility of Cu and Cr under equilibrium conditions and suggests that the metallic inclusions consist of locally heterogeneous Cu-rich and Cr-rich domains. Importantly, the EDS analysis did not reveal the presence of additional regions enriched in oxygen around the metallic particles that could indicate the formation of extensive copper or chromium oxide phases. Similarly, no distinct interfacial reaction layers with clearly differentiated chemical composition were identified within the spatial resolution limits of the applied technique. These observations remain consistent with the XRD results, which confirmed the presence exclusively of  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>, Cu, and Cr phases without detectable secondary reaction products. The elemental mapping additionally confirms the relatively good dispersion of the metallic phase within the ceramic matrix and the absence of large-scale elemental segregation throughout the analysed region. Although some local metallic agglomeration is visible, the overall microstructure remains relatively homogeneous considering the low metallic phase content and the processing route applied. The observed distribution suggests that sintering at 1400°C enabled effective consolidation of the composite while preserving the discrete character of the Cu-Cr metallic inclusions. The EDS results correlate well with the previously discussed densification and mechanical property data. The dense and chemically stable Al<sub>2</sub>O<sub>3</sub> matrix is responsible for the high hardness of the composite. The metallic inclusions likely contribute to increased fracture resistance through mechanisms such as crack deflection and localized stress accommodation. Furthermore, the apparent interfacial interaction associated particularly with chromium may enhance adhesion between the metallic inclusions and the alumina matrix, thereby improving the structural integrity of the composite.

It should be emphasized that the present SEM/EDS and XRD analyses do not provide direct evidence for the formation of continuous reaction layers or specific interfacial compounds at the Al<sub>2</sub>O<sub>3</sub>-metal interface. Although the EDS maps indicate a broader distribution of chromium in the vicinity of metallic inclusions and the SEM observations reveal the absence of extensive interfacial gaps, these findings should be interpreted as indirect indications of possible interfacial interactions rather than definitive proof of enhanced chemical bonding. Furthermore, no additional crystalline phases were detected by XRD within the detection limits of the technique. Therefore, the role of chromium in the investigated composites is discussed primarily on the basis of microstructural observations, densification behaviour, and previously reported literature concerning Cr-containing alumina-metal systems. Verification of possible nanoscale interfacial reaction products or diffusion layers would require higher-resolution characterization methods such as transmission electron microscopy (TEM) combined with local chemical analysis.

The relatively high hardness of the Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composites sintered at 1400°C ( $13.8 \pm 1.6$  GPa) is primarily associated with the formation of a highly densified and fine-grained alumina matrix characterized by very low residual porosity. The stereological analysis demonstrated that the average alumina grain size remained below 1  $\mu$ m, indicating that excessive grain growth was effectively suppressed during sintering. In ceramic materials, fine grain size generally increases resistance to localized plastic deformation and crack initiation due to the larger grain boundary area and reduced probability of critical flaw formation.

Furthermore, the nearly complete elimination of open porosity significantly contributed to the hardness increase, since pores act as local stress concentrators and reduce the effective load-bearing cross-section during indentation. Consequently, the combination of high relative density, limited residual porosity, and stable fine-grained microstructure provided favourable conditions for achieving relatively high hardness despite the presence of a metallic phase.

