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Fabrication and characterization of composite materials based on porous ceramic preform infiltrated by elastomer

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Abstract. The paper presents the experimental results of fabrication and characterization of ceramic- elastomer composites. They were obtained using pressure infiltration of porous ceramics by elastomer As a result the composites in which two phases are interpenetrating three-dimensionally and topologically throughout the microstructure were obtained. In order to enhance mechanical properties of preforms a high isostatic pressure method was utilized. The obtained ceramic preforms with porosity gradient within the range of 20–40% as well as composites were characterized by X-ray tomography. The effect of volume fraction of pores on residual porosity of composites was examined. These results are in accordance with SEM images which show the microstructure of composites without any delaminations and voids. Such composites exhibit a high initial strength with the ability to sustain large deformations due to combining the ceramic stiffness and rubbery elasticity of elastomer. Static compression tests for the obtained composites were carried out and the energy dissipated during compression was calculated as the area under the stress-strain curve. The dynamic behavior of the composite was investigated using the split Hopkinson pressure bar technique. It was found that ceramic-elastomer composites effectively dissipate the energy. Moreover, a ballistic test was carried out using armor piercing bullets.

Key words: ceramic-matrix composite, ceramic, mechanical properties, damage mechanics, scanning electron microscopy.

1. Introduction

For many years successive growth of the interest in composites has been noticed. The progress is determined by continued demand for development of stiffer, lighter and stronger materials. Most works have focused on particle- or fiber reinforced composites [1–3]. Because of their properties, these materials are suitable for many applications. Additionally, they are characterized by very good stiffness, wear resistance and compressive strength by relatively low density. However, there are limitations concerning the possible achievement of volume fraction of reinforcement in the matrix. Moreover, particles or fibers comprise a non-continuous phase and the full interpenetration between components does not occur. Consequently, it can cause decrease of a degree of inter connectivity between the phases. In this context, a new group of materials called Interpenetrating Phase Composites (IPCs) was developed [4, 5]. These materials are called co- continuous or "3-3" composites too. It means that matrix and reinforcement are interconnected in all the three spatial dimensions. Thereby, a material with better mechanical properties can be obtained [6, 7].

With regard to the expensive and complicated manufacturing process of IPCs, new directions in their fabrication are observed. Powder metallurgy and casting methods are typical directions of manufacturing technologies. The pressure infiltration, as a specific modification of casting methods, is more and more used. The porous ceramic, called preform, are infiltrated by liquid materials. A possibility of fabrication the IPCs of precise shape mapping, a high- quality surface and a low- cost production [8–11], is of a particular interest. However, manufacturing of ceramic preform comprised a separate process. Taking into consideration the porous ceramic manufacturing, there are few methods: polymeric sponge method, gel casting of ceramic foams or sintering of mixture of ceramic material and canals structure forming agent. The ceramic preform, as a framework determines the properties and microstructure of the composites. Hence, fabrication of a preforms structure of open pores and joined canals is required. This structure allows to easy flow of the liquid material [12, 13].

Many reviews deal with characterization of ceramic- metal IPCs [14–18]. In many works, mechanical properties of lightweight metal alloys/ alumina composites are examined. The finite element modeling is used to study a microstructure, mechanical and thermal behavior of IPCs. However, the ceramic- polymer "3-3" composites are still insufficiently reported. Because of combination of the ceramic hardness and stiffness with the rubbery entropy–elasticity of the elastomer, novel composites with better characteristic can be obtained [19–21] Due to their advantages such materials can be used not only in transport, aerospace and building applications, but also for lightweight armour in defence systems against ballistic threats. Military industry is one of the most developing branch in the world. Security and military forces have been forced to adapt to the new international terror threats. The application of lightweight and resistant protective systems is required as a military's equipments. Ballistic panels must protect against a range of threats such as direct gun

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fire, shaped charges, fragmentation and land mines [22–24]. Generally, two requirements for materials applied as protective covers are determined. They should exhibit relatively low density and effectively dissipate the energy, which allows to reduce the mass and thickness of the armoured covers. In order to perform ballistic panels, which play protective role and possess energy- consuming properties, materials should be selected appropriately. Vehicles designed in traditional way are manufactured using high strength armour steel. New ideas establish replacement of steel by ceramic or protective sandwich system consisted of two or more layers. The most important ceramic materials for ballistic protection are alumina, silicon carbide, and boron carbide. The composite armour system is usually made of ceramic placed on the strike face and composite backing [22–24].

