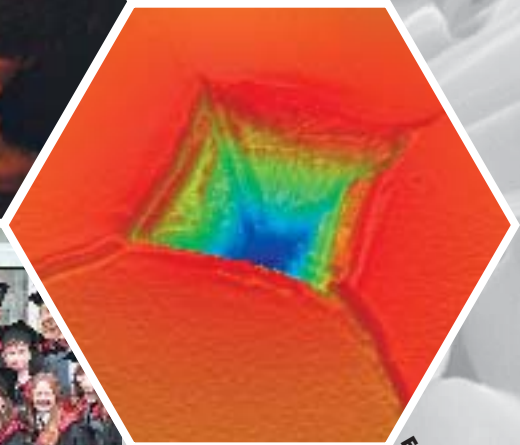


THE SCIENTIFIC-EDUCATIONAL CENTER OF SHS

PHYSICS OF COMBUSTION



MATERIALS SCIENCE



MACROKINETICS



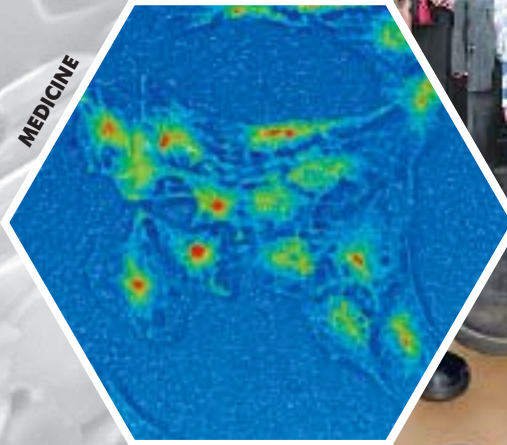
STRUCTURAL



ENGINEERING INDUSTRY



MEDICINE



POWDER METALLURGY



The Scientific-Educational Center of SHS (SHS Center) of the National University of Science and Technology (MISIS) and the Institute of Structural Macrokinetics and Materials Science Problems (ISMAN) was established by the joint resolution № 744/119 dated September 21, 1989 of the State Committee of the Soviet Union for Public Education (approved by G.A. Yagodin) and the Presidium of the USSR Academy of Sciences (approved by G.I. Marchuk). The SHS center was created on the basis of the Moscow Institute of Steel and Alloys (MISIS) and the Institute of Structural Macrokinetics of the USSR Academy of Sciences (today known as ISMAN) to be the research and education complex combining the effort and resources of the higher education institution and those of the academic institution in conducting fundamental research, developing and launching the inventions at plants, and educating and retraining specialists in various aspects of research problems.

The outstanding researchers and organizers of science, Professor A.G. Merzhanov (the founder of SHS and the Institute of Structural Macrokinetics of the USSR Academy of Sciences; today, he is the Academician and research supervisor at the ISMAN) and Professor N.N. Khavskii, who was the vice-rector for research at the MISIS, were the initiators of the idea of integrating the higher education institution and the academic institute. The functions and tasks to be solved, as well as statute and structure of the Center were determined by this resolution. Prof. I.P. Borovinskaya and Prof. N. N. Khavskii were appointed to be the first research supervisors of the SHS Center. E.A. Levashov (today, Doctor of Technical Sciences, Professor, Honorary Professor of the Colorado School of Mines, Director of

the SHS Center, and the Head of the Department of Powder Metallurgy and Functional Coatings of MISIS) was appointed to be the first director and is still in charge for the work of the Center.

Since the very first days of its existence as a structural subdivision the SHS Center has united the leading researchers of the ISMAN and MISIS in the field of chemical physics, physics of combustion and explosion processes, structural macrokinetics, physical materials science, powder metallurgy, metal treatment by pressure, and the theory of metallurgical processes. Only after nine years, the idea of establishing the scientific-educational centers of this type has been implemented and developed within the presidential Federal Targeted Program «State Support of Integration of Higher Education and Fundamental Science for 1997–2000», and after 19 years, within the framework of the Federal Targeted Program «Scientific and Scientific-Pedagogical Personnel of Innovative Russia in 2009–2013».

More than 23 years history of the SHS Center is rich in achievements and has reinforced the position of the Center in Russia and even abroad. Today, the SHS Center has authority for the development of novel materials (ceramics, metal ceramics, intermetallics, composites and functional gradient materials, multicomponent and multilayer nanostructured films, hard tribological coatings, anticorrosion and heat-resistant coatings, multifunctional bioactive nanostructured films, self-lubricating coatings, and nanoparticle dispersion-strengthened materials and coatings), for their fabrication technologies (SHS, powder metallurgy, magnetron sputtering (MS), ion implantation assisted magnetron sputtering, pulsed electrospark de-

position (PED), thermo-reactive electrospark surface strengthening (TRESS)), as well as for certification of mechanical and tribological properties and metrology of nanostructured surfaces.

The SHS Center has conducted research under grants of international funds and programs, such as CRDF, INTAS, NATO-Russia, «Eureka», International Science and Technology Center, UK Royal Society, as well as the Sixth and Seventh EU framework programs.

More than 30 highly qualified researchers, including 6 full doctors of science and professors, 12 PhD and associate professors, 12 engineers, 4–6 postgraduate students, and 7–12 students are employed by the Center. The researchers are the members of the Dissertation Defense Boards D 212.132.05 at the MISIS and D 002.092.01 at the ISMAN, the members of editorial boards of the «Russian Journal of Non-Ferrous Metals», «International Journal of Self-Propagating High-Temperature Synthesis», «Physical Surface Engineering», «Metal Science and Heat Treatment», and International Scientific Journal «Ecological Bulletin of Research Centers of the Black Sea Economic Cooperation». Furthermore, they are the members of the scientific councils and committees, such as the Scientific Council on Combustion and Explosion of the Russian Academy of Sciences; the International Committee on Functional Gradient Materials; the European Joint Committee on Plasma and Ion Surface Engineering; the International Committee of the Conference on Plasma Surface Engineering; the Russian-French International Committee «Advances in Materials Science and Environmental Protection»; «Advanced Coatings and Surface Engineering Laboratory» of Colorado School of Mines; the In-

ternational Committee «Metallurgical Coatings and Thin Films», and the International Committee of the European Conference on Nanofilms.

The staff of the Center have published more than 630 articles in the international and Russian peer-reviewed journals, 67 inventor's certificates and patents (including 20 international patents), and 11 books. The research groups of the SHS Center collaborate with the leading research centers of the USA, Canada, Europe, Israel, Japan, and China.

The education process is carried out in the Center on the following courses: «Structural macrokinetics», «Physical-chemical base for the combustion synthesis of inorganic materials», and «SHS technology» within specialization «Metallurgy» (150100). The Center participates in functioning of the Master School with International Participation «Functional and Nanostructured Materials». Moreover, the leading researchers of the ISMAN participate in the education process.

The SHS Center consists of the following structural subdivisions:

- Scientific-Technological Sector of SHS technologies;
- Group of Mechanical Activation of SHS Mixtures;
- Sector of Pulsed Electrospark Deposition (PED), Thermo-Reactive Electrospark Surface Strengthening (TRESS), and chemical surface treatment technologies;
- Laboratory of Thin Films and Coatings Engineering;
- Laboratory of Functional Surfaces Characterization;
- Group of Nanopowdered Materials;
- Pilot Section for the Ion-Plasma Treatment of Multicomponent Bioactive Nanostructured Coatings (MUBINAF).



