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Friction films analysis and tribological properties of composite antifriction self-lubricating material based on nickel alloy

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This article analyzes the composition and distribution of chemical elements in friction films and their effect on the tribological properties of the self-lubricating, high-temperature antifriction composite based on EP975 powder nickel alloy with CaF_2 solid lubricant. Analysis of the chemical elements by energy-dispersive spectroscopy (EDS) showed their uniform distribution, on both the composite's surface and the counterface's surface. The alloying elements' uniform distribution leads to a uniform distribution of the corresponding phases and structural elements in the antifriction film. This ensures high tribological properties at high temperatures. Analysis of the material's tribological properties, by means of metallographic and micro-X-ray research confirmed the correctness of the technology for producing the composite. Solid lubricant CaF_2, alloying elements, and their corresponding phases form the continuous antiscoring film. The film influences the antifriction properties formation during the friction process and provides a self-lubricating mode under the action of high temperature and oxygen. Antiscoring, self-lubricating CaF_2 films minimize wear of the friction pairs and defend the contact surfaces against intensive wear. The dense antifriction films have smooth microtopography, which stabilizes the high-temperature friction unit operation. Thus, the self-lubrication mode is realized for a long exploitation time. Tribological properties analysis allowed us to determine the ranges of rational exploitation modes for the material being studied: a load up to 5.0 MPa, a slide speed from 0.3 to 1.0 m/s, a temperature up to 800°C, in the air. The results obtained opened the opportunity to control the antifriction film formation and the composite's tribological properties by the choice of the initial ingredients while taking into account the operating conditions.

Keywords: *composite, powder nickel alloy, solid lubricant, chemical elements, antifriction film, self-lubrication*

1. Introduction

Improving the reliability and durability of machines and mechanisms is inseparably linked with an increase in the parts' resistance to wear of various types. It is the wear of the parts' contact surfaces that leads to the destruction of machine parts and of the equipment as a whole. One of the main problems is that of creating and using new materials for contact pairs, particularly antifriction materials operating in difficult operating conditions at high rotational speeds or high temperatures in an oxidizing environment. The application and production process of metal matrix composites (MMCs), which are commonly used in friction pairs, are described in *Composite Materials Engineering, Volume 1: Fundamentals of Composite Materials* [1]. In K. Olaleye et al. and J. Cheng et al. [2, 3], the authors demonstrated the influence of using materials such as nickel-based lubricating composites on the wear rate of the friction surface. As the literature indicates, this is an extremely

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important phenomenon from the point of view of bearings and joints operating at high temperatures and high rotational speeds [4].

Powder metallurgy methods have no alternative for mitigating this problem, but powder technology is not used enough. This is because of insufficient research on the available processes used on friction surfaces, in particular, self-lubricating antifriction materials working without liquid lubricant, when solid lubricant is added to the initial charge. It should also be emphasized that the use of pure metal powders is often very limited due to their high surface energy and high affinity for oxygen. This reduces the efficacy of these composites at high temperatures. Therefore, the process of producing pure metal powders requires additional purification from oxygen. This involves additional technological and financial costs. The well-known high-temperature composites based on pure nickel powders are no exception and have significant disadvantages. In particular, powders of alloying elements W, Mo, Ti, Al, which strengthen the Ni matrix, have a high affinity for oxygen. This leads to the formation of a large amount of oxides in the metal matrix and rapid deterioration of properties (see references [4, 5]). For this reason, existing nickel composites made from pure powders are only able to demonstrate satisfactory tribological properties under limited operating conditions. Therefore, it is more efficient to create high-temperature nickel composites from nickel alloys containing powders rather than from pure powders. An insufficiently complete understanding of the processes that occur on the friction surface of materials during service under extreme conditions (high temperatures and velocities, heavy loading, and aggressive media), and their effect on the wear resistance of the materials is one of the main obstacles to the widespread use of powder technology. Therefore, continued success in the development of new self-lubricating antifriction composites and provision for their optimal operating conditions will depend on profound comprehension of the mechanisms responsible for friction and wear under the influence of external factors and its accompanying phenomena. Dynamic chemical, deformation, and thermal processes constantly occur during friction on the surface of materials, which lead to changes in the functional properties of the materials [4]. The impact of this process has been widely described by the authors of sources in the literature [4, 6, 8–17]. This applies to ceramic materials [6, 8, 11–14], plastics [10, 15], and composites with a metallic lubricating film [9]. Here, the wear resistance of the composites is determined not so much by their initial characteristics as by the properties of the films that form on their surfaces, as indicated by Olaleye et al. [2] and Jamroziak and Roik [6]. Additionally, the effect of using selected solid-based lubricants and the process of forming lubricant films has been extensively described in the literature [8–10]. Because the laws established in many studies for friction processes are special in nature and valid only within the narrow bounds of a specific experiment, further study of the phenomena is of the greatest interest, primarily, the condition of the surface layer, which exerts a decisive influence on the performance of friction couples.

The authors cited [2, 6, 8–14, 18, 19] discovered and confirmed that the wear resistance of antifriction composites depends not only on their initial properties, but also on the properties of the surface friction films that form, which are secondary structures. The nature of such friction films, their elemental and phase composition, directly affect the antifriction behavior of the composites and determine their wear resistance. Such secondary structures are found when CaF_2 additions are used in composites based on a nickel matrix [2, 6] or MoS_2 [8, 13]. It also occurs in ceramic mixtures [11, 12]. Secondary structures and tribological responses are important from the point of view of cooperating elements in the arms industry [18] and railways [19]. It was found that the friction film's composition and structural features are responsible for the level of tribological properties. The formation of this type of lubricant film in composites based on metal matrix and ceramic particles has been extensively described in the literature [6, 8, 9, 11–14], which allowed us to pay particular attention to the percentage of particles and the influence of temperature on the formation of this lubricant film. However, these elements (percentage of particles and temperature) are also important when plastics are used in anti-friction composites [10]. This is especially important under the operating conditions of high temperatures, loads, or sliding speeds. Such working conditions are characteristic of high-speed friction units (above 10,000 rpm), as well as for high-temperature friction units operating at temperatures of 700°C–800°C and loads up to 7.0 MPa in air.

Nickel-based antifriction composites have been developed for such working conditions [2, 6]. This material is also patented through the Ukrainian National Office for Intellectual Property and Innovations (IP office) [20]. New materials demonstrated high tribological properties that allowed authors to recommend these composites for industrial use. The limited data on the study of phenomena on the contact surfaces and the composition of the formed friction films does not allow the purposeful creation of new and effective antifriction materials, especially for severe operating conditions at high temperatures (700°C–800°C) or sliding speeds (above 10,000 rpm), loads (up to 7.0 MPa), and aggressive environment (air). Also, the effect of friction films on the functional properties of composites in the presence of a solid lubricant is still unexplored. This is due to the wide variety of the materials' working conditions, as a result of which the friction films can be either a lubricant or an abrasive. In addition, oxidation processes in air at high temperatures of 700°C–800°C significantly affect the properties of the formed surface friction films. Therefore, studying the composition of friction films, the mechanism of their lubricating action, and their effect on the tribological properties of high-temperature composite antifriction self-lubricating materials based on nickel at various operating temperatures is an urgent task. Such an approach will make it possible to establish the features of the friction film formation for nickel-based composite materials at various temperatures. On the basis of these data, it will become possible to predict the functional properties by purposeful selection of the material's initial components and their quantity. The use of this solid lubricant is found not only in nickel-based composites, as mentioned earlier, but also with the addition of other reinforcing elements. The use of this type of solution allows us to obtain new antifriction materials, for example, WC–TiC–Ni_3Al–CaF_2 [21] or Al_2O_3–Ti(C,N)–CaF_2 [22, 23]. This also indicates multi-track development, not only based on nickel alloys, but also on alloys of other metals.

