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Flammability and acoustic absorption of alumina foam/tri-functional epoxy resin composites manufactured by the infiltration process

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Abstract

Ceramic-polymer composites with an interpenetrating network structure have a lot of advantages, which can be achieved by connecting two quite different phases. Infiltration of liquid epoxy resin into the cellular alumina matrix is the most suitable way of producing this type of materials and offers an opportunity for a precise and simple fabrication method of ceramic-based interpenetrating composites. Alumina foams manufactured by "gel-casting" method were infiltrated by new tri-functional epoxy resin (triglycidylized para-aminophenol) designed for special aircraft applications. Combination of this new epoxy resin with high-porous alumina foams causes higher compressive strength and better flammability of ceramic/polymer composite than either individual component. Additionally, alumina/epoxy composites with partial pores filling ratio showed satisfactory acoustic absorption characteristics. Development of this type of composites is a result of need to research new, lightweight materials with increased mechanical strength, non-inflammable and another functional features, e.g. noise reduction ability, design for the aerospace industry. Therefore, presented paper investigates flammability, acoustic absorption and mechanical properties of resulted composites.

Keywords: A. Ceramic- matrix composites (CMCs), Foams, Resins; B. Thermal properties, Mechanical properties

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1. Introduction

Composites with an interpenetrating network structure are the relatively new class of functional materials, which demonstrated interesting properties and have a potential to be used in numerous industrial application fields. In the literature there are a lot of examples of ceramic-metal composites with an interpenetrating network structure [1-8], but there is a little information about ceramic-polymer composites, where the polymer acts as reinforcement of ceramic function [9-11]. Moreover, presented works show not efficient research of the complexity of mechanical and functional properties of these materials in the context of potential applications in the aerospace industry or related industries. There are also many studies on epoxy resins which are the matrix of polymer/ceramic or polymer/metal composites, because of their low density, excellent adhesion and good mechanical properties [12-17]. Unfortunately, most of epoxy resins are characterized by low flame resistance. Therefore, many efforts have been made to improve this feature by introduction of different types of flame-retardants into polymer matrix [18-21]. On the other hand, multi-functional (tri-, tetra-) epoxy resins with self-extinguishing seem to be a novelty [22]. An introduction of this type of epoxy resin into the ceramic matrix caused additional improvement of the polymer flame resistance. This result can be achieved by continuity of both phases in the infiltrated ceramics/polymer composites, which is described by Newnham theory [23].

With the use of Newnham taxonomy, which is based on phase connectivity, materials are designed as (3-3) composites since both phases have connectivity in three dimensions [23]. Thus, the development of interpenetrating network composites is a logical step in the evolution of this type of materials that began with the fabrication of particulate (0-3) composites. There are some promising advantages resulting from the interconnectivity of the phases. Each phase contributes its own properties to the final composite, the polymer component increasing strength and fracture resistance, while the ceramic component increasing dimensional stability and flammability [23,24].

One method to achieve an interpenetrating ceramic/polymer composite is the infiltration of the liquid polymer into porous ceramic material with continuous open porosity. Providing that structure of the initial porous material can be controlled sufficiently in terms of the degree of porosity, shape and size of pores, size of connections between them and the nature of strut separating them. There is also an opportunity to design and fabricate interpenetrating composites with customized structures. Hence, the infiltration of porous ceramics offers the potential for producing tailored (3-3) interpenetrating composites [25-26].

The aim of this work was manufacturing- of new alumina/tri-functional epoxy resin composites with an interpenetrated network structure having improved flammability and satisfactory acoustic absorption characteristics. In order to produce ceramic/polymer composites, the alumina foams and tri-functional epoxy resin were used.

The choice of alumina foams was driven by good mechanical properties, thermal and chemical resistance and low production costs of them. The alumina matrixes were manufactured by “gel-casting of foams” method. Gel-casting foams are known to possess higher mechanical strength than those produced by a well-known process called replication method [27].

The special tri-functional epoxy resin (triglycidylized para- aminophenol) designed to aircraft applications was chosen because of its low viscosity, adhesion to ceramic, minimal cramp, high compressive strength, thermal and chemical resistance. The structure of resin and hardener (4, 4’- diaminodiphenylsulfone) provides thermal resistant and satisfactory mechanical strength for entire composition after curing process. These features were not observed in commonly used di-functional epoxy resins. The tri-functional epoxy resin with trade name Aradlite MY0510, combined with alumina foams, is a new composite material for applications which were presented in this work and a new material in the aviation area [28].

