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KOMPOZYTY
COMPOSITES

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EFFECT OF SPECIFIC SURFACE FRACTION OF INTERPHASE BOUNDARIES ON MECHANICAL PROPERTIES OF CERAMIC-METAL COMPOSITES, OBTAINED BY PRESSURE INFILTRATION

Ceramic-metal composites, obtained via pressure infiltration of porous ceramics Al_2O_3 by cast aluminium alloy EN AC-AlSi11 (AK11), were studied. As a result, composites of two interpenetrating phases are obtained. They are composed of 30 vol.% of ceramics. The pore sizes of the ceramic preforms varied from 150 to 500 μm. The results of the X-ray tomography proved very good infiltration of the pores by the metal. The residual porosity is approximately 9 vol. %. The obtained microstructure with percolation of the ceramic and metal phases gives the composites good mechanical properties together with the ability to absorb strain energy. Image analysis has been used to evaluate the specific surface fraction of the interphase boundaries (S_v). The presented results of the studies show the effect of the surface fraction of the interphase boundaries of ceramic-metals on the composite compressive strength, hardness and Young's modulus. In addition, the mechanical properties depend on the degree of infiltration.

Compression tests for the obtained composites were carried out, and Young's modulus was measured by application of the DIC (Digital Image Correlation) method. Moreover, Brinell hardness tests were performed.

The composites microstructure was studied using scanning electron microscopy (SEM). SEM investigations showed that the pores are almost fully filled by the aluminium alloy. The obtained results show that the infiltration method can be used to fabricate composites with percolation of the microstructure. However, the research is at its early stage and will be continued in the sphere of the characteristics of interphase boundaries.

Keywords: **porous ceramics, cast aluminium alloy, composite, specific surface fraction of the interphase boundaries**

WPŁYW POWIERZCHNI GRANIC MIĘDZYFAZOWYCH W KOMPOZYTACH Al_2O_3- STOP ALUMINIUM, WYTWARZANYCH METODĄ INFILTRACJI CIŚNIENIOWEJ, NA WYBRANE WŁAŚCIWOŚCI MECHANICZNE KOMPOZYTÓW

Przedmiotem badań były kompozyty ceramika-metal, otrzymywane w wyniku infiltracji porowatej ceramiki Al_2O_3 odlewniczym stopem EN AC-AlSi11 (AK11). W wyniku infiltracji ceramicznych kształtek powstają kompozyty o dwóch wzajemnie przenikających się fazach, przy czym udział objętościowy ceramiki stanowi ok. 30% objętości kompozytu. Makrostruktura kompozytu o perkolacji faz ceramiki i metalu nadaje mu zdolność do absorbowania energii.

Przeprowadzone badania wykazały wpływ udziału granic międzyfazowych ceramika-metal na właściwości mechaniczne kompozytów. Poprzez zmianę wielkości porów w kształtkach ceramicznych infiltrowanych odlewniczymi stopami aluminium, zachowując przy tym stały udział porowatości otwartej, sterowano względną powierzchnią granic rozdziału ceramika-metal. Na wytrzymałość na ściskanie, twardość i moduł Younga oprócz udziału granic międzyfazowych wpływ ma także stopień przeinfiltrowania ceramiki metalem.

Badania te mają charakter wstępny i będą kontynuowane w zakresie charakteryzacji granic rozdziału. Uzyskane wyniki dają możliwość uzyskania tą drogą nowych materiałów o właściwościach łączących pozytywne cechy składników kompozytu, w którym osnową jest tworzywo ceramiczne, a fazą wypełniającą pory metal.

Słowa kluczowe: **ceramika, kompozyty, stop aluminium, perkolacja faz, granica międzyfazowa**

INTRODUCTION

In recent years, an increase of interest in ceramic matrix composites, mostly infiltrated by a light metal has been observed. Such materials have found their application in many industry branches, such as in the aircraft industry, automotive and armaments, as well as in electrical engineering and electronics. However, the applications of these materials is restricted due to their low fracture toughness. Ceramic-metal composites combine ceramic hardness and stiffness with the low elastic modulus, high coefficient of thermal expansion and low wear resistance of light metal alloys [1-5].