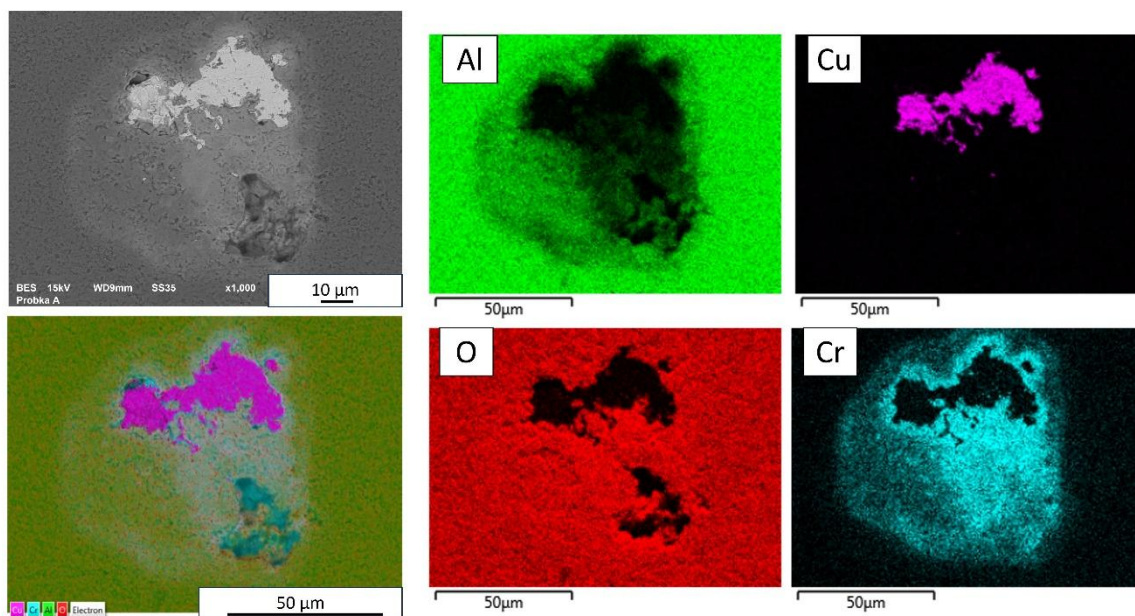


**Fig. 5.** SEM/EDS elemental distribution maps obtained for the  $Al_2O_3$ -Cu-Cr composites containing 2.5 vol.% metallic phase after free sintering at 1400°C

The obtained fracture toughness value ( $4.41 \pm 0.78 \text{ MPa}\cdot\text{m}^{0.5}$ ) indicates a moderate improvement in crack resistance compared with monolithic alumina, where fracture toughness typically remains within the range of approximately 3-4  $\text{MPa}\cdot\text{m}^{0.5}$ . The improved fracture toughness may be associated with crack deflection and local deformation effects related to the metallic phase. Several microstructural toughening mechanisms may contribute simultaneously to the enhanced crack resistance. First, the ceramic-metal interfaces promote crack deflection, forcing propagating cracks to change direction locally and thereby increasing the total fracture path and the energy required for crack propagation. Second, the metallic regions may partially redistribute local stresses, possibly related to their lower elastic modulus and limited plastic deformability relative to the brittle alumina matrix. Additionally, localized crack bridging effects may occur in regions containing elongated or irregular metallic inclusions, where the metallic phase can temporarily retard crack opening during fracture. Differences in thermal and elastic properties between the ceramic and metallic phases may influence local stress distribution during deformation.

The role of chromium within the metallic phase may be important for the mechanical response of the composites; however, the present results do not allow direct confirmation of the underlying mechanisms. The broader chromium distribution observed in the EDS maps suggests possible localized diffusion or segregation effects during sintering. Based on literature reports concerning Cr-containing alumina composites, chromium may contribute to improved interfacial compatibility and more stable metallic phase distribution because of its higher affinity for oxygen and stronger interaction with oxide surfaces compared with copper. Nevertheless, no direct evidence of interfacial reaction products or chemically distinct bonding layers was identified in the present study. Therefore, any beneficial effect of

chromium should be regarded as a plausible interpretation supported by the observed microstructural features and previous literature rather than as a conclusively demonstrated mechanism.

At the same time, several microstructural factors limit further improvement of both hardness and fracture toughness. Despite the high densification achieved at 1400°C, the composite still contains discrete metallic inclusions and isolated residual heterogeneities that may locally reduce hardness due to the significantly lower intrinsic hardness of Cu compared with Al<sub>2</sub>O<sub>3</sub>. The presence of metallic inclusions may locally delay unstable crack propagation. The metallic phase, therefore, acts mainly as a local toughening modifier rather than as a continuous reinforcing network. Complete homogenization of the Cu and Cr constituents was also not achieved, as confirmed by SEM/EDS analysis, indicating the persistence of locally heterogeneous metallic regions. These heterogeneities may promote local stress concentration and contribute to the relatively large standard deviation observed for fracture toughness measurements.