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RESEARCH AREAS

- Theory of SHS. Structural macrokinetics, the mechanisms of phase- and structure formation of heterogeneous chemical reactions products in the combustion wave of SHS systems. Mechanical activation of exothermic mixtures as an efficient method for controlling the process kinetics and product properties.
- Development and synthesis of new classes of nanoparticle dispersion-strengthened materials, as well as ceramic and metal-ceramic materials; dispersion-hardened alloys.
- Development and synthesis of nanostructured composite electrode materials for the processes of electron-ion plasma and ion-beam sputtering, pulsed electrospark deposition, and thermo-reactive electrospark surface strengthening.
- Development of multifunctional and functional gradient materials (FGMs), including diamond-containing tool, electrodes, targets-cathodes, and impact-resistant materials.
- Plasma physics, theory of ion-plasma and ion-beam processes.
- Kinetics and the mechanism of nanostructured thin films and coatings grows (superhard, bio-compatible, heat-resistant, anticorrosion, optical, and resistive ones) deposited by magnetron sputtering, ion implantation, pulsed laser deposition, PED and TRESS using composite SHS targets and electrodes.
- Development of the metrological complex and normative-methodological base to ensure the uniformity of measurements of mechanical and tribological properties of nanostructured surfaces and nanoindustry products.
- Development and synthesis of refractory ceramics for metallurgical applications. Investigation of the regularities in interaction between ceramic materials with metallurgical alloys. Development of new refractory compositions, including the ones used for centrifugal casting of precision alloys for medical applications.
- Development and synthesis of heat-resistant composite materials with intermetallide matrix.

The specified research area is innovation-oriented and corresponds to the priority direction of the development of science and technology «Industry of Nanosystems and Materials», critical technologies: «Nanotechnologies and Nanomaterials», «Technologies for Fabrication and Treatment of Crystalline Materials with Special Properties», «Technologies for Fabrication of Composite and Ceramic Materials», and «Technologies for Fabrication of Biocompatible Materials». The specified directions are developed from the fundamental and problem-oriented scientific research through the development activities, fabrication of pilot product samples and batches, conducting tests, as well marketing and patent research, towards the execution of batch production and rendering research advisory services.

SCIENTIFIC RESULTS ALREADY ACHIEVED

Theory and practice of SHS. Structural macrokinetics

The theoretical models of combustion and structure formation processes have been developed:

- The equation of transition from the diffusion combustion mode to the capillary spreading mode in its criterial form has been obtained for the solid–liquid systems (e.g., the ones based on titanium–carbon). Experimentally confirmed, this equation links the thermophysical, hydrodynamic, and diffusion parameters of the system with mixture composition and dispersity of the initial reagents.

- The competitive filling model for describing the macrokinetic characteristics of the combustion processes in capillary porous systems containing melts of the reagent and inert fill material.

- The model of propagation of a thermal and chemical wave of gas-free combustion in multi-layer systems.

- It was first ascertained via high-speed video imaging of a combustion wave that at the micro level, the combustion zone is a combination of the explosion foci caused by the occurrence of a chemical reaction in separate unit cell, in which the reaction surface was formed.

- The mechanisms for structure formation of ceramic and metal-ceramic compositions in the combustion wave in various heterogeneous systems has been proposed.

- The research principles for controlling the SHS process (elemental synthesis in the solid–liquid systems and filtration synthesis in the solid–gas systems) by powerful ultrasonic fields have been designed. Ultrasound has been demonstrated to be an efficient tool for controlling the structure and properties of the synthesis products based on transition metal carbides, borides, and intermetallides.

Mechanical activation of exothermic mixtures

- The relationship between the structure and properties of the mechanically activated powder mixtures, as well as that between the physicochemical parameters of the combustion reactions and structure of the final products, has been established. Mechanical activation enables carrying out SHS in low exothermic mixtures, including those strongly diluted with an inert additives. Mechanical activation results in an increase in the heat release rate and mixture reactivity due to particle disintegration, formation of the layered structure of grains, reduction of the coherent scattering regions, and an increase in density of structural defects and dislocations.

- The technological regimes of mechanical activation of the reaction mixtures for a number of compositions (Ti–Si, Mo–Si, Ti–Cr–C, Ti–B, Ti–BN, Ti–Si₃N₄, Ti–Cr–B, Cr–B, Mo–B, Ti–Ta–

C, Ni–Al, Ti–Cr–Al–C, Ti–Si–C, Mo–Si–B, Cr–Al–Si–B, etc.) have been optimized.

- It was ascertained that the adsorbed and dissolved oxygen in powder reagents of the Cr–B, Ti–Cr–B, and Ti–Ta–C systems plays an active role. It determines the mass transfer of boron and carbon to the reaction surface and reduces the effective activation energy of the combustion process. An increase in oxygen content in the reaction mixture leads to an increase in contribution of gas transfer. Previously unstudied ternary compounds (Ti₂CrB₂ and Cr₄Ti₉B) were identified among the synthesis products.

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New classes of ceramic and metal-ceramic materials Dispersion-strengthened and dispersion-hardened alloys

Two types of bulk structural materials dispersion-strengthened with nanoparticles, which exhibit the effect of simultaneous nanoparticle strengthening of carbide grains and the metal matrix, have been designed.

1. Dispersion-strengthened-type ceramic materials based on titanium carbide with the effect of simultaneous dispersion strengthening of carbide grains and metal binder as a result of concentration separation (the occurrence of controlled transformations in a solid solution) of supersaturated solid solutions and the release of excess nanodispersed phases both in-

side the carbide grains (e.g., $\text{Me}^{\text{V}}\text{C}$ or Me^{V} -type phases) and metal binding (e.g., the γ -phase). The fundamental novelty of the materials science approach to designing these materials is as follows. Supersaturated solid solutions can be obtained under conditions of high temperature gradients achieved in the combustion wave of the SHS systems. In accordance with the equilibrium state diagrams, alloying elements are accumulated at high concentration in the area of structurization of solid solutions due to high combustion temperature (up to 2500–3500 °C). Upon rapid cooling at rates of $\sim 10^2$ – 10^3 °C/s, these alloying elements cannot leave the crystal lattice; thus, the solid solution becomes supersaturated. However, subsequent thermal treatment results in concentration separation of solid solutions and the release of excess nanodispersed phases. Thermal treatment conditions, degree of supersaturation, and features of the state diagram allow one to control the size of precipitations-excess phases, whose release results in a considerable enhancement of physicomechanical properties. Hardness, crack growth resistance, ultimate strength, and impact resistance increase simultaneously.

2. Ceramic materials (based on carbides, nitrides, and borides) with the modified structure obtained by introducing of nanosized additives of refractory compo-

unds to the initial mixture, which act as modifiers during the primary and secondary structure formation process via the liquid phase, to the initial mixture. The effect of nanosized additives to the macrokinetic combustion parameters and structure formation of various SHS systems was first studied. The effect of strong modification of the structure of synthesis products was revealed; this effect resulted in a simultaneous increase in strength, hardness, and crack growth resistance. The production technology of these materials was implemented under experimental and industrial conditions.

