Structure research of nanoscaled silicon carbide detonation coatings of tribotechnical application

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Abstract

Presented studies are related to the spheres of wearproof coating development. World wear resistance improvement technology experience has accumulated a huge amount of statistical material on the failure due to increased level of parts wear. That is why the issue of research and improvement of anti-wear properties of machine elements is one the components when considering the priority directions of ensuring the reliability of operation motor vehicles and friction units. The SiC coating has been deposited on the medium carbon steel using detonation deposition. It has been established that it is very sensible for modes of coating deposition and different physical and chemical phenomenon have been detected. The structure of the obtained coating has been thoroughly researched on the electronic microscope. The obtained coating has been developed for testing on the friction bench modeling the friction process that is taking place in the couple of main and rod journals of internal combustion engines. The coating has also the corrosion protection. The new magnet modified method of detonation coating deposition has been tested for deposition of nanoscaled coating on mild carbon steel. The optimal modes of the magnet modified coating deposition for silicon carbide powders batch mixture from the viewpoint of structure formation have been detected.

Key words: wear, wear resistance, coating, adhesion, nanoscaled coating, detonation coating deposition.

Introduction

Means of powered equipment and automated equipment of airports are equipped with power plants as energy sources for technological operations on ground handling of aircraft. As such power plants on aircraft ground equipment electric motors, gas turbine engines, internal combustion engines can be used. Operating experience has shown that internal combustion engines have the highest energy efficiency and safety, because electric engines have low starting torques, and gas turbine engines are designed for operation at altitudes of the cruising flight of aircraft and at ground level have low fuel efficiency and stability. Thus, an internal combustion engine (ICE), which can be either fuel or diesel, is the main power unit of aviation ground equipment. The lifetime of the ICE can be improved by wearproof coatings

Review of the latest research

Silicon carbide materials has attracted scientific attention of researchers from many countries. So in the works [1,2] the features of structure formation of ceramic materials of the constituents SiC–C with oxides Al₂O₃ and Y₂O₃, as well as titanium hydride under free sintering and hot pressing are considered. Effect of ceramics dispersion strengthening by nanoscaled particles of silicon carbide and titanium carbide has been established. In the publication [3] micromechanical properties of composition materials made of SiC, acquired by activated sintering has been researched.

Authors of the work [4] has researched high temperature, corrosion resistant, mechanical properties of nanoporous structures made of silicon carbide, which was intended for electrical and technical application.
In the publication [5], researchers had been learning oxidation resistance of composites of the constituents SiC–TiB₂–B₄C, acquired by hot pressing at temperature 2150°C, which have improved physical and mechanical properties. The samples porosity made less than 8%.

The structure formation of ceramics made of constituents SiC–TiB₂, where the silicon carbide was the composite major, was researched in details in works [6, 7]. In particular, optimal technological modes of ceramic materials acquisition concerning the bending strength, crack- and wear resistance had been determined.

Attempts of properties optimization of self-bonded silicon carbide are undergoing nowadays. So, in the works [8] different from conventiona technologies technique of high-dense ceramic products on the basis of self-bonded silicon carbide, which blankpieces are made by the method of slip casting of thermal plastics under the pressure had been developed. The content of multifractional batch mixture and amount of temporary bonds with rheological properties, that provide the high density of products, has been justified.

One of the prominent scientific direction of silicon carbide application is the nanostructured composites and coatings. So, the researchers of article [9] the microstructure and properties of alumina-silicon carbide nanocomposites fabricated by pressureless sintering and post hot-isostatic pressing have been investigated. There the grain growth of Al₂O₃ matrix had been eliminated due to the grain growth inhibition by nano-sized SiC particles (about 150-250 nm). It improved the fracture strength of acquired composite.

In the scientific paper [10] the synthesis of SiO₂ and SiC micro/nanostructures of nanowires (about 100 nm) and nanorod (about 50-200 nm) shapes had been held by thermal evaporation method (CVD). The scientific findings were intended for core-shell coaxial nanocables.

The researchers of work [11] have acquired nanocomposites of C-SiC content. The composite is the carbon fibers covered by SiC nanocoatings. The material was intended for radiation resistant fabric concerning the fabric strength. And the scientists of paper [12] had acquired the nanostructured coatings of silicon carbide by novel method. the thickness of the coating was about 2-9 nm and the coatings were suitable to application in metallurgy, nuclear power engineering, microelectronics and high-temperature stoves.

In order to create the metal ceramic materials, effect of iron millings on the technological modes of acquisition of ceramic materials of the system SiC-Al₂O₃, their structure and properties have been researched. Tribological performances of acquired materials have been tested. Wear rate of these materials together with the steel counterbody is 3.8 microns per kilometer, and together with ceramic counterbody it is 4.1 microns per kilometer. In both cases oxidative wear mechanism takes place [13].