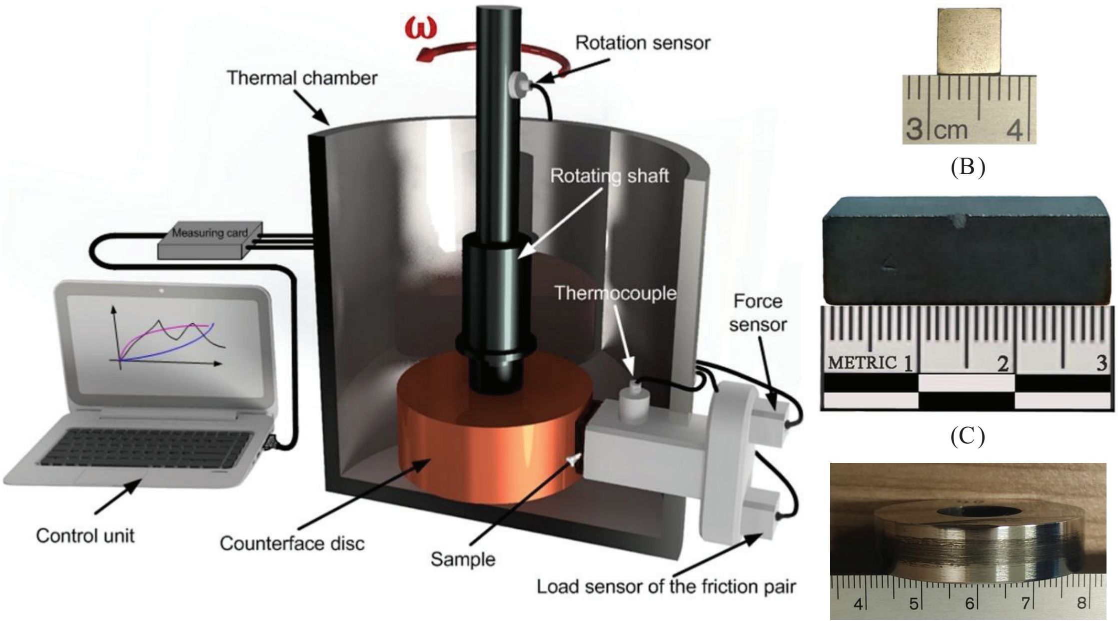
This is the motivation for studying antifriction film composition and its influence on the formation of the functional properties of a self-lubricating, high-temperature composite material based on EP975 powder nickel alloy with solid lubricant additives. The results obtained will open up the possibility of rationally choosing operating conditions for new self-lubricating antifriction composites, which will ensure reliable operation of high-temperature friction units.

The objective of the article is to study friction film composition and its effect on the tribological properties of the self-lubricating, high-temperature antifriction composite based on EP975 powder nickel alloy with CaF_2 solid lubricant.

2. Experimental procedure

Tribological tests were performed on a VMT-1 friction-testing machine (temperature up to 800°C, sliding speed V = 0.3–1.0 m/s and load P = 0.5–5.0 MPa); the counterface is made of stainless high-temperature, high-alloy chrome-nickel steel EI961Sh. A diagram of the test stand is presented below (Fig. 1). The counterface material is EI961Sh steel and corresponded to the material of the real shafts in the high-temperature friction units.

EI961Sh steel has the following chemical composition, wt%: 0.10–0.16 carbon, up to 0.6 silicon, up to 0.6 manganese, 10.5–12.0 chromium, 1.50–1.80 nickel, 1.60–2.00 tungsten, 0.35–0.50 molybdenum, 0.18–0.30 vanadium, up to 0.025 sulfur, up to 0.030 phosphorus, and iron as the base [6]. The microstructure and X-ray microanalysis of antifriction films were studied using scanning electron microscopy (SEM) (Raster microscope Zeiss-EVO SOXVP, EVO50.04.47, Germany) with a secondary electron (SE) detector. Research into the antifriction films' composition was performed



(A) (B) (C) (D)

Fig. 1. Stand and research object: (A) scheme of the test stand, (B) cross-section of the sample, (C) sample for testing, (D) counterface disc

Table 1. Chemical composition of the material based on nickel alloy EP975

Components, wt%									
C	W	Cr	Mo	Ti	Al	Nb	Co	Ni	CaF_2
0.038–0.076	8.65–9.31	7.6–9.5	2.28–3.04	1.71–2.09	4.75–5.13	1.71–2.59	9.5–11.4	basis	4.0–8.0

using SEM and analyzed by energy dispersive X-ray spectroscopy (EDS).

The study focused on new antifriction composite material based on powder nickel alloy EP975. The powder alloy EP975 was chosen as the basis for new materials because this alloy is heat resistant. It has the unique ability to work under extreme conditions of high temperatures, creep, fatigue ability, oxidation, and hot corrosion, which is usually inherent in gas turbines. Powder of the high-alloyed nickel alloy EP975 is the standard industrial alloy that has been produced by the powder spraying method of melted metal by argon stream. As a result, crystallized spherical particles are formed and have dimensions from 10 to 750 μm [6]. In our case, powders of Ni-alloy EP975 had dimensions of 50–200 μm.

A solid lubricant powder of CaF_2 was added to the original mixture. The original charge consisted of metal components such as powders of alloy EP975 and nonmetal components such as CaF_2 powders. Calcium fluoride was chosen for adding to the initial charge of material because this lubricant is well-known as a high-temperature and chemically stable substance. This solid lubricant is an effective substance at elevated and high temperatures (or high speeds) and retains its properties up to 1300°C. This behavior of the material has been investigated and the results published [3, 4, 20]. Additionally, work on the influence of temperature on the operation of anti-friction composites was presented in those sources in which the authors focused on addition and influence of tribological properties of graphite and TiC particles [25], tribological properties of Ni-based materials [26], and addition of solid-based lubricants in Ni-matrix composites [27]. Chemical composition of the material studied is presented in Table 1 [6].

Thus, in our experiments we have studied the antifriction composite material powder nickel alloy, EP975 + (4.0–8.0)% CaF_2. The composite material was manufactured using the powder metallurgy method and hot isostatic pressing technology (HIP), because the traditional powder metallurgy technology doesn't ensure minimum porosity, which is necessary for extreme working conditions [6]. Traditional cast technology for heat-resistant alloys consists of vacuum melting and casting of blanks with the operations of hot-working and final mechanical cutting following in order to obtain various parts with the necessary form. In this case, the coefficient of the use of metal is low enough and only at 15% does this approach become unproductive and expensive. Moreover, this method cannot provide high-quality heat-resistant alloys with a high content of alloying elements in a solid solution because segregation of the alloying elements takes place. This has the effect of reducing the properties of the material obtained using traditional technology [5]. Naim Katea [28] and Kruzhanov [29] focus on powder metallurgy (sintering) technologies. Trends and innovations in lubricating nanocomposites are described by Ogbonna [30] and Ji et al. [31]. Also, Atkinson and Davies [32] and Demptster and Wallis [33] focus on the important foundations of the hot isostatic pressing process. Additionally, Kluczyñski et al. [34] and Śnieźek et al. [35] focused on the behavior of material structures using the hot isostatic pressing method, as well as using various heat-treatment processes. In this context, the powder metallurgy method using the hot isostatic-pressing technology was a tempting prospect for obtaining high-quality composite antifriction heat-resistant material for high-temperature friction units. Hot isostatic pressing technology combines high pressure with a high temperature, which allows unification of the process of forming and sintering [6], which can be also found in articles by Bin et al. [18] and Atkinson [32]. The advantages of the isostatic pressing method consist in the even distribution of pressure and density of blank due to overall (isostatic) pressure. Hot isostatic pressing followed by heat treatment provides a minimum size of grain, which promotes material with the required properties.

