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**Research paper**

# Influence of cyclic frozen and defrost on mechanical properties of polytetrafluoroethylene (PTFE)-coated woven fabrics

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**Abstract:** This research aims to determine the influence of the cyclic process of freezing and defrosting on the mechanical properties of the chosen glass fibres and PTFE-coated woven fabrics. The specimens were subjected to freezing at about −20°C for 4 h and thawing by full immersion into the water at about +20°C for 4 h. The fabric samples after 25 and 50 frozen cycles were air-dried at room temperature for one week and then subjected to uniaxial tensile tests. The same tests have been performed on a reference group of specimens, which were not exposed to temperature change. The authors determined the tensile strength, and longitudinal stiffnesses resulting from performed tests. Although the investigated coated woven fabrics expressed a reduction in the tensile strength in water soaking conditions, the performed frozen cycles don't show a significant decrease in strength under uniaxial tensile tests.

**Keywords:** construction materials, freezer cycles, mechanical properties, PTFE coated woven fabric, uniaxial tensile test

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# 1. Introduction

The technical woven fabrics, called architectural fabrics, are used for roof construction of the membrane, hanging and pneumatic structures [1–4]. The different types of coated woven fabrics are available on the market for application in tensile structures, e.g.: PVC (polyvinyl chloride)-coated woven fabrics with polyester threads or PTFE (polytetrafluoroethylene)-coated woven fabrics with glass threads. The differences between the types of fabrics result from the use of different materials for the base fabric and coatings or differences in the manufacturing process (e.g. Precontraint technology [5, 6]). For the investigated PTFE-coated fabric the base material of the fabric is glass fibre threads. The PTFE-coated woven fabrics were applied for example for the membrane roof of the Forest Opera in Sopot (Poland) [7], Fig. 1.

Fig. 1. The Forest Opera membrane hanging roof in Sopot, Poland – co-designed by the authors

The literature concerning the subject of coated woven fabrics is very extensive. Every year new investigations are performed on comprehensive studies on the coated woven fabrics [8–25]. Performed investigations have shown that the mechanical parameters of the coated woven fabrics are highly influenced by temperature changes [26, 27]. Additionally, research investigations indicated that coated woven fabrics occur a reduction in the tensile strength resulting under water-logged [11, 28, 29]. A more extended survey of literature concerning the investigation carried out on coated woven fabrics is performed by Authors in [11].

In the case of permanent membrane or hanging structures used in changing climatic conditions (especially in winter), the sub-zero temperatures may occur cyclically. Based on these observations, the mechanical properties of the architectural fabrics seem necessary to be verified under the process of freezing and thawing cycles.

Due to these characteristics of coated woven fabrics, the present study is aimed at the determination of the influence of freezer and defrost in cycles on the mechanical properties of chosen PTFE-coated woven fabrics identified from the uniaxial tensile tests.

## 2. Materials and methods

For laboratory tests the authors chose two coated woven fabrics having glass threads coated by PTFE. The properties of the PTFE coated woven fabrics are the same as those investigated by Ambroziak and Kłosowski [11]. The S-type and B-type fabrics' main properties, declared by the producers, are very similar, Table 1. The main difference is related to the manufacturing process, where S-type is produced with the technology, where the threads of the warp and weft are initially strained during coating and B-type fabric with the technology where during coating the fabric is prestressed in warp direction only.

Table 1. Properties of investigated PTFE coated woven fabrics

| Properties | S Type | B Type |
|---|---|---|
| Total Mass per Unit Area (g/m$^2$) | 1540 | 1550 |
| Tensile Strength (kN/m)<br>Warp<br>Weft | <br>170<br>156 | <br>160<br>140 |
| Base Coat | PTFE | PTFE |
| Weight per Unit Area of Base Fabric (g/m$^2$) | 625 | 670 |
| Yarn Count (yarn/cm)<br>Warp<br>Weft | <br>7.1<br>7.5 | <br>8<br>7.5 |
| Translucency at 550 nm (%) | 9 | 8–11 |

