Ministry of Education and Science of the Russian Federation

Federal State Budgetary Institution

TECHNOLOGICAL INSTITUTE FOR SUPERHARD AND NOVEL CARBON MATERIALS

TISNCM

Federal State Budgetary Institution "Technological Institute for Superhard and Novel Carbon Materials"

The Institute was established in 1995 as the Scientific Technological Centre "Superhard Materials" (STC SHM). In 1998 the Institute was reorganized as the Federal State Budgetary Institution "Technological Institute for Superhard and Novel Carbon Materials" (FSBI TISNCM).

FSBI TISNCM is administrated by the the Ministry of Education and Science of the Russian Federation.

*Professor **Vladimir Blank** is the Director of FSBI TISNCM*

Scientific Research Departments at FSBI TISNCM

1. Department of Structural Research
   - Laboratory of Spectral Research
   - Laboratory of Electron Microscopy
   - Laboratory of X-ray structure analysis
   - Theoretical Study Group
2. Department of Single Crystal Growth
   - Laboratory of CVD Diamond Growth
   - Laboratory of Physical Properties of Nanostructures
   - Group of Development and Research of Diamond-based Acoustoelectronic Devices
3. Department of Functional and Constructional Nanomaterials
   - Laboratory of Functional Nanomaterials
   - Laboratory of Synthesis of Novel Superhard Materials
   - Laboratory of Physical and Mechanical Measurements
4. Department of Physical and Mechanical Properties Research
5. Department of Chemical Technologies and Nanomaterials
6. Department of Educational Programmes
7. Physics and Chemistry of Nanostructures Chair in the Moscow Institute of Physics and Technology
8. Auxiliaries

The subdivisions within the Institute are grouped in the following divisions

- FSBI TISNCM Shared-use Equipment Center "Research of Nanostructured, Carbon and Superhard Materials".
- Research and Education Center (REC) "Physics and Chemistry of Nanostructures".
- Joint Laboratory TISNCM–SIEMENS "Functional and Constructional Nanomaterials".

The main task of TISNCM is the development of new materials. With this purpose, the Institute is actively engaged in a range of related projects, including:

- pilot studies;
- R&D;
- certification of materials and products, securing intellectual property rights;
- promoting products to the market.

The Institute possesses the technology for the production of diamond single crystals up to 7 carat in weight, which have no natural analogues (superpure, doped, and semiconductor). This technology is currently being developed commercially.

TISNCM also develops the following types of constructional materials: metals, metal–carbon, carbon–carbon, and nanostructured ceramics. Materials with unique mechanical properties are also produced, including aluminum-, titanium-, and zirconium-based alloys; TiC–ZrC-based hard alloys; ultrahard fullerites; β-Si_3N_4 and UO_2 ceramics. The Institute is also engaged in the production of new nanostructured thermoelectric Bi_2Te_3-based materials.

TISNCM places great value on collaborations with Russian and foreign institutes, including the Scientifi c Center Kurchatov Institute, RAS Institute of Crystallography, GIREDMET, University of Umea (Sweden), University of Manchester (Great Britain), Laboratory of Crystallography of the French Academy of Sciences, and a range of other R&D centers in Russia, Australia, Germany, USA, and Japan.

The personnel are the main resource of any scientifi c institution. The year 2008 saw the establishment of the"Physics and Chemistry of Nanostructures" department at the MIPT Faculty of Molecular and Biological Physics. TISNCM was appointed as the base organization for this department, providing a training in Applied Mathematics and Physics as part of a master's degree in Chemical Physics.

Developments of the Institute were awarded a number of prestigious prizes exhibitions:

- Silver medal of the 27th International Exhibition of Innovation (Geneva, 1999).
- Gold medal of the 5th International special exhibition LaboratoryExpo 2007 (Moscow, 2007).
- Gold medal at the 7th International Salon of Innovations and Investments (Moscow, 2007).
- Diploma and medal of the II International Exhibition and Congress "Advanced Technologies of XXI Century" (Moscow, 2008).
- Diploma of the International Exhibition for Measuring Equipment and Technology (Moscow, 2008).
- Diploma V International Exhibition of Nanoindustry NTMEX 08 (Moscow, 2008).
- Diploma XII St. Petersburg International Economic Forum (St. Petersburg, 2008).
- Diploma of the International Exhibition of 2008 iENa "IDEEN-ERFINDUNGEN-NEUHEITEN" (Nurnberg, 2008).
- Honour Laureate of the 11th International Forum "High Technologies of XXI Century" (Moscow, 2010).
- Gold medal and diploma of the 3rd International Exhibition of Inventions in the Middle East (IIFME- 2010, Kuwait).
- Diploma and Medal of the 9th Specialized Exhibition "Lab Expo" (Moscow, 2011).
- Certificate of the IV Specialized Exhibition of instruments of test and control "Expo Control 2012".

