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Effect of Accelerated Ageing Conditions on the Degradation Process of Dyneema® Polyethylene Composites

Abstract
This research work concerned testing the effect of ageing processes (real-time and accelerated) on the usage properties and safety of ballistic inserts made of a polyethylene sheet called Dyneema® UD SB21 (for soft ballistic inserts). The effect of the ageing process was verified by the assessment of ballistic and mechanical properties. Changes in the structure of the materials tested were estimated with differential scanning calorimetry (DSC) and Fourier Transform Infrared spectroscopy (FT-IR).

Key words: *accelerated ageing, high molecular weight polyethylene, crystallinity.*

Introduction

Ageing is the process of structural changes occurring in a polymer as an effect of the long-term impact of external agents, leading to the degradation of the usage properties of the material. During ageing, irreversible changes occur in material, which are the result of the synergic action of external agents, often some chemical reactions, resulting in cross-linking, oxidation (photo- and thermo-oxidation), degradation, and finally the destruction and disintegration of the material [1 - 4].

Drawing conclusions regarding the resistance of materials to atmospheric conditions requires the execution of ageing tests under natural conditions, which is immensely important for materials, whose parameters are responsible for safety and performance. Because of the long duration of the process, changes that occur by nature are simulated by applying the method of accelerated ageing.

Accelerated ageing is a research method which serves the purpose of determining the usability period of a product (and *de facto* estimation of its performance and safety in the aspect of the time of its usefulness). The method is applied for products which retain their features with no remarkable changes over long periods of time that run into years [5, 6].

Tests of ballistic products in accelerated ageing conditions have been executed many times by manufacturers and users of personal protection goods [7 - 13]. In 1985 the Du Pont company tested 79 bulletproof vests made on the basis of para-aramide fabrics, to be worn under overt clothing and used from 2 to 10 years [7]. Sixteen of them showed a reduction in the ballistic protection limit, expressed as V_{50}[1)], of 8 ÷ 18%, and another - of 24%. Further ballistic tests of such vests [7], also executed by Du Pont, after seven years of intensive usage showed a reduction in V_{50} of 10% compared to the V_{50} of new bulletproof vests. However, sprinkling tests (wet) show an even bigger reduction in the ballistic protection boundary ie. of 20 - 25%. The National Institute of Justice (NIJ, USA) presented the results of research concerning the resistance to ageing of PBO poly(p-phenylene-benzo-bis-oxazole), known as Zylon®, which was applied as a ballistic material for manufacturing bullet- and fragment-proof vests. The mechanical properties of PBO exposed to accelerated ageing under a temperature of 50 °C and humidity of 60% over a period of 50 days had deteriorated by 20% compared to the initial material [8]. The company DSM, which is the manufacturer of Dyneema® UD HB26 and Dyneema® UD HB2, tested composite panels made on the basis of the materials mentioned for the alteration of their ballistic properties, defined by the parameter V_{50}. The panels were exposed to natural ageing under real conditions for a period of 4 years, as well as to accelerated ageing for 20 weeks under a temperature of 65 °C to 90 °C. Ballistic tests of the products showed a insignificant reduction in the V_{50} value compared to the initial samples [9]. Chabba and co-workers [10] tested Dyneema® SK76, Dyneema® UD SB21, SB31 & SB61 under conditions of accelerated ageing. The analysis of the material properties tested proved that the changes occurring inside them during ageing for 8-weeks at a temperature of 65 °C and relative humidity of 80% match those occurring to the same products under conditions of natural ageing over 5 years. Analysis of the bibliography shows that the rarity of changes occurring within PE plastic was estimated on the basis of tests of the mechanical and ballistic properties of these materials [10]. Regarding the data above, it can be concluded that the risk assessment connected with the loss of the performance and safety parameters of protective products during use becomes an important problem mainly with respect to user safety.

