Title: Tribologiczne właściwości ceramiczno-węglowych warstw powierzchniowych otrzymywanych w elektrolitach o różnej zawartości grafitu

Author: Marek Bara, Władysław Skoneczny, Sławomir Kaptacz

Citation style: Bara Marek, Skoneczny Władysław, Kaptacz Sławomir. (2008). Tribologiczne właściwości ceramiczno-węglowych warstw powierzchniowych otrzymywanych w elektrolitach o różnej zawartości grafitu. "Eksploatacja i Niezawodność" (Nr 4, (2008), s. 66-70).

Marek BARA
Władysław SKONECZNY
Sławomir KAPTACZ

TRIBOLOGICZNE WŁAŚCIWOŚCI CERAMICZNO-WĘGLOWYCH WARSTW POWIERZCHNIOWYCH OTRZYMYWANYCH W ELEKTROLITACH O RÓŻNEJ ZAWARTOŚCI GRAFITU

TRIBOLOGICAL PROPERTIES OF CERAMIC-CARBON SURFACE LAYERS OBTAINED IN ELECTROLYTES WITH A DIFFERENT GRAPHITE CONTENT

W pracy przedstawiono tribologiczne właściwości kompozytowych warstw powierzchniowych tlenek glinu-grafit. Warstwy otrzymano metodą elektrolityczną, w elektrolitach o różnym stężeniu grafitu. Wytworzone warstwy skojarzono z tworzywem PEEK/BG w ruchu posuwisto-zwrotnym, w warunkach tarcia bezsmarowego. Przedstawiono rezultaty badań współczynnika tarcia pary ślizgowej i zużycia masowego tworzywa. Celem określenia mikrogeometrii powierzchni warstw tlenku glinu oraz warstw tlenek glinu-grafit przeprowadzono badania struktury geometrycznej powierzchni za pomocą profilografometru stykowego, przed i po teście tribologicznym. Zaprezentowano również obrazy struktury i morfologii powierzchni warstw tlenek glinu-grafit oraz tworzywa PEEK/BG wykonane przy zastosowaniu elektronowej mikroskopii skaningowej.

Słowa kluczowe: *właściwości tribologiczne, warstwa kompozytowa, mikroskopia skaningowa, tworzywo sztuczne, mikrogeometria powierzchni.*

The paper presents the tribological properties of composite aluminium oxide-graphite surface layers. The layers were obtained by the electrolytic method, in electrolytes with a different graphite concentration. The produced layers were coupled with a PEEK/BG material in reciprocating motion, under lubricant-free friction conditions. The results of research regarding the coefficient of a friction couple wear and mass wear of the material are presented. In order to determine the microgeometry of the aluminium oxide layers' surface and of aluminium oxide-graphite layers, investigation was conducted of the geometrical structure of the surface using a contact profilographometer before and after a tribological test. Images are also presented in the paper showing the structure and surface morphology of aluminium oxide-graphite layers and the PEEK/BG material, taken using scanning electron microscopy.

Keywords: *tribological properties, composite layer, scanning microscopy, plastic material, surface microgeomety.*

1. Introduction

The oxide ceramic layers Al_2O_3 produced via hard anodizing belong to a group of materials with a strongly developed surface. They are frequently used for lubricant-free sliding couples with polymers, in the production of servo-motors, compressors or shock absorbers.

Under technically dry friction conditions, the essence of a polymer/aluminium oxide layer, the latter being hard and resistant to wear, is the formation of a polymer sliding film. The film has an ability to significantly reduce resistance to motion in a further friction process causing, however, an increase in mass wear of the material in the initial phase of the couple's interaction [1, 2]. Preventing such undesirable effects has been realized so far through the creation of polymer compositions being a mixture of a basic material with fillers. In the case of PTFE based compositions, fillers in the form of powders, flakes or fibres are used. The most important materials used as fillers in the production process of composite materials with a PTFE matrix include powdered graphite, carbon, molybdenum disulfide, glassy carbon and powders of the following metals: bronze, brass, antimony, nickel, and oxides of some metals. Owing to a laminar structure of the crystal lattice and anisotropic properties of cohesion forces of the fillers, i.e. graphite or molybdenum disulfide, a partial replacement of external friction with internal friction of a low value takes place between the interacting surfaces, which leads to a reduction of the friction forces [3].

