Polish Society of
Composite Materials
Polskie Towarzystwo
Materiałów Kompozytowych

12: 4 (2012) 219-227

COMPOSITES
THEORY AND
PRACTICE

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Received (Otrzymano) 11.04.2012

SELF-LUBRICATING SiC MATRIX COMPOSITES

The research presented here was devoted to the pressureless sintering of composites consisting of a SiC matrix and intentionally introduced particles of solid lubricants as a dispersed phase. The goal of using such materials is to achieve a self-lubrication effect in friction seal elements during sliding contact. In the first part of the investigations, the optimal conditions of sintering, as well as the best composition and concentration of the solid lubricants were established. The second part of the work consisted in characterizing such mechanical and tribological properties of the materials as: hardness (HV and HK), bending strength, abrasive wear resistance, friction coefficient and linear wear in contact parts made of homonymous and heteronymous materials. Based on the tribological and mechanical studies correlated with microstructure investigations (SEM), the material fulfilling the requirements for the construction of self-lubricating seal elements was selected.

Keywords: pressureless sintering, SiC matrix composite, face seal, self-lubricant, solid lubricant

KOMPOZYTY WĘGLIK KRZEMU-GRAFIT O WŁAŚCIWOŚCIACH SAMOSMARUJĄCYCH

Przedstawiono badania poświęcone spiekaniu bezciśnieniowemu kompozytów, w których matrycę stanowił SiC, a fazą rozproszoną były celowo wprowadzane smary stałe. Zadaniem takich tworzyw jest uzyskanie efektu samosmarowania pomiędzy pracującymi w styku ślizgowym elementami uszczelnienia czołowego. W pierwszej części pracy określono warunki wytwarzania kompozytów i dokonano pełnej ich charakterystyki. W tym celu, po przeprowadzonym spiekaniu, na podstawie badań gęstości pozornej i względnej kompozytów, pomiarów składu fazowego i obserwacji mikrostruktury określono warunki spiekania i dokonano wyboru najlepszego smaru stałego. W drugiej części przeprowadzono charakterystykę właściwości mechaniczno-tribologicznych wybranych kompozytów. Określono twardość (HV i HK), wytrzymałość na zginanie, odporność na zużycie ścierne, wartość współczynnika tarcia i zużycia liniowego w styku jedno- i różnoimiennym. Na podstawie wymienionych parametrów tribologicznych połączonych z obserwacjami mikrostruktury wyselekcjonowano kompozyt spełniający wymogi stawiane materiałom służącym do budowy elementów konstrukcyjnych samosmarujących się uszczelnień czołowych.

Słowa kluczowe: spiekanie bezciśnieniowe, kompozyt z matrycą SiC, uszczelnienie czołowe, samosmarowanie, smar stały

INTRODUCTION

Friction and wear are phenomenons, that significantly influence the life time and failure-free use of machine elements, in industry as well as in daily life. In some cases, such occurrences are desirable, in other cases there is an aspiration to eliminate it. Significant lowering of friction is desired in face seals, elements of pumps, especially in self-lubricating face seals [1, 2]. Face seals are made of diverse materials, including from ceramics. They should fulfil a number of requirements which are shown in Table 1 [3].

One of the most commonly used ceramic materials for manufacturing face seals is silicon carbide. Silicon carbide is in common use because it fulfils a number of conditions imposed upon materials for elements of face seals. It is hard (HV = 25÷30 GPa), has a high Young's modulus (400 GPa), low thermal expansion coefficient α ($3{,}5\cdot10^{-6}$ to $4{,}2\cdot10^{-6}$ 1/°C in range of 20÷400°C), very high chemical resistance and good mechanical parameters [4]. It is usually used as a dense, mono-phase sinter but also as a matrix in self-lubricating materials. The self-lubricating effect can be achieved in two ways. The first way is by producing a controlled porous microstructure in the matrix [5, 6]. The intentionally introduced lubricant gathers in pores due to surface tension action, and during sliding it is released and deposits on the friction pair elements and facilitates sliding (Hexoloy® SP material produced by Saint-Gobain Ceramics) [7]. The second way is to produce granular composites in which so-called solid lubricants are present as a dispersed phase. Solid lubricants are materials with a layer structure and low shearing strength e.g. graphite, hBN and MoS_2. As examples of commercial materials which in the silicon carbide matrix there is a dispersed phase of solid lubricant - graphite, could be mentioned: SC-DSG produced by Coorstek and Ekasic® G by ESK Ceramics GmbH [8, 9].

TABLE 1. **Properties of ceramics useful for mechanical seals [3]**

TABELA 1. **Właściwości materiałów ceramicznych użyteczne w uszczelnieniach mechanicznych [3]**

Desired property	Benefit
Hardness	Increased wear resistance
High Young's modulus	High dimensional stability
Bending strength	Work under heavy load
Thermal shock resistance	Work with temperature gradients
Low thermal expansion coefficient	Retain stable dimensions
Corrosion resistance	Possibility of work in highly corrosive environment
Low chemical reactivity	

The aim of this work was to produce by pressureless sintering self-lubricating composites with a silicon carbide matrix and to accomplish full characterisation of their mechanical and tribological properties.

