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Research on Influence of TiSi(N) Reflective Coating Thermal Resistance on Energy Absorption of Fireproof Textile Coupled with Auxetic Fabric

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The research presented in the paper deals with the improvement of firemen protective clothing. The proposed modification of the special textile PROTON is based on the application of TiSi(N) nanocomposite reflective layer, which improves the thermal resistance of the coated material. The second improvement deals with the implementation of auxetic textile into the protective clothing structure. Such material is characterized with the very good blast wave resistance. In the paper both phenomena were studied. The results of thermal resistance of coated and not coated PROTON shown that the application of such structure decreased the temperature acting behind the textile and the application of the auxetic textile significantly increased the PROTON resistance to gas impact. Also the advantages of coupling both improvement methods were discussed, because of the negative influence of high temperature on auxetic behaviour.

Key words: thermal resistance; nanocomposite reflective coating; auxetic textile; firemen clothing.

1. Introduction – aim of research

Textile science is a very wide and still developed part of knowledge. In the presented study the special interest is directed to fireproof textiles improvement. Fireproof textiles are a kind of fabrics more resistant to fire or heat than others through chemical treatment or specially manufactured fibres.

Such fibres could be classified into three categories. The first one are inherently heat and flame retardant fibres (e.g. aramid [1], modacrylic [2], poly-

benzimidazole (PBI) [3], Panox (oxidised acrylic) [4] or semicarbon, phenolic, asbestos, ceramic [5]). The second category are chemically modified fibres and fabrics, in which one can find flame retardant cotton, wool and viscose [6] and synthetic fibres [7], produced by incorporating special additives in the spinning dope before extrusion. The last category of a fireproof textiles are currently developed [8, 9] fibres coated with reflective nanolayer with the use of sputtering method.

In the paper the thermal resistance improvement is based on the TiSi(N) nanocoating application with the use of sputtering technology.

Additionally, it must be noticed that fireproof textiles are not designed to improve the protection against for example gas impact, the possible situation during fire of houses or flats. For this improvement the auxetic textile coupled with fireproof one was proposed.

There are two main disadvantages of auxetics implementation in such constructions as firemen protective clothes. Firstly, they must be stretched during loading to gain the negative Poisson ratio effect. Secondly, the auxetic effect decreases in higher temperatures [10]. Those properties can be a problem when the textile is used for firemen suits. A kind of a protective panel can be the best application.

So the material construction of fireproof textile, additionally coated with reflective nanocoating, and auxetic one can improve both mechanical and thermal protection. The fireproof textile will protect the user against the high temperature and will protect the auxetic textile allowing to use its energy absorption abilities in the most effective way. This phenomenon is studied in the paper.

2. Research background

For most practical applications of plastics, limited flammability is required. The necessity to meet the requirements imposed by the standards defining the resistance of polymeric materials to smoking, forces the addition of anti-pyrenes. Their content in the material may be as high as 50–60% of their weight. Such a high proportion of antipiren, often adversely affecting the physical and processing properties of the material, changes its colour, impairs its transparency, may increase the final price of the product. To limit the flammability of plastics, the most common additives are: aluminum compounds or magnesium hydroxide, bromine, chlorine and phosphorus, antimony and other metals such as tin, zinc, boron, molybdenum oxide [11, 12].

Reduction of the antipiren content and favourable mechanical and processing properties of the polymeric material can be obtained by using the phenomenon of synergism. It consists in enhancing the effect of delaying combustion as a result of the action of a mixture of two or more components and achieving an effect

greater than the sum of individual activities of these components. The addition of graphite in the form of a kind of graphite partitions in the structure (lack of continuity in the structure of the material) prevents the access of flame and oxygen to the entire polymer material. This effect is achieved by using so-called "expandable" graphite, which swells, thus creating a foamed insulating layer. However, by modifying the graphite with melamine salts (cyanurate or phosphate) halogen-free additives are obtained that limit the flammability of polymer materials to a greater extent [13].

Among the large group of polymers, one can distinguish polymers with special properties, which include high temperature polymers [14].

According to the definition under this term heat resistance is understood as the threshold from the maximum temperature at which the given plastic retains its mechanical properties. The measure of the usable properties of a material is its strength, which is why the most often heat resistance is determined on the basis of mechanical properties. This type of test involves the determination of the temperature at which the predetermined deformation occurs under the standard load. For the determination of heat resistance, such methods as: Martensa, Vicata or quite often used in the professional literature temperature of deflection under load (HDT) are used. Heat resistance is achieved by stiffening macromolecule chains, because such polymers are insoluble and do not melt without decomposition of these chains. These materials are made after proper modification (i.e. improving thermal stability by introducing into the macromolecule F, Si, N, B atoms, or e.g. introducing the concentration of aromatic rings) and can be used as a base of fabrics in the production of special clothes for work at high temperatures. Examples of polymers of this group are: polyphenylene ether and poly (p-xylylene) [12, 14, 15].

