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Al_2O_3-Cu-Mo HYBRID COMPOSITES: FABRICATION, MICROSTRUCTURE, PROPERTIES

The work investigated the influence of the share of metallic components on the microstructure and selected properties of Al_2O_3-Cu-Mo composites. Commercial powders were used to produce the composite samples. The composites were obtained by the slip casting method. Three series of composites with a different volumetric composition of metals in the total content of the metallic phase were obtained: Series I - contained 7.5 vol.% Cu - 7.5 vol.% Mo, Series II - contained 10 vol.% Mo - 5 vol.% Cu and Series III - contained 12 vol.% Mo - 3 vol.% Cu. All the series contained 15 vol.% metal particles with respect to the total solid phases. Rheological analysis showed that the slurries used to make the composites were shear thinning fluids. The X-ray analysis showed that regardless of the volume content of copper in the suspensions used to form the composite, all the composites after sintering were characterized by the presence of three phases: Al_2O_3, Cu and Mo. It was found that the microstructure in all the series is characterized by homogeneous distribution of the metal particles. All the samples were characterized by high porosity, which resulted in their low relative density. The volume fractions of molybdenum and copper in the composite slightly do affect the hardness and fracture toughness of the composite. The obtained hardness results indicate that increasing the molybdenum content in the composites causes an insignificant increase in the hardness of the samples.

Keywords: alumina, slip casting, hybrid composites, Al_2O_3-Cu-Mo

HYBRYDOWE KOMPOZYTY Al_2O_3-Cu-Mo: WYTWARZANIE, MIKROSTRUKTURA, WŁAŚCIWOŚCI

W pracy zbadano wpływ udziału komponentów metalicznych na mikrostrukturę i wybrane właściwości kompozytów Al_2O_3-Cu-Mo. Do wykonania próbek użyto komercyjnie dostępnych proszków: Al_2O_3, Cu i Mo. Kompozyty otrzymano metodą odlewania z gęstwy. Wytworzono trzy serie kompozytów o różnym składzie objętościowym metali w całkowitej zawartości fazy metalicznej: Seria I zawierała 7,5% obj. Cu - 7,5% obj. Mo, Seria II zawierała 10% obj. Mo - 5% obj. Cu i Seria III - zawierała 12% obj. Mo - 3% obj. Cu. Wszystkie serie zawierały 15 % obj. cząstek metalu w odniesieniu do całkowitej fazy stałej. Badania reologiczne wykazały, że masy lejne użyte do wytwarzania kompozytów były cieczami newtonowskimi rozrzedzanymi ścinaniem. Badanie składu fazowego wykazało, iż niezależnie od zawartości objętościowej miedzi w masie lejnej użytej do wytworzenia kompozytu wszystkie kompozyty po spiekaniu cechowała obecność trzech faz: Al_2O_3, Cu i Mo. Stwierdzono, że mikrostruktura we wszystkich seriach charakteryzuje się jednorodnym rozkładem cząstek metalu. Wszystkie próbki charakteryzowała wysoka porowatość, co skutkowało ich niskim zagęszczeniem. Udział objętościowy molibdenu i miedzi w kompozycie wpłynął na jego twardość i odporność na kruche pękanie. Uzyskane wyniki twardości wskazują, że zwiększenie zawartości molibdenu powoduje niewielki wzrost twardości próbek.

Słowa kluczowe: tlenek glinu, odlewanie z gęstwy, kompozyty hybrydowe, Al_2O_3-Cu-Mo

INTRODUCTION

Ceramic materials are strongly differentiated, whose properties depend on the type of chemical components from which they were manufactured. Ceramic materials are characterized by high mechanical properties such as hardness or abrasion resistance. In addition, this group of materials is characterized by a high melting temperature and chemical resistance. The disadvantage of these materials is their low fracture toughness, which is a limitation in the wider use of ceramics [1]. The low cracking resistance of ceramic materials can lead to the failure of components working under load and also limits the possibility of using technical ceramics in structural applications. Therefore, in recent years scientists have been looking for solutions to improve the fracture toughness of these materials [2, 3]. One of the concepts is to produce ceramic matrix composites. Technical ceramics, glass or carbon are most commonly used as the matrix in ceramic-based composites [4]. Ceramic-metal composites can be used as constructional and functional materials because the combination of ceramics and metal gives the possibility to fabricate materials with a wide range of properties. The main reason to manufacture ceramic matrix composites is to increase crack resistance while maintaining the high strength proper-

ties that are characteristic of ceramic materials [5]. Additionally, due to the combination of the properties of ceramics and metal, the functional properties of the materials such as thermal, electrical or magnetic can be obtained [6].

