Polish Society of Composite Materials
Polskie Towarzystwo Materiałów Kompozytowych
18: 2 (2018) 110-114
COMPOSITES THEORY AND PRACTICE

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Received (Otrzymano) 8.04.2018

STUDY ON FIREPROOF TEXTILE HEAT RESISTANCE IMPROVEMENT WITH TiSi(N) NANOCOMPOSITE COATING OF VARIOUS THICKNESSES

Fireproof textiles must exhibit high heat resistance. Many methods to improve this property are known. They can be achieved, for example, using special chemical additives during fiber production. In the paper the use of a nanocomposite layer is proposed. NATAN and PROTON fireproof textiles were coated with a TiSi(N) nanocomposite layer using magnetron sputtering technology. Three layer thicknesses of 200, 300 and 400 nm were applied. The thermal barrier effect for heating up to 100 and 330°C was studied on specially designed testing equipment. The influence of the layer thickness on the textile heat resistance was visible at 100°C. For the thickest layer a worse effect was observed, which could be caused by the thermal conductivity of the composite layer. However, the proposed layer raised the heat resistance of the textiles.

Keywords: nanocomposite layer, fireproof textile, heat resistance

BADANIA POPRAWY ODPORNOŚCI CIEPLNEJ TKANIN OGNIOODPORNYCH POPRZEZ UŻYCIE NANOKOMPOZYTOWYCH WARSTW WIERZCHNICH TiSi(N) O RÓŻNYCH GRUBOŚCIACH

Tkaniny ognioodporne muszą charakteryzować się wysoką odpornością cieplną. Znanych jest wiele metod ulepszania tej właściwości. Można ją uzyskać na przykład za pomocą specjalnych dodatków chemicznych stosowanych podczas produkcji włókien. W artykule zaproponowano zastosowanie warstwy nanokompozytowej. Tkaniny ognioochronne NATAN i PROTON zostały pokryte warstwą nanokompozytową TiSi(N) przy użyciu technologii rozpylania magnetronowego. Zastosowano trzy warstwy o grubości 200, 300 i 400 nm. Efekt barierowości termicznej dla ogrzewania do 100 i 330°C został zbadany na specjalnie zaprojektowanym stanowisku eksperymentalnym. Wpływ grubości warstwy na odporność cieplną badanych materiałów był widoczny w 100°C. Dla najgrubszej warstwy zaobserwowano gorszy efekt, co może być spowodowane przewodnością cieplną warstwy kompozytowej. Jednak proponowana warstwa pozwoliła na zwiększenie wytrzymałości cieplnej tekstyliów.

Słowa kluczowe: warstwa nanokompozytu, tkanina ognioodporna, odporność na ciepło

INTRODUCTION - AIM OF RESEARCH

One of the most important properties of fireproof textiles is heat resistance. This factor can be achieved using special fibers, applying additives to the fiber chemical composition or by means of special finishes like coatings.

For heat and flame protection, the requirements range from clothing for situations in which the wearer may be subjected to occasional exposure to a moderate level of radiant heat as part of his/her normal working day, to clothing for prolonged protection, where the wearer is exposed to severe radiant and convective heat or direct flame, for example a firefighter's suit [1]. The influence of high temperature on a textile can produce physical and chemical changes [2-4]. For example in thermoplastic fibers, physical changes occur at the second order transition and melting temperature, and chemical changes take place at pyrolysis temperatures when thermal degradation starts. Moreover, the combustion of textiles is a complex process that involves heating, decomposition leading to gasification, ignition and flame propagation. As mentioned, the fibers could be classified into three categories. The first one is inherently heat and flame retardant fibers (e.g. aramid [5, 6], modacrylic [1], polybenzimidazole (PBI) [7, 8], Panox (oxidised acrylic) [9] or semicarbon, phenolic, asbestos, ceramic [10]). The second category is chemically modified fibers and fabrics, in which one can find flame retardant cotton, wool and viscose [11-13] as well as synthetic fibers [14-16], produced by incorporating special additives in the spinning dope before extrusion. The last category of fireproof textiles is currently being developed [17-19] - fibers coated with a reflective nanocomposite layer by means of the sputtering method.

In the paper a study on the heat resistance of fireproof textiles was carried out. The improvement was made by applying a TiSi(N) nanocomposite coating of three different thicknesses: 200, 300 and 400 nm.

