



ARCHIVES
of
FOUNDRY ENGINEERING

DOI: 10.1515/afe-2017-0021

Published quarterly as the organ of the Foundry Commission of the Polish Academy of Sciences

ISSN (2299-2944)
Volume 17
Issue 1/2017

115 – 120

Preliminary Tests of Cellular SiC/Iron Alloy Composite Produced by a Pressureless Infiltration Technique

B. Lipowska^{a,*}, B. Psiuk^a, M. Cholewa^b, Ł. Kozakiewicz^b^a Institute of Ceramics and Building Materials Refractory Materials Division,
Toszecka 99, 44-100 Gliwice, Poland^b Silesian University of Technology, Department of Foundry Engineering,
Towarowa 7, 44-100 Gliwice, Poland

*Corresponding author. E-mail address: E-mail: b.lipowska@icimb.pl

Received 30.11.2016; accepted in revised form 01.02.2016

Abstract

Preliminary tests aimed at obtaining a cellular SiC/iron alloy composite with a spatial structure of mutually intersecting skeletons, using a porous ceramic preform have been conducted. The possibility of obtaining such a composite joint using a SiC material with an oxynitride bonding and grey cast iron with flake graphite has been confirmed. Porous ceramic preforms were made by pouring the gelling ceramic suspension over a foamed polymer base which was next fired. The obtained samples of materials were subjected to macroscopic and microscopic observations as well as investigations into the chemical composition in microareas. It was found that the minimum width of a channel in the preform, which in the case of pressureless infiltration enables molten cast iron penetration, ranges from 0.10 to 0.17 mm. It was also found that the ceramic material applied was characterized by good metal wettability. The ceramics/metal contact area always has a transition zone (when the channel width is big enough), where mixing of the components of both composite elements takes place.

Keywords: Ceramic-metal composites, Porous ceramics, Cast iron, Silicon carbide

1. Introduction

The main reason for research on developing of metal-ceramic composites is obtaining a material characterised by properties that cannot be achieved by ceramic or metal materials. Modification of the alloy properties by adding ceramics can increase its hardness and rigidity, resistance to tribological wear or decrease creep at elevated temperatures. Ceramics combined with a metal alloy matrix has enhanced resistance to thermal shock and fracture toughness [1].

Metal-ceramic composites are usually obtained by ex-situ and in-situ techniques. These are commonly produced dispersed

composites. Ex-situ composites are most frequently manufactured by the method of mechanical mixing with simultaneous addition of ceramic particles into molten metal or by molten metal infiltration into a densely-packed layer of particles or fibres [2, 3]. As a result, a non-continuous strengthening ceramic phase dispersed in the homogenous matrix of the metal is obtained.

Another solution is to obtain composites characterized by a spatial structure of mutually penetrating cells of the ceramic and metal phase.

Contrary to composites with a dispersed ceramic phase, in this type of materials the properties of the matrix and reinforcement complement each other in a better way [4-8]. One of the methods which allows obtaining this kind of composites is infiltration of

previously prepared ceramic materials having a porous, open structure – so called preforms – by molten metal. Porous ceramics of this type can be obtained e.g. by sintering the fractionated grains [9-11] or gelling a foamed ceramic suspension [12-14]. Another method applied is sedimentation of a ceramic slip on a foamed polymer base [8]. It involves impregnating the elastic organic foam with a polymodal suspension containing ceramic particles in such a way that only the bridges surrounding the pores in the foam are covered. Another way of using a foamed polymer base is making a porous ceramic preform so that its voids are a mapping of the polymer matrix, which is fully immersed in the ceramic suspension and, next, fired [15].

In the case of composites containing a ceramic phase in both a dispersed and continuous form, an important issue is to properly join it with the metal phase. The quality of this joint, determining good transfer of loads between components, depends mainly on the ceramic surface wettability by the molten metal [16]. The problem related to the lack of wettability in the ceramic-metal system is particularly important in the case of obtaining composites by the method of porous preform infiltration without using a higher pressure, in which a vital role is played by capillary forces [17]. A solution is offered by techniques which involve modifying the ceramic surface e.g. by covering it with appropriate coatings (more easily wettable by molten metal) and/or enriching the chemical composition of the metal with additives lowering its surface tension in the liquid phase [18-20].

