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COMPARATIVE STUDIES OF TRIBOLOGICAL PROPERTIES OF CARBON FIBRE REINFORCED POLYMER COMPOSITES (CFRP) IN DRY SLIDING FRICTION CONDITIONS

BADANIA PORÓWNAWCZE WŁAŚCIWOŚCI TRIBOLOGICZNYCH WARSTWOWYCH KOMPOZYTÓW POLIMEROWYCH WZMACNIANYCH WŁÓKNAMI WĘGLOWYMI (CFRP) W WARUNKACH TARCIA ŚLIZGOWEGO SUCHEGO

Key words: carbon fibres reinforced polymer (CFRP), indentation hardness (universal), sliding friction, wear.

Abstract: The aim of the study was to determine the usable and quality features and tribological properties of four Carbon Fibre Reinforced Polymer laminates (CFRP) newly developed by the authors and to indicate the structures with the most advantageous functional properties. The developed material samples were based on two types of prepreg, Kord Carbon (Fiberpreg GmbH) with twill weave and carbon fibres (UD) by the manufacturer (G. Angeloni S.R.L) with a unidirectional system. In the structure of half of the total number of samples, a manufacturer's (NTPT) epoxy adhesive film (AF) layer was used. Tests of indentation hardness (universal) were carried out according to the Oliver-Pharr method. Tests of resistance to abrasive wear were carried out in dry sliding friction conditions, using the ball-disc method. In combination, a ball made of aluminium oxide (Al_2O_3) was used as a cooperating element. The test results indicate a significant impact of the structure of the tested materials on the increase in contact strength and the improvement of the tribological properties that result from the application of the AF layer.

Słowa kluczowe: kompozyty polimerowe wzmacniane włóknami węglowymi (CFRP), twardość indentacyjna (uniwersalna), tarcie ślizgowe, zużycie.

Streszczenie: Celem badań było określenie cech użytkowych i jakościowych oraz właściwości tribologicznych czterech nowo opracowanych przez autorów laminatów o osnowie polimerowej wzmacnianych włóknami węglowymi (CFRP – Carbon Fiber Reinforced Polymer) i wskazanie struktur o najkorzystniejszych właściwościach użytkowych. Opracowane próbki materiałów oparte były na dwóch typach prepregu: Kord Carbon (Fiberpreg GmbH) o splocie twill oraz włókien węglowych (UD) producenta (G. Angeloni S.R.L) o jednokierunkowym układzie. W strukturze połowy ogólnej liczby próbek zastosowano warstwę epoksydowego filmu klejowego (AF) producenta (NTPT). Wykonano badania twardości indentacyjnej (uniwersalnej) wg metody Olivera-Pharra. Badania odporności na zużycie ścierne prowadzono w warunkach tarcia ślizgowego suchego, metodą kula–tarcza. W skojarzeniu jako przeciwpróbkę wykorzystano kulkę wykonaną z tlenku glinu (Al_2O_3). Wyniki badań wskazują na istotny wpływ struktury badanych materiałów na wzrost wytrzymałości stykowej oraz polepszenie właściwości tribologicznych, które wynikają z zastosowania warstwy AF.

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INTRODUCTION

Carbon fibre reinforced polymer composites (CFRP) are increasingly used as construction materials. They are characterized by high specific strength and stiffness **[L. 1, 2, 3]**. Depending on the purpose of the element made of CFRP, in addition to the above-mentioned functional features, the composite should be characterized, e.g., by creep resistance, a high temperature of destruction of the polymer matrix, polymer degradation without the release of harmful low molecular weight compounds or other elements, etc. These materials are not usually used for strictly tribological applications **[L. 4]**. CFRP is commonly used, among others, in the following industries: space, automotive, marine **[L. 5]**, in aircraft structures **[L. 6]** and medical apparatus as well as in armaments and fire fighting techniques. But elements made of CFRP are exposed to sliding frictional contact, because they are often used, e.g., as uncoated or unprotected elements of bodies or housings. This happens, among others, because of the special design, but not exclusively. CFRP are increasingly used in machine construction, e.g., for the manufacture of shafts, grippers and arms of industrial and exploration robots, brake pedals, guides, bearings **[L. 7]**, multilayer leaf springs **[L. 8]**, and in the construction of many lighter machine parts in which contact loads occur. They are also used to strengthen steel and wooden structures.

