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Experimental research on influence of gas impact on thermal and mechanical properties of auxetic material covered with titanium silicide coating

D. MIEDZIŃSKA[1], R. GIELETA[1], E. MAŁEK[1]*, D. ZASADA[2], M. STANKIEWICZ[1], and K. MARSZAŁEK[3, 4]

[1]Military University of Technology, Faculty of Mechanical Engineering, 2 Urbanowicza St., 00-908, Warsaw, Poland
[2]Military University of Technology, Faculty of Advanced Technologies and Chemistry, 2 Urbanowicza St., 00-908, Warsaw, Poland
[3]AGH University of Science and Technology, Faculty of Computer Science, Electronics and Telecommunications, 30 Mickiewicza St., 30-059 Cracow, Poland
[4]DMA Ltd., 7 Łukowa St., 43-300 Bielsko-Biała, Poland

Abstract. The aim of the presented work was to study the auxetic textiles covered with titanium silicide coating. The research was carried out to develop the material structure, which will be used for protective clothing, e.g. for firemen. The new material should be characterized by increased heat resistance coupled with protection against gas pressure impact caused e.g. by gas installation damage. In the paper, an assessment of the change in heat resistance properties of a Ti-Si coated auxetic textile loaded with gas pressure impulse was carried out.

Key words: Ti-Si layers, damage, impact, thermal resistance, SEM, auxetic materials.

1. Introduction

The initial study on applying auxetic materials for protection against detonation was carried out in 2006–2007 and is presented in [1]. The basic idea of an auxetic textile is presented in Fig. 1.

Auxetic yarn is winded as a double spiral built of thin high strength yarn and thick elastomeric core. Under the tensile in auxetic yarn parallel direction the thin yarn tends to straighten, causing bending of the core, which results in negative Poisson's ratio in the direction perpendicular to auxetic yarn. The energy is absorbed by the elastomeric core and increased by its bending. The difference between mechanical properties of auxetic yarn components is very important.

On the basis of this phenomenon, it was decided to use the auxetic textile for antidetonation protection. The protected surface, e.g. window, is equipped with a textile screen fixed onto its edges The blast wave stretches the auxetic yarns, causing elastomeric cores bending and energy absorption. Results of tensile tests of single auxetic yarn were also presented in [1]. There the strong dependence between yarn strength and strain was proved.

A different researched method, whose aim was to study ballistic and explosion debris protection of auxetic textiles, was shown in [2]. Various types of textiles were tested. The tests were carried out with the use of the referenced textile to eliminate mistakes connected with blast wave intensity. Microscopic observation of yarns showed the damage of braid yarns, which were made of contex. The damage was caused by strong bending and debris impact.

The auxetic material behavior was described more comprehensively in [3]. Observations were made with the use of fast cameras, which allowed to assess the stages of deformations when interacting with detonation products.

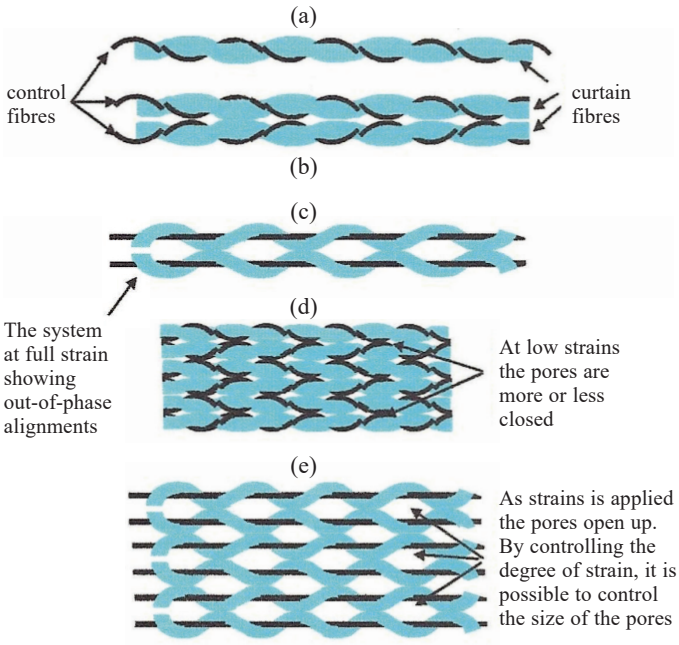

Fig. 1. Basic auxetic textile concept: a) base textile unit – thin yarn winded around thick core, b) two base units aligned opposite each other, c) fully tensiled pair of unit yarns, d) partially tensiled textile, e) fully tensiled textile [1]

*e-mail: ewelina.malek@wat.edu.pl

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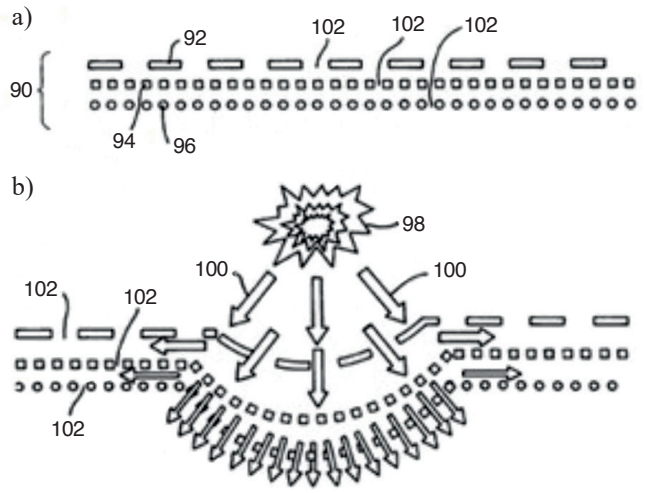

Fig. 2. Blast wave dissipation by auxetic textile a) before detonation, b) during detonation [4]

Also the direction of pressure wave energy absorption by auxetic textile was shown in [4]. According to Fig. 2, initially the flat wave goes through the material pores, changing its shape. Thanks to this phenomenon, less energy impacts the target, which can be deemed energy dissipation.