Alumina or aluminium oxide, Al_2O_3 is a major engineering material. It offers a combination of high strength, hardness, stiffness and good corrosion and thermal stability. Al_2O_3, is the most commonly used ceramic because of acceptable costs compared to other ceramics [25].

The paper concerns the composites fabricated via infiltration of porous alumina ceramics by urea-urethane elastomer. As a result, composites of two interpenetrating phases with three-dimensional connectivity were obtained. Properties of the composites were investigated. The IPCs are distinguished by high compressive strength and capability to dissipate the impact energy. The effect of microstructure and stress- strain characteristic of composites on the properties required for protective panels application was also evaluated experimentally.

2. Materials and methods

Porous ceramics were obtained by sintering of Al_2O_3 powder and Al_2O_3 granulate product mixed with the addition of a pores structure forming an agent. The difference between chemical composition and morphology of the powder and granulate products was found. The components differ in shape and quantity.

The ceramic preforms were fabricated by the three- steps method. Firstly, the mixture of a powder, the granulated product and a pores structure forming an agent were uniaxialy consolidated under the pressure of 10–60 MPa. Three tapes with different porosity from 20 to 40 vol.% were pressed separately. In order to obtain a porosity gradient, lamination of three tapes was carried out respectively 20, 30 and 40 vol.%. In the next step pre-sintering of semi- finished preforms, comprised of the three Ek20, Ek30 and Ek40 tapes, was performed according to parameters presented in Table 1. As a result ceramic preforms with porosity gradient were obtained.

In order to improve fracture toughness and compressive strength of ceramic performs, high isostatic pressure method (HIP) was applied. HIP sintering was performed using 200MPa pressure at 1600°C temperature and argon atmosphere. The process was carried out in one hour.

In the presented studies the urea–urethane elastomer (PU2.5) was synthesized based on three substrates: 4,4'-diphenylmethane diisocyanate (MDI), poly (ethylene adipate) (PEA), with an average molecular weight of 1908 a.u. and dicyandiamide (DCDA), as a chain extender. The urea–urethane elastomers with molar ratio of MDI/(PEA + DCDA) equal to 2.5, which corresponds of hard to soft segments ratio H/S = 1.50, were synthesized by one-shot method [26]. The elastomer structure and properties strongly depend on the hard and soft segments (H/S) molar ratio. The harder segments the better adhesion and compressive strength of the composites [26–28].

Table 1
Parameters of pre- sintering of preforms

T_{start} [°C]*	T_{finish} [°C]**	Time [min]***	Heating velocity [K/h]
20	800	270	453
800	1600	160	573
1600	1600	60	273
1600	20	270	873

* T_{start} [°C] – start time of the process
** T_{finish} [°C] – end time of the process
*** Time [min] – process duration

The Al_2O_3/PU2.5 composites were obtained by the infiltration of ceramic preforms with gradient of porosity with reactive mixture of substrates in the liquid form [29]. The curing process was carried out for 14–16 h at 120°C.

Porosity and absorbability of each Ek20, Ek30 and Ek40 tapes were performed by application of the hydrostatical weight method. The test was carried out according to the Archimedes principle. Microstructure of ceramic performs was also characterized using X-ray tomography type SkyScan 1174. In order to distinctly measure the gradient of porosity, the test was led for every layer of ceramic preform. The application of an image analysis program allowed to investigate the volume fraction of both phases. To determine the geometry of ceramic pores measurements of X-ray tomography were used. Comparison between porosity measured using hydrostatical weight method and X-ray tomography was performed. The microstructure of ceramic-elastomer composites was studied with Scanning Electron Microscopy (SEM) Hitachi TM3000. SEM observations of ceramic-elastomer composites and distribution of Al_2O_3 phase were carried out on samples sections and fracture surfaces. In order to measure a residual porosity of composites X-ray tomography was carried out.