MATERIALS AND METHODS

The first preparation step was to manufacture granulates of solid lubricants or their precursors. Towards this goal, Nowolak MR (*Organika-Sarzyna*) phenol formaldehyde resin was used for binding grains of graphite, graphite mixed with waste wood dust (1:1 vol.), wood dust and hBN. Binding the grains of hexagonal boron nitride was done using acrylic resin. The additive of the Nowolak MR resin, as well as acrylic resin, was about 1 wt.%, per total mixture mass. The granulates were obtained by rubbing the mixtures, after wet homogenisation of the components, through a Nylon 6 sieve, subsequently sieving them through sieve with a mesh size of 0.056 mm. Granulates obtained in this way were mixed in a dry state with a silicon carbide granulate for sintering, Sika FCP 15 (*Saint-Gobain*). The granulates were mixed with the silicon carbide granulate in the amount about 15 vol.%

Another way to obtain a graphite dispersed phase (one of solid lubricants) in a SiC matrix, was to introduce into pure silicon carbide powder an excessive additive of Nowolak MR resin. During the investigation on silicon carbide sintering, it was ascertained that the necessary additives permitting one to obtain dense SiC materials are boron and carbon [10, 11]. The best way to introduce carbon is to use a phenol formaldehyde resin. The resin is also a sliding agent facilitating sample forming, which additionally introduces considerable amounts of carbon. According to the pyrolysis of resin, it is about 50% of the resin mass. Exceeding the resin additive optimal for sintering results in a graphite dispersed phase in a carbide matrix [10]. To obtain a graphite-silicon carbide composite, pure silicon carbide powder with additives of boron (0.5 wt.%) and carbon (4 wt.%) such as the Nowolak MR resin, was combined with an excessive additive of resin (8 wt.%). Homogenisation was conducted in a wet state, in ethyl alcohol. After alcohol evaporation, the mixture was rubbed through a Nylon 6 sieve to obtain a granulate.

Green samples were manufactured from the SiC granulate with an 8 wt.% excessive additive of Nowolak MR resin and from mixtures of solid lubricant granulates with the SiC Sika FCP 15 granulate. Uniaxial, two-sided pressing in a steel die to form green samples with a 20mm diameter and height of 5÷6 mm was used. Sintering was conducted according to the scheme presented in Figure 1.

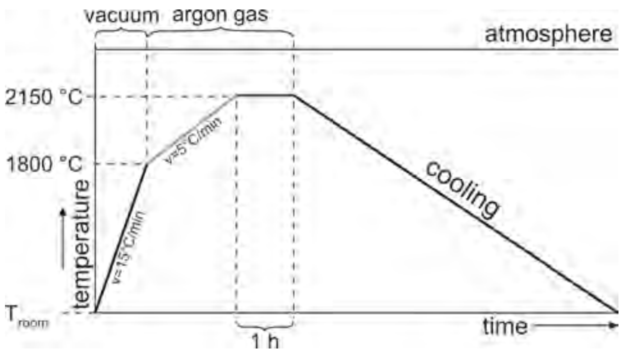


Fig. 1. Composite pressure-less sintering process scheme

Rys. 1. Schemat procesu spiekania kompozytów

The best materials were selected based on the sinter apparent density measurements (Archimedes method), phase composition analysis (XRD method) and microstructure observation (Nikon Epiphot 300 optical microscope; Jeol 5400 and Nova Nano SEM 200, FEI Company scanning electron microscopes).

Subsequently, samples were manufactured from selected materials with differing amounts of solid lubricants or their precursors, to verify the possibility of using them as elements of friction seals, regarding their mechanical and tribological properties.

This investigation concentrated on selecting the best solid lubricant additive and ascertaining the optimal amount of such an additive. The material properties were measured: hardness using the Vickers and Knoop methods (load 9.81 N), bending strength using the biaxial bending method, and elastic properties i.e. Young's modulus, Kirchhoff modulus and Poisson ratio, using the ultrasonic method. The tribological properties were investigated by measuring the abrasion wear resistance with a loose abrasive (*dry sand test*) and the material wear in sliding contact, simultaneously by determining the friction coefficient. The measurements of the friction coefficient of the material wear in sliding contact in contact with homonymous (*pin-on-disc)* and heteronymous materials (*ball-on-disc*) were done. In contact with homonymous materials, both the pin and disc were made from the investigated materials, in the heteronymous pairs, the pin was made from a spherical silicon nitride polycrystal (10 mm in diameter) and the disc was made from the investigated materials. The tribological tests were done at the constant slide velocity of 0.35 m/s and pressure 1 MPa (*pin-on-disc*) and ~1 GPa (*ball-on-disc*). All the research mentioned above was correlated with micro-structure observations using optical and electron microscopy.

The measurements of the mechanical and tribological properties were compared with samples manufactured from monophase silicon carbide. Samples of monophase silicon carbide were manufactured from the granulate for sintering, Sika FCP 15 (*Saint-Gobain*).