In literature, numerous works concerning the fabrication of composites with a percolation of phases can be found. The composite microstructure is characterized by two interpenetrating phases of ceramic and metal. In this paper ceramic-metal composites obtained via the pressure infiltration of porous Al_2O_3 ceramics by an aluminium alloy were studied. [6-8].

Several methods allow one to determine the endurance of interphase boundaries of ceramics/metals and their influence on the mechanical properties of the composite [9-12]. In this study, the effect of the specific surface fraction of the interphase boundaries of ceramic-metals on the composite compressive strength, hardness and Young's modulus was shown.

MATERIALS AND METHODS

The ceramic preforms were manufactured in the Institute of Ceramics and Construction Material by sintering RA-207LS Al_2O_3 powder supplied by Alcan Chemicals. The chemical composition of aluminum oxide was Al_2O_3 (99.8 wt.%), CaO (0.02 wt.%), SiO_2 (0.04 wt.%), MgO (0.04 wt.%), Fe_2O_3 (0.03 wt.%), Na_2O (0.07 wt.%). For each ceramic preform the porosity was at the same level, approximately 70 vol.%. Porous aluminum oxide preforms were formed by the method of copying the cellular structure of the polymer matrix [13]. Three types of polyurethane sponges, differing in density and size of pores were exploited: 45, 60 and 90 pores per inch (ppi). This results in the fabrication of preforms with pore sizes varying from 150 to 500 μm.

The composites were made using pressure infiltration of the ceramic preforms with cast aluminium alloy EN AC-AlSi11 (AK11) by the AGH University of Science and Technology. As a result, three kinds of composites were obtained (Tab. 1).

TABELA 1. **Oznaczenie otrzymanych kompozytów**
TABLE 1. **Designation of ceramic-metal composites**

Designation of Al_2O_3/ AlSi11 composites	Pore size of Al_2O_3 preforms
1-150	150 μm
2-350	350 μm
3-500	500 μm

Filling ceramic forms with liquid metal requires solution of the issue of metal flow through a porous preform. Infiltration of a porous medium with a liquid metal is directly related to the chemical composition and temperature of the liquid metal, as well as its viscosity and surface tension. Liquid metal virtually does not moisturize ceramic surfaces; therefore, infiltration is only possible after the metal has reached a sufficiently high pressure, i.e. penetration pressure or capillary pressure [14, 15]. The lowest pressure p necessary for liquid metal to infiltrate a capillary of r radius, equating the size of the porous preform pores, amounts to [14]:

$$p_k = \frac{2 \cdot \sigma \cdot \cos\theta}{r} \quad (1)$$

where: p_k - penetration pressure [Pa], σ - surface tension of liquid metal [N/m], θ - moisturizing angle, i.e. bordering angle between capillary substance and liquid metal, r - capillary (pores) radius in [m]

$$l = \frac{r}{2} \cdot \sqrt{\frac{\tau \cdot \Delta p}{\eta}} \quad (2)$$

Upon reaching the critical pressure, the depth of liquid (l) metal infiltration depends on the size of the capillaries (r), the variation of pressure along the capillary length (Δp), the time of preserving the metal in a liquid state (t) within the capillary and on the dynamic viscosity of the alloy (η). Filling the capillaries of a porous medium (preforms) with a liquid alloy can be performed by using the technology of creating either overpressure or subpressure. With low-durability porous media and small pore (capillary) size, the use of subpressure is more beneficial. For the purposes of this work, the subpressure technology of preform filling was used. To apply this solution, a sampler was constructed (Fig. 1) and subpressure inside the sampler was created using a vacuum pump. The absolute value of the pressure amounted to 0.02÷0.04 MPa (subpressure of 0.08÷0.06 MPa). To limit the possibility of liquid metal flowing from the sampler to the vacuum pump, a cooling filter system was used (Fig. 1).

