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COMPOSITES THEORY AND PRACTICE

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THE INFLUENCE OF SOLID LUBRICANTS ON MECHANICAL AND TRIBOLOGICAL PROPERTIES OF POLYMER COMPOSITES

The article presents the results of studies on the mechanical and tribological properties of polymer composites intended for the regeneration of sliding machine elements. Chemically cured epoxy resin Epidian 5 constituted the composites' polymer warp. Fe powder with a define content of grains and obtained by means of reduction was used as a basic filler, whereas solid lubricants of a layered structure - graphite and molybdenum disulfide - were used as the sliding additives, and polyaramid fibres in the form of pulp - as a fibrous filler. Three series of composites with different qualitative and quantitative compositions were prepared with the addition of lubricating agents in the amount varying from 10 to 30 parts by weight in a single or combined system. The composites were crosslinked with aliphatic polyamine (triethylenetetramine), and the crosslinking process was conducted at room temperature. Once the composites were cured, their mechanical properties (i.e. resistance to compression and tear, and the module and deformation at the time of bending) were checked with the use of an Instron machine for endurance tests. The tribological characteristics of the composites in dry friction conditions were determined with a T-05 roll-block type tribotester, in which a roller with a composite layer deposited on its surface played the role of a model tribological system. A steel block, on the other hand, played the role of a counter sample. The friction and wear tests were conducted at the constant speed of slide of 0.1 m/s and changeable pressures of 0.9 and 1.5 MPa. Based on the obtained friction and wear properties and the results of investigations in which scanning electron microscopy (SEM) was applied, it was concluded that the best results are displayed when solid lubricants of a layered structure are jointly applied.

Keywords: **polymer composite, solid lubricants, dry friction, friction coefficient, mechanical properties**

WPŁYW SMARÓW STAŁYCH NA WŁAŚCIWOŚCI MECHANICZNE I TRIBOLOGICZNE KOMPOZYTÓW POLIMEROWYCH

Przedstawiono wyniki badań właściwości wytrzymałościowych i charakterystyki tribologiczne kompozytów polimerowych przeznaczonych do regeneracji ślizgowych elementów maszyn. Polimerową matrycą kompozytów jest chemoutwardzalna żywica epoksydowa Epidian 5. Podstawowym napełniaczem jest proszek żelaza (Fe) otrzymywany metodą redukcji o określonym składzie granulometrycznym. Jako dodatki ślizgowe zastosowano smary stałe o budowie warstwowej: grafit i disiarczek molibdenu, a napełniaczem włóknistym są organiczne włókna poliaramidowe w postaci pulpy. Sporządzono trzy serie kompozytów o różnych składach jakościowych i ilościowych, zawierających dodatki smarne od 10 do 30 cz. wag. w układzie pojedynczym oraz w układzie łączonym. Do sieciowania kompozytów zastosowano poliaminę alifatyczną (trietylenotetraaminę). Proces sieciowania prowadzono w temperaturze pokojowej. Po utwardzeniu kompozytów zbadano ich właściwości mechaniczne (wytrzymałość na ściskanie i odrywanie oraz moduł i odkształcenie przy zginaniu) z wykorzystaniem maszyny wytrzymałościowej Instron. Charakterystyki tribologiczne kompozytów w warunkach tarcia technicznie suchego wyznaczono za pomocą testera T-05 typu rolka klocek w styku rozłożonym. Modelowy węzeł tarcia stanowiła stalowa rolka z nałożoną i obrobioną na wymagany wymiar warstwą kompozytów, natomiast przeciwpróbkę stanowił stalowy klocek o geometrii styku w kształcie powierzchni łukowej o polu 1 cm^2. Testy tarciowo-zużyciowe prowadzono przy stałej prędkości poślizgu 0,1 m/s i zmiennych naciskach 0,9 i 1,5 MPa. Na podstawie uzyskanych charakterystyk tarciowo-zużyciowych oraz wyników badań ze skaningowej mikroskopii elektronowej (SEM) i profilografii stwierdzono, że najlepsze rezultaty daje łączne zastosowanie smarów stałych o budowie warstwowej.

Słowa kluczowe: **kompozyt polimerowy, smary stałe, tarcie suche, współczynnik tarcia, właściwości mechaniczne**

INTRODUCTION

Contemporary devices and industrial machines are characterised by advanced design and structure, which enables the execution of complex manufacturing processes. However, their moveable elements are still subject to typical wear and tear, and therefore require periodic inspections and repairs. The structural complexity of machines boosts the costs of repairs arising from the need to replace the used parts. In many cases, the state in which the machine elements are, allows their regeneration without the need for their replacement, which greatly reduces their maintenance costs. At the time of repairs, it is possible to use modern composite materials which ensure suitable operating parameters of the regenerated item.