RESULTS AND DISCUSSION

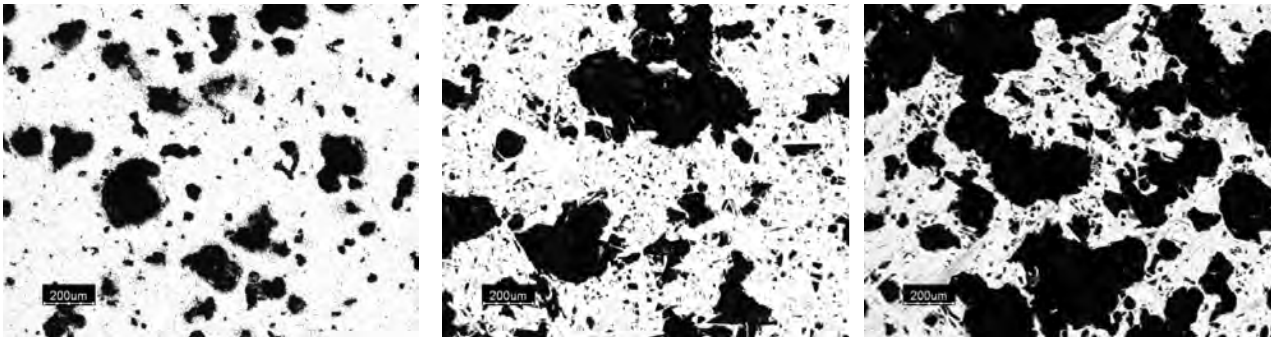
In Table 2 and Figure 2 are presented the results of the density investigations, phase composition and microstructure of composites SiC-15 vol.% different granulates of solid lubricants and composite SiC-8 wt.% of excess amount of Nowolak MR resin. The best solid lubricants for use in silicon carbide matrix were selected based on the presented investigations.

Taking into account the investigation results shown in Table 2 and in Figure 2, the most promising solid lubricants, among the potential additives, are graphite granulate, graphite-wood dust granulate and the excess additive of Nowolak MR resin. The materials with the above-mentioned additives posses a high densification ratio >90% (taking into account amounts of additives introduced into matrix), are uniformly distributed in the SiC matrix and do not tear out during the grinding and polishing of a cross-section (Fig. 2). Moreover the preliminary research confirmed that the phenol formaldehyde resin Nowolak MR and wood dust undergo graphitisation during sintering of the silicon carbide matrix (Table 3 and Fig. 3).

TABLE 2. **Basic properties of SiC matrix composite with additive of various solid lubricants**

TABELA 2. **Podstawowe właściwości tworzyw kompozytowych na bazie SiC z różnymi dodatkami smarów suchych**

Solid lubricants		XRD analysis	Apparent density [kg/m^3]	Theoretical density [kg/m^3]	Relative density [%]
Granulates	(graphite+wood dust 1:1 vol.)/ Nowolak MR	SiC, graphite	2700	3010	90%
	graphite/ Nowolak MR	SiC, graphite	2820	3060	91%
	wood dust/ Nowolak MR	SiC, graphite	2560	2840	89%
	hBN/Nowolak MR	SiC, B_4C	2600	2990	87%
	hBN/acryl resin	SiC, B_4C.	2080	3000	69%
Excess amount of Nowolak MR resin		SiC, graphite	2960	3070	97%


wood dust/Nowolak MR granulate; 15 vol.% hBN/Nowolak MR; 15 vol.% hBN/acryl resin; 15 vol.%

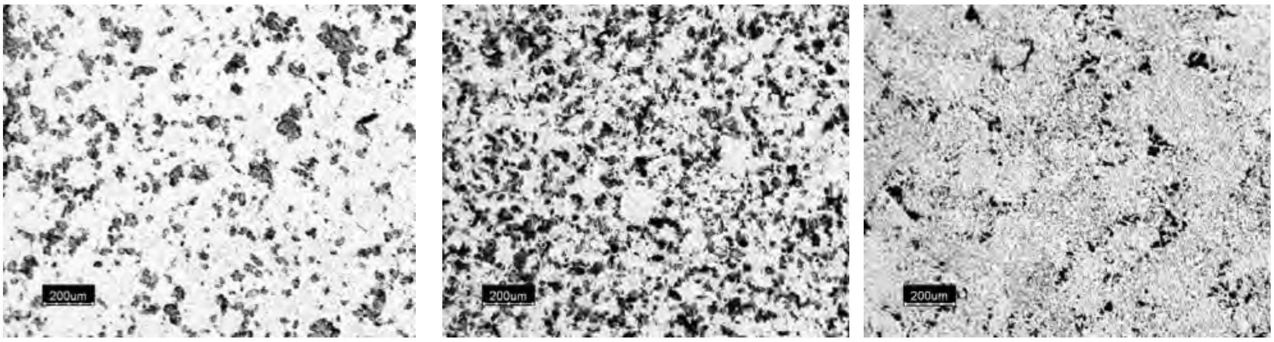

graphite/Nowolak MR; 15 vol.% (graphite+wood dust 1:1 vol.) /Nowolak MR; 15 vol.% excess additive of Nowolak resin; 8 wt%

Fig. 2. Microstructure of SiC composites with various solid lubricant additives (optical microscopy)

Rys. 2. Mikrostruktury kompozytów na bazie SiC z dodatkiem różnych smarów stałych (mikroskop optyczny)

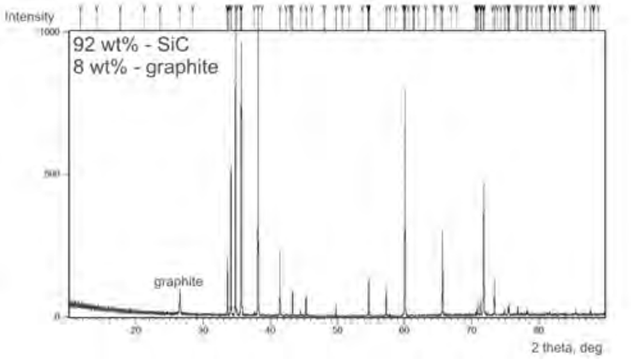


Fig. 3. XRD pattern of sample with excess additive of Nowolak MR resin

Rys. 3. Przykładowy dyfraktogram próbki z nadmiarowym dodatkiem żywicy Nowolak MR

However, in the materials where hBN was used as the potential solid lubricant, the X-ray diffraction did not show the presence of hBN in the sinters, instead, boron carbide was identified. It should be presumed that the introduced hBN is unstable in the temperatures and conditions of SiC matrix sintering. The microstructure of the samples containing the additive of hBN (Fig. 2) shows very numerous pores, which could be a result of the preparation of the metallographic specimens or reaction of the introduced additive of boron nitride with the matrix during sintering.