The measure of flammability or resistance to the material's heat is to test the burned (charred) part of the sample under standard fire or heat conditions. In contrast, the term "thermostability" means the temperature of chemical degradation of the polymer. When classifying polymers in terms of their thermal resistance, various glass transition and melting temperature ranges are used, as well as different time intervals in which the material subjected to a certain temperature should not degrade. According to the Marvel classification, the polymer is considered to be thermostable if it is not degraded during long-term use (up to 25,000 hours) at temperatures up to 300°C and does not change shape or melt during short-term heating (up to 300 hours) at temperatures reaching 500°C. Thermoresistant thermoplastic polymers include those that can be used at very high temperatures (e.g. PBI) as well as examples of materials applicable in a very wide temperature range (e.g. PTFE, for which the operating temperature range is −269°C up to +260°C). To simplify matters, the outer layer is understood as a barrier against heat, water, chemicals, while internal protection is understood

as breathability. During rescue operations firemen are accompanied by stress, rush and increased physical effort. In order not to overheat the body, it produces heat and sweat. An overly tight material layer can cause dangerous effects. One of them are burns [16, 17].

Since a material with all protective properties has not yet been developed, a garment that has a layered structure has been designed. It consists of an outer shell, moisture barrier, thermal barrier and lining, which – depending on the modification – can be a separate layer (four-layer clothing) or be combined with a thermal insulation layer (three-layer clothing) – Fig. 1. Each layer is made of a different material and has characteristic features, which in combination are an effective, innovative solution with a wide range of protection.

Fig. 1. Three-layered arrangement of materials.

The outer shell of special clothing can be made of Nomex or Kermel type aramid fabrics, PBO type polyamide fabrics (these fabrics cannot be colored – appear only in shades of brown), PIB type polyamide fabrics (also they cannot be colored), impregnated cotton fabrics [18].

Aramid and polyamide fabrics under the influence of temperature and flames do not lose their tearing strength, they do not shrink, burn or glow, and do not break, which means that they do not allow the flame to penetrate the inner layers of clothing. Compared to cotton fabrics, they show high resistance to acids and hydroxides. They maintain high mechanical strength and resistance to ignition for a long period of use, regardless of the number of washes and the type of detergent used. Aramid and cotton fabrics appear as a homogeneous structure or reinforced in the form of grids made of thicker fibers – so-called rip stop [18].

The reinforcing material is usually a synthetic rubber Neoprene® produced by the American concern DuPont. It is characterized by high mechanical strength, high resistance to fire, chemical compounds, UV radiation, ozone and weather conditions. Neoprene® is used in industry, including for the production of protective clothing. It is placed on the clothes where the impact on the abrasion is the highest (on the elbows and cuffs of pants and jackets, on the flaps of the pocket, on the collar [18]).

The moisture barrier is the middle layer of special clothing. Its task is to prevent the penetration of liquids and maintain a proper thermal balance. The basic features of the membrane are water tightness – its purpose is to prevent the thermal insulation layer from soaking, permeability to allow the removal of steam generated as a result of sweating of the user. Plastics are used for the production of moisture barriers, e.g. polyurethane, polytetrafluoroethylene (PTFE) polyester [19].

The thermal barrier layer insulates against the external environment and is a barrier against the penetration of thermal radiation into the clothes. This goal is achieved by an appropriate structural solution, based on a model consisting of fibers between which there is air. It is important that the layer is not too thick – and hence heavier – because it can reduce the comfort of wearing and reduce movement. For the production of thermal insulation layers, wool and aramid, aramid-viscose and polyester fibers are used [18].

The lining is a layer of special clothing that is the closest to the body. It can be a separate layer or be combined with a thermal insulation layer. Lining is made of impregnated cotton fabrics, aramid or aramid-viscose fibers [18].

PBI is an organic fiber originally developed in the Apollo project for NASA due to non-flammable properties. Since 1983, they have been recognized as one of the best materials applicable, among others as a protective outer shell. It has characteristic properties: non-flammability (limited oxygen index LOI > 41), non-inflammability, non-shrinkage, thermal stability at high temperature, high chemical resistance to solutions of acids and inorganic bases and organic chemical compounds, retaining elasticity under the flame [14].

Auxsetic fabrics (with negative Poisson's ratio) are currently studied mainly in laboratory conditions. Despite their high energy-absorbing properties, these innovative materials are not used on an industrial scale. One can use them to protect against an explosion wave.

The basic value for describing the behaviour of auxetic materials is the Poisson's ratio described in accordance to the following equation:

$$\nu = -\frac{\varepsilon_{\text{trans}}}{\varepsilon_{\text{load}}}, \tag{2.1}$$

where $\varepsilon_{\text{trans}}$ – transverse strain, $\varepsilon_{\text{load}}$ – strain in the direction of the load.

A common and intuitive phenomenon is the fact that transverse deformations and longitudinal ones have opposite directions. Therefore, a negative sign is used in formula (2.1). In the auxetic materials, there is an unusual situation when Poisson's ratio is negative. In the case of a two-dimensional scheme of behavior of materials with different ratios are shown in Fig. 2.