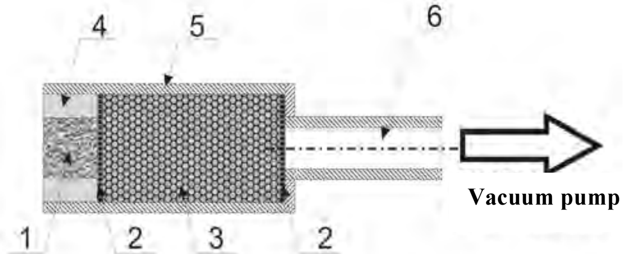


Fig. 1. Design of sampler used for subpressure infiltration of ceramic preforms: 1 - preform sample, 2 - mesh, 3 - filter (cooler), 4 - sample (preform) sealant, 5 - casing, 6 - vacuum pump connection

Rys. 1. Konstrukcja próbnika do podciśnieniowej infiltracji kształtek ceramicznych: 1 - kształtka ceramiczna, 2 - siatka, 3 - filtr (ochładzalnik), 4 - uszczelnienie próbki, 5 - obudowa, 6 - połączenie do pompy próżniowej

The sampler (2) heated to c.a. 300÷350°C was submerged in a melting-pot (1) containing liquid metal (3) and subpressure was created subsequently (Fig. 2). The metal infiltrated the capillaries, filling them in the whole preform volume. Liquid alloy AlSi11 was maintained at a temperature of 770÷780°C during preform filling. Composition of the alloy used for infiltration: Si - 10.8 wt.%, Fe - 0.90 wt.%, Zn - 0.35 wt.%, Al - remainder.

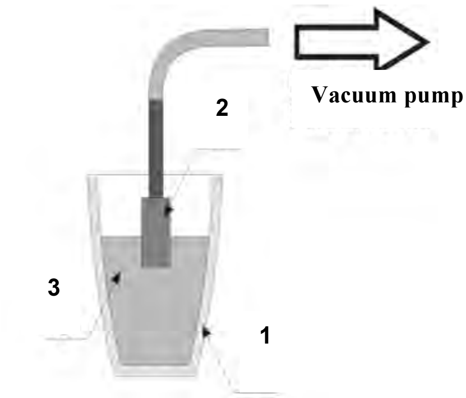


Fig. 2. Transportation by suction of liquid alloy to sampler containing ceramic preform: 1 - melting-pot, 2 - liquid alloy, 3 - sampler

Rys. 2. Zasysanie ciekłego stopu do próbnika z umieszczoną w środku kształtką ceramiczną: 1- tygiel, 2 - ciekły stop, 3 - próbnik

The structure of these composites was studied with Scanning Electron Microscopy (HITACHI S-2600N) quantitatively characterized using image analysis. Such parameters as the volume fraction of phases and distribution of pores were calculated. The specific surface fraction of the interphase boundaries (S_v) was determined using the Micrometer program. The microstructure was characterized using X-ray tomography type SkyScan 1174. Brinell hardness tests were also performed. Furthermore compression tests were carried out using a Zwick 250 machine with application of the Digital Image Correlation (DIC) method. The DIC method was utilized to determine Young's modulus.

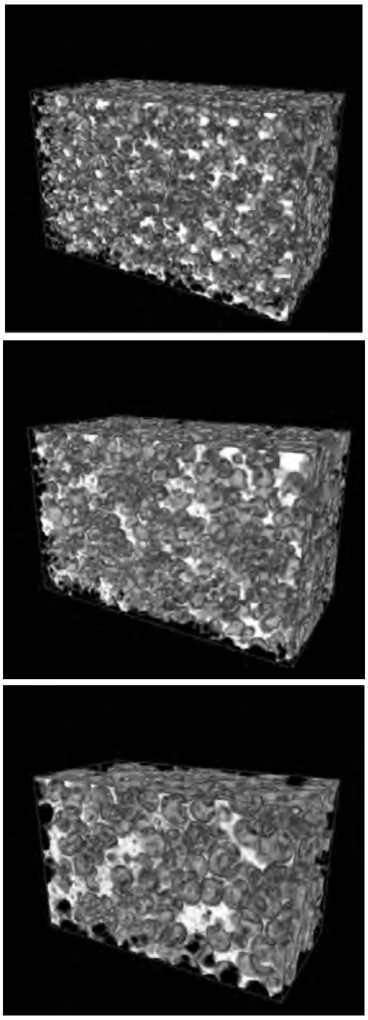


Fig. 3. X-ray tomography images of porous Al_2O_3 ceramics (samples 1 - 150, 2 - 350, 3 - 500, respectively)