Some other studies can be found concerning thermal barrier coating (TBC) testing. The original method of the isolating capabilities of TBC testing was presented in [20]. Differential measurement was proposed. Two samples: with the coating and a reference one were placed in the stove doors. Heating was achieved with an electric stove and the back surfaces of the samples were cooled with air. The temperature was measured with thermocouples. The first thermocouple was placed in the stove (T_0), the second and third one on the back surfaces of the samples (T_1, T_2). After reaching the temperature of 1000°C the isolating capability was calculated using $\Delta t = T_2 - T_1$.

The TBC thermal shock resistance testing stage was presented in [21]. In the experiment continuous cooling of the sample with compressed air or water was assessed. The samples were placed 100 mm from the burner. The measurement was carried out by thermocouples.

The effectiveness of TBC in the context of the combustion of various fuels was presented in [22]. The combustion products heated a steel disc coated with 45 micron 8YSZ - zirconium(IV) oxide-yttria stabilized nanopowder. The disk had space for four thermocouples. Cooling was carried out using a water tank placed in the upper part of the system. During the tests, the temperature of the disc in the lower part from the side of the flame, the upper part from the side of the water tank, flame temperature, temperature of the surface in contact with the flame, as well as the temperature of the water and steam in the tank were recorded.

An interesting discussion of the thermal conductivity of the coating with the designation 8YSZ can be found in [23]. Coatings of this type are used in turbines due to their low thermal conductivity. A furnace with three temperature zones heated the sample placed in its central part. To limit the outflow of heat, the sample was threaded and secured with insulation blocks. Two thermocouples were placed inside the sample and the surface temperature was measured using a third thermocouple. The research was carried out in the temperature range of 200÷1000°C. Temperature measurements were made every 3 hours to ensure stabilized operating conditions of the device.

RESEARCH MATERIAL

Two fireproof textiles were considered NATAN and PROTON. They are dedicated to the production of firemen's suits. NATAN is composed of 75% Nomex, 23% Kevlar, and 2% P140 (carbon fiber), and PROTON is made of para-aramid 58%, PBI 40%, and antistatic fiber 2%.

The TiSi(N) coatings (Fig. 1) were developed by means of the dc magnetron sputtering method. The coating development process was preceded by glow discharge for the purpose of textile degasification. The discharge parameters were as follows: pressure $4 \cdot 10^{-3}$ mbar, electric current under 1 A, and effective power 0.8 kW. The coating development process was carried out in an atmosphere of argon of $2 \cdot 10^{-3}$ mbar from two launchers with a discharge current of 2.5 A and an effective power over 1.5 kW. The temperature of the base was below 70°C. The thicknesses of the developed coatings were 200, 300 and 400 nm.

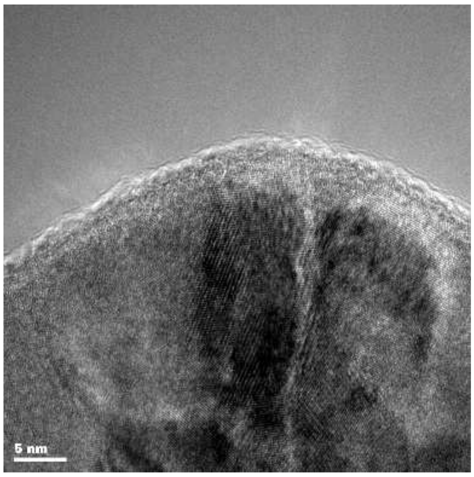


Fig. 1. TiSi(N) nanocomposite layer microstructure visualized with SEM (scanning electron microscope)

Rys. 1. Mikrostruktura kompozytowej warstwy TiS(N) zwizualizowana z użyciem SEM (elektronowy mikroskop skaningowy)

NATAN without and with coating is shown in Figures 2 and 3, and PROTON - in Figures 4 and 5.

a) b)

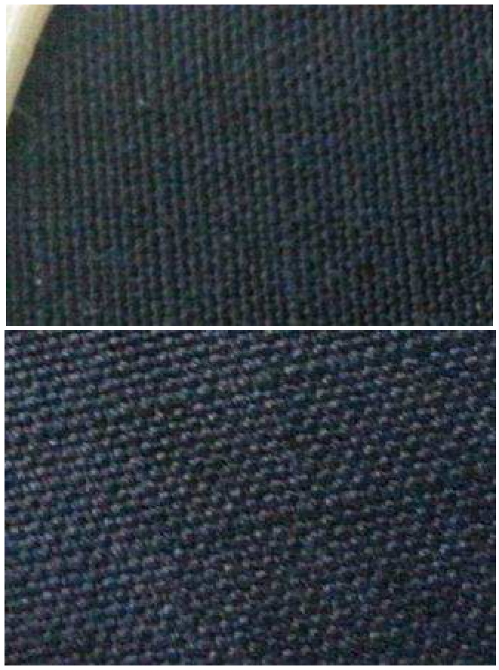


Fig. 2. NATAN without coating (a) and with coating (b)

