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Evaluation of selected properties of PA6-copper/graphite composite

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Properties

ABSTRACT

Purpose: The aim of this study was to investigate the possibility of improving the tribological properties and thermal conductivity of composites with structural modification of polyamides by the additions of copper and graphite.

Design/methodology/approach: The study involved testing polyamide composites containing metallic powder. As the matrix was used polyamide 6 Tarnamid 27 Natural produced by Azoty Tarnów, which strengthened copper, graphite or molybdenum disulfide with varying participation in the composite. Taken tensile test, Brinell hardness, thermal conductivity and the tribological wear resistance. Analyzed the influence of the type of dopant and the participation of the studied property.

Findings: The study showed the desirability of the use of graphite and copper as fillers polyamide. The resulting composites are characterized by satisfactory mechanical properties and thermal conductivity. Applied fillers also showed a positive effect on the value of the coefficient of friction wear him down much.

Research limitations/implications: No synergistic effect of fillers on the properties of a ternary mixture polyamide-graphite-copper property in all test. Tribological wear for this material was significantly higher than for the binary mixtures of polyamide-graphite and polyamide-copper. According to the literature, composite reinforced with graphite and copper should have a best tribological properties, far in excess of other subjects. Low wear resistance of this material disqualify applications on the nodes friction.

Practical implications: The results obtained newly developed composites, combined with the relatively low price of graphite and copper as compared with the price of molybdenum sulfide II may be reasons to use them as analogues of commercial mixtures of PA with MoS_2.

Originality/value: The results are original and valuable cognitive nature. They bring a new and expanded information about the effects of fillers on mechanical and physical properties. Unique data on tribological wear resistance depends on the type and amount of filler.

Keywords: Engineering polymers; Polyamide; Metallic powders; Mechanical properties; Wear resistance

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1. Introduction

One of the main advantages of polymeric materials is susceptible to easy modification of their properties through the introduction of appropriate fillers. With the addition of certain materials can affect a number of characteristics of materials to suit specific application. Particularly good results are observed in the use of fillers in relation to the values of the mechanical properties. It is common to use thermoplastic polymers reinforced with ceramic particles (glass, chalk, montmorillonite) in order to increase the value of the stiffness, strength or creep resistance.

With the addition of the filler can be improved, in addition to mechanical properties, many other characteristics of the materials.

In this paper, the authors have attempted to improve the tribological properties of thermoplastics by using a filler metal and graphite. Macromolecular material is characterized by the multiple-tribological properties depending on the kind of wear resistance and coefficient of friction may vary significantly from each other. In general, however, all the polymers are characterized by a low coefficient of friction pair friction polymer-metal system nodes commonly found in construction of technical facilities. There are much bigger differences when analyzing the coefficients wear-phenomenon will be discussed later in the article [2, 6, 25, 31].

Low friction associated with a favorable coefficient of friction of polymeric materials predispose it to act as a friction element nodes kinematic multiple devices. Widely used are, for example, plastic macromolecular acetabulum. A limitation of the expansion of these elements, however, is their very low (in comparison with similar products with metal or ceramic) thermal resistance. Significantly narrows the range of possible applications to applications where there is, or is not generated high temperatures that can damage the material. Such adverse conditions can occur at high speeds or prolonged friction that prevents heat dissipation resulting [24, 26, 31].

Fig. 1. View samples tested materials in the form of standard fittings Testing

Used to test polyamide 6 PA6 is one of the most widely used polymer for frictional nodes. The unmodified condition characterized by a low coefficient of friction and satisfactory wear resistance and high strength. Another advantage is significant when compared to other polymers, heat resistant, capable of operating under adverse thermal conditions. To improve his and so good tribological properties, producers fill polyamide molybdenum sulphide II-MoS_2. This compound acts as a dry lubricant reducing friction and preventing wear. PA6 compositions of MoS_2, and unmodified PA which were used by the authors as a reference sample in the evaluation of research results.

MoS_2-like activity is characterized by graphite, which thanks to its perfectly layered structure acts as a dry lubricant. However, unlike molybdenum sulfide II, is also an excellent conductor of heat. Noteworthy is also significantly lower price compared to graphite in molybdenum sulfide II [10, 21, 30, 31].