Before the uniaxial tensile tests were carried out the coated woven fabric specimens were subjected to twenty-five and fifty freezer cycles in a freezing chamber with the temperature, and time-controlled refrigerating and heating system. Due to a lack of standard guidelines for the freezer of coated woven fabrics the freezer cycle was performed according to the PN-B-06250 standard [30]. This standard is used generally for the determination of concrete frost resistance [31–33]. The single freezer cycle consisted of freezing at $-18 \pm 2$°C for 4 h and thawing by full immersion in water at +18 ± 2°C for 4 h. After the specified number of freezer cycles, the fabric specimens were air-dried at in-room temperature for one week and then subjected to mechanical tests.

The uniaxial tensile tests were conducted on the Zwick 020 mechanical testing machine. The video extensometer control (based on the digital image correlation method) with the base of the optical extensometer of about 50 mm and flat grips were used. The specimens had 50 ± 1 mm width, and the active length (distance between flat grips) was equal to 200 ± 1 mm, Fig. 2. The total length of fabric specimens was 300 ± 1 mm. The mechanical tests were performed according to the ISO 1421:2016 standard [34] for the strip method, with the displacement rate of the grip equal to 100 mm/min. Each type of test has been repeated at least six times. Three main groups of coated fabric specimens were tested:

the base specimens of the PTFE-coated fabrics, after 25 frozen cycles and after 50 frozen cycles in the direction of the warp and weft threads.

Fig. 2. Uniaxial laboratory tests stand

## 3. Laboratory test results

### 3.1. Uniaxial tensile tests general response

The results of the uniaxial tensile tests are presented in the form of the stress-strain curves in Figs. 3, 4 for the S type and in Figs. 5, 6 for the B type coated fabric, respectively. The denotations S, S_F25 or B_F25 and S_F50 or B_F50 indicated results obtained for the base PTFE-coated fabric material, after 25 freezer cycles and after 50 freezer cycles, respectively.