Knowledge of the kinematic pairs work characteristics is helpful in the selection of durable materials for structural elements. The basic sources of information about the behaviour of the elements of the kinematic pair are its mechanical characteristics, illustrating the relationship of contact deformations with the loads causing them **[L. 9]**. The indentation hardness method is used to determine them. The result of the measurement with this method is to obtain a dependence of force on the penetration depth of the indenter. The registration of these quantities takes place during the phases of normal force and unloading. Based on the obtained curve, it is possible to determine a number of mechanical quantities helpful in the comparative quantitative assessment of the quality of the tested materials **[L. 10]** or surface zones of the same material.

CFRP materials have a multilayer structure. They are characterized by heterogeneity, anisotropy, and the low thermal conductivity of the polymer matrix **[L. 11, 12]**. Carbon fibres, constituting the phase strengthening the CFRP composite structure, due to the relatively high strength and modulus of elasticity, have a significant impact on CFRP friction wear **[L. 13]**.

The wear intensity of composites based on carbon fibres largely depends on the cooperating element of the kinematic pair **[L. 14]**. Studies **[L. 15]** have shown that wear also depends on the architecture and orientation of the fibres relative to the direction of the friction force vector. Fibre orientation relative to the contact excitation vector also affects the nominal contact stress **[L. 16]**. The wear resistance of the outer layers of the laminate structure is of key importance. However, interlaminar strength is also important, depending mainly on the shear strength of the polymer matrix **[L. 17]**. In addition, carbon fibres have self-lubricating properties that reduce friction **[L. 4]**. It is believed that carbon fibres, whose thermal conductivity is greater than the polymer matrix, have a positive effect on tribological wear **[L. 16]**.

CFRP materials predicted for use in medical device joint connections were tested. It has been assumed that the use of an adhesive film (AF) layer in the structure of the tested composites will improve the ability to transfer operational loads – mainly from static and dynamic structural friction (contact oscillations). It was also assumed that the fibrous structures of the layers being deeper in relation to the contact surface are important. Generally, the CFRP consumption process is complex and only partially recognized **[L. 4]**. It increasingly concerns newly developed structures, including "adhesive film" (AF); therefore, initial research was performed and the research objectives were mainly cognitive. Contact strength and tribological parameters of produced laminates were determined, which will allow a comparison of their properties with other materials.

The research was carried out in cooperation with WIT-Composites as research and development work as part of the project entitled „Composite system for fast mechanical connections for the medical industry" co-financed by the European Regional Development Fund, within the framework of the Action 1.1: R&D projects of the enterprise of the Intelligent Development Operational Program 2014–2020.

MATERIAL AND RESEARCH METHOD

As part of the research, 4 materials for comparative tests were made. Test materials are intended for use in medical device components on frictional surfaces. Structures were designed to have the ability to dampen vibrations from structural friction while maintaining good strength, contact rigidity, and wear resistance. Two materials based on a prepreg under the trade name Kord Carbon (Fiberpreg GmbH) were used, i.e. twill weave and a surface weight of 200 g/m^2 with a fibre system of „0". In the first (material labelled „M1"), 4 layers of prepreg and one outer layer made of epoxy adhesive film (AF – Adhesive Film) by the manufacturer NTPT (North Thin Ply Technology sp.z o.o.) were used. The second material „M2" was based on the KC prepreg using a system of 3 layers of prepreg, then 1 layer of AF film and 1 outer layer of KC. In the other two materials, unidirectional carbon fibre (UD) structures were used, which were marked by the manufacturer (G. Angeloni S.R.L), as HS 15/130 DLN2, with a surface mass of 130 g/m^2, KC and AF NTPT.

Laminates were made in the following systems: 3 layers of UD HS 130 DLN2, 1 layer of KC and 1 outer layer of AF (material „M3"), and in a system with a layer of AF as the second and outer layer of KC in material „M4" (**Fig. 1**). Hardness and tribological tests were performed on sample surfaces from the AF layers side. It was assumed that the AF layer has a positive effect on the material's resistance to contact loads; however, it was necessary to determine whether the epoxy film (AF) could be used as the outer layer most exposed to contact service loads or whether a higher composite resistance would be obtained when using the Kord plastic layer Carbon reinforced with carbon fibres.