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Superhard materials

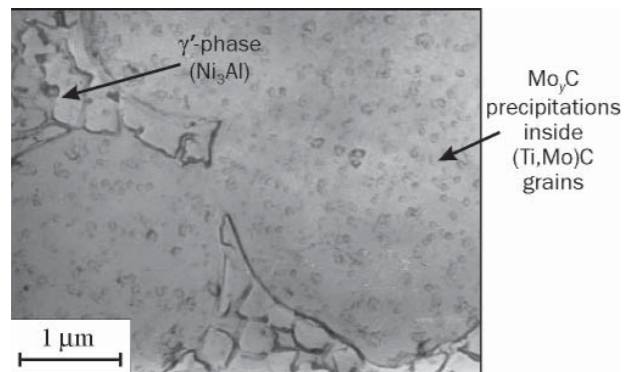
Functional gradient diamond-containing materials with the ceramic matrix (TiB_2 –TiN, TiN– Ti_5Si_3 , TiN–AlN– Ti_5Si_3 , TiC–TiAl, TiC–NiAl, etc.) It was first ascertained both theoretically and experimentally that under certain conditions diamond grains can endure a short-term impact of high-temperature chemical wave without significant changes. The interaction between the initial reagents in the Ti + BN, Ti + Si_3N_4 , and Ti + Al + Si_3N_4 mixtures during the SHS process results in the formation of nitrogen-contain-

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4. *Levashov E.A., Pogozhev Yu.S., Kudryashov A.E., Rupasov S.I., Levina V.V.* TiC–Ni-based composite materials dispersion-strengthened by nanoparticles for electrospark deposition // Russian Journal of Non-Ferrous Metals. 109 (2008). P. 397–403.

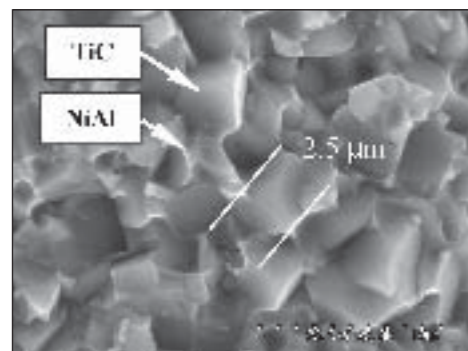
ing atmosphere in the combustion zone, which prevents oxidation and graphitization of the diamond surface.

The diamond cutting tool with the metal matrix composite (MMC) dispersion-strengthened by nanoparticles. The conception of this direction consists in doping the metal binder with strengthening nanoparticles that are to simultaneously solve the following problems:

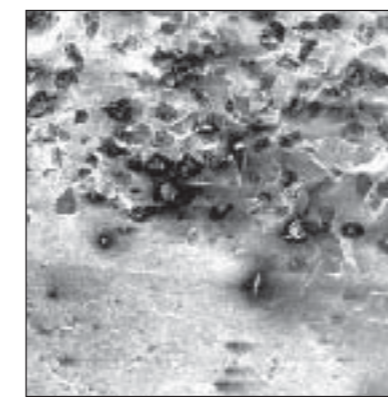
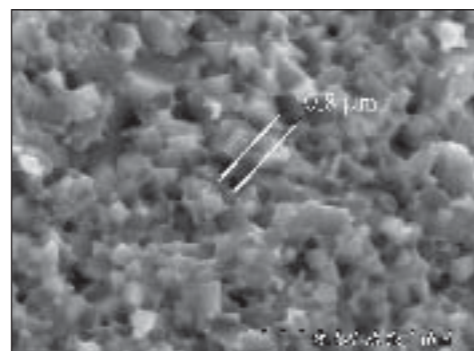
— to change the chemical potential at the diamond–metal binder interphase via doping with re-



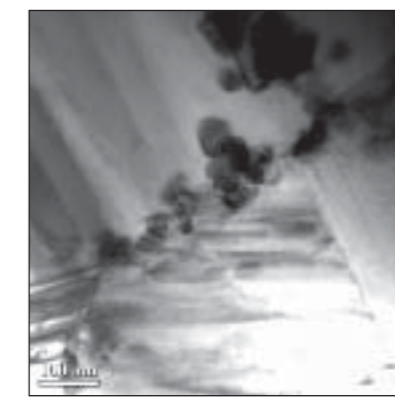
Microstructure of the alloy SHIM-5



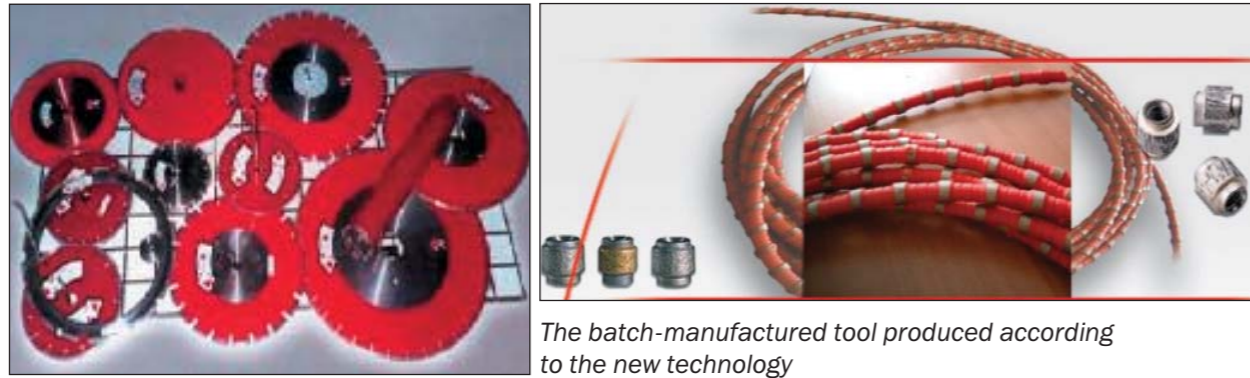
Microstructure of the alloy SHIM-40NA (TiC–NiAl) and the alloy dispersion-strengthened with NbC nanoparticles



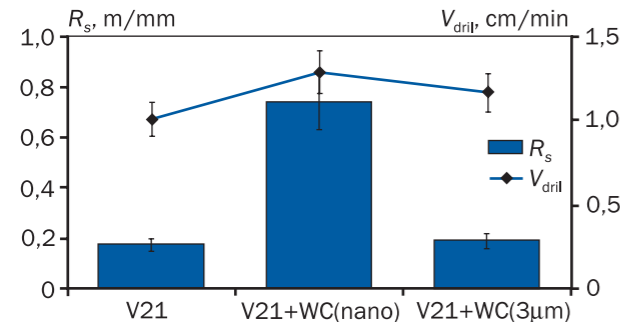
Gradient distribution of diamond grains



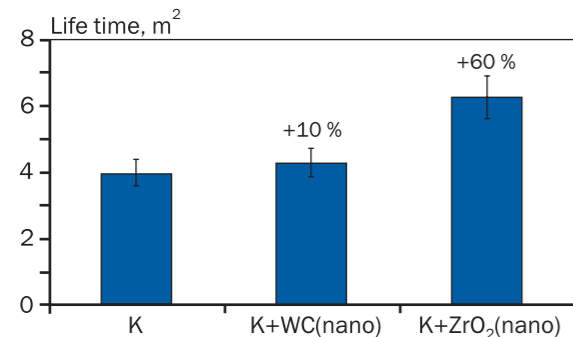
Distribution of ZrO_2 (5–20 nm) and WC (20–80 nm) nanoparticles over grain boundaries and volume of the binder grains (Co extra fine)