One of the interesting groups of composites from the ceramics metal system is hybrid composites. They are composites that emerged as a result of the constant strive to obtain materials with increasingly better properties. Based on the literature, hybrid composites are materials that consist of a matrix and several types of reinforcement [1]. The most commonly known hybrid composites are materials with a polymer matrix and an addition of various types of the fibers [7-12]. One of the main reasons to produce these materials is to obtain the greatest possible toughness and strength. The combination of different fibers allows one to use the properties and advantages of individual components [7-12]. Another type of hybrid materials is laminates made of layers of various materials connected to each other. In recent years, fiber metal laminates (FML) have been the subject of many studies. They are laminates made of thin metal plates and a polymer fiber-reinforced composite bonded with adhesive. They are characterized by a lower specific gravity, better impact strength, damage tolerance and resistance to corrosion than metal [8-12]. Furthermore, in the literature on the subject, there is increasingly more research concerning composites consisting of two ceramic phases and a metallic phase. Exemplary research concerns a composite such the Al_2O_3-ZrO_2 ceramic matrix with the addition of metal particles [13] and Al_2O_3/TiC/Co systems [14]. These experiments reveal that the synergistic effect of reinforcing phases allows materials to be obtained characterized by new and innovative properties compared to single-phase ceramics. Investigations of the fabrication methods as well as the characteristics of hybrid composites are still subjects of basic research. Therefore, the authors of the present article believe that it would be interesting to create and examine new Al_2O_3-Cu-Mo composites. Both Al_2O_3-Cu and Al_2O_3-Mo composites have interesting properties and are the subject of many works [15-22]. The greatest disadvantage of Al_2O_3-Cu composites are the problems associated with their fabrication. The poor wettability of the corundum matrix with liquid copper does not allow the use of classical sintering. The chemical composition of Al_2O_3-Cu composites is difficult to control due to the loss of copper that flows to the surface of the samples. One solution to solve this problem may be to create a hybrid composite by adding molybdenum as a third component. It can be assumed that during the sintering of Al_2O_3-Cu-Mo composites no new phase will appear, which is confirmed by the Cu-Mo equilibrium system [23]. The addition of molybdenum may improve the wettability of the metal phase during the manufacturing of these composites.

Therefore, the aim of this work is to determine the influence of the common ratio of metallic elements on the microstructure, phase structure and the basic properties of Al_2O_3-Cu-Mo composites.

MATERIALS AND METHODS

Commercial powders were used as the starting powders: Al_2O_3, Cu and Mo. The ceramic powder used in the research was Al_2O_3 A16SG (Almatis) of an average particle size equal to 0.5 ±0.1 μm and density of 3.90 g/cm^3. Cu (Sigma Aldrich) was used as the metallic powder: of the average particle size 13.20 ±4.54 μm and density of 8.94 g/cm^3 and Mo (Createc) of the average particle size 21.59 ±5.69 μm and density of 10.28 g/cm^3. The metal powders are characterized by a high purity equal to 99.999%. Figure 1 shows the morphology of the starting powders. The SEM micrograph of the Mo powder shows an irregular morphology. It was found that the ceramic powder has a tendency to form agglomerates. X-ray diffraction patterns of the starting materials are shown in Figure 2. X-ray analysis of the starting materials confirmed the single-phase structure of the used powders. Observation of the XRD pattern for Al_2O_3 (Fig. 2a) reveals that the alumina powder consists of alpha phases [24].

