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EFFECT OF THE POWDER CONSOLIDATION METHOD TYPE ON THE MICROSTRUCTURE AND SELECTED PROPERTIES OF Al_2O_3-Cu-Ni COMPOSITES

The present research is focused on the characterization of the composites from Al_2O_3-Cu-Ni system. Two methods of ceramic-metal composite forming were applied: uniaxial powder pressing and Pulse Plasma Sintering (PPS). To obtain the samples the powder mixtures containing 85 vol.% of Al_2O_3 and 15 vol.% of metal powders were used. Influence of the sintering process on microstructure and mechanical properties of the two series of the composites was analyzed in detail. The selected physical properties of samples were characterized by Archimedes immersion method. Vickers hardness and the fracture toughness of the composites was determined as well. The microstructure of the composites was characterized by XRD, SEM, EDX. Fractography investigation was carried out as well. Independently on composite production method Al_2O_3, Cu, Ni, and CuNi phases were revealed. Fractography investigation results revealed different character of fracture in dependence of fabrication method. Pulse Plasma Sintered samples were characterized by higher crack resistance and higher Vickers hardness in comparison to the specimens manufactured by uniaxial pressing.

Keywords: Composites, Pulse Plasma Sintering, Uniaxial Powder Pressing, Scanning Electron Microscopy

1. Introduction

Composites belong to one of the most rapidly developing groups of structural and functional materials and are of great interest to researchers [1-6]. The development of engineering and technology is increasing the demand for materials with complex properties that cannot be obtained with traditional materials. Composites make it possible to form materials with desired properties and are increasingly used in various industries, such as aviation and automotive [7-10], also for specialized applications such as a crucibles for induction furnace [11]. Composite materials are very diverse and their properties depend on the components they are made from.

Ceramic matrix materials constitute a dynamically developing group of composites [12-17]. They are characterized by very good mechanical properties such as resistance to wear by friction, high hardness and increased strength. Additionally, they are highly resistant to chemical substances and the elements. Unfortunately, one of the disadvantages of these materials is their fragility, which limits their scope of application. As a result, for many years now, solutions have been sought to improve their crack resistance. In order to do so, other materials are introduced into the ceramic matrix, such as metal particles, the presence of which causes the dispersion of crack energy during crack propagation. In addition, by combining ceramics with metal, it is possible to obtain materials with better thermal, electrical or magnetic properties as compared to their original components [18-19]. Combining ceramics with metal yields composites with a wide range of both strength and functional properties.

Despite the already significant share of these composites in the engineering materials group, they are still the subject of fundamental research, which, in particular, deals with studying the relationship between production process parameters and the microstructure achieved as a result of such a process. In the literature on the subject much research is devoted to the subject matter with regard to two-component systems, i.e. Al_2O_3-Cu [20-21], Al_2O_3-Ni [22-24], Al_2O_3-Cr [25-26] and Al_2O_3-Mo [27-28]. Nevertheless, there are few literature reports concerning the production and characterization of ceramic-based composites containing two metallic phases [29]. Therefore, it would be worth extending the scope of experiments conducted so far with new material systems. The authors of the paper believe that it will be worth to investigate a new composite system, namely Al_2O_3-Cu-Ni. The subject matter of the research is innovative

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as there are no literature reports on such materials. Therefore, the obtained results may contribute to broadening the knowledge on ceramic-metal composites.

In light of the above, this paper focuses on determining the influence of the method of powder consolidation on the microstructure and selected properties of Al_2O_3-Cu-Ni composites. For this purpose, two methods of ceramic-metal composite forming were applied: uniaxial powder pressing and Pulse Plasma Sintering (PPS). In order to determine the influence that the methods used in the paper have on the formation of ceramic-metal system composites, a detailed analysis of the produced materials was carried out, which included microscopic observations, chemical and phase composition analyses and determination of selected physical and mechanical properties of the composites. The quantitative characterization of the produced samples involved carrying out a stereological image analysis and determining the volume shares, grain boundary surface areas, equivalent diameters, distances between adjacent particles and shape parameters for the matrix and reinforcement.

As a result, more insight was gained concerning the influence of the applied powder consolidation method on the microstructure and selected properties of Al_2O_3-Cu-Ni composites. The obtained results allowed to determine changes in the microstructure and mechanical properties depending on the type of composite moulding method used, i.e. uniaxial pressing and pulsed plasma sintering.

2. Experimental

Alumina powder (average particle size 100 ± 20 nm, purity 99.99% and a density of 3.96 g/cm^3), cooper (average particle size 13.34 ± 4.7 µm, a density of 8.94 g/cm^3 and purity 99.99%) and nickel (average particle size 25.56 ± 3.51 µm, a density of 9.8 g/cm^3 and purity 99.50%) were used as starting materials in this experiment. Fig. 1. shown typical morphology of powders. SEM images clearly show that the ceramic powder has been described by a tendency to create agglomerates. It has been noticed that the surface of metal particles is highly irregular with numerous cavities.

The Al_2O_3-Cu-Ni composites were fabricated from the powder mixtures containing 85 vol.% of Al_2O_3 and 15 vol.% of metal powders. The ratio of Ni to Cu was 1:1. Two series of samples were prepared using different techniques: the uniaxial pressing (series I) and the pulse plasma sintering (series II).

The process of preparation of materials contained a few steps. In the first period, the powders (Al_2O_3, Ni, Cu) were mixed by ball milling (PM100, Retsch) in ethanol for 2 h with a speed of 300 rpm. Subsequently, the homogenization of the powders was dried in a laboratory oven at 40°C by 60 h and was sieved. Then, depending on the manufacturing method used, the following procedure was followed. In the uniaxial powder pressing method (series I), the poly(vinyl alcohol) (PVA) was added to the powders as the binder and granulation was accomplished.

