ARCHIVES OF METALLURGY AND MATERIALS
Volume 60 2015 Issue 4
DOI: 10.1515/amm-2015-0444

J. LIGODA-CHMIEL*,#, M. POTOCZEK**, R. E. ŚLIWA*

MECHANICAL PROPERTIES OF ALUMINA FOAM/TRI-FUNCTIONAL EPOXY RESIN COMPOSITES WITH AN INTERPENETRATING NETWORK STRUCTURE

WŁAŚCIWOŚCI MECHANICZNE KOMPOZYTÓW PIANKA KORUNDOWA/ TRÓJFUNKCYJNA ŻYWICA EPOKSYDOWA O STRUKTURZE INFILTROWANEJ

Methods of measuring effective material properties, including Young's, Kirchoff's modulus or Poisson's ratio for composites with an interpenetrating network structure, where the both constituent phases have widely different physical properties, do not lead to an unambiguous interpretation. The commonly- known static methods have the basic disadvantage that higher strain values are needed in order to obtain proper results which is generally impossible to achieve in the case of brittle materials, e. g. ceramics or polymers, as well as for composites created by connecting both these components. The measurement of strain values during the stress test, decreases the values of Young's modulus from several per cent to several dozen per cent, due to appearance of micro fractures in the brittle materials. If there are differences in the values, then a special form and an appropriate amount of samples are needed. Dynamic methods of predicting an effective material properties (ultrasonic and impulse excitation of vibration techniques) are much more accurate, and their non- destructive nature mean that the samples can be used again in other experiments.

This paper uses the traditional compression test and ultrasonic and impulse excitation of vibration methods to compare and analyze the experimental material properties, such as Young's modulus, Kirchoff's modulus and Poisson's ratio using alumina foam/tri-functional epoxy resin composites with an interpenetrating network structure.

Keywords: ceramic-polymer composites, infiltration process, Young's modulus, Kirchoff's modulus, impulse excitation of vibration

Metody wyznaczania stałych materiałowych, m.in. modułu Younga, Kirchoffa oraz współczynnika Poissona kompozytu o strukturze infiltrowanej, w którym obie fazy ciągłe charakteryzują się zupełnie innymi właściwościami fizycznymi, nie są jednoznaczne w interpretacji. Powszechnie znane metody statyczne pomiaru mają tę ujemną stronę, że do otrzymania dokładnych wyników, konieczne są większe odkształcenia, co jest z reguły niemożliwe do osiągnięcia dla materiałów kruchych, tj. ceramicznych lub polimerowych, a tym samym dla kompozytów powstałych przez połączenie tych dwóch komponentów. Pomiar odkształcenia względnego, powstającego podczas przyłożonego naprężenia obniża wartość mierzonego modułu Younga od kilku do kilkudziesięciu procent, z uwagi na pojawiające się w próbce mikropęknięcia. Ze względu na duży rozrzut wyników, metoda ta wymaga specjalnego kształtu i odpowiedniej ilości próbek. Dynamiczne metody wyznaczania stałych materiałowych (metoda ultradźwiękowa, metoda wzbudzania impulsu akustycznego) są znacznie dokładniejsze i nie mają wyżej wymienionych niedogodności, ponadto są to pomiary nieniszczące, pozwalające na ponowne wykorzystanie próbek do innych badań.

W niniejszej pracy dokonano analizy porównawczej wartości eksperymentalnych stałych materiałowych, tj. modułu Younga, modułu Kirchoffa kompozytów pianka korundowa/trójfunkcyjna żywica epoksydowa o strukturze wzajemnie przenikających się faz, otrzymanych tradycyjną metodą statycznego ściskania, a także metodą ultradźwiękową oraz metodą wzbudzania impulsu akustycznego.

1. Introduction

In practice, there are two groups of experimental methods to determine the Young's modulus of composite materials. Static method, such as the compression stress test, allow the stress σ and strain ε curves to be plotted during the mechanical test treatment. These curves make it possible to find the proportionality coefficient between σ and ε, which is Young's modulus. The other static methods most often used in practice are the three-point or four-point bending tests. Dynamic methods, based on the acoustic measurements, allow the elastic modulus values to be determined based on the rate of variation of the ultrasonic waves through the length of the sample, or by measuring the resonant frequency of the random damping vibration [1-4].