An interesting type of a structural material which is more frequently used for the manufacture or regeneration of machine sliding elements are powder composites (metal-polymer) based on chemically cured resins, which in numerous cases can replace (not in financial or ecological terms) traditional methods of recovery or galvanic coating. They are used, among others, for the regeneration of averagely loaded, poorly lubricated slide bearings and guiding systems of machine tools. Under certain operating conditions (pressure, slide speed), the tribological system in which the pin of the bearing coated with a composite cooperates with a metal bearing bushing, is not inferior to classic coupling between a steel pin and a bearing bushing made of bearing alloy. A major benefit from the point of view of the repair and regeneration of machine elements that stems from the application of these composites is connected with the fact that the curing time can be freely shaped, and after crosslinking, these components can be subject to typical mechanical treatment. The ease and the short time in which the coatings are formed is another significant factor for the development of polymer-based composite technologies [1-8].

The key characteristics of composites that determine their use for the regeneration of sliding machine elements include suitable mechanical properties, thermal resistance and conductivity, and tribological properties. The improvement of these properties can be obtained mostly by modification of the matrix and/or the use of new and improved fillers. A polymer composite can be used as a bearing material in a tribological system under the following conditions: it is necessary to decrease the friction coefficient and increase the wear resistance through the use of lubricants such as graphite or molybdenum disulfide, as well as increase the heat abstraction from the material of the surface layer, which can be effectively achieved thanks to the use of powder metallic fillers such as iron, copper, or bronze. Works on modification of the tribological properties of composites show, among others, that composites with solid lubricants of a layered structure exhibit better tribological properties than conventional plastics [9-15].

The aim of this study was to investigate the effects solid lubricants with anisotropic cohesion i.e. graphite and molybdenum disulfide, which are used in single or combined systems, have on the mechanical and tribological properties of polymer composites.

SCOPE OF STUDY AND RESEARCH METHODS

The study was focused on metal-polymer composites with restorative properties that were based on chemically cured epoxy resin Epidian 5 (by Sarzyna Chemical Plant). The NC 100.24 Fe powder (by Hoegenes) obtained by means of a reduction method, and with a set chemical composition and grain size, was used as a metallic filler in the amount of 250 parts by weight. A fibrous filler was also introduced (i.e. organic polyaramid fibres in the form of a pulp known under the commercial name of Kevlar 1F651 (by Du Pont) in the amount of 1 part by weight. As lubricating additives, M15-99 graphite and molybdenum disulfide were used. The content of all the fillers is presented in proportions used for 100 part by weight epoxy resin.

Table 1 presents the qualitative and quantitative composition of the tested composite materials.

TABLE 1. **Weight proportion of low friction additives in basic composition of composites**

TABELA 1. **Udział wagowy dodatków niskotarciowych w podstawowym składzie kompozytów**

Type of additive	Weight proportion [PBW]	Composite symbol
Graphite	10	G - 10
	20	G - 20
	30	G - 30
Molybdenum disulfide	10	M - 10
	20	M - 20
	30	M - 30
Combined additives Graphite + Molybdenum disulfide in the proportion of 1:1	10	GM - 10
	20	GM - 20
	30	GM - 30

The way the composites were manufactured consisted in thorough homogenization of the powdered metal fillers, lubricant additives, and aramid fibres with a liquid polymer matrix in a low speed laboratory zeta mixer. The design of the mixer stimulates the occurrence of great shear forces, which ensures adequate dispersion of the fillers in the polymer matrix. The homogenization process was carried out at room temperature, at the speed of 100 rpm. The composites were crosslinked with aliphatic polyamine (triethylenetetramine) in a stoichiometric amount with reference to the epoxy matrix of the composite. The curing process was also conducted at room temperature.

CHARACTERISTICS OF COMPONENTS

The Fe powder with the grain size presented in Figure 1 was used as the powder metallic filler.

The presented properties of the Fe powder show that grains with the dimensions of 100÷125 μm are the most common, whereas grains with a size below 100 μm and over 125 μm constitute respectively 30 and 15% of the total. Certain dispersion in the dimensions of the grains positively influences the properties of a crosslinked composite. The presence of bigger grains increases thermal conductivity, which is particularly important in the case of materials used on sliding machine elements. Smaller filler grains on the other hand prevent sedimentation, spoiling the homogeneity of the composition and the properties of the composite. The SEM image presented in Figure 2 shows that the particles of the Fe powder are irregular in shape and are characterised by large surface development, which is beneficial for the size of mechanical adhesion between the polymer matrix and the filler.