Therefore doing research into ballistic products under conditions of accelerated ageing seems necessary, as well as knowledge expansion of this topic. Developing a comprehensive method for accelerated ageing testing as well as the results obtained from tests allow to define guidelines to change the currently valid standards, which are also helpful to develop new guidelines and standards to be applied for the estimation of the protective properties of ballistic products taking into account the testing methods for ageing processes.

Materials

Tests of accelerated ageing were performed on ballistic protective inserts made of a Dyneema® UD SB21 sheet of ultra high molecular weight polyethylene fibres (UHMWPE). Dyneema® UD SB21 was obtained from DSM High Performance Fibers BV (the Netherlands). The specification of Dyneema® UD SB21 sheets is shown in ***Table 1***.

Another object of testing were 120 images of bullet-proof vests manufactured by the Institute of Security Technology 'MORATEX", used for 5 years in real conditions. The ballistic inserts of the bullet-proof vests were also made of Dyneema® UD SB21 sheet.

Table 1. *Properties of the non-woven PE product Dyneema® UD SB21; * Each sample had a working width of 50 ± 0.5 mm and length of 200 ± 1 mm.*

Parameter	Unit	Value	Test Method
Width	cm	130 ± 0.2	PN-EN ISO 2286-1:2000 [14]
Area density	g/m^2	145 ± 5	PN-EN ISO 2286-2:1999 [15]
Thickness	mm	0.19 ± 0.02	PN-EN ISO 2286-3:2000 [16]
Breaking force*: - lengthwise - crosswise	N	 6 000 ± 500 5 500 ± 500	PN-EN ISO 1421:2001 [17]
Elongation at break*: - lengthwise - crosswise	%	 14 ± 0.5 13 ± 0.5	PN-EN ISO 1421:2001 [17]

Methods

Accelerated ageing test

The testing methods for the resistance of Dyneema® UD SB21 to ageing were reduced to two basic matters: defining a group of properties to be tested, which will be the criteria of resistance assessment and will determine the measurement conditions as well as the set of agents and their intensity effect on the samples being tested. Ageing tests of samples were executed with the application of test methodology based on Standards PN-EN 12280-1:2002 [18] and PN-EN 12280-3:2002 [19].

Accelerated ageing tests were executed inside the following :

1. A thermal chamber - TK 720 from BINDER GmbH, which allow to simulate the pr °Cess of ageing with a heat effect at a temperature of 70 ± 0.5 °C and humidity of 0 + 1.5%.
2. A climatic chamber - KBF 240 from BINDER GmbH, which allows to simulate the pr °Cess of ageing with a heat effect at a temperature of 70 ± 0.4 °C and humidity of 50 ± 1.5%.

Analytical methods

Assessment of mechanical properties

Measuring the breaking force of samples subjected to ageing and those which were not was made according to Standard PN-EN ISO 1421:2001 [17] with a strength tester from Zwick that allowed to determine the tear force up to 50 kN.

The breaking force, based on risk assessment, is the most representative property of ballistic materials, others being the bursting strength, module of elasticity, tearing resistance, etc. to obtain a quick interpretation of changes in the mechanical properties of the samples studied.

Assessment of ballistic properties

Tests were made according to the requirements and methods included in Standard PN-V-87000:1999 [20].

Lead core pistol bullets of 7.62 × 25 mm TT with an impact velocity of 420^{+15} m/s were used for the bullet-proofness tests.

During the tests, the following parameters were determined:

a) the impact velocity of the bullet V_{up} in m/s,
b) the depth of test base deformation U_d in mm,
c) the number of punctured layers.

FT-IR tests

The alteration of the chemical structure was analysed with the Fourier Transform Infrared spectroscopy (ATR – FTIR) method within the range of 500 ÷ 3500 cm^{-1}. IR spectra were made with a Genesis Series FT-IR^{TM} (Unicam). Pill-shaped Dyneema® UD SB21 material was tested in the form of KBr tabs. The pills were made by adding 1 part of very finely comminuted Dyneema® UD SB21 to 99 parts of KBr. The mixture of KBr with the samples was rubbed in a mortar in order to gain a precise distribution of the composite tested, after which all of it was subject to high pressure (over 50 MPa) for several minutes.

