

Magdalena Gizowska^{1*}, Aleksandra Miazga², Katarzyna Konopka², Mikołaj Szafran¹

¹ Warsaw University of Technology, Faculty of Chemistry, ul. Noakowskiego 3, 00-664 Warsaw, Poland

² Warsaw University of Technology, Faculty of Materials Science and Engineering, ul. Wołoska 141, 02-507 Warsaw, Poland

*Corresponding author: E-mail: mgizowska@ch.pw.edu.pl

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THE INFLUENCE OF SINTERING TEMPERATURE ON PROPERTIES OF Al₂O₃-Ni COMPOSITES

The main advantage of ceramic-metal composites is the increase in fracture toughness of the brittle ceramic matrix. The slip casting moulding method is widely used in the ceramic industry, which gives the possibility to obtain products of complicated shapes without green machining. Good quality and homogeneity of powder consolidation are crucial in the ceramic and ceramic matrix composite fabrication process as they influence the properties of the material. In the paper, the results concerning ceramic matrix ceramic-metal composite fabrication via the slip casting method are presented. Composites were formed from a slurry containing alumina and metallic nickel particles. For the studies nickel powder of average particle diameter below 1 μm was used (Aldrich; $d = 8.9 \text{ g/cm}^3$). Two types of alumina powder were applied. The materials formed from alumina powder of an average particle diameter of 0.4 μm (α -Al₂O₃, A16SG, Almatiss; $d = 3.9 \text{ g/cm}^3$) were sintered at a temperature of 1550°C (above nickel melting point; $T_{mNi} = 1455^\circ\text{C}$), whereas composites obtained from alumina powder of an average particle diameter of 0.2 μm (α -Al₂O₃, TM-DAR, Tamei, Japan, $d = 3.8 \text{ g/cm}^3$) undergo densification already at 1300°C (below nickel melting point). The mechanical properties of the composites are dependent on the processes occurring in the sintering process, during which coalescence may lead to the growth of nickel particles. Stereological analysis of the nickel particles in the alumina matrix (in composites sintered below and above nickel melting point) was performed. The description of nickel particle growth in accordance to the sintering temperature was correlated with mechanical properties of the composites.

Keywords: ceramic-metal composite, alumina, nickel, slip casting, stereology

WPLYW TEMPERATURY SPIEKANIA NA WŁAŚCIWOŚCI KOMPOZYTÓW Al₂O₃-Ni

Zapotrzebowanie na tworzywa ceramiczne znacznie wzrosło w ostatnich latach ze względu na ich właściwości fizyczne i mechaniczne. Do tych właściwości zalicza się dużą twardość, sztywność, odporność na ścieranie oraz niską gęstość. Duża kruchość tych materiałów ogranicza obszar ich zastosowania. Jedną z metod zwiększenia odporności na pękanie jest wprowadzenie plastycznych cząstek metalu do osnowy ceramicznej, które powodują rozpraszanie energii rozprzestrzeniającego się pęknięcia. W pracy przedstawiono wyniki badań kompozytów Al₂O₃-Ni otrzymanych metodą odlewania z mas lejnych na bazie proszków metalicznego i ceramicznego. W badaniach jako proszek metaliczny wykorzystano proszek niklu o średnicy cząstek nieprzekraczającej 1 μm (Aldrich; $d = 8,9 \text{ g/cm}^3$). Natomiast fazą ceramiczną był tlenek glinu. W badaniach korzystano z dwóch rodzajów proszku tlenku glinu. Kompozyty otrzymane z proszku tlenku glinu o średniej średnicy cząstek 0,4 μm (proszek α -Al₂O₃, A16SG, Almatiss; $d = 3,9 \text{ g/cm}^3$) zostały spieczone w temperaturze 1550°C (powyżej temperatury topnienia niklu; $T_{topNi} = 1455^\circ\text{C}$). Natomiast kompozyty otrzymane z proszku tlenku glinu o średniej średnicy cząstek 0,2 μm (α -Al₂O₃, TM-DAR, Tamei, Japan, $d = 3,8 \text{ g/cm}^3$) ulegają zagęszczeniu już w temperaturze 1300°C (poniżej temperatury topnienia niklu). Właściwości mechaniczne kompozytów zależą także od warunków procesów spiekania, podczas którego może dojść do znacznego rozrostu cząstek niklu w wyniku ich koalescencji. Obserwacja mikrostruktury oraz analiza cząstek niklu w kompozytach spiekanych powyżej i poniżej temperatury topnienia niklu pozwoliła na opis wpływu temperatury spiekania na rozrost cząstek niklu w osnowie ceramicznej.