First of all, initial components, such as the sprayed nickel alloy EP975 powders and solid lubricant (CaF_2), were mixed. Next, powder mixture was subjected to hot isostatic pressing. The blanks almost didn't have pores, and their relative density was 99.9% after the application of hot isostatic-pressing technology. Then, the heat treatment was carried out after the hot isostatic pressing. The heat treatment consisted of hardening with heat at 1220°C–1240°C, cooling in air, and then aging at 900°C–920°C for 16 hours. This practice is provided by Jamroziak and Roik [6], but a similar approach is also described in the literature by Bin et al. [18] and Atkinson and Davies [33]. After heat treatment, a mechanical operation is applied to produce the finished part. In our case, samples for different types of tests were cut from the heat-treated blank. A diagram of the sample production process is shown in Figure 2.

The composite we studied is a sufficiently dense material that is an important candidate for working in severe operating conditions. The technological operations performed ensured the formation of a composite's homogeneous structure, consisting of the alloyed nickel-based γ-solid solution,

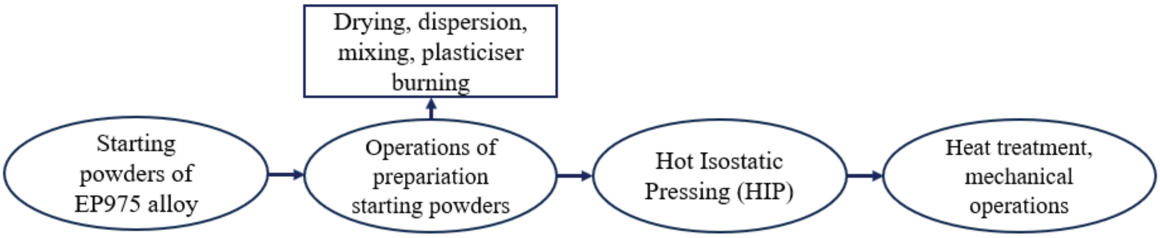


Fig. 2. Technological manufacturing scheme of the antifriction composite EP975 + 6%CaF_2

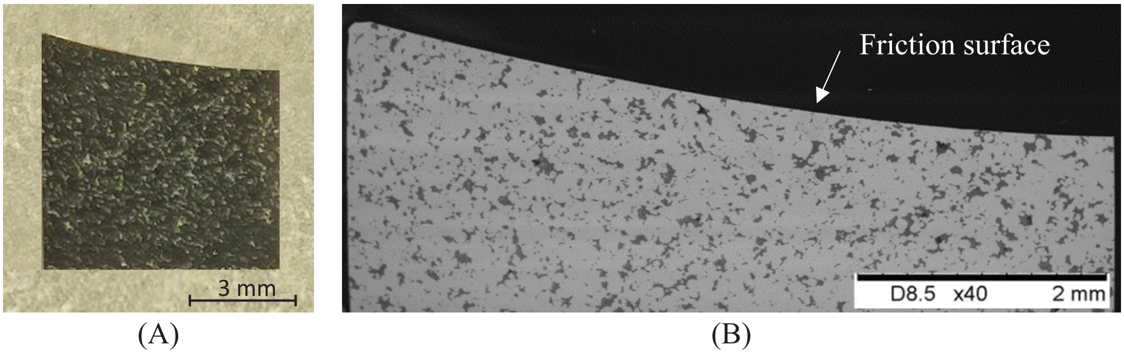


Fig. 3. Tested sample: (A) general view of the metallographic specimen, (B) SEM: view of the structure and friction surface at 40× magnification

strengthened with intermetallics and carbides, as well as calcium fluoride particles (CaF_2) [6]. In this way, samples for tribological tests with an area of 6×6 mm were obtained, which are presented below in Figure 3.

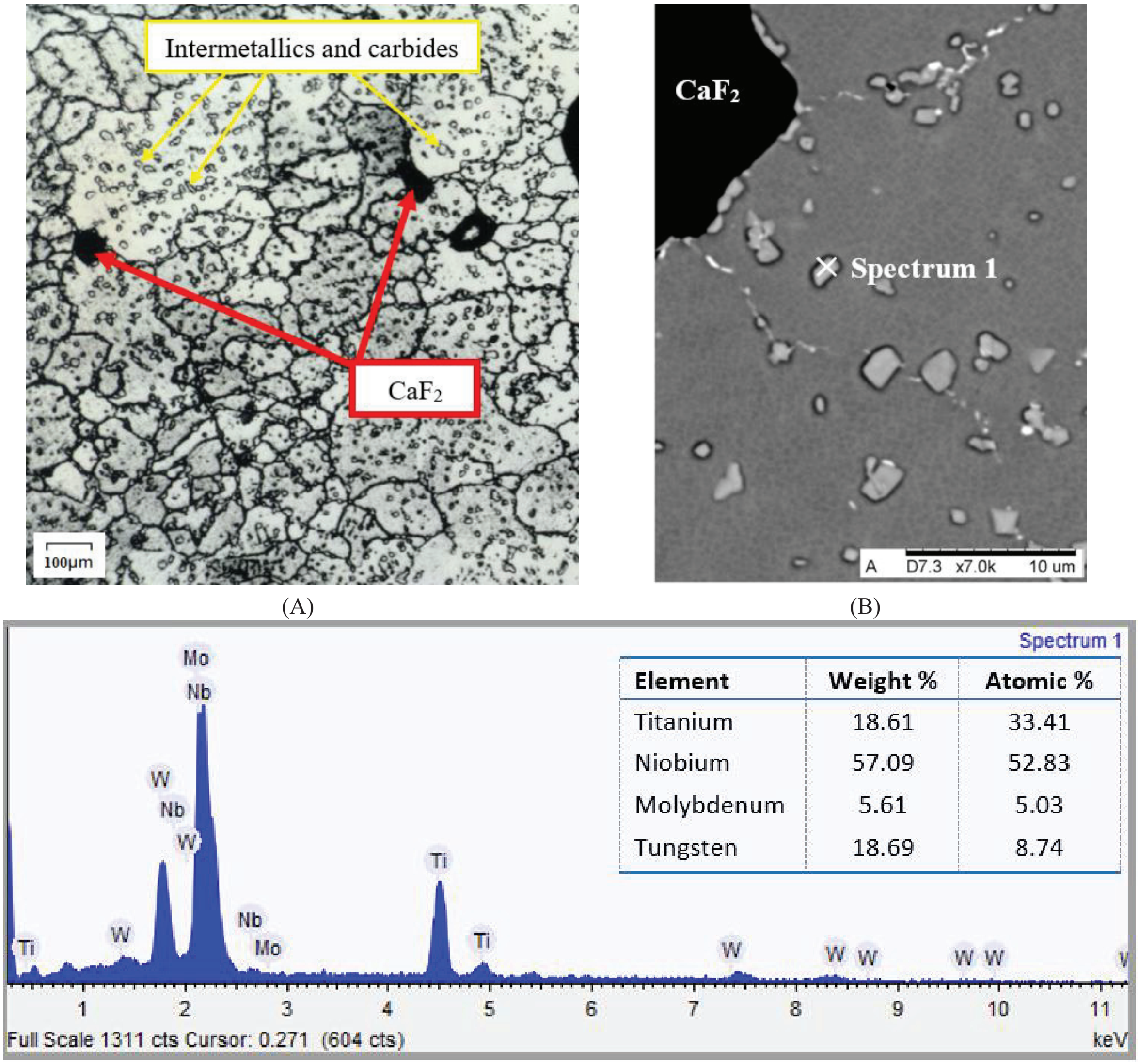
The structure of EP975 + 6%CaF_2 composite after manufacturing including HIP technology is shown in Figure 4. As the SEM analysis presented in Figure 4 shows, the intermetallics in the microstructure are evenly distributed both on the grain boundaries and inside the grains. EDS analysis indicates that most of the precipitates are the stoichiometric composition of Ni-W-Ti-Mo. Some of them also occur with the participation of C and Cr. Phases of this type, present in the materials in the form of dispersed precipitates, contribute significantly to stabilizing the materials during high-temperature operation by increasing their resistance to oxidation similarly to Nb-Ti or Nb-Ti-Si materials [36].