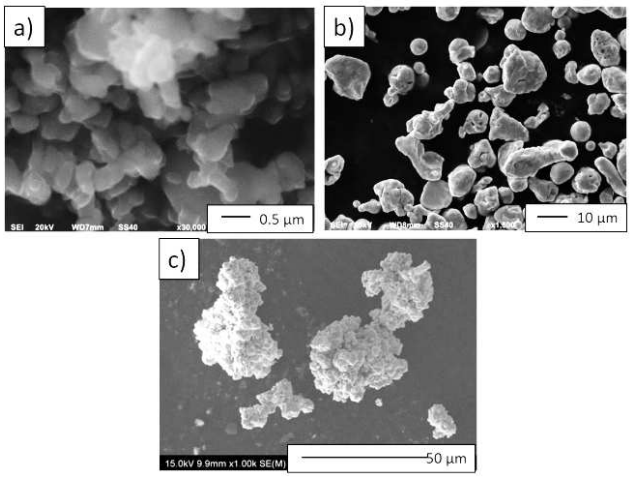

Fig. 1. SEM micrographs of starting powders: a) Al_2O_3, b) Cu, c) Mo

Rys. 1. Zdjęcia SEM przedstawiające wyjściowe proszki: a) Al_2O_3, b) Cu, c) Mo

In the research part, ceramic water-based suspensions with a 50 vol.% solid content and 15 vol.% metal powders with respect to the total solid volume were prepared. Three series of samples differing in the share of copper and molybdenum were prepared: Series I contained 7.5 vol.% Cu - 7.5 vol.% Mo, Series II contained 10 vol.% Mo - 5 vol.% Cu and Series III 12 vol.% Mo - 3 vol.% Cu. The volume of metal was calculated with respect to the total metal volume content in the suspensions. In the experiment, deionized Milli-Q water was used as the solvent. Duramax D3005 was used as the dispersant in the slurries. The suspensions included 1.5 wt.% of the dispersant with respect to the total solid content weight.

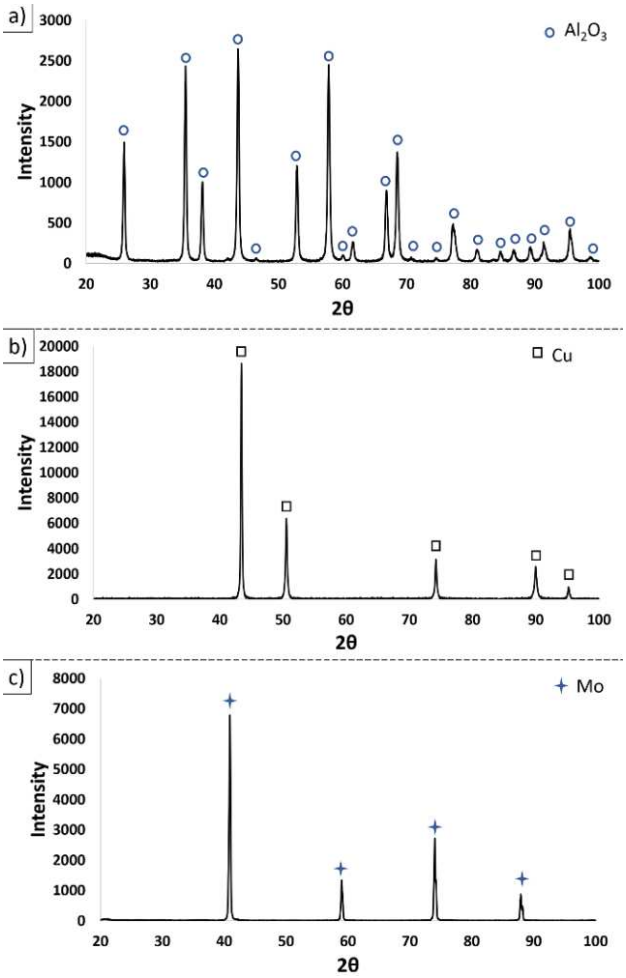

Fig. 2. XRD patterns of starting materials: a) Al_2O_3, b) Cu, c) Mo

Rys. 2. Dyfraktogramy wyjściowych proszków: a) Al_2O_3, b) Cu, c) Mo

The rheological properties of the suspensions were analysed by using a rheometer (Anton Paar). The viscosity was measured as a function of shear rate when increased from 0.1 to 260 s^{-1} and back to 0.1 s^{-1}. XRD scans were made using a Rigaku MiniFlex II diffractometer with Cu Kα radiation. The samples were scanned from 20° to 100° at the scan rate of 0.1°/min. The lattice parameters were determined using the Cohens method and least squares method [25-31].