The indentation hardness tests were carried out according to the Oliver & Pharr (O&P) method **[L. 18]** with a Vickers indenter. The tests were performed on the Micro Combi Tester (Anton Paar). The measurements were carried out with a maximum load force of 3 N and a load / unload speed of 2000 mN/min. Pause at maximum load was 10 s.

In the hardness test, mechanical and elastic parameters of the tested surfaces were determined. The indentation hardness H_{IT} (1) was determined on the basis of the ratio of the highest normal force loading the indenter P_{max} to the indenter contact surface under maximum load A, according to the following formula **[L. 18]**:

$$H_{IT} = \frac{P_{max}}{A} \tag{1}$$

The Vickers conversion hardness was obtained based on the relationship (2) **[L. 18]**:

$$HV_{IT} \approx H_{IT} / 10{,}58 \tag{2}$$

To calculate the modulus of elasticity of the E_{IT} surface, stiffness was determined with the correlation (3) **[L. 18]**:

$$S = \frac{dP}{dh} = \beta \cdot (2/\sqrt{\pi}) \cdot E^{*} \cdot \sqrt{A} \tag{3}$$

The correlation $\frac{dP}{dh}$ was determined from the inductor force-displacement diagram (**Fig. 2**). In Equation (3), the parameter β, in the case of any symmetrical indenter, is set to 1, for the Vickers indenter, the corrected value of the parameter β = 1.0055 **[L. 18]**. Size A is a function of depth h_c (**Fig. 2**) and is dependent on (4), according to the following **[L. 19]**:

$$A = F\left(h_c\right) = 24{,}54 h_c^2 + C_1 h_c^1 + C_2 h_c^{1/2} + C_3 h_c^{1/4} + C_4 h_c^{1/8} + \ldots + C_n h_c^{1/2n} \tag{4}$$

In Equation (4), calculations are based on the C_n constant, which expresses the indenter's geometry. The method of determining the C_n constant is described in **[L. 20]**. In the calculations of stiffness (S), there is a module of the tested surface layer marked with the letter E, this quantity is defined by Equation (5).

$$\frac{1}{E^{*}} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i} \tag{5}$$

In Equation (5), the value E^* indicates the reduced modulus of elasticity and ν indicates the Poisson's number of the sample. The size E^* includes elastic deformation in the tested sample as well as the indenter, and this issue is broadly described in the paper **[L. 18]**. However, E_i and ν_i, respectively, refer to the indenter. The relationship (5) is a general relation that applies to each symmetrical indenter, not limited to a specific geometry, e.g., a cone or pyramid. Equation (5) was originally introduced for the case of elastic contact; however, it also proved to be appropriate for the case of elastic-plastic contact **[L. 18]**.

Based on the value of stiffness S estimated from Equation (3) and the penetration path of the indenter corresponding to the elastic deformations of the test surface – h_c, the E_{IT} parameter was calculated based on the following formula (6):

$$E_{IT} = \frac{\sqrt{\pi} S}{2\beta\sqrt{A \cdot hc}} \tag{6}$$

The wear resistance tests were carried out on the Anton Paar tribotester **(Fig. 3).** The tests were performed using the ball-on-disc method in technically dry friction. Rectangular wall samples with minimum dimensions were used in the tests, i.e. 18 mm x 18 mm x 1 mm (length x width x thickness). Six mm diameter balls were used as cooperative elements.

In **[L. 4],** it was stated that in the case of CFRP materials, in which the matrix is an epoxy resin, friction in the CFRP-steel combination has a lower friction coefficient than in the CFRP-alumina combination. The use of a counter-sample, which was assumed to work together in a kinematic pair, influenced the generation of greater frictional resistance, which translates into more difficult working conditions. This approach was aimed at revealing the impact of the AF layer on the friction and wear process. An additional factor determining the use of the counter-sample with Al_2O_3 was the possibility of cooperation with CFRP in anticipated biomedical