A slightly different mode of action for tribological applications is characterized by a filler in the form of metallic copper powder. According to the authors' intentions, he had to improve the thermal conductivity of the material and, above all, act as a "scaffold" upon which slipped to the second of a pair of friction elements while preventing excessive wear of polymer matrix. This composition is, in a sense, an analogy Babbitt’s metal-bearing structure composed mainly of alloys of tin, copper, lead and antimony. In the alloys of the hard phase Sn_3Sb_2 and Cu_6Sn_5 particles act as carriers, and soft plastic insulating matrix rich in tin and lead. Additional, the expected benefit from the use of copper, according to the authors was to increase the thermal conductivity of the material

1.1. Physical and mechanical aspects of the friction polymers

The friction force is the result of the sum of the other two components of the forces: adhesion and hysteresis. Friction polymer depends on both of them, but at different conditions of the setting process is different their participation. The coefficient of friction of all polymers is inversely proportional to the load, which results from the elastic-plastic nature. Despite the increase in the value of the component of adhesion, friction due to better contact, the effects of stiffening, to change the value of the component of the hysteresis and the resulting decrease in the coefficient of friction. A similar effect can be achieved by changing the temperature or the speed of the phenomenon which causes deformation of the polymer chains [1, 5, 7, 16, 19].

In the case of real technical objects, in which there are all kinds of structural nodes friction is inevitable formation of vibration. In connection with the reciprocal movement of the two elements relative to each other, one of which is made of plastic, this results in a multiplication of the quantity supplied to the power element. The consequence of the construction characteristics of viscoelastic polymers is their ability to store energy in itself, for elastomeric materials which can reach very high values, several times higher than the capacity of such steel. This property is characterized by the hysteresis loop. Stored energy is converted into heat causing the temperature of the increasing and consequently the structural changes in the material [22, 30, 31].

Materials used for the production of components exposed to friction, like most polymers are electrical insulators. In the case of the mutual movement of the two elements relative to one another on their surface forming electrostatic charge. In addition, heat flow, the resulting friction generates a thermoelectric phenomenon, involving the transfer of energy by electrons from warmer to colder places. As shown by the results of investigations an accumulated charge on the polymer component friction pair can reach a very high value, and its effects can significantly affect the conditions of the process of friction by modifying the structure of the polymer. The rapid release of such loads as a result of the penetration of the plastic layer in a flow of current densities of up to several A/cm^2 may be accompanied by additional heat come out capable plasticity, and even spread polymer [17, 19, 28].

1.2. The friction polymer components

In the case of the friction of the polymer component is extremely important to the process of his wear, the mass transfer associated with on the other of the pair of friction. Currently, due to the preferred value of the coefficient of friction, the most common pairs are the slider and the sliding metal-polymer systems. In such a combination is a macromolecular material particles move to the metal element.

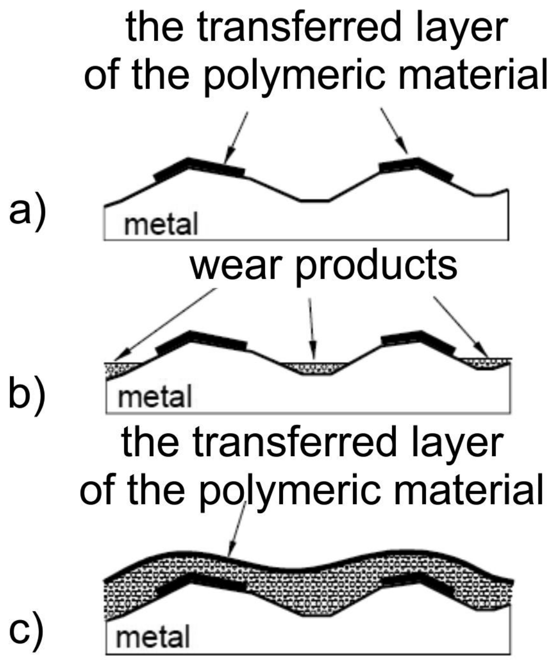

Fig. 2. Model the process of creating a polymer film in collaboration couple polymer-metal sliding a) the beginning of the creation of a polymer film on the metal surface roughness peaks b) wear products fill the spaces between micro peaks c) the final step of forming a polymer film during the friction [3]