On the other hand, the durability of the ceramic-metal joint is determined by the structure and durability of the division surface, also referred to as interface surface; it is worth mentioning at this point that the connection can be mechanical (mutual anchoring of materials), adhesive (intermolecular forces of the adhering phases) or diffusive (mixing of both phases' components, which may also result in the formation of a new phase) [6].

Composites reinforced with a ceramic phase in the form of particles or fibres have been produced for many years. These are usually materials which have a matrix of light alloys, containing particles of Al_2O_3 , SiC or SiO_2 , most frequently applied in the automotive or aircraft industry [2, 21]. There are also ongoing works aimed at obtaining composites with an aluminium alloy matrix through molten metal infiltration of preforms in the form of ceramic alumina foams [22].

The least investigated and produced composites are the ones with a matrix made of iron alloys. The limitations in their application are related to technological difficulties resulting mainly from the temperature of processing, which is higher than a typical temperature for Mg or Al alloys.

In the article the results of preliminary tests aimed at obtaining an SiC/iron alloy composite by the method of molten metal infiltration into the porous ceramic shape have been presented.

2. Methodology

2.1. Manufacture of porous ceramic preforms

In the investigations shapes having the dimensions of 20x20x40mm, made by gel casting from a SiC powder

suspension, have been used. SiC F1000 (Carborex - Washington Mills) with the grain size $d_{50}=4,5\pm 0,8\mu m$ and chemical composition: SiC 98.3%, C 0.25%, Si 0.35%, SiO_2 0.6%, Fe 0.1% has been used. Porous shapes have been obtained by pouring the prepared polymer suspension over a sponge with the porosity of 10 pores per inch (ppi) (Fig. 1).

After gelling, the preforms were subjected to thermal treatment at 600°C, in air atmosphere. In such conditions the foam was completely fired, leaving behind a system of channels mapping its shape inside the preform. To widen the channels, for some tests, the polymer foam bars were thickened by covering their surface with a suspension containing an organic binder, which is completely burnt at 600°C. Next the preforms were fired at 1400°C and a porous material with SiC on an oxynitride bonding was obtained (Fig. 2).

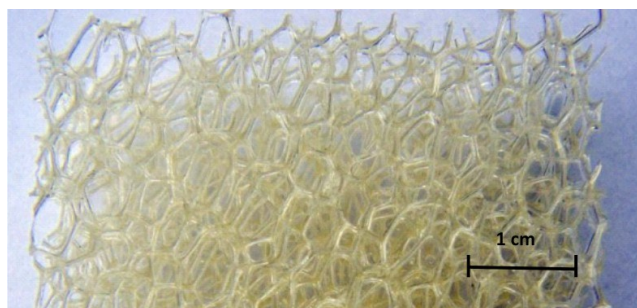


Fig. 1. Polymer foam with the porosity of 10 ppi



Fig. 2. Cross-section of a porous SiC preform

2.2. Molten metal infiltration of preforms

To improve ceramics wettability by molten metal, a water solution of boron and sodium oxides was applied on the preform surface. After drying and thermal treatment at 650°C, the preforms were placed in a casting mould made of synthetic quartz mass with bentonite and subjected to infiltration by grey cast iron with flake graphite having the composition presented in Table 1, at the temperature of 1590°C.

The obtained samples of materials were subjected to macroscopic and microscopic observations as well as investigations into the chemical composition in microareas by means of a Mira 3 scanning electron microscope, produced by Tescan, equipped with an EDS spectroscope (Aztek system produced by Oxford Instruments).

Table 1.

Chemical composition (wt. %) of gray cast iron used for infiltration

C	Si	Mn	P	S	Cr	Ni	Mo	Cu	V	Ti	Al
0.19	0.94	1.57	0.020	0.013	0.32	0.18	0.10	0.06	0.04	0.002	0.03
0.20	0.94	1.57	0.020	0.012	0.32	0.18	0.10	0.06	0.04	0.002	0.17
0.20	0.95	1.56	0.019	0.011	0.32	0.18	0.10	0.06	0.04	0.002	0.15

3. Discussion of results

Based on the macroscopic evaluation of cross-sections of the obtained material samples it was found, that the preforms with channels left behind by the fired, unthickened foam, were not infiltrated, whereas extension of the channels allowed the molten metal permeation. The minimum range of the width of channels filled with metal was established on the basis of microscopic investigations. The image of the composite sample cross-section (Fig. 3 a) indicates that the channels having a minimum transverse dimension of 0.17 mm were infiltrated, whereas metal did not permeate into channels with the dimension lower than 0.1 mm. It was also found that the ceramic material applied was characterized by good metal wettability. The metal-ceramic joint was compact, most frequently without visible cracks.