The batch-manufactured tool produced according to the new technology



Specific service life (R_s) and drilling speed (V_{drill}) of highly reinforced ferroconcrete using diamond segmented mills with V21 binding strengthened with nano- and micro-sized WC particles



Service life of diamond segmented wheels with the bindings dispersion-strengthened with nanoparticles intended for cutting highly reinforced ferroconcrete

active ability nanoparticles (an increase in the adhesion strength of a diamond grain to the binder);

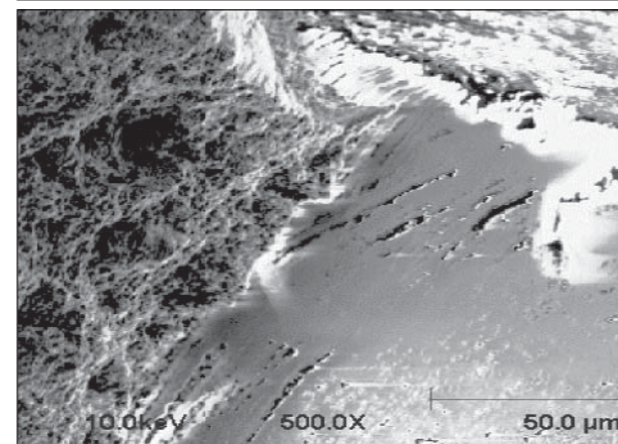
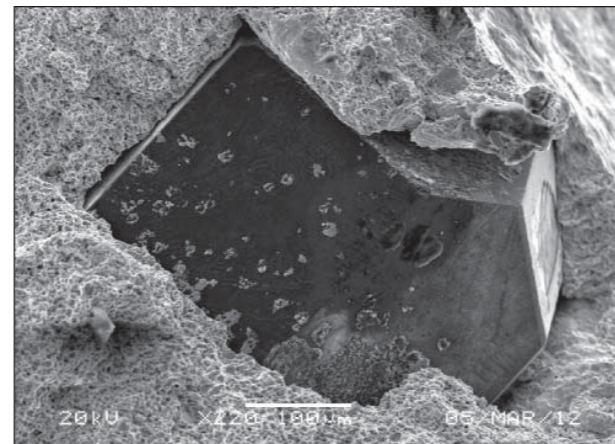
- to ensure dispersion strengthening of the metal binder by introducing nanoparticles into the volume of the grains, which results in the enhancement of hardness, strength, and impact resistance;

- to ensure the doping at the grain boundary of binder by introducing nanoparticles, which results in an decrease in the friction coefficient in the cutting area.

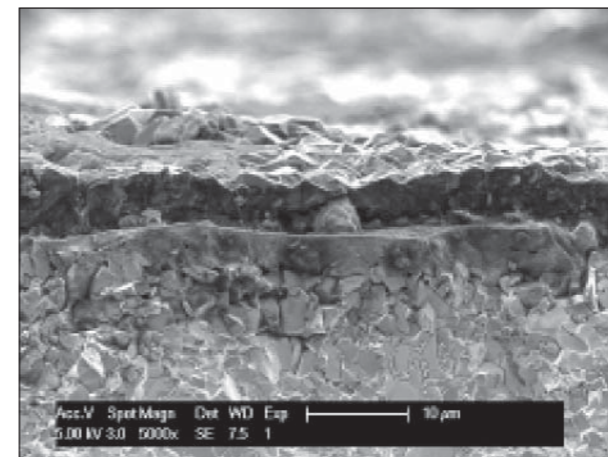
The technology for fabricating diamond-containing materials (including the functional gradient materials) with the binding dispersion-strengthened with nanoparticles has been developed. This technology has been implemented into the batch production of diamond segmented cutting wheels, drills, and rope saws for the building and stone working industries.

CVD-diamond coated WC-Co cutting tool.

It was first demonstrated that the application of pulsed electrospark deposition technology



The fractures of diamond-containing segments demonstrate high adhesion of the diamond to the Co extra fine binding strengthened by nanosized WC



The fracture of the CVD-diamond coated WC-Co insert with the PED-sublayer

using diamond-containing electrodes to form sublayers on the WC-Co cutting tool enables significantly enhancing the adhesion strength

Electrode materials for pulsed electrospark deposition

SHS materials have been successfully used as electrodes in the pulsed electrospark deposition (PED) technology. Electrode materials for multifunctional (e.g., wear- and heat-resistant, anticorrosion, antifriction, possessing enhanced hardness, etc.) coatings on the surface of the products being treated are selected depending on a specific scientific and technical problem to be solved. PED has been successfully used to reinforce or repair the tools and machine elements by varying the frequency–energy regimes and electrode composition (hard alloys, ceramics, intermetallides, metals and their alloys).

The electrode materials can be either rod- or disc-shaped depending on the type of the equipment to be treated. Nanostructured materials produced for mechanized treatment are ring-shaped.

The process of thermoreactive electrospark surface strengthening (TRESS), which combines the PED and the exothermic chemical reaction in the interelectrode gap, has been developed. On one hand, the chemical reaction is maintained by pulsed discharge energy; on the other hand, heat release from the chemical reaction, which is comparable with the discharge energy, increases the total energy of the process. Ad-

ditional heat of the chemical reaction makes it possible to noticeably enhance the efficiency of deposition process.

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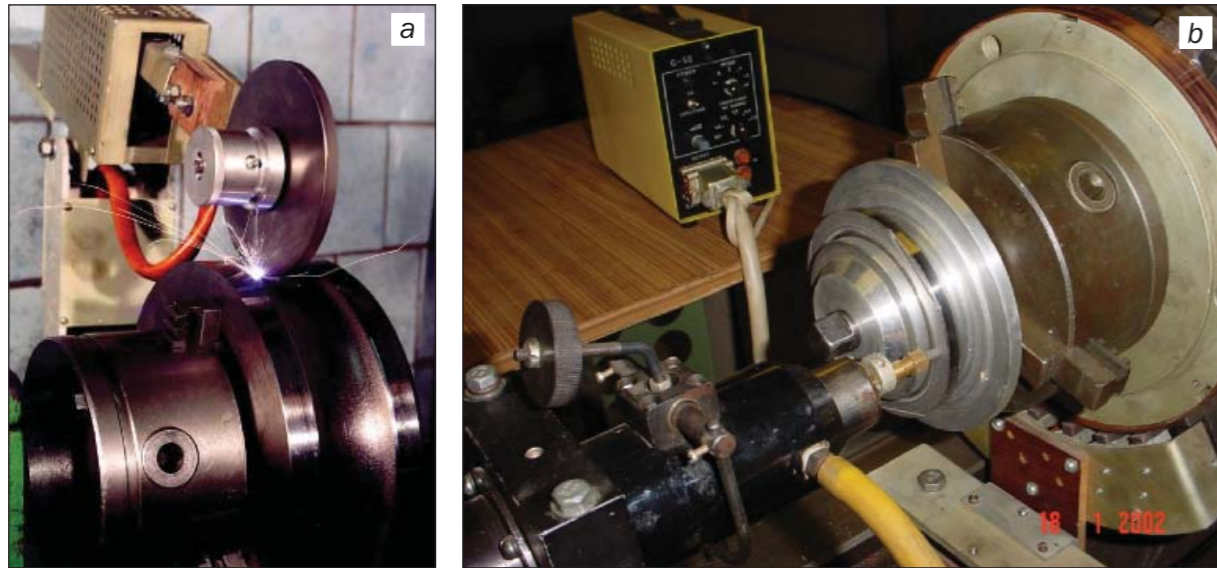
ditional heat of the chemical reaction makes it possible to noticeably enhance the efficiency of deposition process.