Rys. 3. Zdjęcia wykonane mikrotomografem rentgenowskim porowatej ceramiki Al_2O_3 (próbki 1 - 150, 2 - 350, 3 - 500, odpowiednio)

RESULTS

The volume fraction of the ceramics and pores were determined using X-ray tomography (Fig. 3, Tab. 2). Moreover the distribution of size of the pores was |specified. The volume fraction of the ceramic phase is approximately 30 vol.%, the remaining area (70 vol.%) can be filled up with liquid metal. Based on tomography images, a slight difference in the size of ceramic preform pores was observed, as shown in Table 2.

TABELA 2. **Udziały objętościowe ceramiki i porów kształtek Al_2O_3**

TABLE 2. **Volume fraction of Al_2O_3 ceramic and pores**

Designation of Al_2O_3 preforms	Volume fraction of Al_2O_3 ceramics [%]	Volume fraction of pores [%]
1 - 150	30.57	69.43
2 - 350	28.52	71.48
3 - 500	25.58	74.42

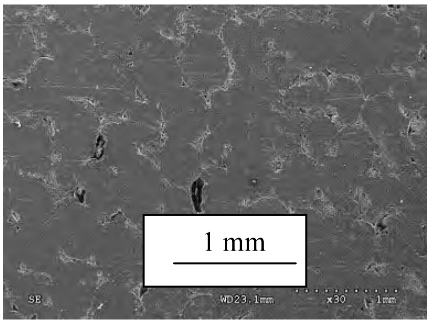
The results of the X-ray tomography proved very good infiltration of the pores by the metal. The residual porosity is approximately 9 vol.% independent of the size of the pores (Tab. 3).

TABELA 3. **Stopień przeinfiltrowania ceramiki stopem aluminium**

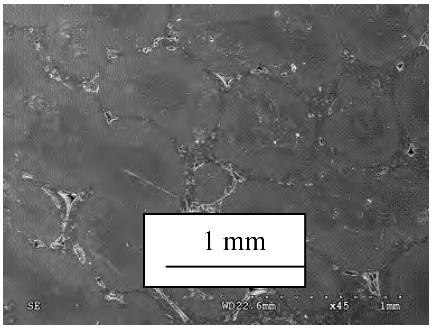
TABLE 3. **Degree of infiltration of ceramic by cast aluminium alloy**

Designation of Al_2O_3/ AlSi11 composites	Residual porosity [%]	Composite volume [%]
1 - 150	9.48	90.52
2 - 350	9.02	90.98
3 - 500	8.99	91.01

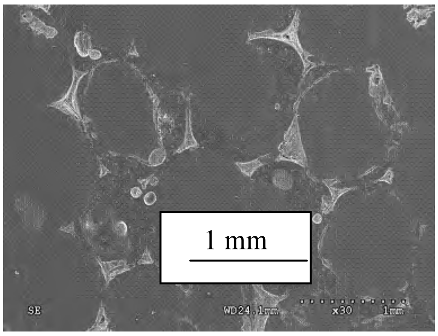
The SEM observations of the composites revealed the percolation type of microstructure; almost all the pores are fully filled by the aluminium alloy. Only small unfilled voids in the microstructure of the composite obtained by infiltration of the preform with the smallest size of pores was observed (Fig. 4).



(1-150)



(2-350)



(3-500)

Fig. 4. Microstructure of Al_2O_3/AlSi11 composites (samples 1 - 150, 2 - 350, 3 - 500, respectively)

Rys. 4. Mikrostruktura kompozytów Al_2O_3/AlSi11 (próbki 1 - 150, 2 -350, 3 - 500, odpowiednio)

The chemical composition of a separated phase observed in the microstructures of the composites were examined using the EDS method (Fig. 5). It was found that on the grain boundaries, separated silicon is visible. For confirmation and the full identification of the separated phases on the boundaries and inside the metallic phase, X-ray and TEM examinations were employed.