Assessment of thermal properties with the method of Differential Scanning Calorimetry (DSC)

Thermal analysis was made in a neutral gas (Nitrogen) atmosphere using a differential scanning calorimeter (Diamond/ Perkin – Elmer). A 7 ÷ 14 mg sample was placed in the kiln of the thermal analyser and heated at a rate of 20 °C/min up to 180 °C. The sample was kept for 5 minutes in such a temperature and later cooled down to a temperature of -20 °C at a rate of 20 °C/min. Afterwards it was heated again up to 180 °C at a rate of 20 °C/min. The temperatures of phase and chemical changes in the test material were determined on the basis of DSC curves.

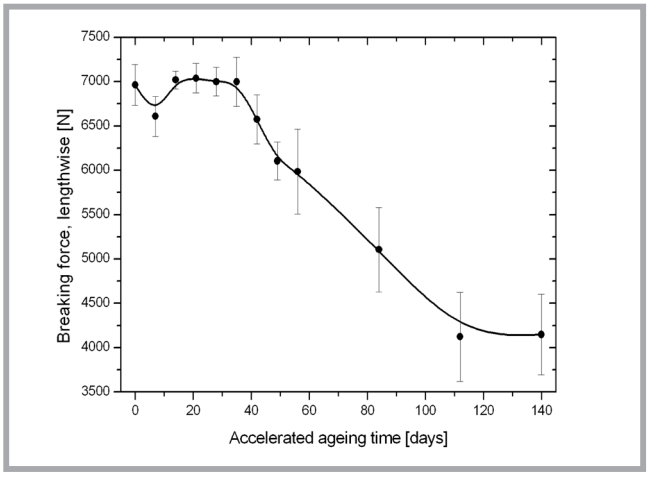

Figure 1. *Relation between the breaking force (lengthwise) and time of the accelerated ageing of Dyneema® UD SB21 at a temperature of 70 °C and humidity of 0%.*

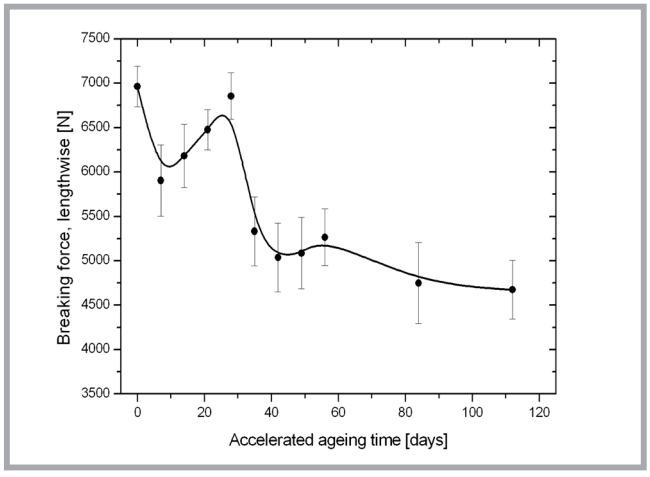

Figure 2. *Relation between the breaking force (lengthwise) and time of the accelerated ageing of Dyneema® UD SB21 at a temperature of 70 °C and humidity of 50%.*

Table 2. *Specific absorption bands within the FT-IR spectrum of Dyneema® UD SB21 after 21 days of accelerated ageing at a temperature of 70 °C and humidity of 0%.*