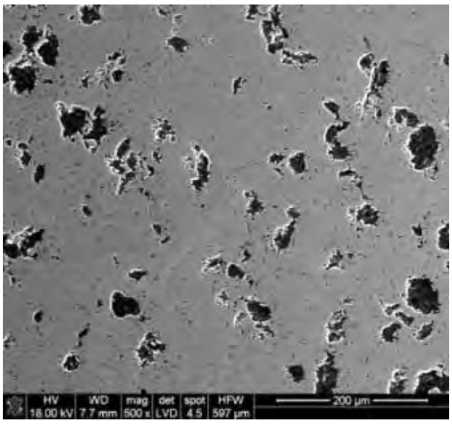
Based on that research, the following additives of solid lubricant or their precursors were chosen for further investigation: graphite granulate, graphite-wood dust granulate and excess additive of Nowolak MR resin. Selected on the basis of the initial investigation, the granulates of solid lubricants or their precursors were introduced in different concentrations into the carbide matrix (Table 3). It was determined that regardless of the content of the additive, all the composites achieved a high densification ratio, above 90% theoretical density (Table 3).

TABLE 3. **Phase composition and densities of SiC composites with different amounts of selected solid lubricants**

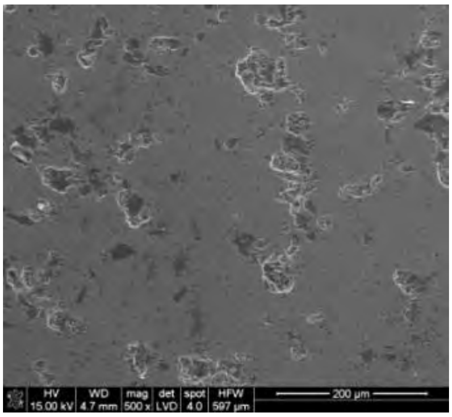
TABELA 3. **Skład fazowy i gęstości kompozytów na bazie SiC z wyselekcjonowanymi dodatkami smarów stałych**

Solid lubricants		Phase composition XRD analysis	Amount of solid lubricant additive	Apparent density [kg/m^3]	Theoretical density [kg/m^3]	Relative density [%]
Granulates	(graphite+ wood dust 1:1 vol/ Nowolak MR	SiC, graphite	10 vol.%	2950	3090	96%
			15 vol.%	2830	3030	93%
			20 vol.%	2700	2970	91%
	graphite/ Nowolak MR	SiC, graphite	10 vol.%	2970	3090	96%
			15 vol.%	2960	3030	98%
Excess amount of Nowolak MR resin		SiC, graphite	8 wt%	2820	3090	91%

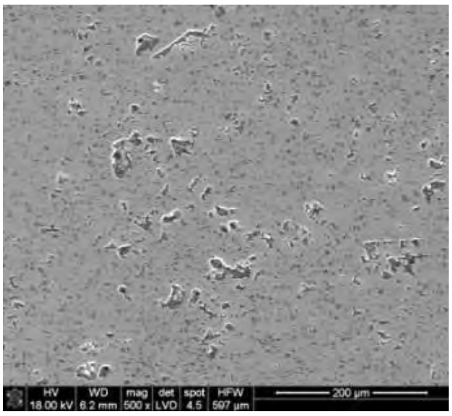
The apparent density decreases with an increasing concentration of additives used as solid lubricants. Such behaviour results from an increase in the volumetric fraction of the graphite phase with a lower density (2000 kg/m^3) in comparison to the density of the SiC matrix (3210 kg/m^3). Silicon carbide and graphite in the material phase composition were identified by the XRD method (Fig. 3 and Table 3). Using the elemental composition analysis EDS (Fig. 5), the phases composing the composites could be differentiated on SEM micrographs (Fig. 5). It could also be noticed that the graphite-dispersed phase is uniformly distributed in the carbide matrix (Fig. 4).