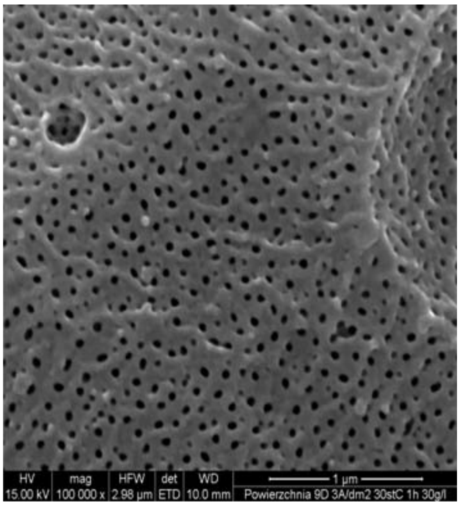
A similar effect of a reduction in resistance to motion during technically dry friction, without excessive mass wear of the material in the initial phase of the couple's interaction, could be achieved by incorporating the graphite into the oxide structure.

2. Ceramic-carbon surface layer

The object of the tribological tests were ceramic-carbon layers of aluminium oxide/graphite, produced on a substrate of aluminium alloy EN AW-AlMg2, of dimensions: 1.8 × 6 cm. The process of surface layers production was preceded by cleaning the aluminium alloy surface through etching in a 5% KOH solution and a 10% HNO_3 solution, followed by rinsing in distilled water.

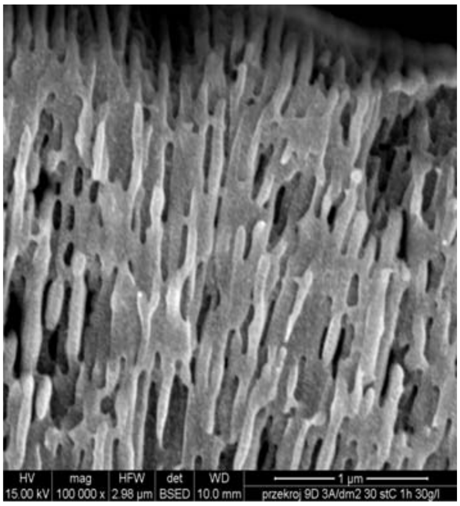
The layers were obtained in an electrochemical oxidation process by the direct-current method, in electrolytes with organic acids' and graphite's additions. Four electrolytes were used, differing with the graphite content from 0 to 30 g/litre of electrolyte. The oxidation process was conducted for an electric charge of 180 Amin for all the surfaces. Current density amounted to 2 - 4 A/dm^2 with oxidation time of 40 - 90 minutes. The bath temperature was 293 and 303 K.

The image of the aluminium oxide-graphite layer's morphology, taken using scanning electron microscopy (Fig. 1), presents, characteristic for aluminium oxides, surface porosity of a cylindrical structure. The porosity is an effect of the collumnar architecture of the structure (Fig. 2), oriented against the electric field [4].



Rys. 1. Morfologia powierzchni warstwy tlenek glinu-grafit

Fig. 1. Surface morphology of aluminium oxide-graphite layer



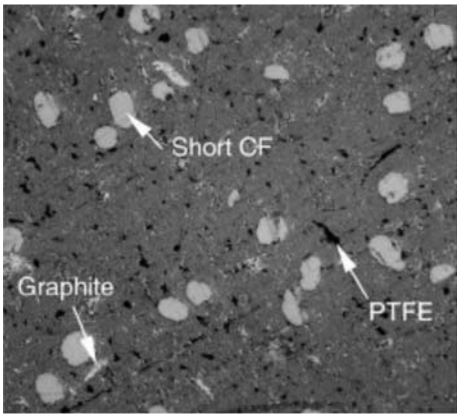
Rys. 2. Struktura warstwy tlenek glinu-grafit

Fig. 2. Structure of aluminium oxide-graphite layer

3. The PEEK/BG material

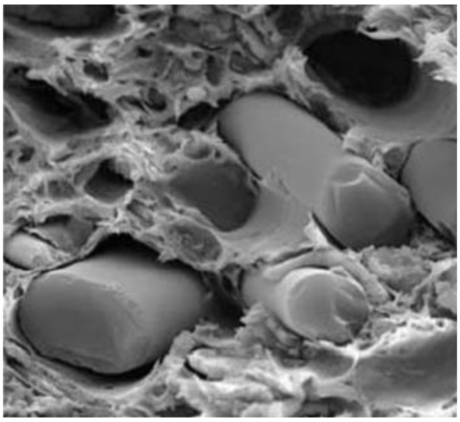
The material interacting with the aluminium oxide/graphite layers was a high-quality polymer composite based on polyetheretherketone. PEEK is a partly crystalline thermoplastic material with very good mechanical properties. It is characterized by a high temperature of continual work, amounting to 523 K, and perfect chemical and radio-resistance. Good sliding properties and low absorbability, ensuring high dimensional stability, complement the properties of this high-parameter material, making it a universal material for a number of applications.