As it was already mentioned above, the auxetic textile would be covered with Ti-Si coating to improve its heat resistance properties. Such TBC – thermal barrier coatings – are used where heat flow limitation is important, e.g. in aerospace (turbines, engines), the automotive industry (valves) and chemistry. Heat transport can be achieved by means of conduction, repossessing and radiation.

Heat conduction is caused by temperature differences and is a transfer of kinetic energy of microscopic motion of neighboring particles.

A variety of research methods of TBS can be found. In [5] the method of differential measurement using thermocouples was shown. In [6] the TBC thermal shock protection study was presented, when in [7] the effectiveness of coatings for different fuels combustion processes was checked.

An interesting description of thermal coating used in turbine properties was presented in [8], while a comparison of different methods using various heat sources, such as ovens or flames, was shown in [9].

2. Research methodology and samples preparation

In the presented work, the auxetic textiles covered with titanium silicide coating will be analyzed. The aim of the study is the development of the material structure which will be used for protective clothing, e.g. for firemen. The new material should be characterized by increased heat resistance coupled with protection against gas pressure impact caused e.g. by gas installation damage. In the paper an assessment of the change in heat resistance properties and structure of Ti-Si coated auxetic textile loaded with gas pressure impulse will be carried out.

A research plan scheme is presented in Fig. 3. As it was mentioned, the auxetic material thus developed will be used for gas pressure impact protection.

Looking at the literature shown below, no such comprehensive and coupled research was carried out to date, so the method of testing proposed in the paper is innovative.

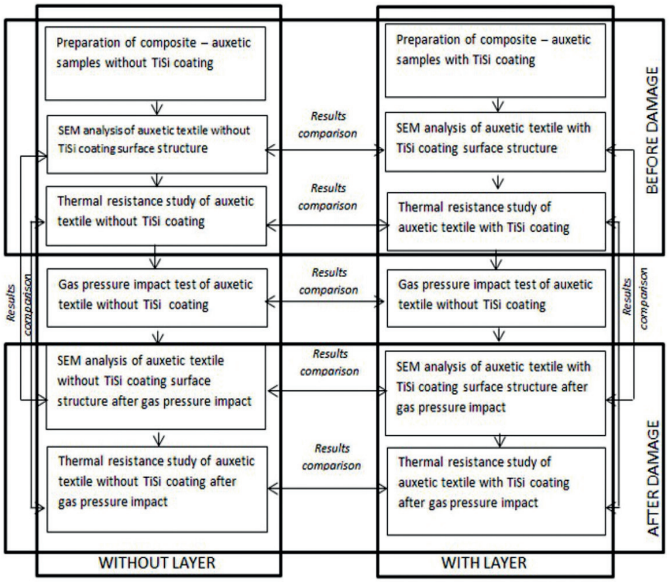

Fig. 3. Research plan scheme

Special samples were prepared for the tests. They were built of auxetic material fixed with the use of ET5428 epoxy glue between two rigid rings made of epoxy-glass composite (Fig. 4a) to achieve the boundary condition for textile auxetic behavior. Sample dimensions are presented in Fig. 4b).

Two series of samples were prepared: without and with Ti-Si coating with special protection against coating pollution during preparation to achieve best SEM analysis results.

Thin Ti10Si coatings were developed with the use of the dc magnetron sputtering method. Deposition was carried out from hot pressed Ti 90% at. – Si 10% at target. A magnetron gun 90Ti10Si hot pressed target was used in both. Two mag-

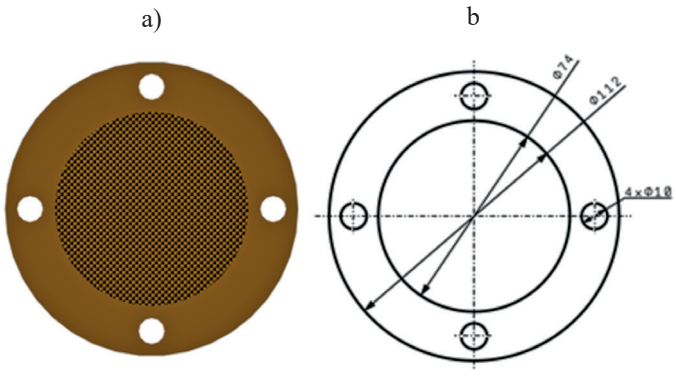

Fig. 4. a) Scheme of tested sample; b) tested sample dimensions

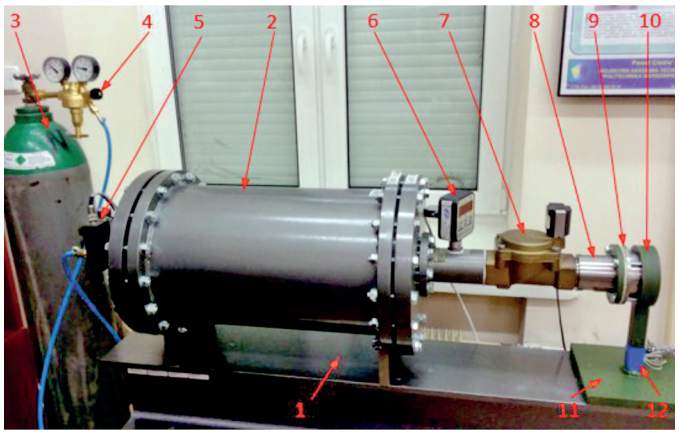

Fig. 5. Testing stand for gas impact influence on auxetic textile study: 1 – basis, 2 – pressure tank, 3 – bottle filled with argon, 4 – pressure reducer, 5 – proportional filling up valve, 6 – control manometer, 7 – trigger valve, 8 – nozzle, 9 – tested sample, 10 – sample holder, 11 – holder basis, 12 – strain measurement system

netron gun systems were designed due to planned long textile deposition. With two guns textile transport velocity could be higher.