Compression tests of composites, ceramic preforms and elastomers were also performed to make a comparison between mechanical properties of these materials. The energy dissipated by composite during loading was evaluated by integrating the area under the stress–strain curve, namely [5, 30].

$$W = \int_0^{\varepsilon_{\max}} \sigma d\varepsilon. \tag{1}$$

The dynamic behavior of the composite was investigated using the split Hopkinson pressure bar technique in conjunction with high-speed photography. Dynamic compression test was conducted using elastic bars system (modified Hopkinson split pressure bar system). Signals from the bars were amplified using LTT500 Tasler emplifier. The Hopkinson split pressure bar characterized by not only its strain excitation system, but mainly by its measuring system which attempts to eliminate elastic wave interference. Ballistic testing was carried out using armor- piercing shell (AP). It is the straightest variety of the armor-piercing ammunition with steel core of the projectile. AP shell is characterized by 7.62–54R mm caliber B-32 type and 854 m/s velocity. The composite armour systems were made of monolithic Al_2O_3 ceramics placed on the strike face and high strength steel as a backing material. Also an intermediate ceramic- elastomer layer was applied.

3. Results and discussion

The obtained Ek20, Ek30 and Ek40 tapes were characterized in terms of porosity and absorbability. The results are presented in Fig. 1. Results of these investigation confirmed that lamination of these tapes allowed to obtain ceramic preform with gradient of porosity. Porosity of each tape varied from 20 to 40 vol.%. The more volume fraction of pores, the higher absorbability.

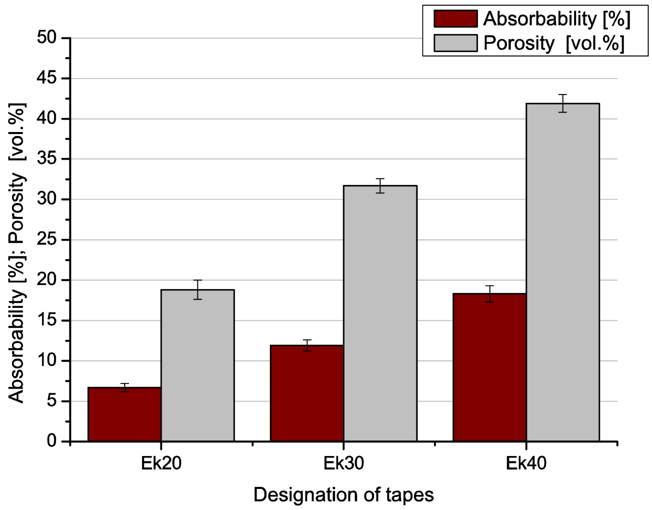

Fig. 1. Porosity and absorbability of Ek20, Ek30 and E40 tapes measured using hydrostatical weight method

The hydrostatical weight method has some limitations in terms of evaluating volume fraction of closed pores. The aim of study was to compare the result of porosity measured by hydrostatical weight method and using X-ray tomography. The difference between the results can be interpreted as a closed porosity. The results of X-ray tomography of Ek20, Ek30 and E40 tapes are presented in Fig. 2.

It is necessary to mention that Ek30 and Ek40 tapes were characterized by open pores. In case of Ek20 tape, a few closed pores can be observed. Using the hot isostatic pressure method for Ek20 tape could cause occlusion of canals between pores. Stereological studies of X-ray tomography images allowed to determined porosity and pore's size of Ek20, Ek30 and Ek40 tapes (Table 2). Difference between the porosity of Ek20, Ek30 and Ek40 tapes measured using hydrostatical weight method and using X-ray tomography were observed, especially for Ek20 tape. On the basis of the X-ray tomography results we were able to total volume fraction of open and closed pores. The pore sizes of the ceramic tapes varied from 20 to 500 μm. Volume fraction of closed pores amounted approximately 5 vol.%.

