Validation procedures for assessing the properties of anti-vandal materials for applications in public transport vehicles
(Sponsored article)

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Abstract: Validation of methods for assessing the properties of anti-vandal hybrid composites modified with inorganic and plant fillers, with reduced flammability and smoke emission and high resistance to vandalism, intended for use in public transport, was carried out. Four standardized procedures, intended for testing personal protective equipment (protection of upper and lower limbs), including impact, and cut resistance and flame resistance, were modified. Since the tested materials were to be used in applications beyond the scope of the original test procedures, each method required re-validation. In this work, systematic and random errors of measurements that could affect the reliability and validity of the methods were identified. Therefore, individual uncertainty components, which characterize the dispersion of values assigned to the measured quantity at a given probability level, were precisely determined and then combined into uncertainty range for individual methods. After the validation, the methods can be successfully used to assess anti-vandal composite materials in the context of resistance to vandalism in public transport.

Keywords: resistance to mechanical and thermal factors, anti-vandal composite material, validation.

Procedury walidacyjne do oceny właściwości materiałów antywandalowych do zastosowań w pojazdach transportu publicznego
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Streszczenie: Przeprowadzono walidację metod oceny właściwości kompozytów hybrydowych antywandalowych, modyfikowanych napełniaczami nieorganicznymi i roślinnymi, o zmniejszonej palności i emisji dymu oraz dużej odporności na akty wandalizmu, przeznaczone do zastosowań w transporcie publicznym. Zmodyfikowano cztery standaryzowane procedury, przeznaczone do testowania środków ochrony osobistej (ochrony kończyn górnych i dolnych), obejmujące odporność na uderzenie i przecięcie oraz płomień. Ze względu na fakt, że badane materiały miały być stosowane w aplikacjach wykraczających poza zakres pierwotnych procedur testowych, każda metoda wymagała ponownej walidacji. W niniejszej pracy zidentyfikowano błędy systematyczne i losowe pomiarów, które mogłyby wpływać na wiarygodność i trafność metod. W związku z tym precyzyjnie określono poszczególne składowe niepewności, które charakteryzują rozrzut wartości przypisanych wielkości mierzonej na danym poziomie prawdopodobieństwa, a następnie połączono je w zakres niepewności dla poszczególnych metod. Po zakończeniu walidacji metody mogą być z powodzeniem stosowane do oceny materiałów kompozytowych antywandalowych w kontekście odporności na akty wandalizmu w transporcie publicznym.

Słowa kluczowe: odporność na czynniki mechaniczne i termiczne, kompozytowy materiał antywandalowy, walidacja.

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An act of vandalism is understood as the intentional destruction of public or private property without a valid reason. Vandalism is considered a social phenomenon that has a detrimental effect on urban public spaces [1]. According to data published by Eurostat, over the past five years the rate of vandalism recorded in the European Union member states has shown maximum fluctuations of up to 12%, with the risk of vandalism in some countries being estimated at close to 20%. In the face of the persistent global problem of vandalism, preventive measures must be taken, especially bearing in mind the increasing repair costs and the development of the technology industry [1]. Generally, there are three approaches to tackling vandalism: increased policing, devising new ways of repairing damage, and developing new highly resistant materials [2]. Given the fast rate of progress in material technologies, the third option seems the most promising.

The design of materials with advanced mechanical parameters for the public transport sector involves two broad categories, i.e., materials to be applied in vehicles and at stationary sites (e.g., passenger shelters), both of which are at risk of vandalism. The most frequently vandalized elements of buses are windows and seats, with upholstered seats being particularly vulnerable to sources of fire, such as a smoldering cigarette or a lighter flame. Even though the number of fires has been on the decrease over the past decade, incidents of fire setting are still the most frequent and important causes of the process of self-sustained combustion of flammable materials in different sectors [3]. According to the literature, much damage can be prevented by the use of composite materials made of polyacrylate or polycarbonate, which are impact resistant due to their physical and chemical properties. While seats in public transport vehicles are made of composite materials incorporating glass fibers, enhancing their resistance to cutting by sharp objects, their smooth structure is susceptible to graffiti, which is difficult to remove [4]. The replacement of synthetic fibers (e.g., glass or carbon) with natural ones, such as flax, jute, or coir, improves mechanical properties including tensile and bending resistance, resulting in lightweight and economical materials [5].

Given the specific applications of the aforementioned composites in public transport vehicles, their properties are tested by methods specified in the European standards which are widely used, e.g., in the railway industry. The materials and elements that are applied in railway vehicles and that are subject to tests according to the standard EN 45545-2:2020 [BS EN 45545-2:2020 Railway applications. Fire protection on railway vehicles Requirements for fire behaviour of materials and components; EN 45545-2:2013, Railway applications – Fire protection on railway vehicles – Part 2: Requirements for fire behaviour of materials and components. Annex B] [6] include electrical cables, coated materials, as well as metallic, non-metallic and glass surfaces. In turn, seats and seat materials are tested in accordance with the requirements of the aforementioned standard in terms of properties such as ignition or cutting. [BS EN 45545-2:2020 Railway applications. Fire protection on railway vehicles Requirements for fire behaviour of materials and components; EN 45545-2:2013, Railway applications – Fire protection on railway vehicles – Part 2: Requirements for fire behaviour of materials and components. Annexes A and B]. In the case of procedures evaluating materials for impact resistance, one can use the European standard PN-EN ISO 7765-1:2005 dedicated to plastics [7], which does not specify any specific application of the tested materials. In designing materials resistant to actual acts of vandalism, one should seek and implement evaluation methods that will reflect vandal behavior as closely as possible. In addition, of great importance is the development of test methods that would be straightforward and enable the evaluation of materials used, e.g., for the development of prototypes and end products.