---

* RZESZÓW UNIVERSITY OF TECHNOLOGY, FACULTY OF MECHANICAL ENGINEERING AND AEROANAUTICS, 2 W. POLA STR., 35-959 RZESZÓW, POLAND,

** RZESZÓW UNIVERSITY OF TECHNOLOGY, FACULTY OF CHEMISTRY, 2 W. POLA STR., 35-959 RZESZÓW, POLAND,

Corresponding author: jligoda@wp.pl

It is well-known that the values of the elastic modulus of composite materials obtained from dynamic methods are higher than the values received using static methods [1]. This may be connected with relaxation phenomena occurring in the sample during the measurements. In acoustic methods, the loads are applied for shorter than the relaxation time and the measured elastic modulus is close to the un-relaxed modulus [5-7]. In turn, during static measurements, often the relaxation phenomena nearly always occurred due to the long duration of loading the material. The elastic limit of the material may be also exceeded during static measurements, and the measured E modulus is undervalued. However, during acoustic measurements, the local stresses associated with acoustic waves are always lower than the elastic limit. During static loading, the speed of exceeding the elastic limit of the material depends on the number of defects there are in the tested material. Ultrasonic measurements are insensitive to relatively large defects, because the length of the ultrasonic wave is larger than the size of defects an order of 1-2 mm [1,6], which is a drawback of this group of methods. The basic problem with projects using E modulus for engineering calculation is, therefore, the choice of method to determine it.

Because static measurements come closer to determining the effective working conditions of materials, some authors postulate that static methods should be used to determine Young's modulus, or if resonance methods are to be used, then the values should be reduced by about 80% [6].

2. Experimental procedure

2.1. Materials

To obtain the alumina foam/tri-functional epoxy resin composites, the following materials were have been used:

- alumina foam preforms with a total porosity in the range of 76 - 92% prepared by using the gel-casting method [8];
- tri-funcional epoxy resin Araldite MY0510 from Huntsman; tri-glycidyl para-aminophenol (TGAP); low viscosity (550-850 mPa·s);
- powdered curing agent (with a melting point 80-90°C): 4,4'-diaminodiphenyl sulfone (DDS).

The choice of using tri-functional epoxy resin during to manufacture the ceramic-polymer composites was due, among other things, to its low viscosity (550-850 mPa·s), its good adhesion to ceramics, its low shrinkage (> 2%), its high compressive strength value after curing (130 MPa), as well as its water and fire resistance (self-extinguishing property). Young's modulus of tri-functional epoxy resin is 3.1 GPa.

2.2. Alumina foam/tri-functional epoxy resin composites preparation

The alumina foam/tri-functional epoxy resin composites were prepared by infiltration in a Nüve EV018 manual vacuum oven using a Vacuubrand RZ 2.5 oil vacuum pump under reduced pressure (10 mbar) for approx. 60 minutes at 80°C. The infiltration phase represented a prepared solution of trifunctional epoxy resin and curing agent. The composites accepted the alumina foam with an overall porosity of 76%, 80%, 86% and 92%. After the infiltration process, the alumina foams impregnated with polymers went through the curing processed using a temperature schedule from 80 to 170°C in a specially designed silicon mould, that prevented the polymer from flowing out of the alumina foam, and allowed better heat dissipation when crosslinking the epoxy resin within the pores of the ceramic material process. The cooling process of the composites involved turning off the oven until they reached room temperature.

As a result of impregnating the alumina foams with tri-functional epoxy resin and curing the entire composition, four series of alumina foam/tri-functional epoxy resin composites were obtained. The excess of polymer on the ceramic matrixes were mechanically removed from the samples. The obtained samples were aged at room temperature for seven days, and then dried in an oven at 40°C for 2 hours. Density of the alumina foam/tri- functional epoxy resin composites were determined by using the Archimedes' method.

2.3. Mechanical characterization

The alumina foams and the alumina foam/tri-functional epoxy resin composite samples were subjected to compressive and flexural strength tests. Measurements were performed on the Instron 5982 testing machine. Samples with dimensions 20x10x10 mm (uniaxial compression test) and 70x10x8 mm (three-point bending test) were tested. The speed of the measuring head displacement was 1.5 mm/min. As the result of compressive and bending strength, the average value of the five measurements was taken. The obtained compressive stress-strain curves of alumina foams and alumina foam/tri- functional epoxy resin composites allow to find the proportionality coefficients between σ and ε, which were the additionally calculated Young's modulus of tested samples.