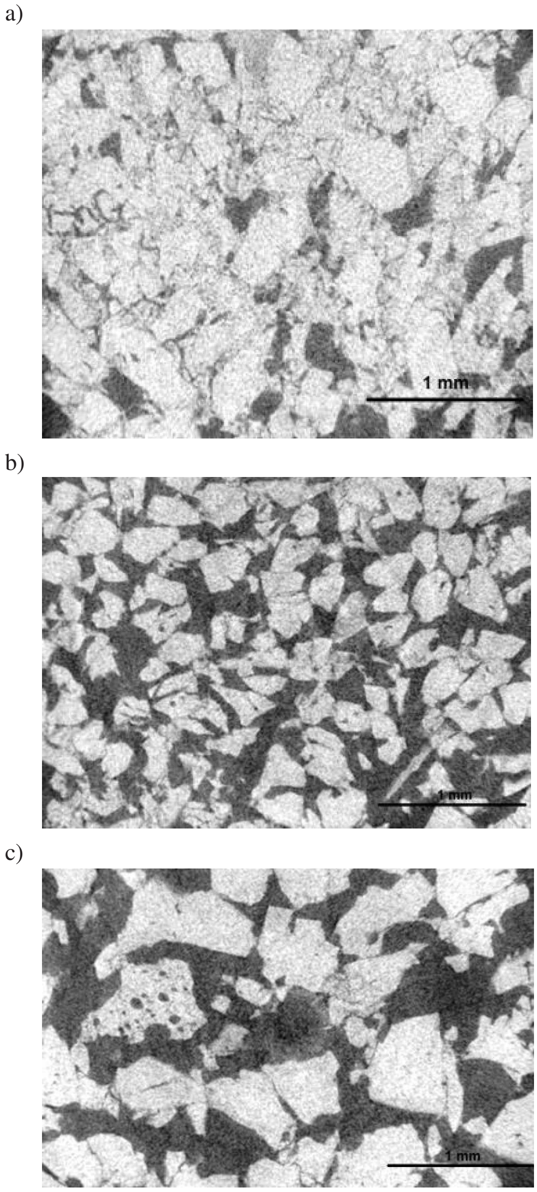

Fig. 2. Images of X-ray tomography of (a) Ek20, (b) Ek30 and (c) Ek40 tapes

Table 2
Porosity and pore's size of Ek20, Ek30 and E40 tapes measured using X-ray tomography

	Ek20	Ek30	Ek40
Porosity [%]	24.2±1.1	32.9±0.7	44±0.9
Pore's size [μm]	20–100	120–250	300–500

In the next step, X-ray tomography of the whole ceramic preform was carried out. Utilization of X-ray tomography images allowed to create a 3D model of ceramic preform as shown in Fig. 3.

