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Effect of Accelerated Ageing on Ballistic Textiles Modified By Plasma-Assisted Chemical Vapour Deposition (PACVD)

DOI: 10.5604/12303666.1167429

Abstract
The functionalisation of textile products and materials using the low temperature plasma technique is increasingly used in industrial practice, mainly because of the many benefits, often unattainable with the use of other techniques. The study focused on the modification of the composite textile materials - sheets of Ultra High Molecular Weight Polyethylene (UHMWPE) fibres (Dyneema® SB51) by applying the surface deposition of silicone-like polymer in an environment of low temperature plasma. The aim of this study was to evaluate changes in the mechanical and physical properties of modified ballistic textiles under simulated storage conditions in accelerated time. Based on experiences in planning the research on accelerated aging, a test programme was developed for the assessment of changes during the aging process in ballistic fibrous materials under various environmental conditions. The process of low temperature plasma modification affected the mechanical properties of the ballistic composite textile materials during the accelerated ageing using temperature or temperature and humidity. The effect of the modification varied with the mechanical properties investigated.

Key words: *UHMWPE fibrous composites, low temperature plasma assisted deposition, accelerated aging.*

Introduction

Low-temperature plasma techniques are superior to the currently common finishing technologies due to the lack of the need to use water nor large quantities of leachable substances, often toxic to humans and the environment; hence they are environment-friendly processes [1].

Moreover in terms of ecology, economy and technology, this process is considerably more efficient than standard finishing ones. In a relatively rapid technological process, the properties of the initial material may be altered in a controlled manner to get an increased effect of wetting and adhesion, to induce hydrophobic and oleophobic properties, change the physical and electrostatic properties, and to purify as well as disinfect even large areas of material [6].

Functionalisation, i.e. increasing the functionality or establishing new targeted properties with low temperature plasma allows for creating thin layers of nanometre thickness, thereby creating new functional groups while minimizing thermal degradation processes and getting a relatively rapid and sustained result [2, 3].

Ballistic inserts for personal body armour are now based on textile materials in the form of woven fabrics of para-aramid fibres, or fibrous sheets of oriented UHMWPE fibres, embedded in a polymer matrix and combined in a multi-layered packet. Qualitative and quantitative composition of the inserts depends largely on the properties of the types of ballistic materials applied and varies with the class of ballistic resistance assumed.

Due to the properties of ballistic inserts currently in use, both soft and hard (composite) , it is impossible to obtain a maximal protection area, high class of ballistic resistance and ergonomics. When designing a personal ballistic armour, a balance is reached between security (ballistic resistance and protection only for the vital organs) and ergonomics.

The second aspect considered in the research work is evaluation of the impact of the time of use/storage of personal armours and the conditions in which they were stored/used, to keep the initial protective parameters. Nowadays most manufacturers of ballistic armours guarantee a minimum 5-year period of safe use, which, in the absence of a real ability to control the usage conditions, results in a risk of uncontrolled loss of ballistic resistance [4].

The structure of fibrous materials is significantly more complex than others. For this reason, the functionalisation processes of fibrous materials are more complex, particularly in terms of their homogeneity and stability.

Struszczyk MH, Puszkarz AK, Miklas M, Wilbik-Hałgas B, Cichecka M, Urbaniak-Domagała W, Krucińska I. Effect of Accelerated Ageing on Ballistic Textiles Modified By Plasma-Assisted Chemical Vapour Deposition (PACVD). *FIBRES & TEXTILES in Eastern Europe* 2016, Vol. 24, 1(115): 83-88. **DOI:** 10.5604/12303666.1167429

Table 1. *Properties of initial UHMWPE fibrous composite - Dyneema® SB51 (initial ballistic textile); (1) No tear of sample was observed, (2) No water permeation was observed.*