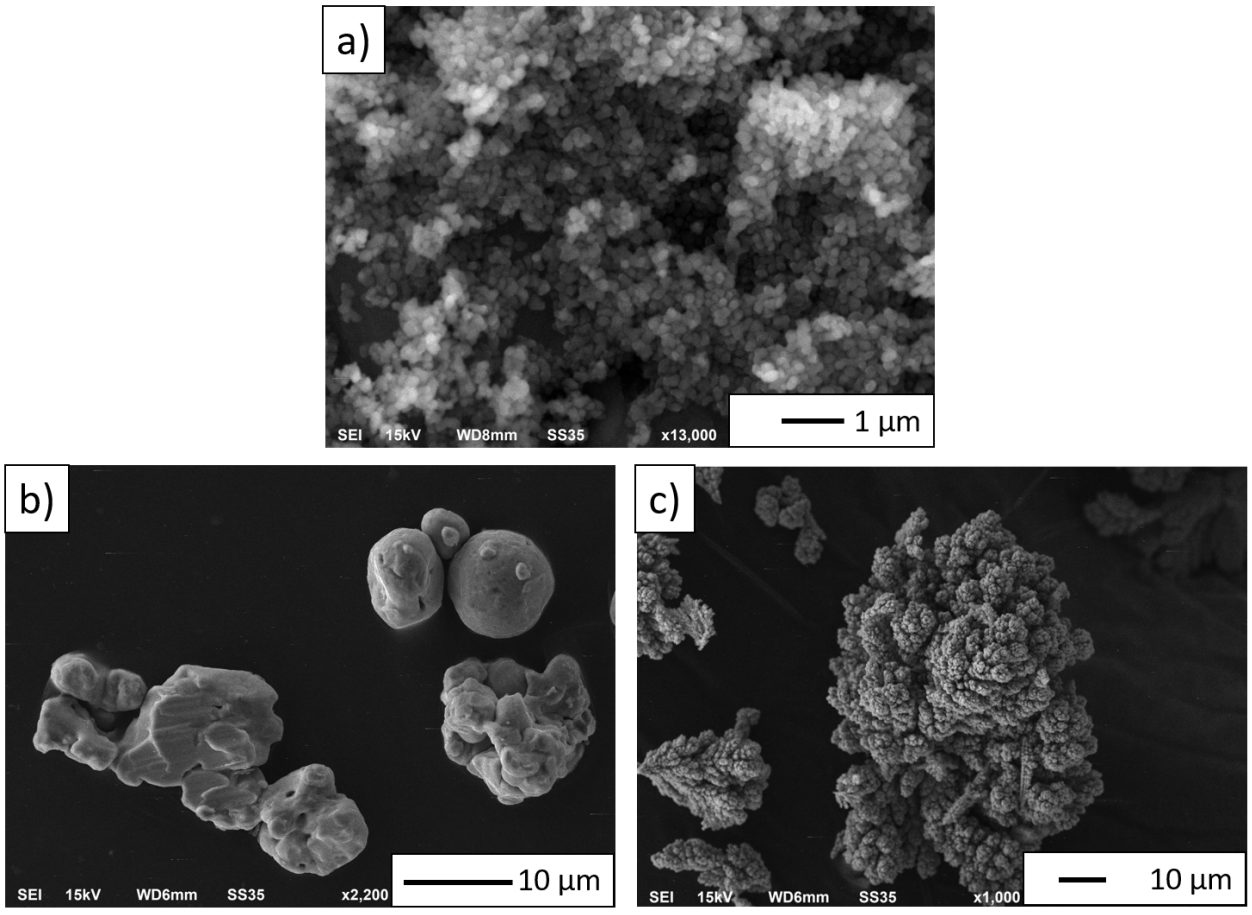

Fig. 1. Morphology of started powders: (a) alumina, (b) cupper, (c) nickel powder

Granulation of the powder with polyvinyl alcohol was carried out through sieves. Subsequently, the specimens were prepared by uniaxial pressing at a pressure of 100 MPa. The samples were sintered in a hydrogen/nitrogen atmosphere at the temperatures of 1400°C. The dwell time was 2 hours. The heating and cooling rate was 5°C/min.

The process of preparation of the samples prepared by the PPS method (series II) were as follows the prepared powders were heated by electric pulses generated periodically by discharging a capacitor battery and, simultaneously, subjected to uniaxial pressing. Powder mixtures of Al_2O_3 with Cu and Ni were sintered in a graphite die in the vacuum of 5×10^{-4} Pa to obtain samples 20 mm in diameter and 3 mm high. The energy of the pulse was 4 kJ and the inter-pulse intervals were 1s. The samples were heated with a ratio of 100°C/min to obtained 1300°C. The samples were kept in a sintering temperature for 3 minutes. In the final stage of the process, the samples were cooled to room temperature in a vacuum without load.

For microstructure investigations prepared composites were cut using a diamond wheel by an automatic cutting precision cut-off machine Secotom 15 (Struers), mounted in epoxy resin and polished. The microstructure and microanalysis of the chemical composition of the fabricated materials were investigated using a JSM-6610 SEM equipped with an EDS detector. Before observations samples were carbon-coated using the Quorum Q150T ESS coating system. SEM was used for fractography investigations, also.

The sinters X-ray diffraction analysis was performed in order to identify the different phases present in the samples. The XRD patterns were obtained with a Rigaku MiniFlex II diffractometer employing CuKα radiation ($\lambda = 1.54175$ Å) in the 2θ range of 20°-100° at sweep rates of 0.02° and counting time 0.5 min^{-1}. The XRD study was performed at the cross sections of all composites.

The selected physical properties of samples were characterized by Archimedes immersion method, i.e. the hydrostatic technique according to standard [30].

