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Spraying of TiB_2/Ti and HfB_2/Ti composite powder wear-resistant coatings

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Abstract. In this paper we studied the synthesis of composite core – shell powders sprayed as wear-resistant metal-ceramic coatings. High-hardness TiB_2 and HfB_2 powders form the core, and the shell is made of titanium. The cladding was applied by iodide transport technology. This cladding method involves deposition by gas transport with iodine as an agent. The TiB_2/Ti and HfB_2/Ti composite powders were sprayed using microplasma technology. In contrast to conventional plasma spraying, it minimizes the phase transformations in the composite powders induced by heating. Analysis of the final coating on polished cross sections revealed that during microplasma spraying, the titanium is oxygenated and it produces a titanium dioxide phase. As a result, the TiB_2/Ti and HfB_2/Ti composite powders are transformed into TiB_2 (TiB)/Ti(TiO_2) and HfB_2/Ti(TiO_2) coatings. We also studied the distribution of the components across the coating. The hardness measurements showed that the titanium diboride coatings obtain microhardness of 1300 HV. The microhardness of the hafnium diboride coatings is about 1600 HV. For abrasion testing of the TiB_2 (TiB)/Ti(TiO_2) and HfB_2/Ti(TiO_2)) coatings we used uncoated alloyed 45Kh steel (similar to EU grade: 41Cr4) and the specified coatings as an abradant material. Despite their lower microhardness, the TiB_2(TiB)/Ti(TiO_2) coating showed the highest abrasion resistance.

Keywords: wear-resistant coatings, clad composite powders, titanium diboride/titanium, hafnium diboride/titanium, microplasma spraying, protective and restorative coatings

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Напыление износостойких покрытий из плакированных порошков TiB_2/Ti и HfB_2/Ti

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Аннотация. В представленной работе приведены результаты по синтезу композиционных плакированных порошковых систем с типом строения «ядро–оболочка» для напыления износостойких металлокерамических покрытий. Для синтеза композиционного порошка в качестве ядра использованы порошки высокотвердых боридов TiB_2 и HfB_2, а для создания оболочки на их поверхности – титан.

Синтез плакирующего слоя осуществляли йодотранспортным методом. Плакирование порошка используемым методом подразумевает осаждение одного компонента на другой посредством газотранспорта, агентом которого выступает йод. Напыление композиционных плакированных порошков систем TiB_2/Ti и HfB_2/Ti осуществляли микроплазменным методом, который, в отличие от классического плазменного напыления, позволяет минимизировать фазовые превращения в композиционных порошках из-за термического воздействия. При исследовании поперечных микрошлифов напыленных покрытий определено, что в процессе микроплазменного напыления титан насыщается кислородом, образуя фазу диоксида титана. В результате плакированные композиционные порошки систем TiB_2/Ti и HfB_2/Ti превращаются в покрытия из систем TiB_2(TiB)/Ti(TiO_2) и HfB_2/Ti(TiO_2). Выявлены особенности распределения компонентов по толщине покрытия. Исследования твердости показали, что у покрытий на основе диборида титана интегральное значение микротвердости составляет 1300 HV. У покрытий на основе диборида гафния интегральная микротвердость составила порядка 1600 HV. При исследовании износостойкости пары с покрытиями TiB_2(TiB)/Ti(TiO_2) и HfB_2/Ti(TiO_2) сопрягались с контртелом образца из стали 45Х без покрытия и совместно друг с другом. Несмотря на менее высокую микротвердость, наиболее износостойким является покрытие системы TiB_2(TiB)/Ti(TiO_2).

Ключевые слова: износостойкие покрытия, плакированные композиционные порошки, диборид титана/титан, диборид гафния/титан, микроплазменное напыление, защитные и восстановительные покрытия

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Introduction

New structural materials and coatings are developed to offer better performance and reliability. New composites extend the service life of products operating at high rates under elevated temperatures, mechanical loads or exposed to aggressive environments.