Selected physical properties of the obtained samples were examined using the Archimedes method. The microstructure observations were performed on a scanning electron microscope (SEM Hitachi TM 1000). The hardness of the composite was measured by the Vickers method on a polished sample surface at a load of 196 N with 15 s holding time. For each series, a minimum of 20 indentations was made. The fracture toughness was calculated based on the Niihara equation for $0.25 < l/a < 2.5$ [32, 33]:

$$K_{Ic} = 0.018 \cdot HV^{0.6} \cdot E^{0.4} \cdot 0.5d \cdot l^{-0.5}$$

where: H - Vickers hardness [GPa], E - Young's modulus [GPa], d - diagonal of the Vickers indentations [mm], l - average crack length [mm] [32, 33].

RESULTS AND DISCUSSION

The rheological properties play a key role during the formation of samples in the slip casting technique. Figure 3 shows the viscosity curves of the prepared composite suspensions. It should be noted that all the analysed slurries were shear thinning fluids. This is very beneficial when casting the suspensions in a porous mould. For the Series I, it was found that the slurry has a maximal viscosity equal to 4.36 Pa·s at a minimal shear rate of 1.29 s^{-1}. Moreover, in the case of Series I it was observed that when the shear rate increases to 260 s^{-1} the viscosity decreases to about 0.113 Pa·s. For Series II it was established that the slurry has a maximal viscosity equal to 4.7 Pa·s at a minimal shear rate of 1.29 s^{-1}; it was also observed that when the shear rate increases to 260 s^{-1} the viscosity decreases to about 0.128 Pa·s. The results of the rheological property tests reveal that the obtained values for Series II are slightly higher than the values for Series I and III. Figure 5c reveals that the slurry for Series III is characterized by a slightly lower viscosity, 3.92 Pa·s at a shear rate of 1.29 s^{-1}, than other the suspensions. However, the differences between the individual series are not big enough to be able to have any influence on the produced composites.

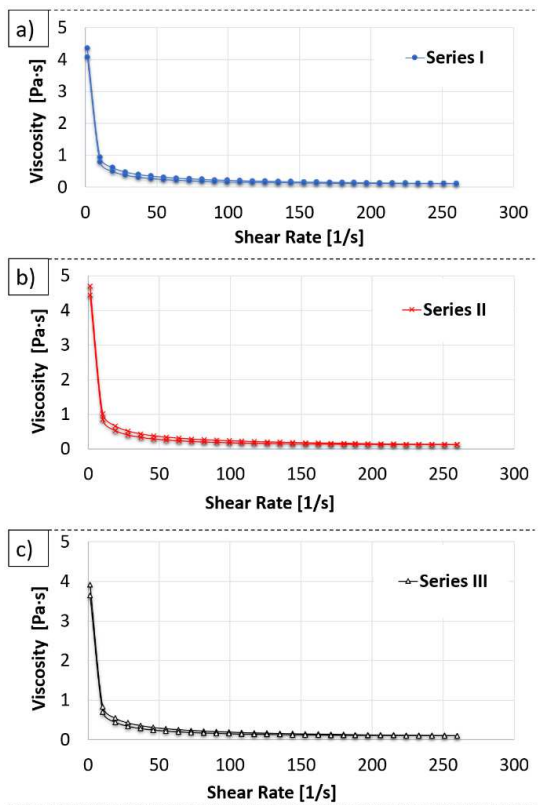

Fig. 3. Viscosity curves of composite slurries: a) Series I with addition of 7.5 vol.% Cu and 7.5 vol.% Mo, b) Series II with addition of 10 vol.% Mo and 5 vol.% Cu, c) Series III with addition of 12 vol.% Mo and 3 vol.% Cu

Rys. 3. Krzywe lepkości zawiesin kompozytowych: a) seria I z dodatkiem 7,5% obj. Cu i 7,5% obj. Mo, b) seria II z dodatkiem 10% obj. Mo i 5% obj. Cu, c) seria III z dodatkiem 12% obj. Mo i 3% obj. z Cu