An addition of PTFE, graphite and carbon fibres to the PEEK material (Fig. 3) yields a material with enhanced mechanical strength, a reduced friction coefficient and improved abrasion resistance. Its optimal tribological properties make the PEEK/BG particularly suitable for applications in friction couples consisting of bearings. Photographs of the material (Figs. 3, 4) have disclosed a unidirectional arrangement of carbon fibres in the structure of PEEK/BG [5-8]. For the tribological tests, a surface of the material was used, in relation to which the fibres were arranged perpendicularly. A PEEK/BG sample prepared for the tests was cube-shaped, with a side of 10 mm.



Rys. 3. Morfologia powierzchni PEEK + 10% PTFE + 10% grafitu + 10% włókien węglowych [6]

Fig. 3. Surface morphology of PEEK + 10% PTFE + 10% graphite + 10% carbon fibres [6]



Rys. 4. Przełom PEEK + 10% PTFE + 10% grafitu + 10% włókien węglowych [6]

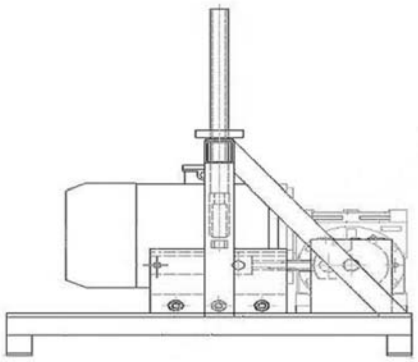
Fig. 4. Fracture PEEK + 10% PTFE + 10% graphite + 10% carbon fibres [6]

4. Research methodology

Tribological tests were conducted on an RS 2007 testing machine, dedicated for testing materials which interact as a sliding couple in reciprocating motion (Fig. 5).

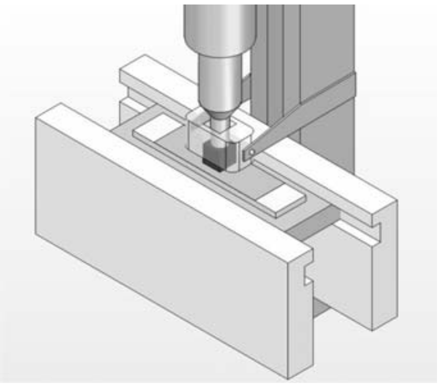
The friction couple in RS 2007 (Fig. 6) reproduces the work of components of a piston-cylinder couple. The specimen used in the test is a sector of a piston gasket ring and the counter-specimen is a sector of a cylinder sleeve. Counter-specimens are fixed in a guide moving with a reciprocating motion, through a crankshaft driven by a motor. The specimens fixed in extensometer transducers are pressed against counter-specimens using bobs.

The tribological tests were carried out in dry friction condi-



Rys. 5. Schemat testera RS 2007

Fig. 5. RS 2007 tester diagram



Rys. 6. Węzeł tarcia testera RS 2007

Fig. 6. RS 2007 tester friction couple

tions, at a constant ambient temperature and constant relative humidity of the air. The tests were carried out for a constant friction distance of 25 km + 10 km, under unit pressure of 30 N and at an average sliding speed of 0.3 m/s.

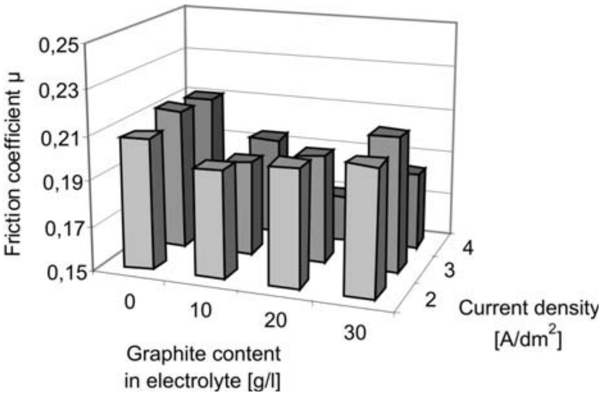
The friction force was recorded by means of a multichannel analog-to-digital converter, *Spider 8*, using the *Catman 4.5*. software. Mass wear of the PEEK/BG material was determined by means of an electronic analytical balance *WA-35* of 0.1 mg accuracy. To determine the influence of the interacting surfaces' microgeometry on the tribological test results, measurement of roughness of the oxide layers was made using a contact profilographometer *Form Talysurf,* with applying the 2D method.