DEPARTMENT OF STRUCTURAL RESEARCH

Department Head Prof. **V. Blank**

Laboratory of Spectral Research

Laboratory Head Dr. **V. Denisov**

Laboratory of Electron Microscopy

Laboratory Head Dr. **B. Kulnitskiy**

Laboratory of X-ray structure analysis

Laboratory Head Dr. **S. Polyakov**

Theoretical Study Group

Group Head Dr. **P. Sorokin**

Fundamental studies

- Fundamental studies of materials in extreme conditions – under high pressure and shear deformation: phase transitions and nano-sized structure formation.
- Research of optical properties of diamonds, including diamonds doped with boron and nitrogen.

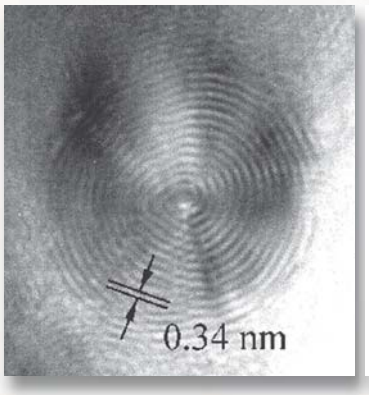
Applied studies

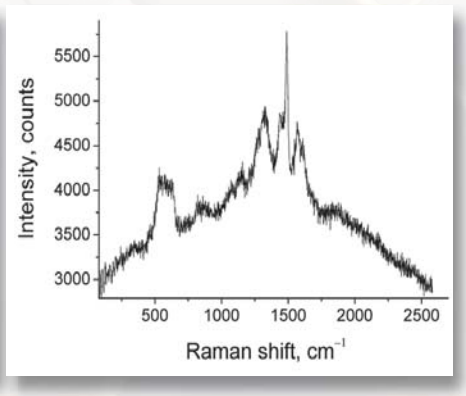
- Investigations of the optical and structural properties of diamonds and carbon nanostructures.
- Certification of synthetic and natural diamonds.
- Nanolithography on a diamond surface with pattern height of few tens of nanometers.

Experimental techniques

- Vibrational spectroscopy:
  - Raman scattering;
  - IR-Fourier spectroscopy in transmission and reflection.
- Electronic spectroscopy:
  - Photoluminescence;
  - Absorption in the UV - visible - near infrared;
  - Electronic Raman scattering.
- Optical studies at low and high temperatures (5 – 700 K).
- X-ray diffractometry and topography.
- Transmission electron microscopy on the JEM-2010 microscope with element analysis employing energy dispersive X-ray spectroscopy (EDS) unit.

Shear diamond anvil cells with electric drive developed and produced by TISNCM

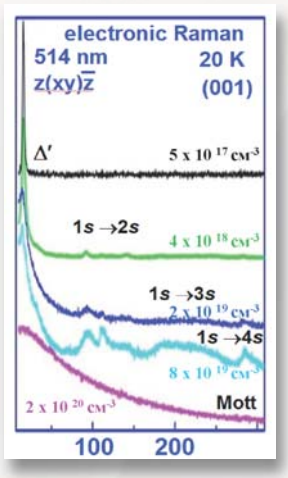




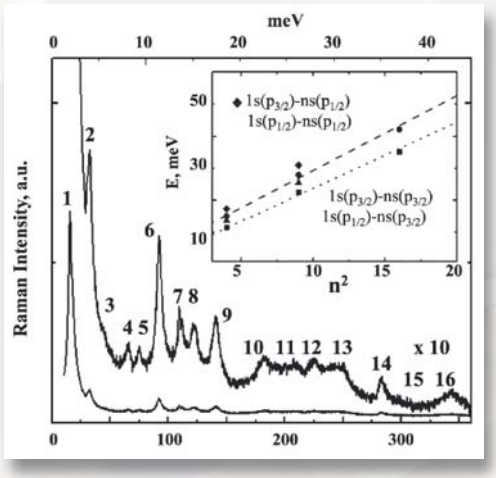
The carbon onions were synthesized from graphite in a diamond anvil cell under a high pressure and shear deformation treatment. Raman scattering experiment revealed a fullerene-type structure of the created carbon onions.

Evolution of electronic Raman spectra of boron clusters in diamond crystals doped with boron, depending on the concentration of boron.

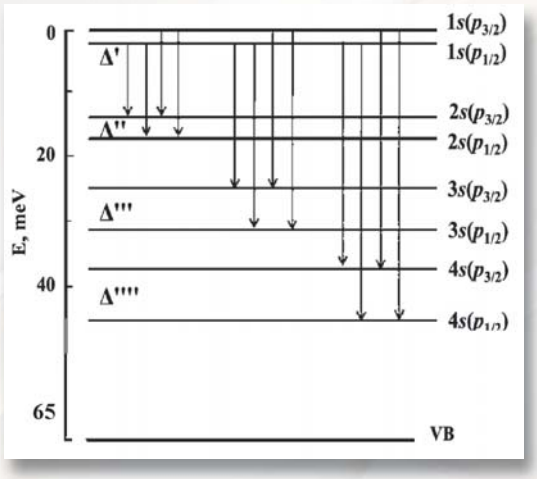
Electron transitions between the acceptor levels of boron clusters in boron-doped diamond were discovered for the first time.