Based on a literature review, the following four test procedures were selected to evaluate composite materials in terms of:

– resistance to cuts resulting from knife strikes (pursuant to PN–EN 1082-1:1999) [8],

– flame resistance (pursuant to PN–EN ISO 15025:2017-02) [9],

– resistance to cutting by sharp objects (pursuant to PN-EN ISO 13997:2003) [10],

– impact resistance (pursuant to PN-EN ISO 20344:2012 and the internal procedure NONB-33) [11].

The above methods of testing the cut, impact, and flame resistance of protective gloves were chosen because of their potential for simulating real-life conditions. The proposed modified methods constitute new tools for the evaluation of anti-vandal composite materials used in trains and buses. In accordance with the standard PN-EN ISO/IEC 17025:2018-02, General requirements for the competence of testing and calibration laboratories, validation should be performed for standardized methods which have been modified or which are applied beyond their original scope. The measures presented in this paper indicate that the results obtained and documented for the tested composites are satisfactory and that the methods are metrologically useful and can be deployed commercially.

EXPERIMENTAL PART

Materials

The following fabrics were used to prepare the composites: carbon fabric BIAX 400 g/m^2 from Saertex (Saerbeck, Germany), which is a sewn, two-way (+45/-45°) fabric with a weight of 410 g/m^2; aramid fabric 110 g/m^2 from P.P.H.U. SURFPOL (Rawa Mazowiecka, Poland), which is a linen weave (2/2 twill) fabric of 110 g/m^2 made from 42tex fibre; BIAX 400 g/m^2 glass fabric from Saertex (Saerbeck, Germany) and 200 g/m^2 from Interglass (Kraśnik, Poland), which is a bi-directional fabric (+45/-45°) of 411 g/m^2 made from E-glass; Basfiber® BT11/1 from Kamenny Vek (Dubna, Russia), which is a twill fabric with a weight of 380±25 g/m^2; linen fabric from Safilin (Szczytno, Poland), which is a twill fabric with a weight of 500 g/m^2 made from tex400 fibre. Powder fillers such as raw vermiculite of superfine fraction with a grain size in the range 0.3–1 mm (at least 80%) purchased from Vermeko Sp. z .o.o. (Lublin, Poland) were also used. The bulk density of the mineral is 110–130 kg/m^3 and the admixture of other rocks does not exceed 10 %; CO_2-filled glass beads with a diameter of 30–115 μm and a mass of approximately 120 g/l, with the trade name MIKROBALON DT-99; hazelnut shells obtained from the company AGRO Jarosław Seroczyński (Nadbrzeż, Poland).

Preparation of samples

The study involved anti-vandal composite materials made by vacuum bagging in the following way. The resin and hardener were mixed in a proLAB 075 dissolver from GlobimiX Ltd (Ząbkowice Śląskie, Poland). The resin was applied successively to fabric samples that were previously cut out and arranged on flat, parallel polyethylene plates and PTFE film. The resin was evenly distributed using a spatula, brushes, and laminating rollers. Subsequently, delaminating and breathable fabric layers were placed on the base fabrics, and the whole assembly was covered with a polymer vacuum bag, whose edges were sealed with tape. After removing air with a vacuum pump, the fabric layers were saturated with resin. The prepared composites were left to harden, and after 4 days they were cured at 70°C for 3 h. The composites also contained reinforcement in the form or flax, aramid, basalt, carbon, and glass fabrics, as well as powder fillers in various configurations.

Some of the composites had powder fillers in their composition, which were added to the epoxy resin on the occasion of some fabrics, as follows:

– vermiculite was added instead of one layer of aramid or carbon fabric, glass beads instead of one layer of glass fabric, hazelnut shells instead of one layer of linen fabric.

The order of materials used is indicated by the letters a-e respectively. The W-6 composite, on the other hand, is the reference material and was made from six layers of glass fabric.

The composition and manufacture of the composites tested are protected by patent application P.442733. Pictures and description of the composites are shown in Table 1.

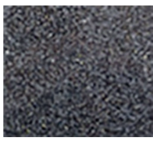
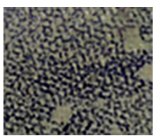
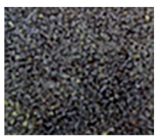
Methods

The selection of validation parameters for anti-vandal composite materials depended on their applications and was based on literature reports indicating that public transport vehicle components are most susceptible to damage due to burns, impacts with heavy objects and cutting by sharp objects. For this reason, composite materials were evaluated for two mechanical factors (impact and cutting) and one thermal factor (flame).

Resistance to cuts by knife strikes

The test procedure for determining the resistance of anti-vandal composite materials to cuts resulting from knife strikes was developed pursuant to the standard PN-EN 1082-1:1999 [8]. For this purpose, the experimental station was modified to increase strike energy. The test method was designed for 120×120 mm samples of anti-vandal materials and involved a striker weighing 2 kg (mb) that was positioned 580 mm above the sample (l) and that impacted it in free fall with an energy of 11.3 J.

Table 1. Results of simulation

Sample	W-0	W-1	W-2	W-3	W-4	W-5	W-6
							
Carbon fabric	2a	2a	1a	2b	2b	2c	–
Aramid fabric	2b	2b	2b	2a	1a	1a	–
Glass fabric	2c	2c	1c	2c	1c	2b	6a
Basalt fabric	2d	2d	2d	2d	2d	2d	–
Linen fabric	2e	2e	1e	2e	1e	1e	–
Vermiculite	–	–	1a	–	1a	1a	–
Glass beads	–	–	1c	–	1c	–	–
Nut shells	–	–	1e	–	1e	1e	–
Flame retardant	–	+	+	+	+	+	+

1, 2, 6 is a number of layers, while a–e is an order.

Flame resistance

The test procedure for determining the flame resistance of anti-vandal materials was developed pursuant to the standard PN-EN ISO 15025:2017-02 [9]. For this purpose, the original test procedure was expanded in terms of measuring the temperature of materials before and after the test. The method was designed for 70×70 mm samples of anti-vandal composite materials exposed to a 35±2 mm high flame for 60 s. The distance from the burner to the sample was 25±1 mm. The samples were mounted at an angle of 45±5° to the horizontal.