As shown in Figure 4, the structure of the composite is dense and free of pores and segregations. Two factors such as homogeneous structure (Fig. 4) and absence of segregation are main advantages that promote higher operating properties of both friction coefficient (μ) and wear rate (Fig. 5). Known material was tested in combination with the same counterface [5, 6].

The data in Figure 5, shows the significant advantages of the new composite compared with the known material tested by Jamroziak and Roik [5] and [6], which has high porosity of 12%–14% and is currently used in similar conditions. This is due to the low alloying degree of the known material and the differences of these composites' manufacturing technology. Such differences have a significant effect on the formation of structure and properties.

Moreover, the new antifriction composite can work under more severe operating conditions. Analyzing the data of Figure 5, shows that the new composite's tribological characteristics improve at increasing sliding speed and load. This is due to the intensification of the antifriction film formation with increasing loading factors, which leads to stabilization of the friction pair operation and realization of the self-lubricating mode. A balance has been observed between film wear rate and the film's formation rate for new areas under these conditions. That's why it is extraordinarily important to provide the forecasting of the new material's functional properties and proper characteristics of friction surfaces under such operating conditions. For this purpose, an analysis of antifriction films was performed after tribological tests. The surface microstructure before the abrasion process (Fig. 6) and after the abrasive wear process (Fig. 7) is presented below.

The trends of the effect of abrasion parameters on the obtained values of abrasion coefficient and abrasive wear, shown in Figure 5, can be explained by microscopic studies showing the state of the surface of the samples after the abrasion tests (Fig. 7). In order to highlight the changes in the mechanism of formation and degradation of oxide layers and friction films at different parameters,



Element	Weight %	Atomic %
Titanium	18.61	33.41
Niobium	57.09	52.83
Molybdenum	5.61	5.03
Tungsten	18.69	8.74

(A) (B)

(C)

Fig. 4. Structure of the developed composite after manufacturing, wt%: EP975 + 6%CaF_2: (A) surface electrolytic etching of metallographic microscopy – light microscopy, (B) SEM (detector SE): microstructure with a view of grain boundaries and intermetallic precipitates, (B, C) EDS analysis

samples operating at the lowest and highest coefficient of friction were selected. In each of the cases considered, abrasion of the composites on the surface of the EI961Sh counterexample results in the formation of a band-oriented structure on the friction surfaces along the direction of abrasion, with visible jambs and dissolved adhesively bonded lubricating elements. The state of the friction surface for composites subjected to abrasion under a load of 5.0 MPa and a velocity of v = 1.0 m/s (Fig. 7B) indicates a more balanced operation of the friction pair causing more effective grinding of lubricating elements on the friction surfaces in contrast to composites subjected to abrasion under a load of 2.0 MPa and a velocity of 0.3 m/s (Fig. 7a).

On the surfaces of specimens operating under higher loads and speeds, observations confirm a greater number of smaller more evenly distributed lubricating elements. On the other hand, at lower loads and process speeds within wide areas on the abrasion surfaces, an increased amount of fine,

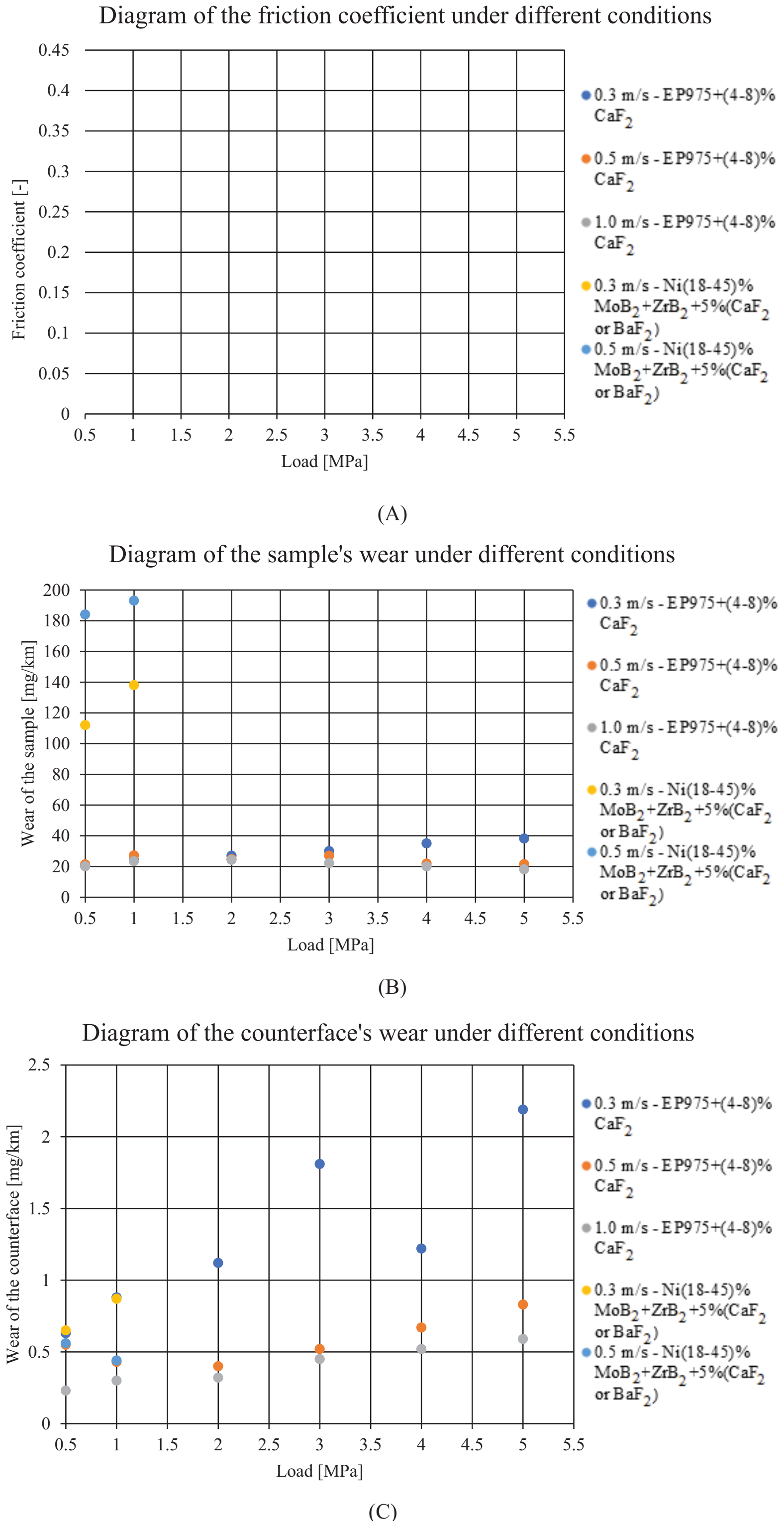
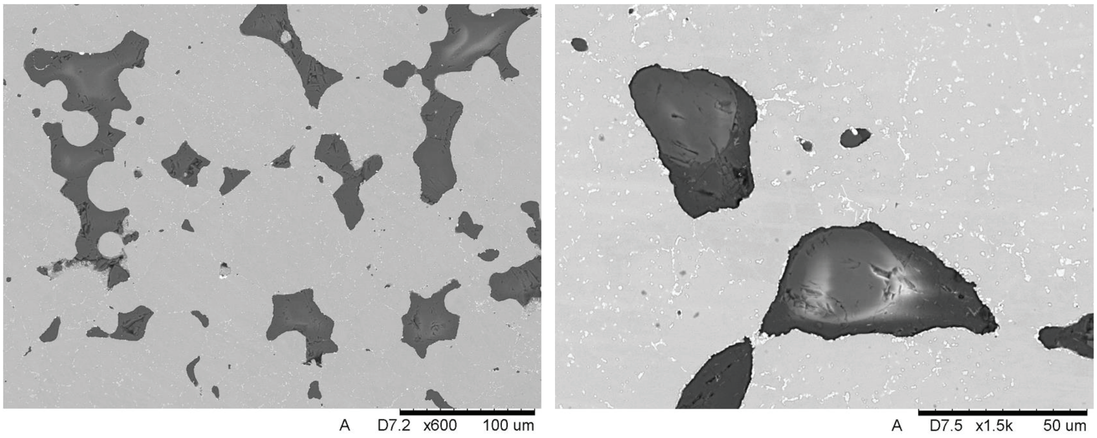
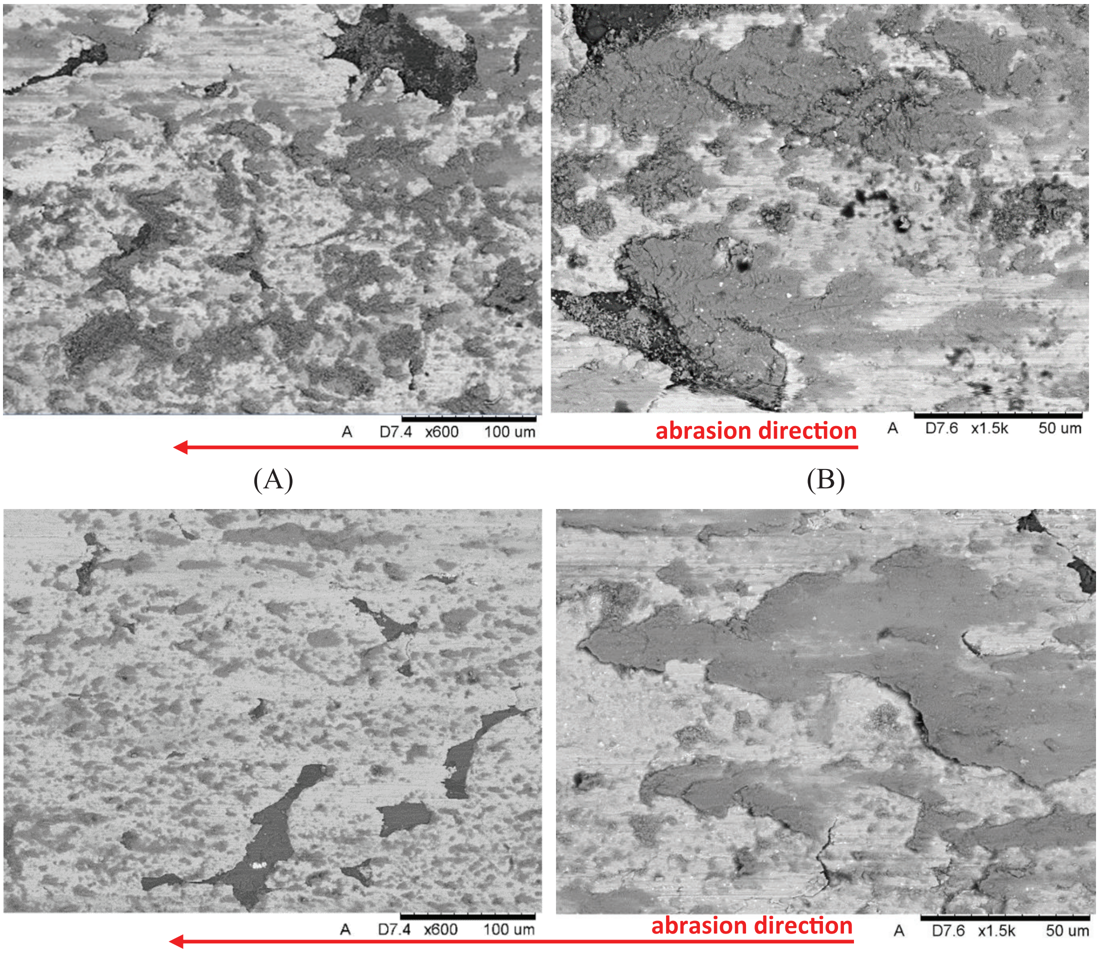