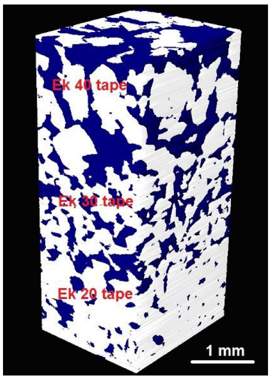

Fig. 3. X-ray tomography model 3D of Al_2O_3 gradient preform

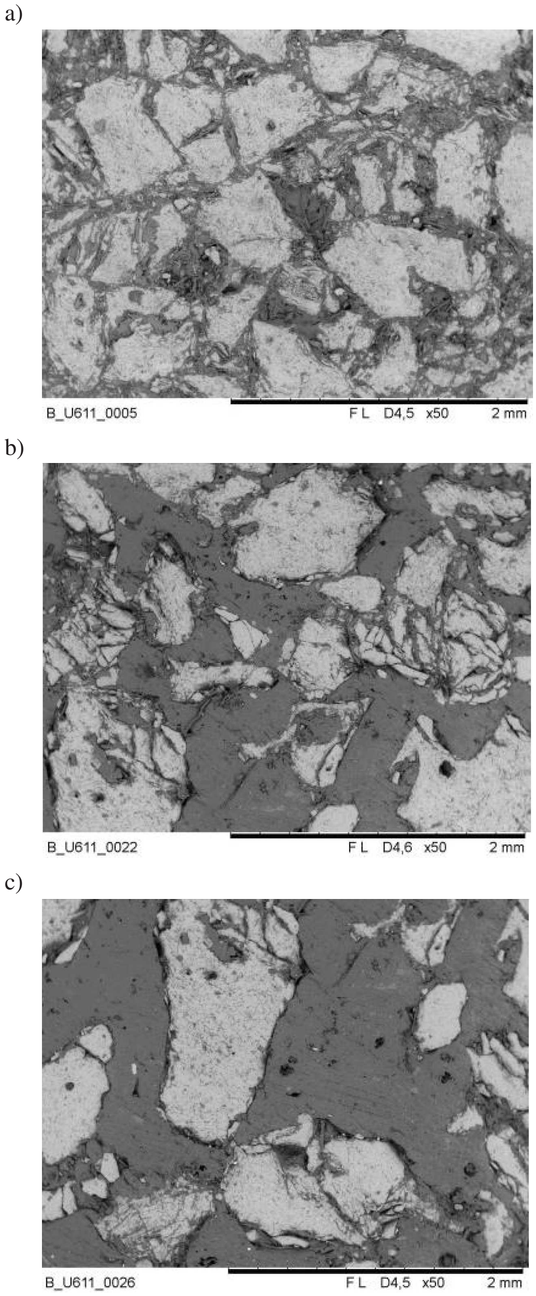

Fig. 4. SEM images of ceramic- elastomer composite, respectively (a) Ek20, (b) Ek30 and (c) Ek40 layers

Microstructures of Al_2O_3/PU2.5 composites are shown in Fig. 4. The observations were performed for each part of composite differing in porosity of ceramic preform used for composite fabrication. The microscopic observations of composites indicated that the pores of matrix are filled by the elastomer, independent of the ceramic preform porosity used for the composites fabrication. Only small voids located at ceramic- elastomer boundaries can be observed. Additionally, SEM investigations revealed that pressure used to infiltration of elastomer does not generate cracks in the perform.

The main problem during particles reinforced composites fabrication that a filler sedimentation leads to non-homogeneous microstructure [1]. In contrast to the traditional composite, IPC consisted of two continuous interpenetrating networks. SEM observations of the ceramic- elastomer composites proved that the phases are interconnected. Isotropic microstructure of Al_2O_3/PU2.5 composites are observed in Fig. 4.

SEM observation of Al_2O_3/PU2.5 composite's microstructure after compression tests was carried out (Fig. 5). It was found that the network of cracks in the ceramic phase was formed. Moreover, the observations indicated that microcrack tips are blocked by the elastomeric phase and a bridging effect is ensured. Also crack branching was deflected. Similar results were attained by Steier [31]. The crack path mainly occurred along the ceramic- elastomer interface. Hence, adhesion between ceramic and elastomer affected on a crack growth. Improvement adhesion between the phases was conducted to decrease anetwork of cracks and increase mechanical strength of a composite. For this purpose using an adhesion promoter was recommended, especially silanes [31].