Resistance to cutting by sharp objects

The measurement procedure for determining the resistance of anti-vandal materials to cutting by sharp objects was conducted pursuant to the standard PN-EN ISO 13997:2003 [10]. However, the experimental stand was modified to increase blade pressure on the sample. The test method was designed for 100×70 mm samples of anti-vandal composite materials bent along the shorter edge at a radius of 38 mm, subjected to a cutting force of 250 N applied over a constant length of 65 mm.

Impact resistance

The measurement procedure for determining the impact resistance of anti-vandal materials pursuant to the standard PN-EN ISO 20344:2012 [11]. In this case, the experimental stand was modified to include strikers of different shapes. The test method was designed for 100×100 mm samples of anti-vandal composite materials. The apparatus involved a sample holder, modeling clay cylinders, and three 20 kg steel strikers with different impact heads:
– in the shape of a hemisphere with a radius of 25±1 mm;
– in the shape of a semicylinder with a radius of 10±1 mm;
– in the shape of a wedge with an angle of 90° and a tip radius of 3.0±0.1 mm.

Validation and statistical analysis

According to the standard PN-EN ISO/IEC 17025:2018-02, it is necessary to revalidate standard test methods which have been modified or which are to be used for purposes outside their original intended scope. The validation of each of the four procedures encompassed determining the components of standard measurement uncertainty affecting the results of tests conducted pursuant to the selected standards [12], and then estimating an uncertainty range indicating the percentage contributions of each factor to relative combined standard uncertainty. The revalidation process was conducted on the basis of the results obtained from the tests of composite materials W-0 – W-6 performed using suitably modified measurement methods. The mechanical tests of resistance to knife strikes and impacts were conducted in triplicate for each composite material, while the tests of resistance to cutting by sharp objects was conducted in quintuplicate, pursuant to the standard (no outliers were found on the basis of the Grubbs statistical test). Finally, thermal tests were performed in one replicate for each material. The methodology of the revalidation process is presented below in four segments – one for each measurement procedure.

Resistance to cuts by knife strikes

The measurement uncertainty of the presented method for evaluating the resistance of anti-vandal composite materials to cuts by knife strikes consists of the following uncertainty components (the calculations shown are for composite W-3).

The standard measurement uncertainty of striker weight (u_1) was estimated from the Equation (1):

$$u_1 = \frac{0.0112}{2} = 0.0056 \text{ [kg]} \tag{1}$$

The striker was weighed using a digital newton meter, whose expanded measurement uncertainty was ±1.12 g at a confidence interval of approx. 95% and a coverage factor of $k = 2$. Its contribution to relative combined standard uncertainty was determined on the basis of the Equation (2):

$$w_1 = \frac{u_1}{m_b} = \frac{0.0056}{2} = 0.0028 \tag{2}$$

The standard measurement uncertainty of the distance between the striker and the sample (u_2) was estimated using the Equation (3):

$$u_2 = \frac{0.3}{2} = 0.15 \text{ [mm]} \tag{3}$$

The distance between the striker and the sample was measured using a semi-rigid graduated ruler, whose expanded measurement uncertainty was ±0.3 mm at a confidence interval of approx. 95% and a coverage factor of k = 2. Its contribution to relative combined standard uncertainty was found from the Equation 4:

$$w_2 = \frac{u_2}{l} = \frac{0.15}{580} = 0.000259 \tag{4}$$

The standard measurement uncertainty of sample cut depth (u3) for the composite material W-3 was estimated from the Equation (5):

$$u_3 = \frac{0.02}{2} = 0.01 \text{ [mm]} \tag{5}$$

Cut depth was measured using an electronic caliper whose expanded measurement uncertainty was ±0.02 mm at a confidence interval of approx. 95% and a coverage factor of $k = 2$. Its contribution to relative combined standard uncertainty was found using the Equation (6):

$$w_3 = \frac{u_3}{h} = \frac{0.01}{8.5} = 0.00118 \tag{6}$$

The standard uncertainty of the dispersion of measurement results (u_4) was estimated from the Equation (7):

$$u_4 = \frac{S}{\sqrt{n}} = \frac{0.5}{\sqrt{3}} = 0.289 \text{ [mm]} \tag{7}$$

Standard deviation (S) was calculated for $n = 3$, where n is the number of replicates. Its contribution to relative combined standard uncertainty was computed for the composite material W-3 from the Equation (8):

$$w_4 = \frac{u_4}{l} = \frac{0.289}{8.5} = 0.034 \tag{8}$$

Expanded uncertainty was calculated using the Equation (9):

$$U_{95} = 2 \cdot u_4 \text{ [mm]} \tag{9}$$

Expanded uncertainty for three measurements ($n = 3$) of the depth of cuts in the anti-vandal composite material W-3 was 8.5±0.6 mm at a confidence interval of approx. 95% and a coverage factor of $k = 2$.

Measurement uncertainty for the resistance of anti-vandal composite materials to cuts by knife strikes was estimated on the basis of experimental data.

Flame resistance

Measurement uncertainty for the presented methodology of evaluating the flame resistance of anti-vandal composite materials consists of the following components (the calculations shown are for the composite material W-0).