The auxetic threads are made of two types of fibers. The first of them is inextensible and has a relatively small diameter. It is entwined around the elastic

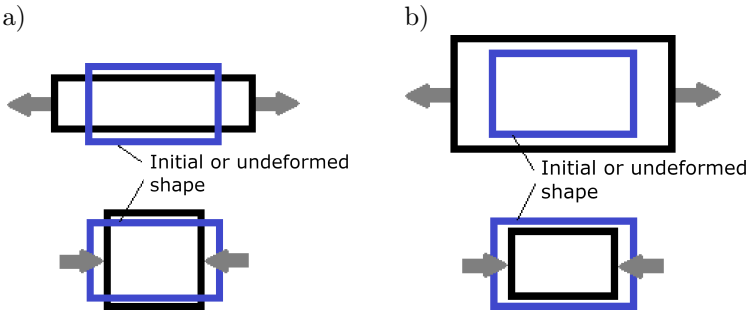

FIG. 2. 2D scheme of material behavior: a) typical material, b) auxetic material.

fiber with a larger diameter in the so-called the "screw" way (Fig. 3). When the thread is stretched in the longitudinal direction, the inextensible fiber straightens and in this way the initially straight thread becomes wavy. Of course, the elastic fiber reduces its diameter, but due to the sideways spreading it gives the effect of a negative Poisson's ratio. If the thread arrangement is consistent with the one shown in Fig. 3c, the stretching causes the neighbouring threads to be pushed. Ultimately, it results in widening of the area occupied by the whole group (Fig. 3). It is important to put together neighbouring threads that should remain in counter phase. One can see that if they are arranged in phase, as shown in Fig. 3e, the observable auxetic effect is minimized [20, 21].

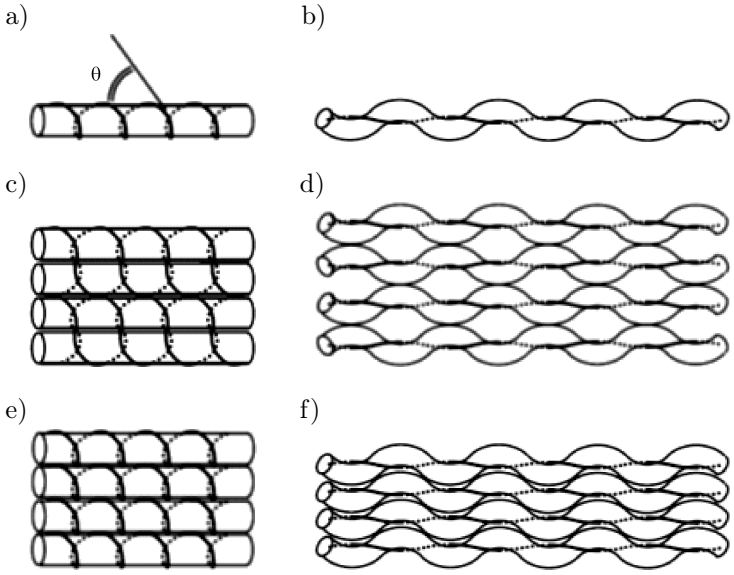

FIG. 3. Concept of building a single fiber and a group of fibers: a) single thread before stretching, b) stretched thread c) threads arranged in counter phase, d) stretched threads in counter phase presenting the effect of auxeticity, e) threads arranged in a phase, f) ineffective thread arrangement resulting in a reduction of the effect of auxeticity [20].

Among many techniques that increase the durability of engineering materials, the most important role in industrial practice is played by [22]:

- PVD (Physical Vapour Deposition),
- CVD (Chemical Vapour Deposition),
- PS (Plasma Spraying),
- hybrid (multiplex) methods, enabling full control over composition, structure and properties, using the characteristics of individual methods.

The PVD technique consists in converting a solid material into a gas phase by means of various physical processes. The obtained steam is cooled and then deposited on the substrate material. A magnetron spray is one from varieties of PVD techniques.

According to the current state of knowledge, reactive magnetron deposition of layers is carried out most often by means of: sputtering with high frequency AC, medium frequency, HIPIMS (High Power Impulse Magnetron Sputtering) and MPPMS (Modulated Pulse Power Magnetron Sputtering) [22].

3. Researched materials

For the purpose of proposed research PROTON fireproof textile was selected. PROTON is made of paraaramid 58%, PBI 40%, and antistatic 2% (Fig. 4a,b). The textile was covered with TiSi(N) reflective nanolayer (Fig. 4c,d).

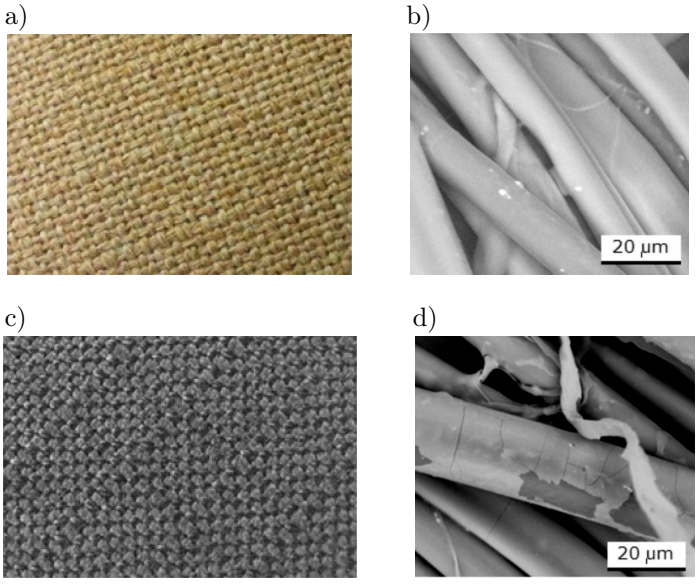

Fig. 4. PROTON fireproof textile: a) view of as received textile, b) SEM image of as received textile, c) textile coated with TiSi(N) layer, d) SEM image of textile coated with TiSi(N) layer.