Surface density, g/m^2	Thickness, mm	Tear resistance	Tensile strength, N		Elongation at maximal force, %		Bursting strength, N	Average water absorbability, %	Average water permeability, cm^3/dm^2	Water tightness, cm H_2O
PN-EN ISO 2286-2:1999	PN-EN ISO 2286-3:2000	PN-EN ISO 4674-1:2005	PN-EN ISO 1421:2001				PN-EN 863:1999	PN-EN 29865:1997		PN-EN 20811:1997
			lengthwise	crosswise	lengthwise	crosswise				
257 ± 3	0.29 ± 0.01	-(1)	8679 ± 715	9216 ± 817	4.10 ± 0.36	3.7 ± 0.5	224.0 ± 6.9	7.62 ± 0.37	-(2)	611 ± 24

Table 2. *Properties of UHMWPE fibrous composite - Dyneema® SB51 modified by low temperature plasma deposition of silicone; (1) No tear of sample was observed, (2) No water permeation was observed.*

Surface density, g/m^2	Thickness, mm	Tear resistance	Tensile strength, N		Elongation at maximal force, %		Bursting strength, N	Average water absorbability, %	Average water permeability, cm^3/dm^2	Water tightness, cm H_2O
PN-EN ISO 2286-2:1999	PN-EN ISO 2286-3:2000	PN-EN ISO 4674-1:2005	PN-EN ISO 1421:2001				PN-EN 863:1999	PN-EN 29865:1997		PN-EN 20811:1997
			lengthwise	crosswise	lengthwise	crosswise				
258 ± 1	0.29 ± 0.01	-(1)	9047 ± 880	7850 ± 1020	4.3 ± 0.18	4.1 ± 0.2	182.2 ± 7.8	9.18 ± 1.71	-(2)	654 ± 43

Due to applying UHMWPE to composites and ballistics, two trends are currently observed in modifying the properties of that polymer:

- surface adhesive properties, for the quality of composites created;
- tribological and strength properties (mainly an increase in the resistance of the surface layer to wear), also in potential ballistic applications.An increase in the adhesion of UHMWPE to other materials is generally obtained by oxidation of the polymer surface layer, implemented by methods of:
- processing the polymer in glow discharges [5];
- processing in the ozone environment with UV [6 - 8].An increase in resistance to mechanical wear is obtained in various ways, e.g.:
- forming a mixture of UHMWPE with a more mechanically resistant component. e.g. an aramide-co-polyester in the presence of a stabiliser - resulting in an important increase in the durability of tribological usage at a contact pressure of 600 - 2500 kPa [9];
- processing the polymer in glow discharges. Plasma techniques applied to polymer modification are an appropriate tool to modify the surface due to the thermal sensitivity of polymers. By using low-temperature plasma, avariety of reactions can be carried out safely on the polymer surface, such as digestion, cross-linking and thin coatings.Improvement in the mechanical properties of UHMWPE was obtained with the following plasma technologies:
- in gas discharges of the direct-current (DC) type, and in radio (RF) and microwaves, which lead to an increase in cross-linking of the surface layer. The UHMWPE of the top layer showed increased hardness and elastic modulus, reduced mechanical wear from friction and increased resistance to scratching [8, 10, 11].
- in nitrogen plasma ion deposition [12, 13], where increases in surface hardness, elastic module and wear resistance were observed. The results were proportional to the dose applied;
- in nitrogen plasma ion implantation and formation of a titanium layer [14]. The process involved two stages - pre-implantation of N^+ at elevated temperature of the substrate and then a layer of titanium oxides and titanium nitride was formed. An increase in the surface wettability was observed, with the friction coefficient decreasing five-fold, and resistance to mechanical wear increasing approximately 40-times after modification;
- plasma deposition process of the carbon layer [15]. A hard carbon layer was created in the methane plasma using hydrogen as a carrier gas, which increased the hardness of the UHMWPE product, and with an increase in the friction coefficient, reduced tribological wear of the polymer was observed.

In summary, it should be noted that the available publications focus only on modification of simple material systems - fibres made of polyethylene or para-aramid.