The standard measurement uncertainty of flame application (u_1) was estimated using the Equation (10):

$$u_1 = \frac{0.13}{2} = 0.065 \text{ [s]} \tag{10}$$

Flame application time was measured using a mechanical stopwatch whose expanded measurement uncertainty was ±0.13 s at a confidence interval of approx. 95% and a coverage factor of $k = 2$. Its contribution to relative combined standard uncertainty was determined on the basis of the Equation (11):

$$w_1 = \frac{u_1}{t} = \frac{0.065}{60} = 0.00108 \tag{11}$$

The standard measurement uncertainty of temperature before flame application (u_2) was estimated from the Equation (12):

$$u_2 = \frac{0.2}{\sqrt{3}} = 0.1156 \text{ [°C]} \tag{12}$$

Temperature was measured using a 176H1 temperature and humidity probe with an accuracy of ±0.2°C. Its contribution to relative combined standard uncertainty was found from the Equation (13):

$$w_2 = \frac{u_2}{T} = \frac{0.1156}{25} = 0.00462 \tag{13}$$

The standard measurement uncertainty of temperature after flame application (u_3) was estimated from the Equation (14):

$$u_3 = \frac{0.2}{\sqrt{3}} = 0.1156 \text{ [°C]} \tag{14}$$

Again, temperature was measured using a 176H1 temperature and humidity probe with an accuracy of ±0.2°C. Its contribution to relative combined standard uncertainty was determined using Equation (15):

$$w_2 = \frac{u_3}{T} = \frac{0.1156}{66} = 0.00175 \tag{15}$$

Expanded uncertainty was calculated on the basis of the Equation (16):

$$\begin{aligned} U_{95} &= 2 \cdot T \cdot \sqrt{w_1^2 + w_2^2 + w_3^2} = \\ &= 2 \cdot T \cdot \sqrt{0.00108^2 + 0.00462^2 + 0.00175^2} = 0.01 \cdot T \text{ [°C]} \end{aligned} \tag{16}$$

Expanded uncertainty for a single temperature measurement during tests of the anti-vandal composite material W-0 was 66.4±0.7°C at a confidence interval of approx. 95% and a coverage factor of $k = 2$.

Measurement uncertainty for the flame resistance of anti-vandal composite materials was estimated on the basis of experimental data.

Resistance to cutting by sharp objects

Measurement uncertainty for the presented methodology of evaluating the resistance of anti-vandal composite materials to cutting by sharp objects consists of the following components (the calculations shown are for the composite material W-0).

The standard uncertainty of force applied (u_1) was estimated from the Equation (17):

$$u_1 = \frac{0.5}{\sqrt{3}} = 0.289 \text{ [N]} \tag{17}$$

The expanded measurement uncertainty of the device was 0.5 N, and its contribution to relative combined standard uncertainty was calculated from the Equation (18):

$$w_1 = \frac{u_1}{F} = \frac{0.289}{250} = 0.001156 \tag{18}$$

The standard uncertainty of cut length (u_2) was estimated from the Equation (19):

$$u_2 = \frac{0.02}{2} = 0.01 \text{ [mm]} \tag{19}$$

Cut length was measured using an electronic caliper whose expanded measurement uncertainty was ±0.3 mm with a confidence interval of approx. 95% and a coverage factor of k = 2. Its contribution to relative combined standard uncertainty was found using the Equation (20):

$$w_2 = \frac{u_2}{l} = \frac{0.01}{15.8} = 0.000633 \tag{20}$$

The combined uncertainty of the blade sharpness correction factor (u_3) was estimated using the exact differential method, taking into account the mathematical properties of the blade sharpness factor, calculated using Equation (21), which is used in Equation (22):

$$C = \frac{K}{l} + dC \tag{21}$$

$$\begin{gathered}\frac{\delta C}{\delta l} = \left|-\frac{K}{l^2}\right| = \left|-\frac{20}{l^2}\right| = \left|-\frac{20}{24.8^2}\right| = 0.0325\\ u_l = \frac{0.1}{2\sqrt{3}} = 0.029\,[\mathrm{mm}]\\ u_{dC} = \frac{S}{\sqrt{\ }} = \frac{1.0478}{\sqrt{10}} = 0.3313\,[\mathrm{mm}]\\ u_3 = \sqrt{\left(-\frac{20}{l^2}\right)^2 \cdot u_l^2 + u_{dC}^2} = \sqrt{0.0325^2 \cdot 0.029^2 + 0.3313^2} = 0.3342\,[\mathrm{mm}]\end{gathered} \tag{22}$$

Its contribution to relative combined standard uncertainty was determined from the Equation (23):

$$w_3 = \frac{u_3}{l} = \frac{0.3342}{24.8} = 0.01348 \tag{23}$$

The standard uncertainty of the dispersion of measurement results (u_4) for the composite material W 0 was estimated on the basis of the Equation (24):

$$u_4 = \frac{S}{\sqrt{n}} = \frac{1.82}{\sqrt{5}} = 0.8139\ [\mathrm{mm}] \tag{24}$$

Standard deviation (S) was calculated for n = 5, where n is the number of replicates. Its contribution to relative combined standard uncertainty was computed from the Equation (25):

$$w_4 = \frac{u_4}{l} = \frac{0.8139}{15.8} = 0.0515 \tag{25}$$

Expanded uncertainty was calculated using the following Equation (26):

$$\begin{gathered}u_c = l \cdot \sqrt{w_3^2 + w_4^2} = 15.8 \cdot \sqrt{0.01348^2 + 0.0515^2} = 0.84\ [\mathrm{mm}]\\ U_{95} = 2 \cdot u_c = 2 \cdot 0.84 = 1.7\ [\mathrm{mm}]\end{gathered} \tag{26}$$

Expanded uncertainty for a single measurement of cut length in the anti-vandal composite material W-0 was 15.8±1.7 mm at a confidence interval of approx. 95% and a coverage factor of k = 2.

Measurement uncertainty for the resistance of anti-vandal composite materials to cutting by sharp objects was estimated on the basis of experimental data.

Impact resistance

Measurement uncertainty for the presented methodology of evaluating the impact resistance of anti-vandal composite materials consists of the following components (the calculations shown are for the composite material W-0).