5. Reserach results

Based on data obtained from the tribological test carried out for couples of ceramic-carbon surface layers with the PEEK/BG material, the influence was determined of oxide layers' formation conditions (current density, graphite content in electrolyte, bath temperature) on the friction coefficient value and mass wear of the material. The results are presented in a form of charts for particular temperatures of electrolyte.

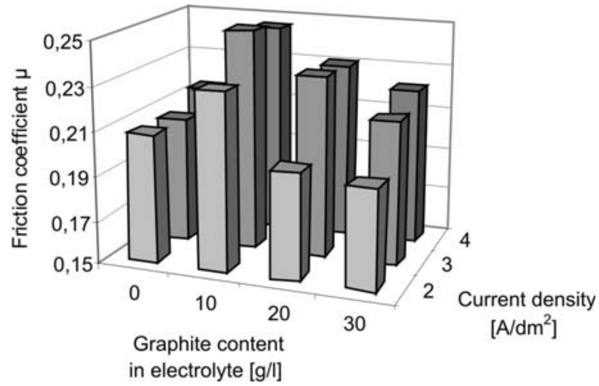
An analysis of the friction coefficient for layers produced at a temperature of 293 K (Fig. 7) suggests that a graphite addition to electrolyte results in decreasing the value of friction coefficient. An increase in current conditions during the electrolysis process results in a decrease of the friction coefficient value for layers produced in the electrolyte with graphite.

In the case of layers formed at a temperature of 303 K (Fig. 8), one can observe an inverse nature of changes of the friction coefficient as a function of current conditions and electrolyte composition**.** For the layers formed in electrolyte with graphite, while the current density value decreases, the friction coefficient decreases as well. An increase of graphite content in the bath results in a decrease of the friction coefficient value. A layer produced at current density of 4 A/dm^2 in electrolyte of a temperature of 293 K and graphite content of 20 g/l achieved the lowest friction coefficient value, i.e. 0.17, among the layers produced in electrolytes containing graphite. The friction coefficient for the layer formed in the same current conditions, in electrolyte of the same temperature but without graphite, amounted to 0.2 l.



Rys. 7. Zależność współczynnika tarcia od gęstości prądowej i rodzaju elektrolitu dla skojarzenia tworzywo PEEK/BG - tlenek glinu-grafit wytwarzanego w temperaturze 293 K

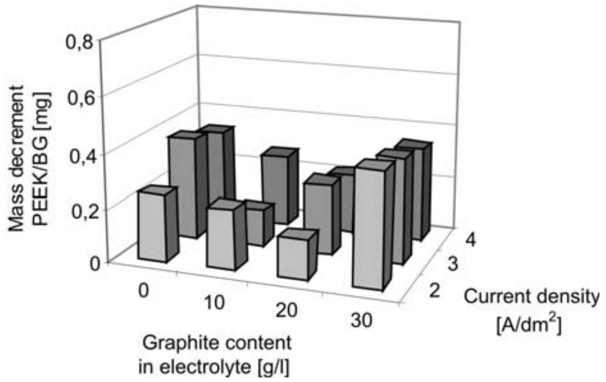
Fig. 7. Dependence of friction coefficient on current density and electrolyte type for the couple: PEEK/BG -aluminium oxide – graphite, formed at a temperature of 293 K



Rys. 8. Zależność współczynnika tarcia od gęstości prądowej i rodzaju elektrolitu dla skojarzenia tworzywo PEEK/BG - tlenek glinu-grafit wytwarzanego w temperaturze 303 K

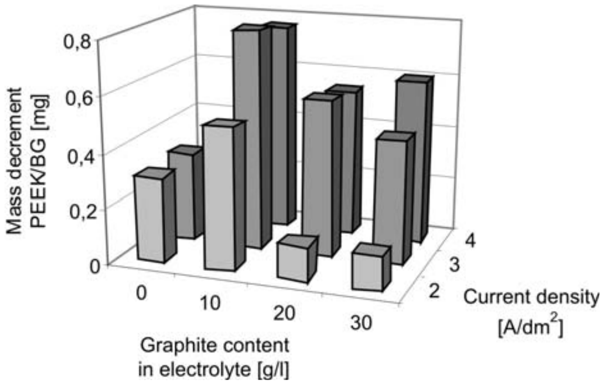
Fig. 8. Dependence of friction coefficient on current density and electrolyte type for the couple: PEEK/BG -aluminium oxide – graphite, formed at a temperature of 303 K

The nature of the PEEK/BG material wear as a function of current conditions and electrolyte composition for layers produced at 303 K (Fig. 9) is identical to the nature of changes in the friction coefficient (Fig. 7). The lowest PEEK/BG mass decrement, i.e. 0.12 mg, was recorded for a specimen interacting with the layers produced at current density of 4 A/dm^2 in electrolyte at a temperature of 303 K with graphite content of 20 and 30 g/l. The mass decrement of a specimen interacting with the layer formed in the same current conditions, in electrolyte of the same temperature but without graphite, amounted to 0.33 mg.