Fig. 5. Antifriction properties of composites studied under different loads and velocity of friction: (A) diagram of the friction coefficient under different loads, (B) diagram of the sample's wear under different loads, (C) diagram of the counterface's wear under different loads



(A) (B)

Fig. 6. SEM (SE): view of the sample surface before abrasive wear testing



(A) (B)

(C) (D)

Fig. 7. SEM (SE): Friction surface of the EP975 + 6% CaF_2 composite after testing with the following parameters: (A, B) P = 2.0 MPa and v = 0.3 m/s, (C, D) P = 5.0 MPa and v = 1.0 m/s

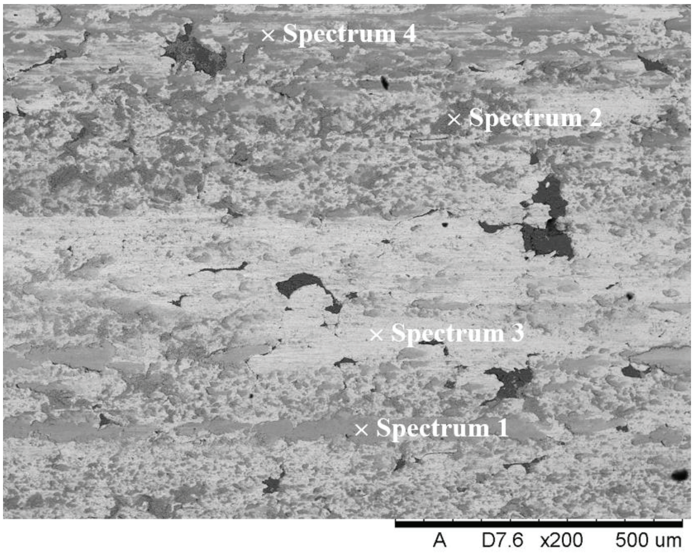


Fig. 8. SEM (SE): friction surface of the EP975 + 6% CaF_2 composite with marked EDS analysis points of elemental composition

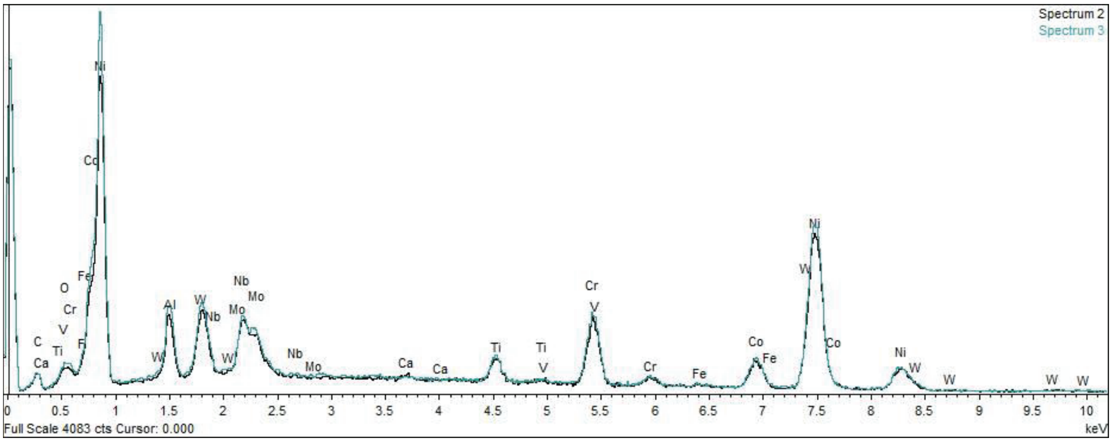


Fig. 9. Spectra from the selected areas on the composite's friction surface

fragmented wear products accumulated, located mainly in the area of lubrication pockets and in the layer of smeared grease. These elements intensify the abrasion process by increasing the brittleness of the lubricating layers leading to an increase in the intensity of their tearing and delamination with simultaneous detachment from the substrate. The detached fragmented material elements moving between the friction vapor act as an abrasive, increasing the furrowing and damaging the previously formed oxide layer and lubricating film.