Rys. 2. NATAN bez warstwy wierzchniej (a) i z warstwą (b)

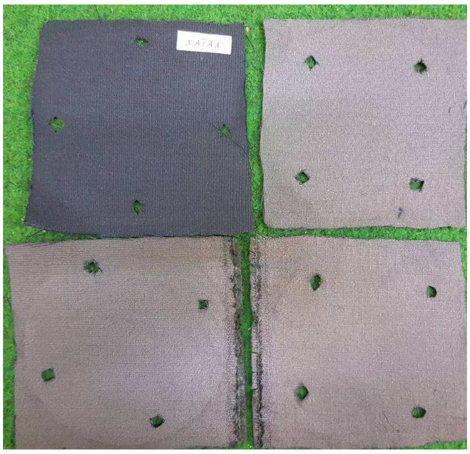


Fig. 3. Samples made of NATAN with coating of various thicknesses: N - without coating, N1 - 200 nm, N2 - 300 nm, N3 - 400 nm

Rys. 3. Próbki wykonane z tkaniny NATAN pokrytej warstwą powierzchniową o różnej grubości: N - bez warstwy, N1 - 200 nm, N2 - 300 nm, N3 - 400 nm

a)

b)

Fig. 4. PROTON without coating (a) and with coating (b)

Rys. 4. PROTON bez warstwy wierzchniej (a) i z warstwą (b)

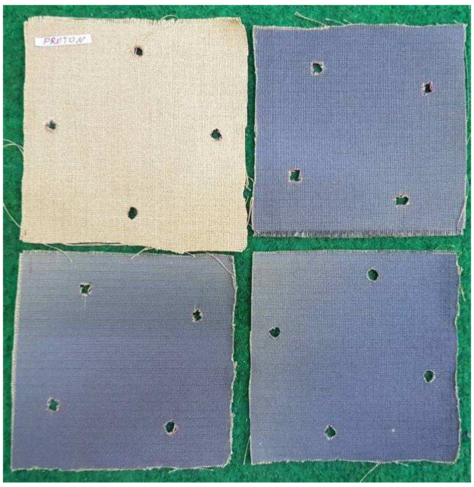


Fig. 5. Samples made of PROTON with coating of various thicknesses: P - without coating, P1 - 200 nm, P2 - 300 nm, P3 - 400 nm

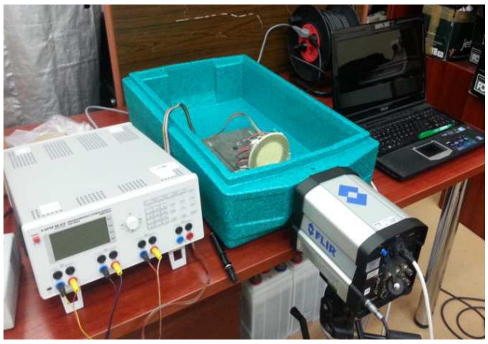
Rys. 5. Próbki wykonane z tkaniny PROTON pokrytej warstwą powierzchniową o różnej grubości: N - bez warstwy, N1 - 200 nm, N2 - 300 nm, N3 - 400 nm

RESEARCH METHODOLOGY

Two test stations were constructed to study the thermal resistance of the coated textiles at temperatures of 100 and 330°C.