The coating development process was preceded by glow discharge for the purpose of textile degasification. Discharge parameters were as follows: pressure of $4 \cdot 10^{-3}$ mbar, electric current under 1 A, effective power of 0.8 kW. The coating development process was carried out in the atmosphere of argon of $2 \cdot 10^{-3}$ mbar from two launchers of discharge current 2.5 A and effective power over 1.5 kW. Temperature of the basis was under 70°C. The developed coating thickness was 180 nm.

3. Gas impact tests

Research on gas impact influence on auxetic textile was carried out on a specially designed testing stand, as shown in Fig. 5.

Pressurized gas was applied for the purposes of pressure impact execution. Pressure tank 2 was filled with argon from bottle 3 by means of pressure reducer 4 and proportional filling up valve 5. Control manometer was used for checking pressure

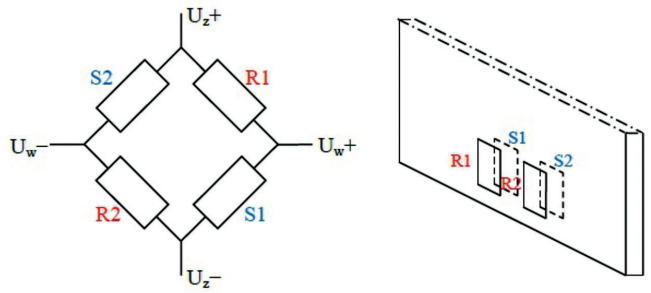

Fig. 6. Strain measurement system scheme

value in the tank. After sample 9 fixing in holder 10 and fixing holder basis 11 to stage basis 1, the test was carried out with the trigger valve 7 opening. The pressurized gas impacted the researched sample by means of nozzle 8. Loading system was controlled with the use of a special computer code. The initial pressure value was 8 bar, while time of gas impact was 200 ms. The strain measurement system allowed to carry out quantitative and qualitative analysis of gas pressure impact on tested materials. Strains were registered and archived with the use of the special computer code. The electric scheme of the strain measurement system is presented in Fig. 6. A full strain gauge bridge sensitive to bending was applied.

Output signal of strain gauge bridge was described as:

$$U_w = U_Z \cdot \frac{1}{4} \cdot N \cdot k \cdot \varepsilon \tag{1}$$

where:

U_w – output voltage [V],
U_z – supply voltage [V] (in tests $U_z = 5$ V),
N – configuration constant of a bridge, N = 4 for full bridge,
k – strain gauge constant (k = 2,09 for the strain gauges used),
ε – measured strain [m/m].

Strain measurement system scaling was effected by bypassing one of the bridge branches with resistance of 100 kΩ. The averaged results are presented in Fig. 7. and Fig. 8. The maximum values of forces and strain are shown in Table 1.

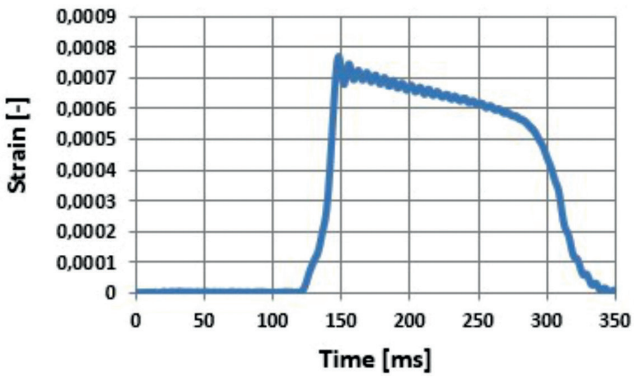

Fig. 7. Gas pressure impact test results for sample without Ti-Si coating

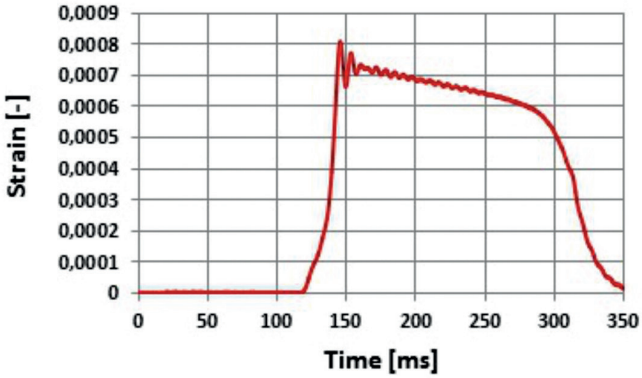

Fig. 8. Gas pressure impact test results for sample with Ti-Si coating

Table 1
Comparison of gas pressure impact test results

	Sample without Ti-Si coating	Sample with Ti-Si coating
Maximum force [N]	2809.4	2821.6
Maximum strain [–]	0.000772	0.000775

Strain vs. time values registered on the testing stand are almost the same for textiles without and with Ti-Si coating. The Ti-Si coated auxetic material absorbs energy of gas from pressure tank in the same way as the textile without coating. On this basis it can be concluded that application of such coating is negligible for gas impact energy absorption of auxetic material.