2. Experimental techniques

2.1. Materials

To prepare the alumina foam/epoxy composites the following materials were used:

- alumina foams (Al_2O_3) manufactured by the gel-casting method and having different porosity level in the range of 76 - 92%;
- tri-funcional epoxy resin; tri-glycidyl para-aminophenol (TGAP) with trade name Araldite MY0510 (Aldrich) and diamino diphenyl sulphone (DDS) with trade name Aradur 976-1 (Aldrich) as the curing agent.

2.2. Alumina foams preparation by the gelation technique

This method is a combination of foaming of ceramic suspension and stabilizing the foam structure. The essential components of the process were alumina powder (Al_2O_3) with an average particle size 0.7μm (CT 3000 SG, Almatis, Germany), agarose as a gelling agent (Fluka Biochemika), foaming agents (Tergitol TMN-10, Aldrich, Germany and Simulsol SL-26, Seppic, France), dispersant (Darvan 821A, R.T. Vanderbilt Company Inc. U.S.A.) and demineralised water. The foam was prepared by mechanical frothing of ceramic suspension by a high share mixer. The next steps of the process were: gelling, drying, calcination of the organic binder and sintering for 2 h at 1575°C. This technique allows producing the ceramic foams with porosity ranging from 60 - 95% [29].

The microstructure of the alumina foams was observed by scanning microscopy (Jeol JSM-550 LV). The total porosity and density of prepared alumina foams were measured by Archimedes' method. Theoretical density of fully densified alumina (3.98 g/cm^3) was used as a reference to calculate the total volume fraction of porosity.

2.3. Composites processing and preparation

Tri-functional epoxy resin (TGAP) was used for manufacturing alumina/epoxy resin composites by the infiltration of liquid polymer and curing agent (DDS) solution into alumina foams (Al_2O_3). Infiltrating phase was prepared by mixing resin and curing agent with appropriate amounts in plastic or glass beakers. Because of the curing agent powder structure (particles size ~150 µm), it was necessary to dissolve it in epoxy resin, which was done by mixing epoxy resin and curing agent at 80°C.

Next, dried ceramic foams were placed in the crystallizers and dipped in the earlier prepared solution of epoxy resin and curing agent. The specimens were saturated for 60 minutes in the vacuum chamber under 1 bar pressure at 80°C (Fig. 1). After foams infiltration, the cure process was carried on at atmospheric pressure conditions. The cure temperature was controlled in the range of 80 - 175°C, according to the manufacturer’s recommendation. Cure experiments were made in special silicone forms in order to avoid polymer outflowing from porous matrixes and decrease an exothermic reaction.

Insert Fig.1

After curing procedure, all the samples were seasoned at room temperature for 7 days and then resin additions were removed from ceramic matrixes by mechanical tooling.

The density and the pore filling ratio of alumina/tri-functional epoxy composites were determined by using Archimedes’ method. The pore filling ratio (F_r) was given by:

$$F_r = \frac{Po - Pp}{Po} \cdot 100\% \quad (1)$$

where *Po* and *Pp* were the open porosity of alumina foams and open porosity of alumina/tri-functional epoxy resin composite, respectively.

Microstructure of the cross-sectioned alumina/tri-functional epoxy resin composites was observed by scanning electron microscopy (Joel JSM-55-LV).

The flammability test was performed in accordance with the IEC 60695-11-10 standard [30]. The samples were supported in a horizontal position and tilted at 45°. The flame was applied to the end of the specimen with 1 inch mark for 30 seconds, after that, the burner was taken off and the burning time of each sample was observed. Epoxy bars were also tested, for comparison.

The acoustic absorption measurements of alumina/tri-functional epoxy resin composites and alumina foam alone, for comparison, were performed using the so-called transfer function method in accordance with ISO 10534-2:1998 standard [31]. The measurement was performed from 500 to 6400 Hz. The alumina foam and composites samples were cylinders 29 ± 0.1 x 15 ± 0.1mm^2 with total porosity of alumina foam matrix 88%. Each material was measured five times. Acoustic absorption capacity depends on the open porosity of material. It should be noted, that alumina/epoxy composites had a not-fully filled structure in this experiment. It was due to obtaining higher acoustic absorption of partially filled alumina/epoxy composite than that of fully-filled composites. The partial pore filling ratio was controlled by modification of the infiltration time of alumina foams by tri-functional epoxy resin.

The reported compressive strength of the interpenetrating alumina/tri-functional epoxy composites was the average of at least five determinations. The rectangular-shaped samples for compression test had a length of 20 mm and thickness of 10 mm. The cross head speed was 1.5 mm/min for all the samples. A universal testing machine Instron 8080 was used.