graphite granulate, 15 vol.%



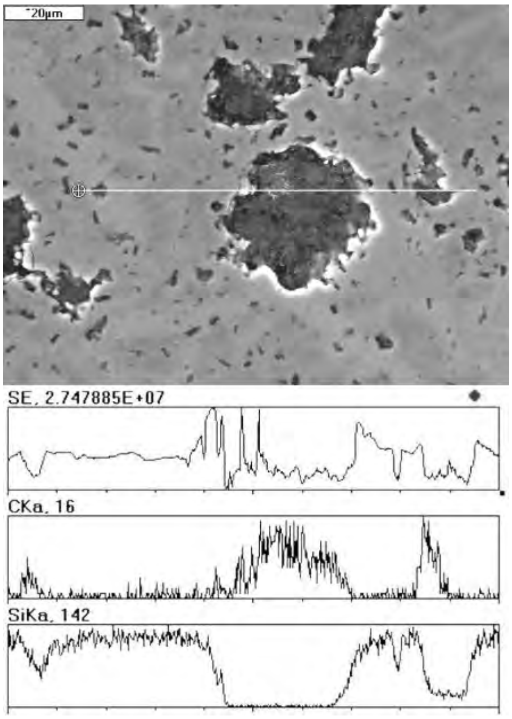
graphite-wood dust granulate, 15 vol.%



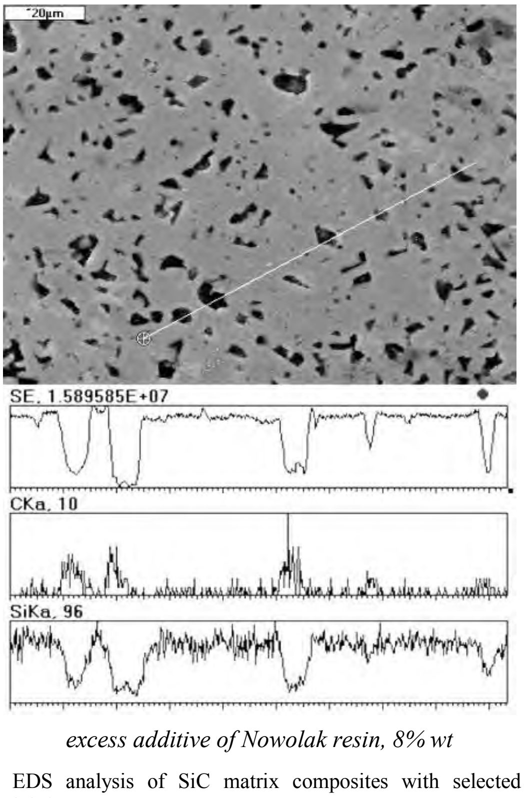
Excess additive of Nowolak resin, 8% wt

Fig. 4. SEM microstructures of SiC matrix composites with selected solid lubricants

Rys. 4. Mikrostruktury kompozytów na bazie SiC z wyselekcjonowanymi smarami stałymi



graphite-wood dust granulate, 15 vol. vol.%



excess additive of Nowolak resin, 8% wt

Fig. 5. EDS analysis of SiC matrix composites with selected solid lubricants

Rys. 5. Analizy EDS kompozytów SiC z wyselekcjonowanymi smarami stałymi

In almost the materials there are numerous large dispersed phases of carbon present. Smaller, but no less numerous dispersed phases of carbon (carbon originated in Nowolak MR pyrolysis) are present in the material with the resin excess additive. It could be presumed, that the presence of a graphite phase influences all the investigated mechanical and tribological properties.

Hardness is a resultant of the volumetric fractions of phases forming the composite, i.e. graphite and SiC. A lower hardness value was the consequence of a bigger volume fraction of a softer composite phase - graphite (Table 4).

The introduction of graphite and graphite-wood dust granulates results in lowering of the sinter density (Table 3). A higher amount of graphite or graphite-wood dust additives results in a higher porosity of the samples (Table 3), higher volumetric fraction of graphite in the samples and lower bending strength (Fig. 6). The highest strength (~300 MPa) among the group of graphite-carbide composites is achieved by the samples with a graphite dispersed phase, whose precursor was the graphite-wood dust granulate (10÷15 vol.%) and with graphite resulting from pyrolysis of the excess additive of Nowolak MR resin.

TABLE 4. **Mechanical properties of SiC matrix composites with different additives of selected solid lubricants**

TABELA 4. **Właściwości mechaniczne kompozytów na bazie SiC z różnymi dodatkami wyselekcjonowanych smarów suchych**

Samples		Amount of solid lubricant additive	Young Modulus E [GPa]	Vickers Hardness HV1.0 [GPa]	Knoop Hardness HK1.0 [GPa]
monophase SiC (*Sika*)		-	450 ± 9.2	23.5 ± 1.4	19.2 ± 0.8
SiC+solid lubricants					
Granulates	(graphite+ wood dust 1:1 vol.)/Nowolak MR	10 vol.%	304.97 ± 15.04	20.1 ± 3.8	15.3 ± 2.7
		15 vol.%	306.06 ± 15.01	20.2 ± 5.7	12.5 ± 2.5
		20 vol.%	325.14 ± 8.93	13.4 ± 4.7	10.8 ± 3.9
	graphite Nowolak MR	10 vol.%	284.80 ± 11.24	21.8 ± 4.2	18.4 ± 2.4
		15 vol.%	231.69 ± 0.05	18.0 ± 3.9	13.6 ± 3.7
Excess amount of Nowolak MR resin		8 wt%	292.19 ± 7.92	19.4 ± 2.1	15.0 ± 0.9

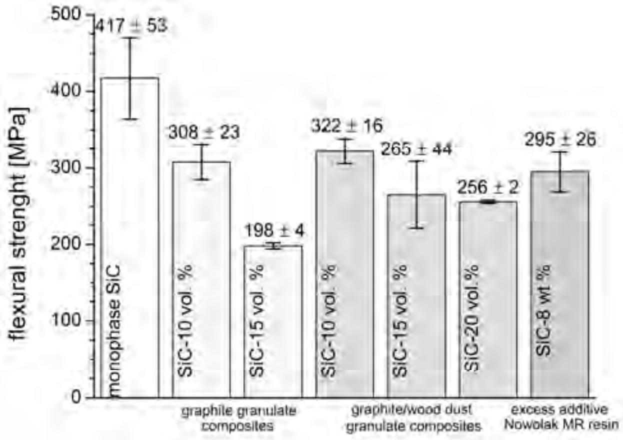


Fig. 6. Bending strength of composites SiC - solid lubricants

Rys. 6. Wytrzymałość na zginanie kompozytów SiC - smary stałe

In Figure 7 the abrasion wear of graphite-silicon carbide composites is presented. The highest abrasion wear resistance (least amount an abrasion wear) among the group of graphite-silicon carbide composites were the samples where respectively 10 and 15 vol.% graphite-wood dust granulate was introduced. Subsequently, the lowest abrasion wear resistance was achieved by the material into which the excess additive of Nowolak MR resin was introduced. The reason for such a state of things could be the differences of ways that the SiC matrix is formed and the size of the resulting graphite dispersed phase (Figs. 4 and 5).