The hardness of the composites was determined by using Zwick/Roell Vickers hardness tester. Vickers hardness was measured on the polished surface. In the experiments, a load of 196 N with 15 s holding time was used. For each series, a minimum of 25 indentations was made. The fracture toughness has been calculated based on Niihara equation (1) for $0.25 < l/a < 2.5$ [31-32]:

$$K_{1C} = 0.018 \cdot HV^{0.6} \cdot E^{0.4} \cdot 0.5d \cdot l^{-0.5} \quad (1)$$

Where: HV – Vickers hardness [GPa], l – average crack length [mm], a – stands for half of the average indentation diameter [mm], E – Young's modulus [GPa], d – diagonal of the Vickers indentations [31-32]. Young's Modulus in K_{1C} calculations was determined with the use of the rule of mixtures. According to that rule, Young's Modulus of the composite samples constitute the sum of the components modulus of elasticity with respect to their total amount in the specimen. In this research Young's Modulus calculated for composites from the ternary Al_2O_3-Cu-Ni system with 15 vol. % of metal content and equal percentage amount of metal components in the metal phase was 347.6 GPa. Modulus of elasticity values used in the calculations for Cu, Ni and Al_2O_3 were equal 128 GPa [33], 200 GPa [34] and 380 GPa [35], respectively.

3. Results and discussion

Analysis of the obtained results indicated that the lowest relative density value was noticed for samples obtained by uniaxial powder pressing. The Archimedes measurements show that the series I (uniaxial powder pressing method) characterized by relative density equal to 90.75 ± 1.32%. While for samples produced by the PPS method (series II), the relative density was 95.54 ± 0.95%. Direct observations of samples allowed us to notice that in the case of series I was observed leakage of liquid metal during sintering. It was found that approximately 2-3% of metal (copper) loss in the sintered samples in the case of series I. The leakage of liquid copper during the sintering process has not been observed for series II samples. This may be the reason for the lower density of composites obtained uniaxial pressing than composites fabricated by plus plasma sintering. These results lead to the conclusion that the gained values of density especially with respect to the theoretical density are not satisfying and have to be optimized in order to execute enhanced mechanical properties like a hardness and fracture toughness of fabricated samples.

Macroscopic observations indicate that the both composites obtained by uniaxial powder pressing (Fig. 2a) and pulse plasma sintering (Fig. 2b) revealed no pores and no cracks on their surface. At typical microstructure of samples shown at Fig. 2. The dark areas correspond to the ceramic matrix, while the bright areas are the metallic phase. The scanning electron microscopy research has revealed the homogeneous distribution of the metallic phase in obtained composites.

Energy-dispersive X-ray spectroscopy was carried out to reveal the differences in chemical composition of the samples regarding to method of fabrication. Distribution of the elements on the surfaces of the composites shows the concentrations of aluminium, nickel, copper, and oxygen from the cross-section of composites obtained by powder pressing (Fig. 3a) and pulse plasma sintering (Fig. 3b). The observation obtained in this study indicated that analysed bright areas contain both nickel and copper independently on fabrication method. Concentration of aluminium and oxygen corresponds to composite matrix. Unfortunately EDX measurements do not allow to estimate is the bright areas correspond to solid solution. To reveal phases at the composites XRD measurements were performed (Fig. 4).

The phase composition of the obtained samples is shown in Fig. 4. In both kinds of samples the XRD patterns reveal four phases: Al_2O_3, Cu, Ni, and CuNi. Direct measurements allowed us to observed that characteristic patterns for composites obtained by uniaxial powder pressing the picks at $2\theta = 43.32°$, 50.46°, 74.14°, 89.96° and 95.15° correspond to the (111), (200), (220), (311) and (222), respectively, of the Cu atomic plane