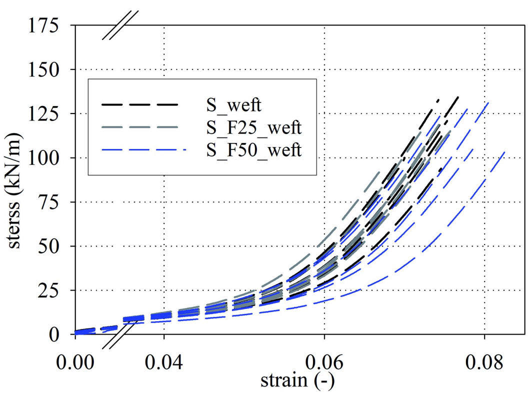


Fig. 3. Uniaxial tensile test results – weft S type fabric

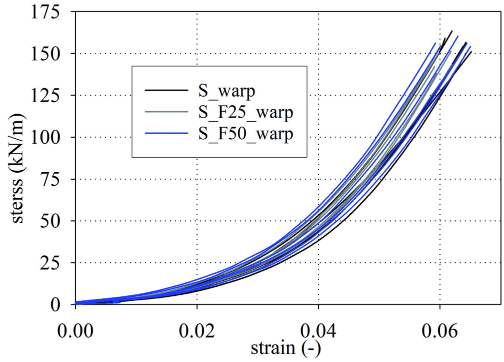


Fig. 4. Uniaxial tensile test results – warp S type fabric

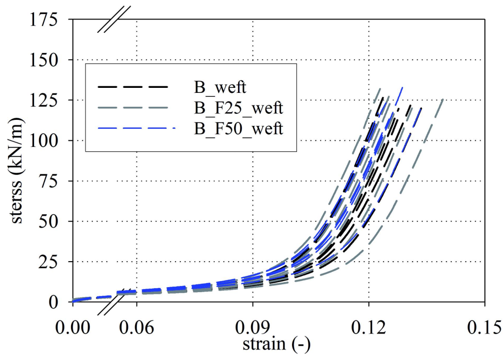


Fig. 5. Uniaxial tensile test results – weft B type fabric

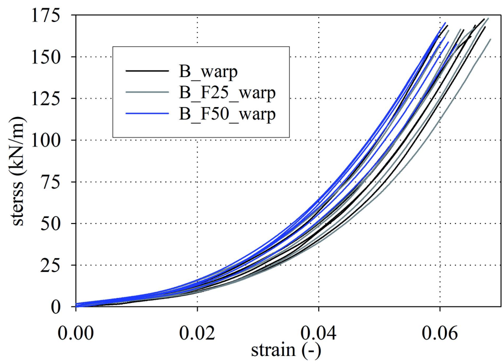


Fig. 6. Uniaxial tensile test results – warp B type fabric

The shape of the stress-strain curves is typical for the investigated type of architectural fabrics with glass threads and PTFE coating [6]. The B type fabric exhibits twice the time higher deformations in the weft direction under the uniaxial loads due to differences related to the manufacturing process. In the S type fabric, where the threads of warp and weft are initially strained, the difference between deformation in warp and weft direction is much smaller.

### 3.2. Elongation at break and tensile strength

Based on the performed uniaxial tensile tests two main mechanical properties (the elongation at break and tensile strength) for each tested specimen are determined and collected in Tables 2, 3 for B type fabric and in Tables 4, 5 for the S type fabric. The mean values of the tensile strength obtained during tests are generally lower than specified

Table 2. Elongation at break and tensile strength – base specimens B type fabric

| Specimen No. | Weft | | Warp | |
|---|---|---|---|---|
| | Rupture Strain [–] | Tensile Strength [kN/m] | Rupture Strain [–] | Tensile Strength [kN/m] |
| 1 | 0.1312 | 123.8 | 0.0679 | 173.3 |
| 2 | 0.1226 | 117.4 | 0.0643 | 170.7 |
| 3 | 0.1277 | 114.3 | 0.0654 | 170.2 |
| 4 | 0.1238 | 127.6 | 0.0622 | 171.3 |
| 5 | 0.1277 | 119.7 | 0.0658 | 163.4 |
| 6 | 0.1343 | 124.1 | 0.0680 | 172.7 |
| mean | 0.1279 ± 0.0013 | 121.1 ± 2.0 | 0.0656 ± 0.0009 | 170.3 ± 1.5 |
| median | 0.1277 | 121.7 | 0.0656 | 171.0 |

Table 3. Elongation at break and tensile strength – freezer cycles, B type fabric

| Specimen No. | Weft – F25 | | Weft – F50 | | Warp – F25 | | Warp – F50 | |
|---|---|---|---|---|---|---|---|---|
| | Rupture Strain [–] | Tensile Strength [kN/m] | Rupture Strain [–] | Tensile Strength [kN/m] | Rupture Strain [–] | Tensile Strength [kN/m] | Rupture Strain [–] | Tensile Strength [kN/m] |
| 1 | 0.1261 | 127.1 | 0.1338 | 122.3 | 0.0641 | 170.5 | 0.0600 | 162.6 |
| 2 | 0.1399 | 128.8 | 0.1261 | 118.2 | 0.0656 | 167.1 | 0.0613 | 172.3 |
| 3 | 0.1233 | 132.1 | 0.1288 | 133.1 | 0.0695 | 175.5 | 0.0621 | 166.7 |
| 4 | 0.1272 | 120.7 | 0.1247 | 110.4 | 0.0615 | 166.4 | 0.0642 | 163.7 |
| 5 | 0.1323 | 124.1 | 0.1228 | 119.5 | 0.0615 | 165.2 | 0.0606 | 167.3 |
| 6 | 0.1257 | 130.0 | 0.1244 | 123.2 | 0.0686 | 162.0 | 0.0618 | 161.3 |
| mean | 0.1291 ± 0.0025 | 127.1 ± 1.7 | 0.1268 ± 0.0016 | 121.1 ± 3.0 | 0.0651 ± 0.0014 | 167.8 ± 1.9 | 0.0616 ± 0.0006 | 165.6 ± 1.6 |
| median | 0.1266 | 127.9 | 0.1254 | 120.9 | 0.0649 | 166.7 | 0.0616 | 165.2 |