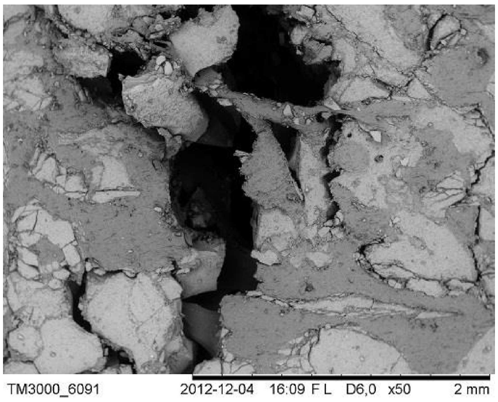

Fig. 5. SEM image of Al_2O_3/PU2.5 composites after compression test

In order to investigate that co- continuous composites are characterized by inter connectivity between the phases, several methods have been used. Microscopic observations of cross-section of material are typical selected. Moreover, a SEM/EDS map and a XRD analysis of obtained composite can be utilized to illustrate its interpenetrating network. In case of metal- ceramic IPC electrical conductivity measurements may be done. Although X-ray tomography is recommended by many authors [32, 33].

X-ray tomography also allowed to determine residual porosity of composites (Fig. 6). Ceramic was visible as a white phase, elastomer as a grey phase and pores as a black one.

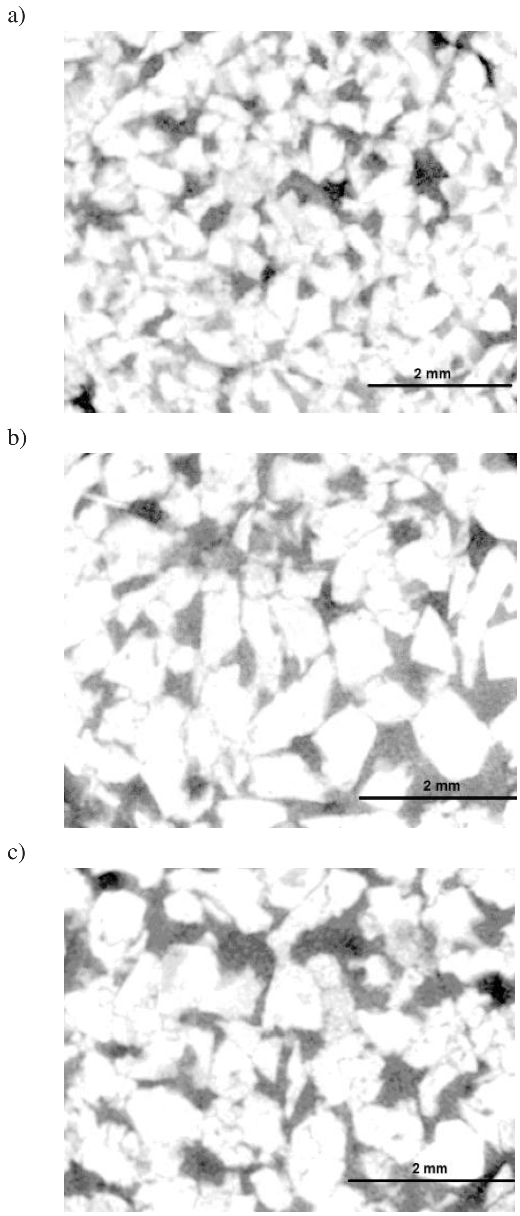

Fig. 6. Images of X-ray tomography of ceramic- elastomer composite, respectively (a) Ek20, (b) Ek30 and (c) Ek40 layers

These studies allowed to quantitatively evaluate both phases and voids. For the porosity ranged from 20 to 40 vol.% the residual porosity averaged between 12 and 5 vol.%, respectively (Fig. 7). Moreover, one can observe decrease of the investigated residual porosity of Al_2O_3/PU2.5 composites with regards to the increasing of preform porosity used for IPCs fabrication. In order to explain the tendency, it is worth to mention that the depth of liquid infiltration can depend on the size of the capillaries, the variation of pressure along the capillary length, the time of preserving the liquid state within the capillary and on the dynamic viscosity [34, 35]. In the case of Ek20 tape, insufficient size of the pores and closed canals can cause increase of infiltration depth.