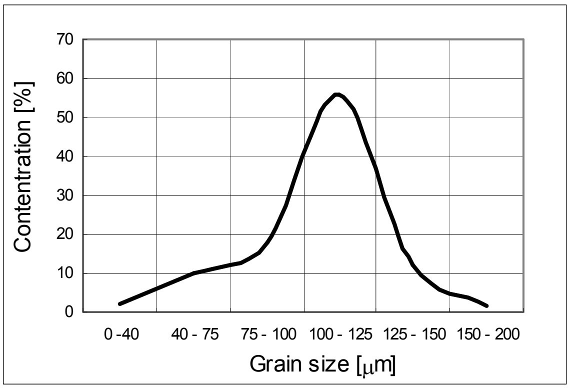

Fig. 1. Grain size of used metallic filler

Rys. 1. Rozkład granulometryczny napełniacza metalicznego

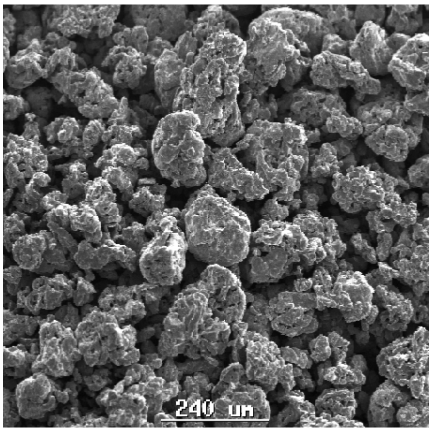

Fig. 2. Microscopic image (SEM) of Fe powder

Rys. 2. Obraz mikroskopowy (SEM) proszku żelaza (Fe)

When selecting lubricant additives and fibrous fillers, emphasis was put on the technological conditions in which composites are manufactured and the influence they have on the mechanical and tribological properties. For this reason, as additives reducing friction and wear, solid lubricants with anisotropic cohesion, i.e. graphite and molybdenum disulfide, were used.

Figures 3-5 present the images of tribological additives and polyaramid fibres which were taken with a scanning electron microscope (SEM).

Graphite is an allotropic variation of carbon with a layered structure, which is characterised by well-defined slide and cleavage planes running along the individual layers. The layered structure of the crystal lattice connects atoms arranged in the planar layer by means of strong covalent bonds. The interactions between the planes are of an electrostatic type, which means that they are much weaker.

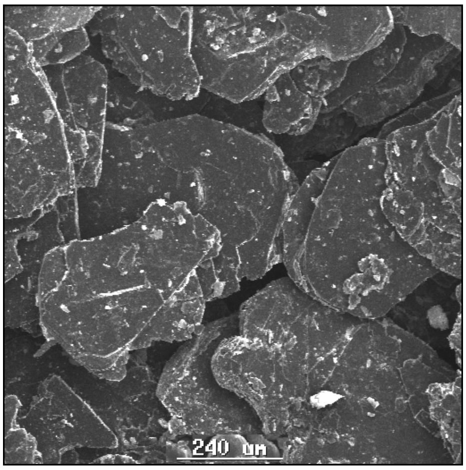

Fig. 3. Graphite microscopic image

Rys. 3. Obraz mikroskopowy grafitu

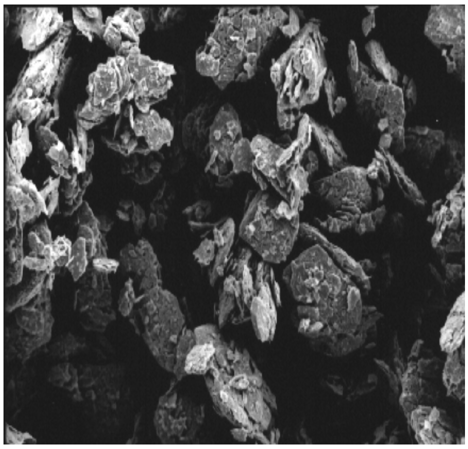

Fig. 4. Molybdenum disulfide microscopic image

Rys. 4. Obraz mikroskopowy disiarczku molibdenu

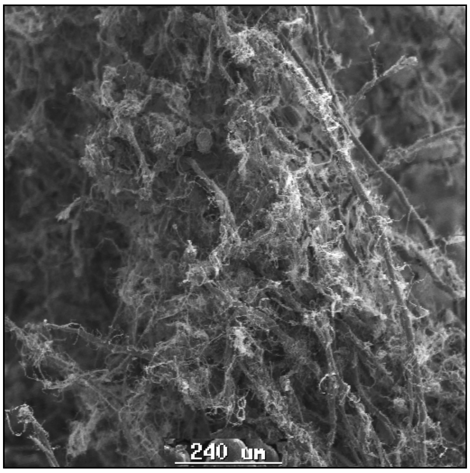

Fig. 5. Polyaramid fibre microscopic image

Rys. 5. Obraz mikroskopowy włókien poliaramidowych

The average grain size of 10 μm improves the lubricity. The crystals of molybdenum disulfide, similarly to graphite crystals, also have a layered structure and are easily influenced by shear forces parallel to these layers. At the same time, they are characterised by high adhesion to metal, which makes them a particularly effective lubricant. The average grain size of the additive is about 3÷4 μm, and the maximum - about 15 μm. The fibrous filler used was an organic polyaramid fibre with the length of ca. 2 mm, which gave the composites the desired thixotropy and improved their mechanical and friction-wear properties. The fibrous filler was fed in the form of pulp (Fig. 5).