Słowa kluczowe: kompozyt ceramika-metal, tlenek glinu, nikiel, masa lejna, stereologia

INTRODUCTION

Ceramic-matrix composites due to their unique properties find applications in diverse areas. The presence of a ductile metal phase hinders the propagation of cracks in a ceramic matrix originating from a strain exhibited in the material. This results in, among others, an increase in fracture toughness and thermal shock resistance in comparison to ceramic material.

In recent years, the colloidal processing of ceramic matrix ceramic-metal composites has gained importance [1-5]. Slip casting, listed among the wet processing methods, is a method where slurry poured into a porous mould is filtrated so that the solidified material settles at the mould surface replicating its shape. The slip casting method gives the possibility of product

shape control without green machining. It also guarantees good quality and homogeneity of powder consolidation in ceramic and ceramic matrix composites. These parameters influence the properties of the material.

In the paper, results concerning ceramic matrix alumina-nickel composites obtained by means of the slip casting method are presented. The composites were sintered at temperatures above and below the nickel melting point. The results of microstructural analysis are presented. Mechanical parameters such as hardness, fracture toughness and bending strength were evaluated and compared to the values obtained for alumina material (obtained in the same conditions as composites). Changes in the mechanical properties of the composites were considered in aspect of the microstructural analysis of nickel inclusions.

EXPERIMENTAL PROCEDURE

In order to investigate the influence of the sintering temperature on the structural and mechanical properties of composites, two kinds of alumina powder were used in the study. The chosen alumina powders differ in their sintering temperature. Sintering active TM-DAR α -alumina powder of a particle diameter size of $d_{50} = 0.21 \mu\text{m}$, provided by Tamei Chemicals (Japan), sinters already at the temperature of 1300°C , which is below the melting point of nickel ($T_m = 1455^\circ\text{C}$). The α -alumina, A16SG, powder obtained from Almatris Inc. (USA) of a particle diameter size $d_{50} = 0.42 \mu\text{m}$ sinters at the temperature of 1550°C , which exceeds the melting point of nickel. Nickel powder of an average particle diameter of $d_{50} = 0.6 \mu\text{m}$ (Sigma-Aldrich) was used. The basic parameters of the used materials are listed in Table 1.

TABLE 1. Basic parameters of used powders
TABELA 1. Podstawowe parametry stosowanych proszków

	$\alpha\text{-Al}_2\text{O}_3$ (TM-DAR)	$\alpha\text{-Al}_2\text{O}_3$ (A16SG)	Ni (Sigma-Aldrich)
Specific surface area (BET)	14.5 m ² /g	8.9 m ² /g	2.11 m ² /g
Density	3.800 g/cm ³	3.926 g/cm ³	8.9 g/cm ³
Particle diameter (d_{50})	0.21 μm	0.42 μm	0.60 μm
Sintering temperature	1300°C	1550°C	-
Melting point	-	-	1455°C

The average size of particle diameter of the powders (Table 1) was measured by means of the Zetasizer Nano ZS (Malvern Instruments).