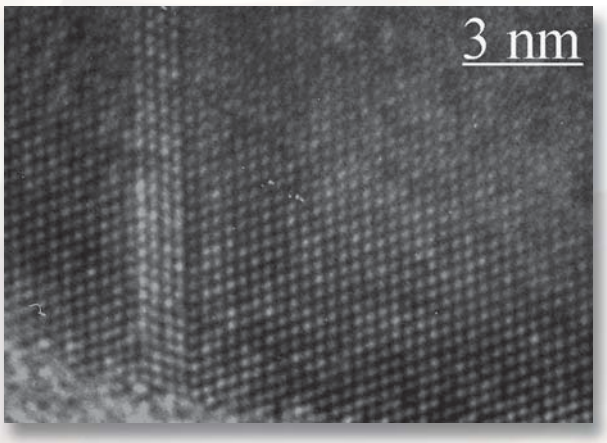
Raman frequency shift, cm-1



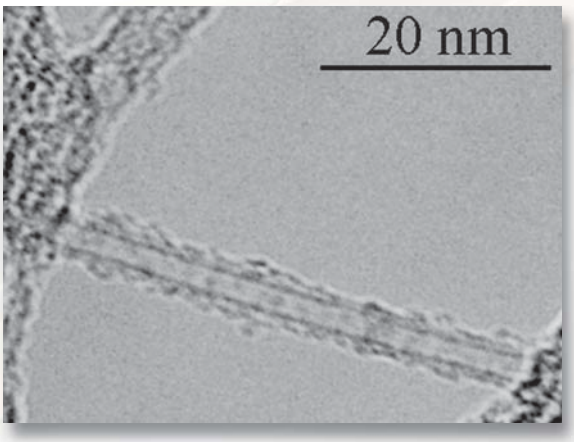
Raman frequency shift, cm-1



The scheme of acceptor levels of boron clusters in boron-doped diamond



Twin in a silicon nanoparticle.



Fluorinated single-wall nanotube.

Laboratory of Electron Microscopy

Laboratory Head Dr. **B. Kulnitsky**

Laboratory equipment:

- **JEM 7600F Scanning Electron Microscope** with attachments for EDS and WDS analysis of spectra characteristic of chemical elements;
- **JEM 2010 Transmission Electron Microscope** with an attachment for EDS analysis of characteristic spectra and GIF Quantum attachment for EELS and EFTEM;
- There is also an equipment for samples preparation for transmission electron microscope.

Scientific research areas

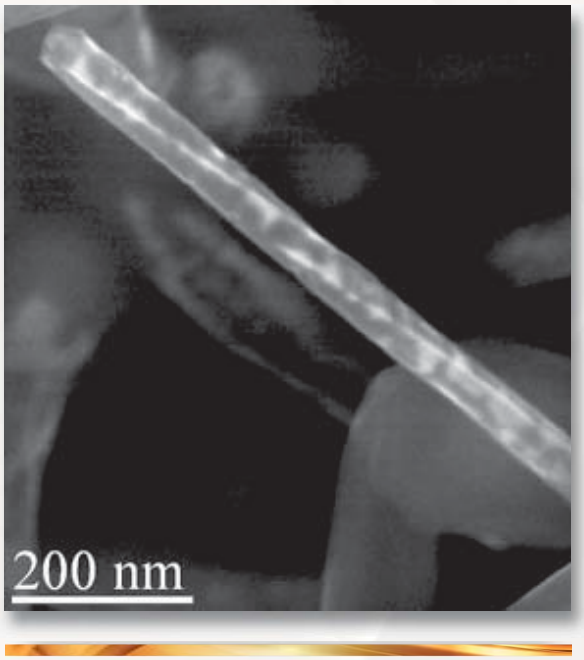
Studies of carbon nanostructures

FSBI TISNCM has an extensive experience in synthesis of various carbon nanostructures (nanotubes, nanofibers, carbon bulbous structures (onions) and others). Transmission electronic microscopy allows us to study the morphology of these structures, particular qualities of their texture, especially the catalytic growth of nanotubes and nanofibres, various defects. The results of such studies are particularly important for optimizing synthesis conditions of carbon nanostructures, and besides, they can establish a relationship between the structure and properties of the object and a single carbon macroscopic material properties of these objects. Electron energy loss spectroscopy allows in some cases to draw the conclusions about the nature of chemical bonds between carbon atoms.

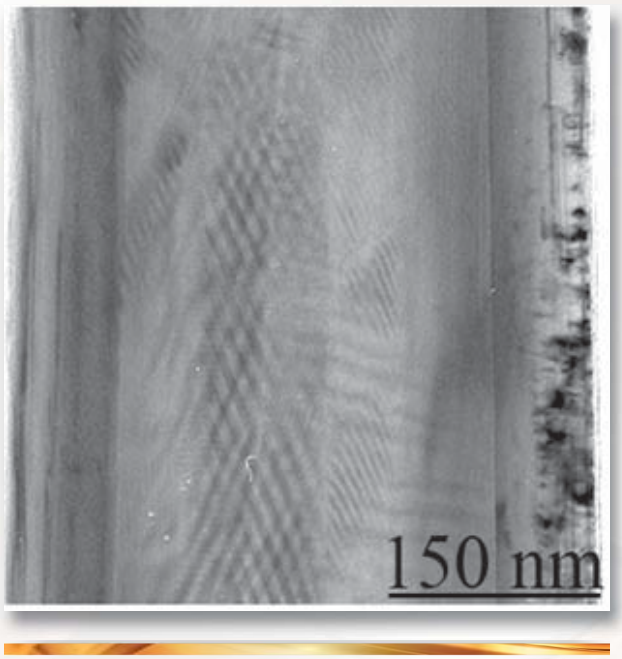
Study of nanoparticles of metals, alloys, ceramics and other nanocrystalline objects and non-carbon nanotubes

Nanocrystals of these materials are used in a variety of tasks, such as creating composite materials with advanced mechanical and electronic properties of the catalytic growth of carbon and other nanostructures.