3. Results and discussion

3.1. Characterization of alumina foams

The total porosity of alumina foams was found to be from 76 to 92%. The difference between total and open porosity was less than 1% (Table 1). This fact is really important in the infiltration process, where open pores connectivity in the ceramic foam structure is needed to introduce a polymer solution. The density of alumina foams varied between 0.39 and 1.02 g/cm^3 (Table 1).

Insert Table 1.

Fig. 2. shows the microstructure of alumina foams with porosity ranging from 76 to 92%. The alumina foams were typically composed of approximately spherical macropores, so-called "cells", interconnected by large interpore connections, so-called "windows". The presence of windows makes the foam able to infiltrate liquid polymer. The average cell size and window size increased with increasing the porosity level of the foam. Within the porosity range of 76 - 92% the average sizes were found to be 160 - 460 μm and 25 - 130 μm, respectively.

Insert Fig. 2.

3.2. Characterization of alumina/tri-functional epoxy composites

The composites obtained by tri-functional epoxy resin infiltration into the alumina foams within total porosity range 76 - 92% were characterized by low density and high degree of pore filling ratio (Table 2).

Insert Table 2.

The microstructure of alumina/epoxy resin interpenetrating composites is presented in Fig. 3. Due to open cell structure of the alumina foams, macropores in alumina preforming in whole porosity range (76 - 92%) were completely filled by polymer (Figs. 3a and 3b). Alumina foams with 76% total porosity are characterized by open porosity yet. Below 76%, the presence of closed pores is observed and complete filling of the ceramic matrix is not possible [29]. The fracture-section of the composite (Fig. 3c) shows that the dense walls of alumina cells do not allow the epoxy resin to infiltrate but leave fully dense struts in the composite to form a three-dimensionally continuous ceramic phase while the polymer phase forms the other. "In situ" polymerization of epoxy resin into macropores of ceramic foam appeared to provide a sound interface between the epoxy resin and the ceramic strut. The observations of the composite microstructure show the continuous polymer phase in the alumina foam matrix, with good adhesion at the interface of alumina /epoxy resin at the same time. The microscopic analysis, including the assessment of two components connection quality seems to be satisfactory and allows to adequately assessing the connection and microstructure of the ceramic - polymer composite. The quality of the adhesive connection has an influence on mechanical features. In order to solve the flammability and acoustic absorption issues, the connection assessment based on the microscopic examination analysis [32-36] were presented in this paper.

Insert Fig. 3 a,b,c

3.3. Flammability of alumina/tri-functional epoxy composites

One of the reasons to connect ceramics with polymers is the improvement of polymer component flammability. Tri-functional epoxy resin is classified as a self-extinguishing material. The flammability test showed that samples stop burning after 10 s of taking off the burner from the resin bars. Additionally, the head of flame did not reach the 1 inch mark (Fig. 4a). The self-extinguishing property of tri-functional epoxy resin is the result of it and hardener structure. The presence of the sulfur atom in 4,4'-diaminodifenylosulfonu (DDS) and the epoxy group linked with the p-aminophenol nitrogen atom, reduce the flammability of this cured system.

The infiltration of tri-functional epoxy resin into the alumina foams structure with porosity range 76 - 92% additionally inhibited the burning process. The flammability of alumina/epoxy composites was increased with decreasing the porosity in alumina foam matrix (Fig. 4b). The best result was obtained for alumina/epoxy composite based on the alumina foam matrix with total porosity of 76% (Fig. 4b). In this case ceramic material with the smallest pore size provided the best protection by creating some kind of buckler, where the flame cannot be easily spread. The presence of cells and windows with the smallest diameters makes that only resin formed on the wall of the composite is burned, the flame does not penetrate to the inside and thus is quickly extinguished. Composites based on the alumina foam with porosity 92% show slower flame extinguishing, but the area of burn is comparable to the result obtained for the pure resin. Burning time decreases with increasing of volume fraction of the alumina foam. It is caused by decreasing the sizes of pore and the connections between them, along with decrease of alumina foam porosity, which acts a matrix function.

Insert Fig. 4.

3.4 Mechanical characteristics of alumina foams and alumina/tri-functional composites

The main disadvantage of ceramic foam material is its brittleness. The compressive stress-strain plot shows a typical behavior for ceramic foams, i.e. subsequent brittle crushing and densification following the fractures [24]. Within the porosity range of 92 – 76% the compressive strength was found to be from 5 to 30 MPa, respectively (Fig. 5). There is a simple relationship between porosity and compressive strength, when the porosity increases, the compressive strength decreases. The obtained compressive strengths are not sufficient for load-bearing parts. Nevertheless, the applied gel-casting technique resulted in highly porous materials with better mechanical strength than those obtained by other routes. It was caused by the spherical shape of large pores associated with a densified polycrystalline matrix of the material walls.