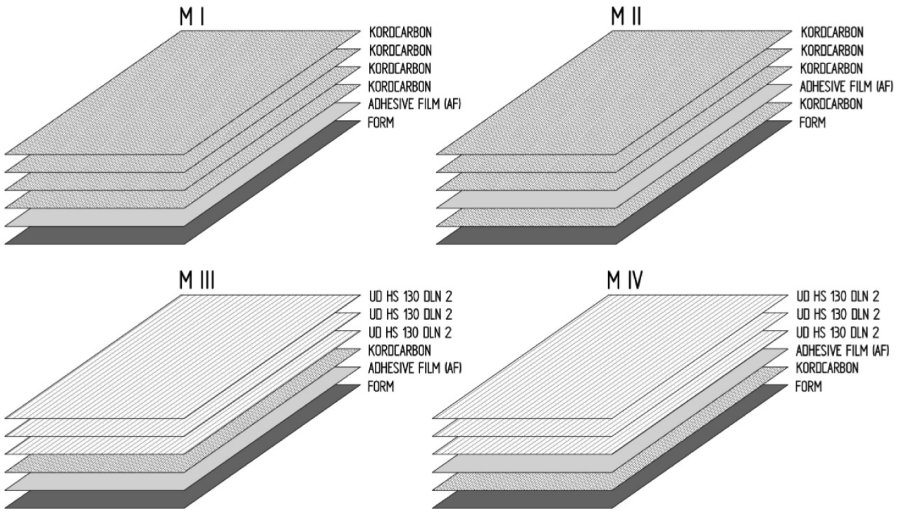

Fig. 1. Diagram of the layers in the manufacturing process of the materials used in the tests
Rys. 1. Schemat układu warstw w procesie wytwarzania materiałów wykorzystanych w badaniach

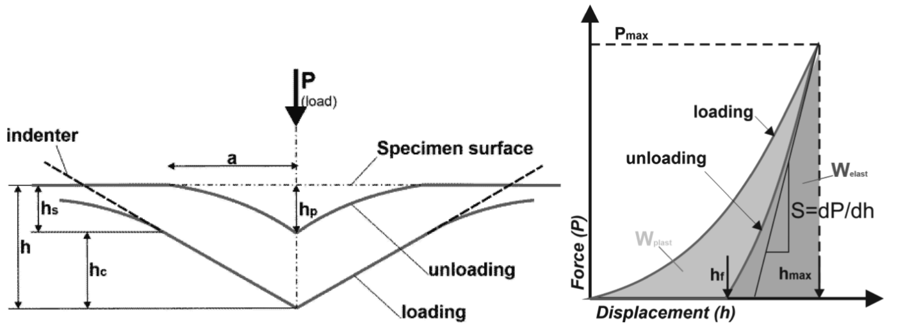

Fig. 2. Diagram of indenter contact with the surface being tested and theoretical force-displacement characteristics with important test parameters marked [L. 21]
Rys. 2. Schemat kontaktu wgłębnika z badaną powierzchnią oraz charakterystyka teoretyczna siła-przemieszczenie z zaznaczonymi ważnymi parametrami próby **[L. 21]**

applications. Therefore, balls made of alumina (Al_2O_3) with a hardness of approx. 2000 HV (CSM Instruments S.A. Switzerland). The tests were carried out under a 5 N load. A friction path radius of 5 mm was assumed. The total friction path in the test was 2000 m. The change in friction coefficient as a function of friction cycles of the ball with the sample was recorded. The friction cycle corresponded to one complete revolution of the CFRP sample. Often weight loss **[L. 22]** or the characteristics of the wear surface (furrow, trace of friction) on the sample surface are assumed to be the measure of wear as a result of the kinematic frictional contact of the sample and counter-sample. The subject tests determined volumetric wear as the volume of the furrow formed on the friction surfaces of CFRP samples.

The wear profile geometry was tested using a Veeco Dektak 150 contact veilometer (Veeco Instruments). The contact profilometer allows measurements of a 2D profile and 3D surface with a resolution of 0.01 µm in the vertical axis. The cross-sectional area of the wear trace was determined in 10 places along the furrow's circumference. Volume consumption was determined as the product of the average value of the cross-sectional area of the wear profile (furrow) and the circumference of the circle formed by the furrow. The surface roughness of the samples was determined on an optical profilometer (Bruker) on a surface measuring 5 mm x 5 mm and 1 mm x 1 mm in the centre of the sample. The arithmetic mean value of the profile deviations from the mean line – *Ra*, the height of the highest roughness profile elevation – *Rp*, the square mean of the ordinates of the roughness profile – *Rq*, the total height of the roughness profile – *Rt,* and the depth of the lowest recess of the roughness profile – *Rv*. The tests were carried out in accordance with the PN-EN ISO 4287: 1999 standard **[L. 23]**.