by manufacturers. Only the result of tensile strength for the B type fabric in the direction of the warp is about 7% greater than given in Table 1. This difference may be due to the application of the flat grips or the influence of a long storage period of fabric specimens before tests. It should be noted that in the case of the flat grips, damage to specimens close to the grips may be observed. The curved grips cause a more homogeneous stress distribution near the fixing; therefore, higher stress values may be reached under uniaxial tensile tests. On the other hand, the usage of the extensometer restricts the experiment area to the middle part of the fabric specimen only. This part is far from fixing; thus, for the identification of material parameters, the influence of the grips can be neglected [24].

Table 4. Elongation at break and tensile strength – base specimens S type fabric

| Specimen No. | Weft | | Warp | |
|---|---|---|---|---|
| | Rupture Strain [–] | Tensile Strength [kN/m] | Rupture Strain [–] | Tensile Strength [kN/m] |
| 1 | 0.0750 | 96.8 | 0.0664 | 156.6 |
| 2 | 0.0755 | 121.9 | 0.0646 | 159.1 |
| 3 | 0.0701 | 101.6 | 0.0650 | 160.9 |
| 4 | 0.0751 | 115.6 | 0.0609 | 159.8 |
| 5 | 0.0743 | 133.3 | 0.0623 | 165.4 |
| 6 | 0.0772 | 137.8 | 0.0648 | 153.1 |
| mean | 0.0746 ± 0.0010 | 117.8 ± 6.7 | 0.0640 ± 0.0008 | 159.1 ± 1.7 |
| median | 0.0751 | 118.8 | 0.0647 | 159.4 |

Table 5. Elongation at break and tensile strength – freezer cycles, S type fabric

| Specimen No. | Weft – F25 | | Weft – F50 | | Warp – F25 | | Warp – F50 | |
|---|---|---|---|---|---|---|---|---|
| | Rupture Strain [–] | Tensile Strength [kN/m] | Rupture Strain [–] | Tensile Strength [kN/m] | Rupture Strain [–] | Tensile Strength [kN/m] | Rupture Strain [–] | Tensile Strength [kN/m] |
| 1 | 0.0738 | 114.0 | 0.0792 | 108.5 | 0.0625 | 155.6 | 0.0642 | 154.7 |
| 2 | 0.0679 | 98.7 | 0.0748 | 126.3 | 0.0608 | 158.9 | 0.0593 | 156.5 |
| 3 | 0.0733 | 103.9 | 0.0780 | 128.8 | 0.0626 | 146.5 | 0.0655 | 157.0 |
| 4 | 0.0748 | 122.0 | 0.0837 | 110.8 | 0.0601 | 153.6 | 0.0651 | 160.0 |
| 5 | 0.0758 | 115.4 | 0.0696 | 94.9 | 0.0591 | 142.4 | 0.0605 | 156.7 |
| 6 | 0.0725 | 117.4 | 0.0810 | 135.0 | 0.0600 | 141.5 | 0.0638 | 165.4 |
| mean | 0.0730 ± 0.0011 | 111.9 ± 3.6 | 0.0777 ± 0.0020 | 117.4 ± 6.2 | 0.0608 ± 0.0006 | 149.7 ± 2.9 | 0.0631 ± 0.0010 | 158.4 ± 1.6 |
| median | 0.0736 | 114.7 | 0.0786 | 118.5 | 0.0605 | 150.0 | 0.0640 | 156.8 |

### 3.3. Longitudinal stiffnesses

For the S and B types of PTFE-coated fabrics, the longitudinal stiffnesses are specified in Tables 6–9. The longitudinal stiffnesses $F_i$ (kN/m) are determined from the linear approximation of the stress-strain curves using the piecewise linear model [24, 35]. The graphical concept of the piecewise linear model identification is presented in Fig. 7. The