The studies presented focused on modifying composite textile materials, fibrous sheets of UHMWPE fibres (Dyneema® SB51), by applying the surface deposition of silicone polymer assisted by low temperature plasma [16, 17].

The aim of this study was to evaluate changes in the mechanical and physical properties of modified ballistic textiles under the impact of simulated storage conditions in accelerated time. Based on the studies [18 - 20] in planning the research on accelerated aging, a test system was developed which allows for assessment of changes in the aging process of textile materials under various environmental conditions.

Materials

Ballistic textiles

The Dyneema® SB51 fibrous composite (DSM/the Netherlands) was used in the study [17, 21]. The main properties of initial UHMWPE fibrous composite - Dyneema® SB51 are presented in ***Table 1***.

Modified ballistic textiles by low temperature plasma assisted deposition (PACVD)

The Dyneema® SB51 fibrous composite (DSM/The Netherlands) was modified by the low temperature plasma assisted deposition (PACVD) of silicone-like polymer according to [16] in the PACVD system: CD 400 PLC R/R model (Europlasma/Belgium) using hexametylodisiloxane, (HMDSO) Sigma Aldrich as a substrate.

The main properties of the modified UHMWPE fibrous composite - Dyneema® SB51 are shown in ***Table 2***.

Methods

Accelerated ageing

The process of the accelerated ageing of modified and unmodified UHMWPE fibrous composites was carried out in an ageing system based on the TK 720 apparatus (BINDER GmbH/Germany) for thermal ageing at 70.0 ± 0.5 °C and low as possible relative humidity of 15.0 ± 1.5%. Due to the low humidity of the test environment, the temperature was established as the main accelerated aging agent.

The second accelerated ageing process was carried out in KBF 240 apparatus (BINDER GmbH, Germany) at a temperature of 70.0 ± 0.5 °C and relative humidity of 65.0 ± 1.5%.

The periods of ageing were established based upon the research of [17] for 28, 35 or 42 days. For the acceleration aging tests, a ballistic package was designed consisting of 22 layers of the UHMWPE fibrous composite (unmodified and modified), 250 mm × 250 mm in size, covered by polyester woven fabrics. The package simulated soft ballistic inserts dedicated for ballistic protection vests.

Properties of the unmodified and modified UHMWPE textile composites

Properties of the ballistic materials were determined in the range described in ***Tables 1 – 2***, i.e.: surface density (according to PN-EN ISO 2286-2:1999), thickness (according to PN-EN ISO 2286-3:2000), tear resistance (according to PN-EN ISO 4674-1:2005), tensile strength and elongation at the maximal force (according to PN-EN ISO 1421:2001), bursting strength (according to PN-EN 863:1999), average water absorbability and average water permeability (according to PN-EN 29865:1997), and resistance to water penetration (according to PN-EN 20811:1997).

The properties to be determined were selected on the basis of the risk analysis elaborated, showing the main usage parameters essential for qualifying the behaviour of the fibrous composite modified.