The Al_2O_3-Cu-Mo composites were fabricated using the slip casting technique. The production scheme is shown in Figure 4. At the beginning, the dispersant and subsequently the ceramic (Al_2O_3) and metallic (Cu and Mo) powders were added to the water. Next, the slurries were mixed and degassed in a planetary centrifugal mixer THINKY ARE-250. The optimum parameters of the process were first established in a series of trials. The prepared suspension was poured into a plaster mould. Then the water from the slip was absorbed by the porous plaster mould and a green cast was obtained. Afterwards, the plaster mould with the sample inside was dried in a vacuum chamber at 25°C for 24 hours. The dried and shrunk composite was removed from the plaster mould. Then, the composite was sintered at 1400°C in an H_2/N_2 atmosphere.

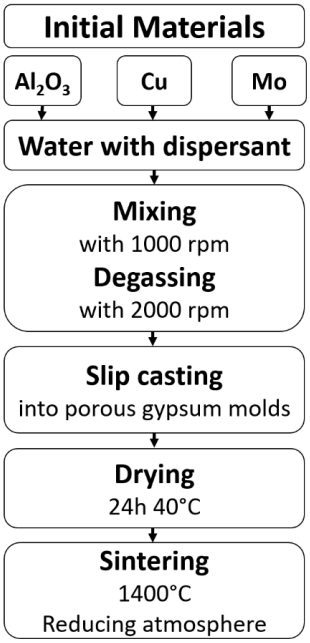

Fig. 4. Fabrication scheme of Al_2O_3-Cu-Mo composites

Rys. 4. Schemat wytwarzania kompozytów Al_2O_3-Cu-Mo

The samples obtained by the slip casting method are shown in Figure 5. Preliminary macroscopic observations of the samples from all the series show that the composites after sintering have no visible cracks on the surface.

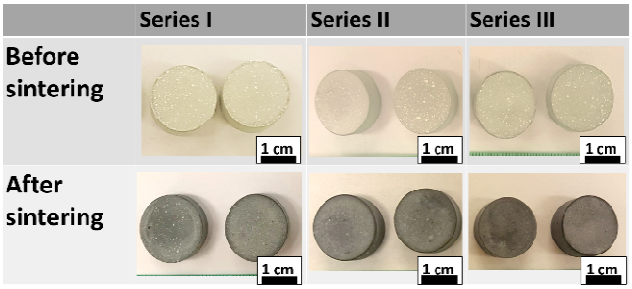

Fig. 5. Green body and sintered Al_2O_3-Cu-Mo composites obtained by slip casting

Rys. 5. Kompozyty Al_2O_3-Cu-Mo w stanie surowym i po spiekaniu uzyskane metodą odlewania z gęstwy

Based on the obtained X-ray diffractions, it was shown that all the series consist of three phases: Al_2O_3, Cu and Mo (Fig. 6). Due to the use of a reducing atmosphere during sintering, no presence of metal oxides or spinel phase ($MoAl_2O_4$) in the obtained materials was found. In order to check whether the change of additions of the metallic components affects the phase structure of the composites, the lattice parameters of the Cu and Mo powders were determined based on the Cohens and least squares analysis of the diffractograms [25-31]. Table 1 presents the results of the calculated lattice parameters. The obtained data show that the change of additions of the metallic components does not significantly affect the phase composition of the composites. It was found that slight changes in the lattice parameters for the Mo and Cu phases occurred in all the test series. These changes can be caused by changes difficult to unambiguously determine in the materials due to the temperature during the sintering process because the lattice parameters of pure substances undergo small changes depending on the conditions of the sintering process.

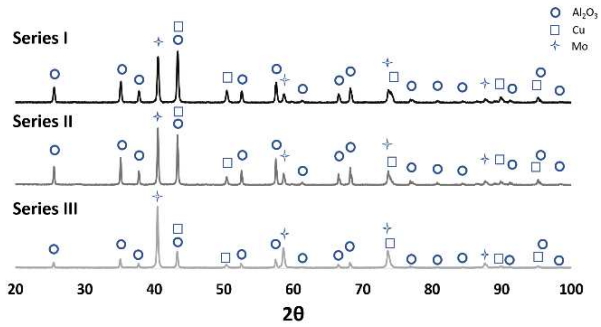

Fig. 6. X-ray diffraction patterns of three series of Al_2O_3-Cu-Mo composites after sintering in reducing atmosphere