As have already been mentioned the specific way acquired batch mixture of SiC-Al₂O₃ content was used for acquisition of wearoff composites [13] the same batch mixture was applied for coating deposition by detonation method modified by magnetic field [14]. So it had been established on direct polarity of coil magnet the microparticles of silicon carbide and alumina had been deposited on the substrate. The coating had demonstrated not only high wear resistance [14], but also high wear resistant at elevated temperatures [15]. The last mentioned composition had been widely used for coating deposition by different technique and several results had been acquired. And changing the polarity of coil magnet only the nanosized particles of silicon carbide had been deposited on the substrate to which investigation this scientific paper is devoted.

**Research aim**

Scientific development of nanoscaled composition coatings for crank shaft journal of internal combustion engines of aircraft ground support equipment.

Originating from the aim of article papper the following tasks of research were preset:

1. Outlook of the reference sources on the topic of the article paper and on its basis the topic urgency had been confirmed.
2. Algorythm development of the complex scientific research, selection of the necessary laboratory equipment for coating deposition and tribotechnical research.
3. Preparation of the coating batch mixture, manufacture of the specimens and deposition of the coating.
4. Research of the coating structure and its description.

**Research methodology**

For study of interactions between properties of coatings with their phase composition and structure, and also an external factors influence the choice of research methods has the great importance. The receiving of reliable results of research in this work is provided with the use of modern equipment and devices, approved methodologies, necessary productivity of experiments, by careful treatment of specimens before and after the experiment, strict adherence of order of experiment carrying out.

For receiving a charge of carbide silica ceramics with aluminum oxide admixtures, the starting powders were used: silicon carbide grade 64С (ГОСТ 26 327-84) with an average size of 45-55 μm, aluminum oxide (ТУ 6-09-03-350-73) with particles of average size 45 - 50 microns.

The chemical composition of the starting powders is given in Table 1.
Table 1

<table>
<thead>
<tr>
<th>Powder name</th>
<th>Al</th>
<th>Si</th>
<th>Mg</th>
<th>Fe</th>
<th>Ni</th>
<th>Cr</th>
<th>Ti</th>
<th>Ca</th>
<th>Zr</th>
<th>Ag</th>
<th>Cu</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiC</td>
<td>10^{-3}</td>
<td>major.</td>
<td>10^{-4}</td>
<td>10^{-3}</td>
<td>-</td>
<td>-</td>
<td>10^{-4}</td>
<td>-</td>
<td>-</td>
<td>10^{-4}</td>
<td>10^{-3}</td>
</tr>
<tr>
<td>Al_{2}O_{3}</td>
<td>major.</td>
<td>-</td>
<td>10^{-3}</td>
<td>&gt;0.1</td>
<td>&gt;1</td>
<td>0.01</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>10^{-4}</td>
<td>10^{-3}</td>
</tr>
</tbody>
</table>

An integral operation for the formation of composite materials from the initial powders is their mutual mixing and grinding.

To obtain a SiC-based ceramic charge with Al_{2}O_{3} admixture, the powder components in the appropriate proportions were mixed with simultaneous grinding for 5 hours in the laboratory planetary mill Sand-1 in an alcohol medium.

In this case, the rotational speed was 648 rpm, the drum rotation frequency was 1620 rpm. To prepare the charge, laminated aluminum oxide and steel drums of 340 cm³ and steel grinding media from steel of ШX15 with a diameter of 10-15 mm and SiC-Al_{2}O_{3} ceramics grinding media were used.

The ratio of the mass of the charge to the mass of grinding media is 1: 3. After grinding, the charge was dried and sifted. The granulometric composition of the resulting mixtures after milling was determined in aqueous media on a laser microanalyzer "SK Lazer Micron Sizer PRO 7000".

Coatings in the work were applied by the detonation method on the installation described below. The “Dnepr-3M” (Table 2.) detonation-gas installation is intended for coating metal powders, hard alloys, ceramics and composite materials on the surfaces of machine parts, devices, apparatuses and tools during their manufacture, as well as reconditioning.

According to their purpose, coatings can be wear-resistant, frictional, antifriction, corrosion-resistant, heat-resistant, electrically conductive, electrical insulating, etc.