Wave number [cm^{-1}]		Group or bond	Vibration
Found	Literature data [26-27]		
2928	2926	CH_2	ν_{asym} C-H
2850	2853	CH_2	ν_{sym} C-H
1700	1698 - 1700	-C=O	-C=O
1648	1675 ÷1665	RR'C=CHR	ν C=C
1473	1470 ÷ 1420	CH_2	δ scissoring C-H
1384	1380	CH_3	δ_{sym} C-H
1326	1300	CH_2	δ wagging and twisting CH_2
1099 ÷ 1047	1020 ÷ 1000	CH_2	ν CH_2 framework
910	950 ÷ 800	C=CH	δ C=C
719	719	$(-CH_2-)_{n>4}$	CH_2 r °Cking
730	730	CH_2	CH_2 r °Cking amorphous phase

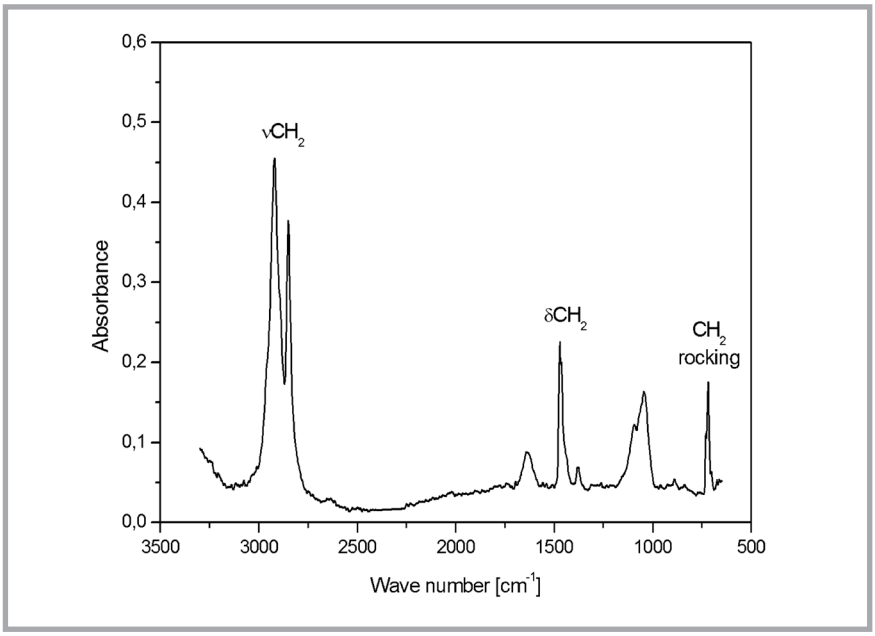

Figure 3. *FT-IR spectrum of Dyneema® UD SB21 after 35 days of accelerated ageing at a temperature of 70 °C and humidity of 0%.*

Using the DSC method, the crystallinity rate, x_C, was calculated for the samples tested according to formula [21]:

$$x_c = \frac{\Delta h_m}{\Delta h_m^0} * 100\%$$

where:

x_c – crystallinity rate

Δh_m – fusion heat of the polymer sample

Δh^0_m – fusion heat of a completely crystalline polymer – for polyethylene $\Delta h^0_m = 293$ J/g

Results and discussion

Mechanical properties

During the assessment of the mechanical properties of the Dyneema® UD SB21 sheet subjected to accelerated ageing for certain time periods of exposure, it was was observed that after the initial reduction in the breaking force, its increase comes about in the course of further ageing (***Figures 1 - 2***). Such a diversity of mechanical properties is linked to the occurrence of the parallel processes of the oxidation and cross-linking of the material [22, 23]. Other authors of publications [24, 25] attribute this effect to the large number of defects that appear on the surface of the sample, which loses its ability to transfer stress deep into the material. The remarkable deterioration of the mechanical properties of Dyneema® UD SB21 happens after 42 days of sample conditioning at a temperature of 70 °C and humidity of 50% (accelerated ageing), as shown in ***Figures 1 – 2***. The intervals between consecutive tests of the mechanical properties of the samples throughout the course of ageing were short enough to allow to precisely detect the moment when the material reaches the minimum breaking force, thus allowing to determine the real time of plastic usability. The method for assessing the mechanical properties of Dyneema® UD SB21 applied resulted in a curve of plastic durability, which allowed to determine the critical time of ageing when a certain change arises in the value of a test parameter that affects the safety provided by the protective product.