4. SEM investigation of Ti-Si coating

The investigation of textile surface without and with Ti-Si coating before and after the gas pressure impact was carried out with the use of the Quanta 3D FEG Dual Beam scanning microscope using different values of magnification. Acceleration voltage of 20 kV was used. The samples were observed in secondary electrons contrast SE(L) and SE(M). It must be mentioned that the samples were observed in the same regions marked on their surfaces before and after the pressure test, as shown in Fig. 9.

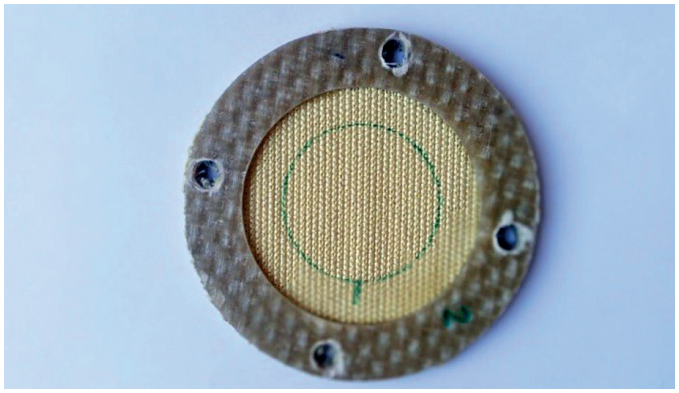

Fig. 9. SEM tested area

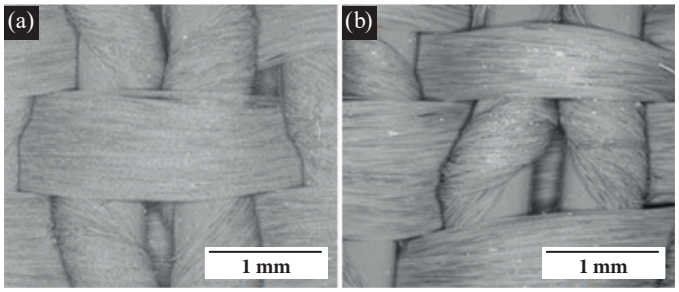

Fig. 10. SEM images of selected area of auxetic sample at magnification 50× a) before gas pressure test, b) after gas pressure test

The results are presented in Fig. 10–12 as surface visualizations for the sample without Ti-Si coating.

The results are presented in Fig. 13–15 as surface visualization for the sample with Ti-Si coating.

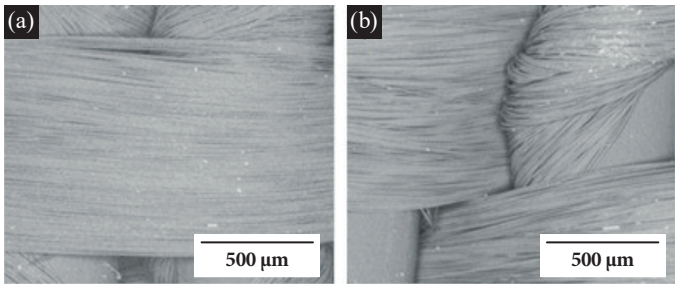

Fig. 11. SEM images of selected area of auxetic sample at magnification 100× a) before gas pressure test, b) after gas pressure test

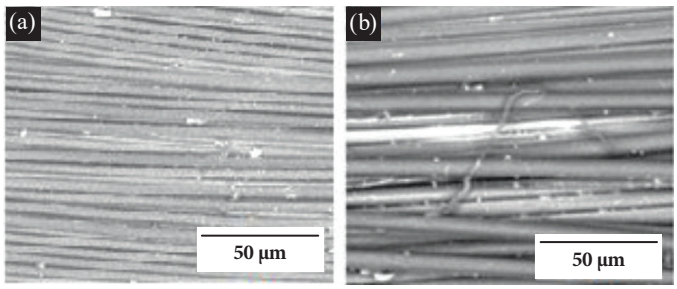

Fig. 12. SEM images of selected area of auxetic sample at magnification 500× a) before gas pressure test, b) after gas pressure test

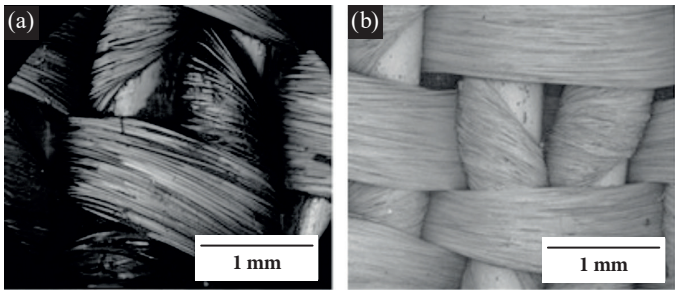

Fig. 13. SEM images of selected area of auxetic sample at magnification 50× a) before gas pressure test, b) after gas pressure test

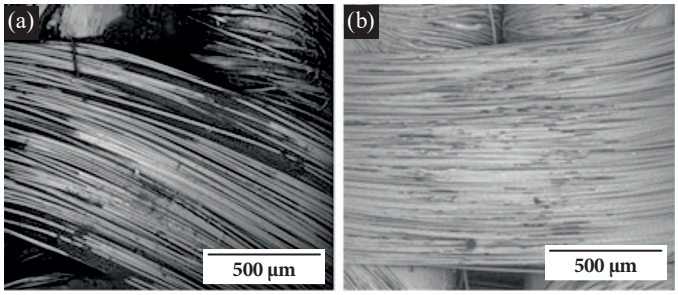

Fig. 14. SEM images of selected area of auxetic sample at magnification 100× a) before gas pressure test, b) after gas pressure test