Transmission electron microscopy and high-resolution electron diffraction allow us to find, submit and describe various lattice defects (stacking faults, twinning, grain structure boundaries, dislocations, etc.) and in some cases to indicate their influence on material properties, the influence on the process when using a catalytic growth.



Faceted and twisted nanotube composition of boron-carbon-nitrogen



Faceted nanotube composition of boron-carbon-nitrogen. Moire patterns.

Study of diamond, nano-diamond and other materials

Diamond is a unique material with outstanding mechanical, chemical and electronic properties. Doping diamond with different elements in some cases allows us to control some of these properties. The study of diamond particles of nano-diamond by method of high-resolution transmission electron microscopy and electron energy loss spectroscopy allows us to analyze the influence of defects and impurities in diamond on its various properties. In addition, the laboratory has also been exploring nitride boron and other solid materials.

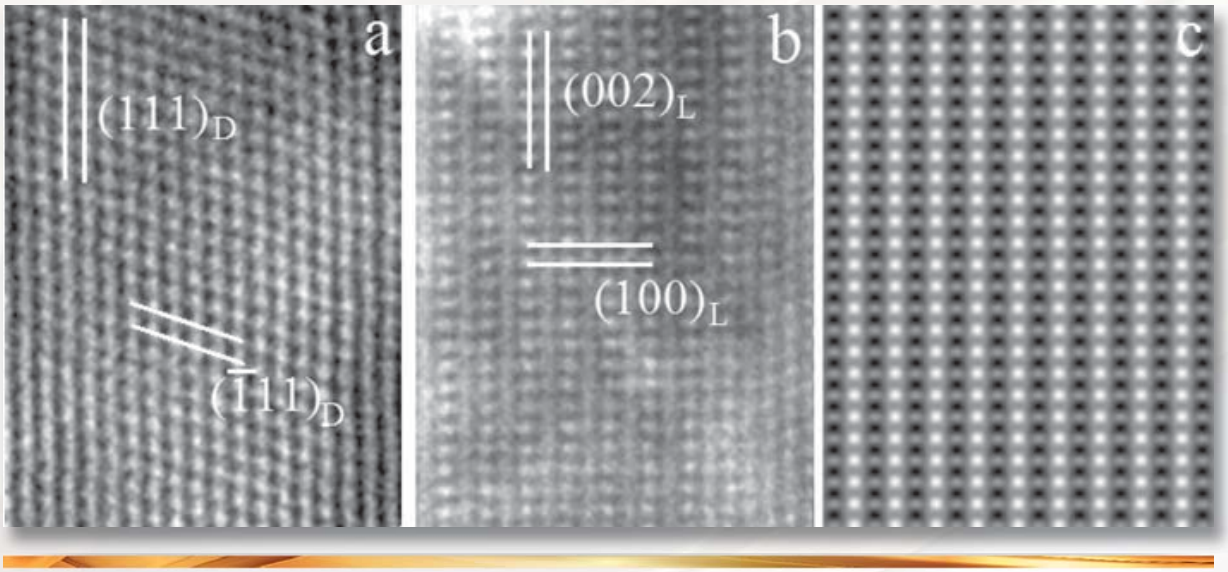


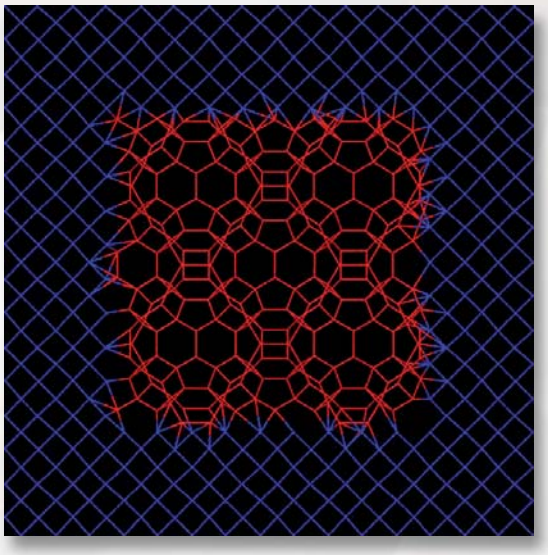
Image obtained with high resolution:
a) Diamond, the interplanar distances are 0.206 nm;
b) lonsdalite, interplanar distances are 0.206 nm and 0.218 nm;
c) High-resolution modeling of the same section of lonsdaleite.

Theoretical Study Group

Group Head Dr. **P. Sorokin**

FSBI TISNCM Theoretical group is the creative team of young scientists and students led by Dr. Pavel Sorokin.