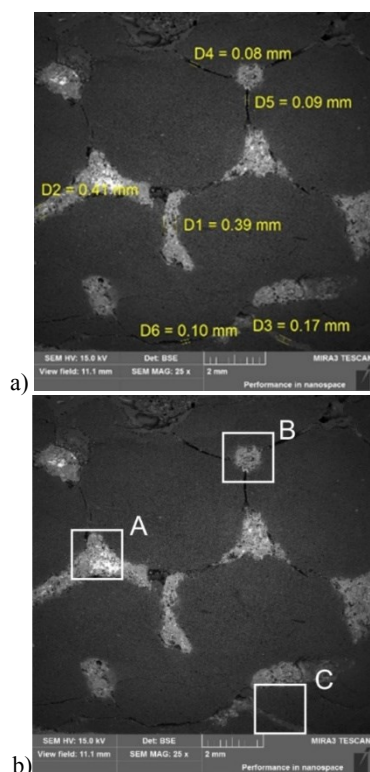


Fig. 3. Microstructure of the composite sample cross-section: a) dimension of channels in the preform, b) selected areas of infiltration subjected to analysis

Selected areas of infiltration with various transverse dimensions of the channels in which infiltration actually occurred (A, B and C in Fig. 3 b) were observed at higher magnifications: 200x and 1000x.

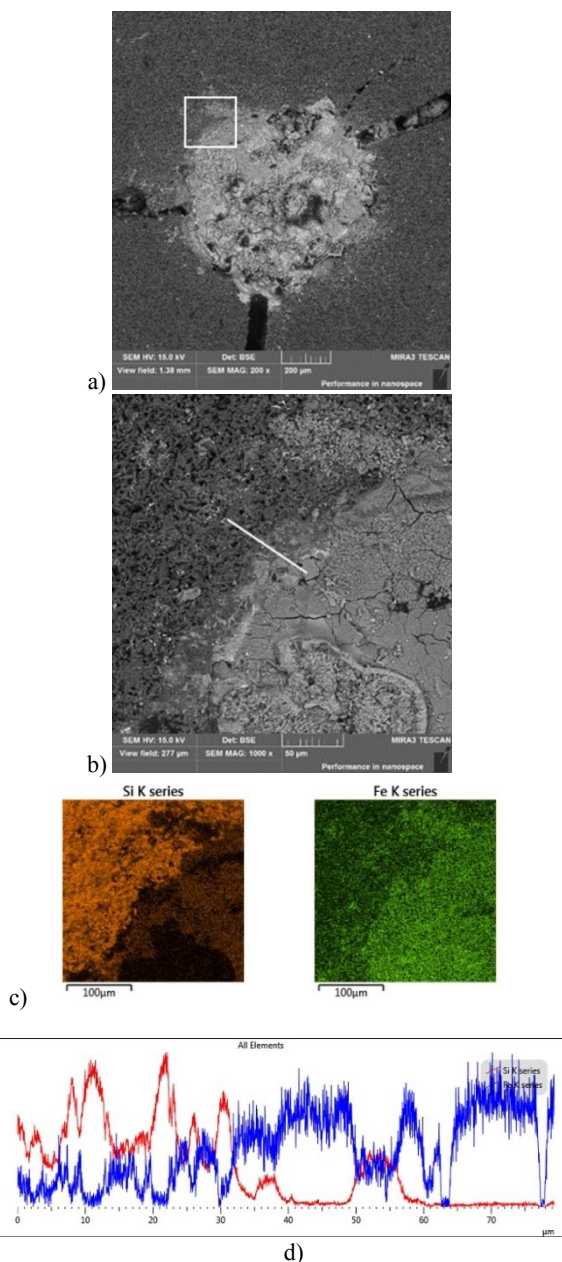


Fig. 4. Area A - BSE micrographs of magnification: a) 200x, b) 1000x and analysis of chemical composition in microareas: c) distribution of Si and Fe, d) linear profiles of Si and Fe contents drew perpendicular to the ceramic-metal contact line