Ballistic properties

On the basis of the results of the research, conducted according to Standard PN-V-87000:1999, it was proved that the 3rd class of bullet-proof vests ie. protecting against 7.62 × 25 mm TT lead core, 5.5 g pistol bullets at an impact velocity of 420^{+15} m/s, as well as their inserts made of Dyneema® UD SB21, subjected to ageing in a laboratory reatain the protective properties declared.

Additionally, in the course of the ballistic test cycle at various bullet impact velocities, the number of punctured layers was determined in the bullet-proof vests as well as in the inserts made of Dyneema® UD SB21 subjected to laboratory ageing. The research results show that the maximum increase in the punctured layers of a packet after 5-years of usage is about 13% compared to unused vests, which corresponds with the 6 layers of ballistic inserts of Dyneema® UD SB21 applied. Analysis of the results obtained show that there is an increase in shot-punctured layers depending on the time of ageing in laboratory conditions.

Structure studies

FT-IR

The FT-IR spectra of every sample of Dyneema® UD SB21 sheet, both for the initial sample and that subjected to accelerated ageing or in real time, the absorption bands found were within similar ranges of wave numbers. The absorption bands corresponding to the vibrations of $–CH_2–$ groups ($\lambda = 2920$ cm^{-1}, $\lambda = 2858$ cm^{-1}, $\lambda = 1473$ cm^{-1}, $\lambda = 1380$ cm^{-1}, $\lambda = 719$ cm^{-1} and $\lambda = 730$ cm^{-1}) were present within the FT-IR spectra (***Table 2***) [26-27]. Moreover, in the FT-IR spectra of Dyneema® UD SB 21 there appeared absorption bands corresponding to the vibrations of atoms

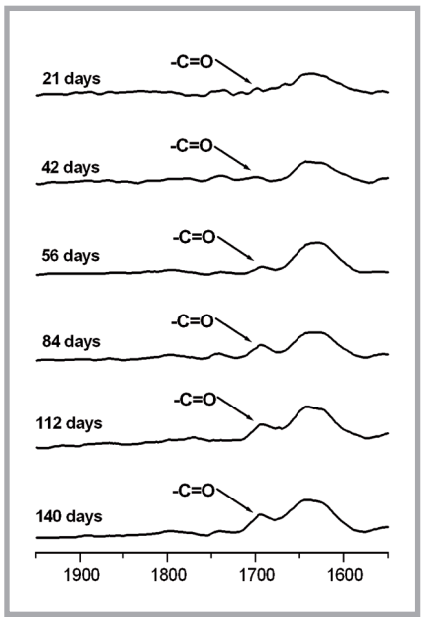

Figure 4. *Absorption change in carbonyl groups of the material depending on the various time periods of the accelerated ageing of Dyneema® UD SB 21 at a temperature of 70 °C and humidity of 0%.*

of –C=C– groups within a wave number range of 1640 ÷ 1660 cm^{-1}. The presence of these bindings may be explained by the composition of Dyneema® UD SB21, which is as follows [28]:

- 73% fibres (High Molecular Weight Polyethylene, HMWPE),
- 10% foil-matrix (Low Density Polyethylene),
- 17% binder (a chloroform-soluble residue - polyisoprene).

The composition of Dyneema® UD SB21 is the topic of our institute's research [28]. An example of the FT-IR spectrum of Dyneema® UD SB21 after 35 days of accelerated ageing at a temperature of 70 °C is shown in ***Figure 3***.

As is known from the mechanism of the degradation process initiated thermally and running with the participation of oxygen from air, a remarkable component of changes in the structure of polymeric chains is oxidation causing the creation of carboxylic groups of acidic, ketonic and ester types [29].