Scientific group was formed to conduct basic research in novel materials. The main task of the group is a theoretical study of properties of various meso- and nano-materials. The main tool of theoretical research is a computer simulation using a wide range of existing theoretical methods (ab initio, semi-empirical methods, classical potentials).

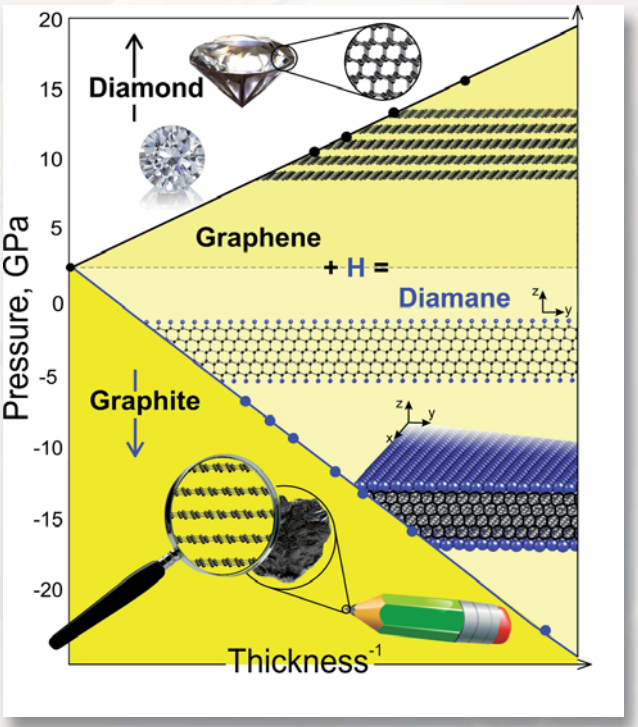


Model of ultrahard fullerite

Primary research fields:

- Study of superhard and novel materials
- Study of properties of diamond based nanostructures
- Study of properties of new nanomaterials with advanced properties
- Support for experimental studies

For modeling of materials properties the group uses a «T-Platforms» high performance computing cluster (2.94 TFLOPS) based on T-Blade 1.1 and specialized software.



Dependence of phase transition pressure on reciprocal thickness of ultrathin diamond films

«T-Platforms» computing cluster

DEPARTMENT OF SINGLE CRYSTAL GROWTH

Department Head Dr. **S. Terentiev**

Laboratory of CVD Diamond Growth

Laboratory Head Dr. **N. Kornilov**

Laboratory of Physical Properties of Nanostructures

Laboratory Head Dr. **S. Buga**

Group of Development and Research of Diamond-based Acoustoelectronic Devices

Group Head Professor, Dr. **B. Sorokin**

Primary research and technological fields

- Development of high-pressure apparatus and control systems for the synthesis, thermobaric treatment, and sintering of superhard materials.
- Improving of technologies for manufacture of large diamond single crystals with special properties.
- Improving of chemical vapor deposition (CVD) technique of polycrystalline and homoepitaxial single-crystal diamond films on boron-doped diamond substrates.
- Development of technology for the thermobaric treatment of superhard materials.
- Manufacturing of the products of a predetermined shape made of natural and synthetic diamonds.
- Study of the properties of multilayer diamond structures.

A principal scope of the department is growing of large diamond single crystals that have no natural analogues: "nitrogen-free", superpure and precisely doped by boron semiconducting.

Self-designed growth units are used for the research and pilot production.

A promising research direction is further modification of diamond single-crystal films growth technology using CVD technique.

Universal high-pressure growth units (model URS-01/V).

Parameters of growth processes:

Pressure 5 -7 GPa Temperature 1250 - 1600 ° C Time up to 300 hours

Diamond single crystals

superpure

doped by boron semiconducting

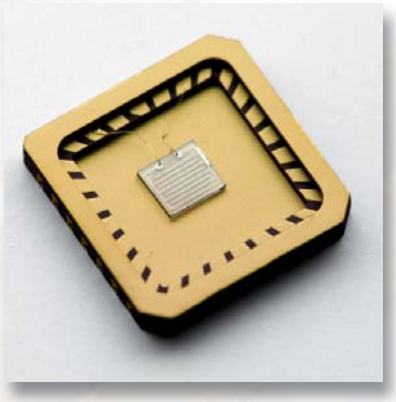
Products from superpure single-crystal diamond

Properties of superpure single-crystal diamond:

- Size up to 8 mm;
- Low nitrogen concentration: 0,5 – 2,0 ppm;
- High thermal conductivity up to 2200 W/(m·K);
- Wide range of optical transparency from 225 nm to 25 μm (with specific absorption in 1600–4000 cm^{-1});
- High perfection of crystalline structure;
- Low luminescence ~15–30 (ratio of the second-order Raman spectrum intensity to background luminescence) ;
- High specific resistance up to 10^{12} Ohm·cm;
- Hardness not less than 105 GPa.

Applications:

- optical windows for powerful lasers;
- gauges for ultraviolet and X-ray radiation, and high-energy particles;
- parts for optoelectronic devices;
- diamond anvils for research of material properties and phase transitions at superhigh pressures (up to 2.5 Mbar);
- heatsinks for electronic devices;
- probes for scanning probe microscopes;
- substrates for homoepitaxial CVD diamond growth;
- jewelry;
- water-jet cutting nozzles.