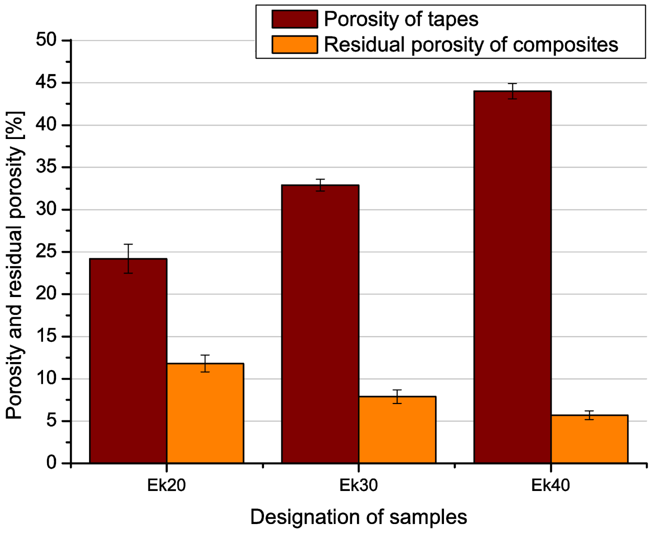

Fig. 7. Comparison of tapes porosity used for composites fabrication and residual porosity of IPC, respectively Ek20, Ek30 and Ek40 layers

The compressive strength and dissipated energy of the elastomer, porous ceramic and ceramic- elastomer composites are shown in Fig. 8. The strain's velocity of the static compression test was 0.001 s^{-1}. The composites exhibit significantly higher compressive strength and dissipated energy in comparison to ceramic preform. The increase of mechanical properties of composites was caused by combination of ceramic stiffness and high elasticity of elastomer. While under compression tests the porous ceramics were totally destroyed, the composite's failure was not observed due to the presence of elastomer in the ceramic pores. Similar results were obtained for IPC fabricated using infiltration aluminum preform by epoxy- based syntactic foam [4, 5].

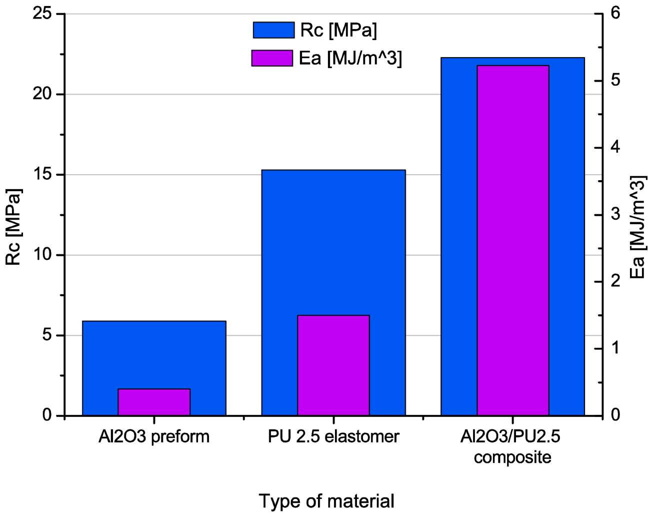

Fig. 8. Compressive strength and energy dissipated by PU2.5 elastomer, Al_2O_3 ceramic preform and Al_2O_3/PU2.5 composites

In comparison static and dynamic compression tests were utilized (Fig. 9). Strain's velocity of static test were 0.001 $s^{-1}$and 0.1 s^{-1}, while for dynamic test 500 s^{-1}. Dynamic tests were carried out via generation of two-dimensional waves in uniaxial strain state. Results of both tests proved that increase of strain velocity caused improvement of compressive strength. Increase of strain's velocity ensured higher compressive strength and capacity of energy dissipation by the composites (Fig. 9).