2.4. Elastic property characterization

The elastic properties of alumina foams and alumina foam/tri-functional epoxy resin composites were determined by two independent dynamic methods: the ultrasonic velocity method, testing the transit time of ultrasonic wave through the length of samples, in accordance with ASTM E494 [9], measuring 10x10x20mm, and the resonance method by measuring its natural frequencies to determine the elastic properties of the alumina foams and composites using samples measuring 70x35x8mm by using an RFDA basic resonance frequency and damping analyzer from the Belgian producer Integrated Material Control Engineering in accordance with standard ASTM C1259-01 [10],

The ultrasonic velocity method (Fig. 1), involves testing longitudinal and transverse wave velocities in material with known density and velocity- path thickness. It consist of entering the ultrasonic longitudinal wave impulse with a specific frequency value (2,25 MHz) by the sending-head tool inside of the tested sample. After that, the ultrasonic wave impulse transits through the whole length of sample and received by the receiving-head tool on the opposite side of the sample. The velocity of longitudinal and transverse waves were noted.

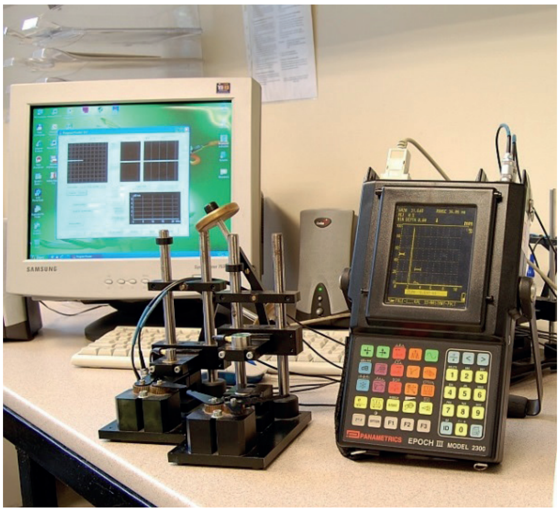


Fig. 1. Ultrasonic velocity measurement devices to determine the elastic properties of materials

The RFDA basic apparatus (Fig. 2) is a measuring system used to determine of selected mechanical properties of materials (Young's modulus, Kirchoff's modulus, Poisson's ratio). The RFDA Basic Software uses powerful algorithms to determine the resonant frequency of vibrations and their damping (damped harmonic motion) after initial stimulation by sending a mechanical impulse through test samples in the shape of cuboids, cylinders and discs. The stimulated longitudinal vibration (to calculate Young's modulus) and transverse vibration (to calculate Kirchhoff's module) are detected by a sensitive microphone with a USB connection, processed in a CPS (digital signal processing) into an electrical signal using the Fourier transform, which is directed to a computer, where it is analysed.

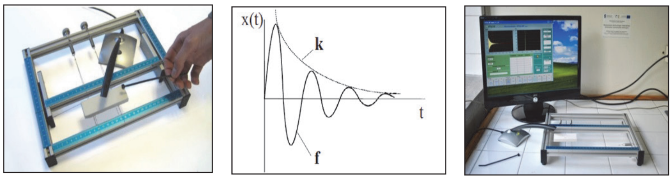


Fig. 2. Scheme of the impulse excitation of vibration technique (resonance method) to determine the elastic properties of materials

The values for Young's modulus, E, were calculated from flexural frequencies, f_{flex}, using the relation:

$$E = 0.9465 \frac{mL^3}{bt^3} T_1 f_{flex}^2$$

where L is the specimen length, b the width, t the thickness, m the mass and $T1$ the geometry constant determined from b and t [10]. The relationship used to calculate shear modulus, G, from the torsional frequency, f_{tors}, was:

$$G = \frac{4mLf_{tors}^2}{bt}\left[\frac{B}{(1+A)}\right]$$

where A and B are geometry constants [10] determined from L, b and t. By determining the Young's modulus and Kirchoff's module, the program calculates the value of Poisson's ratio according to the formula:

$$\nu = \frac{E}{2G} - 1$$

The values of Young's modulus as a result of mixing two phases were also calculated from the mixing rule, to show that these kind of materials don't apply to this law.