Thus far, V groups of electrode materials have been designed.

Group 1 – electrodes based on a hard alloy, an intermetallide, or an infusible component. This group includes basic materials of SHIM grade (synthetic hard instrumental materials) consisting of a hard phase and the binder (20–60 %). The following alloys are the examples of materials belonging to this group: SHIM-20N (TiC–Ni), SHIM-3B (TiC–Cr₃C₂–Ni), SHIM-3V (TiC–Cr₃C₂–Fe), SHIM-2/40NZh (TiC–nickel alloy KhN70Yu), SHIM-40NA (TiC–NiAl), SHIM-40TAA



Rod and disc electrodes for PED and TRESS



Mechanized electrospark treatment (a) of a forming roll with a disc SHS-electrode and (b) of the critical part of a rocket engine

(TiC-Ti₃AlC₂), SHIM-9/20A (TiB₂-TiAl), SHIM-2/30NM, (TiC-Ni-Mo), SHIM-60NT (TiC-TiNi), SHIM-9/20NA (TiB₂-NiAl), and ZhS-3NT.

This group also comprises dispersion-hardened ceramics with the effect of simultaneous dispersion strengthening of carbide grains and the metal binding due to nanosized precipitations result in concentration separation of supersaturated solid solutions.

Depending on their composition, electrode materials are referred to as CNT – double titanium–niobium carbide (Ti,Nb)C, CZT – double titanium–zirconium carbide (Ti,Zr)C; and CTT – double titanium–tantalum carbide (Ti,Ta)C. Metal binder in amount 10-60 % of various compositions (Ni-Co-Al-Cr, Ni-Al, Ni) are added to the electrodes to improve the quality of the coatings being formed. Materials of Group 1 also include heat-resistant electrodes of compositions Cr-Al-B-Si, Mo-Si-B and Cr-Al-B. These materials are intended for coating operating at temperature around 1500 °C.

Furthermore, Group 1 includes the materials synthesized on the basis of MAX-phases, infusible oxygen-free compounds, such as Ti_xCr_yAlC, possessing layered structure and the unique combination of properties of both a metal and ceramics. Materials based on MAX-phases are characterized by relatively low density, high thermal and electrical conduction, high strength, reduced modulus of elasticity,

as well as high corrosion and heat resistance (up to 2000 °C).

Group 2 – electrode materials based on a hard alloy or an intermetallide containing functional nanosized additives: ZrO₂, Al₂O₃, NbC, Si₃N₄, W, WC, WC-Co, Mo-Al₂O₃, ultradispersed diamond. The introduction of an infusible nanodispersed component into the SHS mixture results in significant modification of the alloy structure and formation of nanosized grains of carbides, borides, silicides or intermetallides.

The positive effect of the nanostructure on the coating formation process can be attributed to the increase in erosion capacity of the electrode. The pulsed discharge energy results in an increase in temperature of the electrode (anode) working tip up to the melting of the binder. Provided that the pulsed discharge energy remains constant, the intensity of electrode erosion increases with decreasing grain size. The erosion rate increases due to a stable flow of fine fragments, which are comparable with the grain size of the electrode material. The quality of the coating formed (density, thickness, uniformity) is improved, as well.

Group 3. Nanostructured cemented carbide in the WC-Co system fabricated using the pressing-sintering and hot pressing techniques using nanosized powders. The average size of carbide phase in the electrode material is ~ 80 nm. The electrodes from Group 3 are referred to as SNM

(sintered nanostructured material) or HPNM (hot-pressed nanostructured material).

Group 4. Glass-forming materials containing transition metal carbides (Fe₃C, WC) are specifically prepared near-eutectic precursors of the amorphous phase (e.g., fast-quenched Fe-Mn-Ni-Si-C cast iron doped with boron in order to enhance its glass-forming ability). Fast quenching of the melt results in the suppression of formation of infusible crystallites, reduction of the size of structural components and the melting temperature of the electrode. These electrodes have a high potential for applying nanostructured coatings onto titanium alloys.

Group 5 – electrodes for the TRESS process. The electrodes from this group are com-

pact materials consisting of the pressed or sintered reactive mixture. TRESS electrodes are also produced by drawing of powder mixture in aluminum, steel, or copper claddings. Several electrode compositions have been developed, such as Ti-B, Ti-C-Ni-Al, Ti-C-Al, Ti-Al, Ni-Al, Ti-Al-diamond, Ti-B-diamond, etc.

Diamond-containing compositions of the TRESS electrodes are used to create sublayers on various support materials (titanium alloys, steels, hard alloys) for the further deposition of CVD-diamond films.

The electrode materials and setups designed have found application in strengthening and repairing cutting, forming, pressing, and rolling tools, critical elements of machine parts, as well as parts of aerospace vehicles.

Coatings obtained using the PED and TRESS techniques

The regularities of formation of the PED and TRESS coatings on various support materials (titanium and nickel alloys, carbide steels, high-speed steels, die steels, and white cast iron) upon variation of pulsed discharge parameters (current intensity, frequency, and duration). The optimal energy regimes and treatment duration characterized by high rate of coating formation at satisfactory roughness of the surface layer have been determined.

Thus, the coatings on titanium alloy OT4-1 applied using the SHIM-40 TA electrodes (the TiC-Ti₃AlC₂ system) consist of titanium carbonitride grains Ti(C,N) with size varying from 70 to 500 nm.

The coatings produced using the electrodes

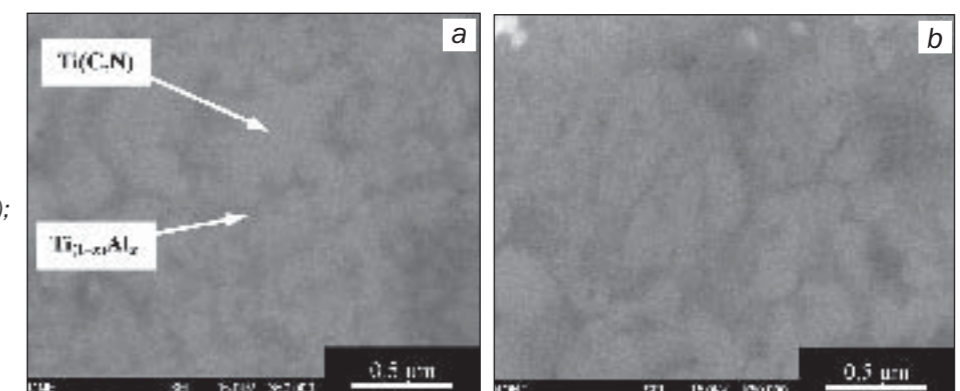
based on titanium carbide and diboride are characterized by high density (up to 100 %), microhardness (up to 20,7 GPa), as well as heat and wear resistance.