Nanocomposite TiSi(N) layer was applied in the specially designed magnetron gun. The development of the technology of obtaining titanium-silicon sinters with contents of up to 10% at. Si was the innovative idea implemented in the construction. The titanium-silicon sinters formed the basis of a coating-forming material in accordance with patent PL215960B1 for obtaining TiN-Si_3N_4 composite coatings. The final goal of this task was to create cathodes for linear magnetrons. It is worth noting here that previously composite layers applied with one of the PVD (or CVD) techniques required the use of at least two independent element sources (separately silicon, separately titanium) forming the layer. The use of a cathode with a specific chemical composition provides much better reproducibility and stabilization of the conditions during the reactive deposition of layers.

The starting materials for the production of sinters were titanium and silicon microparticles. The titanium and silicon micropowders were purchased in Atlantic Equipment Engineers company. The titanium micropowder was characterized by medium graining, according to sieve analysis, 325 mesh, which corresponds to the 44 μm in metric scale. According to the manufacturer's data, titanium microparticles had analytical purity not lower than 99.90%.

The Ti and Si micropowders to be sintered were observed on a scanning microscope. Figure 5 shows the image of titanium micropowder.

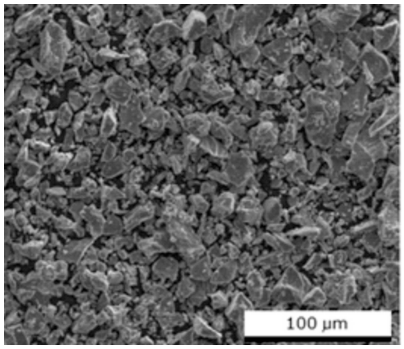

FIG. 5. Titanium microparticles.

A mixture of titanium and silicon micropowders in an amount of 90% at. Ti and 10% at. Si, or so-called reaction bed, was homogenized.

Figure 6 shows the so-called pressing curve conditions for sintering the prepared powder bed. The first of the curves illustrates the course of changes in the applied pressure. It can be seen that the maximum pressure of approx. 11 Torr was obtained after 35 min and maintained at this level for 85 min. The course of temperature changes is illustrated by the second of the curves visible in Fig. 6. The maximum temperature (about 1300°C) was reached after 80 minutes and it was kept for almost 40 minutes. The third curve (the "stepped" one) shows the

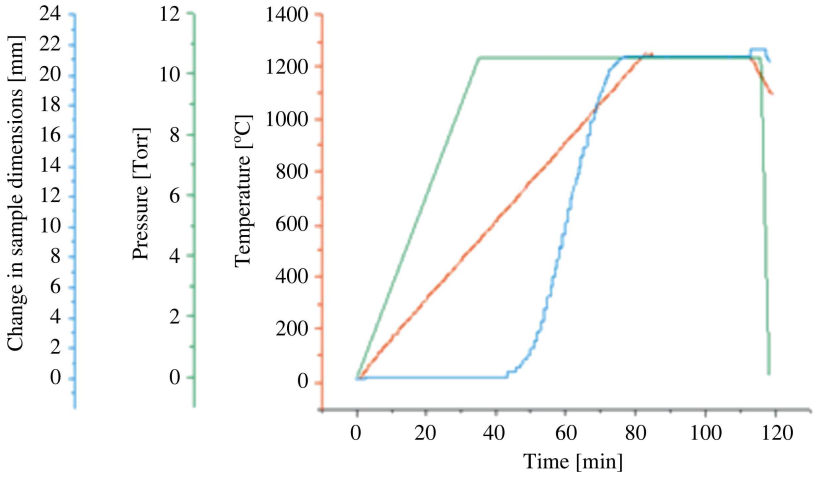

FIG. 6. Pressing curve of composition of 90% at Ti and 10% at Si in three-inch matrix.

change in the position of the piston, which illustrates the process of the deposit compacting. It can be seen that the piston displacement stopped after reaching a temperature of approx. 950°C. The process was carried out in a protective atmosphere of argon. After the furnace was cooled, the sinter was removed from the matrix and mechanically treated to remove graphite foil.

The obtained sinters were examined using a scanning microscope equipped with a chemical composition microanalyzer. Figure 7a shows the obtained surface image of the sinter and Fig. 7b shows a finished TiSi disk with a diameter of 50 mm.