Rys. 6. Dyfraktogramy uzyskane dla trzech serii kompozytów Al_2O_3-Cu-Mo spieczonych w atmosferze redukującej

TABLE 1. **Lattice parameters of phases detected in Al_2O_3-Cu-Mo composites**

TABELA 1. **Parametry sieci faz występujących w kompozycie Al_2O_3-Cu-Mo**

Al_2O_3-Cu-Mo composites	Lattice parameters [Å]			
	Calculated using Cohens method		Calculated using least squares method	
	Cu	Mo	Cu	Mo
	[Å]	[Å]	[Å]	[Å]
Series I (7.5 vol.% Cu and 7.5 vol.% Mo)	3.6118	3.1316	3.61129	3.1218
Series II (10 vol.% Mo and 5 vol.% Cu)	3.61025	3.147715	3.61022	3.14567
Series III (12 vol.% Mo and 3 vol.% Cu)	3.61577	3.14597	3.61421	3.14285

From these data, it can be concluded that irrespective of the amount of copper addition in the suspensions

for the prepared composites, the lattice parameter for Mo was from 3.1218 Å to 3.147715 Å, whereas for Cu it was from 3.61022 Å to 3.61577 Å. It can be assumed that the differences in the lattice parameters for Mo and Cu may result from the stress difference in the composites.

Selected physical properties of the sintered specimens were measured by the Archimedes method. Series I specimens with 7.5 vol.% Cu and 7.5 vol.% Mo metallic phase had the highest relative density among the analysed samples, equal to 85.51%. It was found that the lowest relative density of 77.09% was obtained for Series III with 12 vol.% Mo and 3 vol.% Cu. The experimental studies have shown that changing the proportion of copper and molybdenum in the metallic phase caused changes in the efficiency of the densification process. It was observed that increasing the amount of molybdenum in the metallic phase results in a decrease in sample density. Lower linear and volume shrinkage was also observed. The research revealed that the higher content of molybdenum in the metallic phase caused an increase in the number of open pores in the material, which resulted in its higher absorptivity. A higher proportion of molybdenum with a particle size larger than the copper particles could have influenced the lower density of these samples in the green body state, which in turn resulted in the lower relative density of these samples after the sintering process. It can be stated that during the sintering process with the participation of Mo, it is a solid state process for the Mo particles (melting point 2623°C), and this is explains the presence of lower density and higher porosity for the sintered composites with a greater amount of molybdenum.

TABLE 2. **Selected physical properties of Al_2O_3-Cu-Mo composites after sintering process**

TABELA 2. **Wybrane właściwości fizyczne kompozytów Al_2O_3-Cu-Mo po procesie spiekania**

Al_2O_3-Cu-Mo composites	Relative density [%]	Absorptivity [%]	Open porosity [%]	Linear shrinkage [%]	Volume shrinkage [%]
Series I (7.5 vol.% Cu and 7.5 vol.% Mo)	85.51 ±1.25	4.1 ±0.21	15.79 ±1.21	10.36 ±0.14	27.69 ±1.24
Series II (10 vol.% Mo and 5 vol.% Cu)	80.83 ±1.65	4.23 ±0.64	16.46 ±1.65	9.92 ± 0.65	26.99 ±1.41
Series III (12 vol.% Mo and 3 vol.% Cu)	77.09 ±1.85	5.05 ±0.55	18.78 ±1.41	9.38 ±0.41	24.58 ±1.05

In Figure 7 the distribution of metal particles in the Al_2O_3-Cu-Mo composites is shown. The grey area is the ceramic matrix (Al_2O_3) and the bright areas are metal particles. Based on the obtained SEM micrographs, it can be concluded that no effect of the metallic phase on the microstructure of any of the composites was noticed. It was found that the microstructure in all the series is characterized by a compact, non-cracking structure with homogeneous distribution of the metal particles.