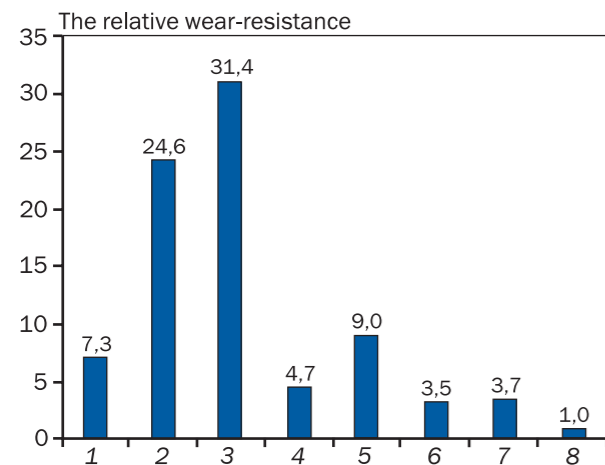
The application of the PED coatings on tool steel Kh12MF results in the enhancement of wear-resistance by a factor of 30 (after the running-in of the coating). In practice, strengthening die tooling is a rather efficient procedure.

The application of nanostructured electrode materials and those dispersion-strengthened with nanoparticles results in a decrease in coating roughness and the friction coefficient K_{fr} . Scratch testing at stress up to 130 N demonstrated no signs of adhesive fractures on the

Microstructure of PED coating on titanium alloy OT4-1

a – SHIM-40TA (TiC-Ti₃AlC₂);
b – SHIM-40TAOKn (TiC-Ti₃AlC₂ + ZrO₂^{nano})





The relative wear-resistance of the coatings on steel Kh12MF

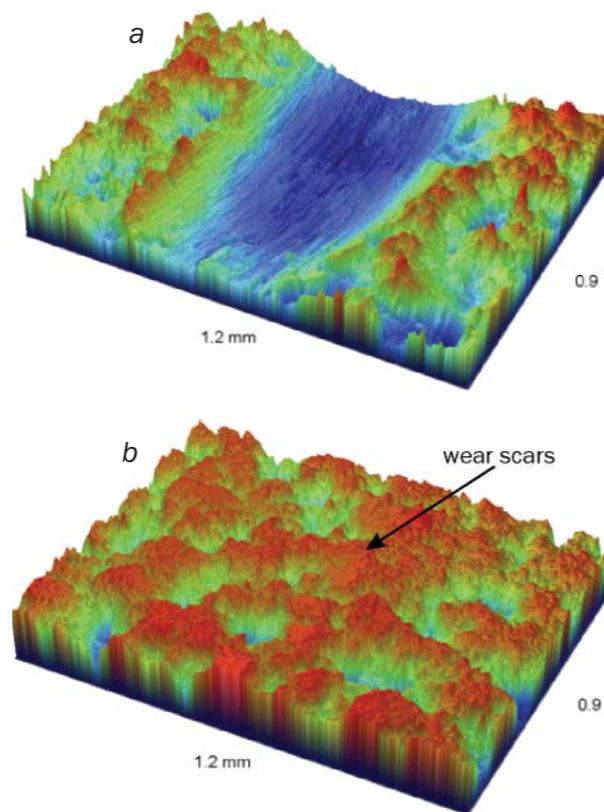
1 – SHIM-9/20A + WSe₂; 2 – SNM 8 (WC-Co) + WSe₂; 3 – SNM 8; 4 – SHIM-40NA (TiC-NiAl); 5 – SHIM-40NAKNn (TiC-NiAl-NbC^{nano}); 6 – SHIM-20NMon (TiC-Ni-Mo^{nano}); 7 – secondary PED: first by SHIM-40TA (TiC-TiAl) and second by graphite; 8 – steel Kh12MF

coatings on titanium alloy. The elastic recovery value for these coatings is 40–50 %.

The use of secondary electrospark treatment with carbon-containing materials was proposed to further improve the operating properties of the products. The use of carbon-carbon electrodes facilitates the reduction and stabilization of K_{fr} and the improvement of wear-resistance.

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Surface topography of the coating on titanium alloy VT6 after the tribological tests

a – a wear track on the coating made of alloy SHIM-40NAKNn (TiC-NiAl+NbC^{nano}); b – a wear track on the coating after the secondary treatment with graphite

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Targets for ion-plasma sputtering

A wide range of SHS-composite targets in the TiC–TiB₂, TiB₂–Al₂O₃, TiC_α, TiB₂–Ti₅Si₃, TiB–Ti, TiN–TiB₂, TiN–Ti₅Si₃, TiC–TiB₂–TiC_xN_y–Ti₅Si₃, TiC–Ti₃SiC₂–TiSi₂(SiC), TiB₂–TiAl, TiC–Cr₃C₂, TiC–TiAl, Ti₅Si₃–Ti, TiB₂–CrB₂, CrB₂, (Ti,Mo)C–Mo₂C, TiC–TaC–Mo₂C, TiC_α–CaO(ZrO₂), TiC_α–Ca₃(PO₄)₂, Cr_xTi_{2-x}AlC, (Ti,Ta)C_α, (Ti,Nb)C_α, (Ti,Zr)C_α, (Ti,Ta)C_α–Ca₁₀(PO₄)₆(OH)₂, Ti(C,N)+Ti₅Si₃+TiAl₃, CrB₂ + Cr₅Si₃ + Cr₄Al₉, MoB_α + Mo_xSi_y, etc. systems have been developed for the technologies of ion-plasma sputtering of nanostructured multifunctional coatings (magnetron sputtering and pulsed cathodic arc evaporation). Disc and planar targets with wide geometrical configurations are produced using the force SHS-pressing technology.

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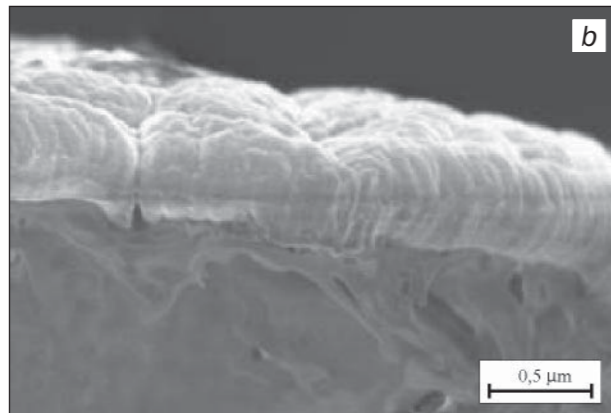
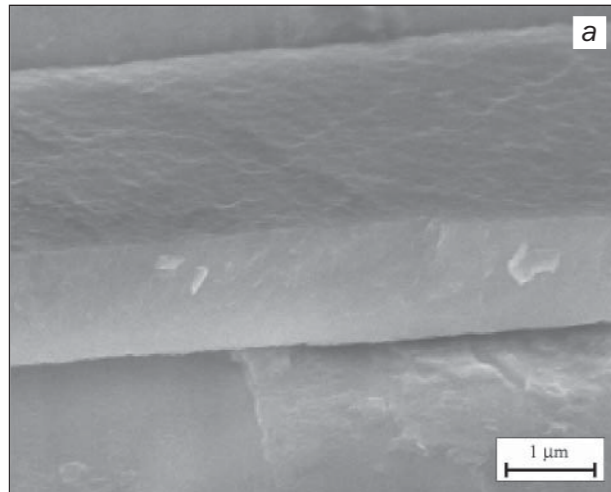
Disc and planar targets for magnetron sputtering

Multicomponent bioactive nanostructured films

Researchers of the SHS Center have developed a new approach to the design of thin-film biomaterials for load-bearing metallic and polymer implants [1–5]. Multicomponent bioactive nanostructured films (MuBiNaFs) were deposited by magnetron sputtering of composite targets based on non-stoichiometric titanium carbide TiC_{0.5} with various inorganic additives (CaO, TiO₂, ZrO₂, Si₃N₄, Ca₃(PO₄)₂, and Ca₁₀(PO₄)₆(OH)₂).