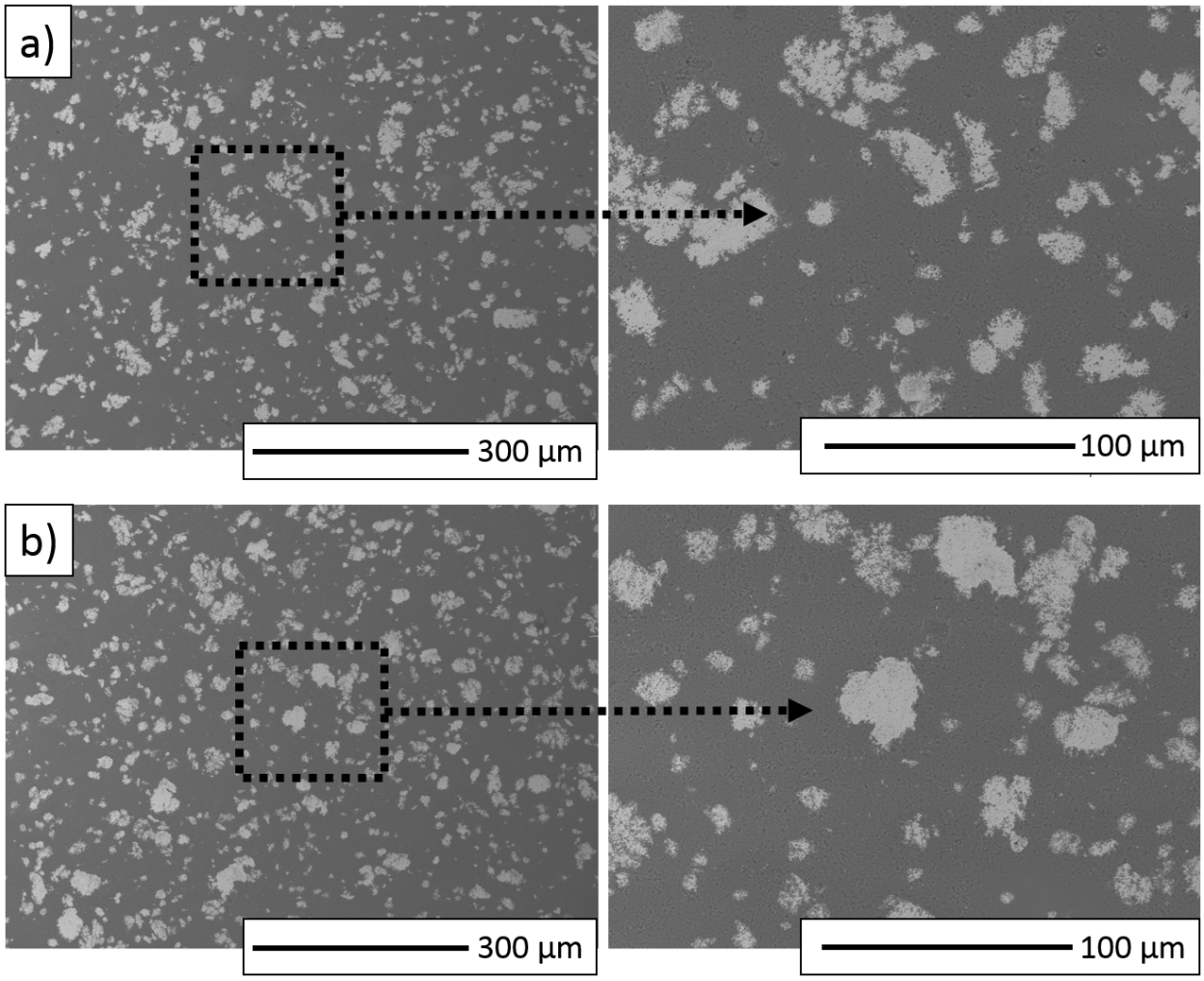

Fig. 2. SEM micrographs of cross-section of Al_2O_3-Cu-Ni composite obtained by: (a) uniaxial powder pressing and (b) pulse plasma sintering

(JCPDS No. 04-004-8452). While, for samples produced by the PPS method the picks at $2\theta = 43.47°$, 50.62°, 74.38°, 90.26° and 95.50° correspond to the (111), (200), (220), (311) and (222), respectively, of the Cu atomic plane (JCPDS No. 04-003-5318). Base on the obtained XRD results, it can be concluded that the diffraction lines for uniaxial powder pressing the picks at 2θ = 44.04°, 51.34°,75.63°, 91.97° and 97.40° correspond to the (111), (200), (220), (311) and (222), in sequence, of the Ni atomic plane (JCPDS No. 04-0016-4592). Whereas, it was found that for composites fabricated by the PPS technique the picks at $2\theta = 44.16°$, 51.46°, 75.75°, 92.09° and 97.52° correspond to the (111), (200), (220), (311) and (222), respectively, of the Cu atomic plane (JCPDS No. 04-016-4592). The results obtained in this study indicated that the XRD pattern of the CuNi exhibits new diffraction lines that are situated between the standard Ni and Cu diffraction lines, suggesting that the mutual insertion of Ni and Cu atoms was created rather than segregated [36-37]. The XRD experimental results indicate that the solid solution CuNi characterized by the cubic structure (Fd-3m) in both series. The obtained results revealed that in the composites obtained by the uniaxial powder pressing characteristic peaks at 43.97°, 51.21°, 75.30°, 91.46° and 96.81° in CuNi conformed to the plane indices (111), (200), (220), (311), (222), respectively (ICDD 04-005-6651). While, for samples produced by the PPS technique the picks at 2θ = 44.12°, 51.38°, 75.55°, 91.79° and 97.16° correspond to the (1 1 1), (2 0 0), (220), (311) and (2 2 2), respectively, of the CuNi atomic plane (JCPDS No. 04-004-6750).

The XRD analysis exhibits that a slightly small in all diffraction patterns there is a slight increase in the 2 theta angle. In Table 1 shown differences between the 2θ peak locations in samples obtained different methods. According to the authors, there can be two reasons for differences. The first reason may be the different locations of the sample relative to the axis of rotation of the goniometer. In this case, the shift of each peak was the same or similar. Another reason may be changing interplanar spacing, which is the separation between sets of parallel planes formed by the individual cells in a lattice structure, depends on the radii of the atoms forming the structure as well as on the shape of the structure of foreign atoms. In this case, the shift of each peak may increase for larger angles.

In the case of the Ni phase, we observe a similar value of the difference between the angles regardless of the fabrication method used. This may be due to the design of the handle on the device. We have previously established that some Ni particles are isolated from Cu and do not react with it. Perhaps this is the signal from these Ni particles. For Cu and CuNi peaks, the offset