The composites were prepared by means of the slip casting method. Ceramic water-based slurries with a 50 vol.% solid content were prepared with 0 and 5 vol.% of nickel powder with respect to the total solid volume. Depending on the alumina powder type, the slurries varied in the composition of the used additives.

In all the slurries, a mixture of citric acid (CA; p.a., POCH Gliwice) and diammonium citrate (DAC; p.a., Aldrich) were added in the role of a deflocculant. Binders were added to the slurries in order to increase the green body strength. For slurries containing TM-DAR alumina an acrylic binder (DuramaxTM B-1000, Rohm and Haas) was used. To the slurries containing the A16SG alumina powder ($d = 0.42 \mu\text{m}$), polyvinyl alcohol (Aldrich) was introduced in the role of binder. In both cases, the addition of the polymer was 0.5 wt.% with respect to the alumina powder weight and 0.47 wt.% with respect to the alumina-nickel powder mixture weight. A surface active agent - defoamer (octanol, Reachim) was added as well. The ingredients were homogenized in a planetary mill with a rotating speed of 250 r.p.m. for 80 min. Afterwards, the air absorbed at the particles surface was removed by submitting the slurries to low pressure (10 hPa) for about 15 min.

All the samples were fired in a tube furnace in argon flow as a protective atmosphere. Depending on the ceramic type, two sintering procedures were applied. Alumina and alumina-nickel samples formed from the TM-DAR alumina powder were sintered at the temperature of 1300°C for 5 h. The ceramic and composite samples obtained from A16SG alumina were sintered at 1550°C for 1 h. The physical parameters (porosity, apparent and relative density) of the sintered samples were estimated by means of the hydrostatic method. The theoretical density used to determine relative density was calculated from the rule of mixtures equation. Shrinkage was calculated from a change in sample dimensions.

The bending strength was estimated by the ball-on-ring test. The values were computed using Kirstein and Wooley's equation:

$$\sigma_{\max} = \frac{3P(1-\nu)}{4\pi t^2} \cdot \left[1 + 2 \ln \frac{a}{b} + \frac{(1-\nu)}{(1+\nu)} \left\{ 1 - \frac{b^2}{2a^2} \right\} \frac{a^2}{R^2} \right] \quad (1)$$

where P is the load, t - disk thickness, a - radius of the circle of the support points, b - radius of the region of uniform loading at the center, R - radius of the disk and ν - Poisson's ratio [6, 7].

In order to estimate the fracture toughness value, a method based on the measurement of crack length on the sample surface, originating from a Vickers diamond indentation, was used. The indentations were made under a load of 98, 196 and 294 N. However, the hardness and fracture toughness only for indentations obtained under a load of 196 N are given. Only central cracks were taken into account and the K_{IC} value was computed using equation (2):

$$K_{IC} = 0.067 \cdot \left(\frac{E}{H_V} \right)^{0.4} \cdot \left(\frac{c}{a} \right)^{-1.5} \cdot H_V \cdot \sqrt{a} \quad (2)$$

where a is the half of the indent diagonal ($a = 0.5d$), H_V - Vickers hardness ($H_V = 1.8544F/d$), E - Young modulus, c - crack length [8].

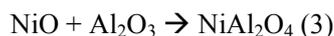
The microstructure observations was performed by means of a LEO 1530 scanning microscope. For better contrast of the composite components, the back scattered electrons detection technique was used. The stereological analysis of the nickel particles in the alumina matrix was performed by means of the computer program MicroMeter written by Tomasz Wejrzanowski [9].

RESULTS AND DISCUSSION

Alumina and nickel show great electrokinetic incompatibility that cause agglomeration of the powders in water systems. The applied composition of deflocculants lessens the effect, however, in the composite slurry minor agglomeration occurs. Alumina-nickel agglomerates promote the stabilization of nickel particles in the composite slurry and composites with a homogenous distribution of nickel particles were obtained. The agglomeration processes in composite slurries is extensively described elsewhere [10].