3. Results and discussion

3.1. Characterisation of alumina foams

The alumina foam as the matrix of composites, was tested for compression (uniaxial compression test) and bending test (three-point bending test). The compressive strength and flexural strength characteristics of the alumina foams are presented in the form of graphs showing the compressive strength- strain curves (Fig. 3a) and the flexural strength as a function of total porosity (Fig. 3b).

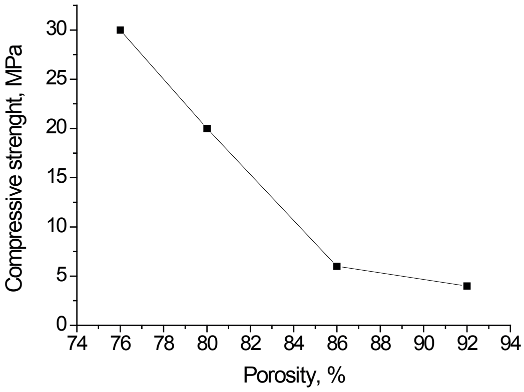


Fig. 3a. Compressive stress - strain curves of the alumina foams.

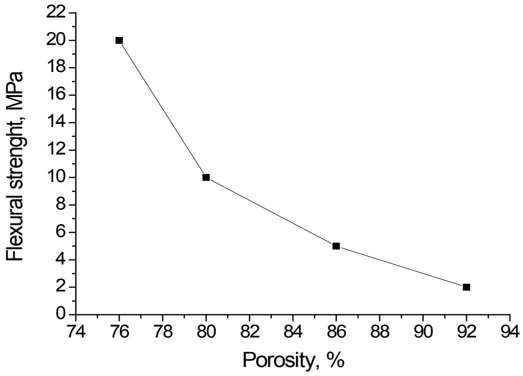


Fig. 3b. Flexural strength of the alumina foams in the total porosity range of them

The porosity, density and mechanical parameters of alumina foams were also summarised in the form of a table (Table 1). These results show that the alumina foams are characterised by good mechanical parameters for as such a type of porous materials while also being low density. The uniaxial compression test and the three-point bending test show that the compressive strength and flexural strength of the alumina foam decreases as porosity increases. The obtained values of the mechanical parameters result from a well- compacted ceramic skeleton formed by the expansion of the alumina slurry with the dissolved agarose. The values of density for alumina foams with total porosity in the range of 76% to 92% was in the range of 0.34 g/cm^3 to 0.98 g/cm^3.

TABLE 1

The physical and mechanical parameters of the alumina foams designed to ceramic-polymer composites manufacture

Porosity of alumina foams, **Pc [%]**	**Density of alumina foams,** **ρ [g/cm^3]**	**Compressive strenght,** **Rc [MPa]**	**Young's modulus,** **E [GPa]**	**Flexural strenght,** **Rg [MPa]**
76	0,98	30	7,0	20
80	0,77	20	2,5	10
86	0,52	6	0,8	5
92	0,34	4	0,2	2

3.2. Characterisation of alumina foam/tri-functional epoxy resin composites

Samples of alumina foam/tri-functional epoxy resin composite were subjected to the test of resistance to compression and bending. The results are illustrated in the form of graphs, showing the compressive strength - strain (Fig. 4a) and flexural strength - strain (Fig. 4b).

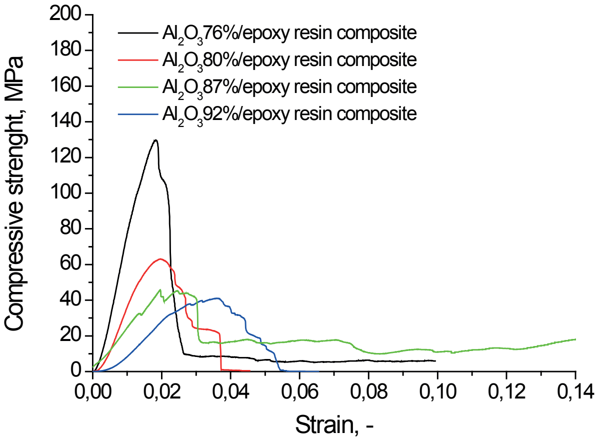


Fig. 4a. Compressive stress - strain curves of the alumina foam/tri-functional epoxy resin composites