The standard uncertainty of striker weight (u_1) was estimated using Equation (27):

$$u_1 = \frac{3}{2} = 1.5\ [\mathrm{g}] \tag{27}$$

The striker was weighed using a digital newton meter whose expanded measurement uncertainty was ±3 g at a confidence interval of approx. 95% and a coverage factor of k = 2. Its contribution to relative combined standard uncertainty was determined from the Equation (28):

$$w_1 = \frac{u_1}{m_b} = \frac{1.5}{20094} = 0.000075 \tag{28}$$

The standard measurement uncertainty of the distance between the striker and the sample (u_2) was estimated from the Equation (29):

$$u_2 = \frac{0.12}{2} = 0.06\ [\mathrm{mm}] \tag{29}$$

The distance between the striker and the sample was measured by means of a semi-rigid graduated ruler whose expanded measurement uncertainty was ±0.12 mm at a confidence interval of approx. 95% and a coverage factor of k = 2. Its contribution to relative combined standard uncertainty was calculated from the Equation (30):

$$w_2 = \frac{u_2}{l} = \frac{0.06}{500} = 0.00012 \tag{30}$$

The standard measurement uncertainty of the height of the clay cylinder (u_3) before tests for the composite material W-2 was estimated from the Equation (31):

$$u_3 = \frac{0.009}{2} = 0.0045\ [\mathrm{mm}] \tag{31}$$

Cylinder height was measured using a dial thickness gauge whose expanded measurement uncertainty was ±9 μm at a confidence interval of approx. 95% and a coverage factor of k = 2. Its contribution to relative combined standard uncertainty was determined using the Equation (32):

$$w_3 = \frac{u_3}{h} = \frac{0.0045}{40} = 0.0001125 \tag{32}$$

The standard measurement uncertainty of the clay cylinder after tests (u_4) for composite W-2 was estimated from the Equation (33):

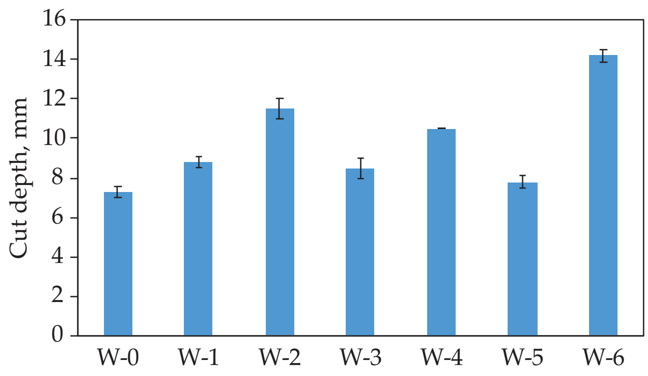


Fig. 1. Material resistance to cuts by knife strikes

$$u_4 = \frac{0.009}{2} = 0.0045\ [\text{mm}] \tag{33}$$

Again, cylinder height was measured using a dial thickness gauge whose expanded measurement uncertainty was ±9 μm at a confidence interval of approx. 95% and a coverage factor of $k = 2$. Its contribution to relative combined standard uncertainty was determined using the Equation (34):

$$w_4 = \frac{u_3}{h} = \frac{0.0045}{16.1} = 0.00028 \tag{34}$$

The standard uncertainty of the dispersion of measurement results (u_5) was estimated from the Equation (35):

$$u_5 = \frac{S}{\sqrt{n}} = \frac{S}{\sqrt{5}} = 0.447\,S = 0.1028\ [\text{mm}] \tag{35}$$

Standard deviation (S) was calculated for $n = 3$, where n is the number of replicates for the composite material W-2. Its contribution to relative combined standard uncertainty was computed from the Equation (36):

$$w_5 = \frac{u_5}{l} = \frac{0.447 \cdot 0.23}{16.1} = 0.00639 \tag{36}$$

Expanded uncertainty was calculated from the Equation (37):

$$U_{95} = 2 \cdot u_5 = 2 \cdot 0.103 = 0.2\ [\text{mm}] \tag{37}$$

Expanded uncertainty for cylinder height measurements ($n = 3$) after impact tests of the anti-vandal composite material W-2 was 16.1±0.2 mm with a confidence interval of approx. 95% and a coverage factor of $k = 2$.

Measurement uncertainty for the impact resistance of anti-vandal composite materials was estimated on the basis of experimental results.

RESULTS

Based on the obtained results, this section presents the illustrations of uncertainty range for each test procedure separately and provides the percentage contributions of the various sources of error (associated with the apparatus as well as the operator) that may affect the evaluation of anti-vandal composite materials pursuant to the methods described in this text.

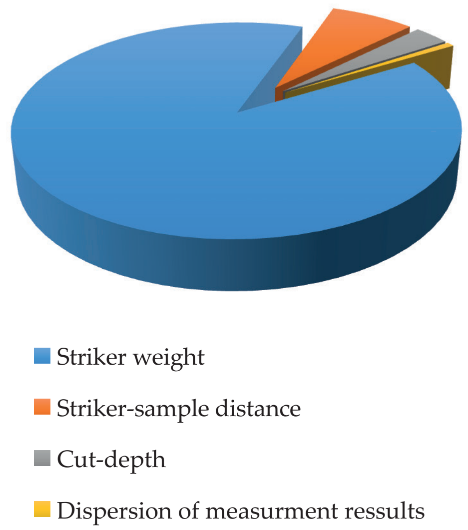


Fig.2. Uncertainty range for the method for determining the W-0 anti-vandal composite resistance to cuts caused by knife strikes

Resistance to cuts by knife strikes

Figure 1 presents the obtained results for resistance to cuts resulting from knife strikes for each of the studied anti-vandal composite materials.

In turn, Figure 2 shows the uncertainty range for the resistance of one of the studied anti-vandal composite materials (W-3) to cuts by knife strikes.

Flame resistance

Figure 3 presents the uncertainty range for flame resistance testing of one of the studied anti-vandal composite materials (W-0).