The film is a multiphase conglomerate of the compounds formed from chemical elements of composite, counterface, and oxygen. It is obvious that in addition to the contact pair's alloying elements and calcium fluoride, atmospheric oxygen takes an active part in the formation of a self-lubricating film at a temperature of 800°C. This suggests the oxidation processes are activated by friction in air at the high temperatures; this is a positive factor in our case.

X-ray microanalysis of the film from the composite's surface confirmed this (Figs. 8 and 9, Table 2).

The surface antifriction films formed were investigated both on a specimen from a new

Table 2. EDS results of the wear track in selected areas on composite's friction surface

Analysis point	**Element, wt%**											
	O	**F**	**Al**	**Ca**	**Ti**	**Cr**	**Fe**	**Co**	**Ni**	**Nb**	**Mo**	**W**
Spectrum 1	17.24	4.81	3.21	1.94	1.01	5.00	14.1	5.84	36.86	0.78	2.22	6.99
Spectrum 2	16.45	26.45	0.85	16.87	0.21	1.24	8.48	1.31	25.21	0.37	0.97	1.68
Spectrum 3	9.12	0.99	4.12	0.07	1.71	8.04	0.24	10.41	52.01	1.24	2.51	9.54
Spectrum 4	16.04	4.51	3.02	2.01	1.04	5.54	13.46	6.23	38.02	0.65	2.45	7.03

composite and on a counterface. The structure of the antifriction layer on the surface of the composite material samples was revealed using elemental decomposition performed using linear EDS analysis. The tests were performed on the transverse surface (Fig. 10). Chemical composition tests were carried out along the scanning lines at a depth of up to 14 μm below the abrasion surface. The analysis confirmed a significant increase in the amount of oxygen in the surface layer on the abraded surface compared to its amount in deeper areas. The presence of oxygen along with other elements confirms the presence of oxide phases in these areas that create an anti-friction film. That the presence of Ca and F was demonstrated also confirms the active participation of the CaF_2 lubricant in the process of cooperation between the sample and the countersample. The increased Fe content indicates the active participation of elements originating from the countersample in the process of building friction layers.

Detailed determinations of chemical composition were made for the differences in the distribution of elements in the surface layer shown in the tests in Figure 10, occurring when the depth from the surface changes. The research was carried out based on point and area EDS analysis (Fig. 11, Table 3).

The surface of the specimen is completely covered with conglomerates and compounds from the chemical elements of the friction pair and oxygen. This indicates the next balance of the friction process under such operating conditions: the film's new areas are constantly being formed on worn-out areas of the contact surface. Therefore, a balance is observed between film wear and film's new sections formation on worn-out areas. Such phenomena of balanced wear of the friction film and formation of its new sites is confirmed by high antifriction properties of the investigated composite. The process of delamination and fragmentation of the foil with simultaneous reconstruction of the layer using CaF_2 lubricating elements taken from open pockets is illustrated in Fig. 12.

As can be seen from Figures 8–12 and Tables 2 and 3, the presence of oxygen in a significant amount in the film confirms the assumption about the oxidative nature of wear at high temperatures and loads in air.

Similar features are evidenced by data of X-ray microanalysis and element maps also obtained from the surface of the counterface which is made of EI961Sh steel.

EDS results of the wear track in selected areas from the counterface (Figs. 12, 13, Table 4) after tribological tests at 880°C, load 3.0 MPa, and speed of 0.5 m/s are given in Table 4.

Fe is present in the surface film as the main element of steel (Table 4). The high content of molybdenum is associated with its presence both in the composite and in the counterface. Moreover, the molybdenum high content confirms the effect of mass transfer during friction. This also applies to Ca on the surface of the counterface that confirms CaF_2 mass transfer from composite to counterface's surface. These results (Fig. 14, Table 4) confirm the phenomenon of mass transfer to the surface of the counterface and the presence of the same antifriction film on its surface, which is a positive factor for the stable operation of the friction pair.

It is likely much stronger signals from elements, such as Fe and Mo, shield other alloying elements in this spectrum from the counterface's surface (Fig. 14). The high oxygen concentration on the counterface's surface should be emphasized. This

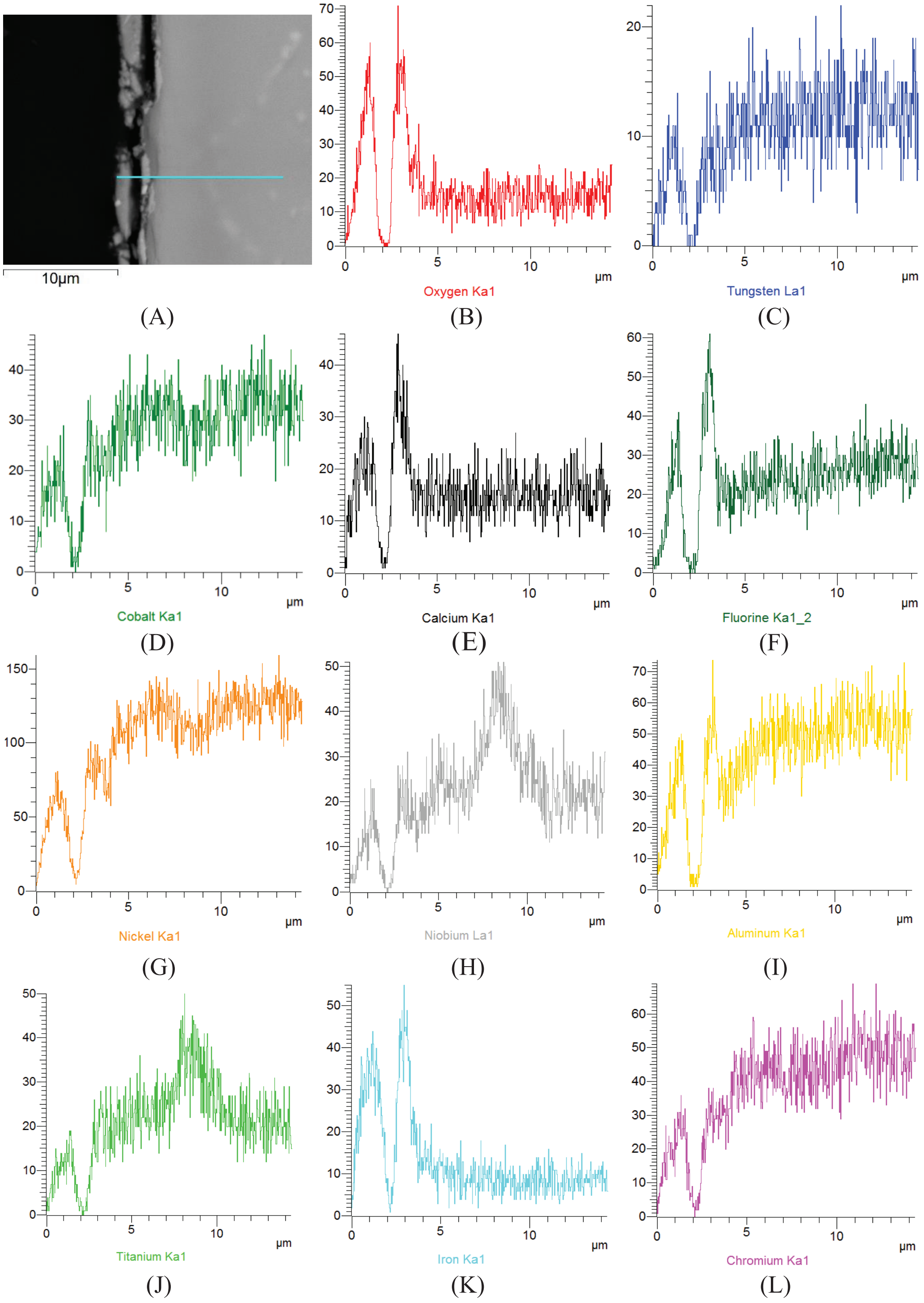
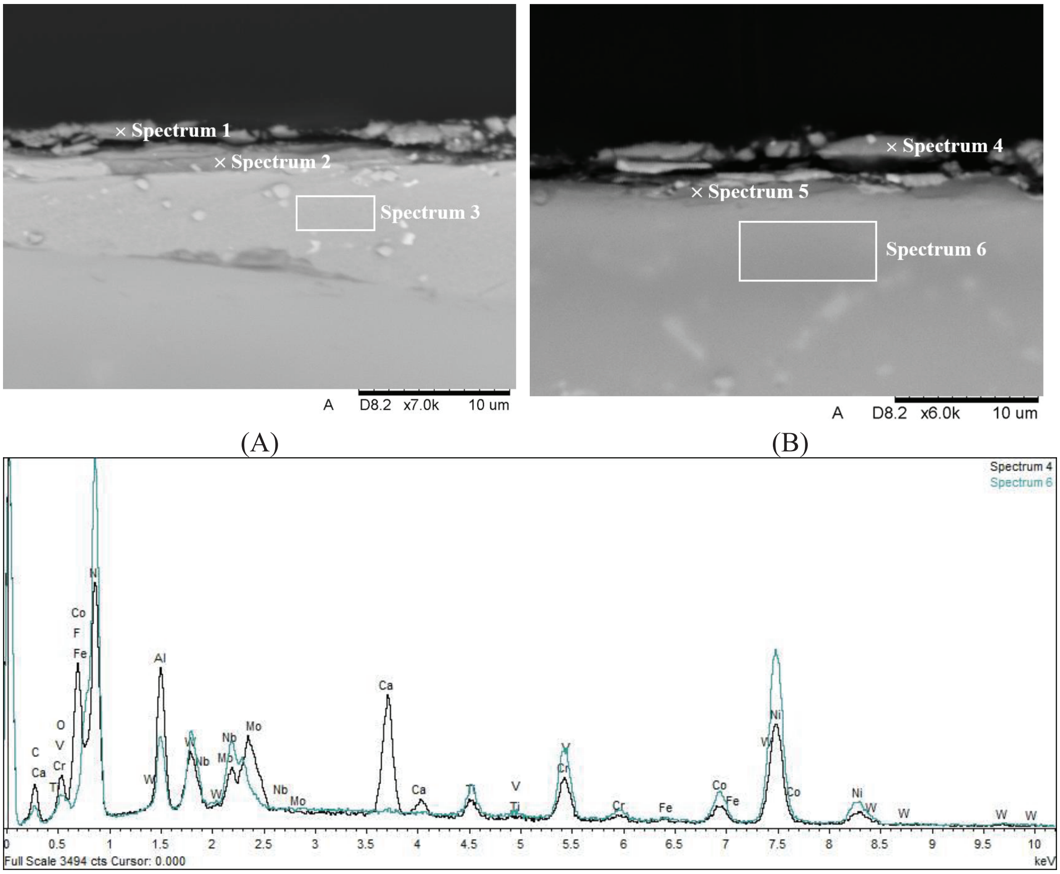