Alumina samples formed by the slip casting method, both from A16SG and TM-DAR alumina, show high green density. The composite samples in both cases show a lower green density which might be a result of agglomerates present in the composite slurries.

During the sintering process at the surface of all the composite samples, a new phase (NiAl₂O₄) is created which originates from the reaction of alumina and nickel oxide. The surface of the metallic nickel particles after processing in water-based slurries undergoes oxidation and hydroxylation. According to literature, the layer thickness of corrosion products does not exceed a few nanometers [11]. During the heat treatment the corrosion product convert into nickel oxide that is one of the substrates in the reaction of spinel phase formation:



The reaction takes place already below the temperature of 1200°C [12]. The nickel-aluminate spinel is present only at the surface of the samples. The bulk of the samples is free from other species than α -Al₂O₃ and Ni (which was also observed in [13]). None of the samples showed open porosity, which indicates that the spinel layer is dense and non-porous.

The sintering procedure was designed in regard to the optimal conditions for the sintering of selected alumina powders. Thus the influence of nickel particles in an alumina matrix on densification process could be estimated.

Alumina - A16SG type - sintered at 1550°C shows a density of 99.0%, whereas, the composites obtained from alumina A16SG with a 5 vol.% of nickel sintered at the same conditions show a density of 94.5% (Table 2). The presence of nickel particles causes a decrease in the densification process of the alumina matrix. A lower shrinkage of the composite samples was also observed.

TABLE 2. Basic physical properties of green and sintered Al₂O₃ and Al₂O₃-5 vol.% Ni samples depending on sintering temperature

TABELA 2. Podstawowe właściwości fizyczne otrzymanych kształtek surowych oraz spieków z Al₂O₃ oraz Al₂O₃-5% obj. Ni w zależności od temperatury spiekania

	$T_{\text{sintering}} = 1300^\circ\text{C}$		$T_{\text{sintering}} = 1550^\circ\text{C}$	
	Al ₂ O ₃ (TM-DAR)	Al ₂ O ₃ (TM-DAR)+ 5 vol.% Ni	Al ₂ O ₃ (A16SG)	Al ₂ O ₃ (A16SG) + 5 vol.% Ni
Green bodies properties				
d_{rel} [%]	63.2 ± 1.0	60.7 ± 1.3	62.3 ± 1.4	60.5 ± 1.1
Sintered bodies properties				
d [g/cm ³]	3.83 ± 0.03	4.03 ± 0.02	3.89 ± 0.01	3.95 ± 0.02
d_{rel} [%]	99.6 ± 0.7	98.4 ± 0.5	99.0 ± 0.1	94.5 ± 0.5
S_i [%]	14.2 ± 1.0	14.9 ± 0.6	12.9 ± 0.4	11.8 ± 0.6

This phenomena is caused by the fact that during sintering, the nickel particles are in a liquid phase. The diffusion coefficients of the Al₂O₃-Ni phases are low [14-16], thus the mass transport related to the sintering process of alumina does not proceed by means of the liquid in the second phase. Nickel particles hinder the sintering process. Relative density of alumina sintered at 1300°C is 99.7%. TM-DAR alumina is sintering active, thus the influence of nickel particles on densification of composite bodies is less significant. Density of alumina-nickel composites equals 98.4% (Table 2).

In Figure 1, a typical microstructure of the composites (green body and sinters) is presented. The distribution of nickel particles is homogenous. The nickel particles are well dispersed in the green bodies (Fig. 1a), as the average particle diameter (expressed as average spherical equivalent - d_2) evaluated by stereological analysis, is 0.56 μm (with standard deviation of 0.26 μm ; Fig. 2b), which is close to the value of the average particle diameter of the used 0.51 μm powder (Fig. 2a).