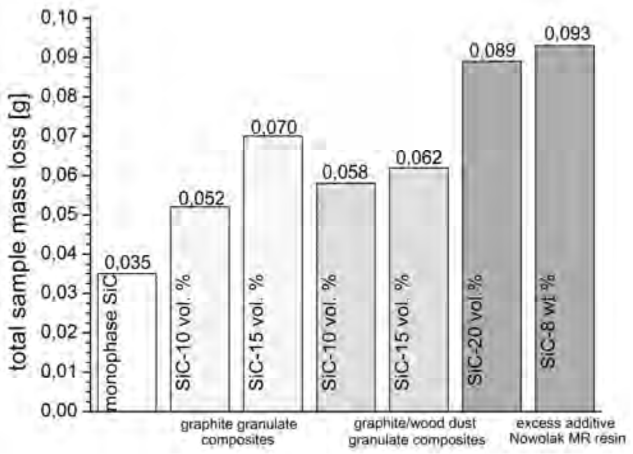


Fig. 7. Results of dry-sand-test for composites SiC-solid lubricants

Rys. 7. Wyniki dry-sand-testu kompozytów SiC - smary stałe

Tests in the pin-disc configuration carried out in contact with homonymous materials (Fig. 8) unequivocally show that the pair made of the material into which 15 vol.% of graphite-wood dust granulate was introduced demonstrates the most stable character of interaction. This is attested by the narrowest range of friction coefficient values, as well as in comparison to other materials, the lowest modal value of μ coefficient. Besides, among all the investigated materials, the abovementioned one showed the lowest linear wear during testing. The opposite character of interaction is found in the pair made of monophase silicon carbide. The character of interaction in the monophase SiC is aggressive, which is confirmed by instability (sudden step increases of μ) and the wide range of friction coefficient values from 0.33 to 0.68. The linear wear of the SiC-SiC pair is significant and amounts to 49.9 μm, and its course has a step character - there could be differentiated step changes in the value. Subsequently, in the other investigated materials the character of interaction varies, with sudden jumps in the friction coefficient value (material with excess additive of Nowolak MR resin), significant linear wear of the sample (material with 20 vol.% of graphite-wood dust granulate), or both of those behaviours simultaneously (material with 10 vol.% graphite granulate), but with a lower intensity than in the monophase SiC sample.

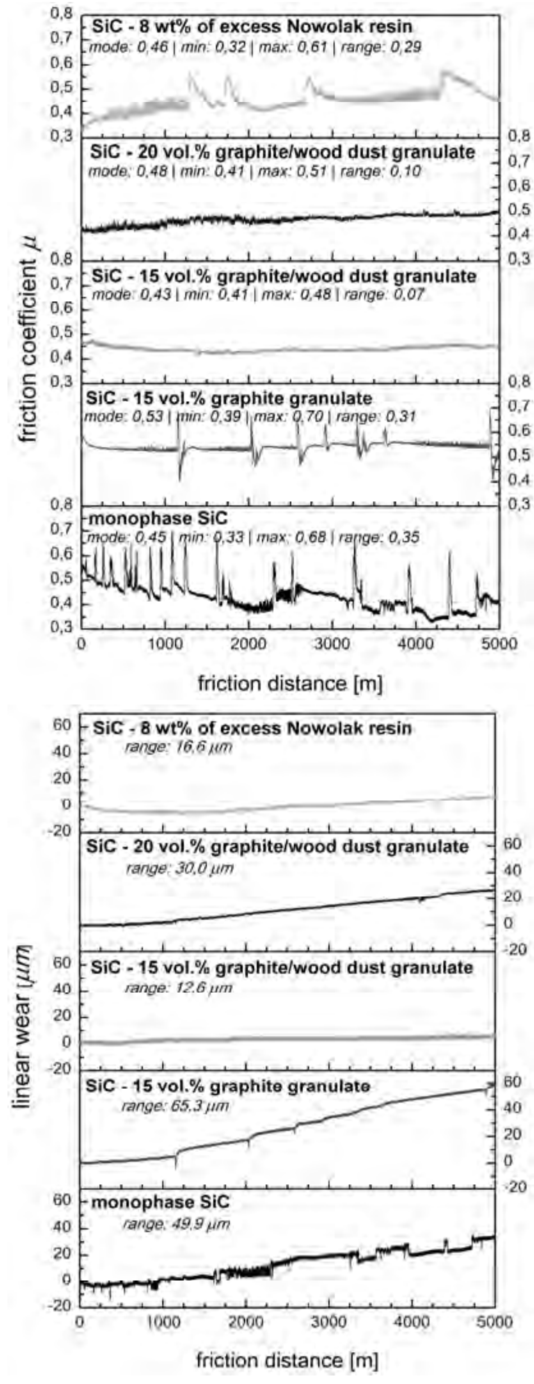


Fig. 8. Friction coefficient and linear wear as function of friction displacement, determined for contact of homonymous materials