Therefore the research was focused on the analysis of bands specific to carbonyl groups when estimating alterations in the chemical structure of Dyneema® UD SB21 samples subjected to accelerated ageing in a laboratory as well as those after 5 years of real usage. The analysis resulted in finding an increase in the absorbance of bands within a wave number range of 1698 ÷ 1700 cm^{-1} along with an increase in the period of accelerated ageing (***Figure 4***). A significant increase in the absorbance of carbonyl groups was observed after 42 days of accelerated ageing at a temperature of 70 °C. This result shows structural alterations in macromolecules, which brings about the cracking of chains, leading to a reduction in molecular weight, thus causing a deterioration of mechanical properties.

DSC

In order to determine the thermal properties of Dyneema® UD SB21 subjected to ageing, some DSC thermal images were made (meltdown and crystallisation). The results of DSC analysis are given in ***Table 3***. The DSC curves of selected samples of Dyneema® UD SB21 (D-SB21-49D) material are shown in ***Figure 5***.

The curve form of the DSC charts and the detailed analysis of the heat effects of samples subjected to testing prove that the content of the crystalline phase of the melting point above 160 °C is only specific to Dyneema® UD SB21 subjected to either accelerated or natural ageing. Under accelerated ageing at a temperature of 70 °C and humidity of 50%, Dyneema® UD SB 21 undergoes its crystalline phase, which consists of various types of crystalline structures. During the first heating, the endothermic peaks of the DSC curve of test samples subjected to ageing are assigned the meltdown of the following crystalline structures [21]:

- rhombic crystals – melting point 149 °C,
- pseudohexagonal meso-phase - melting point 155 °C,
- monocrystals - melting point 160 °C,

During the second heating, an endothermic peak was observed, which came from the meltdown of undulating lamellas at 135 °C, while the peaks of crystalline forms appearing in the first stage of

Table 3. *Results of the DSC analysis of Dyneema® UD SB21 material in a neutral gas atmosphere; 1 - melting point of „undulating lamellas", 2 - melting point of rhombic crystals, 3 - melting point of the pseudohexagonal meso-phase, 4 - melting point of monocrystals, 5 - melting point of low molecular weight PE, 6 - temperature of crystalisation of very high molecular weight PE, 7 - temperature of crystalisation of low molecular weight PE. Tm - melting temperature, Δhm - fusion heat of polymer sample, x_C - crystallinity rate, Tk - temperature of crystalisation.*

Symbol of sample	Ageing time, days	The 1st heating						The 2nd heating				cooling	
		T_m, °C				Δh_m, J/g	x_C, %	T_m, °C		Δh_m, J/g	x_C, %	T_k, °C	
		2	3	4	5			1	5			6	7
D-SB21-0	-	-	154.59	160.63	123.82	228.25	77.9	138.02	123.94	136.40	46.5	114.66	-
D-SB21-14D	14	150.11	156.49	-	123.83	212.72	72.6	137.20	123.98	138.15	47.1	116.15	107.74
D-SB21-21D	21	150.08	158.45	-	123.82	216.45	73.9	138.20	124.31	139.15	47.5	115.17	107.54
D-SB21-28D	28	149.09	157.79	-	123.98	216.84	74.0	137.52	124.13	137.70	47.0	115.99	107.24
D-SB21-28DW	28	149.80	159.17	-	123.50	217.63	74.3	139.40	124.35	135.42	46.2	114.40	105.65
D-SB21-35D	35	149.75	158.97	-	123.99	222.30	75.9	138.86	125.14	141.33	48.2	115.00	105.92
D-SB21-42D	42	152.57	159.29	-	125.32	208.86	71.3	139.35	125.63	133.16	45.4	114.49	105.57
D-SB21-49D	49	151.75	156.27	-	124.98	207.70	70.9	137.68	123.79	127.72	43.6	115.63	106.88
D-SB21-56D	56	149.82	156.16	-	123.85	221.46	75.5	137.75	123.85	137.29	46.8	115.55	106.80
D-SB21-56DW	56	149.49	158.02	-	123.18	210.73	71.9	138.25	124.51	133.13	45.4	115.37	106.63
D-SB21-84D	84	150.29	159.66	-	123.84	209.47	71.5	139.56	125.24	132.52	45.2	114.07	105.48
D-SB21-84DW	84	151.40	157.75	-	123.59	220.88	75.3	137.13	123.57	135.61	46.2	115.91	107.16
D-SB21-112D	112	151.81	158.48	-	124.69	212.38	72.4	139.24	125.00	134.58	45.9	114.23	105.65
D-SB21-112DW	112	150.04	157.23	-	123.92	220.63	75.3	138.14	124.07	137.03	46.8	115.40	106.83
D-SB21-140D	140	149.65	156.82	-	124.02	217.18	74.1	137.58	124.18	136.6	46.6	115.88	106.70
D-SB21-140DW	140	150.36	155.73	-	124.09	220.05	75.1	138.30	124.08	136.33	46.5	115.09	106.67
D-SB21-5L	5 lat	151.24	158.78	-	123.64	205.90	70.3	138.85	123.78	132.17	45.1	114.15	106.07