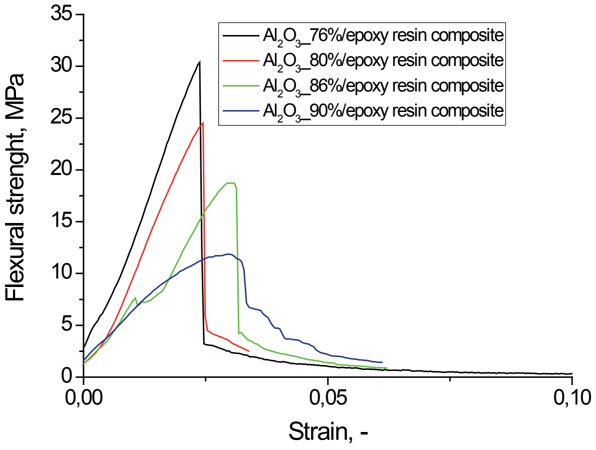


Fig. 4b. Flexural stress - strain curves of the alumina foam/tri-functional epoxy resin composites

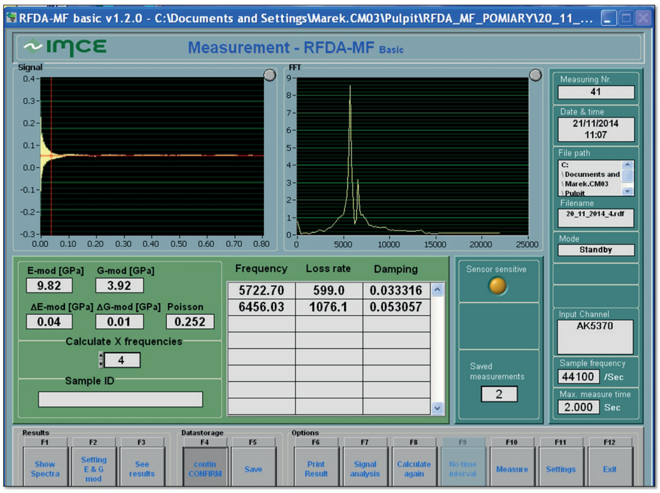
The density of the composites and the results obtained from the static mechanical measurements are also summarised in the form of a table (Table 2). The introduction of epoxy resin into the pores of the ceramic matrix resulted in more than six-fold increase in compressive strength , as compared to the ceramic foam. The epoxy resin displays the characteristics of a brittle body, therefore a high strain was not observed in this composite.

TABLE 2

Density, compressive and flexural strength of the alumina foams/ epoxy resin composites

Porosity of alumina foams **Pc [%]**	**Density of composite,** **ρ [g/cm^3]**	**Compressive strenght,** **Rc [MPa]**	**Flexural strenght,** **Rg [MPa]**
76	1,90	129	30
80	1,72	63	24
86	1,62	45	19
92	1,50	40	12

Elastic properties such as Young and shear modulus and Poisson's ratios of alumina foam/tri-functional epoxy resin composites were determined by two independent methods dynamic, which allowed to determine the Young's and Kirchoff's modulus and Poisson's ratio values based on the velocities of ultrasonic waves passed through the sample (ultrasonic method), or by measuring the resonant frequencies and their damping (resonance technique). The example of the second one's measurement result is presented as the spectrum of the resonant frequencies and their damping (Fig. 5).



RFDA-MF basic v1.2.0 - C:\Documents and Settings\Marek.CM03\Pulpit\RFDA_MF_POMIARY\20_11_...

Measurement - RFDA-MF Basic

Measuring Nr. 41

Date & time 21/11/2014 11:07

File path C: \Documents and \Marek.CM03 \Pulpit

Filename 20_11_2014_4.rdf

Mode Standby

Input Channel AK5370

Sample frequency 44100 /Sec

Max. measure time 2.000 Sec

E-mod [GPa] 9.82

G-mod [GPa] 3.92

ΔE-mod [GPa] 0.04

ΔG-mod [GPa] 0.01

Poisson 0.252

Calculate X frequencies 4

Sample ID

Frequency	Loss rate	Damping
5722.70	599.0	0.033316
6456.03	1076.1	0.053057

Saved measurements 2

Fig. 5. The print screen of the RFDA $_{\text{basic}}$ measurement of the alumina foam/tri-functional epoxy resin composite elastic properties with 92% porosity of alumina preform

Fig. 5

Young's modulus for the alumina foams and the composites were also determined based on the curve of compressive stress-strain from the slope of the straight sections in the range of linear deformation – elastic. The results of the measurements of Young's modules concerning the alumina foams are shown in Fig. 6.