Borides of transition metals, such as TiB_2 and HfB_2 are promising ones since they have a high level of hardness, heat, wear and corrosion resistance [1 – 5]. There are few boride coating application technologies. Jayaraman S. et al. [6] produced HfB_2 and Hf–B–N coatings by chemical vapor deposition (CVD). The hardness of the coatings is up to 40 GPa. The CVD technology is also used to apply ultra-high-temperature transition metal boride coatings onto porous substrates. The application technology for such coatings consist of thermal gas-phase decomposition of titanium, zirconium and hafnium borohydride solutions in high-boiling saturated hydrocarbons as borohydride and solvent vapor are passed through porous materials preheated to 250 °C, and then placed in a tubular reactor under vacuum (Dugin S.N. et al. [7]).

Composite HfB2/SiC coatings protect from oxidation in various aggressive environments [8 – 12] and are therefore of a great interest. Silicon carbide alloying with hafnium diboride significantly increases the structural strength at high temperatures, thermal conductivity and heat resistance while reducing the thermal expansion coefficient. Such coatings are extensively used under high temperatures and contact loads, e.g. in solid-propellant rocket engine components [13].

Spark plasma sintering (SPS) is used for sintering ceramic powders. This technology produces ultra-high-temperature and high-strength ceramics [14 – 16]. The standard temperature of HfB_2–SiC spark plasma sintering is 1800 – 2100 °C [17].

Despite their excellent performance and physical/structural properties, such coatings are not widely used due to their high brittleness and the lack of direct application technologies. Therefore, it is advisable to use boride composites with binding metals.

Gas cladding is an efficient technology for making highly dispersed composite powders; one component is converted into a gas phase and then deposited on the surface of the other component. Iodine is used as a transport agent. Bogdanov S.P. [18; 19] studied the iodine transport of cladding components in detail.

As components operated under high contact loads are restored a layer up to 1 mm thick [20] is mostly applied.

Thermal spraying is considered a suitable technology for making coatings of such thicknesses [21]. In particular, the microplasma technology can spray composite powders while maintaining their high physical and structural properties. 20 – 300 μm thick coatings are applied with a single pass.

The above-mentioned composite powders can be used as protective and repair coatings for mechanical parts exposed to high contact loads, variable temperatures and aggressive or corrosive environments in heat exchangers, steam generators, pipelines, valves and jet engines.

The purpose of this study is to apply the iodine transport technology to TiB_2/Ti and HfB_2/Ti composite pow-

Table 1

Chemical composition of PTOM-1 powder

Таблица 1. **Химический состав порошка марки ПТОМ-1**

Powder grade	Titanium	Wt. % (max)					
		nitrogen	carbon	hydrogen	iron + nickel	silicon	calcium
PTOM-1	Core	0.08	0.05	0.40	0.40	0.10	0.08

ders, fine-tune the microplasma spraying process, and estimate the properties of the resulting coatings.

Materials and Methods

We used the following materials:

– PTOM-1 titanium powder, 10 – 100 μm grade from POLEMA, AO (see Table 1 for the chemical composition);

– titanium diboride powder, 1 – 4 μm grade (the chemical composition is: 68.3 % titanium; 30.2 % boron; 0.1 % carbon; 0.05 % iron).

– HfB_2 hafnium diboride powder, 3 – 12 μm grade, 99.8 % purity (the chemical composition is: boron 29 %; the rest is hafnium).

Fig. 1 shows the SEM images of the powders. The powders were mechanically mixed. The MeB_2:Ti mass equivalent ratio was 50:50 %, where *Me* is Ti or Hf. The mixtures were then synthesized by the iodine transport technology. The titanium was converted into a gas phase by a chemical reaction with iodine vapor and carried to the surface of ceramic particles. The mass transfer intensity depends on the temperature and the holding time. The temperature was 700 °C, and the holding time was 3 h. The resulting TiB_2/Ti and HfB_2/Ti composite powders, 20 to 80 μm grade were deposited with the microplasma technology on UGNP-7/2250 machine equipped with Kawasaki FS003N robot manipulator. The plasma generator power reached 2.8 kW and the arc current was 35 to 40 A at 40 V. Argon (2 l/min flow rate) served as the transport agent and plasma gas.

We studied the structure of the powders and the polished cross-sections of the coatings on Tescan Vega 3 scanning electron microscope (SEM). PMT-3 microhardness tester was used for the coating microhardness testing by Vickers method.