Rys. 9. Zależność zużycia tworzywa PEEK/BG od gęstości prądowej i rodzaju elektrolitu po teście tribologicznym z ceramiczno-węglowymi warstwami wytwarzanymi w temperaturze 293 K

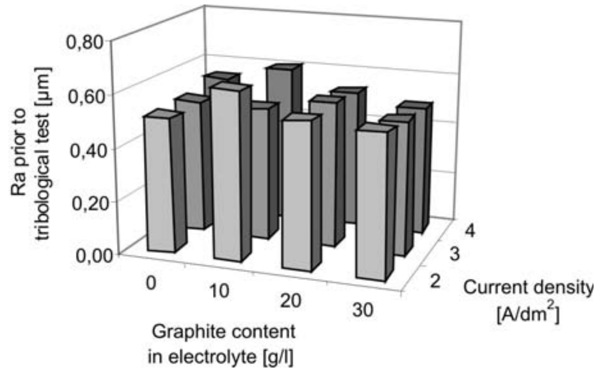
Fig. 9. Dependence of PEEK/BG wear on current density and electrolyte type after tribological test with ceramic-carbon layers produced at a temperature of 293 K



Rys. 10. Zależność zużycia tworzywa PEEK/BG od gęstości prądowej i rodzaju elektrolitu po teście tribologicznym z ceramiczno-węglowymi warstwami wytwarzanymi w temperaturze 303 K

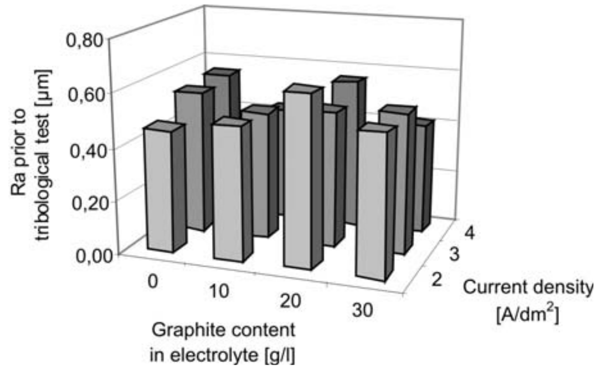
Fig. 10. Dependence of PEEK/BG wear on current density and electrolyte type after tribological test with ceramic-carbon layers produced at a temperature of 303 K

By determining the influence of the microgeometry of ceramic-carbon surface layers on the friction coefficient value and wear of the PEEK/BG material, the basic parameters of the profile were analyzed. Figures 11 and 12 present the measurement of mean arithmetical deviation of the layers' roughness profile before friction. A comparison of parameter Ra of all the oxide layers (0.42-0.64) shows insignificant differences in roughness. The results of the profilographometric examination corroborate that the geometric structure of the layers' surface has no influence on changes of the friction coefficient or wear of the PEEK/BG material during tribological interaction.



Rys. 11. Średnie arytmetyczne odchylenie profilu chropowatości Ra, ceramiczno-węglowych warstw wytwarzanych w temperaturze 293 K

Fig. 11. Mean arithmetic deviation of roughness profile Ra, of ceramic-carbon layers produced at a temperature of 293 K



Rys. 12. Średnie arytmetyczne odchylenie profilu chropowatości Ra, ceramiczno-węglowych warstw wytwarzanych w temperaturze 303 K

Fig. 12. Mean arithmetic deviation of roughness profile Ra, of ceramic-carbon layers produced at a temperature of 303 K

6. Conclusions

Based on the conducted studies and analysis of test results it has been concluded that as a result of a lubricant-free tribological test for a couple consisting of ceramic-carbon surface layers with PEEK/BG, the polymer material is transferred onto the oxide surface. Incorporation of graphite into the structure of oxide layers formed at a temperature of 293 K at high current density values decreases the friction forces in tribological couples with the PEEK/BG material, whereas incorporation of graphite into the structure of oxide layers produced at a temperature of 303 K at low current density values, reduces the wear of the material.

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This paper was financed from KBN funds in years 2007-2009 as Scientific Project N504 051 32/3948.

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Mgr Marek BARA
Prof. dr hab. Władysław SKONECZNY
Mgr Sławomir KAPTACZ
University of Silesia
Department of Materials Science
40-200 Sosnowiec, Śnieżna 2.
e-mail: skoneczn@us.edu.pl