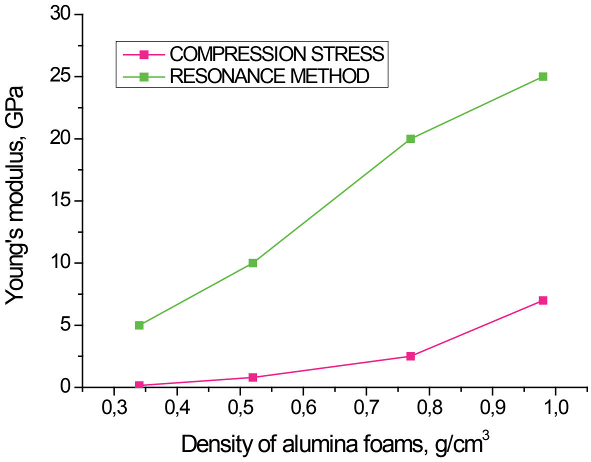


Fig. 6. Young's modulus of the alumina foams based on the compressive strength- strain curves and determined with the resonance method

Young's modulus of composites was also calculated using the basic rule of mixture, namely:

$$E_{composite} = E_{Al2O3} \cdot f_{Al2O3} + E_{epoxy} \cdot f_{epoxy}$$

where the value of Young's modulus for densify ceramic material E=380 GPa, and the measured value of the Young's modulus for tri-funcional epoxy resin is E= 3.1 GPa. These results are presented as the Young's modulus composite density curves (Fig. 7). For a clearer overview of these findings, the measurements were also broken down in Table 3.

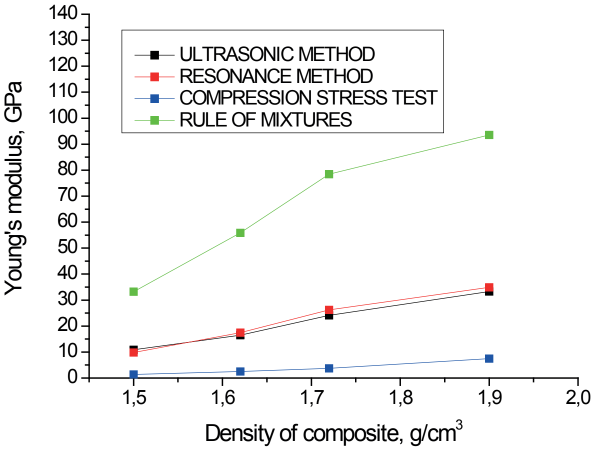


Fig. 7. The comparison of the Young's modulus of the alumina foam/tri-functional epoxy resin composites determined on the compression stress-strain curves and with two independent dynamic methods - ultrasonic and resonance tests. The Young's modulus values calculated from the mixing rule were also submitted.

The results from measurements of Kirchoff's modulus and Poisson's ratio using the ultrasonic and resonance methods were presented in Table 4. The value of Poisson's ratio of alumina material was υ=0.20 and for tri-functional epoxy resin υ=0.35.

TABLE 3

Young's modulus of the alumina foam/epoxy resin composites obtained by several experiments: ultrasonic velocity, impulse excitation of vibration measurements and based on the compression stress-strain curves and using the mixing rule

Density of composite,	**Young's modulus obtained from resonance method,**	**Young's modulus obtained from ultrasonic method,**	**Young's modulus obtained from compression stress test**	**Young's modulus counted from rule of mixtures**
ρ [g/cm³]	**E [GPa]**	**E [GPa]**	**E [GPa]**	**E [GPa]**
1.90	34.9	33.3	7.50	93.56
1.72	26.2	24.1	3.69	78.48
1.62	17.5	16.5	2.50	55.87
1.50	9.8	10.9	1.35	33.25

TABLE 4

Kirchoff's modulus and Poisson's ratio values of the alumina foam/tri-functional epoxy resin composites obtained by using the impulse excitation of vibration and ultrasonic velocity measurements.