Image analyses have been used to measure the specific surface fraction of the interphase boundaries (S_v) using the Micrometer program. The measured S_v parameter increases together with the reduction of the sizes of the ceramics performs pores (Tab. 4).

A decrease of the size of the performs pores results in a growth of the volume fraction of ceramics-metal interphase boundaries, at a permanent value of the porosity.

Compression tests for the samples of porous ceramics and composites were carried out. The compressive strength of the ceramic and composites was determined. The character of the stress-strain curves of the ceramic and composites was compared. The values of the compressive strength are shown in Table 4.

TABELA 4. **Względna powierzchnia granic ziaren (S_v), wytrzymałość na ściskanie, twardość HB, moduł Younga i energia absorpcji kompozytów Al_2O_3/ AlSi11**

TABLE 4. **Surface fraction of interphase boundaries (S_v), compressive strength, hardness HB, Young's modulus and energy absorption of Al_2O_3/ AlSi11 composites**

Designation of Al_2O_3/ AlSi11 composites	S_v [1/mm]	Compressive strength [MPa]	Hardness HB	Young' modulus [GPa]	Energy absorption [MJ/m^2]
1 - 150	9.79	146	90.8 ± 2.6	3.829	7.85
2 - 350	8.33	83,6	76.1 ± 1.9	2.725	5.07
3 - 500	5.98	68	65.6 ± 3.5	1.866	2.47

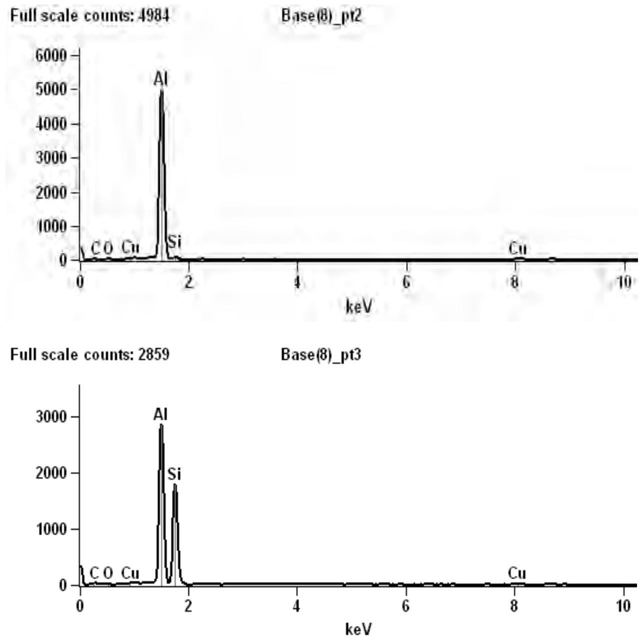


Fig. 5. Chemical composition of aluminium AlSi11 alloy (1) and interphase boundary (2)

Rys. 5. Skład chemiczny stopu aluminium AlSi11(1) i granicy międzyfazowej (2)

As shown in Figure 6, composites exhibit much higher compressive strength in comparison to the porous preform. The composite fabricated by infiltration of the preform with the smallest pores is characterized by the highest compressive strength (Table 4). Also the slope of the stress-strain curves for ceramics and composites changed. A distinct decrease in stresses on the stress-strain curves of the composites was not observed, while ceramics failed to pass the test. The obtained microstructure with percolation of the ceramic and metal phases gives the composites high mechanical properties together with the ability to absorb strain energy.