This process results in the postponement of the thin polymer layer and change the layout of the polymer-metal on polymer-polymer. Is initiating tack adhesive particles and pulling the two surfaces of the polymer fragments. This implies a decrease heat of friction node, while resulting in improved damping and reduced friction – that layer acts as a lubricant. Difficult outflow of heat will raise the temperature and the intensification of the process of transferring the polymer. With the increase in speed of transfer of matter, increases the thickness of the plastic film, causing further deterioration of heat dissipation. In extreme cases, the thermodynamic imbalance can cause such a system to melt the plastic (Fig. 2). The rate of growth of the polymer layer is closely related with the adhesion interactions. It can be reduced by treating the metal element such as erosion smoothing techniques, or applying a ceramic PVD coatings [8, 11, 14, 16, 18, 28].

Under the conditions of thermodynamic equilibrium is established after the establishment of the polymer film layer of the metal element is the deposition of the next layer, but for a plastic element. At this stage, any tribological effects occur in the outer layers of the film. This means that, under such conditions should theoretically result in a complete stop of wear of both. This does not, however, due to the continuous erosion and of thermal destruction of the layer film. Extremely dangerous is also at this stage the presence in the layer between the components the hard inclusions, which can cause abrasive wear. They may come from the same polymer filled with ceramic particles often. A similar effect can be use in case of inaccurate machined piece of metal and high surface roughness. In conditions such consumption will be dominated by the frictional adhesion [8, 9, 11, 16, 22, 28].

1.3. Tribological wear mechanisms of polymeric materials

The process is closely connected to the friction process of destruction of the material. This process is caused by abrasion, erosion, and cracking of the particulate material and the adhesion surface of the working elements and chemical reactions occurring on the surface of the friction elements. In the case of polymer cooperation with metals the most common types of wear are:

- **Adhesive wear**. Associated with adhesion friction surfaces. It causes local tacking these surfaces and transfer of the polymer to the surface of the counter element. In the next phase of movement followed by detachment of the material transferred from the counter element and removes it from the friction zone. This process is cyclical and will wear the particular polymeric material. Substantial effect on the adhesion of a polymeric material surface energy and surface roughness against a counter element.
- **Abrasive wear.** Occurs in mate with the surface of the polymeric material with a high roughness. Then projecting harder material inequalities serve as microblades. The loss of material due to the microcutting, drawing or fissures. This process also takes place when in the friction area associated components are loose or firmly established abrasive particles.In the case of abrasive wear of sliding pairs polymer-metal can apply to both the polymer and the metal. The second case occurs when the polymer is filled with hard particles such as glass fiber, quartz powder, etc. Then, the hard particles of the filler may machine the surface of the metal causing it to wear. Abrasive wear is characterized by high intensity and often causes strong heating of the polymer

material. Therefore accompany him - except the mechanical- also thermal and chemical processes of wear.

- **Wear fatigue**. The reason for this type of wear is cyclic, variable deformation of the surface layer. For this reason, the surface micro-cracks appear, which the further development and connect causes spalling of the material. Wear is growing fast as the deformation of the polymeric material are plastic. In the case of polymers wear fatigue causes cracking macromolecular chains. This contributes to reduce the molecular weight of the polymer in the surface layer and the reduction in the crystallinity of the polymer.
- **Erosive wear.** Wear is formed by impact of fine particles on the surface of the material. This process is not well known and it is assumed that, for polymeric materials can be treated as fatigue wear.
- **Chemical Wear.** Wear occurs as a result of chemical reactions between the mating materials. It may also be related to reactions between the material and the environment (such as oxidation), in which there is a process of friction. Degradation processes have a major impact on the intensity of the response of the material. Chemical Wear usually accompanied by other types of wear and is intensified by heat-generating other tribological mechanisms.
- **Thermal Wear.** This Wear is the result of the evolution of large amounts of heat, which causes softening and plasticizing the material in the surface layer. This process may occur even bonding surface friction [3, 11, 15, 22].