Sensing element of ultraviolet radiation

Diamond single crystal gear

Optical window for lasers

Nozzles in an assembly with a working part of a diamond single crystal

Anvils of high-purity diamond single crystals

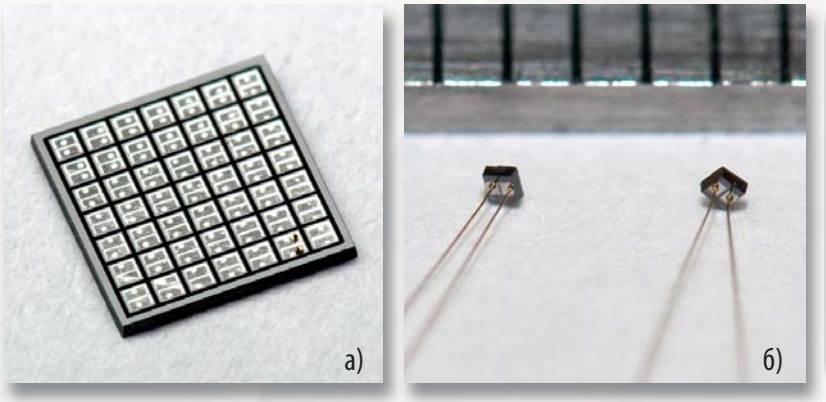
Products from semiconductor diamond single crystals

Properties of II b-type semiconductor diamond single crystals :

- Size up to 8 mm
- Acceptor ionization energy 0.19 - 0.37 eV;
- Wide range of specific resistance $0.1–10^9$ Ohm·cm;
- Color – light blue to black;
- Boron concentration up to 300 ppm.

Применение:

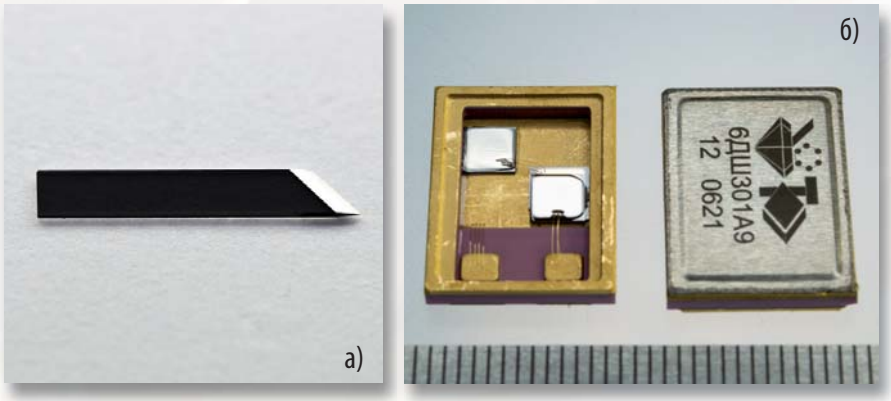
- high-sensitivity temperature gauges;
- Shottky diodes;
- high-frequency devices and elements (diodes, transistors);
- liquid- and gas-consumption gauges for the petrol and gas industry;
- low-inertia heat elements;
- probes for scanning probe microscopes;
- substrates for homoepitaxial CVD diamond growth;
- jewelry;
- microsurgical blades.



Sensible elements of temperature gauges:

a) Matrix of sensible elements of temperature gauges produced by a group technique from a plate of boron-doped diamond single crystal.

b) 0.5×0.5×0.2 mm diamond low-inertia sensible element of a temperature gauge.

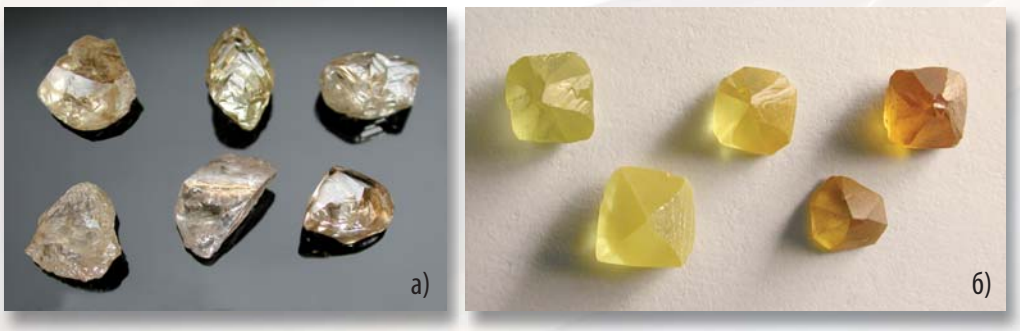


a) Microsurgical blades made of black diamond.

b) Diamond Schottky diode in case.

Термобарическая обработка природных кристаллов алмаза

Thermobaric treatment designed in the department effects brown initial color of several types of natural diamonds with weights up to 20 carat, yielding colorless, fantasy yellow, green, pink, blue, and orange colors, thereby improving their appeal to consumers.