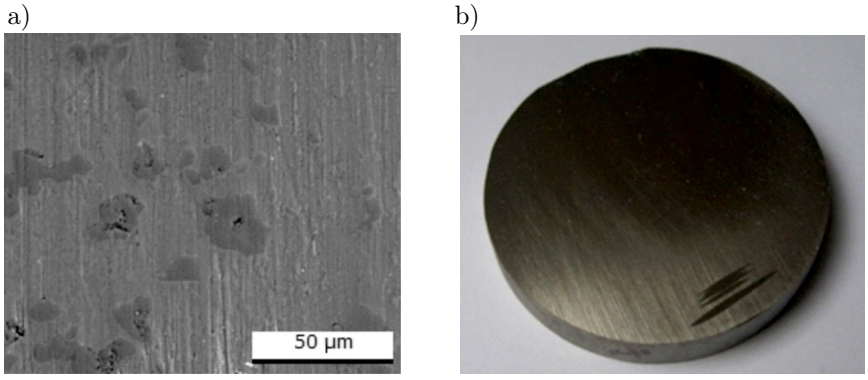

FIG. 7. a) Image of titanium and silicon with 10% at. Si sinter surface, b) TiSi sintered disc with diameter of 50 mm.

Figure 8 shows a ready-made linear magnetron. On the surface of the sinters, the spraying area of the tested prototype linear magnetron is visible. The size of

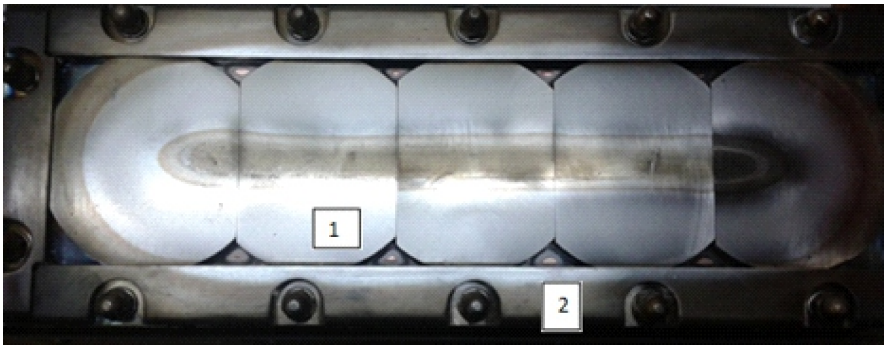

FIG. 8. Linear magnetron: 1 – titanium-silicon sinters, 2 – holders for fixing sinters.

the sprayed area is about 70% of the target surface (magnetron cathode). The presented magnetron is characterized by the so-called "virtual anode" (no material anode). The presented linear prototype magnetron was installed in a vacuum chamber.

The length of the cathode was 30 cm and the width was 11 cm. The base for mounting the launcher had a height of 12 cm and a vacuum port with a diameter of 32 mm through which a high voltage supply pipe and a water supply for cooling the cathode was introduced. The layering parameters were as follows: partial pressures of gases $P_{\mathrm{Ar}} = 0.116$ Pa, $P_{N2} = 0.101$ Pa; total pressure $P_{\mathrm{Ar+N2}} = 0.217$ Pa; partial pressures ratio $P_{\mathrm{N2}}/P_{\mathrm{Ar}} = 0.87$; Magnetron M1 – $I = 1.14$ A, $M_{\mathrm{eff}} = 0.87$ kW, $M_{cr} = 0.40$ kW; Magnetron M2 – $I = 1.07$ A, $M_{\mathrm{eff}} = 0.71$ kW, $M_{cr} = 0.37$ kW; layer composition Ti, Si 30 nm/(Ti, Si)N 114 nm; layer total thickness 144 nm; time of layering 1.5 min in Ar, 19.5 min in Ar $+N_2$; final temperature 196°C.

The applied auxetic fabric is composed of elastomer core and Kevlar braid (Fig. 9).

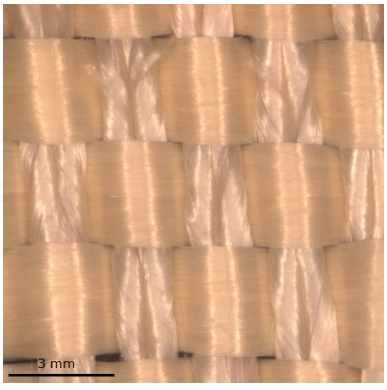

FIG. 9. Structure of auxetic textile.

4. Experimental research

Two kinds of tests were carried out:

- comparative study on PROTON thermal resistance with and without TiSi(N) coating,
- comparative study on PROTON and PROTON – auxetic structure gas impact resistance.

Special testing stands were built to achieve those aims. It must be mentioned that there is no influence of coating on PROTON gas impact resistance causing the layer local damages, what was shown in [23].

The applied TiSi(N) coating was 200 nm thick.

4.1. Thermal resistance testing

As it was mentioned a special testing stage was built to study the thermal resistance of textiles.