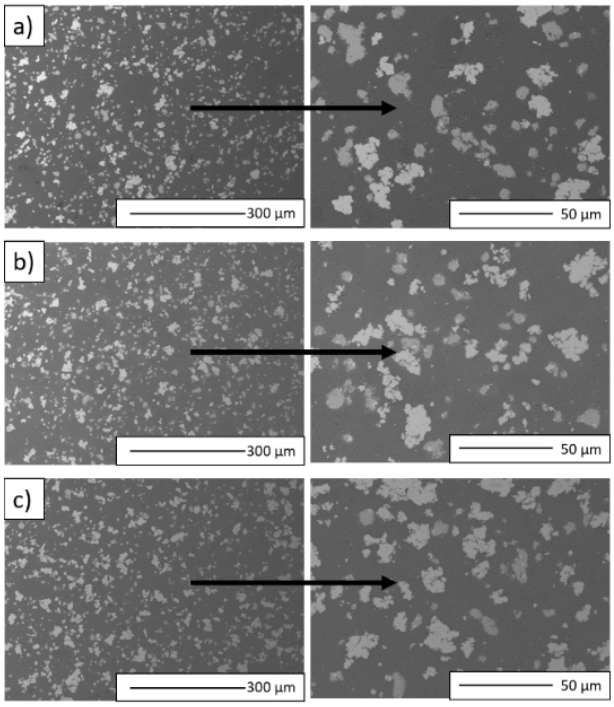

Fig. 7. SEM microphotographs of Al_2O_3-Cu-Mo composites: a) Series I, b) Series II, c) Series III

Rys. 7. Mikrofotografie SEM kompozytów Al_2O_3-Cu-Mo: a) seria I, b) seria II, c) seria III

The values of Vickers hardness were measured and the fracture toughness was calculated. It was found that the Vickers hardness of the sample obtained from Series I equaled 4.29 ± 0.09 GPa. For Series II a higher hardness was observed than Series I, equal to 4.90 ± 0.13 GPa. The highest hardness was obtained by Series III equal to 4.98 ± 0.04 GPa. The obtained hardness results indicate that increasing the molybdenum content causes a slight increase in the hardness of the samples. The fracture toughness was determined using the Vickers indentation fracture toughness test. The results of the fracture toughness measurements revealed that the K_{IC} of the examined sample using the Niihara formula were equal to 4.30, 4.24, 4.61 $MPa \cdot mm^{-1/2}$ for Series I, Series II and Series III, respectively. On the other hand, for the samples fabricated from pure alumina sinters, it was found that the KIC values calculated on the basis of the Niihara equations was equal to 4.02 ±0.65 $MPa \cdot mm^{-1/2}$. It can be concluded that the addition of 15 vol.% metal particles increased the fracture toughness in comparison to the specimens formed from pure alumina. This may be due to several to several factors. Owing to the presence of plastic metal particles of Mo and Cu the cracks were deflected, which prevented their continued propagation [34].

TABLE 3. **Vickers hardness and fracture toughness of different sintered series of samples**
TABELA 3. **Twardość Vickersa i odporność na kruche pękanie dla spieczonych serii próbek**

Al_2O_3-Cu-Mo composites	Vickers hardness [GPa]	K_{IC} [MPa·mm$^{-1/2}$]
Series I (7.5 vol.% Cu and 7.5 vol.% Mo)	4.29 ±0.09	4.30 ±0.15
Series II (10 vol.% Mo and 5 vol.% Cu)	4.90 ±0.13	4.24 ±0.40
Series III (12 vol.% Mo and 3 vol.% Cu)	4.98 ±0.04	4.61 ±0.41

SUMMARY AND CONCLUSIONS

This study describes the fabrication and characterization of innovative hybrid alumina matrix composites. It was established that the slip casting method enables the production of ceramic-metal specimens from the Al_2O_3-Cu-Mo system. The results of the X-ray analysis show that the all the series of composites after sintering contained three phases: Al_2O_3, Cu, Mo. The SEM observations revealed that the metal phase is uniformly distributed in the composite matrix in all the series. It was found that all the samples were characterized by high porosity, which resulted in their low relative density. The volume fractions of Mo and Co in the specimens slightly do affect the composite hardness and fracture toughness. The addition of metal particles decreases the hardness of the ceramic matrix. The results of Vickers hardness tests reveal that increasing the molybdenum content in the composites causes an insignificant increase in the hardness of the samples.

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