Effect of thermobaric treatment on the color of natural diamonds:

a) Initial color.

b) Color after treatment.

Laboratory of CVD Diamond Growth

Laboratory Head Dr. **N. Kornilov**

One of the promising areas of the research is enhancing the method of synthesis of poly- and single-crystal diamond films by Chemical Vapor Deposition (CVD).

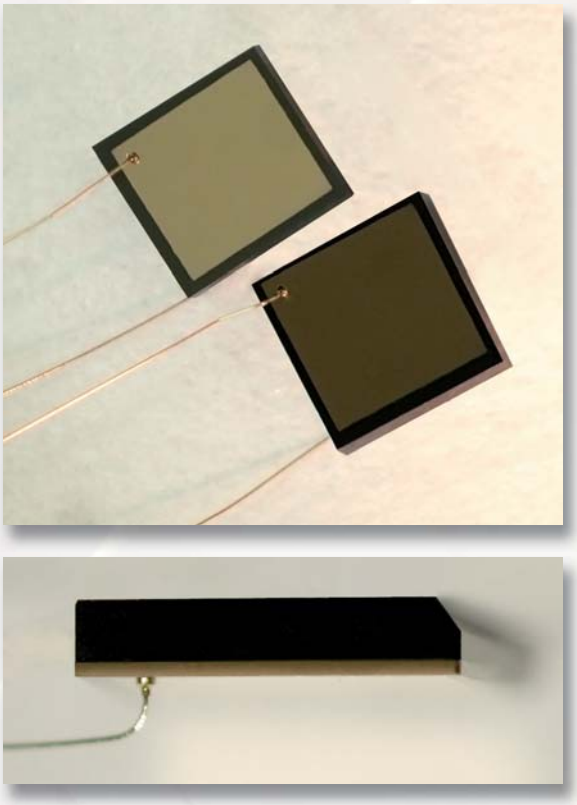
Research areas

- Improving of doping technologies (with nitrogen, boron, phosphorus) of homoepitaxial monocrystalline CVD diamond layers in order to develop an application basis of diamond as a material for electronics.
- Synthesis of defect-free single crystal homoepitaxial CVD diamond layers.
- Improving of technology of rapid growth of monocrystalline and polycrystalline CVD diamond plates.
- Development of process of CVD diamond synthesis for use as a structural material, in particular for devices for X-ray optics.
- Improving of technology of CVD synthesis of polycrystalline and homoepitaxial monocrystalline diamond films on substrate of boron-doped diamond.

CVD diamond plates and films properties:

- The thickness can vary from tens to hundreds of microns.
- The polycrystalline CVD diamond plate size is up to 50mm (diameter).
- The single-crystal CVD diamond plate size is determined by the size of the substrate (in this case, up to 8 mm).
- The nitrogen content is less than 1 ppb.

Plassys BJS 150 diamond deposition reactor



Sensing element of ionizing radiation detector made of CVD single crystals doped with diamond substrates

Laboratory of Physical Properties of Nanostructures

Laboratory Head Dr. **S. Buga**

Primary research areas

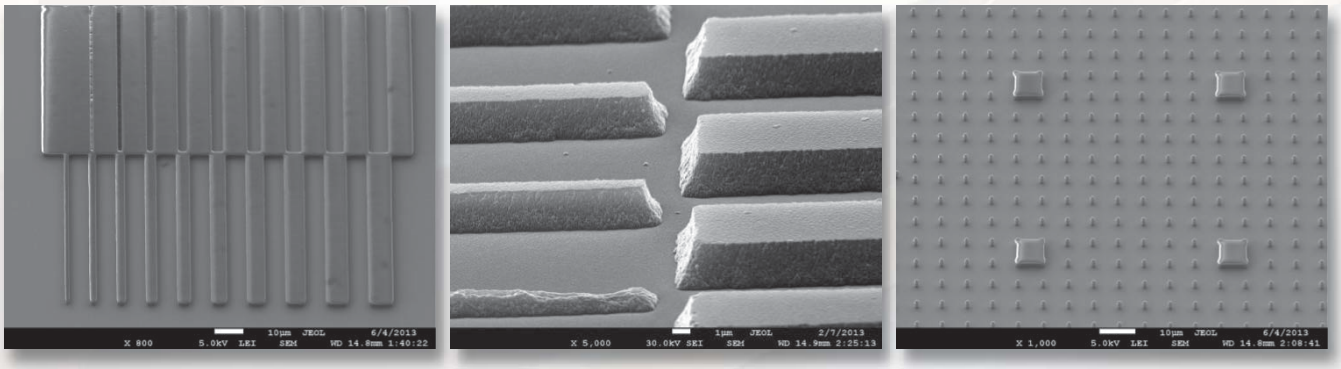
1. Developing of technological basis for production of multilayer structures based on synthetic single crystal diamond.
2. Development of design of the elements of electronic component base of a new generation high power diamond electronics.
3. Research of electrophysical and transport properties of semiconductor synthetic single crystals diamond, in particular under the effect of the optical and ionizing excitation.
4. Development of numerical models for calculating the characteristics of electronic devices based on synthetic diamonds.
5. Studies of electrical properties of composite nanostructured functional materials, such as thermoelectric alloys doped with fullerene C_{60} and others.
6. Study of nanoscale effects in the new electronic media, such as topological insulators, etc.