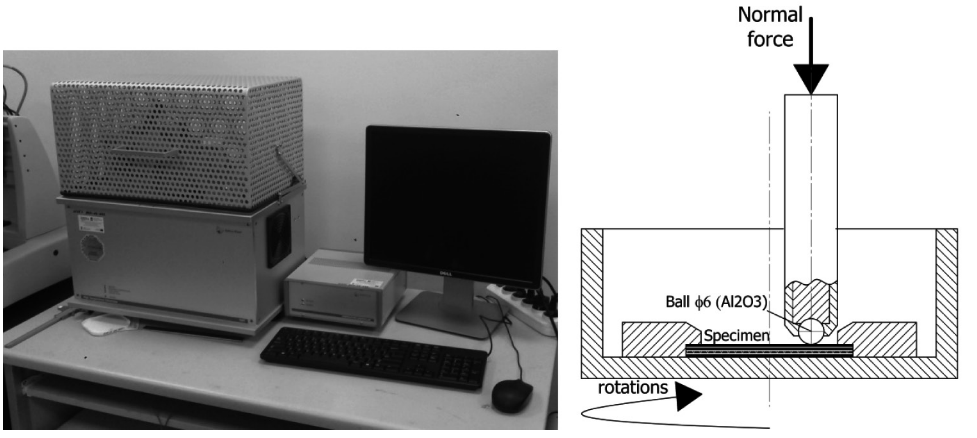

Fig. 3. Anton Paar tribometer and ball-disk carbon composite test scheme
Rys. 3. Tribometr Anton Paar i schemat badania metodą kula–tarcza kompozytu węglowego

RESEARCH RESULTS AND DISCUSSION

The results of indentation (universal) hardness tests are presented in **Table 1**, where the mean value, standard deviation, and median values of measured values are given. The material marked as M1 had the highest hardness among the tested materials. The lowest hardness was demonstrated for M4 material.

Elastic properties are one of the basic features necessary in the design process of mechanical systems. Elastic properties are described by the E_{IT} surface elasticity module. For the tested materials, the highest modulus of surface elasticity was found in materials M1 and M2.

In some works, researchers use the ratio of hardness to elasticity (H/E) **[L. 24]**. This factor describes the ability to the highest possible elastic deformation (low modulus) and the ability to the least permanent deformation (high hardness). For the tested materials, the values of this relationship were as follows: "M1" – $61.94 \cdot 10^{-3}$; "M2" – $59.71 \cdot 10^{-3}$; "M3" – $66.72 \cdot 10^{-3}$; and "M4" – $50.27 \cdot 10^{-3}$. The most favourable value of this relationship was obtained for M3 and M1 materials with an outer layer of AF.

Table 2 presents sample roughness parameters. The sample roughness expressed by the *Ra* parameter was in the range of 0.216–0.451 µm. Material 4 had the highest roughness, while the total height of the roughness profile (*Rt)* of the M2 material was the highest. **Figure 4** presents selected profile profiles of samples, and differences in sample roughness affect the real contact surface in frictional contact. Higher average values of *Ra* and *Rt* parameters were shown for materials in which the AF layer was used under the KordCarbon layer.

Table 3 summarizes the results of wear tests on the surface of carbon composites. The best materials for tribological wear were M3 and M1. M4 material had the worst resistance.