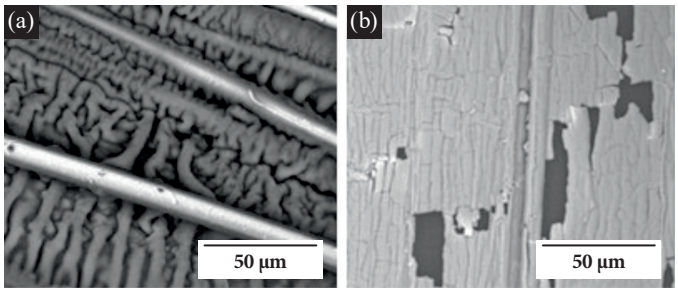

Fig. 15. SEM images of selected area of auxetic sample at magnification 1 000× a) before gas pressure test, b) after gas pressure test

On the basis of the results achieved, the main differences in analyzed material structures were observed. Prior to applying pressure, the auxetic textile without Ti-Si covering can be characterized by light uniform color and proper yarn distribution without any discontinuity or damage. In the case of the same textile but with Ti-Si coating the material is metallic grey, the yarns are damaged and their cohesion is disturbed. Also Ti-Si coating is not homogenous – lighter and darker regions are visible, which can serve as an indicator of deepness of embedded coating. Also it can be seen that some elastomeric yarns are melted, which can be a result of not sufficiently diligent selection of the covering process parameters.

After the pressure test the distribution of yarns in auxetic textile changed. They lost their cohesion and were distributed loosely. In the case of the Ti-Si coated structure, the damage of yarns and covering was observed. After the gas impulse the coating actually cracked. However it still protected the textile against yarns incoherence.

5. Thermal resistance study

The aim of the research was to study the influence of Ti-Si coating on the infrared radiation (thermal) resistance generated in a specially designed testing stand. Radiation intensity was registered with the use of a thermo-vision camera. Samples the same as for previous tests were used. The test was carried out before and after the gas pressure impact.

The researched material is characterized by slack weave, which is presented in Fig. 16, where the samples were illuminated with white light.

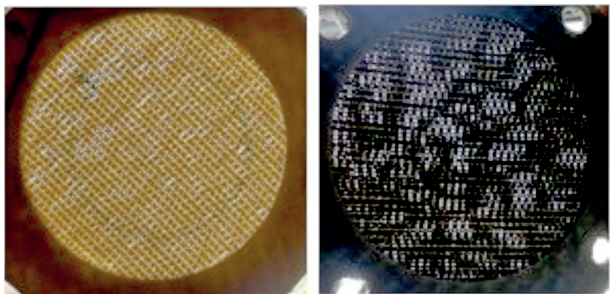

Fig. 16. Samples illuminated with white light: without coating (left) and with Ti-Si coating (right)

The thermal resistance testing stand was set in a thermo-isolated box (Thermobox) made of polypropylene (EPP) for the temperature scope of –40°C to +120°C – Fig. 17. The testing stand was designed and built specially for the purpose of the experiment. Its detailed construction is shown in Fig. 17–19.

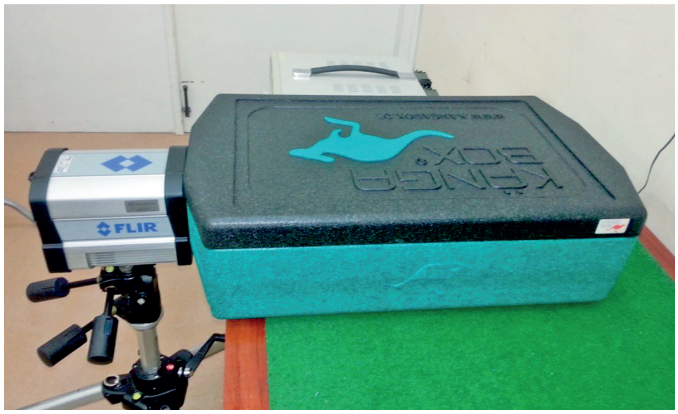

Fig. 17. External view of thermal resistance testing stand

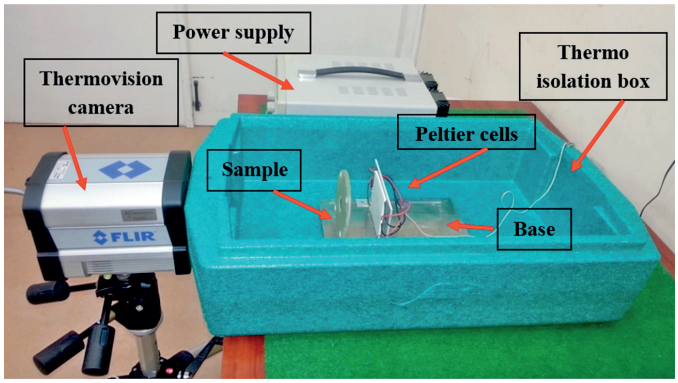

Fig. 18. Internal construction of thermal resistance testing stand

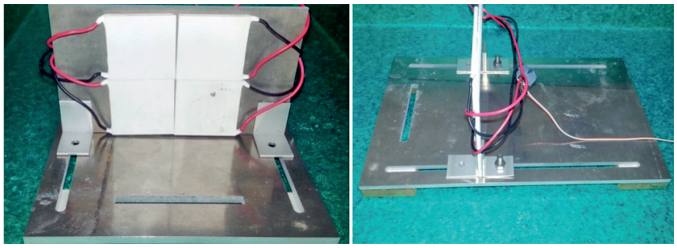

Fig. 19. Peltier cells fixed onto movable base

The material heating process was carried out with the use of so-called “black body” built of four Peltier cells – Fig. 19, fixed onto the movable base, which allowed to change the distance between the heat source and sample within the range of 0–150 mm. Cells control was achieved by the power supply unit (change of the unit voltage resulted in cells temperature change). Temperature level measurement was carried out with

a thermo-vision camera, whose lens was situated inside the thermo-isolation box.