Table 6. Longitudinal stiffnesses – base specimens B type fabric

| Specimen No. | Weft | | Warp | |
|---|---|---|---|---|
| | $F_1$ [kN/m] | $F_2$ [kN/m] | $F_1$ [kN/m] | $F_2$ [kN/m] |
| 1 | 88.4 | 4750 | 418.1 | 5230 |
| 2 | 108.6 | 5140 | 568.8 | 5310 |
| 3 | 89.7 | 4860 | 518.5 | 5240 |
| 4 | 115.9 | 5080 | 614.8 | 5510 |
| 5 | 89.9 | 4970 | 504.5 | 4980 |
| 6 | 79.6 | 4710 | 433.2 | 5450 |
| mean | 95.3 ± 13.8 | 4918 ± 175 | 509.7 ± 76 | 5286.7 ± 187.9 |
| median | 89.8 | 4915.0 | 511.5 | 5275.0 |
| $\varepsilon_{p1}$ | 0.1053 | | 0.0369 | |

Table 7. Longitudinal stiffnesses – freezer cycles, B type fabric

| Specimen No. | Weft – F25 | | Warp – F25 | | Weft – F50 | | Warp – F50 | |
|---|---|---|---|---|---|---|---|---|
| | $F_1$ [kN/m] | $F_2$ [kN/m] | $F_1$ [kN/m] | $F_2$ [kN/m] | $F_1$ [kN/m] | $F_2$ [kN/m] | $F_1$ [kN/m] | $F_2$ [kN/m] |
| 1 | 96.3 | 5040 | 505.9 | 5480 | 100.9 | 4520 | 665.4 | 5080 |
| 2 | 69.2 | 4740 | 494.1 | 5030 | 117.6 | 4650 | 593.6 | 5110 |
| 3 | 125.0 | 4960 | 418.0 | 5210 | 113.1 | 4810 | 599.0 | 5080 |
| 4 | 94.2 | 5000 | 637.0 | 5220 | 124.4 | 4640 | 530.2 | 5050 |
| 5 | 85.1 | 5050 | 518.0 | 5210 | 132.4 | 4670 | 628.7 | 5100 |
| 6 | 109.2 | 4840 | 407.6 | 5100 | 137.9 | 4710 | 562.3 | 5100 |
| mean | 96.5 ± 19.3 | 4938 ± 123 | 496.8 ± 82.9 | 5208 ± 171 | 121 ± 13.5 | 4667 ± 95 | 596.5 ± 47.7 | 5087 ± 22 |
| median | 95.2 | 4980 | 500 | 5210 | 121 | 4660 | 596.3 | 5090 |
| $\varepsilon_{p1}$ | 0.1056 | | 0.0363 | | 0.1035 | | 0.0329 | |

Table 8. Longitudinal stiffnesses – base specimens S type fabric

| Specimen No. | Weft | | Warp | |
|---|---|---|---|---|
| | $F_1$ [kN/m] | $F_2$ [kN/m] | $F_1$ [kN/m] | $F_2$ [kN/m] |
| 1 | 226.4 | 5320 | 434.4 | 4346* |
| 2 | 239.7 | 5630 | 379.8 | 5190 |
| 3 | 254.1 | 5650 | 368.4 | 5190 |
| 4 | 227.8 | 4970 | 405.5 | 5380 |
| 5 | 258.2 | 5460 | 421.3 | 5320 |
| 6 | 245.1 | 5380 | 310.1 | 5370 |
| mean | 241.9 ± 13.2 | 5402 ± 249 | 386.6 ± 44.9 | 5290 ± 94 |
| median | 242.4 | 5420 | 392.7 | 5255 |
| $\varepsilon_{p1}$ | 0.0553 | | 0.0366 | |