COMPOSITE TEST METHODS

Important parameters of composites intended for the surface layers of lathe runners include e.g. high adhesion to the substrate material, and high resistance to amorphous deformation. Therefore, the scope of the study on the assessment of the influence of additives on the endurance properties of metal-polymer composites was particularly concentrated on, inter alia, the adhesion to steel as measured indirectly by means of measuring compression strength and the resistance to tear (which were tested in accordance with the PN EN ISO 604:2004 norm). The module and the deformation at the time of bending were also examined, and these parameters were determined according to the PN-EN ISO 178:2011 norm using an Instron testing machine.

METHODOLOGY OF TRIBOLOGICAL TESTS

The tribological characteristics of the composites were determined with the T-05 roll-block type tribotester (Fig. 6a), in which the roller with the composite layer deposited on it surface, and block made of hardened and tempered carbon steel (Fig. 6b) played the role of a model tribological system representing a pin-bushing pair in the slide bearing.

a)

b)

Fig. 6. Roll-on-block tribological tester T-05 (a), model tribological system (b)

Rys. 6. Tester tribologiczny T-05 typu rolka-klocek (a), modelowy węzła tarcia (b)

The friction and wear tests were carried out in the following conditions: roller with different types of composites, counter sample - carbon steel, dry friction, constant sliding motion, constant speed of 0.1 m/s, constant pressure of 0.9 and 1.5 MPa, sliding distance 2400 m and distributed contact. The tests were focused on measurement of the frictional force, the temperature of the block, and the change in weight as the difference in the measurements of the weight of the sample before and after the test.

METHODOLOGY OF MICROSCOPIC TESTS

The S 2460N SEM microscope by Hitachi, enabling tests in low vacuum, and the energy-dispersive (EDS) detector by Noran with the Norvar window, and SiLi crystal with the resolution of 133 eV that was electronically coupled with the microscope, were used to investigate the surface conditions of the composites. The test parameters were as follows: accelerating voltage of 15 kV, high vacuum, SE detector, 70x zoom. The pictures were taken after gold was deposited on the surface of the composite so as to enable observations of the surface in high vacuum and with use of the SE detector.

METHODOLOGY OF TESTS ON THE GEOMETRIC STRUCTURE OF THE SURFACE

The Form Talysurf PGI 830 profilograph by Taylor Hobson with the system enabling the roughness, waviness and shape of the surface to be measured with a contact method was used particularly for the analysis and recording of the roughness and topography of the surface of test elements, and the recording offriction. A laser sensor with a 830 PGI head with nose radius R = 2 μm, and tool nose angle of 90° moving along the surface of the elements was used in the test. The Ultra and Platinum TalyMap software enabling both 2D (profiles) and 3D (topography) analyses was used as well.

RESULTS OF TESTS FOCUSED ON MECHANICAL PROPERTIES OF COMPOSITES

The study on the mechanical properties of composites allowed their initial characterisation and constituted an important material selection criterion. The results of the mechanical property tests, as shown in Figures 7-10, are the arithmetic mean of at least five measurements of the measured parameters.

The layered solid lubricants, which are the main modifiers of the tribological properties of metal--polymer composites and differently affect their mechanical properties, were used in the study. Based on the analysis of the obtained results of tests of mechanical properties, the authors concluded that the composites containing graphite have a much lower compressive strength and resistance to tear than composites containing molybdenum disulfide and composites involving combined solid lubricants of these two types.