Heating of the material was carried out by CAT HC17.5D device, having a heating plate made of a ceran with dimensions of 125×125 mm, which can be heated up to 500°C. The device was equipped in a microprocessor temperature controller type FUZZY-LOGIC, ensuring temperature stabilization at the level of up to ±1.0°C. A sample was placed 5 mm far from heating plate. The sample was attached to the stand with a rotating base (Fig. 10a). An aluminum plate, 90×90 mm, was fixed to the heating plate to uniformly distribute the heat and uniformly heat the test sample. A thermovision camera observing the entire area of the test sample was placed in front of the stage.

a) b)

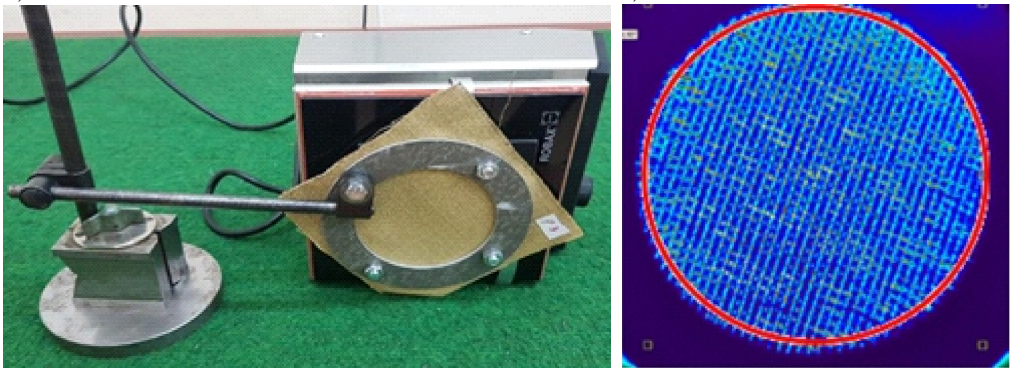

Fig. 10. a) Thermal resistance testing stage; b) sample measuring area.

The device was heated to 330°C and after reaching the thermal equilibrium a material sample was applied to a predetermined distance. The value of the set

temperature was dictated by the technical capabilities of the thermal imaging camera which current measuring range was 340°C. The sample was heated for 5 minutes. The sample measuring area was shown marked in Fig. 10b (marked in red). The mean temperature value was read from the selected area.

Temperature maps achieved for particular time moments were shown in Figs 11 and 12. The graph of the recorded temperature increase for the PROTON material with the surface layer and without the layer was presented in Fig. 13.

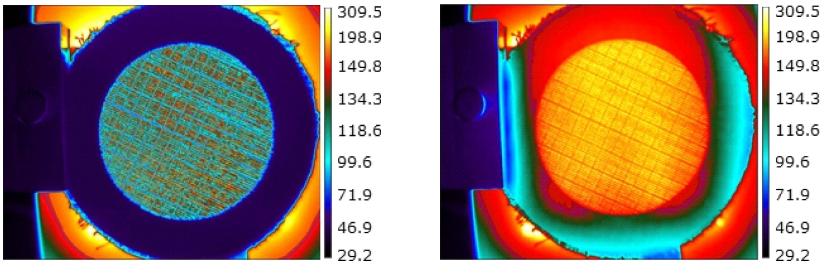

FIG. 11. Temperature [°C] maps for PROTON without TiSi(N) layer heating: test beginning (left), test end (right).

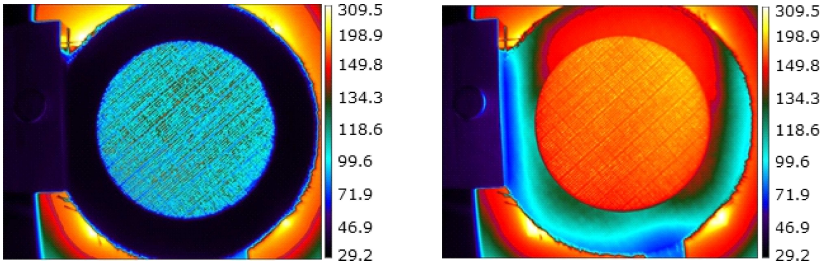

FIG. 12. Temperature [°C] maps for PROTON with TiSi(N) layer heating: test beginning (left), test end (right).

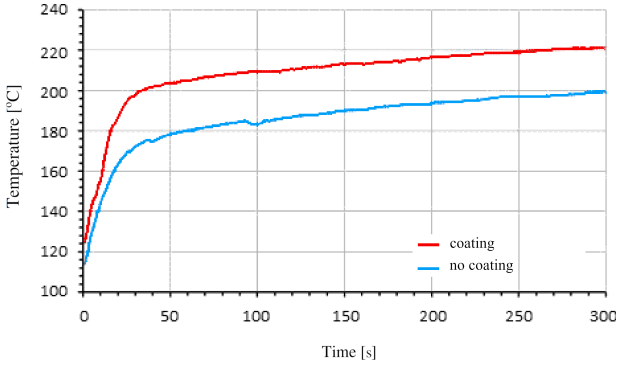

FIG. 13. Recorded temperature vs. time chart for PROTON material with and without surface layer.