Modification of tribological properties of polymers

In order to improve the tribological properties of the polymeric materials used to modify their structure and the filling of different types of particles. Increasing the contribution of the crystalline phase in the polymeric material to increase the stiffness modulus and strength of the material, preventing wear fatigue, and increasing capacity node design. In addition, the crystalline phase transition in the amorphous macromolecular material requires the delivery of thermal energy. Therefore, materials having a crystal structure are more resistant to brief, sudden increase in the temperature of the work compared to the same materials with amorphous structure. Increasing the share of ordered crystalline phase in the structure of the product can be accomplished by the use of appropriate chemical modifiers or heat treatment at the production stage - the higher forms temperature and longer time of cooling and after the step of molding such as by heating and slow cooling. An additional amount of energy associated with a higher temperature facilitates the movement of the polymer chains by giving them the opportunity to cross-orientation [11, 12, 22, 30].

The most popular and easiest ways to improve the tribological properties of the material to be placing various types of fillers. The most commonly used are: molybdenum disulfide, graphite, fiber and glass beads and various ceramic particles. They have a self-lubricating effect rubbing surfaces (graphite and MoS_2), or are reinforcing structure on which slides the second surface rubbing. In the literature one can find a brief description of the modification of tribological properties of polymeric additives aramid fibers, basalt, and even low-melting alloys. The most interesting fillers are of certain textile and leather waste from the textile industry. They are subject to pre-process of saturation of organic oils and greases, in the state hitting the polymer matrix. Such fillers are generally used for the curable material exhibiting self-lubricating action [1, 4, 11-13, 20, 22, 23, 27, 29].

The aim of this study was to investigate the possibility of improving the tribological and thermal properties of the products of the structural modification of polyamides with their copper and graphite additives. Currently on the market there are blends of polyamides with improved tribological properties by additions of molybdenum sulfide II. However, these materials are relatively expensive, and an obstacle to their use are poor thermal properties, especially the thermal conductivity of low value, to prevent their application in structural nodes are exposed to elevated temperatures. An alternative to the above-described materials may become newly developed materials.

2. Material and experimental methodology

The study was used as the matrix polymer polyamide 6 Tarnamid 27 Natural produced by Azoty Tarnow, filled with copper, graphite and molybdenum disulphide in different concentrations. For all of the filler particle size testing is done by X-ray or laser (graphite), with the following results median particle size of filler:

- Copper-19.95 microns,
- molybdenum sulfide II-14.6 microns,
- graphite (technical graphite C-12.01)-58.3 microns.

Images of the powders particles fillers used, are shown in Figures 3–5. Observations of the filler particles was performed using an optical microscope Opta-Tech MN800, in reflected light at a magnification of 40 ×.

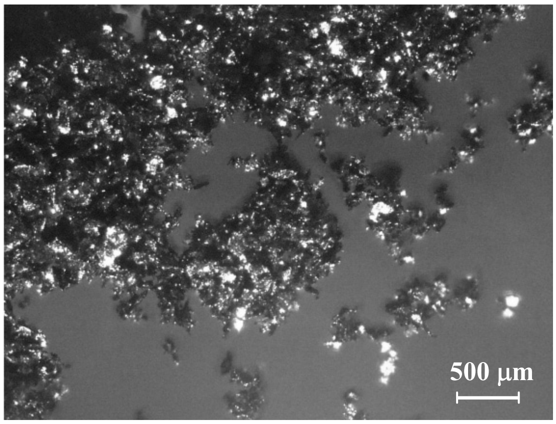

Fig. 3. An image of the graphite powder particles

Test compounds were pre-extrusion moulding using a twin-screw extruder, a counter-Goetfert thereby obtaining granules. The use of this type of extruder allowed to obtain good homogenization of the material. This was particularly important when mixtures containing copper powder, which, due to its large density tended to sediment in the hopper. The technology of extrusion granules obtained were performed using an injection molding machine Battenfeld Plus 35/75 test fitting.