The fundamental research was started in 2004 and as early as in 2010 the films have been approved by the Russian Ministry of Health for medical application in the Russian Federation.

The comparison of the properties of MuBiNaFs with those of bulk materials (Ti-, Ni-, and Co-based alloys, stainless steel, ceramics) and thin films produced by alternative methods demonstrated a noticeable advantage from the view-



TiCaPCON film on the surface of Ti (a) and polytetrafluoroethylene (b)

point of the entire combination of physical, mechanical, tribological, and biological properties:

- Nanocomposite structure with various functional groups on its surface;
- High hardness – 25–40 GPa;
- Reduced Young's modulus – 230–350 GPa {TiN, 440GPa; TiC, 480GPa; SiC, 450 GPa; Al₂O₃, 390 GPa; stainless steel, 200 GPa; Ti, 120GPa};
- High resistance to plastic deformation (H^3/E^2) up to 0,9 GPa;
- High resistance to long elastic strain to failure (H/E) as an indicator of coating durability and wear resistance;
- High fatigue strength 350 MPa;
- High adhesion strength up to 50 N;
- High percentage of elastic recovery up to 75 %;
- Low coefficient of friction 0,12–0,22;
- Low wear rate – $10^{-6} \div 10^{-7} \text{ mm}^3 \text{N}^{-1} \text{m}^{-1}$;
- Low roughness $R_{rms} = 0,13 \div 1,5 \text{ nm}$;

- Negative surface charge at pH = 7;
- Positive values of the corrosion potential with low current density in biological solutions;
- Hydrophilic nature of MuBiNaFs surfaces;
- Biocompatibility;
- Bioactivity.

The more recent research has been focused upon the surface modification of Ti alloys using a combination of various techniques (such as cold spray, selective laser sintering, pulsed electroerosion treatment, and magnetron sputtering) to control surface topography, porosity, surface chemistry, and wettability; i.e., the characteristics affecting osteointegration.

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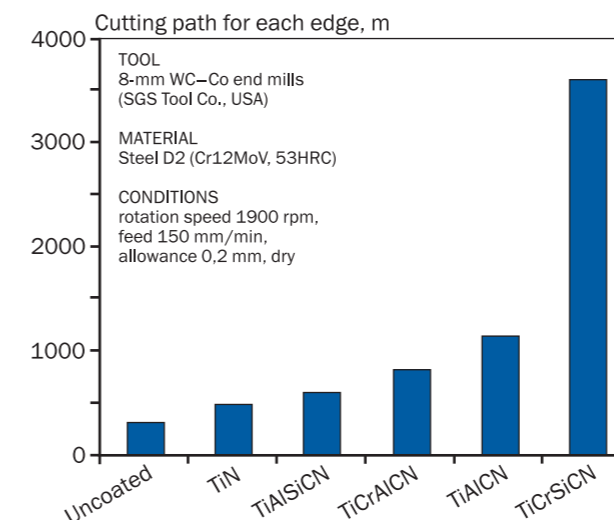
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Multifunctional (hard, anticorrosion, heat-resistant, self-lubricating, resistive) films and coatings fabricated using PVD techniques

A great variety of nanostructured coatings exhibiting superb chemical and mechanical properties have recently been obtained in our department. Among other coatings, the ones in the TiZrCON [1], Ti-(Al, Cr)-SiCN [2], CrAlSiBN [3],



Comparison of lifetime of coated and uncoated tools in dry milling operations



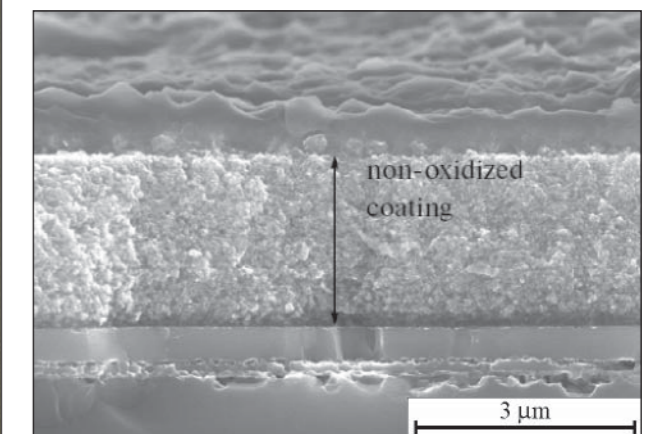
Coated end mills

(Ti, Al, Cr)-CN [4], and TiCrBN/WSe [5] systems are worth mentioning.

The TiZrCON coating exhibits the highest hardness of 40 GPa and good oxidation resistance up to 600 °C; therefore, it is suitable for tribological applications in the temperature range of 25–500 °C.

The TiAlSiCN coatings possess high hardness above 37 GPa in a very broad range of temperatures from 25 to 1300 °C. The TiCrSiCN coatings demonstrate the hardness above 34 GPa up to 1100 °C. The coatings in the Ti-(Cr, Al)-SiCN system show high oxidation resistance up to 1000 °C. The end mills with Cr-doped TiSiCN coatings shows superior dry cutting performance against high-chromium steel.

The Cr-Al-Si-B coating exhibits the optimal combination of properties: hardness of 30 GPa, the modulus of elasticity of 410 GPa, and the oxidation resistance up to 1200 °C. Cr-Al-Si-B-(N) coatings can be used as thin protective layers on items for tribological high temperature application.



Cross-section micrographs of the CrAlSiBN coatings oxidized for 1h at 1100 °C

The TiAlCN and TiCrAlCN coatings exhibit high hardness of 31–35 GPa, the low and stable friction coefficient against WC–Co in the range from 0,15 to 0,25. Within this system with the optimal chemical composition, the coatings exhibit high thermal stability up to 1200 °C.

The TiCrBN/WSe_x coatings are characterized by hardness of 30 GPa. The incorporation of WSe_x into a TiCrBN coating decreases the friction coefficient in air from 0,5 to 0,2–0,25 over a wide temperature range of 25–550 °C.