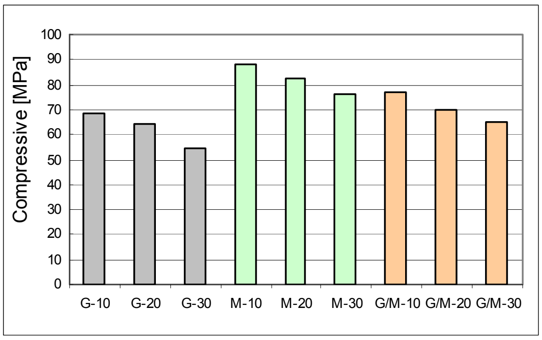

Fig. 7. Compressive strength of polymer composites

Rys. 7. Wytrzymałość na ściskanie kompozytów polimerowych

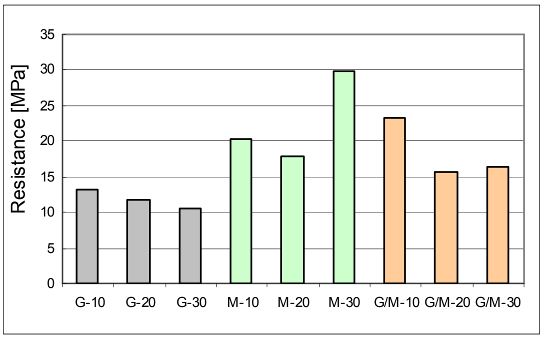

Fig. 8. Resistance to tear of polymer composites

Rys. 8. Wytrzymałość na odrywanie kompozytów polimerowych

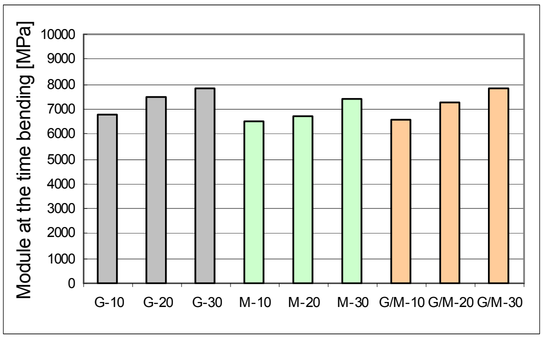

Fig. 9. Module at time of polymer composites bending

Rys. 9. Moduł przy zginaniu kompozytów polimerowych

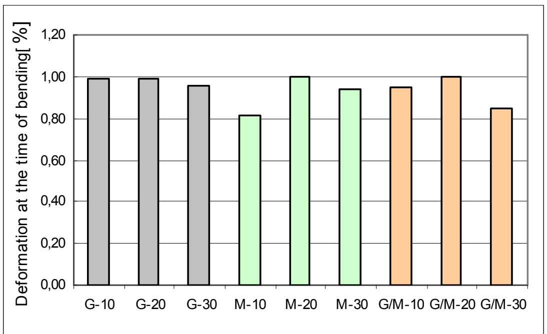

Fig. 10. Deformation at time of polymer composites bending

Rys. 10. Odkształcenie przy zginaniu kompozytów polimerowych

The lower specific gravity of the graphite-based modifier, compared to the molybdenum disulfide-based one, significantly increased the volume of the modifier in a crosslinked composite, which may have adversely affected the wettability of the filler particles, and thereby caused some structural defects manifested by the deterioration of its mechanical properties. Compressive strength and adhesion to a metal substrate, measured indirectly by means of the measurement of the resistance to tear for all composites, except for the M-30 composite, decreased with an increasing mass fraction of solid lubricants in the total mass of the composite. High adhesion to the substrate of composites containing molybdenum disulfide, both in a single and binary system, may have stemmed from the polarity of molybdenum disulfide and the presence of additional electrostatic interactions between the matrix and the fillers, as well as the composite and the substrate material.

Different results were, on the other hand, obtained in the case of the determination of the module at the time of bending. The increased content of solid lubricants caused a nearly proportional increase in this parameter. The best results were obtained for the composites containing graphite. The influence of solid lubricants on the deformation at the time of tear was varied and therefore it is difficult to speak of a specific connection. The smallest deformation could be observed for composites that contain molybdenum disulfide in a single or combined system.

TRIBOLOGICAL PROPERTIES OF COMPOSITES

What decides on the quality of interactions between materials in a tribological system, apart from the level of wear and the value of friction coefficient, is the persistence over time of these factors. Figures 11-13 illustrate the examples of changes in the friction coefficient and the temperature of the tribological system.

The recorded changes in the friction coefficient and temperature of the composite*steel tribological system exhibit a monotonic course in the entire sliding distance, which indicates stable friction cooperation in the node between the materials, despite the absence of external lubrication. Figures 14 and 15 show the influence of layered solid lubricants on the friction coefficient in the rotation of the roll with a layer of a metal-polymer composite cooperating with a steel counter sample (block) at pressures of 0.9 and 1.5 MPa. Based on the obtained results, it can be stated that the highest possible friction coefficient is exhibited, in terms of cooperation with steel, by the composites containing molybdenum disulfide. In addition, an increase in the weight fraction of this modifier of tribological properties of the composite boosts the resistance to motion. A visibly lower friction coefficient was obtained for composites containing graphite and its combined additives (graphite and molybdenum disulfide). Increasing their weight fraction decreased the friction coefficient. The lowest friction coefficient was exhibited by the composites containing respectively 20 and 30 parts by weight of the combined lubricant additives. An important advantage of the tested composite materials is the small dependence of the friction coefficient on unit pressures.

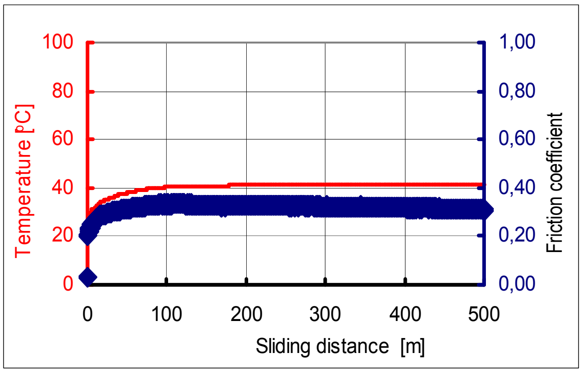

Fig. 11. Friction coefficient and temperature of tribological system for composite G-20 - steel (p = 0.9 MPa)

Rys. 11. Przykładowy przebieg zmian współczynnika tarcia i temperatury węzła dla skojarzenia kompozyt G-20 - stal