Before each stage of processing, the test compounds were dried at 80°C in order to remove moisture. This procedure was necessary because of the base polymer used, which has the highest of all commonly used engineering plastics water absorption of about 10%. The release of moisture during compound processing of extrusion technology or injection could result in the formation of voids that reduce the value of the mechanical and physical properties .

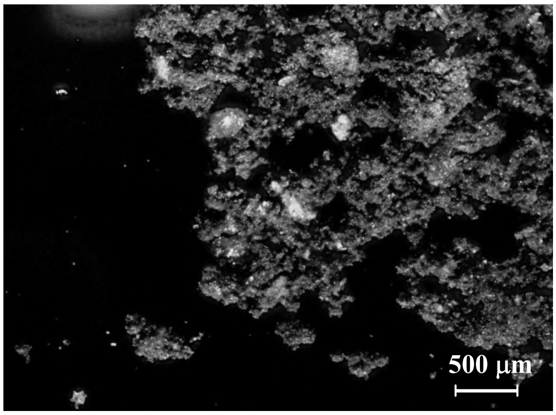

Fig. 4. An image of the MoS_2 powder particles

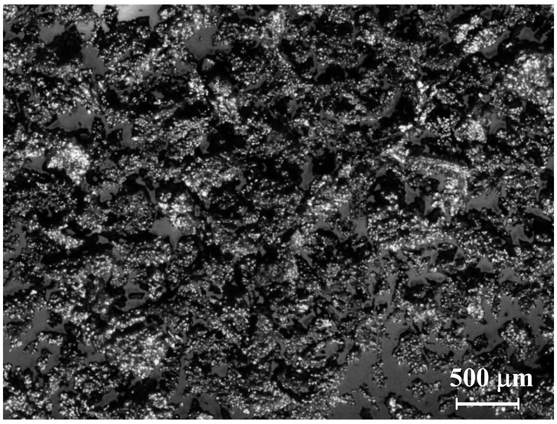

Fig. 5. An image of the copper powder particles

The test materials were characterized by the following volumetric composition (part additive given in% by volume):

- 100% PA6;
- 98%PA6+ 2% Cu;
- 95% PA6+ 5% Cu;
- 98%PA6+ 2% graphite;
- 95% PA6+ 5% graphite;
- 96%PA6+ 2%Cu+ 2% graphite;
- 98%PA6+ 2%MoS_2.

The mixture does not contain any modifier, and includes MoS_2 accounted the reference materials for the other materials.

3. Results

The study aimed to determine the functional properties determining the suitability of the material for tribological applications in construction engineering. In particular, the tensile strength, elongation at break, Brinell hardness, thermal conductivity and the rate of wear of the friction pair of polymer-metal.

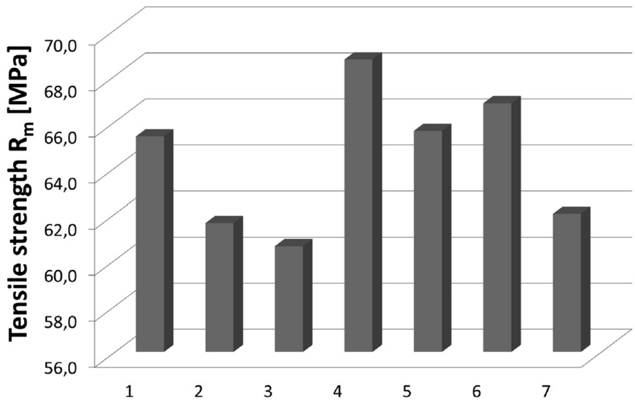

Fig. 6. Tensile strength test composites: 1 – PA6, 2 – PA6+ 2%Cu, 3 – PA6+5%Cu, 4 – PA6+2% graphite, 5 – PA6+5% graphite, 6 – PA6+2% graphite + 2%Cu, 7 – PA6+2%MoS_2