Table 1. Descriptive statistics of indentation hardness and modulus of elasticity of the surface of tested materials
Tabela 1. Statystyki opisowe twardości indentacyjnej i moduł sprężystości powierzchni badanych materiałów

Material		M1	M2	M3	M4
H_{IT}	Mean	498.611	456.830	301.643	238.305
[MPa]	Std.dev.	84.627	79.512	140.595	92.932
	Median	461.236	445.801	358.447	230.593
H_{VIT}	Mean	47.062	43.118	28.471	22.493
[Vickers]	Std.dev.	7.988	7.505	13.270	8.771
	Median	43.534	42.077	33.832	21.765
E_{IT}	Mean	8.050	7.646	4.516	4.743
[GPa]	Std.dev.	0.716	0.568	1.715	2.356
	Median	8.148	7.569	4.161	5.705

Table 2. Roughness test results (average values)
Tabela 2. Wyniki badań chropowatości

Materials	Roughness parameters				
	Ra [µm]	Rp [µm]	Rq [µm]	Rt [µm]	Rv [µm]
M1	0.216	25.630	0.286	50.765	-25.130
M2	0.401	30.522	0.677	71.940	-41.418
M3	0.383	29.383	0.548	53.835	-24.452
M4	0.451	29.352	0.676	65.734	-36.382

Table 3. Tribological wear in geometric terms of tested materials
Tabela 3. Zużycie tribologiczne w ujęciu geometrycznym badanych materiałów

Materials	N	Distance [m]	Load [N]	Average wear in cross section [mm^2]	Average volumetric wear [mm^3]
M1	4	2000	5	0.020	0.629
M2	4	2000	5	0.024	0.759
M3	4	2000	5	0.017	0.522
M4	4	2000	5	0.029	0.922

Table 4 summarizes the values of friction coefficients. The minimum, maximum, average, and standard deviation of friction coefficients are presented. At a constant normal load value in the friction tests, the coefficient of friction varied. The average values of friction coefficients ranged from 0.149 (M1_pr4) to 0.438 (M4_pr3).

Figure 5 presents representative cross-sections of the signs of wear of the tested materials. At the bottom of the furrows of the M4 and M3 material samples, clear unevenness can be seen, which can be associated with adhesive wear and fibre damage.

The presented study of indentation hardness and tribological wear in the conditions of sliding friction of

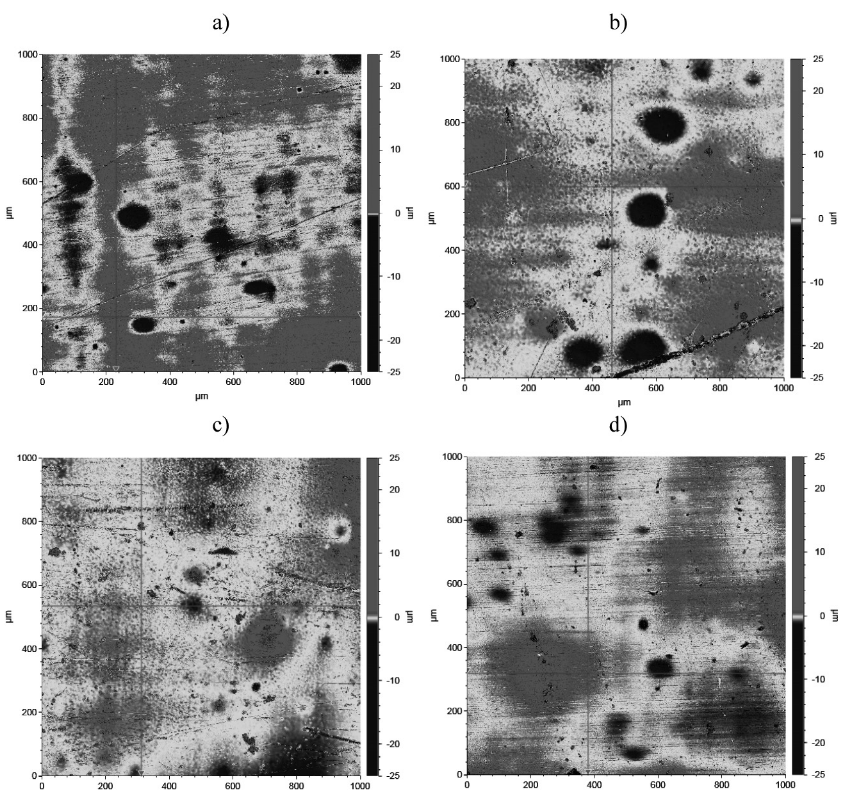

Fig. 4. Summary of selected surface profile profiles of the tested samples: a) M1 material, b) M2 material, c) M3 material, d) M4 material
Rys. 4. Wybrane struktury geometryczne powierzchni badanych próbek: a) materiał M1, b) materiał M2, c) materiał M3, d) materiał M4

new CFRP composites led to obtaining test results that indicate that the values of friction coefficients depend on the degree of wear and the surface wear mechanisms of the samples tested. For the M1 material, the lowest average friction coefficient values were recorded, which is reflected in the wear mechanism (**Fig. 6**). The bottom surface of the furrows in M1 and M2 composites has less roughness and is less frayed than in M3 and M4 materials.