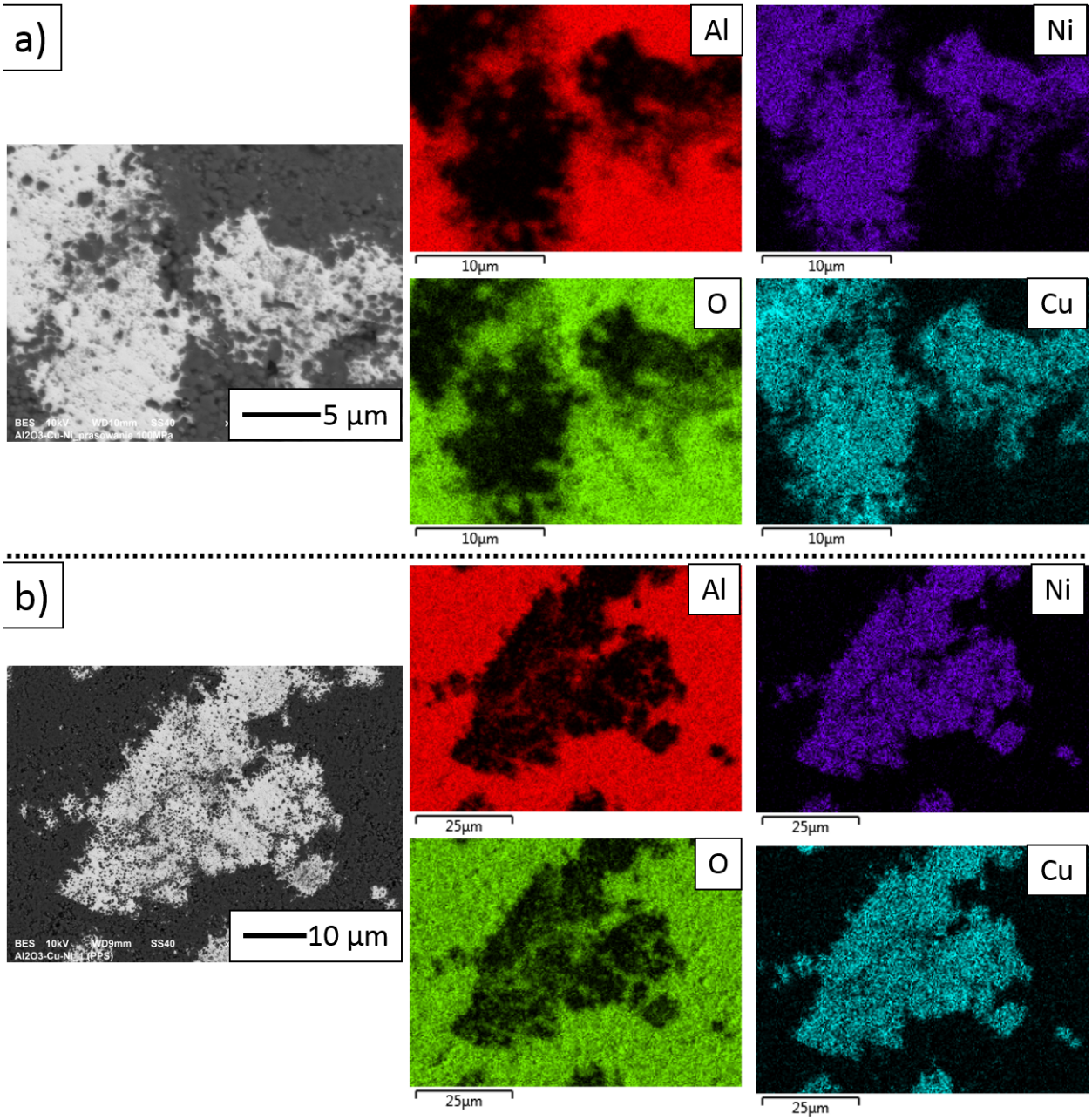

Fig. 3. Distribution of elements on surface of composite specimens obtained by: (a) uniaxial powder pressing and (b) pulse plasma sintering

increases with the angle. It can be assumed that when consolidating with the PPS method, liquid copper better penetrates the areas between Al_2O_3 particles and meets more Ni particles that dissolve in it. Thus, it appears that in PPS samples the CuNi solution may be richer in Ni. The areas we call Cu are actually copper with a small amount of nickel dissolved in it. There is more nickel in PPS samples. All this confirms the thesis that in the case of PPS the samples are more homogeneous. Copper under pressure dissolves better.

Fractography investigation was performed to reveal the mechanism of fracture of the obtained composites in dependence of fabrication method. To observe the fracture surfaces of samples were used. SEM images in Fig. 5a show the fracture surface of the composite obtained by uniaxial powder pressing while Fig. 5b show fracture surface of the composite after pulse plasma sintering. Fractography analysis results revealed that in most cases the bonds between the ceramic matrix and metallic phase are the areas of cracking initiation. Character of cracking is brittle was indicate on poor adhesion of metallic phase and matrix. In the case of pulse plasma sintered composite plastic fracture was observed. Cracking was initiated in the metallic phases what indicate that metallic phases which were plasticity fractured during the test. Fracture observation allowed to conclude that the Al_2O_3 matrix surface is characterized by the brittle fracture mechanism.

The EDS analysis for selected areas on the fracture of the sample obtained by uniaxial powder pressing (Fig. 5a) and pulse plasma sintering (Fig. 5b) was carried out. The results of the concentration measurements of aluminum, oxygen, copper, nickel are presented in Table 2. EDS spectra were collected from two