Insert Fig. 5

The infiltration of epoxy resin into alumina foams improved the mechanical strength of the material. The strength-strain plots for alumina/epoxy composites are shown in Fig. 7. Compressive strength of the composites was found to be 4 - 7 times higher than that obtained for the alumina foams (Fig. 6). The best result was obtained for alumina/epoxy composite with total porosity of alumina matrix 76%, which showed maximum strength of 130 MPa. For alumina/epoxy resin composite with total porosity of alumina matrix 92%, this value was of 40 MPa. The maximum stress carried by composites is significantly higher in compare to the results obtained for ceramic foams. These results indicate that it depends on the porosity and pore size of the ceramic matrix.

Insert Fig.6

3.5. Acoustic absorption of alumina foam and alumina/tri-functional epoxy composites

Ceramic foams are able to absorb the sound even with 99%. The size and distribution of foam cells make that sound with different frequency is absorbing with different effectiveness. If the body has a porous structure, air vibrations are suppressed as a result of the multiply reflection created by the tubular walls [33, 34]. Acoustic absorption plots of the alumina foam and the alumina foam/epoxy resin composites which have open pore structure are given in Fig. 7. The open-cell alumina foam with open porosity of 88% has a characteristic absorption peak of 90 - 99%, around 2000 - 3000 Hz. For higher frequencies (3500 – 6400 Hz), the acoustic absorption is around 80%. On the other hand, the alumina foam epoxy resin composites with open porosity of 12 and 37% are characterized by absorption peaks of approximately 25 – 80% around 1500 – 3000 Hz. For higher frequencies (3500 – 6400 Hz) the acoustic absorption of alumina/epoxy composites with open porosity of 37 and 12% are found to be 20 and 40%, respectively (Fig. 7). The partial pore filling ratio in alumina/epoxy resin composites appears to provide several open channels inside the composite specimen, where the sound energy can enter and attenuate as in the case of open cell ceramic foam. With an increase in open-cell volume in composites samples there is an increase in the number of connected porous channels; hence higher acoustic absorption was observed.

The acoustic absorption characteristics of alumina/tri-functional epoxy composites are not satisfactory in comparison to alumina foam results. However, in conjunction with high mechanical strength of the composites they can be used in this area where the increased mechanical strength is needed.

Insert Fig.7

4. Conclusions

Introduction the tri-functional epoxy resin with a low viscosity into the alumina foams and polymerization inside the ceramic foams allows obtaining the composites with full filling and good adhesion at the ceramic/polymer boundary.

The most positive property of alumina foam/tri-functional epoxy resin composite was their lower burning range in compare to the cured tri-functional epoxy resin. The flammability test showed that composites based on the alumina foams with the smallest porosity – 76% are the most suitable for fire barriers, because of the shortest burning time and self-extinguishing properties.

Alumina foam/epoxy resin composites were characterized by higher compressive strength than alumina foams, which is a result of connection two rigid components. The maximum compressive strength values for composites were in the range of 40 - 120 MPa. These results indicate that the stresses transmitted by the composite depend on the porosity of ceramic matrix. In addition, the extent deformation of composites samples was observed, which shows the rightness of connection of these two components.

The alumina/epoxy resin composites with open pore structure showed an ability of acoustic absorption in some frequency ranges. An acoustic absorption coefficient increased with increasing of the open porosity in the composite.

5. Acknowledgments

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Caption for Figures:

Fig. 1. Scheme of infiltrating and curing processes.

Fig. 2. Microstructure of the alumina foams having total porosity of: a) 76%, b) 80%, c) 86%, d) 92%.