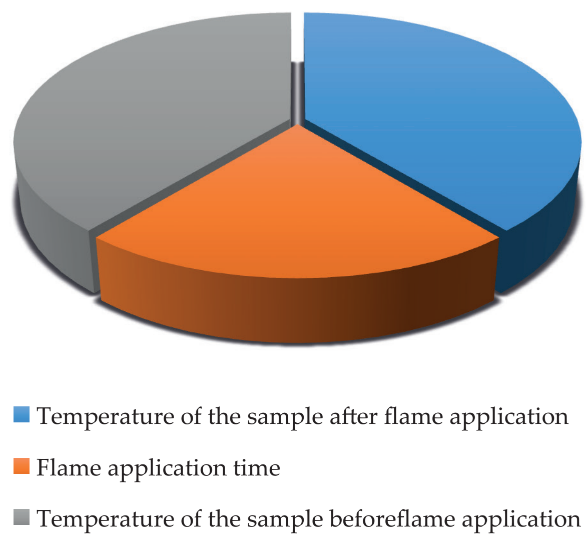


Fig. 3. Uncertainty range for the method for determining the fire resistance of the W-0 anti-vandal composite material

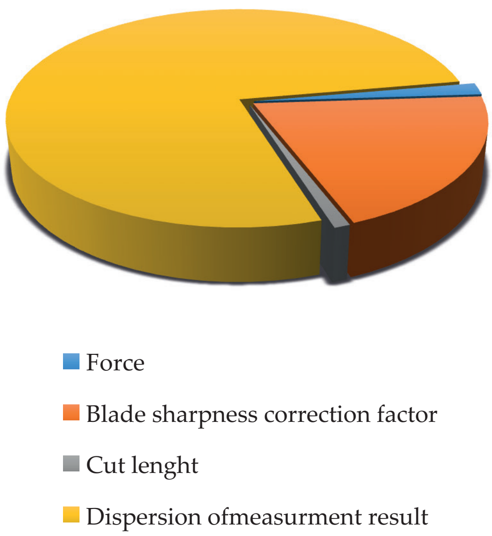


Fig. 4. Uncertainty range for the method for determining the W-0 anti-vandal composite resistance to cuts caused by knife cuts

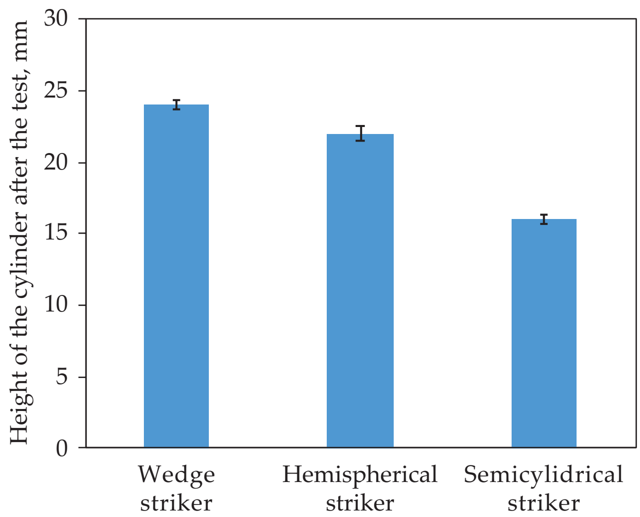


Fig. 5. Results of impact resistance tests for material W-2 conducted using three types of strikers

Resistance to cutting by sharp objects

Figure 4 shows the uncertainty range for the resistance of one of the studied anti-vandal composite materials (W-0) to cutting by sharp objects based on the results obtained from tests.

Impact resistance

Figure 5 presents the results of impact resistance tests performed using three strikers of different shapes for one of the studied anti-vandal composite materials (W-2).

Figure 6 presents the uncertainty range based on the results obtained from impact resistance tests performed using three strikers of different shapes for one of the studied anti-vandal composite materials (W-2).

Figure 7 shows the contributions of operator- and apparatus-related factors for all four measurement procedures.

Resistance to knife strikes was determined by means of a procedure dedicated to testing chain mail gloves, mostly used in the meatpacking industry. In contrast to the test method for cutting by sharp objects, this procedure additionally enables the determination of the resistance of anti-vandal composite materials to strikes or cuts by hand knives, which are commonplace in acts of vandalism [13]. In the validation process, tests for resistance to cuts by knife strikes were carried out pursuant to the standard PN-EN 1082-1:1999 (Fig. 1). Based on the specific characteristics of the tests, we determined their components affecting systematic errors, such as measurement uncertainty associated with the newton meter used for

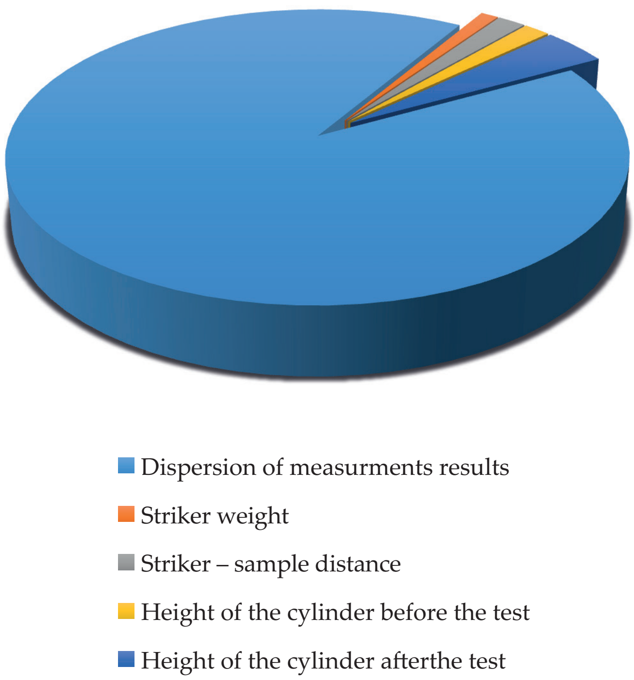


Fig. 6. Uncertainty range for the method of determining the resistance of the anti-vandal composite material W-2 to impact resistance tests