The average particle diameter of nickel inclusions in the alumina matrix sintered at 1550 °C is almost two times greater than before sintering and the average spherical equivalent diameter equals $d_2 = 1.12 \mu\text{m}$ ($\pm 0.79 \mu\text{m}$). The nickel particles melt before the sintering temperature is achieved ($T_m(\text{Ni}) = 1455^\circ\text{C}$), thus it may migrate in the porous structure of the matrix (which densifies at sintering temperature) and create bigger aggregates. In Figure 2 a cumulative curve of nickel particles is presented. Curve 'd' in Figure 2 refers to the distribution of nickel inclusions diameter (expressed by d_2 parameter) in the composite sintered at the temperature of 1550°C. The cumulative curve is related to the volume fracture of metallic inclusions. From the curve it can be concluded that the major volume of the metallic phase is exhibited as inclusions of $d_2 > 2 \mu\text{m}$ and particles of a diameter of about 4.5 μm can rarely be observed.

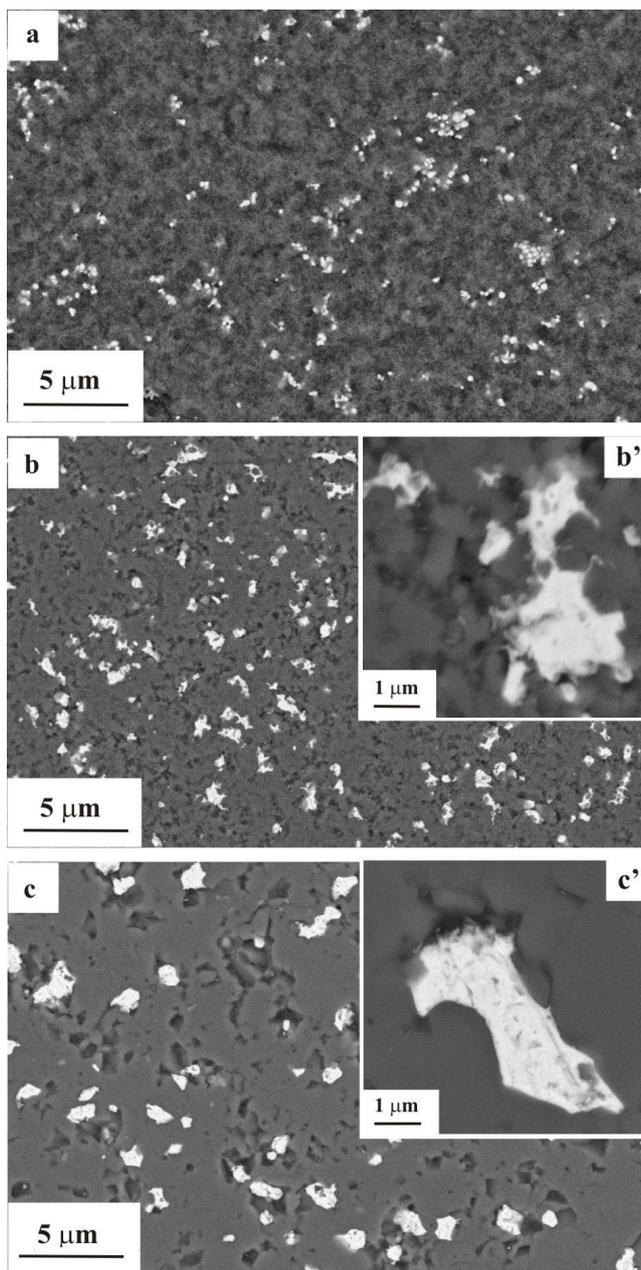


Fig. 1. Typical microstructure of Al_2O_3 -Ni composite obtained by slip casting method (white areas - nickel; dark areas - alumina): a) green body (Al_2O_3 (TM-DAR) - 5 vol.% Ni), b, b') Al_2O_3 (TM-DAR) - 5 vol.% Ni composite sintered at 1300°C , c, c') Al_2O_3 (A16SG) - 5 vol.% Ni composite sintered at 1550°C