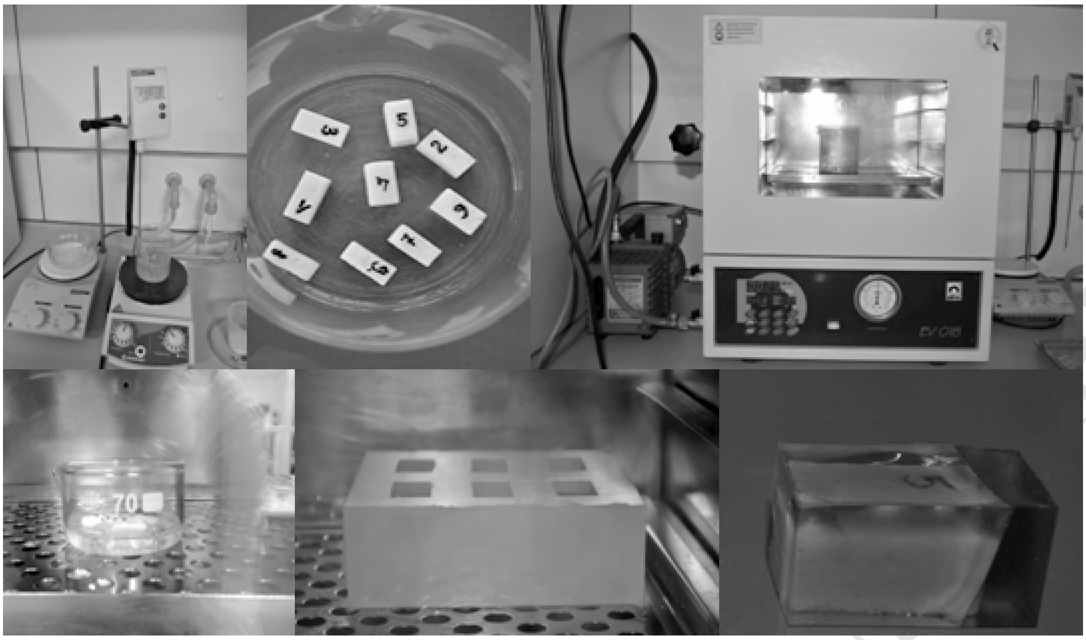
Fig. 3. Microstructure of alumina foam/tri-functional epoxy resin composites obtained by infiltration of tri-functional epoxy resin into alumina foams having porosity of: a)76%, b) 92%, c) alumina matrix/epoxy resin connection.

Fig. 4. a) Tri- functional epoxy resin bars before and after flammability test, b) alumina/tri-functional epoxy composites based on cellular alumina matrixes with total porosity of: (1) 92%, (2) 86%, (3) 76% after flammability test.

Fig. 5. Compressive stress - strain curves of alumina foams having porosity of: (1) 76%, (2) 80%, (3) 86%, (4) 92%.

Fig. 6. Compressive stress - strain curves of alumina/tri- functional epoxy composites obtained by infiltration of tri-functional epoxy resin into alumina foams having porosity of: (1) 76%, (2) 80%, (3) 86%, (4) 92%.

Fig. 7. Acoustic absorption-frequency curves for alumina foam and composite samples. S1- alumina foam having open porosity of 88%, S2- alumina/epoxy composite having open porosity of 34% (pore filling ratio 61%), S3- alumina/epoxy composite having open porosity of 12% (pore filling ratio 86%)



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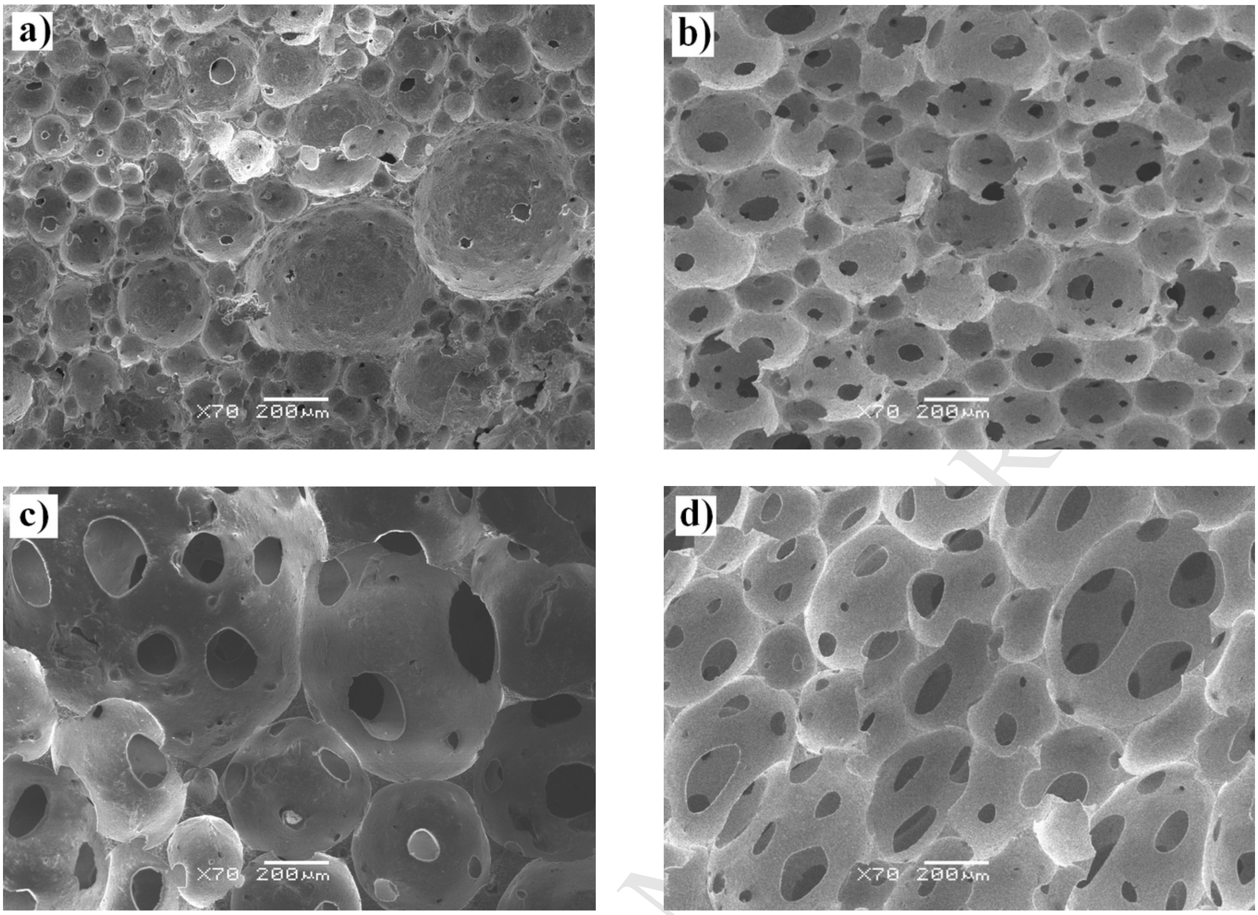