It should be emphasized that the fracture toughness values reported in the present work were obtained using the indentation fracture method and should therefore be interpreted with appropriate caution. Although this technique is widely used for comparative evaluation of ceramic materials, the calculated values are strongly influenced by factors such as crack morphology, residual stresses, surface preparation, indentation load, and the empirical model used for data analysis. Consequently, the obtained  $K_{IC}$  values should be regarded primarily as approximate indicators of the relative crack resistance of the investigated composites rather than as absolute material properties. The main significance of the present results lies in the comparison of the fracture response with that reported for similar alumina-based ceramic-metal composites and in identifying general trends associated with the developed microstructure. More rigorous determination of fracture toughness would require standardized methods based on pre-cracked specimens, such as single-edge notched beam (SENB) or chevron-notch testing.

Further improvement of fracture toughness would likely require optimization of several parameters simultaneously, including metallic phase content, particle size distribution, interfacial engineering, and control of residual stresses generated during cooling. However, increasing the metallic fraction excessively could adversely affect the hardness, stiffness, and dimensional stability of the composite due to the softer nature of the metallic constituents. Therefore, the obtained mechanical properties reflect a compromise between maintaining the intrinsic hardness and structural rigidity of the alumina matrix while introducing localized metallic toughening mechanisms through the Cu-Cr phase.

The compression curve of the composite (Fig. 6) shows a characteristic brittle response typical of ceramic-based materials. The load increased almost linearly with piston displacement in the initial stage, indicating elastic deformation and stable stress transfer within the composite structure. With increasing displacement, a slight reduction in the slope of the curve was observed, suggesting the initiation of microcracks and local stress redistribution at the ceramic-metal interfaces. The maximum load reached 4.19 kN at a displacement of 0.24 mm, corresponding to a compressive strength of 29.76 MPa. After reaching the maximum value, a sudden drop in load occurred, indicating unstable crack propagation and catastrophic failure of the specimen. The fracture behaviour suggests predominantly brittle failure controlled by rapid crack propagation through the alumina matrix. However, the slight nonlinearity before fracture and limited post-peak load retention indicate that the Cu-Cr metallic phase contributed to local toughening mechanisms. The fracture process was therefore probably governed by a combination of transgranular cracking, interfacial decohesion, and crack deflection around metallic particles. The obtained results are

consistent with literature reports concerning Al<sub>2</sub>O<sub>3</sub>-Cu-Cr and Al<sub>2</sub>O<sub>3</sub>-Cu-Ni composites. Previous studies demonstrated that the addition of chromium or nickel improves the microstructure and mechanical behaviour of Al<sub>2</sub>O<sub>3</sub>-Cu composites by reducing copper leakage during liquid-phase sintering and improving metallic phase distribution within the ceramic matrix [50]. Compared with Al<sub>2</sub>O<sub>3</sub>-Cu-Ni composites produced by uniaxial pressing, the present material exhibited lower mechanical performance and retained a more brittle character, which is associated with the relatively low amount of metallic reinforcement. In Al<sub>2</sub>O<sub>3</sub>-Cu-Ni systems containing 5–15 vol.% metallic phase, higher densification and improved crack-bridging effects were observed due to the formation of Cu-Ni solid solutions during sintering [51]. A similar tendency was reported for gradient Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composites manufactured by centrifugal slip casting, where chromium addition improved the ceramic–metal interface and limited excessive copper outflow during sintering [52]. In those materials, improved crack deflection and better metallic phase distribution contributed to enhanced structural integrity. Furthermore, studies on hybrid Al<sub>2</sub>O<sub>3</sub>-Cu-Me composites confirmed that introducing a second metallic component, including Cr or Ni, positively affects compressive strength and limits the formation of large metallic agglomerates compared with binary Al<sub>2</sub>O<sub>3</sub>-Cu composites [50]. Therefore, despite the relatively low compressive strength obtained in the present study, the compression behaviour indicates that even a small Cu-Cr addition can partially modify crack propagation mechanisms and improve the overall integrity of alumina-based composites.