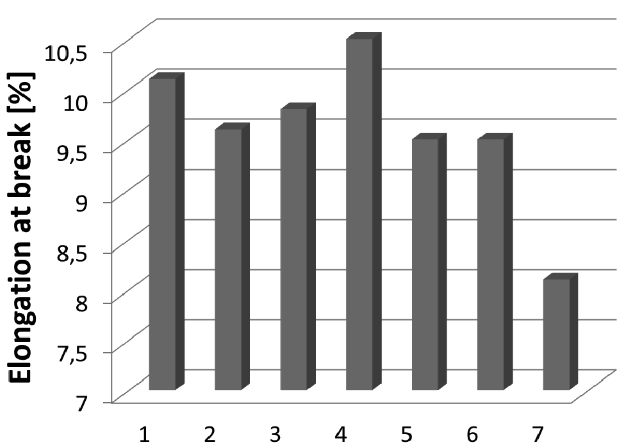

Fig. 7. Elongation at break of the composites tested: 1 – PA6, 2 – PA6+ 2%Cu, 3 – PA6+5%Cu, 4 – PA6+2% graphite, 5 – PA6+5% graphite, 6 – PA6+2% graphite + 2%Cu, 7 – PA6+2%MoS_2

Figures 6 and 7 show the results of the mechanical property measurements carried out using a universal testing machine Instron TT-CM 80 They show that the additives fillers have a significant influence on the strength of the material. Graphite in amounts added to the base polymer increases the strength. With the largest increase observed for concentrations of 2%. Slightly lower tensile strength value is a mixture of 5% of the content of graphite. This effect is probably due to the excess of the optimum filler content in the material and the creation of the agglomerates. Inclusions such exhibits a notch mechanical, and reduces the actual cross-section plastic stress transfer. It is worth noting that the graphite in a mixture of copper has a positive effect on the mechanical properties of this material, even though the binary mixtures of polyamide with copper reduction of value tensile strength was recorded compared to unfilled polyamide. This is due to poor adhesion of the metal particles to the polymer matrix. In spite of this negative phenomenon, their strength is still high and fully allows the application of this material as a construction material. Another filler product which causes a decrease in the mechanical properties is molybdenum sulfide II. This modifier also reduces the greatest elongation at break material containing it. In addition two-component mixture of MoS_2 PA6, the elongation at break of all other mixtures containing fillers, was close to the value of elongation at break of unfilled plastic.

The hardness of the material used for the elements of friction pairs are literal effect on the properties and stability of such a utility node design. Less susceptible to deformation of the material is also more resistant to fatigue compared to less rigid element. An additional benefit of using this material it is possible to increase the capacity of the item made out of it, and the generation of oscillations in the case of modules that rapid rotation. In the case of deformable element, such movement in conjunction with the rotating element imbalance causes permanent movement of the deformed zone around the axis of rotation of generating vibration and causing misalignment of the shaft axis and the axis of rotation, ie. wobble. This phenomenon generates the formation of the system of additional radial force with a value dependent on the size of the unbalance and speed. The presence of such a force can contribute to excess capacity and component damage.

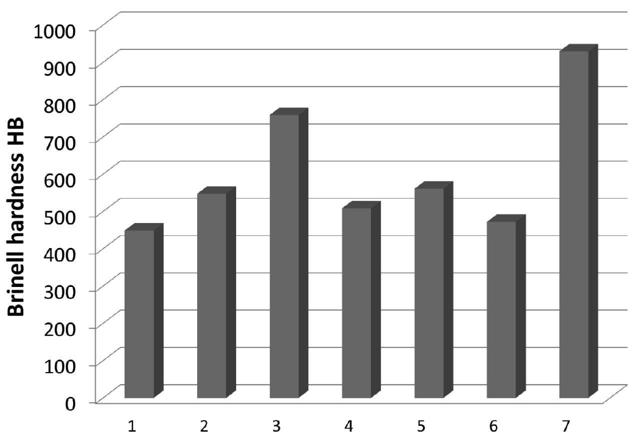

Fig. 8. Brinell hardness test composites: 1 – PA6, 2 – PA6+ 2%Cu, 3 – PA6+5%Cu, 4 – PA6+2% graphite, 5 – PA6+5% graphite, 6 – PA6+2% graphite + 2%Cu, 7 – PA6+2%MoS_2

All blends containing the modifiers were characterized by a higher hardness than the matrix of pure base polymer, which is illustrated in Figure 8 The highest hardness and thus resistance and undergo deformation have a blend containing molybdenum disulphide. Slightly lower hardness of the material (although still relatively high) is characterized by the addition of 5% of copper. For mixtures containing the filler correlation between increase of hardness and modifier content is most pronounced. Graphite as copper increases hardness value blend with added, but the increase is not as big as in the case of using the modifier metal. Somewhat surprising were the results of a ternary mixture hardness polyamide-graphite-copper, for which it was expected to get the cumulative effect of the two fillers. This mixture has a hardness of less than two-component materials. This effect will be the subject of further research.