Rys. 1. Typowa mikrostruktura kompozytu Al_2O_3 -Ni otrzymanego metodą formowania z mas lejnych (ciemne obszary - osnowa ceramiczna; jasne obszary - nikiel): a) kompozyt w stanie surowym (Al_2O_3 (TM-DAR) - 5% obj. Ni), b, b') kompozyt Al_2O_3 (TM-DAR) - 5% obj. Ni spiekany w 1300°C , c, c') kompozyt Al_2O_3 (A16SG) - 5% obj. Ni spiekany w 1550°C

The sintering of composites below the melting point of the metallic phase does not exclude the growth of nickel particles. The average diameter of nickel inclusions in the alumina (TM-DAR) matrix sintered at the temperature of 1300°C equals $d_2 = 0.81 \mu\text{m}$ ($\pm 0.54 \mu\text{m}$). However, from the cumulative curve (Fig. 2c) it can be concluded that only a small volume fraction of nickel inclusions are characterized by $d_2 < 1 \mu\text{m}$, and inclusions with d_2 up to $4 \mu\text{m}$ can be found.

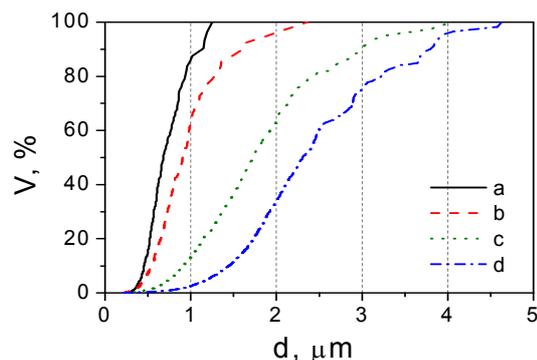


Fig. 2. Cumulative volume curves of nickel particles equivalent spherical diameter: a) nickel powder (raw material), b) nickel particles in green body (Al_2O_3 (TM-DAR) - 5 vol.% Ni), c) nickel particles in Al_2O_3 (TM-DAR) - 5 vol.% Ni composites sintered at 1300°C , d) nickel particles in Al_2O_3 (A16SG) - 5 vol.% Ni composites sintered at 1550°C

Rys. 2. Krzywe kumulatywne rozkładu wielkości cząstek (średnicy równoważnej) niklu: a) proszku niklu, b) cząstek niklu w kompozycie surowym (Al_2O_3 (TM-DAR) - 5% obj. Ni), c) cząstek niklu w kompozycie Al_2O_3 (TM-DAR) - 5% obj. Ni spiekany w temperaturze 1300°C , d) cząstek niklu w kompozycie Al_2O_3 (A16SG) - 5% obj. Ni spiekany w 1550°C

In Table 3, the basic mechanical parameters of the alumina and alumina-nickel composite materials obtained by the slip casting method are presented. The composites show a lower bending strength than the ceramic materials obtained in the same conditions. A decrease in bending strength results from the nickel inclusion growth. Between the alumina and nickel particles dispersed in the ceramic matrix, a great mismatch between the thermal expansion coefficient occurs. The presence of particles that are characterized by a higher thermal expansion coefficient than the matrix cause the matrix to be compressed in the region nearby the particle. The bigger the inclusion, the greater the forces resulting from the thermal expansion mismatch occur. A certain size of nickel inclusion may cause cracking of the ceramic matrix. According to literature [17], nickel particles of a diameter above $2 \mu\text{m}$ may cause the occurrence of microcracks in alumina at the nickel-alumina interphase. However, such microcracks have not been observed under SEM in the studied composites.