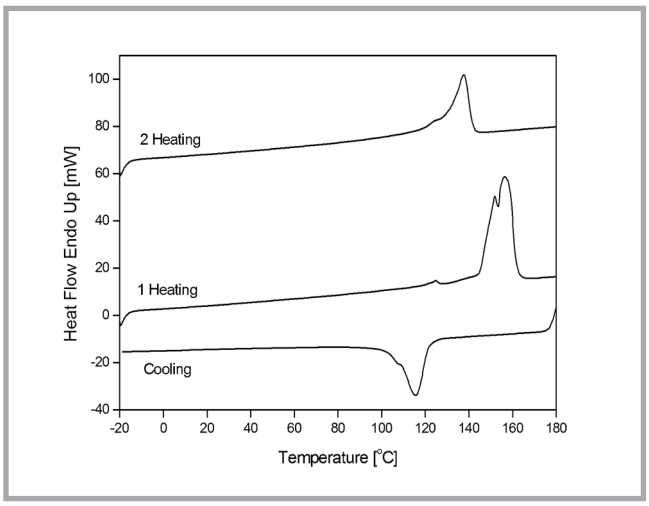

Figure 5. *DSC curves of Dyneema UD SB 21(D-SB21-49D) after 49 days of accelerated ageing at a temperature of 70 °C and humidity of 0%.*

heating were not observed. Presumably, during the second heating of the test samples at a constant rate, unification of the crystalline structure of Dyneema® UD SB21 occurred. Moreover, a peak from the crystalline structure with a melting point of 123 - 125 °C appears on the DCS curves of both the first and second heating. The appearance of this peak on the DSC curve is linked to the presence of the crystalline phase of low molecular weight polyethylene included in compounds of Dyneema® UD SB21.

The experimental data show that the ageing processes visibly affect the change in the crystallinity rate (***Figures 6 - 7***). The crystallinity rate of a Dyneema® UD SB21 sheet exploited under natural conditions for 5 years and that of one subjected to accelerated ageing at a temperature of 70 °C and humidity of 50% amount to 70% ÷ 76%. The crystallinity rate of the material calculated after the second heating amounts to 45% ÷ 48%. It seems possible that the difference between the crystallinity rate of Dyneema® UD SB21 obtained during the first and second heating is determined by the effects of time and by the temperature of the material tested. As early as after 14 days of the exposure of Dyneema® UD SB21 samples subjected to accelerated ageing (T = 70 °C), a remarkable increase in the crystallinity rate occurs both during the first heating and the second. This increase in the crystallinity rate is also visible for the samples subjected to accelerated ageing at a temperature of 70 °C for 21, 28, 35, 56, 84, 112 and 140 days. According to Yanai [30], the reason for the above phenomenon is possible degradation taking place in amorphous areas, causing chains to break, which may indirectly contribute to the improvement in orderliness, i.e. an increase in the sample's crystallinity rate. Analysing the results of the tests of a Dyneema UD SB21 sheet, we can observe a decrease in the crystallinity rate after 42 and 49 days of accelerated ageing at a temperature of 70 °C, which is caused by the processes of branching and cross-linking occurring in the material tested, which are in opposition to the processes of chains cracking [30].