Table 4. Statistical values of the friction coefficient

Tabela 4. Wartości statystyczne współczynnika tarcia

Material_specimen	Min.	Max.	Mean	Std.Dev.
M1_pr1	0.019	0.527	0.330	0.166
M1_pr2	0.100	0.565	0.373	0.047
M1_pr3	0.015	0.471	0.240	0.119
M1_pr4	0.018	0.544	0.149	0.081
M2_pr1	0.029	0.521	0.438	0.049
M2_pr2	0.063	0.537	0.296	0.108
M2_pr3	0.006	0.535	0.424	0.103
M2_pr4	0.045	0.466	0.292	0.092
M3_pr1	0.027	0.427	0.313	0.057
M3_pr2	0.000	0.467	0.378	0.049
M3_pr3	0.000	0.472	0.376	0.056
M3_pr4	0.000	0.483	0.372	0.039
M4_pr1	0.030	0.479	0.373	0.039
M4_pr2	0.014	0.498	0.383	0.076
M4_pr3	0.018	0.504	0.406	0.037
M4_pr4	0.003	0.510	0.385	0.059

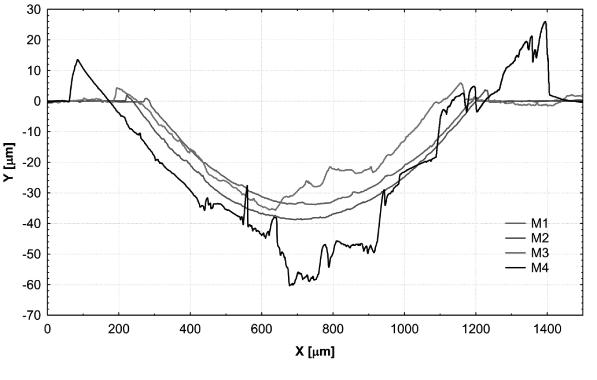

Fig. 5. Representative cross-sections of signs of wear on the surface of material samples: M1("1"), M2("2"), M3("3"), M4("4")

Rys. 5. Przekroje śladów zużycia na powierzchni próbek materiałowych (materiały: M1, M2, M3, M4)

From the studies of tribological wear, it can be stated that the structure of the KC prepreg promotes the formation of a "tribo-layer" **[L. 4]** between fibres involved in friction contact. The likely cause is the fine products of carbon fibre wear in the form of dust forming a graphite film **[L. 16]**. This film is created not only on the surface of the CFRP composite, but also applied to the surface of the mating element. The KC structure is not conducive to adhesive damage. The outer layer AF was used in M1 and M3 materials, and the lowest surface wear was observed for these materials (average volume wear was 0.5216 mm^3 and 0.6296 mm^3, respectively). These materials significantly differed in terms of hardness, which was probably determined by the prepreg used.

CONCLUSIONS

The obtained research results allowed us to formulate the following conclusions:

Higher hardness was demonstrated for samples containing the KC prepreg (M1 and M2). However, in the groups classified according to the prepreg (KC and UD), samples with the outer layer AF (M1 and M3) were harder. The results of indentation hardness tests confirmed the beneficial effect of the AF layer on the surface contact strength.

In the presented tests, the ratio of hardness to elasticity (H/E) depends to a large extent on the location of the AF layer in the structure of the tested composites. H/E values are clearly better for composites with an outer AF layer.

The use of an additional polymer layer has achieved the intended effect, both in terms of hardness and abrasion resistance.

The research results obtained are encouraging, and it seems reasonable to continue the work already begun. In subsequent stages of work, we hope to perform friction tests in reversing motion and to take into account the impact of a humid environment and elevated temperature. It is also planned to make composites containing a layer of damping impulse forced made of elastomeric material.

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