The tests conditions were as follows:

– Cells temperature:
  - Procedure 1: 60°C,
  - Procedure 2: 80°C,
  - Procedure 3: 100°C;
– Distance between textile and Peltier cells was 5 mm for each scenario (Fig. 20a);
– Time of sample heating process – 5 minutes;
– Measurement area on the sample was shown in Fig. 20b. The average temperature was taken from this area.

The test steps were as follows:

– heating the cell to a predetermined temperature,
– stabilization of temperature on the Peltier cells,
– placing the sample in measuring position and measurement.

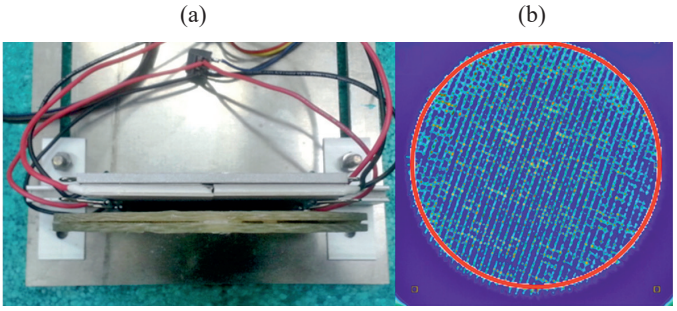

Fig. 20. a) Top view of sample on testing stand, b) sample measurement area

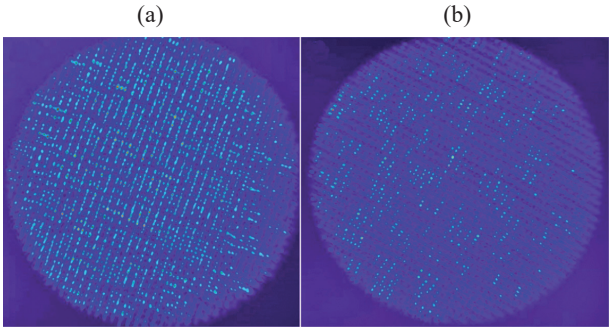

Fig. 21. Visualization of heat radiation passing through sample a) without coating, b) with Ti-Si coating

After the test, it was observed that the amount of heat radiation passing through the sample with Ti-Si coating was smaller than for the sample without coating. An example of that phenomenon is shown in Fig. 21, where the test for Peltier cells temperature of 100°C and time of heating of 1 s is visualized.

The test results for samples before gas pressure impact for Procedure 1 as a temperature vs. time chart is shown in Fig. 22, for Procedure 2 – in Fig. 23 and for Procedure 3 – in Fig. 24.

After the analysis, it was concluded that the differences in temperatures achieved for textile without and with coating at the beginning and at the end of the test were as follows:

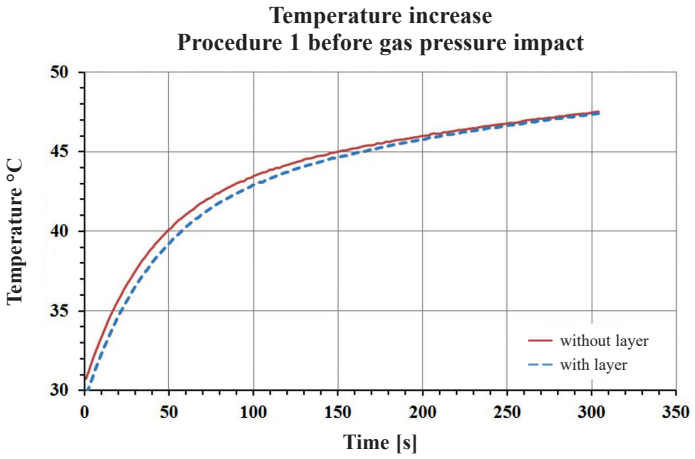

Fig. 22. Test results for Procedure 1 before gas pressure impact as temperature vs. time

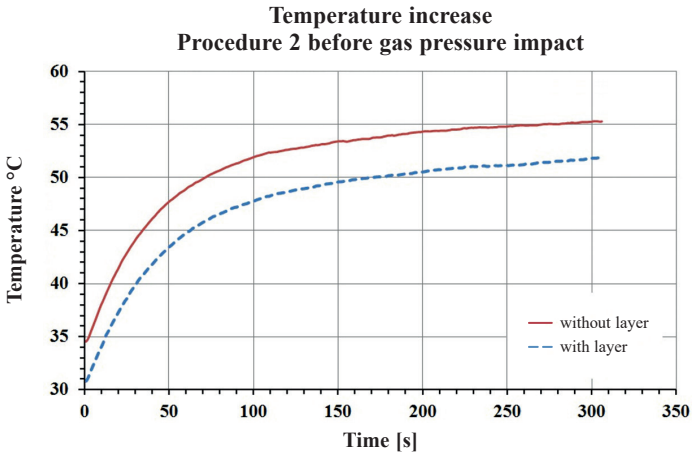

Fig. 23. Test results for Procedure 2 before gas pressure impact as temperature vs. time chart