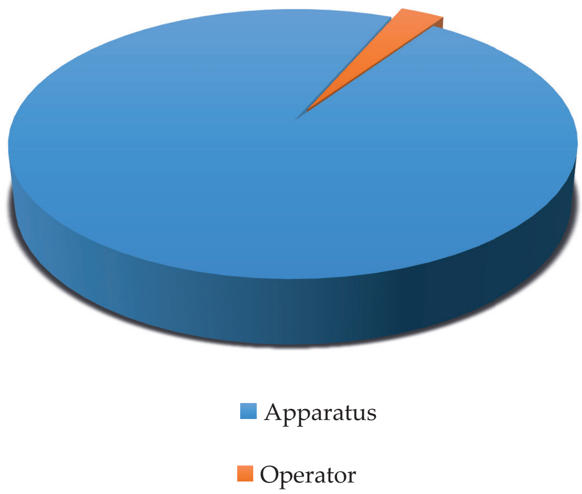


Fig. 7. Contributions of factors for the method of determining the resistance of the anti-vandal composite material W-2 to impact resistance tests

weighing the striker, the graduated ruler used for measuring the distance between the striker and the sample, and the digital caliper used for ascertaining sample cut depth. Random errors were accounted for as the dispersion of the obtained results, represented as standard deviation for a series of three tests. Subsequently, based on the estimated standard uncertainties (u_1–u_4), an uncertainty range was defined including its individual components (Fig. 2). Analyzing the formulas W1–W4, it should be noted that test results may be affected by some constant factors, whose relative combined standard uncertainty will be independent of the studied composite material under constant experimental conditions, i.e., striker weight and the striker–sample distance. In turn, the contributions of factors determining cut depth and the dispersion of results to the uncertainty range will be directly linked to the test results. Figure 2 shows the uncertainty range for the composite material W-3, indicating the aforementioned four components. The largest percentage contribution to that range is associated with component W4, which affects the final results to the greatest extent. Therefore, in estimating expanded measurement uncertainty (U95) one can ignore the remaining components (Eq. 9).

According to its original scope, the procedure for determining flame resistance makes it possible to determine the properties of a material in terms of flame spread as a result of the direct contact of samples with a low-intensity flame. Information about the behavior of anti-vandal composite materials in response to flames is important due to the frequent incidents of fire setting by vandals. Of special value here are data on afterflame and afterglow time as well as the maximum temperature reached by the composite (owing to the risk of pyrolysis of the polymer). Flame resistance tests were conducted only once for each composite (W-0–W-6) because of the limited availability of the materials studied. Each element of the measurement system was considered individually to identify the factors that may affect measurement results. These include flame application time (which should be the same during all tests) as well as the temperature of the composite before and after the test, which depends on the characteristics of individual samples. Based on this, we determined standard and relative combined standard uncertainties, which had maximum values of the order of 10-3. Figure 3 shows the percentage contributions of each of the three components affecting measurement results in the procedure. The results indicate that each factor does influence measurement, and so expanded uncertainty was calculated taking into account all three parameters. They are of special importance with a view to estimating allowable flame temperature and flame application time before ignition of the composite material.

The determination of resistance to cutting by sharp objects was conducted in quintuplicate and involved the measurement of cut length for each composite pursuant to the standard PN-EN 1082-1:1999. A significant difference between the European standard and the presented method being validated for railway applications is the much greater force with which the blade is applied to the material. In addition, the new procedure provides for cut length measurement only at one, constant pressure on the sample, which is 250 N (nearly 100 N more than in the European standards in the railway sector). Test results were affected by parameters such as the force with which the blade acts on the sample, cut length, the blade sharpness correction factor, as well as the dispersion of results around the mean for five replicates. The first of these uncertainty components is uncorrelated with measurement results, while the magnitudes of the remaining components are sample-dependent. Figure 4 presents the percentage contribution of each parameter associated with the apparatus or random factors for the composite material W-0. By far the most important parameters in the uncertainty range defined for measuring resistance to cutting by sharp objects are the blade sharpness correction factor and the dispersion of measurement results, and so the extended measurement uncertainty for this procedure is deemed to consist only of these two parameters (Eq. 26). Errors associated with the applied cutting force or cut length are systematic and cannot be eliminated, but their effects on the measurand are negligible.

The procedure determining impact resistance according to PN-EN ISO 20344:2012 was originally intended for testing toecaps in safety footwear designed for protecting users from objects falling with a certain energy and pressure [11]. With respect to composite materials, this procedure enables their evaluation in terms of resistance to impact by objects of different shapes. Figure 5 presents the effect of the shape of the impact head on results for the composite material W-2 together with a standard deviation for three replicates. Evaluation revealed five parameters that may potentially affect measurement results (Fig. 6), of which the most important one is the random factor, that is, dispersion around the mean. As the effects of the remaining factors are negligible, they do not influence the reliability or effectiveness of the measurement procedure, and so they were ignored in calculating expanded uncertainty.

DISCUSSION

In summary, statistical analysis of the four presented measurement procedures for anti-vandal materials revealed errors associated with both the operator and apparatus. The latter include striker weight, force applied, striker–sample distance, etc., while the former corresponds to the dispersion of results around the mean. Taking into account all parameters, an uncertainty range was prepared for each of the measurement methods for anti-vandal materials taking into account the two crucial elements of the operator and apparatus, encompassing all the factors identified for them. As can be seen from Figure 7, the factors associated with the apparatus may exert an effect on the measurement amounting to a total of 3%, while the largest errors potentially affecting the final measurement results are associated with the operator's actions. Nevertheless, the estimation of an uncertainty range, on the basis of which factors comprising expanded uncertainty were identified, makes it possible to present the end result of the study at an appropriate confidence level. Due to the insufficient number of samples obtained we were unable to estimate parameters such as reproducibility or precision.