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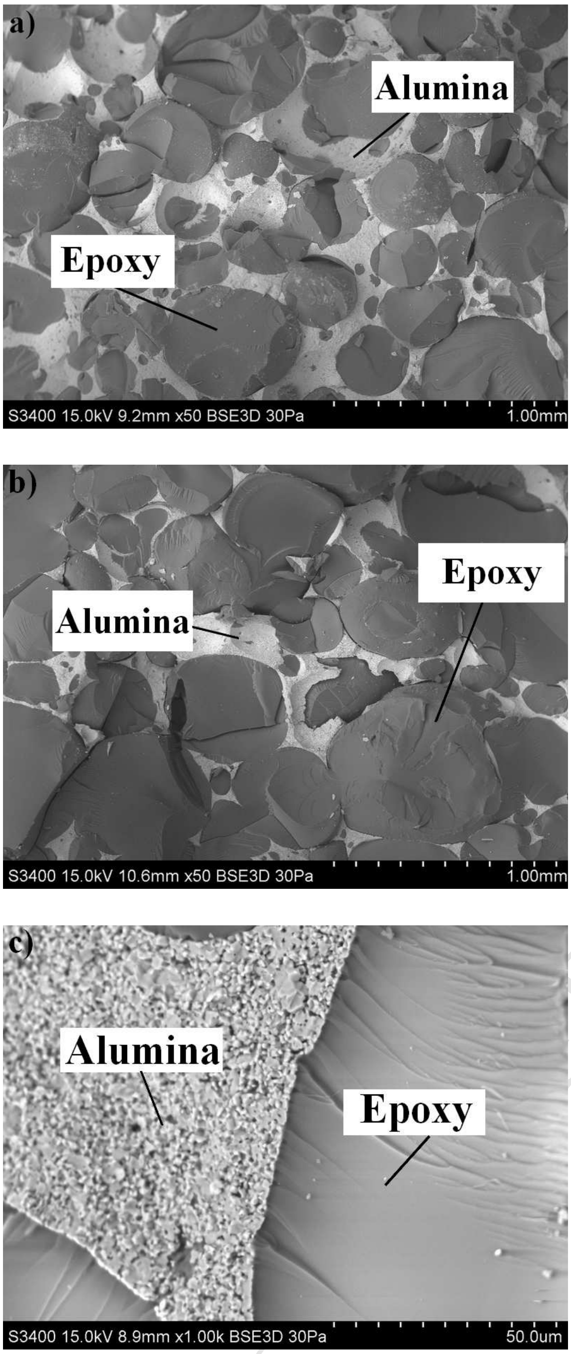