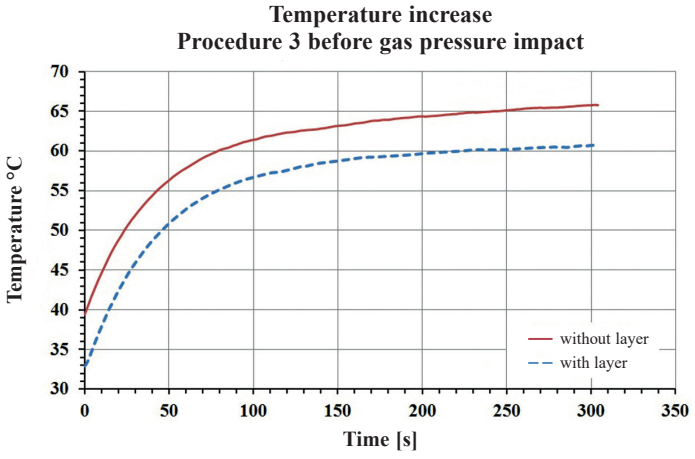

Fig. 24. Test results for Procedure 3 before gas pressure impact as temperature vs. time chart

– Procedure 1 before gas pressure impact: beginning of the test – 0.88°C; end of the test – 0.12°C,
– Procedure 2 before gas pressure impact: beginning of the test – 3.7°C; end of the test – 3.36°C,

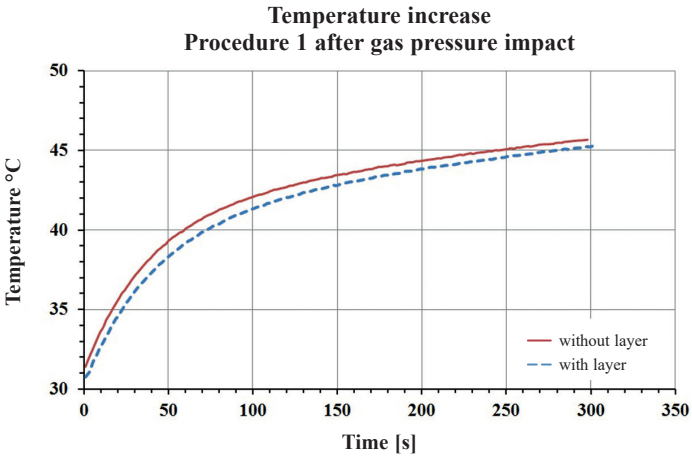

Fig. 25. Test results for Procedure 1 after gas pressure impact as temperature vs. time chart

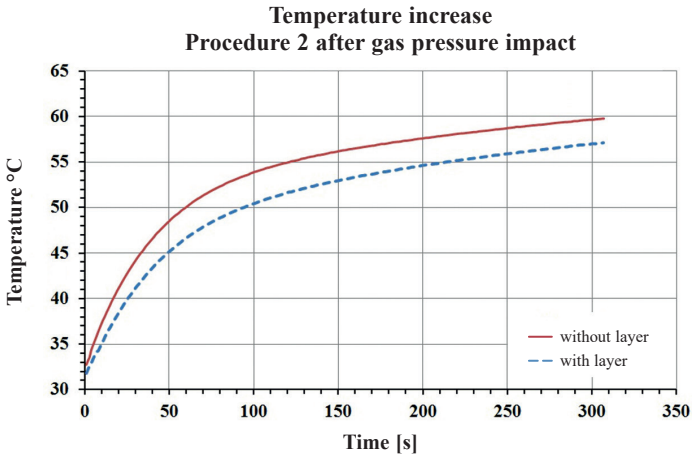

Fig. 26. Test results for Procedure 2 after gas pressure impact as temperature vs. time chart

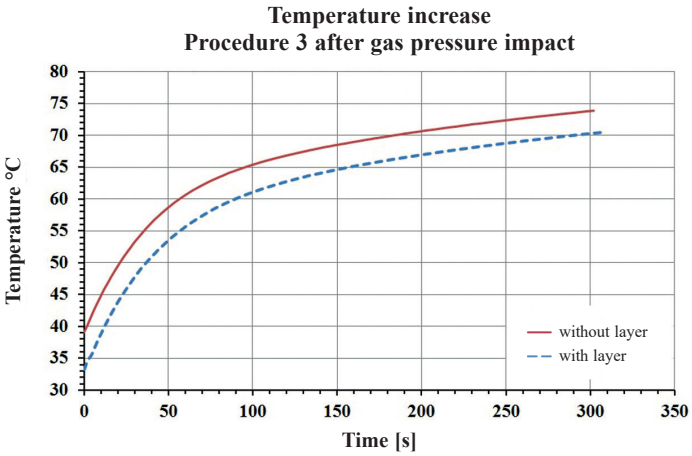

Fig. 27. Test results for Procedure 3 after gas pressure impact as temperature vs. time chart

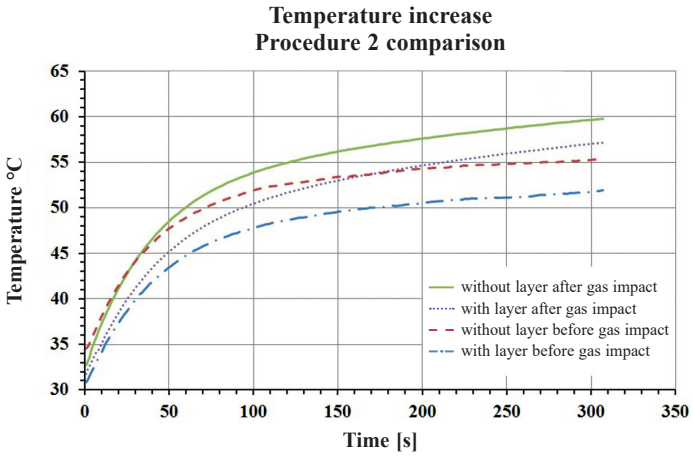

Fig. 28. Comparison of temperature increase vs. time charts for tests before and after gas pressure impact for Procedure 2

– Procedure 3 before gas pressure impact: beginning of the test – 5.21°C; end of the test – 4.98°C.

The next step of the thermal resistance study was carrying out the test for the samples after gas pressure impact.