Table 9. Longitudinal stiffnesses – freezer cycles, S type fabric

| Specimen No. | Weft – F25 | | Warp – F25 | | Weft – F50 | | Warp – F50 | |
|---|---|---|---|---|---|---|---|---|
| | $F_1$ [kN/m] | $F_2$ [kN/m] | $F_1$ [kN/m] | $F_2$ [kN/m] | $F_1$ [kN/m] | $F_2$ [kN/m] | $F_1$ [kN/m] | $F_2$ [kN/m] |
| 1 | 247.2 | 6140 | 380.2 | 5480 | 209.5 | 5290 | 375.6 | 5130 |
| 2 | 313.5 | 5670 | 437.8 | 5170 | 296.2 | 4810 | 501.4 | 5340 |
| 3 | 223.2 | 5530 | 374.8 | 5020 | 247.7 | 5270 | 373.0 | 5160 |
| 4 | 255.4 | 5830 | 402.7 | 5370 | 201.3* | 5840 | 385.1 | 4950 |
| 5 | 226.1 | 5700 | 394.2 | 5200 | 259.4 | 5500 | 466.0 | 5090 |
| 6 | 269.9 | 5820 | 417.9 | 5320 | 240.5 | 5680 | 420.2 | 5160 |
| mean | 253.1 ± 33.3 | 5782 ± 207 | 401.3 ± 23.7 | 5260 ± 163 | 250.7 ± 31.5 | 5398 ± 363 | 420.2 ± 53.2 | 5138 ± 126 |
| median | 251.3 | 5760 | 398.5 | 5260 | 247.7 | 5395 | 402.7 | 5145 |
| $\varepsilon_{p1}$ | 0.0561 | | 0.0350 | | 0.0587 | | 0.0351 | |

value $\varepsilon_{p1}$ (the strain for change of stiffness in the biaxial model) can be calculated according to the formula

$$\varepsilon_{p1} = \frac{\sigma_r - F_2 \cdot \varepsilon_r}{F_1 - F_2} \tag{3.1}$$

where: $\varepsilon_r$ is the mean rapture strain and $\sigma_r$ is the mean tensile strength (from Tables 2–5).

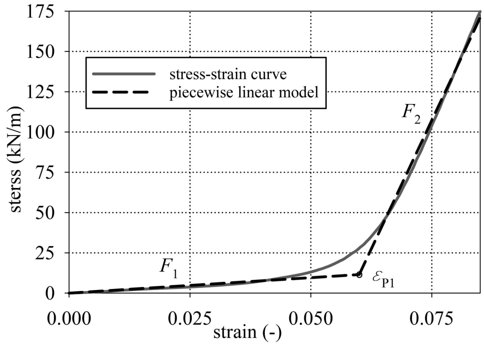


Fig. 7. Graphical concept of the piecewise linear model identification

# 4. Discussion

The one-way analysis of variance (ANOVA) was used to determine, whether there are any statistically significant differences between the means of the three investigated specimens groups [36, 37]. To perform ANOVA analysis it was necessary to check basic assumptions, which were the normal distribution of the results in the groups and the equal variance of the coated fabric specimens groups. The normal distribution was checked by the Shapiro–Wilk test [38], and the equal variance by the Brown–Forsythe test [39]. It should be noted, that if one of these tests fails the other variance analysis method should be used (e.g. Kruskal–Wallis One-Way Analysis [40]). To obtain the normal distribution of results a few results of the individual tests (with values much different from the others) have to be excluded from the ANOVA investigation (values with "*" in Tables 8–9). After that refinement, all results fulfilled the normal distribution and equal variance tests and therefore could be the subject of the ANOVA analysis.

The One-Way ANOVA compares the means between the groups you are interested in and determines whether any of those means are statistically significantly different from each other. Specifically, it tests the null hypothesis:

$$H_0 : m_1 = m_2 = m_3 = \ldots m_n \tag{4.1}$$

where: $m_i$ – the group mean and $n$ – the number of groups.

If, however, the One-Way ANOVA returned a statistically significant result, it is accepted the alternative hypothesis (H1), which is that there were at least two group means that were statistically significantly different from each other. To determine which two groups were statistically significantly different it was necessary to perform one of the post hoc tests. Here the Holm-Sidak method was adopted [41]. In Table 10 the results of the testing hypothesis of significant differences in group results are presented. If the difference is statistically important it is indicated in Table 10 with the word YES, otherwise the word NO is used.