The first station allowing heating to 100°C is presented in Figure 6. It consisted of four Peltier cells fixed on a movable base, which allowed changing of the distance between the heat source and sample in the range of 0÷150 mm. Control of the cells was achieved by the power supply unit (a change in the unit voltage resulted in a temperature change of the cells). The setup was placed in a thermal insulated box.

a)



b) c)

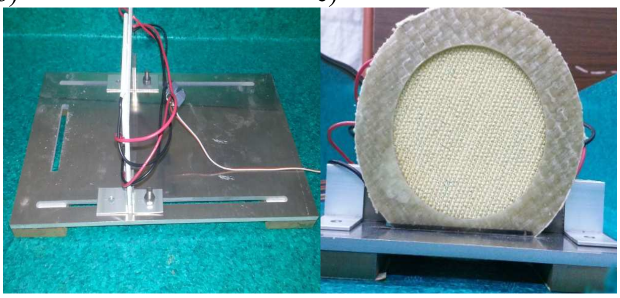


Fig. 6. Low temperature testing stage: a) general view, b) Peltier cells, c) sample

Rys. 6. Stanowisko do badań niskotemperaturowych: a) widok ogólny, b) ogniwa Peltiera, c) próbka

In the second stage (Fig. 7) heating was performed by a CAT HC17.5D device equipped with a CERAN® hot plate allowing the temperature to reach up to 500°C. The sample was placed on a stand with a rotating base. In addition, an aluminum plate was placed on the heating device surface to achieve uniform heat distribution. The test temperature was 330°C because of the measurement range of the thermovision camera driver configuration.

In both cases the sample was placed at the distance of 5 mm in front of the heat source. The each test was divided into the following stages: heating the equipment to the desired temperature level (100 or 330°C), temperature stabilization, placing the sample in the heating device. Observation of the heated sample lasted 300 s in each case. The sample temperature change was observed with the thermovision camera FLIR Sc6000. The temperature was measured at each time step and the observed values were averaged (the arithmetic average from the temperature values in each pixel) within the

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selected measurement area by means of Flir Tools data processing software.

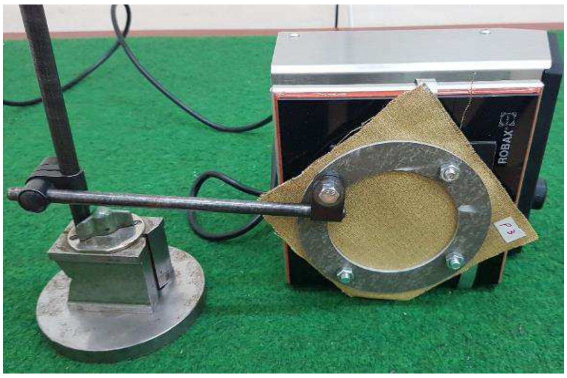

Fig. 7. High temperature testing station

Rys. 7. Stanowisko do badań wysokotemperaturowych

RESULTS AND DISCUSSION

As mentioned, the average temperature observed and measured on the sample surface during heating was calculated at each time step. The results were as presented as temperature vs. time charts (Figs. 8-11).

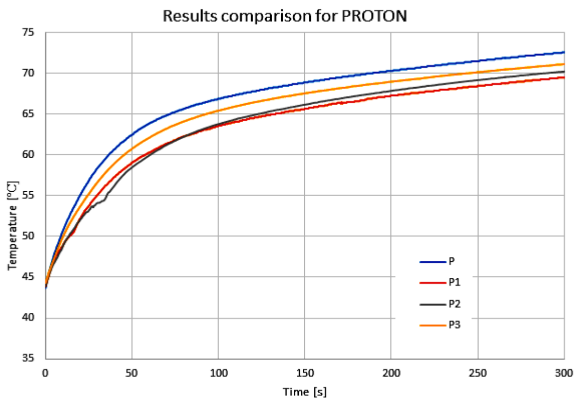

Fig. 8. Temperature vs. time chart for sample made of PROTON for heating to 100°C

Rys. 8. Wykres przebiegu temperatury w funkcji czasu dla próbek z tkaniny PROTON ogrzewanych do 100°C

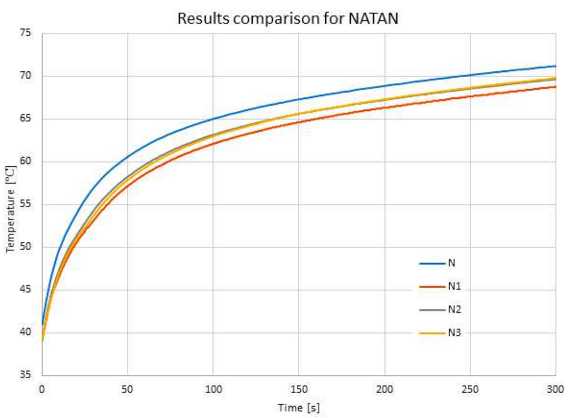

Fig. 9. Temperature vs. time chart for sample made of NATAN for heating to 100°C

Rys. 9. Wykres przebiegu temperatury w funkcji czasu dla próbek z tkaniny NATAN ogrzewanych do 100°C