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Assurance of the uniformity of measurements of mechanical and tribological properties of nanostructured surfaces

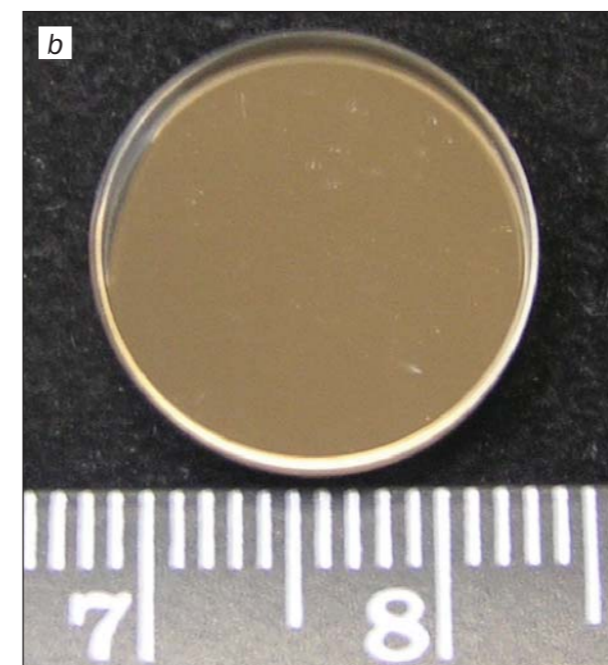
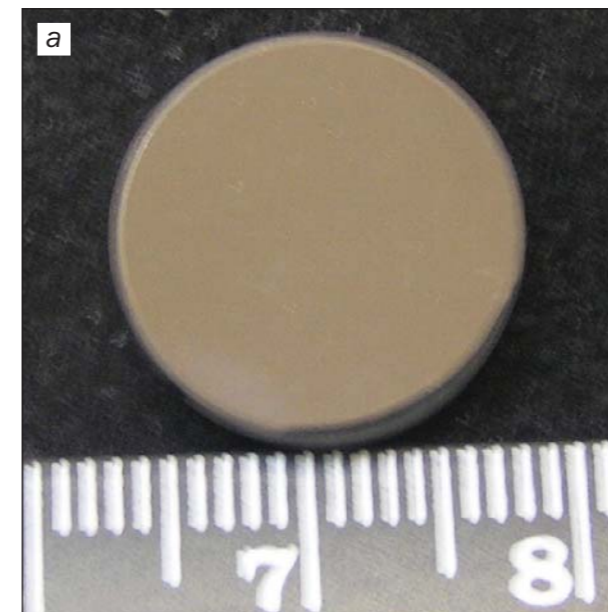
Assurance of measurement uniformity of functional properties of nanomaterials such as hardness, modulus of elasticity, adhesion strength, friction coefficient, wear rate and roughness is a key question of positive development and commercialization in nanoindustry. To achieve this purpose the metrological complex based on modern equipment such as nano-hardness tester, scratch tester, tribometer, atomic force microscope, optical and mechanical profilers, impact tester, scanning nanoindenter have been created.

The metrological complex is included into the Testing Laboratory of Functional Surfaces (accredited by association of analytical centers «ANALYTICA» and also accredited by ROSNANO). So the measurements of hardness, modulus of elasticity, elastic recovery, adhesion strength, friction coefficients and wear rate etc., as well as surface roughness and topology of nanoindustry products has been provided.

The procedures for measuring mechanical and tribological characteristics of nanomaterials and calibrating the corresponding measuring tools have been developed and certified. The state reference materials ensuring adequate calibration and traceability of measuring mechanical and tribological properties of nanostructured surfaces have been certified. Two projects of the National Standards (GOST R) for the methods for measuring the elastic modulus and the adhesion/cohesion strength of nanostructured coatings have been developed.

The practical results of instrumented indentation, scratch and tribological tests of nanostructured surfaces of the most typical commercialized nanomaterials and nanostructured coatings have been summarized.

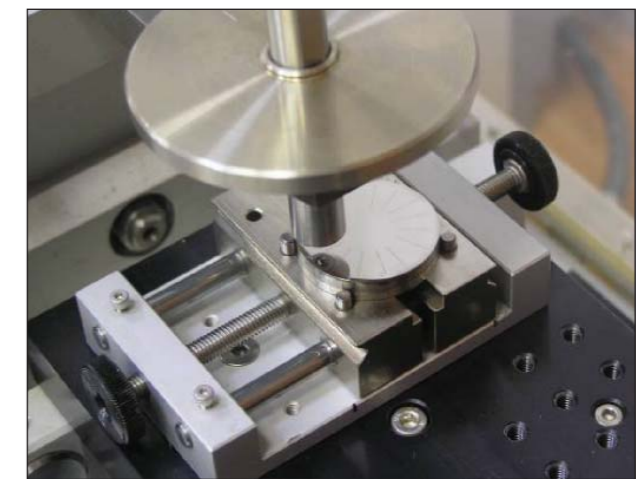
The complex of technological normative documents, including the practical recommendations for measuring hardness, the modulus of



State reference material of Young modulus of the nanostructured TiCCaPON coating with the established value $E = 202$ GPa and the extended uncertainty of 9,8 %: the side with coating deposited on fused silica (a) and the side without coating (b)

elasticity, elastic recovery, plastic resistance, elastic failure strain resistance, crack resistance, has been elaborated.

A website contained the recommendations on good practice, normative documents and procedures to ensure the uniformity for measuring of mechanical and tribological properties of nanostructured surfaces has been launched (<http://>



State reference material of sliding friction coefficient of nanocrystalline material (KT-NKM-0.25) with the established value $f = 0,194$ and the extended uncertainty of 8 % at the stage of tribological testing for metrological certification

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LIST OF THE RECENT INTERNATIONAL PATENTS



Аналитика

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Valid to
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Accreditation Body AAC "Analitica" certifies that
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is accredited in accordance with the recognized International Standard ISO/IEC 17025:2005 (GOST R ISO/IEC 17025 - 2006). This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer Joint ISO-ILAC-IAF Communiqué dated January 2009).

The scope of laboratory (center) accreditation is described in the Appendix.

Head of
Accreditation body



I. Boldyrev

15 July 2010

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LIST OF THE AWARDS RECEIVED AT PRESTIGIOUS FORUMS AND INTELLECTUAL PROPERTY EXHIBITIONS

- Honoured diploma and gold medal for the invention «Biocompatible Multicomponent Nanostructured Coatings for Medicine», presented at International Salon of Inventions on April 5–9, 2006 in Geneva, Switzerland. Authors: E. Levashov, D. Shtansky, N. Gloushankova, I. Reshetov
- Honoured diploma and silver medal for the invention «FGM Target for Deposition of Multicomponent Nanostructured Coatings for Medicine», presented at International Salon of Inventions on April 5–9, 2006 in Geneva, Switzerland. Authors: E. Levashov, V. Kurbatkina, D. Shtansky, B. Senatulin
- Honoured diploma and silver medal for the invention «Biocompatible Multicomponent Nanostructured Coatings for Medicine», presented at the International Innovation Exhibition: Ideas, Inventions and Innovations, 2006, Nurnberg, Germany. Authors: E. Levashov, D. Shtansky, N. Gloushankova, I. Reshetov
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- Honoured diploma and gold medal for the Invention «Biocompatible Multicomponent Nanostructured Coatings for Medicine», presented at the IX International Salon of Industrial Property «Archimed-2006». Authors: E. Levashov, D. Shtansky, N. Gloushankova, I. Reshetov
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- Honoured diploma and gold medal with Nicola Tesla image for the Invention «Biocompatible Multicomponent Nanostructured Coatings for Medicine», presented at Belgrade Salon of Innovation and Business on November 11–December 01, 2007, Serbia
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