It can be seen that in almost all cases of analysis for S fabric the differences are statistically not important. That means that the properties of the fabric are not changing significantly. That means the fabric has good resistance to cyclic temperature changes.

Table 10. Results of ANOVA analysis

| | Fabric B Weft | | | Fabric B Warp | | |
|---|---|---|---|---|---|---|
| | Tensile Strength | Long. Stiff. $F_1$ | Long. Stiff. $F_2$ | Tensile Strength | Long. Stiff. $F_1$ | Long. Stiff. $F_2$ |
| Base/F50 | NO | YES | YES | NO | NO | NO |
| Base/F25 | NO | NO | NO | NO | NO | NO |
| F25/F50 | NO | YES | YES | NO | NO | NO |
| | Fabric S Weft | | | Fabric S Warp | | |
| | Tens. Strength | Long. Stiff. $F_1$ | Long. Stiff. $F_2$ | Tensile Strength | Long. Stiff. $F_1$ | Long. Stiff. $F_2$ |
| Base/F50 | NO | NO | NO | NO | NO | NO |
| Base/F25 | NO | NO | NO | YES | NO | NO |
| F25/F50 | NO | NO | NO | YES | NO | NO |

For B fabric good frozen cycles resistance in the warp direction also can be confirmed. Nevertheless, the weft direction situation is the opposite. When more than 25 freeze cycles have been applied the changes in material properties should be considered. The explanation for that is the way how both fabrics have been manufactured. During the coating process of S fabric, both directions of the threads have been pre-tensioned. During coating of B fabric, only the threads in warp direction have been pre-tensioned. This causes larger strain values in this direction during exploitation as well as changes in temperature resistance.

## 5. Conclusions

In the present paper, the investigation of the influence of freezer and defrost in cycles on the mechanical properties of chosen PTFE-coated woven fabrics under uniaxial tensile tests is performed. Based on performed investigation the following conclusions may be drawn:

– A significant reduction in the tensile strength and longitudinal stiffnesses for investigated PTFE-coated woven fabrics resulting from performed frozen cycles was not observed.

– A significant difference in the elongation at break for PTFE-coated fabric under frozen cycles was not assigned.

The present study can be treated as a base for the new investigations on the influence of freezer and defrost in cycles on the architectural coated fabrics. The obtained results encourage the Authors to carry out broad investigations with a larger number of fabric specimens and usage of the curved grips.

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## Wpływ cyklicznego zamrażania i rozmrażania na właściwości mechaniczne tkanin technicznych powlekanych politetrafluoroetylenem (PTFE)

**Słowa kluczowe:** materiały konstrukcyjne, mrozoodporność, właściwości mechaniczne, tkanina techniczna powlekana PTFE, próby jednoosiowego rozciągania

### Streszczenie:

Artykuł opisuje badania tkanin technicznych wykonanych z włókana szklanego i powlekanych politertafluroetylenem (PTFE) (ang. *PTFE-coated woven fabric*), które mają na celu określenie wpływu cyklicznych procesów zamrażania i rozmrażania na właściwości mechaniczne wybranych tkanin Próbki tkanin technicznych zostały poddane zamrażaniu w temperaturze około −20°C przez 4 godziny i rozmrażaniu przez całkowite zanurzenie w wodzie w temperaturze około +20°C przez 4 godziny. Próbki tkanin technicznych po 25 i 50 cyklach zamrażania suszono na powietrzu w temperaturze pokojowej przez tydzień, a następnie poddano próbom jednoosiowego rozciągania. Na podstawie testów jednoosiowego rozciągania określono wytrzymałość na rozciąganie i moduły sztywności. Pomimo, iż w badanych tkaninach powlekanych następuje spadek wytrzymałości na rozciąganie w warunkach ich namoczenia w wodzie, to wykonane cykle zamrażania nie wykazują znacznego spadku wytrzymałości w próbach jednoosiowego rozciągania.

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