4.2. Gas impact resistance test

Samples made of a multi-layered protective structure of fireman's clothing supplemented with a layer of auxetic material were the subject of experimental research. Samples were marked with numbers from 1 to 4. Experimental investigations included tests of the energy dissipation capacity of gas pressure impact acting on multilayer structures. The samples provided for testing were presented in Fig. 14. They were specially prepared by gluing fabrics between composite rings to ensure the tension of the auxetic fabric during the experiment.

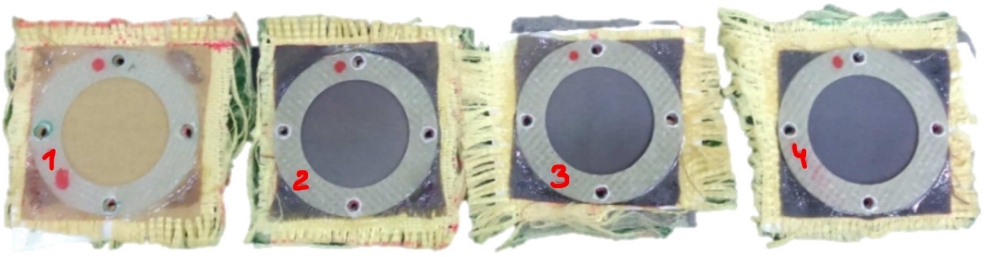

FIG. 14. Samples for gas impact resistance test.

The tests of the impact of the gas pressure on the fabric were carried out on the measurement stand shown in Fig. 15. Pressure was applied with the use of compressed gas. The pressure tank 2 was filled with argon from the bottle 3 via a pressure reducer 4 and a filling proportional valve 5. A pressure gauge 6 was used to control the pressure in the tank. After mounting the sample 9 in the holder 10 and fixing the base of the holder 11 to the base of the station 1, a test was carried out by opening the drain valve 7. The compressed gas through the

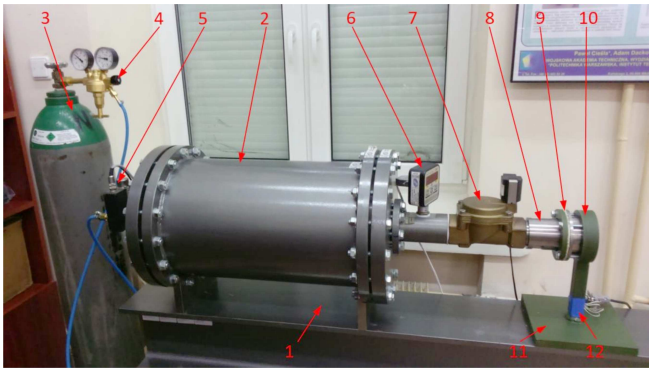

FIG. 15. Measuring stand for testing fabrics loaded with a gas pressure impulse: 1 – stand basis, 2 – pressure tank, 3 – gas cylinder (argon), 4 – pressure reducer, 5 – filling proportional valve, 6 – gauge control, 7 – valve, 8 – nozzle, 9 – sample, 10 – sample clamp, 11 – handle base, 12 – force measurement system.

nozzle 8 acted on the test sample. Control of the loading system was carried out using a specialized computer program. The tests assumed the initial pressure of 8 bar and the loading pulse duration of the 200 ms. The force measuring system 12 allowed a qualitative and quantitative assessment of the impact of the pressure pulse on the tested materials. Electrical signals were recorded and archived using a computer program.

The results of gas impact resistance test were shown in Fig. 16 as force – time chart.

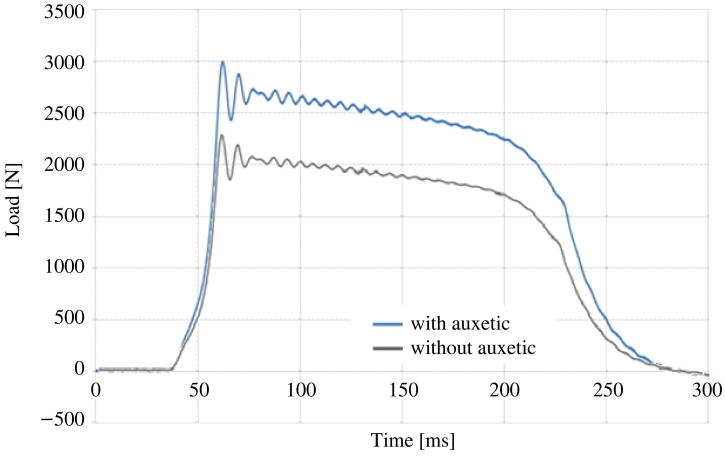

Fig. 16. Gas impact resistance test results.

5. Results analysis and conclusions

As it was shown in [10] the temperature increase from 20 to 180°C causes the decrease in auxetic effect of even 13.2%.