Fig. 10. EDS analysis of the distribution of elements along the selected scanning line illustrating the structure of the anti-friction layer on the surface of the EP975 + 6%CaF_2 composite as a function of the depth from the surface: (A) transverse line of which EDS was taken, (B) oxygen content, (C) tungsten content, (D) cobalt content, (E) calcium content, (F) fluorine content, (G) nickel content, (H) niobium content, (I) aluminum content, (J) titanium content, (K) iron content, (L) chromium content



(A) (B)

(C)

Fig. 11. SEM (SE): Microstructure of the abrasive layer in the composite cross-section view EP975 + 6% CaF_2. (A) EDS analysis measurement locations EP975 + 6%CaF_2 $P = 4.0$ MPa, $v = 0.5$ m/s, (B) $P = 2.0$ MPa, $v = 0.3$ m/s, (C) results obtained for areas of the composite friction layer

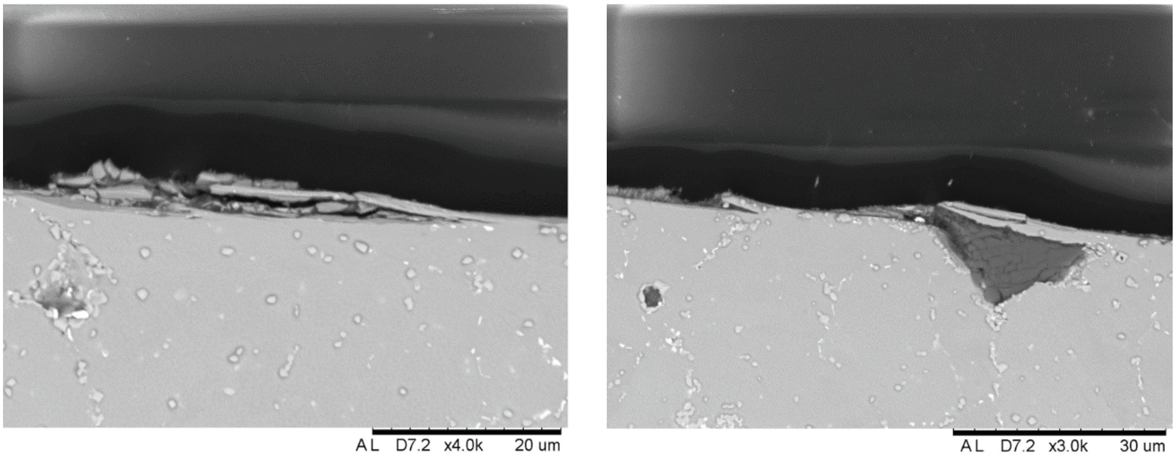
Table 3. Results of the EDS analysis in selected areas (marked in Fig. 11) of the cross-section of the friction layer

Analysis point	**Element, wt%**											
	O	**F**	**Al**	**Ca**	**Ti**	**Cr**	**Fe**	**Co**	**Ni**	**Nb**	**Mo**	**W**
Spectrum 1	16.64	4.68	3.49	2.12	0.99	4.97	14.68	6.14	35.96	0.94	2.38	7.01
Spectrum 2	14.38	3.71	3.07	1.96	1.15	5.62	16.49	7.04	38.44	0.49	1.59	6.06
Spectrum 3	0.11	1.04	3.99	0.07	1.64	8.32	0.18	11.16	61.11	0.86	2.48	9.04
Spectrum 4	16.74	4.91	3.73	1.98	1.03	5.11	13.67	7.08	36.93	0.49	1.62	6.71
Spectrum 5	14.91	5.79	3.29	2.17	1.11	5.56	14.14	6.73	37.60	0.71	1.67	6.32
Spectrum 6	0.09	1.58	4.77	0.09	2.01	8.06	0.10	10.43	57.31	1.94	3.01	10.61

once again confirms the occurrence of oxidative wear at high temperatures in air.

Analysis of the elements' maps from the counterface's surface showed the alloying element's uniform distribution. This indicates a uniform distribution of the corresponding phases and structural elements in the antifriction film.