Table 2

<table>
<thead>
<tr>
<th>#</th>
<th>Specification</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Working gases</td>
<td>oxygen, acetylene, nitrogen, compressed air.</td>
</tr>
<tr>
<td>2</td>
<td>Pressure of working gases, MPa</td>
<td>0.2, 0.14, 0.4, 0.4</td>
</tr>
<tr>
<td>3</td>
<td>The consumption of working gases per shot, m³</td>
<td>27<em>10^{-5}, 23</em>10^{-5}, 5<em>10^{+}, 5</em>10^{+}</td>
</tr>
<tr>
<td>4</td>
<td>Powder consumption per shot, m³</td>
<td>1.5*10^{-5}</td>
</tr>
<tr>
<td>5</td>
<td>Water consumption, m²/s</td>
<td>3 *10^{5}</td>
</tr>
<tr>
<td>6</td>
<td>Frequency of fire, Hz</td>
<td>1-10</td>
</tr>
<tr>
<td>7</td>
<td>The diameter of the booster section of the barrel, m</td>
<td>0.022</td>
</tr>
<tr>
<td>8</td>
<td>Coating thickness per shot, microns</td>
<td>5-20</td>
</tr>
<tr>
<td>9</td>
<td>Productivity at a coating thickness of 10 microns, m³/h</td>
<td>0.8-3.5</td>
</tr>
<tr>
<td>10</td>
<td>Installation</td>
<td>remote control</td>
</tr>
<tr>
<td>11</td>
<td>Overall dimensions, m</td>
<td>1.8<em>0.6</em>1.1, 1.8<em>0.6</em>0.61, 0.5<em>0.3</em>0.22</td>
</tr>
<tr>
<td>12</td>
<td>Mains supply:</td>
<td>50-60, 220, 200</td>
</tr>
<tr>
<td>13</td>
<td>Sound pressure level, dB (A)</td>
<td>140</td>
</tr>
<tr>
<td>14</td>
<td>Relative humidity of air,%</td>
<td>40-75</td>
</tr>
</tbody>
</table>

For getting such compositions the following conditions have been determined. Working gas is a mixture of C₂H₂-O₂. Consumption of C₂H₂ is 30 points, O₂ is 70 points. Powder supply is 30 points. Blowing the barrel at the end of the cycle is air. Scavange gas is air. Shots speed is 4 shots per second. The diameter of the spot is 22
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mm. Spraying distance is 170 mm. For research of the effect of a constant magnetic field a cylindrical solenoid at the output has been used, which provided the magnetic field strength \( H = 150 \text{ A/m} \). For deposition of nanoparticles of the coating the reverse magnetic field had been applied during the shots (fig. 1.).

![Diagram of magnet modified detonation installation](image)

**Fig. 1.** Magnet modified detonation installation simplified diagram:
1 – carrying gas; 2 – charge bin; 3 – spark plug; 4 – gun tube of plant; 5 – substrate (specimen); 6 – coating; 7 – flushing gas between shots; 8 – combustible gas; 9 – solenoid coil (direct polarity when it is toward substrate, reverse polarity when it is backward from substrate.).

For research of structure and phase composition of the structure and phase storage of ceramic on the basis of crystalline materials, the friction surfaces was conducted by metallography, X-ray-phase (RPA) and micro X-ray spectral (MRSA) analyses. The metallography analysis of the investigated materials was carried out on the optical microscopes МИМ-8 and «НЕОФОТ».

Radio-phase analysis of specimens was executed on the X-ray diffractometers ДРОН-2.0 (see fig. 2.5.1.) in CuK\(_\alpha\) radiation. Micro X-ray spectral analysis and receiving of electronic images of surfaces was conducted on electron microscope РЭМ-106И

### Research results and discussion

Composite coatings SIAL-M32 (SiC-Al\(_2\)O\(_3\) – 32 hours milling) has a high wear resistance in a compact form due to the formation of films of complex oxide systems SiO\(_2\), Al\(_2\)O\(_3\) on the surface of the friction, which, as a result of the dissolution of iron oxide, form secondary structures. In the process of grinding in the steel vessels, steel milling bodies in the batch mixture form pieces of iron, which have a size from 250–400 nm (fig. 2.). Large particles can be easily removed by magnetic clean. Nanoscale particles cannot be removed from the charge with magnetic clean. Particles of this size are evenly distributed in the batch mixture without bundle and segregation, and their size is not sufficient to absorb larger particles of silicon carbide during the formation of silicides. The presence in the batch mixture of particles of iron millings should intensify the process of coating deposition using the magnetic field flux (double sided).

![SEM images](image)

**Fig. 2.** SEM image of SiC-Al\(_2\)O\(_3\) composite powders after mixing and grinding in steel vessels by steel milling bodies for 32 hours; a – 4000 zoom; b – 10,000 zoom. (250–400 nm particles are detected).