Conclusions

1. On the basis of the results of research conducted according to Standard PN-V-87000:1999, it was proved that the 3rd class of bullet-proof vests after 5-years of usage as well as inserts made of Dyneema® UD SB21 subjected to accelerated ageing in a laboratory retain the protective properties declared;
2. Regardless of the factors which cause ageing, time has a significant impact on the course of ageing, both for real conditions and accelerated ageing.
3. The effect of temperature and humidity causes a remarkable reduction in the mechanical and ballistic properties of Dyneema® UD SB21 material. The effect increased during prolonged exposure.
4. The progressive ageing of the time of exposure causes alterations in the chemical structure of macromolecules, confirmed by the presence of absorption bands arising from –C=O groups within the FT-IR spectra of the material tested, as well as by the changing values of the crystallinity rate (determined by DSC methods) occurring along with the time of ageing.

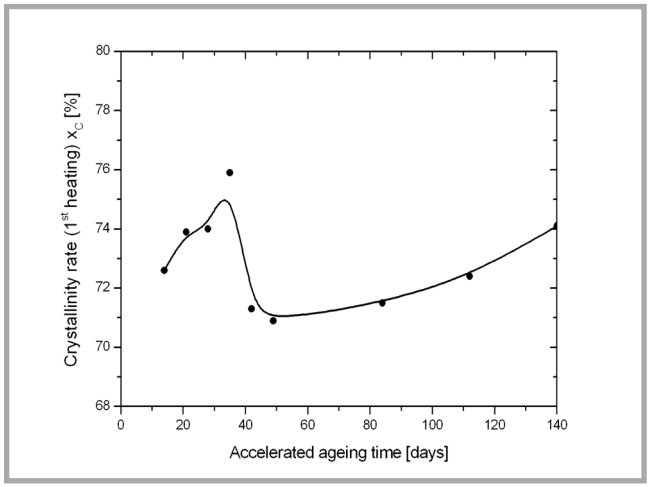

Figure 6. *Crystallinity rate (1st heating) dependence on the time of the ageing of Dyneema® UD SB 21 at a temperature of 70 °C and humidity of 0% (measurement of the crystallinity rate was made for one sample at a given time of exposure).*

Figure 7. *Crystallinity rate (2nd heating) dependence on the time of the ageing of Dyneema® UD SB 21 at a temperature of 70 °C and humidity of 0% (measurement of the crystallinity rate was made for one sample at a given time of exposure).*

5. The correlation between the critical parameters of the Dyneema® UD SB21 inserts was determined in real conditions of use and for materials subjected to accelerated aged. Analysis of the mechanical, ballistic and structural properties of the material tested shows that the changes occurring in it throughout the course of accelerated ageing between the 35th and 45th day at a temperature of 70 °C and humidity of 50% correspond to changes found in the material after having been in natural ageing conditions for 5 years. The research demonstrated that a reduction in the mechanical and ballistic properties of Dyneema® UD SB21 inserts occurs after about 42 days of accelerated ageing.
6. In accordance with the aim of the experiment, a time frame was determined after which the alteration of the properties of ballistic material °Ccurs. The properties of the high-strength material Dyneema® UD SB 21 tested allow to mark out a curve of plastic durability.

Editorial note

1) Ballistic Limit: For a given bullet type, the velocity at which the bullet is expected to perforate the armour 50% of the time. The ballistic limit is typically denoted as the V_{50} or V_{50} value - according to the Ballistic Resistance of Body Armor NIJ Standard-0101.06.

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