TABLE 3. Selected mechanical parameters of Al_2O_3 and Al_2O_3 -5 vol.% Ni composites

TABELA 3. Wybrane parametry mechaniczne spieków z Al_2O_3 i kompozytów Al_2O_3 -5% obj. Ni w zależności od temperatury spiekania

	$T_{\text{sintering}} = 1300^\circ\text{C}$		$T_{\text{sintering}} = 1550^\circ\text{C}$	
	Al_2O_3 (TM-DAR)	Al_2O_3 (TM-DAR) + 5 vol.% Ni	Al_2O_3 (A16SG)	Al_2O_3 (A16SG) + 5 vol.% Ni
σ_{max} [MPa]	554 ± 107	379 ± 65	322 ± 123	241 ± 56
H_{V196N} [GPa]	15.27 ± 0.58	13.95 ± 0.50	14.03 ± 0.31	13.68 ± 0.37
K_{IC} [$\text{MPa}\cdot\text{m}^{1/2}$]	4.88 ± 0.48	6.09 ± 0.49	4.35 ± 0.53	5.72 ± 0.89

Both types of composites (sintered at 1300 and 1550°C) show a minor decrease of hardness in regard to the hardness of the alumina material (Table 3). Furthermore, an increase in fracture toughness of the composites in comparison to alumina was observed. The presence of nickel particles in the composites sintered at 1300°C contributed to an increase in the K_{IC} coefficient by 25%, as the fracture toughness coefficient for alumina (TM-DAR) is 4.88 MPa·m^{1/2} and for the composite it is 6.09 MPa·m^{1/2}. Alumina type A16SG, sintered at the temperature of 1550°C shows a lower value of fracture toughness coefficient (which is 4.35 MPa·m^{1/2}), however, the presence of a mere 5 vol.% of nickel phase causes an increase in the K_{IC} by about 32% and K_{IC} coefficient equals 5.72 MPa·m^{1/2} (Table 3).

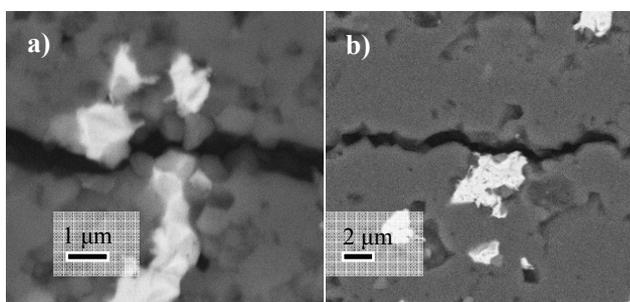


Fig. 3. Deflection of crack path (originating from Vicker's indentation) by nickel particle: a) Al₂O₃ (TM-DAR) - 5 vol.% Ni, b) Al₂O₃ (A16SG) - 5 vol.% Ni

Rys. 3. Ugięcie pęknięcia wychodzącego z naroża odcisku po wglębniku Vikersa: a) Al₂O₃ (TM-DAR) - 5% obj. Ni, b) Al₂O₃ (A16SG) - 5% obj. Ni

In the ceramic matrix composites reinforced by ductile metal particles, the an increase in fracture toughness results from the inhibition of propagating cracks due to a few mechanisms, i.e.: crack bridging, crack path deflection by particles and crack relaxation via local plastic flow [18]. In the studied composite materials, observation of the interaction of a propagating crack with metallic particles proved that for the dissipation of crack energy, the deflection of the crack path is usually responsible (Fig. 3). This kind of crack-inclusion interaction is also a result of the distribution of residual stresses in the composite materials that stem from the thermal expansion mismatch [19].

CONCLUSIONS

- Slip casting method can be successfully applied to fabrication of alumina-nickel composites, in which distribution of nickel particles was homogenous.
- Composites sintered at temperature below melting point of nickel showed average nickel particle diameter below 1 μm.
- Presence of nickel particles aggregates of high diameter in ceramic matrix negatively influence bending strength of alumina.

- Presence of nickel particles in composites (both sintered below and above melting point of nickel) cause dissipation of propagating crack energy which results in increased fracture toughness value measured for composites in comparison to the value obtained for alumina.

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