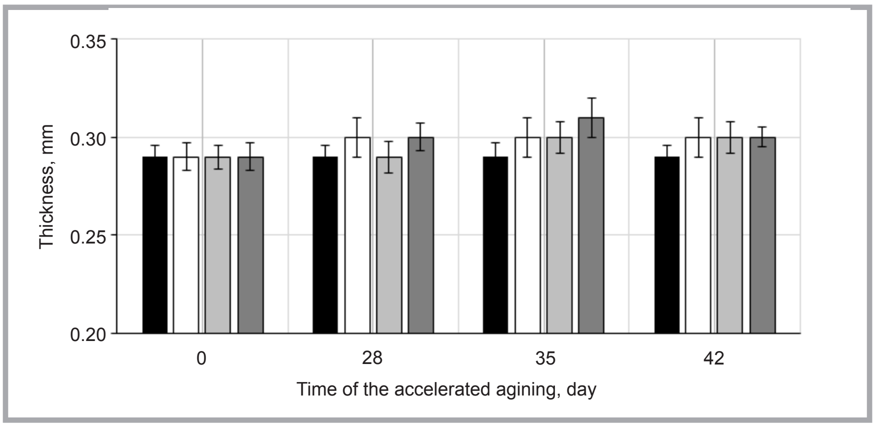

Figure 1. *Effect of accelerated aging at temperature of 70 °C or temperature of 70 °C and relative humidity of 65% on the thickness of the modified and unmodified UHMWPE fibrous composites;* ■ *- unmodified UHMWPE composite after accelerated aging at temperature of 70 °C and relative humidity - 65%,* □ *- modified UHMWPE composite after accelerated aging at temperature of 70 °C and relative humidity - 65%,* ▨ *- unmodified UHMWPE composite after accelerated aging at temperature of 70 °C,* ▩ *- modified UHMWPE composite after accelerated aging at temperature of 70 °C.*

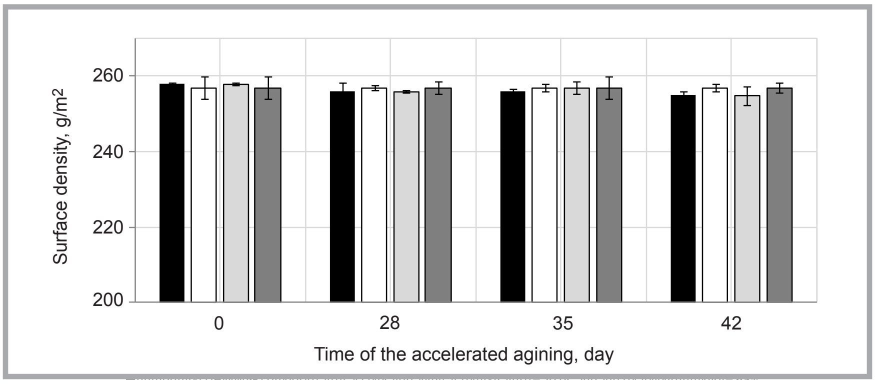

Figure 2. *Effect of accelerated aging at temperature of 70 °C or temperature of 70 °C and relative humidity of 65% on the surface density of the modified and unmodified UHMWPE fibrous composites; designation of the bars as in* ***Figure 1****.*

Results and discussion

Modification with low temperature plasma assisted deposition caused significant changes in a relatively short time in the surface behaviour of the textiles. However, the long term stability of the resulting properties is a crucial matter for estimation of the practical usefulness of the textiles designed. Accelerated aging is the most optimal process for simulation in relatively short time, with good validity of ballistic textile stability [17].

The process of silicone-like polymer deposition on UHMWPE fibrous composites with low temperature plasma treatment yielded insignificant changes in the thickness and surface density, as shown in ***Figures 1 – 2*** and in [16]. The above phenomenon confirms the absence of the influence of the process parameters on the structure of the ballistic materials, i.e. stretching or elongation.

The process of accelerated aging using temperature or synergistically - temperature and humidity, as the aging factors did not influence the changes in the main physical properties of the unmodified and modified UHMWPE fibrous composites (***Figures 1 – 2***).

Both modified and unmodified samples did not tear in test conditions (***Tables 1 – 2***). The accelerated aging conditions: temperature as well as temperature and humidity, did not cause any changes in the tearing behaviour of the modified or unmodified UHMWPE fibrous composites. The above observation underlined the absence of the impact of the accelerated aging agents on the most important property (tearing), taking into the account the ballistic behaviour of the material studied.