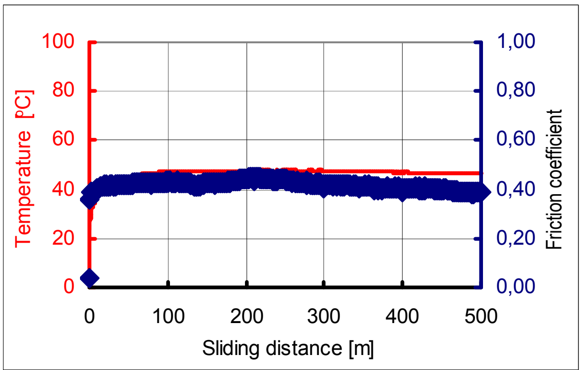

Fig. 12. Friction coefficient and temperature of tribological system for composite M-20 - steel (p = 0.9 MPa)

Rys. 12. Przykładowy przebieg zmian współczynnika tarcia i temperatury węzła dla skojarzenia kompozyt M-20 - stal (p = 0.9 MPa)

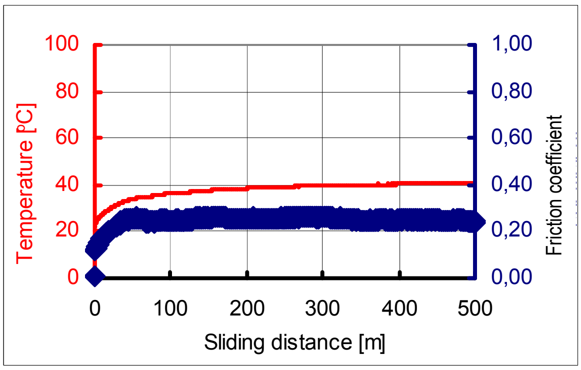

Fig. 13. Friction coefficient and temperature of tribological system for composite GM-20 - steel (p = 0.9 MPa)

Rys. 13. Przykładowy przebieg zmian współczynnika tarcia i temperatury węzła dla skojarzenia kompozyt GM-20 - stal (p = 0,9 MPa)

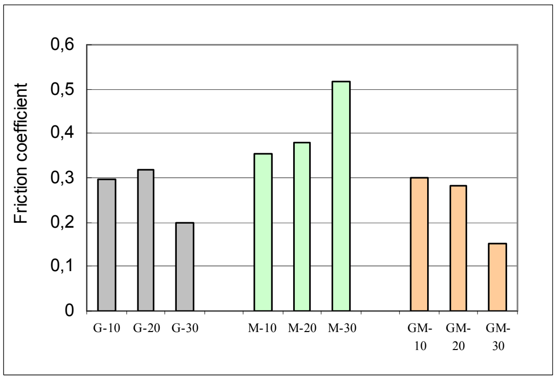

Fig. 14. Average friction coefficient of tribological system composite-steel (p = 0.9 MPa, v = 0.1 m/s)

Rys. 14. Średni współczynnik tarcia skojarzenia kompozyty - stal (p = 0,9 MPa, v = 0,1 m/s)

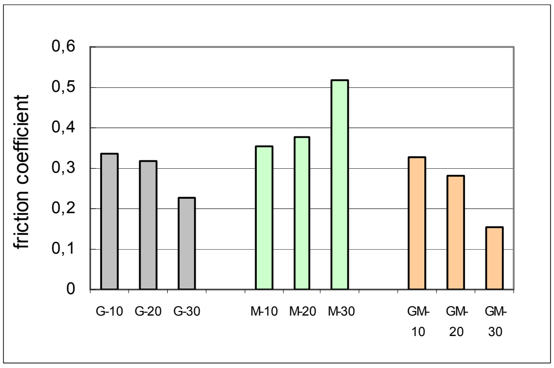

Fig. 15. Average friction coefficient of tribological system composite-steel (p = 1.5 MPa, v = 0.1m/s)

Rys. 15. Średni współczynnik tarcia skojarzenia kompozyty - stal (p = 1,5 MPa, v = 0,1 m/s)

The analysis of the data presented in Figures 16 and 17 facilitates the conclusion that, as in the case of the friction coefficient, the composites containing molybdenum disulfide are characterised by the highest level of wear. Both the increase in the resistance to motion and the weight proportion of the additive of this type cause accelerated wear of the composite material. The greatest resistance to wear can be observed when solid lubricants of anisotropic cohesion_are combined in a metal-polymer composite.