The 2168 UMT abrasion testing machine performed accelerated wear resistance tests. It is a general-purpose machine with replaceable friction pairs and multiple available motions in a wide range of rpm and loads. Lubricants can be delivered to the friction area. The samples had coating sprayed on their ends. They were arranged as a ring-on-ring friction pair with continuous water cooling. The independent variables were the normal load applied to the ring, the ring rpm and the test time. All the samples were examined under the 0.5 MPa load at 100 rpm for 5 h.

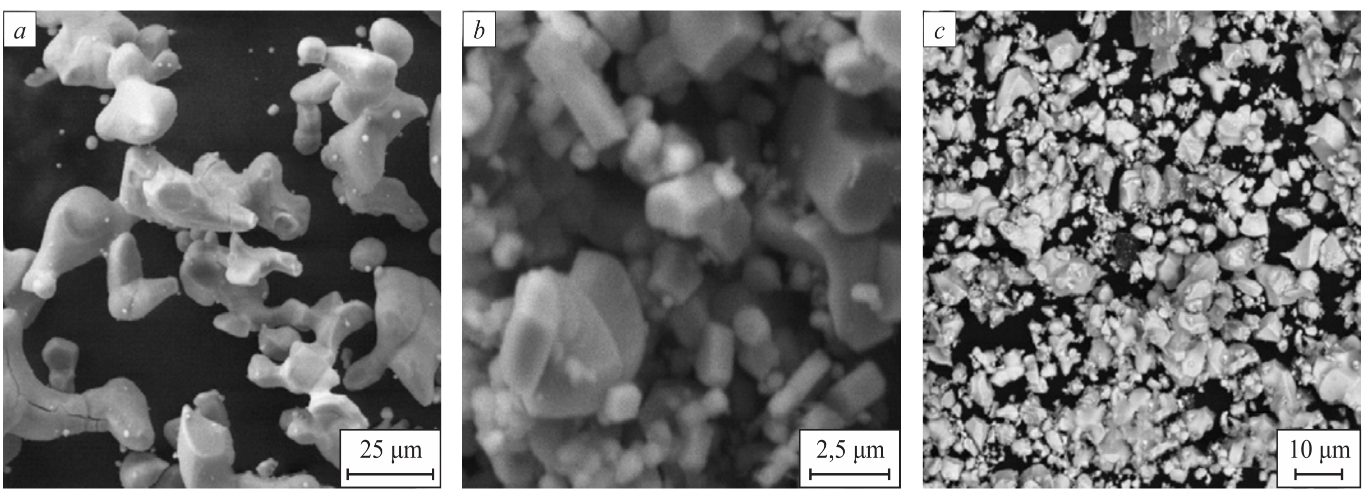

Fig. 1. SEM micrographs of the powders:
a – PTOM-1, *b* – TiB_2, *c* – HfB_2

Рис. 1. РЭМ-микрофотографии исходных порошков:
a – ПТОМ-1; *b* – TiB_2; *c* – HfB_2

Results and Discussion

We examined the structure of the TiB_2/Ti and HfB_2/Ti composite powders processed by the iodine transport technology. The typical structure is shown in Fig. 2.

The composite particles mostly inherit the shape of the original ceramic components, while some of the particles remain unclad and they are merged in agglomerates.