In the literature devoted to polymeric materials and composites, studies are often aimed at investigating and expanding knowledge on properties such as tensile strength, bending, or elastic modulus upon bending [14, 15]. In the case of developing polymeric composites with improved resistance, the static and standard tests that are now used for determining the required properties of such materials, cannot fully reflect parameters crucial with respect to acts of vandalism. Therefore, studies such as those conducted by Krzyżak *et al.* [16] and Al-Shammari *et al.* [17], which evaluate polymeric composites in terms of their mechanical properties relevant for applications in vehicles, in accordance with PN-EN ISO 7765-1:2005 and PN-EN ISO 527-1:2020-01, are indispensable. The aforementioned standard PN-EN ISO 7765-1:2005 specifies procedures for measuring the impact resistance of plastic films and sheets by means of a dart with a hemispherical head. The document describes two test methods differing in terms of dart head diameter and drop height, but it does not provide any indications as to the applicability of different dart head shapes [6]. Since from the standpoint of the present work the shape of the impacting object may matter, it is necessary to use impact heads of more than one shape (especially rounded). Another test method in the area of impact resistance is specified in PN-EN ISO 6272-1:2011. It is designed to evaluate materials in terms of the resistance of paint and varnish coatings to cracks or peeling as a result of deformations caused by the fall of a 2 kg spherical indenter with a diameter of 20 mm [18]. Unfortunately, the method is limited to examining changes on the surface of the material, and additionally the sample preparation procedure involves coatings subjected to deformation by the pressing of materials [19]. In the case of simulating actual situations any deformations of the analyzed object will carry the risk of potential errors and inappropriate application of the polymeric composite. In the scope of materials for seats in public transport vehicles one should refer to the methods used by the Railway Research Institute. One of the standards it has adopted is EN 45545-2:2020 (E) replacing EN 45545-2:2013, which specifies requirements concerning, e.g., the fire behavior of materials and components used in railway vehicles. According to clause B.4 Fire test method for seating of the previous edition of this standard (EN 45545-2:2013), which was binding prior to the implementation of European Union fire protection requirements in the railway sector as of January 1, 2018 as well as the current implementation standard EN 16989:2018, which is a refinement of Annexes A and B of the previous edition of the standard EN 45545-2, flame resistance testing involves an entire object (complete passenger seat) including the armrest and headrest, as well as any separate cushions [20, 21]. The selection and optimization of polymeric composites for anti-vandal applications makes it necessary to provide large samples of a defined shape for each test, which involves considerable material loss and is not always possible (EN 45545-2:2013). The literature also describes test procedures for the last of the parameters discussed in this paper, that is, cut resistance pursuant to EN 45545-2:2013, Annex A Standard vandalism test for seat covering, dedicated to coated materials. Here, test methodology involves evaluation of cuts to material samples larger than 50 mm acted upon with blades made of carbon-chromium steel with a hardness of at least 800 HV5 at a force of 150 N. It should be noted that this procedure is dedicated only to flat materials, while seats contain a number of rounded elements, which may behave completely differently in contact with a blade. Additionally, in developing new methods for testing the strength parameters of damage-resistant materials, one should take into account the advancement of modern technologies offering tools characterized by greater strength and weight, which can better reflect real-life conditions. One solution is to increase the force with which the tool acts on the studied material, or to increase the weight of the tool. Currently, in the research literature there is a lack of publications about materials for applications in public transport vehicles that would feature improved anti-vandal properties. Much more attention has been devoted to public transport vehicle materials that are produced in an environmentally friendly way, with decreased production costs and material weight, which translates into superior economic efficiency and lower emissions during the operation of vehicles [5]. While similar solutions have been used in composite materials, including the incorporation of natural fibers in the polymeric matrix, the application scope of the resulting products is completely different. As a result, the existing measurement procedures do not make it possible to evaluate material resistance under real-life conditions. Moreover, the existing test methods dedicated to the public transport sector (EN 45545-2:2020 (E)), or the impact resistance method specified in PN-EN ISO 527-1:2020-01, cannot be used for samples produced in laboratory settings due to their application, the evaluation of objects at risk of damage, or an inappropriate form of the composite produced. One way to solve this problem is to apply methods designed for the testing of resistance of personal protective equipment (PPE) to hazardous factors in the workplace during the performance of routine tasks because procedures evaluating PPE are very often developed in such a way that the test conditions reflect the actual occupational tasks to the extent possible.

The presented validation of anti-vandal composite materials as well as the compiled literature data indicate that the modified and validated measurement procedures enable:

– ensure accurate evaluation of polymeric composites in accordance with PN-EN ISO 17025:2018-02 for standard methods in the context of mechanical and thermal properties crucial for the resistance of polymeric composites to acts of vandalism occurring in public transport vehicles, such as fire setting, impact, and cutting;

– enable the development of new tools for the evaluation of the mechanical and thermal resistance of composites, characterized by a hardness of approx. 90°Sh D, with measurement procedures that make it possible to conduct tests under realistic conditions (e.g., by using different striker shapes or applying force and power values corresponding to human movements);

– ensure the maintenance of metrological consistency among the obtained and presented results.

CONCLUSIONS

The current results for composite materials indicate that the standard measurement procedures, which were modified and validated in this work, are suitable for the evaluation of anti-vandal composite materials. This is confirmed by the expanded uncertainties obtained for the measurands. Analysis of standard uncertainties and uncertainty range with the percentage contributions of their constituent components, as well as estimated errors, indicate that the developed procedures are appropriate for the assessment of composite materials in terms of impact, cut, and fire resistance. A sufficient confidence level of results is crucial in the case of developing new composites and analyzing them in terms of their composition, which has a direct bearing on the properties of the resulting materials (e.g. for prototyping). The intended objects of study are materials that can be used in public transport vehicles potentially exposed to harsh external mechanical and thermal factors. Thus, it is all the more important to conduct research with the use of validated measurement procedures to make sure that the results are repeatable and reflect actual situations

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Authors contribution

E.I. –methodology, validation, investigation, writing-review and editing, visualization; K.M. – conceptualization, methodology, writing-review and editing; K.S. – conceptualization, methodology, supervision.

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Conflict of interest

The authors declare no conflict of interest.

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