**Fig. 6.** Load-displacement curves were obtained during uniaxial compression testing of Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composite systems containing a 2.5 vol.% metal phase

The strain distribution maps presented in Figure 7 provide important insight into the deformation and failure behaviour of the Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composite containing 2.5 vol.% metallic phase during uniaxial compressive loading. The digital image correlation (DIC) analysis provides important insight into the deformation and failure mechanisms of the Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composites and constitutes one of the key aspects of the present study because it enables direct visualization of strain evolution preceding catastrophic fracture. Unlike conventional mechanical testing, which provides only global load–displacement information, the DIC technique allows spatially resolved analysis of local strain heterogeneities and damage development within the composite during compressive loading. Consequently, the DIC results establish a direct relationship between the microstructural architecture of the ceramic-metal composite and its deformation behaviour under external stress. The strain maps reveal that deformation in the investigated composites evolves progressively from an initially relatively homogeneous strain distribution toward highly localized deformation zones as the applied load increases. At low deformation levels, the effective strain field remains

comparatively uniform across the specimen surface, although minor local fluctuations are already detectable. These local heterogeneities most likely originate from microstructural features such as residual pores, ceramic grain boundaries, metallic inclusions, and local stiffness differences between the alumina matrix and the Cu-Cr metallic phase. The early appearance of nonuniform strain fields demonstrates that deformation within the composite is intrinsically heterogeneous even before macroscopic damage initiation occurs. With increasing compressive stress, the strain localization process becomes progressively more pronounced. Distinct high-strain regions begin to form and subsequently evolve into narrow localization bands characterized by significantly elevated effective strain values compared with the surrounding matrix. The progressive intensification of these localized regions indicates the accumulation and coalescence of microdamage within the composite structure. Importantly, the deformation process does not remain spatially uniform during loading but instead transitions toward increasingly heterogeneous behaviour prior to catastrophic failure. This observation is characteristic of brittle ceramic systems in which local stress concentrations govern crack initiation and propagation processes. The onset of unstable deformation can be associated with the formation of a dominant strain concentration band visible shortly before final fracture. The DIC maps demonstrate that this localization band develops progressively rather than instantaneously, suggesting that damage accumulation occurs continuously during loading. The localization region becomes narrower and more intense as the applied displacement increases, indicating that the material gradually loses its ability to redistribute stresses uniformly throughout the microstructure. The evolution of the strain field, therefore, reflects the transition from stable microcrack formation toward unstable crack propagation leading to catastrophic collapse. An important observation is the strong correlation between the regions exhibiting the highest effective strain and the final fracture trajectory observed after failure. The dominant localization bands identified by DIC correspond closely to the eventual crack propagation path, confirming that strain concentration zones act as preferential sites for macroscopic fracture initiation. In most analysed specimens, the localization bands propagated approximately parallel to the loading direction, although local deviations in orientation were also observed. These deviations are likely associated with microstructural heterogeneities and interactions between propagating cracks and metallic inclusions dispersed within the alumina matrix. The finite width of the localization zones additionally suggests that fracture initiation is not confined to a single sharp crack at the early stages of deformation, but instead involves the accumulation of distributed microdamage within a localized deformation region. The presence of Cu-Cr metallic inclusions appears to influence the strain localization kinetics and may partially delay the onset of unstable deformation. The metallic phase locally modifies the stress distribution within the composite, possibly related to its lower elastic modulus and limited plastic deformability relative to the brittle alumina matrix. As a consequence, metallic inclusions may act as local stress-relaxation regions that reduce the intensity of stress concentrations at crack tips and retard rapid crack propagation. This effect is particularly important during the intermediate stages of deformation, where the DIC maps reveal progressive rather than instantaneous localization development. The metallic phase, therefore, contributes to limited stabilization of the deformation process by promoting local stress redistribution and partial energy dissipation before catastrophic failure occurs. At the same time, the DIC analysis confirms that the investigated composites still exhibit predominantly brittle deformation behaviour characteristic of alumina-based materials. Once the strain localization reaches a critical intensity, unstable crack propagation occurs rapidly and leads to abrupt specimen collapse with minimal post-peak deformation. The progressive evolution of localization bands suggests that the metallic inclusions may influence strain redistribution during loading. The

deformation process remains governed primarily by crack initiation, crack coalescence, and localized damage accumulation within highly stressed regions. The DIC analysis significantly expands the interpretation of the mechanical behaviour of the Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composites by demonstrating that failure is preceded by progressive spatial evolution of strain heterogeneities rather than by instantaneous uniform fracture. The results confirm that the deformation behaviour of the investigated composites is strongly controlled by microstructural heterogeneity and by interactions between the ceramic matrix and dispersed metallic inclusions. Consequently, the DIC observations provide valuable mechanistic insight into the relationship between microstructure, strain localization, and fracture evolution in ternary ceramic-metal composites processed by conventional powder metallurgy.