The test results for samples after gas pressure impact for Procedure 1 as temperature vs. time chart are shown in Fig. 25, for Procedure 2 – in Fig. 26 and for Procedure 3 – in Fig. 27.

The comparison of temperature increase vs. time charts for tests before and after gas pressure impact is presented in Fig. 28 (Procedure 2) and Fig. 29 (Procedure 3). The differences in temperature values are strongly visible. For Procedure 2, the difference was about 5°C, while for Procedure 3 it was even 10°C for samples tested before gas pressure impact.

In Procedure 1 the difference in temperature achieved after 5 minutes of heating was almost negligible, however at the beginning of the test the registered temperature value for the sample with coating was lower than in the case of the sample without coating. It was also noticed that the process of heating was slower for the sample with Ti-Si coating and it achieved the same temperature as the material without coating at the end of the test.

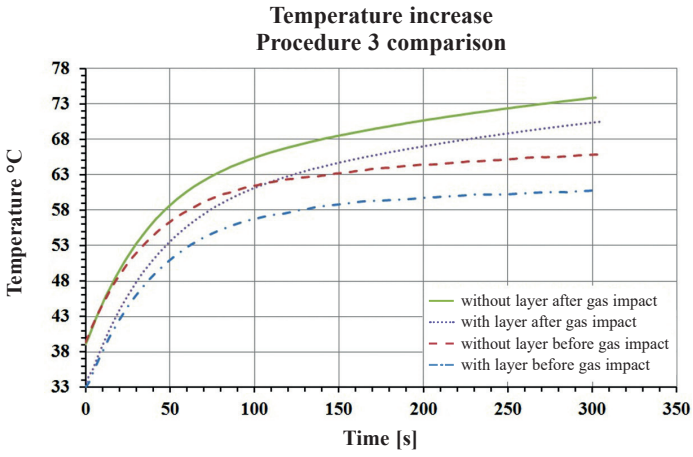

Fig. 29. Comparison of temperature increase vs. time charts for tests before and after gas pressure impact for Procedure 3

In Procedure 2 and 3 considerable difference in the results achieved was noticed. The change in temperature value in the initial phase was 3.7°C (Procedure 2) and 5.21°C (Procedure 3),

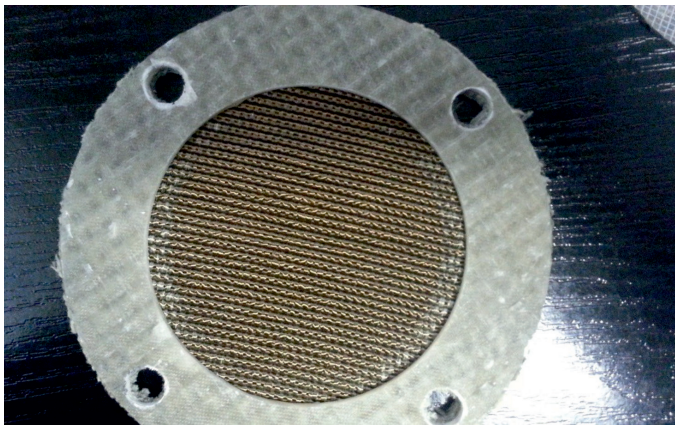

Fig. 30. Sample with visible coating destruction caused by gas pressure impact

Table 2
Comparison of results of differences in temperatures achieved for textile without and with coating at the beginning and at the end of the test before and after gas pressure impact

	Before gas pressure impact		After gas pressure impact	
	Temp. at the beginning [°C]	Temp. at the end [°C]	Temp. at the beginning [°C]	Temp. at the end [°C]
Procedure 1	0.88	0.12	0.58	0.40
Procedure 2	3.70	3.36	0.94	2.64
Procedure 3	5.21	4.98	4.78	3.38

respectively, and at the end of the test it stood at 3.36°C (Procedure 2) and 54.98°C (Procedure 3). The difference at the beginning of the test decreased minimally as compared with the end of the test. In each scenario influence of the Ti-Si coating is visible, especially at the beginning of the test, when partial reflection of heat radiation can also be noticed. The higher the temperature of Peltier cells was, the more the above-described effect became visible.

The samples subjected to gas pressure impact were characterized by lower heat radiation resistance. This was caused by the fact that the gas wave going through the textile destroyed the tight arrangement of fibers, increasing the distance between them, which allowed the heat transfer through it. However in this case the Ti-Si coating also influenced textile heat resistance.

It was also marked that the Ti-Si coating was very fragile at it was easy to remove. The sample with visible coating destruction caused by gas pressure impact is presented in Fig. 30.

After the analysis, it was concluded that the differences in temperatures achieved for textile without and with coating at the beginning and at the end of the test were as follows:

– Procedure 1 after gas pressure impact: beginning of the test –0.58°C; end of the test –0.4°C,
– Procedure 2 after gas pressure impact: beginning of the test –0.94°C; end of the test –2.64°C,
– Procedure 3 after gas pressure impact: beginning of the test –4.78°C; end of the test –3.38°C.

A comparison of the results achieved is presented in Table 2. It can be concluded that the differences in temperature values are usually lower for textiles which were subjected to gas pressure impact.

6. Summary

In this paper a comprehensive study of thermal resistance properties of auxetic material covered with Ti-Si coating and subjected to pressure gas impact was carried out. The study showed the differences and changes in textile and coating structure discovered by means of SEM analysis. It was shown that the coating did not change the mechanical properties of the auxetic textile, however the gas impact damaged the Ti-Si structure, decreasing heat resistance properties. It must be concluded that the developed innovative method of experimental testing showed good results and in the future will be extended to include microwave resistance study and then applied for testing special textiles for firemen clothing covered with other types of ceramic coatings.

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