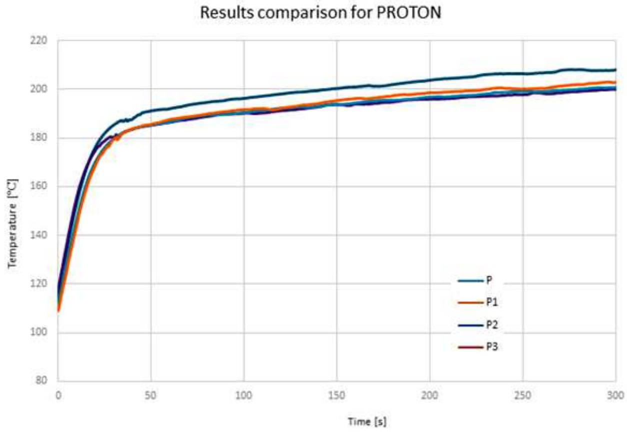

Fig. 10. Temperature vs. time chart for sample made of PROTON for heating to 330°C

Rys. 10. Wykres przebiegu temperatury w funkcji czasu dla próbek z tkaniny PROTON ogrzewanych do 330°C

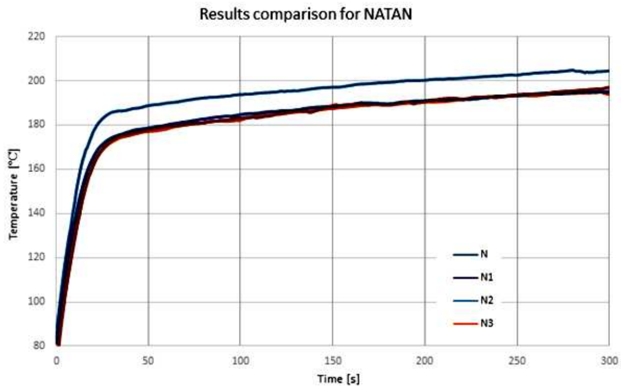

Fig. 11. Temperature vs. time chart for sample made of NATAN for heating to 330°C

Rys. 11. Wykres przebiegu temperatury w funkcji czasu dla próbek z tkaniny NATAN ogrzewanych do 330°C

It can be noticed that the results of the measurements at 100 and 330°C differ from each other. In the case of the first type of measurement (heating up to 100°C) of both the PROTON and NATAN fabrics, the temperature differences obtained with and without the top layer are small and do not exceed 5°C. The applied layer reduces the thermal radiation to a small extent. The obtained graphs show that the best thermal radiation reflection is for the thinnest layer, 200 nm thick, for thicker layers the effect decreases, which may be related to the high thermal conductivity of the metal from which the layer is built, whose effect exceeds the reflexivity effect in those temperatures. Depending on the fabric, there is a different starting temperature, which leads to the conclusion that the NATAN material has a greater ability to reflect thermal radiation.

In the case of the second type of measurement (heating up to 300°C) also at the beginning of the test, the samples reached different initial temperatures resulting from the penetration of thermal radiation through the structure of the heated fabrics (see fabric characteristics). A temperature of 80°C (mean temperature for all the samples) was recorded for the NATAN fabric, and for the PROTON fabric it was 118°C. Different layer thicknesses do not affect temperature changes. The temperature rise curves for individual layers coincide with each other within the`2 given type of fabric. The temperature difference is visible between the types of fabrics. The difference between the PROTON and NATAN fabrics with the applied layers at the end of the test is about 6°C.

CONCLUSIONS

As a result of the research, it was found that the applied TiSi(N) nanocomposite layer has an influence on improving the heat resistance properties of both NATAN and PROTON fabrics. This is not a significant effect because these fabrics are characterized by a rather loose fiber strand weave, and the applied layer does not affect the fabric structure, where the NATAN and PROTON fibers begin to "swell" and tighten the weave [7, 8]. An interesting conclusion is that at lower temperatures the effect of the thermal conductivity of the metal may outweigh the effect of the reflective layer and degrade the barrier. For higher temperatures this effect loses its significance and the thickness of the layer does not increase the heat resistance of the special fabric.

Acknowledgements

The paper was supported by grant No DOB-BIO6/04/104/2014 financed in the years 2014-2017 by The National Centre for Research and Development, Poland.

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