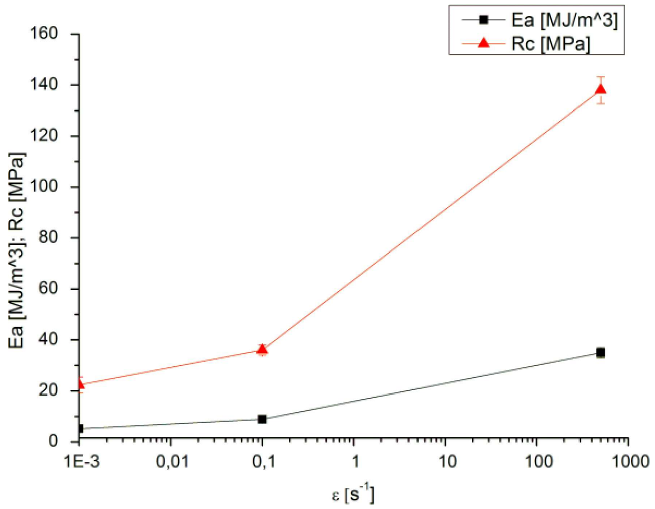

Fig. 9. Effect strain's velocity on compressive strength and dissipated energy of Al_2O_3/PU2.5 composites

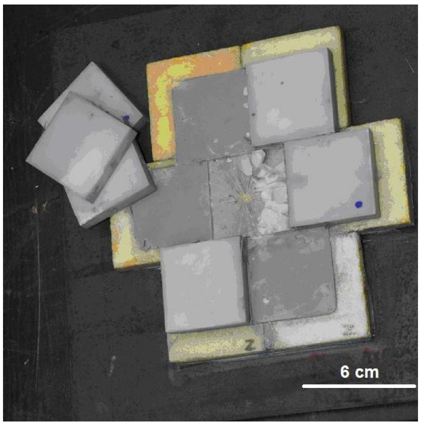

Fig. 10. The front view of the panel with ceramic and composite layers after ballistic test

A typical armour system consists of ceramic and steel layers. The ceramic is normally placed on the strike face [36, 37]. In these paper ballistic panels contained ceramic and intermediate composite layers was investigated. The armour configuration included also an Armox 500T high strength steel. The armour systems were impacted by an armour piercing (AP) projectile 7.62 mm calibre AP 7.62x54R B32 type with steel core. In Fig. 10, the results of ballistic tests are shown. When the projectile impacted the surface of the ceramic, its kinetic energy was greatly reduced due to penetration of ceramics. Moreover, the shattered fragments of the projectile completely penetrate the ceramic. As a result of breakup of ceramic body, kinetic energy of the AP projectile was dissipated. However, composite's layer has not been destroyed (Fig. 10). The residual energy was absorbed via elastic strain of composite plates. It can suggested that steel layer may be replaced by IPC layer.

4. Summary

Fabrication, microstructures and mechanical properties of ceramic- elastomer composites is demonstrated. The obtained results of SEM observations and mechanical tests showed that the infiltration method is useful for fabrication of composites with percolation of the phases, which exhibit higher compressive strength and sustain large deformation. SEM observations of the microstructure of the composites indicated that the porous ceramic preform was successfully infiltrated by elastomer. It was concluded that infiltration with elastomer significantly increases compressive strength of porous ceramics.

The static and dynamic compressive stress-strain curves of composites have been studied. Compressive strength and dissipated energy were investigated. It is possible to fabricate composites with the ability to absorb the impact energy by combining the porous ceramics stiffness and entropy-elasticity of elastomer.

In this paper, the ballistic behavior of the alumina ceramic and Al_2O_3/PU2,5 composites panel was investigated. The application of ceramic-elastomer composite as an interlayer leads to the more effective dissipation of the impact energy. Elastomers are commonly known as materials that can dissipate the energy due to large deformation (based on their entropy changes). The combination of entropy-elasticity of elastomer with ceramic stiffness in the material with 3D connectivity of phases leads to much higher values of energy absorbed during impact in comparison to other commonly known materials. It is worst to mention that if material absorb the energy it can defeat projectile and ensure optimal degree of safety. Composite material located on the back of the ceramic can be used as the replacement of steel. According to the experimental results, utilization ceramic and composite layers ensured fully ballistic protection.

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