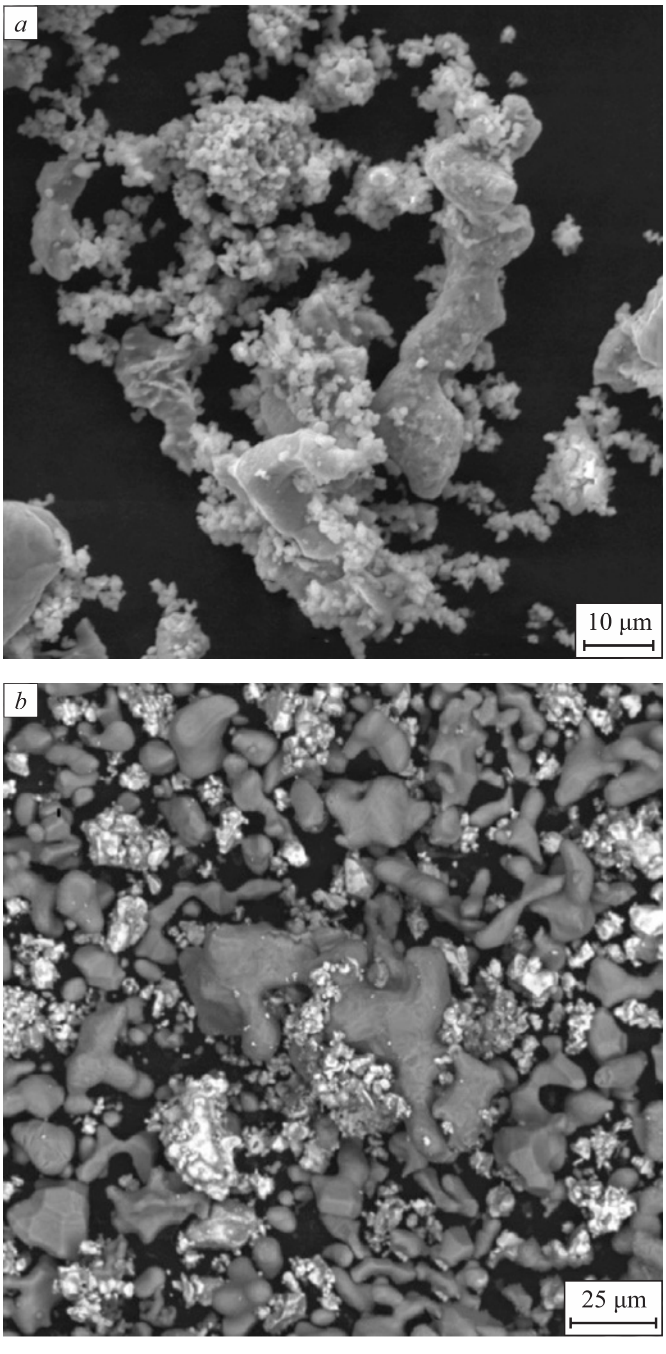

Fig. 2. SEM images of the clad powders:
a – TiB_2/Ti, *b* – HfB_2/Ti

Рис. 2. РЭМ-микрофотографии плакированных порошков:
a – TiB_2/Ti; *b* – HfB_2/Ti

Fig. 3 and 4 show the SEM images of the TiB_2/Ti and HfB_2/Ti composite powder coating polished cross-sections respectively.

The images in Fig. 3 and 4 indicate that the coatings have clear interfaces with the substrate, without through pores. The dark and bright areas in the backscattered electron SEM images indicate that the titanium diboride and hafnium diboride particles are preserved in the coating. Fig. 3 shows that the light areas are titanium and the dark areas are titanium diboride. In the SEM images of the coatings (Fig. 4) the darker areas are rich in titanium and the lighter areas are rich in hafnium, due to the higher atomic number of the latter. The titanium diboride coating changes its phase composition during spraying. The diboride is partially converted into titanium monoboride. A similar effect was given in [22; 23]. The hafnium diboride powder coating does not feature such transformations. Besides, the titanium dioxide phase is formed in the coatings due to the oxygen diffusion during spraying. It can be identified as the areas with an intermediate contrast between the areas containing the ceramic and the binder.

We examined the microhardness of the polished cross-sections of the coatings. The hardness of TiB_2(TiB)/Ti(TiO_2) coating is 1320 HV under 100 g load and 1.0 rms deviation. The hardness of HfB_2/Ti(TiO_2) is 1654 HV under 200 g load and 3.2 rms deviation.

The results indicate high hardness values of the ceramic components in the coatings. HfB_2/Ti has higher hardness because the ceramic component does not have a phase transition. As mentioned above, the titanium diboride is saturated with titanium at spraying, which resulted in monoboride formation. It slightly decreases the final hardness, since the hardness of the titanium diboride reaches 35 GPa, while that of the titanium monoboride is 28 GPa [24; 25] only.

We also tested the sprayed coatings for abrasion resistance. First, we used 41Cr4 steel as an abradant material (Table 2, tests *1* and *2*). Then we tested the abrasion resistance of two samples with two types of coatings (Table 2, test *3*). Table 2 shows the weight changes, weight wear and wear rates for each friction pair.

The weight wear of TiB_2(TiB)/Ti(TiO_2) coating is 28 %, which is less than that of the steel part. The mass losses of the sample and the abradant materials were low. The similar test with a friction pair of a hafnium diboride coating sample and 41Cr4 abradant material showed that the sample mass loss increases while the steel body wear is 690 % higher than that of the coated sample. The reason is the higher hardness of HfB_2/Ti(TiO_2) coatings.