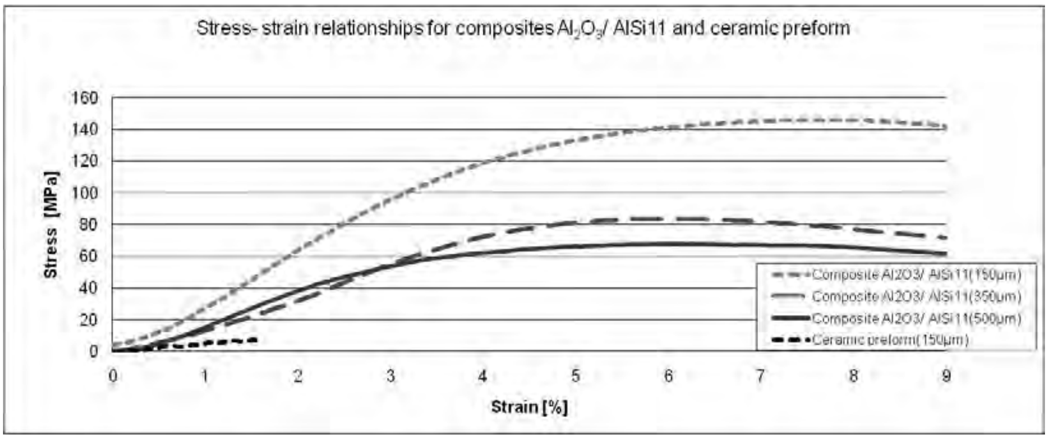


Fig. 6. Typical stress-strain curves for ceramic and Al_2O_3/ AlSi11 composites

Rys. 6. Wykresy ściskania porowatej ceramiki i kompozytów Al_2O_3/ AlSi11

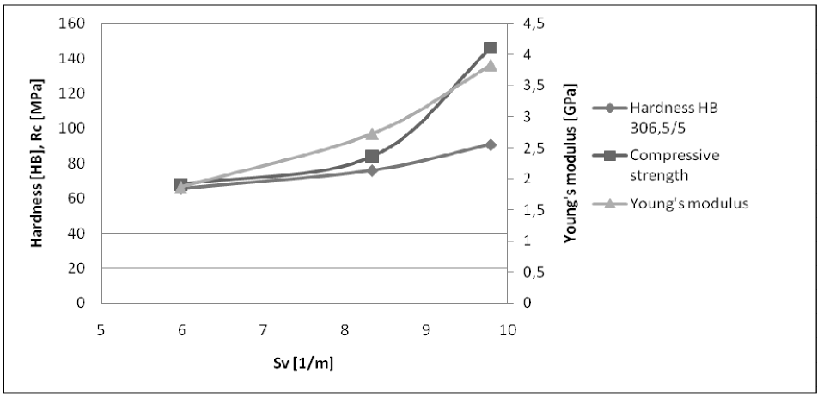


Fig. 7. Effect of specific surface fraction of interphase boundaries on mechanical properties of Al_2O_3/ AlSi11composites

Rys. 7. Wykresy zależności właściwości mechanicznych od względnej powierzchni granic ziaren (S_v) kompozytów Al_2O_3/ AlSi11

The results of the Brinell hardness tests of the Al_2O_3/ AlSi11 composites are shown in Table 4. The hardness of the composites grew together with increasing quantities of interphase boundaries. The ceramics with a higher fraction of interphase boundaries had a higher resistance while pressing a steel bullet, used for the Brinell hardness tests. Composites with a greater number of thin walls have a higher hardness than those with a smaller number of thick walls.

Young's modulus (E) was determined as a slope coefficient of the stress-strain curve within the elastic range. An increase of the fraction of interphase boundaries caused an increase of Young's modulus (Tab. 4). The composite obtained by infiltration of the preform with the smallest pores by the aluminium alloy exhibits the highest Young's modulus.

The influence of the fraction of interphase boundaries was determined by the relation of mechanical properties of Al_2O_3/ AlSi11 composites, such as hardness HB, compressive strength and Young's modulus as a function of the specific surface fraction of the interphase boundaries (*Sv*) (Fig. 7).

The hardness HB, compressive strength and Young's modulus increase with an increase of the S_v parameter. The curves have exponential character. A double increase of the fraction of interphase boundaries causes closely twice an increase of the mechanical properties of the composites with percolation of the microstructure.