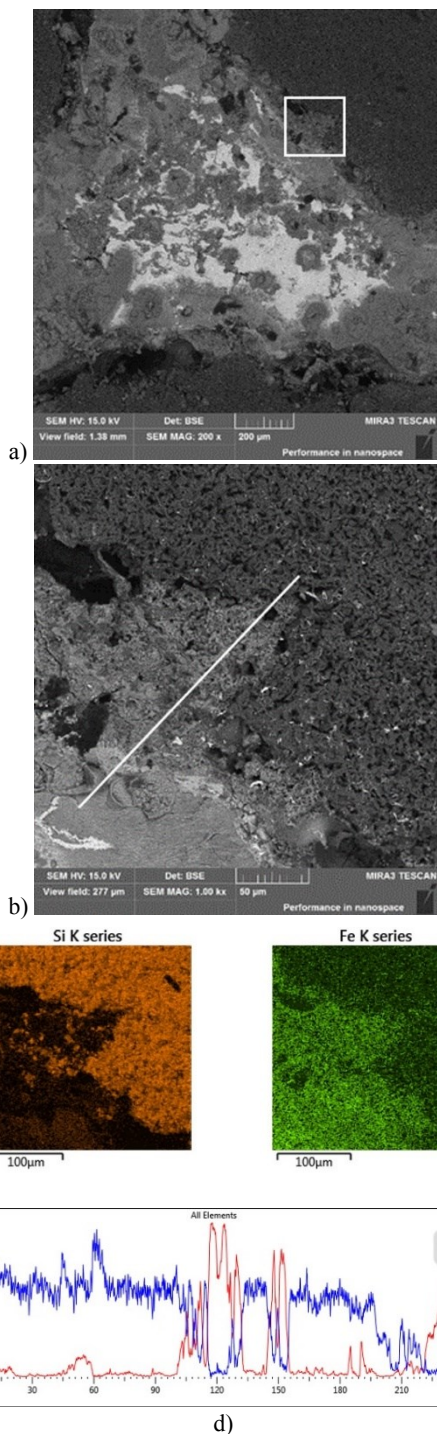


Fig. 5. Area B - BSE micrographs of magnification: a) 200x, b) 1000x and analysis of chemical composition in microareas: c) distribution of Si and Fe, d) linear profiles of Si and Fe contents drew perpendicular to the ceramic-metal contact line

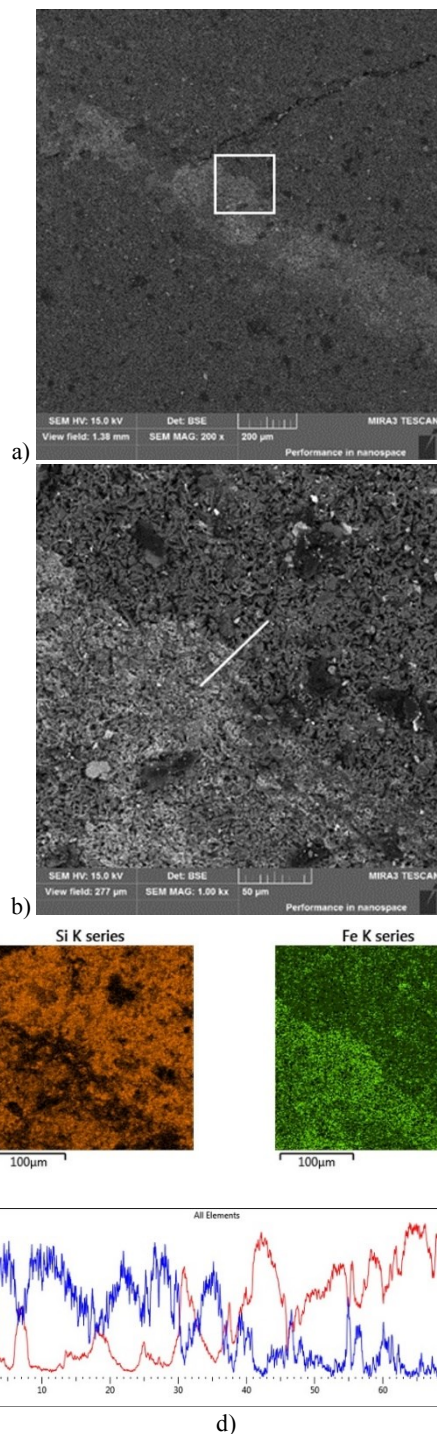


Fig. 6. Area C - BSE micrographs of magnification: a) 200x, b) 1000x and analysis of chemical composition in microareas: c) distribution of Si and Fe, d) linear profiles of Si and Fe contents drew perpendicular to the ceramic-metal contact line