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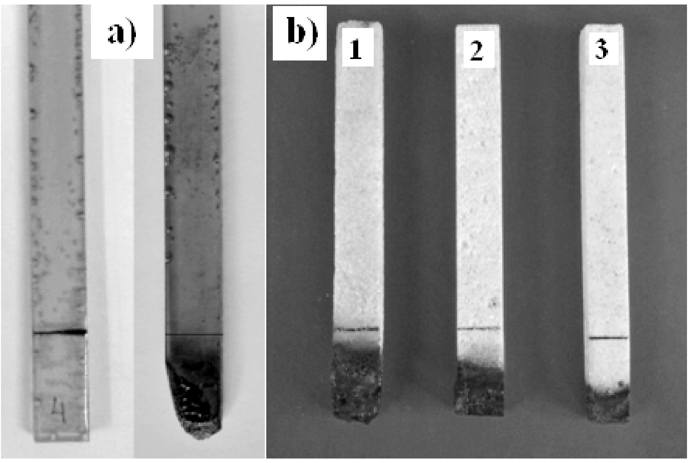

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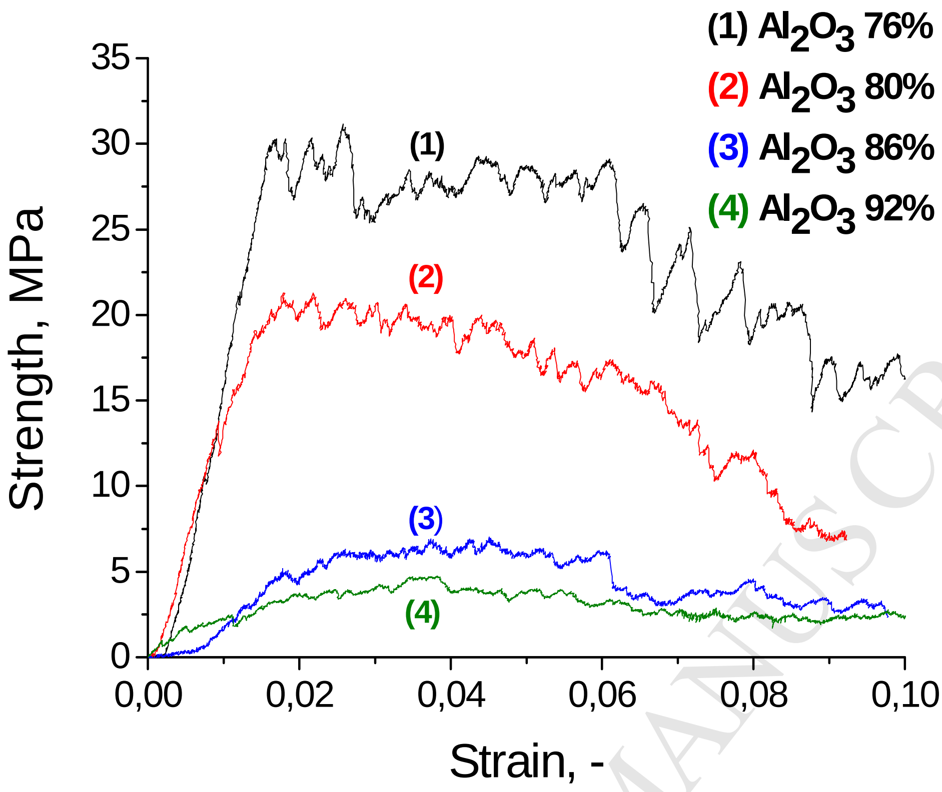

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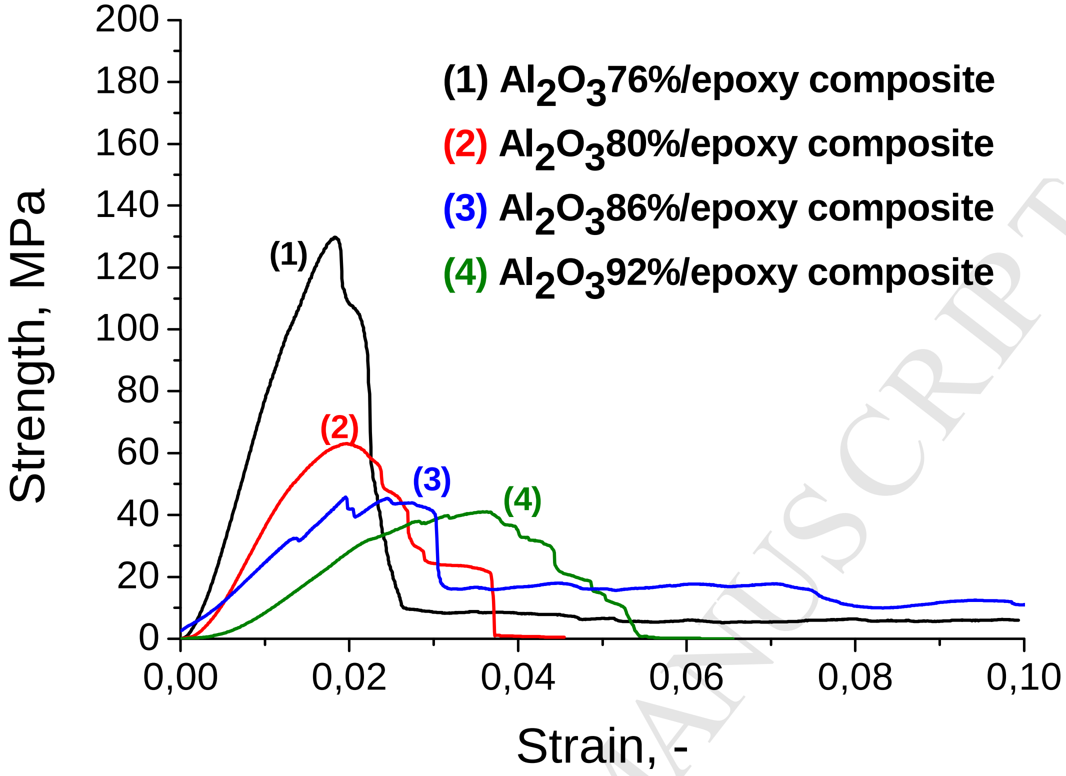