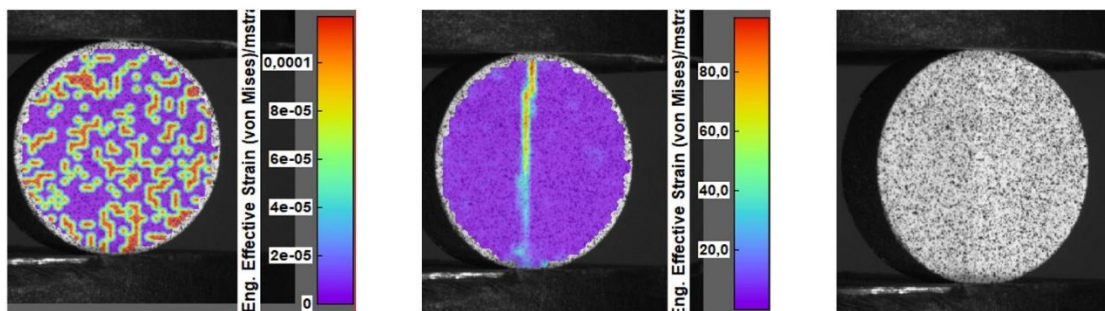


**Fig. 7.** Representative effective strain distribution maps obtained by digital image correlation during uniaxial compressive loading of Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composites containing 2.5 vol.% metallic phase fabricated by uniaxial pressing and free sintering at 1400°C. The maps illustrate the progressive evolution of local strain fields and the development of deformation localization preceding catastrophic fracture. Increasing strain concentration leads to the formation of a dominant localized deformation band, indicating the initiation and propagation of damage within the ceramic-metal composite structure

Figure 8 presents a representative SEM micrograph of the fracture surface of the Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composite containing 2.5 vol.% metallic phase after compressive failure, together with the stereological analysis of the alumina grain size distribution determined from quantitative image analysis. The observations provide important information regarding the fracture behaviour of the composite and the microstructural stability of the alumina matrix after free sintering at 1400°C. The fracture surface micrograph reveals a dense and relatively homogeneous ceramic microstructure composed predominantly of fine equiaxed alumina grains. The observed morphology is characteristic of well-sintered alumina-based materials consolidated under conditions promoting effective densification while suppressing excessive grain growth. The fracture surface exhibits predominantly brittle fracture features, which are typical for Al<sub>2</sub>O<sub>3</sub> ceramics subjected to compressive loading. Numerous fractured grains with sharp-edged cleavage-like surfaces are visible, indicating that crack propagation occurred mainly through the ceramic grains and along selected grain boundaries. The absence of extensive intergranular decohesion suggests relatively strong grain boundary cohesion within the alumina matrix. The alumina grains exhibit relatively regular morphology with only limited anisotropy in grain shape. Most grains appear tightly packed with good intergranular contact, confirming the high densification level previously identified by density and porosity measurements. Only isolated microvoids are visible on the fracture surface, indicating that residual porosity remained very low after sintering at 1400°C. Such a dense microstructure is beneficial for mechanical performance because residual pores in brittle ceramics frequently act as preferential crack initiation sites during loading. The grain size distribution histogram demonstrates that the microstructure is dominated by submicrometer alumina grains. The equivalent grain diameter determined by stereological analysis reached:  $d_2 = 0.87 \mu\text{m} \pm 0.28 \mu\text{m}$ . The obtained average grain size indicates that the sintering process enabled effective