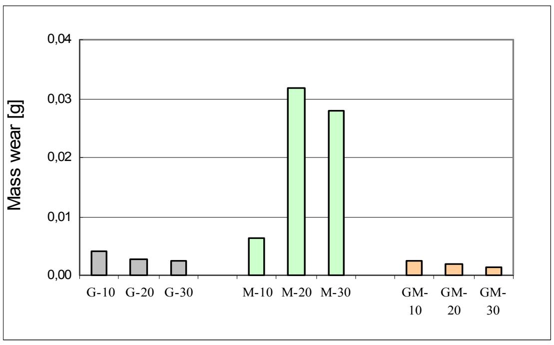

Fig. 16. Mass wear of rolls with composite layer (p = 0.9 MPa, v = 0.1 m/s)

Rys. 16. Zużycie wagowe rolek z warstwą kompozytów (p = 0,9 MPa, v = 0,1 m/s)

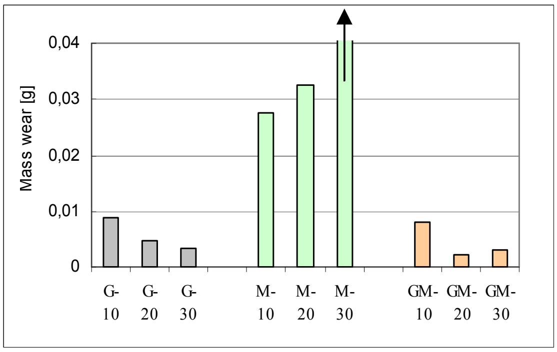

Fig. 17. Mass wear of rolls with composite layer (p = 1.5 MPa, v = 0.1 m/s)

Rys. 17. Zużycie wagowe rolek z warstwą kompozytów (p = 1,5 MPa, v = 0,1 m/s)

The greater wear of the composite containing molybdenum disulfide can result from the key importance of the adhesion process (according to the adsorption theory of solid lubricant friction (apart from structural theory)), that is the ability to adhere to the surface of the steel counter sample, as in the case of the tests conducted by the authors. As for the composites containing molybdenum disulfide, adhesion during friction between the crystals of the modifier and the steel counter sample can be greater than the adhesion between the components of the composite. The increased adhesion of the composite samples with molybdenum disulfide also stems from the polar structure of this additive and the electrostatic interactions between the surfaces that are in contact. As a result, increased wear of the composites material can be observed. The use of solid lubricants in a binary system can reduce the adhesion force of the composite, without elimination of the slide between the individual layers of the lubricant, and positively influence its internal cohesion, and therefore reduce the wear of the composite material during friction.

Summarizing, it can be stated that the interactions between graphite and molybdenum disulfide that are jointly introduced to the structure of the composite, lead to the creation of a surface layer that effectively separates the initial layers of the composite and steel, which increases their tribological properties.

MICROSTRUCTURE OF THE COMPOSITE SURFACE

Using a scanning electron microscope, the state of the surface layer of composite samples was assessed after the tribological experiment. Figure 18 shows an image of the surface of a roll with the composite layer deposited on it after friction-wear tests were conducted. The area to the right of the line (Fig. 18) shows the surface subject to friction, whereas the area to the left presents an image of the composite outside the friction area.

Comparing the microscopic images of composites with different qualitative compositions that were subject to tribological tests, visible differences in the appearance of the surface can be observed. The surface containing the binary system of lubricant additives (Fig. 18a) is smooth, with no signs of cracking, surface scratches or excessive wear. The surface of the composites with single lubricant additives on the other hand exhibits clear signs of wear. On the surface of the composite containing molybdenum disulfide (Fig. 18c), visible cracks parallel to the friction path can be observed, together with surface defects arising from the spalling of the metallic filler from the chemically hardened polymer matrix of the composite, which confirms the higher wear of this composite. Such effects and signs of wear are not observed for the composite sample containing combined lubricant additives with a layered structure. In experiments aimed at the determination of wear mechanisms for composite associations, the surface of the composites was observed with a scanning electron microscope, and the topography and roughness of the surface of the tested composite samples was measured.

a) b) c)

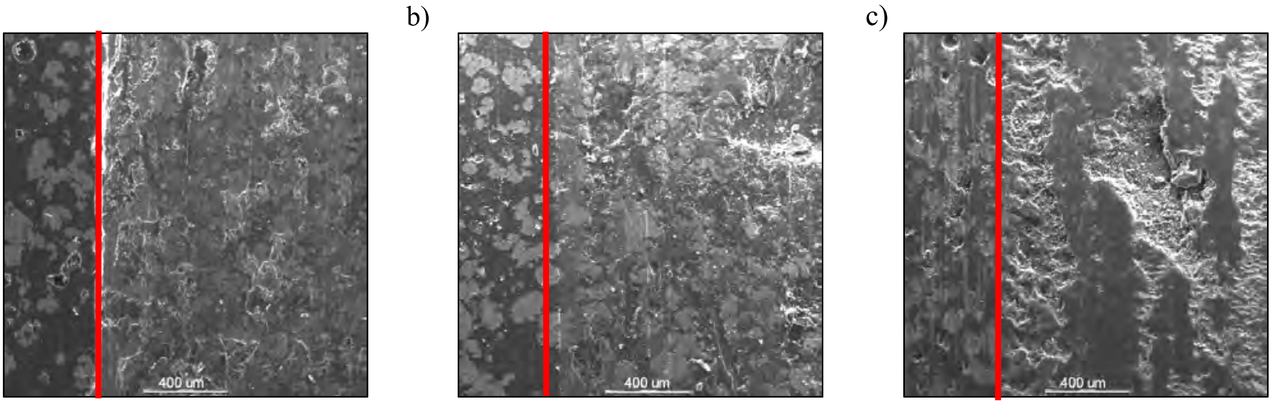

Fig. 18. Microscopic images of composite samples after tribological tests: a) GM-20 composite, b) G-20 composite, c) M-20 composite