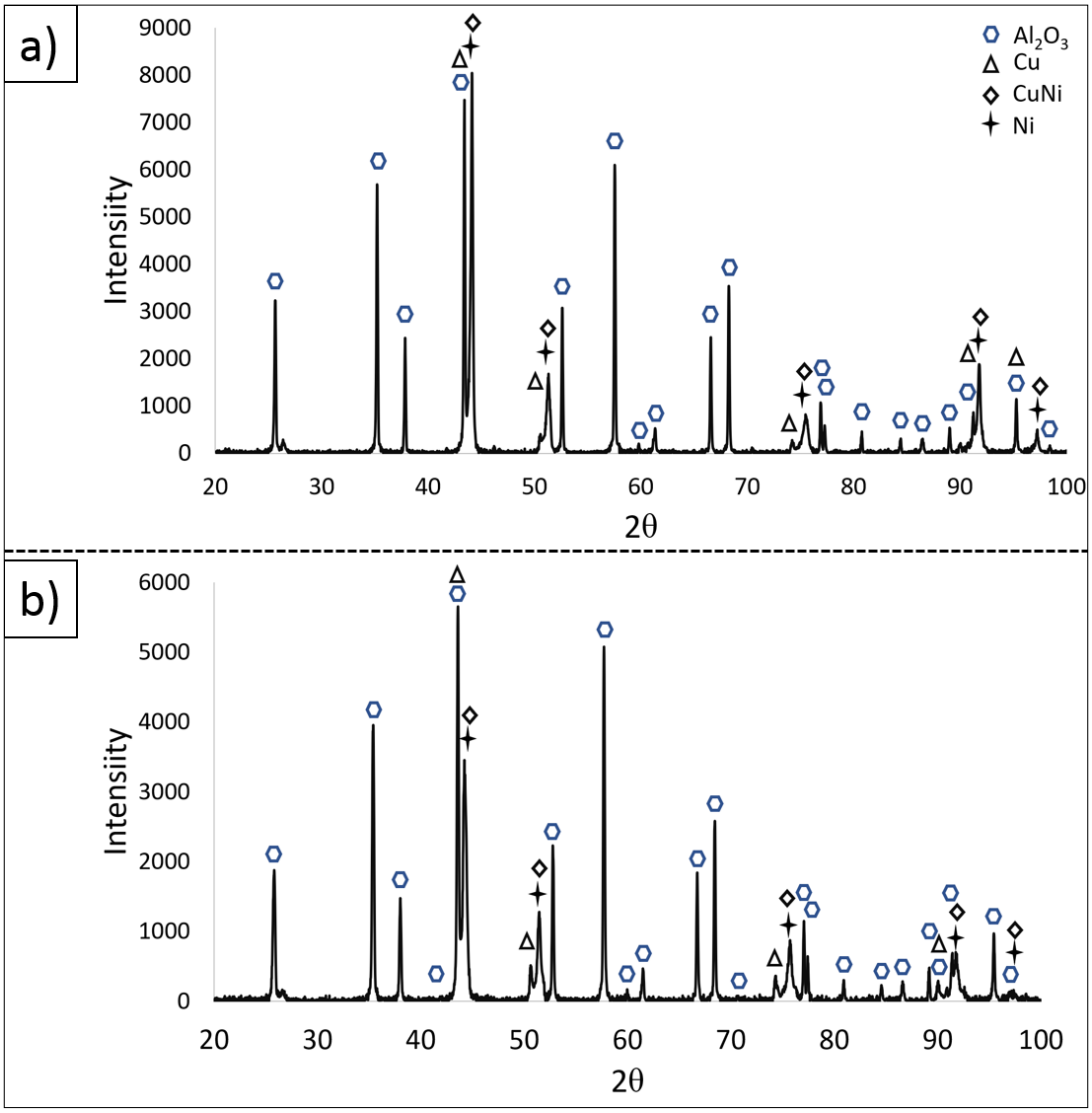

Fig. 4. XRD patterns of the obtained composites fabricated by: (a) uniaxial powder pressing, (b) pulse plasma sintering

TABLE 1

Differences between the 2 theta angle peak locations in samples obtained different methods

Phase	Uniaxial powder pressing	Pulse plasma sintering	Differences between the 2 theta angle peak locations
Cu	43.32	43.47	0.15
	50.46	50.62	0.16
	74.14	74.38	0.24
	89.96	90.26	0.3
	95.15	95.5	0.35
Ni	44.04	44.15	0.11
	51.34	51.46	0.12
	75.63	75.75	0.12
	91.97	92.09	0.12
	97.4	97.52	0.12
CuNi	43.97	44.12	0.15
	51.21	51.36	0.15
	75.3	75.55	0.25
	91.46	91.79	0.33
	96.81	97.16	0.35

different locations at a specimen. The study was conducted on the fracture, so the tested surface is not flat which can generate some measurement error. To limit error of the measurement only point analyses were performed with a high number of counts. The results showed that in the case of the sample obtained by uniaxial powder pressing, matrix at point 1 was revealed and particle made of copper and nickel was revealed at point 2. In the case of the sample obtained by pulse plasma sintering, pure alumina was revealed at the measurement point 1. As opposed to the uniaxial powder pressed sample in the pulse.

Vickers hardness and fracture toughness analysis showed the dependence between obtained results and the specimens manufacturing method used. The obtained values for composites were summarized in Table 3. Samples prepared with use of Pulse Plasma Sintering technique were characterized by higher hardness values in comparison to the samples prepared by combination of uniaxial pressing and pleasureless sintering. Average hardness measured for specimens obtained with use of Pulse Plasma Sintering and uniaxial pressing were equal 9.38 ± 0.98 GPa and 7.69 ± 0.35 GPa, respectively.