densification without inducing excessive grain coarsening of the alumina matrix. The relatively low standard deviation ( $\pm 0.28 \mu\text{m}$ ) indicates moderate dispersion of grain sizes and suggests a comparatively uniform microstructure throughout the analysed material. The histogram exhibits a unimodal distribution with the highest frequency of grains located approximately within the range of  $0.65\text{--}0.95 \mu\text{m}$ . The modal grain size appears near approximately  $0.75\text{--}0.85 \mu\text{m}$ , where the histogram reaches its maximum frequency of nearly 8-9%. This indicates that the majority of alumina grains remained concentrated within a narrow size interval, reflecting stable grain growth kinetics during sintering. The relatively symmetric central region of the histogram suggests that normal grain growth dominated during thermal treatment. At the same time, the histogram displays slight positive skewness toward larger grain diameters. Although most grains are below approximately  $1.2 \mu\text{m}$ , a limited population of coarser grains extending locally up to approximately  $2.1 \mu\text{m}$  is also visible. However, these larger grains constitute only a very small fraction of the total analysed population. Importantly, no secondary maxima or bimodal distribution features are observed. This indicates that abnormal grain growth did not occur during sintering and that the microstructure remained microstructurally stable despite the elevated sintering temperature of  $1400^\circ\text{C}$ . The statistical significance of the obtained grain size distribution is high due to the large number of analysed grains used during stereological evaluation. As stated previously in the methodology section, at least 1000 individual alumina grains were measured for each material condition. Such a large statistical population substantially reduces random measurement uncertainty and minimizes the influence of local microstructural heterogeneities on the final distribution. Consequently, the calculated mean grain size and standard deviation can be considered representative of the entire composite microstructure rather than for isolated local regions. The relatively smooth and continuous character of the histogram additionally indicates the reliability of the image analysis procedure and indicates that the selected equivalent circle diameter approach was appropriate for describing the irregular grain geometries visible on the fracture surfaces. The absence of discontinuities or artificial clustering within the histogram suggests good reproducibility of the stereological measurements. From a microstructural perspective, the obtained fine-grained alumina matrix is highly beneficial for the mechanical behaviour of the investigated composites. Fine alumina grains generally contribute to increased hardness and improved resistance to crack initiation due to the larger total grain boundary area and reduced probability of critical flaw formation. Simultaneously, the absence of exaggerated grain growth helps maintain structural uniformity and limits the formation of preferential fracture paths. The observed grain size distribution, therefore, correlates well with the relatively high hardness ( $\sim 13.8 \text{ GPa}$ ) and the stable compressive behaviour of the  $\text{Al}_2\text{O}_3\text{-Cu-Cr}$  composites sintered at  $1400^\circ\text{C}$ . The fracture surface observations and quantitative stereological analysis confirm that the applied processing route, uniaxial pressing followed by free sintering at  $1400^\circ\text{C}$ , enabled the fabrication of highly densified  $\text{Al}_2\text{O}_3\text{-Cu-Cr}$  composites characterized by a stable fine-grained alumina matrix, limited residual porosity, and statistically homogeneous grain size distribution.

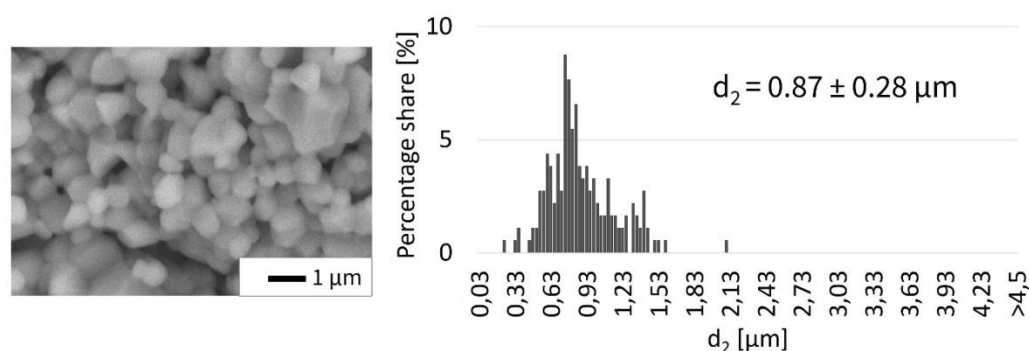


**Fig. 8.** Representative fracture surface micrograph of an Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composite after compressive failure, together with an analysis of the alumina grain size

## SUMMARY

The present study introduces several important scientific and technological innovations in the field of ceramic-metal composites, particularly within alumina-based multiphase systems. The research advances current knowledge not only through the development of a previously insufficiently explored Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composite system, but also through a comprehensive analysis of the relationships between processing conditions, microstructural evolution, densification mechanisms, and mechanical behaviour.