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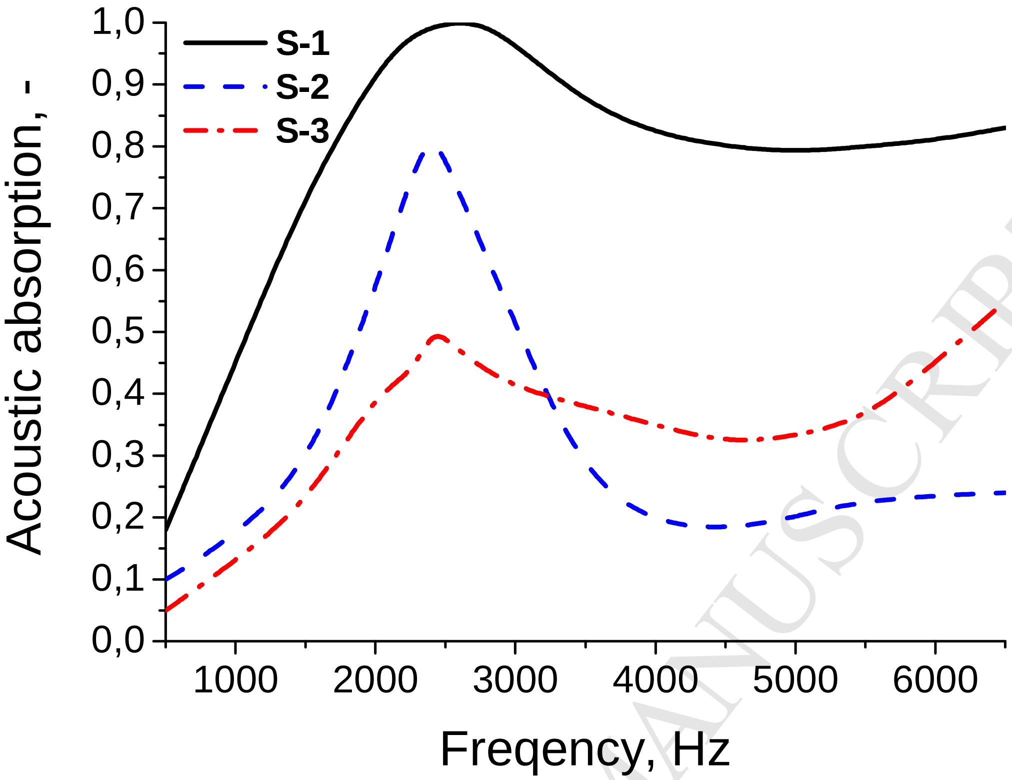

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Caption for Tables:

Table1. Total porosity and density of alumina foams.

Table 2. Density and pore filling ratio of alumina/tri-functional epoxy composites based on alumina foam matrixes with total porosity in the range of 76 - 92%.

Table1. Porosity and density of alumina foams.

Porosity, %		Density, g/cm^3
Total	Open	
75,9	75,7	1,02
80,4	80,1	0,79
86,1	85,6	0,59
92,3	91,6	0,39

Table 2. Density and pore filling ratio of alumina/tri-functional epoxy composites based on alumina foam matrixes with total porosity in the range of 76 - 92%.

Total porosity of alumina foam matrix, %	**Density , g/cm^3**	**Pore filling ratio, %**
76	1,90	98
80	1,72	98
86	1,62	99
92	1,50	99