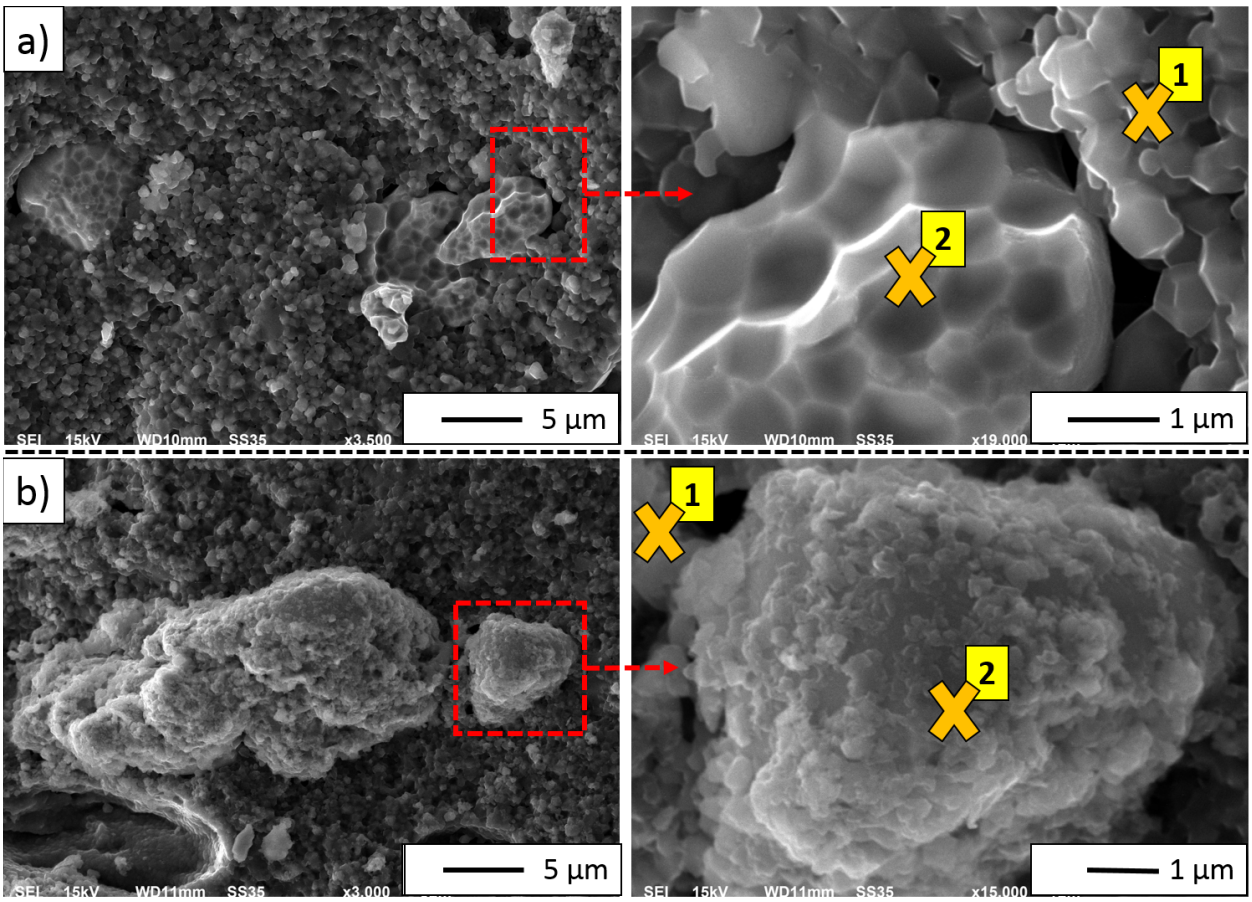

Fig. 5. Fracture Surface of sample obtained by: a) Uniaxial powder pressing and b) pulse plasma sintering

TABLE 2

EDX results of measurements points presented in Fig. 5

Kind of method used for fabrication composites	Points (Fig. 5.)	Weight [%]			
		Al	O	Cu	Ni
Uniaxial powder pressing	1	55.5 ± 0.1	44.5 ± 0.1	0.0 ± 0.0	0.0 ± 0.0
	2	0.0 ± 0.0	0.0 ± 0.0	28.9 ± 0.4	71.1 ± 0.4
Pulse plasma sintering	1	51.2 ± 0.1	48.8 ± 0.1	0.0 ± 0.0	0.0 ± 0.0
	2	9.2 ± 0.1	8.9 ± 0.1	44.3 ± 0.3	37.5 ± 0.3

TABLE 3

The selected mechanical properties of composites

	Pulse plasma sintering	Uniaxial powder pressing
Vickers Hardness [GPa]	9.38 ± 0.98	7.69 ± 0.35
Fracture toughness K_{1C} [MPa·m$^{0.5}$]	6.03 ± 0.68	5.62 ± 0.67

Similar correlation can be observed in case of average fracture toughness calculated on the basis of Vickers indentation technique. Specimens obtained by Pulse Plasma Sintering were characterized by higher crack resistance with slightly higher average K_{1C} value, equal 6.03 ± 0.68 MPa·m$^{0.5}$, in comparison to the specimens manufactured by uniaxial pressing. The K_{1C} value for the latter was equal 5.62 ± 0.67 MPa·m$^{0.5}$. Regardless the manufacturing process, fracture resistance enhancement were observed for both series in comparison to alumina ceramics, which K_{1C} value in the literature is determined as 3.20 MPa·m$^{0.5}$ [38].

4. Conclusion

Two series of Al_2O_3-Cu-Ni composites via different methods: uniaxial powder pressing and Pulse Plasma Sintering (PPS) were fabricated. From the observations of SEM images, it may be concluded that regardless of the method used to form the samples, the composites had a homogeneous microstructure. The direct XRD measurements showed that in both kinds of samples were obtained four phases: Al_2O_3, Cu, Ni, and CuNi. The results reported shows that the samples prepared with use of Pulse Plasma Sintering method were characterized by higher hardness values and fracture toughness in comparison to the

composites prepared by combination of uniaxial pressing and pleasureless sintering.

The obtained results of the research could help to understand and design more effective processes of the production of ceramic-metal composites. The obtained experimental data made it possible to establish the changes in microstructure and mechanical properties vis-a-vis the kind of samples forming technique used, i.e. uniaxial pressing and PPS.

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REFERENCES

[1] J.S. Moya, S. Lopez-Esteban, C. Pecharroman, Prog. Mater. Sci. **52** (7), 1017-1090 (2007), DOI:10.1016/j.pmatsci.2006.09.003.

[2] P.D. Pastuszak, A. Muc, Key Eng. Mater. **542**, 119-129 (2013), DOI:10.4028/www.scientific.net/KEM.542.119.

[3] B. Zhu, Y.J. Cai, Int. J. Impact Eng. **133**, 103340 (2019), DOI:10.1016/j.ijimpeng.2019.103340.

[4] B. Haghpanah Jahromi, A. Ajdari, H. Nayeb-Hashemi, A. Vaziri, Compos. Struct. **92** (8), 1813-1822 (2010), DOI:10.1016/j.compstruct.2010.01.019.

[5] L.K. Yang, P. Shen, R.F. Guo, Y.L. Li, Q.Ch. Jiang, Scr. Mater. **167**, 101-104 (2019), DOI:10.1016/j.scriptamat.2019.04.004.

[6] A. Smirnov, P. Peretyagin, J.F. Bartolomé, J. Eur. Ceram. Soc. **39** (12), 3491-3497, DOI:10.1016/j.jeurceramsoc.2019.02.044.

[7] D.V. Praveen, D.R. Raju, M.V.J. Raju, Mater. Today: Proceedings, (2019), DOI:10.1016/j.matpr.2019.05.392 (in-press).