The observations also revealed (Fig. 4-6) that these areas were non-homogenous, forming a transition zone between the components. Based on the analysis of elements' distribution in the microareas, it can be concluded that the transition zone is formed due to the process of mixing the metal components and the ceramic material. The phenomenon of mixing the components in the transition zone is also confirmed by an analysis of chemical composition changes along the set line (linear profiles present changes for Si and Fe – main components of the composite in ceramics and metal, respectively). The transition zone thickness is changeable, ranging from 30 to 120 μm in the areas subjected to observation (Fig. 4 and 5). If the infiltration area is narrower than this range, it already contains components characteristic of the transition zone and the region of pure metal is absent (Fig. 6). Abrupt fluctuations of Si and Fe contents in the transition zone suggest that this is where the components of the ceramic phase and metal are mechanically mixed. Another noteworthy thing is considerable compactness of the area discussed, in particular the lack of cracks on the boundary between ceramic grains and the surrounding metal. It can therefore be presumed that chemical and physical reactions occur in the process of transition phase formation, which results in the formation of new phases on the ceramics/metal boundary. The obtained results confirm good wettability of ceramic grains by molten metal.

Because in this article the only results of preliminary tests aimed at obtaining an SiC/iron alloy composite by the method of molten metal infiltration into the porous ceramic shape have been presented, detailed determining the dominant type of connection in the presented composite will be the subject of further investigations. However, whether the composite components have mainly mechanical, adhesive or diffusive joints, the presented results indicate that it is possible to obtain a stable cellular SiC/iron alloy composite by the method of pressureless infiltration.

4. Summary

Preliminary tests aimed at obtaining a cellular SiC/iron alloy composite with a spatial structure of mutually intersecting skeletons, using a porous ceramic preform have been conducted. The possibility of obtaining such a composite joint using a SiC material with an oxynitride bonding and grey iron with flake graphite has been confirmed.

It was found that the minimum width of a channel in the preform, which in the case of pressureless infiltration enables molten cast iron penetration, ranges from 0.10 to 0.17 mm.

The ceramics/metal contact area always has a transition zone (when the channel width is big enough), where mixing of the components of both composite elements takes place.

The results are the basis for the continuation of metal-ceramic composite materials' research, i.e. mechanical properties or tribological wear behaviours.

Acknowledgements

The work has been financed from the resources for the statutory activity of the Institute of Ceramics and Building Materials – Refractory Materials Division in Gliwice No. 2N010S15 in the year 2015.