The mechanical behaviour of the modified UHMWPE fibrous composites during the accelerated aging was more stable compared to the initial materials

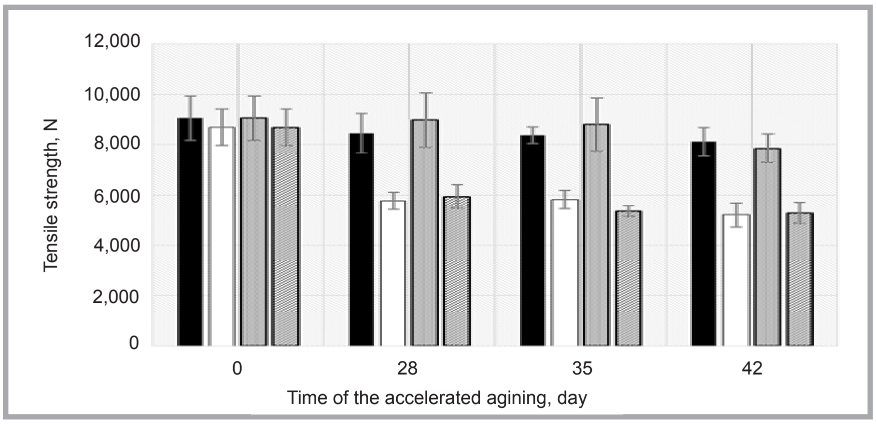

Figure 3. Effect of accelerated aging at temperature of 70 °C or temperature of 70 °C and relative humidity of 65% on the tensile strength in the longitudinal direction of the modified and unmodified UHMWPE fibrous composites; designation of the bars as in ***Figure 1****.*

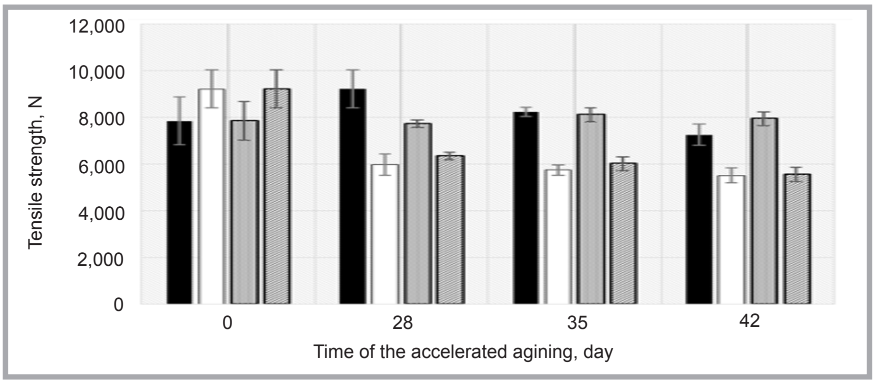

Figure 4. Effect of accelerated aging at temperature of 70 °C or temperature of 70 °C and relative humidity of 65% on the tensile strength in the vertical direction of the modified and unmodified UHMWPE fibrous composites; designation of the bars as in ***Figure 1****.*

(***Figures 3 – 7***). The tensile strength, both lengthwise and crosswise, showed significantly higher values after acceleration aging, compared to the unmodified samples (***Figures 3 – 4***).

If the temperature and humidity were used as the aging factors, the modified textile composite was characterised by the highest tensile strength. The tensile strength in the longitudinal direction of the modified samples slightly decreased with the aging periods, whereas the mechanical strength of the unmodified textile composite was drastically reduced after 28 days of accelerated aging, if the temperature or temperature and humidity were used as the aging factors. Prolongation of the accelerated aging (after 28 days) of the unmodified textiles resulted in a statistically insignificant reduction in the tensile strength. A similar effect was observed for the elongation at the maximal load (***Figures 5 – 6***).

Surface modification with the silicone-like polymer yielded no changes in the above parameters if the temperature and humidity were used as the aging factors. The application of temperature with low humidity as the aging factors to the modified samples resulted in a slight decrease in elongation at the maximal load.

The unmodified samples were characterised by a significant reduction in elongation at the maximal force after 28 days if temperature and humidity were used as the aging factors. The phenomena observed are directly connected to the modification performed, and they confirmed the protection effect of the silicone-like polymer layer against the accelerated aging agents.

The initial modified sample used as a starting material for the accelerated aging test was characterised by lower (by approx. 20%) bursting strength (***Figure 7***).