In order to investigate the formation of a grinding of iron in the process of grinding of the components of SiC-Al\(_2\)O\(_3\) batch mixture, the kinetics of the change in the content of particles of iron millings was determined experimentally, depending on the duration of grinding. As a result of the grinding of SiC-Al\(_2\)O\(_3\) batch mixture powders with varying the duration of process, the following regularities were established (table 3.).
Table 3

<table>
<thead>
<tr>
<th>Grinding time, h</th>
<th>1</th>
<th>2</th>
<th>4</th>
<th>8</th>
<th>16</th>
<th>32</th>
</tr>
</thead>
<tbody>
<tr>
<td>Material designation *</td>
<td>SIAL M1</td>
<td>SIAL M2</td>
<td>SIAL M4</td>
<td>SIAL M8</td>
<td>SIAL M16</td>
<td>SIAL M32</td>
</tr>
<tr>
<td>Average size of charge particles, μm</td>
<td>28,7</td>
<td>14,9</td>
<td>6,8</td>
<td>4,8</td>
<td>2,2</td>
<td>2,1</td>
</tr>
<tr>
<td>The content of iron particles, % wt</td>
<td>1,5</td>
<td>3,4</td>
<td>6,8</td>
<td>10,9</td>
<td>16,3</td>
<td>19,3</td>
</tr>
</tbody>
</table>

The phase content of the milled batch mixture is shown on fig. 3.

Fig. 3. X-ray diagram of powder SIAL-M32 composition.

The comprehensive detonation coating research is on the fig. 4. There the zoom kinetics and general imagination of the coating surface roughness and pattern is shown.
While milling the silicon carbide the nanosized particles can be obtained [4]. During the milling the metal particles are acquired in the batch mixture and they are affecting crucially on the coating formation in the magnetic fields. In particular, applying the magnetic field the microsized coatings are acquired and well investigated [9]. This coating is acquired by the technique described in paper [9,10] on the direct flux (from S to N) of the magnetic field, merely, enspeeding the metal particles to the substrate. Changing the polarity of the magnetic flux (from N to S) permanent magnet coil the microscopic particles are retained in the detonation shots stream and only nanosized particles of the batch mixture are reaching to the surface and depositing there (fig. 4. and 5.).

The scanning electronic image (SEM) is supplied on the fig 5. Within the 20 000 electronoc zoom (fig. 5. a) the surface of the coating appeared to be very rough and containing the nanosized particles. Within the 40 000 electronoc zoom (fig. 5. b) the particles of 70,9, 115,2, 76,5, 54,0, 50,5, 65,6, 82,9 and 73,0 nanometers are acquired.

The content of the nanoparticles is about 85% of SiC, 10% of Al₂O₃ and 5% of Fe₂C. No metallic particles were detected in the coating content. Coating thickness was about 50-60 micrometers.

Conclusions

Using the gas detonation deposition from the batch mixture which contains the silicon carbide and aluminium oxide particles, which have a size from 250-400 with the steel millings, on the direct polarity the fine grained microstructure is acquired and on the reverse polarity the nanostructure the particles of 70,9, 115,2, 76,5, 54,0, 50,5, 65,6, 82,9 and 73,0 nanometers had been acquired.
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Довгаль А.Г., Приймак Л.Б. Дослідження структури нанорозмірних карбідокремнієвих покриттів триботехнічного призначення

Представлене дослідження пов’язане з розробкою зносостійких покриттів. Світовий досвід підвищення зносостійкості накопичив велику кількість статистичного матеріалу відомих деталей через зношування. Тому питання дослідження та покращення зносостійких властивостей деталей машин є одним із приоритетних компонентів при розгляді напрямків забезпечення надійності експлуатації транспортних засобів та вузлів тертя. Покриття зі SiC були нанесені на середньовуглецеву сталь детонаційним напиленням. Було встановлено, що воно дуже чутливе до режимів нанесення покриттів та були виведені різні фізичні та хімічні явища. Структура отриманих покриттів була вивчена методами електронної мікроскопії. Отримані покриття були розроблені для подальших триботехнічних випробувань на машинах тертя, що моделюють процеси тертя, що мають місце у парах корінних та шатунних шийок двигунів внутрішнього згоряння. Покриття мають також і коррозійну захисну властивість. Було випробувано новий магнітно модифікований метод детонаційного напилення нанорозмірних покриттів на середньовуглецеву сталь. Було визначено оптимальні режими магнітномодифікованого нанесення покриттів для карбідокремнієвої шихти стосовно структури

Ключові слова: знос, зносостійкість, покриття, адгезія, нанорозмірне покриття, детонаційне нанесення покриттів