Rys. 8. Współczynnik tarcia i zużycie liniowe w funkcji drogi tarcia określone dla par jednoimiennych

The character of interaction in the investigated contact directly influences the surface quality in the place of contact after wear (Figs. 9 and 10). In all the investigated materials the presence of chemical compounds resulting from wear could be identified. The EDS analysis shown in Figure 9, illustrates that they are probably the effect of SiC matrix oxygenation, i.e. silicon oxide (IV) SiO_2. This product deposits on surface irregularities or on places where graphite was taken out. It could be said that in the case of rubbing contact made of monophase silicon carbide, abrasion wear caused by micro cutting and tearing out or crumbling out of grains in the working surface dominates (Fig. 10a). Additionally wear is magnified by particles (wear debris) present between the interacting surfaces of the sample and counter-sample. In the case of the material with the best tribological properties (Fig. 10b) the wear is limited, probably due to an adequate concentration of the graphite dispersed phase. The wear surface of the material is devoid of distinct irregularities (Fig. 10b). It could be supposed that the graphite present in the dispersed phase is taken out of material surface and greased during wear. Thus it successfully fulfils the role of solid lubricant.

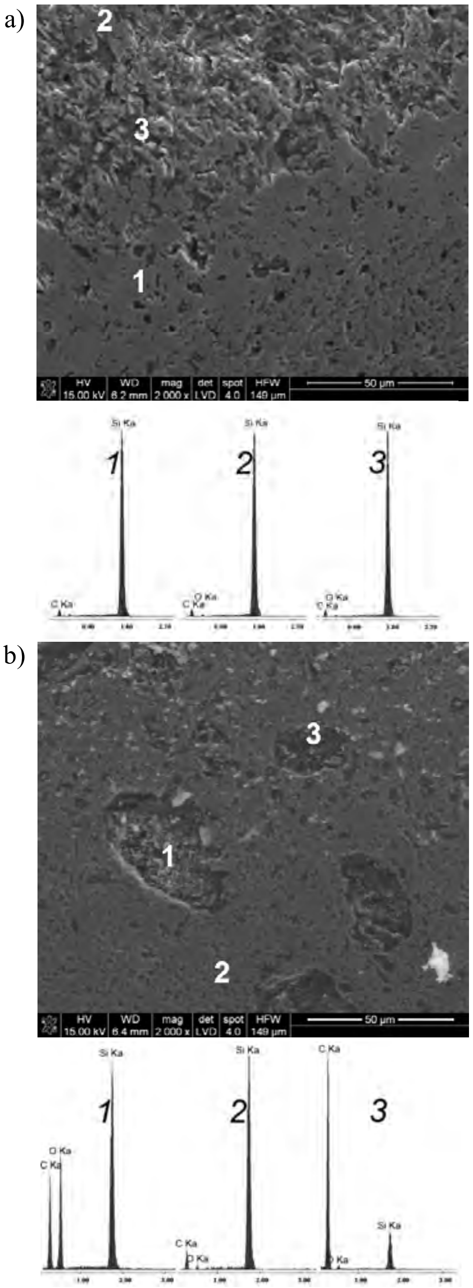


Fig. 9. Results of EDS analysis of friction surface in samples made of monophase SiC (a) and composite containing 15 vol.% graphite wood dust granulate (b)

Rys. 9. Wyniki analizy EDS powierzchni tarcia próbek wykonanych z czystego SiC (a) i kompozytu zawierającego 15% obj. granulatu grafit-pył drewniany (b)

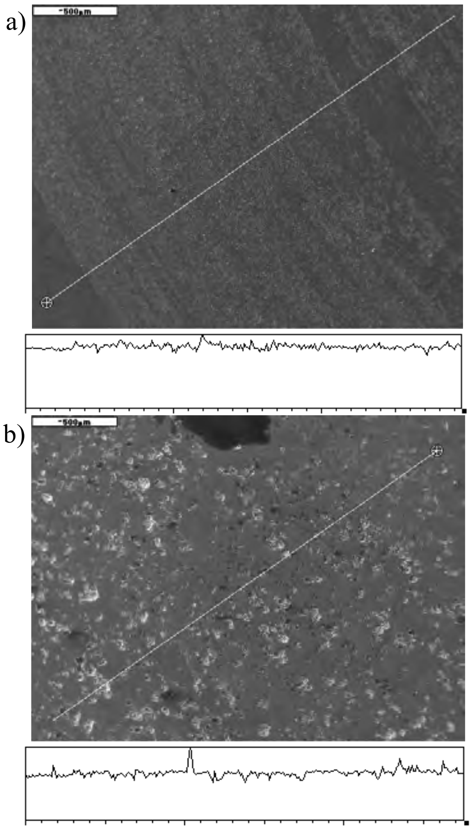


Fig. 10. Profile of friction surface on discs made of monophase SiC (a) and composite containing 15 vol.% graphite wood dust granulate (b)

Rys. 10. Profil powierzchni tarcia tarcz wykonanych z czystego SiC (a) i kompozytu zawierającego 15% obj. granulatu grafit-pył drewniany (b)

On the best material, the tribological tests in contact parts made of heteronymous materials (*ball-on-disk*) were conducted. The results of the measurements are shown in Figure 11.