Figures 4–14 opens up the possibility of understanding the processes on the working surfaces of the friction pair during high-temperature friction. These data show the self-lubrication mechanism of the new composite at high temperatures. The distribution of chemical elements from all friction participants indicates reliable data obtained by a



(A) (B)

Fig. 12. SEM (SE): View of the microstructure of the composite in cross-section after the abrasion process at P = 5.0 MPa and v = 0.5 m/s: (A) formation of a friction film, (B) delamination and fragmentation of the oxide layer

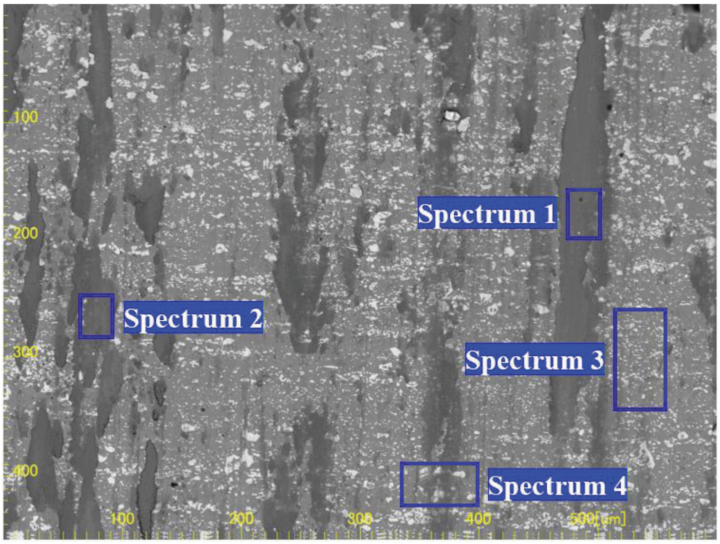


Fig. 13. SEM (SE): View of the counter-sample surface after the abrasion test at a pressure of P = 3.0 MPa and a speed of 0.5 m/s; designation of measurement areas of EDS elemental composition analysis

Table 4. Results of the EDS analysis of the chemical composition in selected areas (marked in Fig. 13) of the friction surface of the EI961Sh counter-sample after the abrasion test of the EP975 + 6%CaF_2 composite. Friction conditions: P = 3.0 MPa and v = 0.5 m/s

Analysis point	**Element, wt%**												
	O	**F**	**Al**	**Ca**	**Ti**	**Cr**	**Fe**	**Co**	**Ni**	**Nb**	**Mo**	**W**	**V**
Sp. 1	19.23	3.99	2.39	4.21	0.79	5.54	21.95	4.84	26.79	0.15	2.59	7.09	0.44
Sp. 2	18.02	3.53	2.3	4.02	0.65	5.62	25.86	4.89	23.55	0.12	2.56	8.54	0.34
Sp. 3	12.31	1.84	0.44	0.42	0.14	6.51	61.16	1.56	7.26	0.11	0.65	6.58	1.02
Sp. 4	15.07	2.87	1.08	0.98	0.26	4.11	50.66	1.89	14.92	0.12	1.02	6.66	0.36

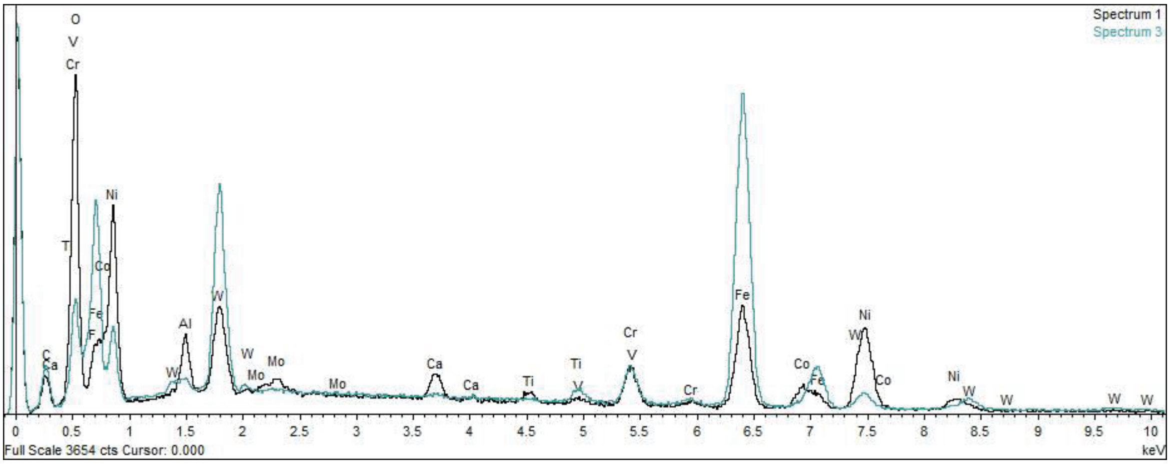


Fig. 14. Spectra from the selected area on counterface's friction surface

variety of methods. These include the special role of solid lubricant and air oxygen (Figs. 4–14, Table 2) and the formation of the conglomerates (Figs. 8, 11) that form the antifriction film, providing high tribological properties (Fig. 5). a It is also confirmed by preliminary industrial tests of antifriction bushings made from the composites when they work in friction units of gas turbine equipment at 700°C–800°C.

In summary, it can be argued that the nature of the distribution of chemical elements on both contact surfaces significantly affects the structural features of the antifriction film. The alloying elements' uniform distribution contributes to a uniform distribution of phases in the film. In turn, this ensures high tribological properties at high temperatures. The studied morphological features that arise inside the antifriction film favorably affect the self-lubrication mechanism, ensuring the contact pair's stable operation under severe operating conditions. Antifriction films consist of the conglomerates from the complex, including oxide and fluoride formations which cover the contact surfaces and prevent the composite's volume oxidation.

Further studies will be devoted to determining the friction film's phase composition depending on the antifriction composite's initial chemical composition and the temperature conditions of the friction process. The data obtained will make it possible to rationally select the operating conditions of the material, with which it will demonstrate the highest tribological properties.

3. Conclusion

Composition and chemical element distribution in antifriction films and their effect on the tribological properties of self-lubricating high-temperature antifriction composite consisting of EP975 powder nickel alloy with CaF_2 solid lubricant. SEM analysis of the chemical elements using EDS showed their uniform distribution, both on the composite surface and the counterface surface, which positively affects the level of functional properties in the operation modes corresponding to real conditions, such as a load up to 5.0 MPa, a slide speed 0.3–1.0 m/s, and a temperature up to 800°C, in air.

For the first time the experimental studies performed showed the nature of the chemical elements' distribution on both contact surfaces has a significant effect on the structural features of the antifriction film. The internal chemical reactions occur in friction films during the whole friction process under the actions of high temperature and oxygen. The alloying elements' uniform distribution leads to a uniform distribution of the corresponding phases and structural elements in the antifriction film. Solid lubricant CaF_2 alloying elements, and their corresponding phases form the continuous antiscoring film. This ensures high tribological properties at high temperatures.

The internal processes in the films are very complex and largely depend on the manufacturing technology and operating and lubricating conditions. Analysis of the material's tribological properties using metallographic and micro-X-ray research, confirmed the suitability of the technology for producing the composite. Anti-seize self-lubricating films with calcium fluoride minimize wear of the friction pair. Antifriction films have smooth microtopography, which stabilizes the high-temperature friction unit operation. Thus, the self-lubrication mode is realized for a long exploitation time in these operational modes.

The results obtained make it possible to rationally choose operating conditions for new self-lubricating antifriction composites with the highest properties. Tribological properties analysis allowed us to determine the ranges of rational exploitation modes for the material studied: a load up to 5.0 MPa, a slide speed 0.3–1.0 m/s, a temperature up to 800°C, in the air. These data open the opportunity to control the antifriction film formation and the composite's tribological properties by choosing the initial ingredients while taking into account the expected operating conditions. This will allow for the formation of the necessary metal basis and the addition of the required amount of the solid lubricant in order to ensure the reliability and durability of the high-temperature friction unit. Bearing bushings made of new self-lubricating nickel-based composites were successfully tested at 800°C in friction units of turbine equipment and are recommended for application. Studies have also shown that new antifriction composites based on EP975 powder nickel alloy with CaF_2 solid lubricant are multifunctional and can be recommended for use not only for high-temperature friction units, but also for units of high-speed printing machines operating up to 10,000 rpm.

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