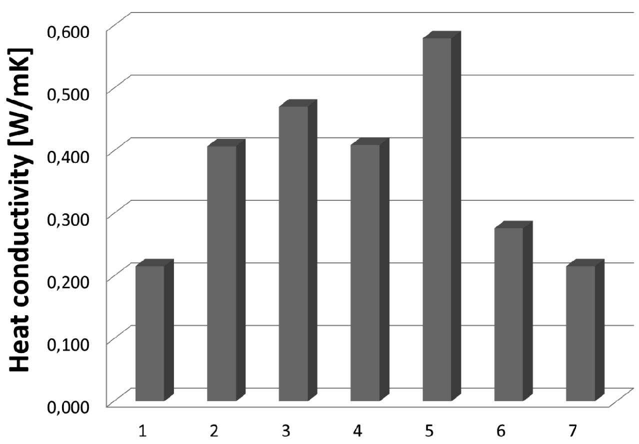

Fig. 9. The heat conductivity of the composites tested: 1 – PA6, 2 – PA6+ 2%Cu, 3 – PA6+5%Cu, 4 – PA6+2% graphite, 5 – PA6+5% graphite, 6 – PA6+2% graphite + 2%Cu, 7 – PA6+2%MoS_2

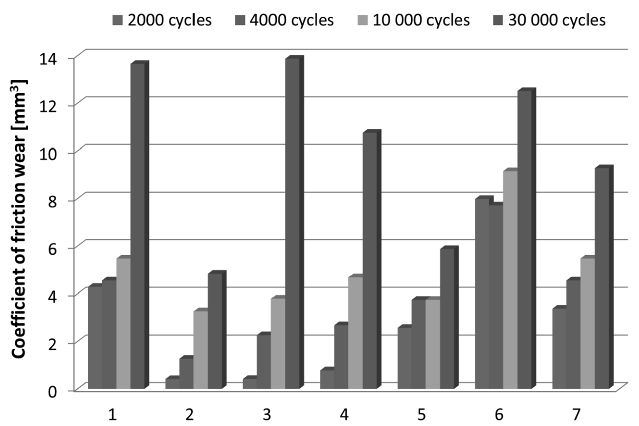

Fig. 10. Tribological wear test composites; 1 – PA6, 2 – PA6+ 2%Cu, 3 – PA6+5%Cu, 4 – PA6+2% graphite, 5 – PA6+5% graphite, 6 – PA6+2% graphite + 2%Cu, 7 – PA6+2%MoS_2

As expected additions of copper and graphite significantly raised the thermal conductivity of the test materials, as shown in Figure 9. For a content of 2% of both fillers λ thermal conductivity of approximately 0.4 W / m • K, the differences in conductivity were seen only for the modifier content of 5%.

Greater thermal conductivity was characterized by a mixture of graphite. This fact is probably due to a better distribution of the graphite in the polymer matrix and the creation of energy-efficient heat conduction paths. Copper, in spite of much higher thermal conductivity than graphite, did not improve as much as graphite thermal conductivity of the polymer blend. This effect, as in the case of the breaking strength, can be explained by the formation of filler agglomerates separated by layers of plastic base materials that prevent good conductivity. Improved thermal conductivity of the composite with graphite are the result of the cleavage. After the addition of graphite to the polymer is divided into flakes along the crystallographic direction [002] and stacking in this manner in the polymer creates a "path" conductivity. Molybdenum sulfide II as a modifier of tribological properties, did not affect the thermal conductivity. This is understandable, since MoS_2 as salt, I do not have very good thermal conduction properties. As in the case hardness, so in the test of thermal conductivity, a three-component blend of polyamide-graphite-copper, showed no synergistic effect of fillers on the test parameter, causing only a slight increase in the thermal conductivity.