The primary innovation of the work lies in the fabrication and characterization of ternary Al<sub>2</sub>O<sub>3</sub>-Cu-Cr composites containing a low metallic phase fraction (2.5 vol.%) produced by uniaxial pressing followed by pressureless free sintering in a reducing atmosphere. While previous studies have predominantly focused on binary Al<sub>2</sub>O<sub>3</sub>-Cu or Al<sub>2</sub>O<sub>3</sub>-Cr systems, the simultaneous incorporation of Cu and Cr into an alumina matrix remains insufficiently investigated. The presented research, therefore, fills an important scientific gap concerning the synergistic interaction between copper and chromium during sintering and microstructure formation in alumina-based composites.

A significant novelty of the study is the demonstration that chromium addition effectively modifies the ceramic-metal interfacial behaviour within the Al<sub>2</sub>O<sub>3</sub>-Cu system. The results indicate that Cr contributes to improved interfacial compatibility, enhanced densification, and stabilization of the metallic phase distribution during sintering. SEM and EDS analyses revealed the formation of chemically stable and well-bonded ceramic-metal interfaces without detectable formation of undesirable oxide or spinel phases, even after sintering at 1400°C. This finding is particularly important because poor wettability and interfacial instability are major limitations in conventional Al<sub>2</sub>O<sub>3</sub>-Cu composites.

Another innovative aspect of the research is the identification of the strong influence of sintering temperature on densification efficiency and microstructural stabilization of the Al<sub>2</sub>O<sub>3</sub>-Cu-Cr system. The study demonstrated that increasing the sintering temperature from 1200°C to 1400°C enabled a transition from insufficiently consolidated porous structures to nearly fully dense composites with a relative density approaching 98% and residual open porosity below 0.1%. Simultaneously, shrinkage behaviour, pore elimination mechanisms, and phase stability were systematically correlated with thermal processing conditions, providing valuable insight into the sintering kinetics of ternary ceramic-metal systems.

The work also introduces an important methodological contribution through the combination of conventional mechanical characterization with full-field digital image correlation (DIC) analysis during compressive loading. The application of DIC enabled direct

visualization of strain localization and damage evolution in the ceramic-metal composite prior to catastrophic fracture. This approach provided new insight into the deformation mechanisms of brittle alumina-based composites reinforced with discrete metallic inclusions and demonstrated the role of the Cu-Cr phase in local stress redistribution and crack stabilization. An additional innovation of the study is the successful fabrication of a highly densified fine-grained alumina matrix without abnormal grain growth despite high-temperature sintering at 1400°C. Quantitative stereological analysis confirmed that the composites maintained a stable submicrometer grain structure with an average equivalent grain diameter of approximately 0.87  $\mu\text{m}$ . The composites exhibited an estimated indentation fracture toughness of  $4.41 \pm 0.78 \text{ MPa}\cdot\text{m}^{0.5}$ , indicating moderate improvement in crack resistance compared with monolithic alumina.

The research further contributes to the field by demonstrating that even a relatively low metallic phase content can significantly influence crack propagation behaviour and mechanical integrity in alumina-based composites. The Cu-Cr metallic regions acted as local toughening sites promoting crack deflection, stress redistribution, and limited energy dissipation mechanisms while preserving the intrinsic hardness and structural stability of the alumina matrix.

From a technological perspective, the study is innovative because it demonstrates the feasibility of producing advanced  $\text{Al}_2\text{O}_3$ -Cu-Cr composites using an industrially scalable and economically attractive processing route based on uniaxial pressing and free sintering. The ability to obtain highly dense and chemically stable ceramic-metal composites without the need for advanced pressure-assisted sintering techniques represents an important advantage for future industrial implementation.

The presented research significantly expands current understanding of ternary alumina-based ceramic-metal composites by establishing clear relationships between powder morphology, sintering behaviour, interfacial phenomena, microstructural stability, and mechanical performance. The obtained results provide a scientific foundation for the future design of multifunctional ceramic-metal materials with improved structural reliability and potential applications in wear-resistant, thermally stable, and mechanically demanding engineering environments.

## ACKNOWLEDGEMENT

The study was made possible thanks to the funds allocated by the Engineering Warsaw University of Technology within the framework of the research statute work - Project Manager: Ph.D. Eng. Justyna Zygmuntowicz.

### *Conflict-of-interest statement*

The authors certify that they have no affiliations with or involvement in any organization or entity with any financial interest, or non-financial interest in the subject matter or materials discussed in this manuscript.

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