References

- [1] Rosso, M. (2006). Ceramic and metal matrix composites: Routes and properties. *Journal of Materials Processing Technology*. 175, 364-375. DOI: 10.1016/j.jmatprotec.2005.04.038.
- [2] Ibrahim, I.A., Mohamed, F.A. & Lavernia E.J. (1991). Particulate reinforced metal matrix composites - a review. *Journal of Materials Science*. 26, 1137-1156. DOI: 10.1007/BF00544448.
- [3] Raether, F. (2013). Ceramic matrix composites – an alternative for challenging construction tasks. *Ceramic Applications*. 1(1), 45-49.
- [4] Clarke, D.R. (1992). Interpenetrating Phase Composites. *Journal of the American Ceramic Society*. 75(4), 739-759. DOI: 10.1111/j.1151-2916.1992.tb04138.x.
- [5] Binner, J., Chang, H. & Higginson, R. (2009). Processing of ceramic-metal interpenetrating composites. *Journal of the European Ceramic Society*. 29(5), 837-842. DOI: 10.1016/j.jeurceramsoc.2008.07.034.
- [6] Szafran, M., Rokicki, G., Lipiec, W., Konopka, K. & Kurzydłowski, K. (2002). Porous ceramic infiltrated by metals and polymers. *Kompozyty*. 2(5), 313-317. (in Polish).
- [7] Mattern, A., Huchler, B., Staudenecker, D., Oberacker, R., Nagel, A. & Hoffmann, M.J. (2004). Preparation of interpenetrating ceramic-metal composites. *Journal of the European Ceramic Society*. 24, 3399-3408. DOI: 10.1016/j.jeurceramsoc.2003.10.030.
- [8] Shouren, W., Haoran, G., Jingchun, Z. & Yingzi, W. (2006). Interpenetrating microstructure and properties of $\text{Si}_3\text{N}_4/\text{Al-Mg}$ composites fabricated by pressureless infiltration. *Applied Composite Materials*. 13(2), 115-126. DOI: 10.1007/s10443-006-9015-x.
- [9] Konopka, K. & Szafran, M. (2006). Fabrication of $\text{Al}_2\text{O}_3\text{-Al}$ composites by infiltration method and their characteristic. *Journal of Materials Processing Technology*. 175, 266-270. DOI: 10.1016/j.jmatprotec.2005.04.046.
- [10] Rödel, J. (2002). Mechanical properties of metal-ceramic composites: Model microstructures, macroscopically homogenous and graded material. *Anales de Macánica del la Fractura*. 19, 13-22.
- [11] Olejniczak, J., Wiśniewski, P., Ciupiński, L., Tarnowski, M., Grabian, J. & Mizera, J. (2012). The investigations on obtaining aluminium-silicon carbide composites. In Conference: XL Szkoła Inżynierii Materiałowej, 24-27 September 2012. Krakow, Poland: AGH University of Science and Technology. DOI: 10.13140/RG.2.1.3196.1368. (in Polish).
- [12] Lemster, K., Delporte, M., Graule, T. & Kuebler, J. (2007). Activation of alumina foams for fabricating MMCs by

- pressureless infiltration. *Ceramics International*. 33, 1179-1185. DOI: 10.1016/j.ceramint.2006.04.002.
- [13] Chang, H., Higginson, R. & Binner, J. (2010). Microstructure and property characterisation of 3-3 Al(Mg)/Al₂O₃ interpenetrating composites produced by a pressureless infiltration technique. *Journal of Materials Science*, 45(3), 662-668. DOI: 10.1007/s10853-009-3983-9.
- [14] Potoczek, M., Myalski, J., Śleziona, J. & Śliwa, R.E. (2009). Gelcasting of alumina foams as preforms for metal infiltration. *Inżynieria Materiałowa*. 30(6), 536-539. (in Polish).
- [15] Lange, F.F., Velamakanni, B.V. & Evans, A.G. (1990). Method for processing metal-reinforced ceramic composites. *Journal of the American Ceramic Society*. 73(2), 388-393. DOI: 10.1111/j.1151-2916.1990.tb06523.x.
- [16] Nogi, K. (2010). The role of wettability in metal-ceramic joining. *Scripta Materialia*. 62, 945-948. DOI: 10.1016/j.scriptamat.2010.03.007.
- [17] Krauß, G., Kübler, J. & Trentini, E. (2002). Preparation and properties of pressureless infiltrated SiC and AlN particulate reinforced metal ceramic composites based on bronze and iron alloys. *Materials Science and Engineering A*. 337(1), 315-322. DOI: 10.1016/S0921-5093(02)00044-8.
- [18] Hashim, J., Looney, L. & Hashmi, M.S.J. (1999). Metal matrix composites: production by the stir casting method. *Journal of Materials Processing Technology*. 92-93, 1-7. DOI: 10.1016/S0924-0136(99)00118-1.
- [19] Gawroński, J., Cholewa, M. & Szajnar, J. (1994). Aluminium – SiC ceramic particles composites. The technology of shape composites production. *WIT Transactions on Engineering Sciences*. 4, 321-328. www.witpress.com. ISSN 1743-3533.
- [20] Cholewa, M., Gawroński, J. (1988). PL 157721. Urząd Patentowy Rzeczypospolitej Polskiej.
- [21] Sobczak, J. & Wojciechowski, S. (2002). The current trends in the practical application of metal matrix composites. *Kompozyty*. 2, 24-37. (in Polish).
- [22] Myalski, J. & Hekner, B. (2015). Aluminium matrix composites reinforced by ceramic foams. *Inżynieria Materiałowa*. 36, 5, 220-223. DOI: 10.15199/28.2015.5.3. (in Polish).