Abrasion test was performed by pin-on-plate using a steel penetrator diameter 8.7 mm and a mass of 630g. In order to evaluate the tribological properties were performed 2, 4, 10 and 30 thousand abrasion cycles. After each stage, using an optical microscope was evaluated as a loss of material in the sample volume of the worn groove. View of an exemplary abrasion that occurred on the sample composition of PA6 5% graphite is shown in Fig. 10.

The smallest coefficient of friction wear is characterized by a mixture having a content of 2% copper. Higher friction wear of blend containing 5% of the modifier after 30 thousand abrasion cycles is probably due to the pulling out of the filler particles in the polymer matrix. However, in the earlier stages of the study (2, 4, 10 thousand cycles) of this mixture showed satisfactory tribological properties compared to the pure base material (Fig. 11).

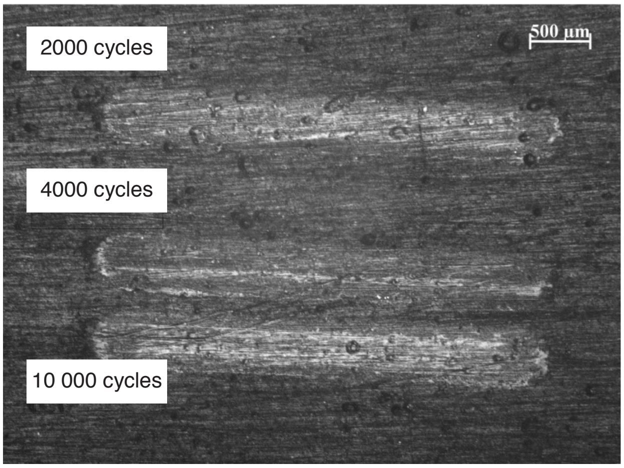

Fig. 11. Trace wipe samples PA6 with 5% graphite

This effect could be caused by abrasion of the polymer matrix and excessive exposing modifier particles which have lost their stable fixation and have been removed from the area under the influence of friction forces acting. Quite different from the mixture of copper mechanism for improving the tribological properties are characterized by a mixture of graphite. Graphite acts as a lubricant in them to prevent accelerated wear. Therefore, the observed reduction in friction wear, along with increasing the content of the filler.

Against the expected behavior of the sample found in their composition containing molybdenum disulfide, and mixtures of the ternary polyamide-graphite-copper. In the first of them was quite small in relation to the pure PA reduced susceptibility to wear after 30 thousand abrasion cycles. However, during the first three cycles, the wear of the material was much higher than for the binary mixtures of polyamide and polyamide-copper-graphite. This fact is to clash with the literature, according to which the mixture is to be characterized by the best tribological properties, far in excess of other subjects. In the case of a ternary mixture confirmed once again the lack of a synergistic effect of fillers on the properties of the mixture. It was characterized by a relatively low resistance to abrasion, which disqualifies this material for use in friction nodes.

4. Conclusions

The study showed the desirability of the use of graphite and metallic copper as fillers polyamide, as materials for use in construction nodes friction. The described compounds are characterized by satisfactory with respect to mechanical properties of the polymer base in case of graphite higher strength and toughness, and hardness of the copper. Both modifiers significantly improve the thermal conductivity of the material. This is a highly desirable feature for materials which are to be made from elements that require the ability to quickly dissipate the heat from the working area. Test fillers also showed a positive effect on the value of the coefficient of friction wear him down much. The above-mentioned advantages, combined with the relatively low price of graphite and copper as compared with the price of molybdenum sulfide II may be reasons to use them as analogues of commercial mixtures of MoS_2 with PA.

In the future we plan to undertake research on the modification of the filler and the polymer matrix which, to improve the adhesion between them. Stronger embedded of copper grains in the material macromolecular would prevent them from pulling out of the warp and the ensuing increase in wear observed after 30 thousand abrasion cycles in presented in this paper study.

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