[8] J. Zhao, The use of ceramic matrix composites for metal cutting applications, in: I.M.Low (Eds.), Advances in Ceramic Matrix Composites 2018, Elsevier (2018).

[9] S. Song, Z. Dong, C. Fernandez, Z. Wen, L. Lu, Mater. Lett. **236**, 13-15 (2019), DOI:10.1016/j.matlet.2018.10.059.

[10] X. Zhu, F. Kong, X. Ma, Ceram. Int. **45** (2), 1940-1945 (2019), DOI:10.1016/j.ceramint.2018.10.086.

[11] J.R. Martinelli, F.F. Sene, Ceram. Int. **26** (3), 325-335 (2000), DOI:10.1016/S0272-8842(99)00059-0.

[12] X. Zhang, T. Yu, J. Zhao, Inter. J. Mech. Sci. (2019), DOI:10.1016/j.ijmecsci.2019.105314 (in press).

[13] J. Du, H. Zhang, Y. Geng, W. Ming, K. Liu, Ceram. Inter. **45** (15), 18155-18166 (2019), DOI:10.1016/j.ceramint.2019.06.112.

[14] B. Mainzer, C. Lin, M. Frieß, R. Riedel, D. Koch, J. Eur. Ceram. Soc. (2019), DOI:10.1016/j.jeurceramsoc.2019.10.049.

[15] P. Zhang, D. Jia, Z. Yang, B. Yang, G. Wang, Mater. Charact. **142**, 59-67 (2018), DOI:10.1016/j.matchar.2018.05.024.

[16] Y. Arai, R. Inoue, K. Goto, Y. Kogo, Ceram. Inter. **45** (12), 14481-14489 (2019), DOI:10.1016/j.matchar.2018.05.024.

[17] S.V. Raj, Ceram. Inter. **45** (3), 3608-3619 (2019), DOI:10.1016/j.ceramint.2018.11.021

[18] A. Fathy, F. Shehata, M. Abdelhameed, M. Elmahdy, Mater. Des. (1980-2015), **36**, 100-107 (2012), DOI:10.1016/j.matdes.2011.10.021.

[19] R. Ritasalo, X.W. Liua, O. Soderberg, A. Keski-Honkola, V. Pitkanen, S.P. Hannula, Procedia Eng. **10**, 124-129 (2011), DOI:10.1016/j.proeng.2011.04.023.

[20] Y. Shi, W. Chen, L. Dong, H. Li, Y. Fu, Ceram. Int. **44** (1), 57-64 (2018), DOI:10.1016/j.ceramint.2017.09.062.

[21] M. Stratigaki, W. Pabst, V. Nečina, M. Hajíček, A.D. Gotsis, SN Appl Sci **1**, 40, (2019) doi:10.1007/s42452-018-0037-4.

[22] C.R. Raghavendra, S. Basavarajappa, I. Sogalad, Colloid Interfac. Sci. **27**, 18-25 (2018), DOI:10.1016/j.colcom.2018.09.003.

[23] A. Yazdani, T. Isfahani, Adv. Powder Tech. **29** (5), 1306-1316 (2018), DOI:10.1016/j.apt.2018.02.025.

[24] J. Zygmuntowicz, M. Wachowski, A. Miazga, K. Konopka, W. Kaszuwara, Compos. Part B-Eng. **156**, 113-120 (2019), DOI:10.1016/j.compositesb.2018.08.079.

[25] W. Węglewski, M. Basista, M. Chmielewski, K. Pietrzak, Compos. Part B-Eng. **43** (2), 255-264 (2012), DOI:10.1016/j.compositesb.2011.07.016.

[26] L. Zhou, S. Cui, Y. Zhai, F. Luo, Y. Dong, Ceram. Inter. **41** (10), 14908-14914 (2015), DOI:10.1016/j.ceramint.2015.08.024.

[27] I.J. Shon, Ceram. Inter. **44** (2), 2587-2592 (2018), DOI:10.1016/j.ceramint.2017.10.120

[28] Y. Zhou, Y. Gao, S. Wei, K. Pan, Y. Hu, Int. J. Refract. Met. H. **54**, 186-195 (2016), DOI:10.1016/j.ijrmhm.2015.07.033.

[29] J. Zygmuntowicz, A. Łukasiak, P. Piotrkiewicz, W. Kaszuwara, Composites Theory and practice **19** (2), 43-49 (2019).

[30] Methods of test for dense shaped refractory products. Determination of bulk density, apparent porosity and true porosity. Technical report, Standard by DIN-adopted European Standard DIN EN 993-1 (1995).

[31] K. Niihara, J. Mater. Sci. Letters **5** (2), 221-223 (1983). DOI: 10.1007/BF00725625.

[32] K. Niihara, R. Morena, D.P.H. Hasselmann, J. Mater. Sci. Letters **1** (1), 13-16, (1982). DOI:10.1007/BF00724706.

[33] J. Dolbow, M. Gosz, Mech. Mater. **23**, 311-321 (1996).

[34] S. Basrour, L. Robert, P. Delobelle, Mater. Sci. Eng. A **288** (2), 160-163 (2000).

[35] A. Ruys, Alumina Ceramics Biomedical and Clinical Applications, Woodhead Publishing (2019).

[36] M. Biswas, A. Saha, M. Dule, T.K. Mandal, J. Phys. Chem. C **118**, 22156-22165 (2014), https://doi.org/10.1021/jp5071874.

[37] Z. Piyong, Z. Gongchang, S. Ting, H. Shaobin, W. Tingting, Z. Heping, J. Catal. **369**, 267-275 (2019), https://doi.org/10.1016/j.jcat.2018.11.003.

[38] D. Chakravarty, S. Bysakh, K. Muraleedharan, T. Narasinga Rao, R. Sundaresan, J. Am. Ceram. Soc. **91** (1), 203-8, (2008). DOI: 10.1111/j.1551-2916.2007.02094.x.