The accelerated aging (using the temperature and humidity) yielded a continuous, in aging periods, reduction of the bursting strength of the unmodified sample, whereas the application of temperature and humidity as the aging factor caused a drastic reduction after 28 days of the test. After that the parameter was statistically unchanged. Surface modification of UHMWPE fibrous composites by silicone-like polymer with low temperature plasma assisted deposition yielded higher resistance against the aging factors described by the burst strength parameters. However, the action of the temperature with low relative humidity caused an increase in bursting strength by approx. 15% after 28 days, and after prolongation of the aging, a significant reduction in the above parameter to alevel similar to that obtained with the non-aged sample.

The phenomenon of an increase in the bursting strength after the first period of aging is probably connected with structural changes (remodelling) in the modified textile surface as a result of the action of relatively high temperature.

The initial materials exposed to low plasma modification showed higher water tightness by approx. 8% compared to the unmodified sample (***Tables 1 – 2; Figure 8***).

The synergistic use of temperature and humidity caused an increase in the parameter discussed by approx. 10% after accelerated aging. Additionally the application of temperature only during the ageing process resulted in insignificant lowering of the water tightness.

The accelerated aging of the unmodified samples using both temperature or temperature and humidity caused a significant reduction in the parameter discussed by approx. 10% (aging factors: temperature and humidity) or approx. 19% (aging factor: temperature).

During the accelerated aging test, the modified and unmodified UHMWPE fibrous composites do not allow water to pass, which indicates the absence of changes in the macroscopic structure of the composite. Such an observation confirmed the absence of the impact of the processing parameters on the structure of the materials that are resistant to the accelerated aging agents, both temperature or temperature and humidity.

The process of aging of the unmodified sample in the accelerated conditions caused an increase in the capability to absorb water (***Figure 9***).

A higher increase in the water uptake after the accelerated aging was found if temperature was used as the aging factor. The application of temperature and humidity yielded an increase in water absorption up to 35 days of the test preformed. Applying the humidity as an additional aging factor showed the protection effect on the water absorbability level. Prolongation of the aging test affected the loss of the dynamic change in the parameter discussed. The modified sample showed more stable behaviour in terms of water absorbability during the accelerated aging. The level of water absorption was statistically stable during the aging test.

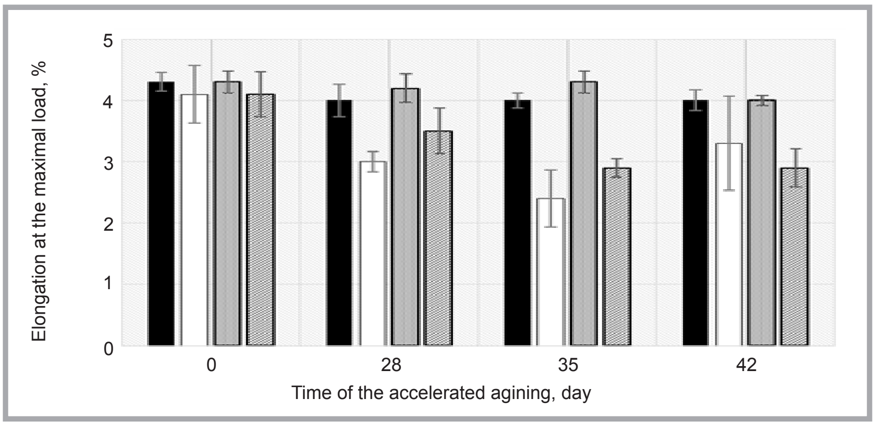

Figure 5. *Effect of accelerated aging at temperature of 70 °C or temperature of 70 °C and relative humidity of 65% on the elongation in the longitudinal direction of the modified and unmodified UHMWPE fibrous composites; designation of the bars as in* ***Figure 1****.*