CONCLUSIONS

Ceramic-metal composites, obtained via pressure infiltration of porous Al_2O_3 ceramics by cast EN AC-AlSi11 (AK11) aluminium alloy are characterized by a large degree of filling up pores by the metal. As a result of ceramics infiltration, composites of two interpenetrating phases are obtained. The obtained microstructure gives the composites high mechanical properties together with the ability to absorb strain energy.

The mechanical properties of the composites depend on the specific surface fraction of the interphase boundaries (S_v) and the degree of infiltration. The composite obtained via infiltration of the ceramics preform with the smallest pores (150 μm) has the highest value of compressive strength, hardness and Young's modulus. It was found that the energy absorption ability of the composites increases treble with the growth of the fraction of interphase boundaries.

Due to combining ceramics and metal, composites with higher mechanical properties compared to porous ceramics can be obtained. Moreover such composites do not lose their cohesion during compression, while the ceramics samples were totally broken.

It was proved that the developed technology of fabricating composite material with a ceramics matrix infiltrated by aluminium alloy ensures the required microstructure, and moreover, the obtained composite materials can be applied in practice.

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REFERENCES

[1] Sobczak J., Wojciechowski S., Współczesne tendencje praktycznego zastosowania kompozytów metalowych, Kompozyty (Composites) 2002, 2, 3.

[2] Binner J., Chang H., Higginson R., Processing of ceramic-metal interpenetrating composites, Journal of the European Ceramic Society 2009, 29, 837-842.

[3] Sobczak J., Kompozyty metalowe, Wydawnictwo Instytutu Odlewnictwa i Instytutu Transportu Samochodowego, Kraków-Warszawa 2001.

[4] Scherm F., Völkl R., Neubrand A., Bosbach F., Glatzel U., Mechanical characterisation of interpenetrating network metal-ceramic composites, Materials Science and Engineering A 2010, 527, 1260-1265.

[5] Pampuch R., Kompozyty ceramiczne, Kompozyty (Composites) 2002, 2, 3, 10-14.

[6] Szafran M., Konopka K, Rokicki G, Lipiec W., Kurzydłowski K.J., Porowata ceramika infiltrowana metalami i polimerami, Kompozyty (Composites) 2002, 2, 5, 313.

[7] Potoczek M., Śliwa R.E., Myalski J., Śleziona J., Kompozyty metalowo-ceramiczne wytwarzane przez infiltrację ciśnieniową metalu do ceramicznej preformy o budowie piany, Rudy Metale 2009, 54, 11.

[8] Śleziona J., Podstawy technologii kompozytów, Wydawnictwo Politechniki Śląskiej, Gliwice 1998, 28.

[9] Sulima I., Mikułowski B., Wytrzymałość i struktura granicy rozdziału połączeń AlSi11/Al_2O_3, Inżynieria Materiałowa 2005, 6, XXVI.

[10] Pagounis E., Talvitie M., Lindroos V.K., Influence of the metal/ceramic interface on the microstructure and mechanical properties of hiped iron-based composites, Composites Science and Technology 1996, 56.

[11] Konopka K., Olszówka-Myalska A., Charakterystyka powierzchni międzyfazowej ceramika-metal w materiale kompozytowym Al_2O_3-Fe, Inżynieria Materiałowa 2004, 3, 140, 149.

[12] Konopka K., Olszówka-Myalska A., Szafran M., Ceramic–metal composites with an interpenetrating network, Materials Chemistry and Physics 2003, 81, 329-332.

[13] Ozięblo A., Jaegerman Z., Traczyk S., Dziubak C., Porowata ceramika do wytwarzania kompozytowych materiałów metalowo-ceramicznych metodą infiltracji ciśnieniowej ciekłymi stopami aluminium, Szkło i Ceramika 2006, 57.

[14] Staronka A., Holtzer M., Podstawy fizykochemii procesów metalurgicznych i odlewniczych, Skrypt AGH nr 1228, Kraków 1991.

[15] Szreniawski J., Niedźwiecki Z., Analiza głębokości wnikania ciekłego żeliwa w pory masy formierskiej i wysokości chropowatości powierzchni odlewów, Zeszyty Naukowe Politechniki Łódzkiej 1975, 218, Mechanika, z. 42.