Abrasion tests of TiB_2(TiB)/Ti(TiO_2) and HfB_2/Ti(TiO_2) coatings showed that the titanium diboride coating had higher wear resistance.

The weight wear of HfB_2/Ti(TiO_2) coating is 290 % higher than that of TiB_2 (TiB)/Ti(TiO_2) coating despite its higher microhardness. This can be explained by better elasticity of titanium diboride coatings, since spraying forms titanium monoboride. The substance creates a chemical bond between crystal lattices of initial phases, which prevents the ceramic component removal by friction. The hafnium diboride coating lacks such a bond. As a result, HfB_2/Ti(TiO_2) coating wear rate is higher than that of TiB_2 (TiB)/Ti(TiO_2) coating under identical contact loads.

Conclusions

We synthesized TiB_2/Ti and HfB_2/Ti metal-ceramic powders. The initial mass ratio of the components in the mixture was 50:50 %. The synthesis lasted for 3 h at 700 °C.

We studied the properties of TiB_2/Ti and HfB_2/Ti composite powder coatings applied with the microplasma technology.

The structural examination showed that the particles of ceramic components are fixed in the matrix without any through pores. During spraying the energy input changes the phase composition of titanium diboride composite powders from TiB_2/Ti to TiB_2 (TiB)/Ti(TiO_2).

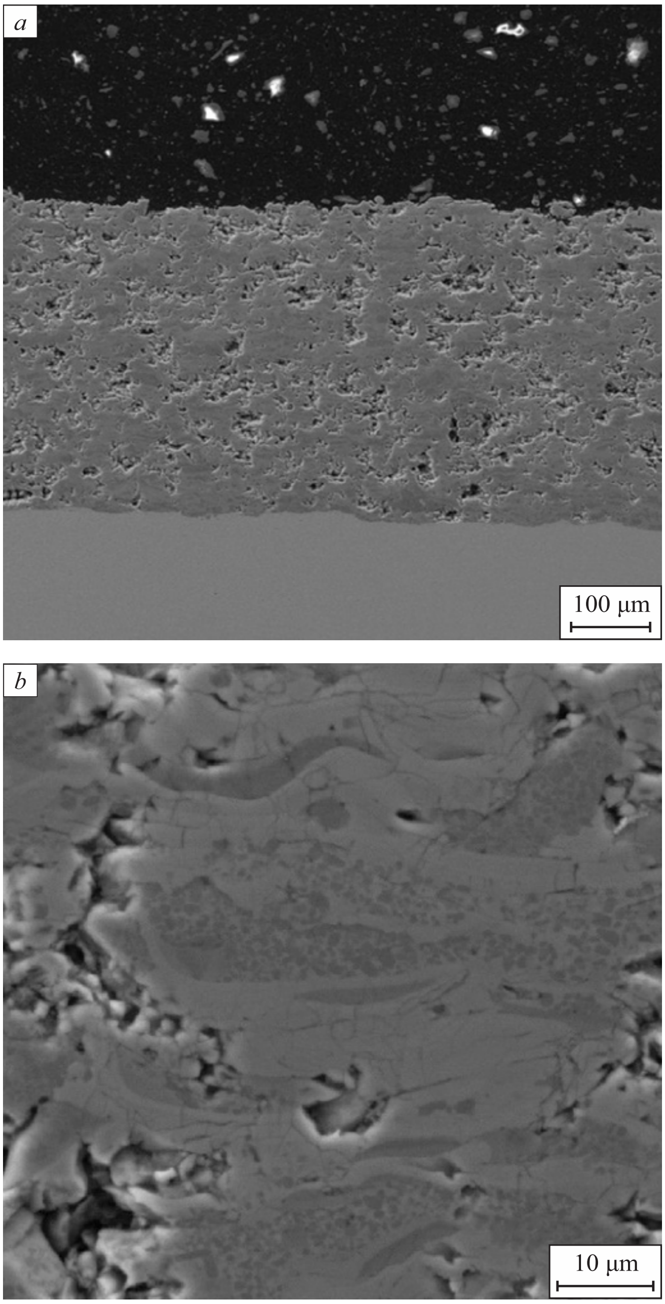

Fig. 3. Backscattered electron SEM images of TiB_2/Ti coating polished cross-section: *a* – ×300, *b* – ×3600