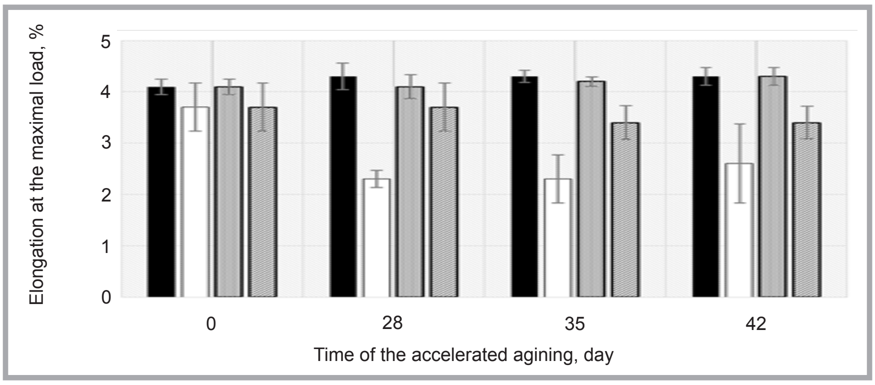

Figure 6. *Effect of accelerated aging at temperature of 70 °C or temperature of 70 °C and relative humidity of 65% on the elongation in the vertical direction of the modified and unmodified UHMWPE fibrous composites; designation of the bars as in* ***Figure 1****.*

Conclusions

Low temperature plasma assisted deposition (PACVD) of the silicone-like polymer onto the surface of the UHMWPE fibrous composite showed promising results if material is tested directly after the PACVD modification. However, the resultant effect of the PACVD modification should be stable during the usage and storage of the final products. Thus simulated accelerated aging is needed to determine the real effectiveness of the PACVD modification process of the UHMWPE fibrous composite.

The process of the accelerated aging performed with the use of two conditions (the temperature and low relative humidity or temperature and high relative humidity) confirmed:

- the protection effect of the silicone-like polymer layer put onto the surface of the UHMWPE fibrous composite against the aging factors;
- the significant impact of low temperature plasma assisted deposition of the silicone-like polymer on the crucial usage parameters of the ballistic fibrous composites, helpful for prolonging the life span of ballistic products;
- the results of the accelerated aging were different if the temperature with low humidity or temperature with high humidity were applied. The phenomenon indicated the significant impact of the increase in the humidity during the accelerated aging test.
- the mechanical behaviour of the aged materials also varied depending on the materials used – unmodified or modified by low temperature plasma;
- PACVD modification of the UHMWPE fibrous composite significantly improved the usage properties that support stability during the accelerated aging process, which confirms the effectiveness of the deposition of the silicone-like polymer onto the ballistic composite. The results of the accelerated ageing tests presented show the worst case, compared to aging in real conditions – the stability of the PACVD effect on the UHMWPE fibrous composite shows high probability, to be confirmed in real conditions of use.

Verification of the ballistic properties of the modified and unmodified fibrous systems based on the requirements of PN-V-87000 Standard (bullet proofness: K2 class and fragment proofness: O3 class) for the initial materials as well as after accelerated aging will be the next stage of the research. Additionally the effect of potential changes in the structural properties after the accelerated aging test will be also studied in detail (DSC, DMTA, ATR-FTIR, SEM-EDS).

Acknowledgements

The research was supported by the National Science Centre under research project No. N N508 629940 „THE STUDIES ON THE FUNCTIONALISATION OF BALLISTIC MATERIALS".