Gęstość kompozytu, ρ **[g/cm^3]**	**Kirchoff's modulus obtained by resonance method,** **G [GPa]**	**Poisson's ratio,** υ **[-]**	**Kirchoff's modulus obtained by ultrasonic method,** **G [GPa]**	**Poisson's ratio,** υ **[-]**
1.90	14.5	0.21	13.8	0.21
1.72	10.6	0.23	9.7	0.24
1.62	7.1	0.24	6.7	0.24
1.50	3.9	0.25	4.4	0.25

4. Conclusions

The alumina foams obtained had a total porosity of 76% to 92%. The apparent density of the obtained samples ranged from 0.34 g/cm^3 to 0.98g/cm3. The process for preparing the ceramic slurries, and to inspect the physical and chemical processes during foaming and gelation allowed the produced alumina foam to have a characteristic microstructure.

The created alumina foam with varied geometrical parameters was available as a matrix for infiltration by tri-functional epoxy resin, which allowed composites to be obtained with a higher compressive strength (40 – 129 MPa) and flexural strength (12 – 30 MPa) while retaining low density (1.5 – 1.9 g/cm^3), compared with the results obtained for the ceramic material.

Compression tests showed that the extent deformation of the composite samples was significantly greater than that of the alumina foams, which proves the validity of combining these two components. The higher compressive strength of the alumina foam/tri-functional epoxy resin composite results from the combination of two rigid components. The compressive strength of the composite depends on the porosity of the ceramic matrix, as it decreases, the compressive strength increases.

Three-point bending demonstrated that the flexural strength of the alumina foam/tri-functional epoxy resin composites is several times higher when compared to the values obtained for alumina foams. In the experimental studies it was shown, that the bending stress in the composite material is carried by the ceramic skeleton, and the polymeric filling provides some improving of the flexural strength.

Young's modulus values of composites, obtained by using the dynamic methods, were several times higher than the values of elastic modules obtained from static methods. This could be due to relaxation phenomena occurring in the sample during the measurement, and thus the appearance of the micro cracks in the composite samples during the compression stress test. Young's modulus calculated using of the rule of mixtures law, do not agree with the results obtained experimentally.

The calculated values of Poisson's ratios of alumina foam/tri-functional epoxy resin composites showed that the manner of deformation in the composites under the applied load is influenced by the behavior of the ceramic material acting as the matrix of these types of composites.

Received: 15 September 2015.

Acknowledgements

Financial support of Structural Funds in the Operational Programme - Innovative Economy (IE OP) financed from the European Regional Development Fund - Project No POIG.0101.02-00-015/08 is gratefully acknowledged.

REFERENCES

[1] A.A. Wereszczak, R.H. Kraft, J.J. Swab, Flexural and torsional resonances of ceramic tiles via impulse excitation of vibration, Metals and Ceramics Research Branch, U.S. Army Research Labolatory.

[2] R.C. Bradt, A.N. Scott, Elastic properties of refractories: their roles in characterization, Refractories Applications and News, 3, 2007,

[3] S. Tognana, W. Salgueiro, A. Somoza, A. Marzocca, Measurement of the Young's modulus in particulate epoxy composites using the impulse excitation technique, Mater. Sci. Eng. A **527** (2010).

[4] R.R. Atri, K.S. Ravichandran, S.K. Jha, Elastic properties of in-situ processed Ti–TiB composites measured by impulse excitation of vibration, Mater. Sci. Eng. A **271** (1999).

[5] J. Podwórny, A. Wrona, Nieniszczące metody badań materiałów ogniotrwałych i perspektywy ich rozwoju, [w:] Materiały ogniotrwałe w metalurgii, wytwarzanie, metody badań, zastosowanie. Materiały XI Międzynarodowej Konferencji Hutniczej, pod red. J. Czechowskiego, J. Wojsy, Polskie Towarzystwo Ceramiczne, Kraków 2005.

[6] J.M. Gómez de Salazar, M.I. Barrena, G. Morales, L. Matesanz, N. Merino, Compression strength and wear resistance of ceramic foams–polymer composites, Materials Letters, 60, (2006).

[7] M. Potoczek, Gelcasting of alumina foams using agarose solutions, Ceramics International, 34, (2008).

[8] ASTM E494 – Standard Practice for measuring Ultrasonic Velocity in Materials. Annual Book of ASTM Standards, **3.03**, American Society for Testing and Materials, West Conshohocken, PA, 2000.

[9] ASTM C 1259-01 – Standard test method for dynamic Young's modulus, Shear modulus and Poisson's ratio for advanced ceramics by impulse excitation of vibration. Annual Book of ASTM Standards, **15.01**, American Society for Testing and Materials, West Conshohocken, PA, 2001.