Рис. 3. РЭМ-микрофотография в отраженных электронах поперечного микрошлифа покрытия из TiB_2/Ti: *a* – ×300; *b* – ×3600

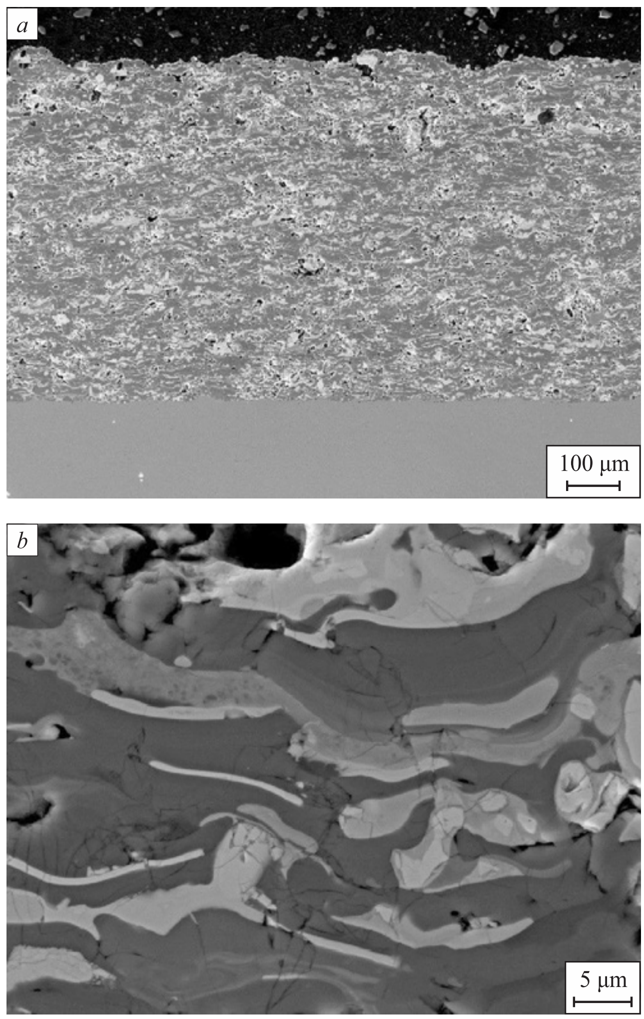

Fig. 4. Backscattered electron SEM images of HfB_2/Ti coating polished cross-section: *a* – ×300, *b* – ×3600

Рис. 4. РЭМ-микрофотография в отраженных электронах поперечного микрошлифа покрытия из HfB_2/Ti: *a* – ×300; *b* – ×3600

Table 2

Wear resistance properties of the coatings

Таблица 2. **Показатели износостойкости исследуемых систем материалов**

Test number	Friction pair	Sample weight, g			Weight wear, g/km	Wear rate, g/h
		before testing	after testing	Δm		
1	45H	37.4536	37.4522	0.0014	0.000619250	0.00028
	TiB_2(TiB)/Ti(TiO_2)	36.9823	36.9813	0.0010	0.000442321	0.00020
2	45H	37.4775	37.4632	0.0143	0.006325195	0.00286
	HfB_2/Ti(TiO_2)	37.3925	37.3907	0.0018	0.000796178	0.00036
3	TiB_2(TiB)/Ti(TiO_2)	37.0552	37.0451	0.0101	0.004467445	0.00202
	HfB_2/Ti(TiO_2)	37.6530	37.6130	0.0400	0.017692852	0.00800

Hardness of TiB_2 (TiB)/Ti(TiO_2) coating is 1320 HV and the hardness of HfB_2/Ti(TiO_2) coating is 1654 HV. The abrasion tests indicated that for the steel grade 45H abradant material, the weight wear of the coatings is 28 % less than the wear of the steel body. TiB_2 (TiB)/Ti coating offers the best performance with the lowest wear under a contact load with a steel body.

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S. P. Bogdanov – synthesis of composite clad powders of the studied systems.
A. V. Krasikov – participation in tests to determine wear resistance.

M. V. Staritsyn – study of materials on a scanning electron microscope.
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