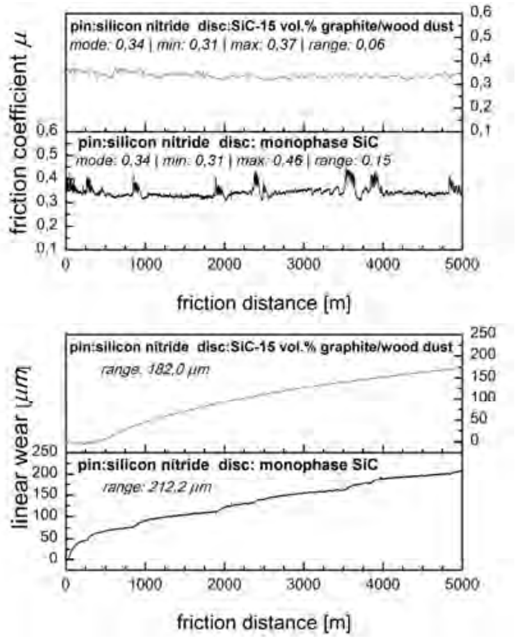


Fig. 11. Friction coefficient and linear wear vs. friction distance determined for contact of heteronymous materials

Rys. 11. Współczynnik tarcia i zużycie liniowe w funkcji drogi tarcia określone dla par różnoimiennych

The linear wear shown in Figure 11, in both the analysed cases is much higher than during the tests with contact of homonymous materials. The most probable reason for such a state of things is the geometry of the friction pair at the concentrated point of contact. Analogous to the results of the investigation of the composite with the 15 vol.% additive of graphite-wood dust granulate in contact with homonymous materials, also in this case it could be presumed that the dispersed phase of graphite acts like a solid lubricant, which could explain the stability and narrow range of the friction coefficient value. As in the contact of homonymous materials, also in this configuration, graphite is most probably taken out of the dispersed phase and distributed on the wear track, which could be seen on the microscopic pictures (Fig. 12).

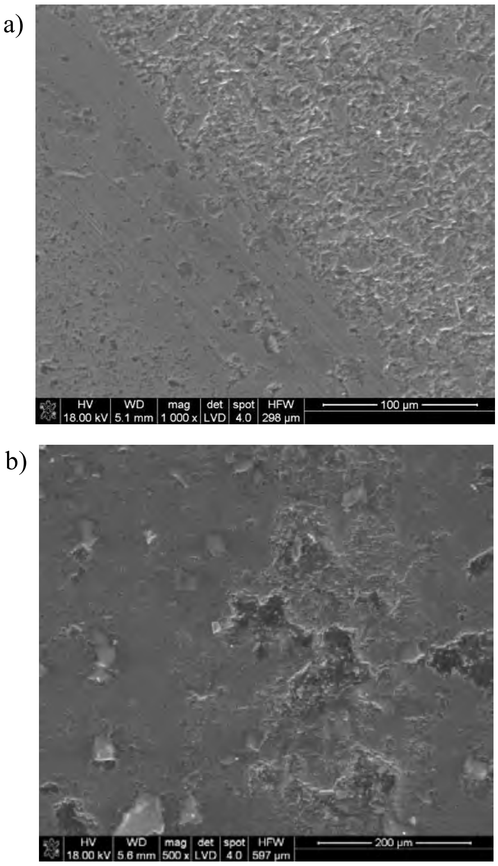


Fig. 12. SEM micrographs of wear track surface in contact of heteronymous materials made of pure SiC (a) and composite with 15 vol.% graphite-wood dust granulate (b)

Rys. 12. Mikrofotografie SEM powierzchni toru tarcia w różnoimiennym skojarzeniu ciernym tarcz wykonanych z czystego SiC (a) i kompozytu zawierającego 15% obj. granulatu grafit-pył drewniany (b)

Subsequently in the contact zone, relatively soft wear products which do not cause significant surface destruction are also present. As a result of such action by the graphite lubricant, the contact surface is devoid of irregularities, in contrast to the significantly degraded surface of the monophase SiC (Fig. 12). The greatly worn out interaction surface and sudden increases in the μ coefficient (Fig. 11) may attest to the domination of abrasion wear as a result of cracking and tearing grains out of the surface during the interaction of the pair of pure SiC-Si_3N_4. During the wear of both contact parts made of heteronymous materials, just as during the interaction of contact of homonymous materials, a similar product of chemical reactions accompanying the interaction of the tribologic pair are generated. As stated in literature, they can have a beneficial influence on the tribologic pair interaction and lessen the friction between the structural elements.

CONCLUSIONS

In this work it was shown that:

1. It is possible to manufacture by pressureless sintering SiC matrix composites with self-lubricating properties.
2. Among the various additives that can be used as solid lubricants, the best are graphite and wood dust granulate, in which phenol formaldehyde Nowolak MR resin was used as the binder.
3. Both precursors i.e. wood dust and resin, undergo graphitisation during carbide matrix sintering.
4. The optimal amount of solid lubricant additive was about 15 vol.% and guarantees the best combination of mechanical and tribological properties of the obtained composite, i.e. high hardness (HV ~19 GPa, HK ~14 GPa), good mechanical strength (~300 MPa), abrasive wear resistance at an acceptable level with a stable interaction character in the contact of homonymous and heteronymous materials, additionally with low linear wear.

Acknowledgments

The paper reports some results of research sponsored by the European Union Structural Funds in Poland under contract UDA-POIG.01.03-12-024/08-00 and realized by a scientific industrial consortium, the research being partly dedicated to complex investigations of the properties of new generations of cutting tools with the goal to develop cost-effective and environmentally friendly methods for ceramic materials designed for high speed machining of difficult workable work pieces and Statutory Researches no. 11.11.160.364.

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