The results of thermal resistance of coated and not coated PROTON shown that the application of such structure can decrease the temperature acting behind the textile from 180 to 100°C (45%). It can be explained by the effect of the reflective layer application. One of the more interesting properties of c-TiN/a-Si_3N_4 layers applied on aramid, Nomex or Kevlar type fabrics is the ability to significantly improve the transmission resistance of the energy stream. The research, the results of which were partially presented in several works [24–26], showed that the temperature of 60°C – defining the so-called pain threshold, with a thermal energy stream of approx. 36 kW/m^2 from a source with a temperature of 600°C, is achieved with Nomex type fabric in a few to several seconds, and covering this fabric with, for example, c-TiN/a-Si_3N_4 extends this time from about 10 seconds to about 120 seconds. Hence the practical applications of composite layers on fabrics can be widen to production of clothing for all types of emergency services. Extending the time of possible evacuation from the danger

zone is of enormous importance for the protection of life of rescuers. The use of such modified fabrics for the production of fire curtains, image screens in museums, furniture coverings in theater and cinema halls, etc. is also foreseen. There is another area of potential applications – fabrics for making suits for F1 drivers. These suits undergo very rigorous tests before they get the approval of the International Automobile Federation (FIA). The fabrics are not only washed and chemically cleaned 15 times, but must additionally pass a real fire test and withstand a temperature of 820°C for 10 seconds.

The next tests showed that the application of the auxetic textile can increase the PROTON resistance to gas impact of 18.2%. The method of dissipating the energy of the pressure wave, including the explosive wave, through the auxetic fabric was described in the patent [27]. It presents many possible applications of the type of discussed fabric, but the description of the essence of the explosion energy dissipation is particularly important. According to the diagram shown in Fig. 17, initially flat wave penetrating the resulting pores in the material changes its shape. As a result, less energy reaches the target and can be treated as its dissipation.

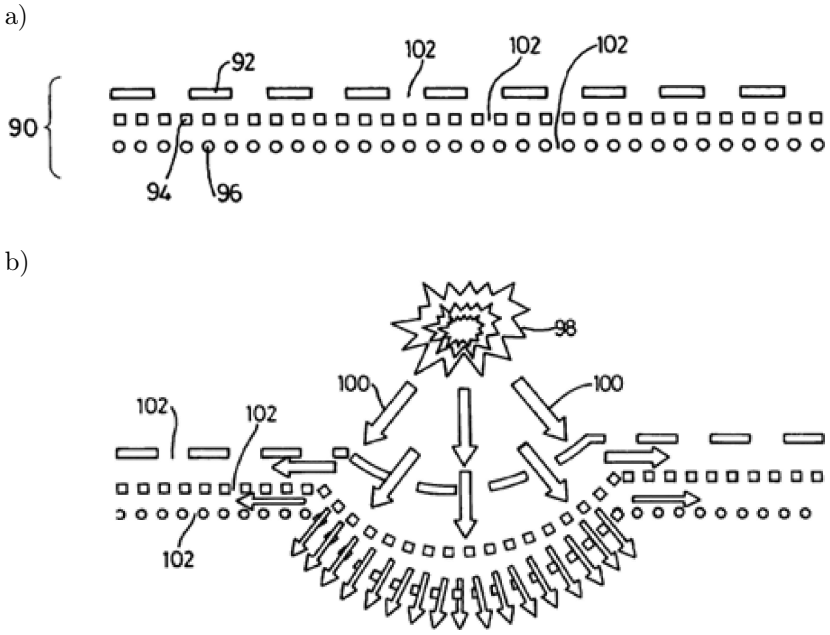

Fig. 17. Dissipation of the explosive wave through auxetic fabric: a) pre-explosion state, b) fabric under pressure [27].

Both achieved results allow to consider the coupling of these phenomena in one construction. The effect of proposed coupling scheme was shown in Fig. 18.

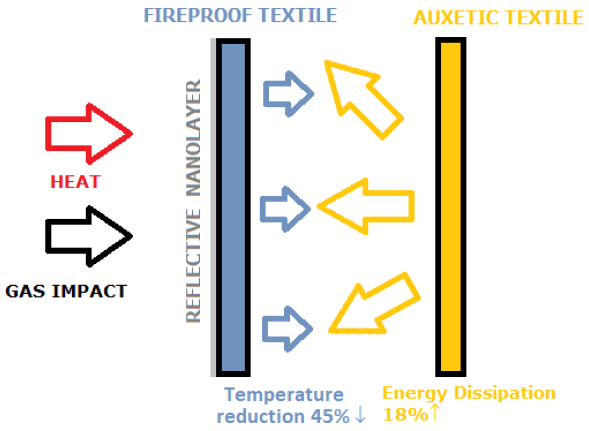

FIG. 18. Scheme of fireproof and auxetic textiles coupling.

Looking from the point of view of high temperature and pressure wave threat due to the use of these two materials (PROTON with reflective layer and auxetic fabric), a double protective effect can be obtained. Fireproof textile covered with reflective coating protects against the rapid temperature increase, the auxetic textile protects against the pressure impact. But the auxetic phenomenon decreases with the temperature increase – so the fireproof textile allow to keep its energy dissipation high protection. In the same time auxetic textile improves the gas impact protection ability of fireproof one.

Finally on the base of achieved results it can be concluded that using both improvement: TiSi(N) nanocoating and auxetic fabric can improve energy absorption and thermal resistance of fireproof textile.

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