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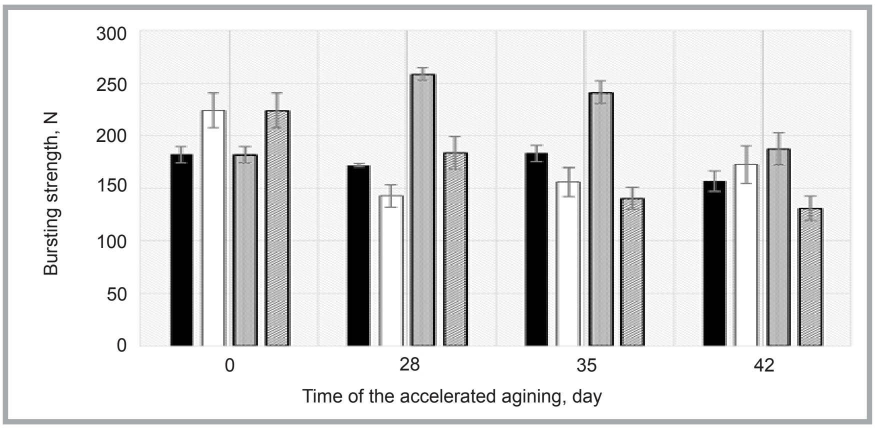

Figure 7. *Effect of accelerated aging at temperature of 70 °C or temperature of 70 °C and relative humidity of 65% on the bursting strength of the modified and unmodified UHMWPE fibrous composites; designation of the bars as in* ***Figure 1****.*

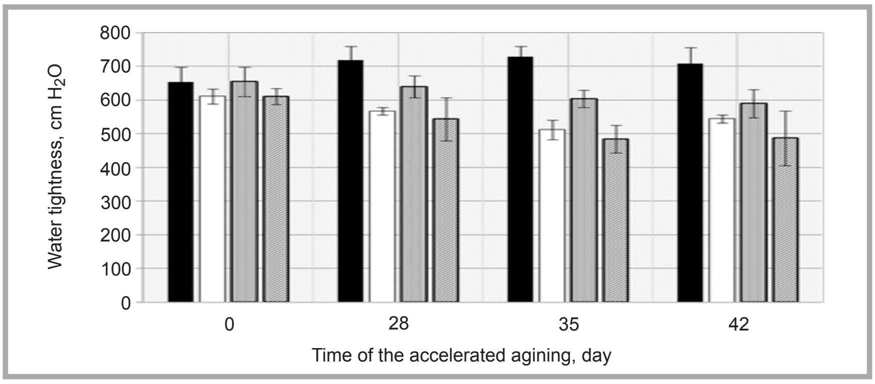

Figure 8. *Effect of accelerated aging at temperature of 70 °C or temperature of 70 °C and relative humidity of 65% on the water tightness of the modified and unmodified UHMWPE fibrous composites; designation of the bars as in* ***Figure 1****.*

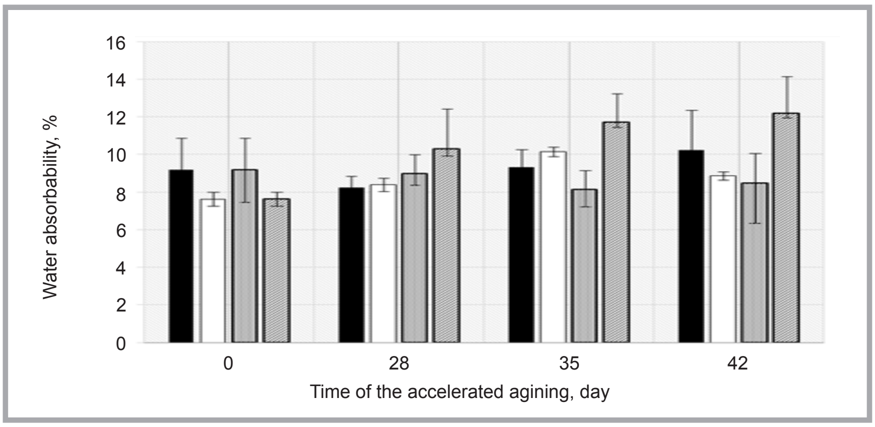

Figure 9. *Effect of accelerated aging at temperature of 70 °C or temperature of 70 °C and relative humidity of 65% on the water absorbability of the modified and unmodified UHMWPE fibrous composites; designation of the bars as in* ***Figure 1****.*

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Received 20.03.2014 Reviewed 09.06.2015