Rys. 18. Obrazy mikroskopowe próbek kompozytów po badaniach tribologicznych: a) kompozyt GM-20, b) kompozyt G-20, c) kompozyt M-20

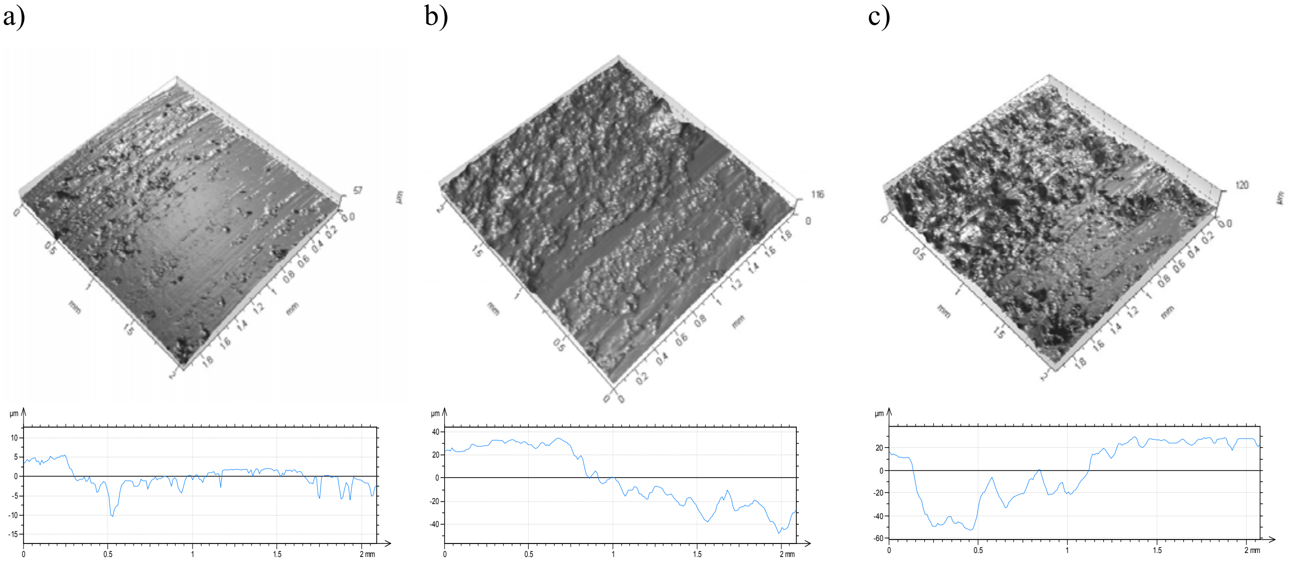

Fig. 19. Geometric structure of surface and roughness profile of roller after tribological tests: a) GM-20 composite, b) G-20 composite, c) M-20 composite

Rys. 19. Struktura geometryczna powierzchni i profil chropowatości rolki po badaniach tribologicznych: a) kompozyt GM-20, b) kompozyt G-20, c) kompozyt M-20

The results of the surface investigations (Fig. 19) indicate significant dependence between the surface and the type of modifiers of tribological properties used in the composite. As presented in the above images, the main wear mechanisms are in fact connected with the scratching and ridging of the surface and the spalling of fillers from the polymer matrix of the composite.

This may be due to the combined interactions of normal forces and forces that are tangential to the surface of the composite that take place during friction. However, in the case of compressive strength tests, only normal forces (perpendicular to the surface) affect the surface. This fact can also be used to explain the differences between the mechanical and tribological properties of the tested composites.

The smallest roughness and the surface that is least damaged after the execution of tribological tests in which no lubricants were added, can be observed for the composite containing combined lubricant additives (graphite and molybdenum disulphide). The highest surface roughness and pathological signs of wear can on the other hand be observed on the surface of the composite containing molybdenum disulfide only.

CONCLUSIONS

Based on the results obtained for metal-polymer composites, it can be concluded that mechanical, particularly tribological properties, depend on the type and amount of lubricants with anisotropic cohesion. The tribological experiment in dry friction conditions showed that the composite samples containing a binary system of additives - graphite and molybdenum disulphide - cooperating with a steel counter sample exhibit high resistance to wear and a low friction coefficient.

The results of friction-wear tests, and the study on the condition of the surface layer of the composite samples, suggest that in the composite containing more than one lubricant additive, an operational surface layer helping to reduce wear and eliminating any resistance to motion, is created. The presence of graphite further reduces the susceptibility of the composite to adhesive interlocking.

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