Research techniques

1. Measuring of electrical resistivity and Hall voltage in the range of 0.1 mohm - 200 Gohm at temperature range of 1.8 K - 800 K and magnetic fields up to 9 T, in particular under the effect of the optical excitation.
2. Measuring of the heat capacity and thermal conductivity of solids within the temperature range of 1.8 K - 400 K.
3. Measuring of static electrical characteristics of diode and triode structures. Measured voltage range is from 1 nV to 400 V, current range is from 1 fA to 10 A.
4. Study of trapping centers in semiconductors structures by methods of the current and capacitance spectroscopy of deep levels with temperature variation from 77 to 800 K.
5. Study of the relaxation and transport mechanisms of nonequilibrium charge by measuring the Hall effect under the optical excitation.

Technological capabilities

1. Formation of contact and dielectric structures by direct and inverse lithography with a minimum element size of 1 micron.
2. Magnetron sputtering of metal coatings with controlled thickness in the range from 1 to 1000 nm.
3. Deposition of oxide and nitride coatings (including high-k) of thickness under 2 microns by reactive RF sputtering .
4. Forming of contact pads by annealing metal and intermetallic coatings to 800 ° C in high vacuum (less than 10^{-6} Torr).
5. Reactive ion etching of semiconductor materials, including synthetic diamond using broad spectrum of gases (Ar, O_2, H_2, CF_4, SF_6).



Examples of structures produced by using lithography and reactive ion etching (RIE)

Results obtained

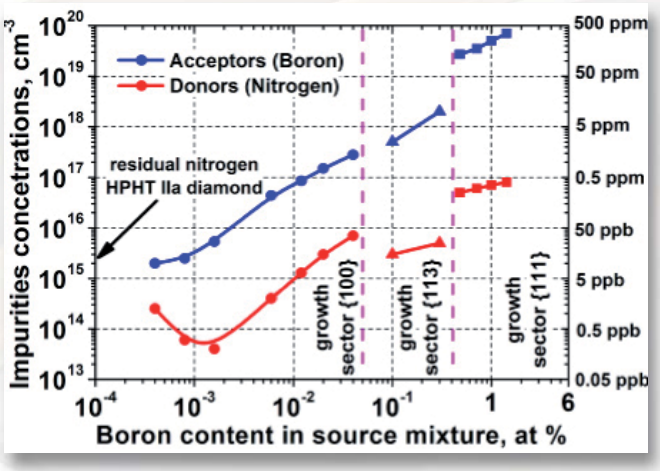
1. The technique of high-speed radiation resistant Schottky diodes based on synthetic single crystal diamonds was developed and mastered.

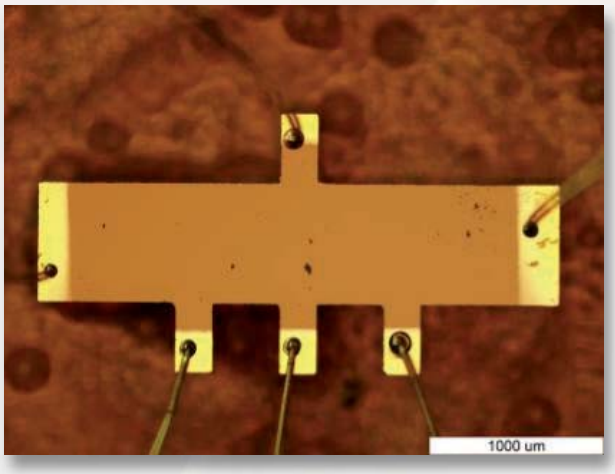
The main characteristics of the diodes:

- reverse voltage, Urev, **> 1000 V**
- average forward current, Ifw, **> 2 A**
- leakage current at Urev = 1000 V, **< 100 μA**
- forward voltage drop at Ifw = 2 A, **< 4 B**
- operating temperature **from -60 to 250 °C**
- recovery time, **less than 10 ns**

Schottky diodes based on synthetic diamond

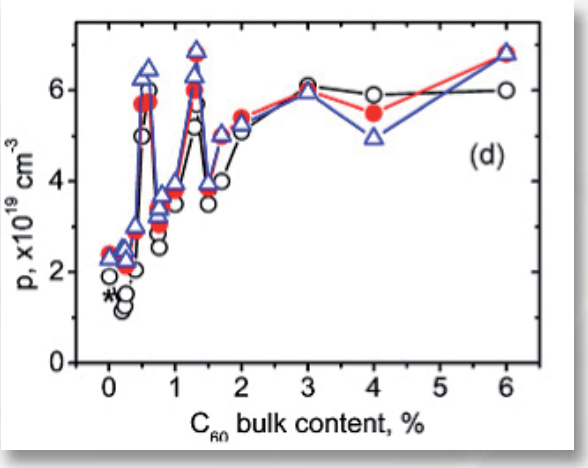
2. The electrical properties of synthetic HPHT single crystal diamonds doped with boron were studied. The relationship between electrical properties and the synthesis parameters were determined. The optimal parameters for synthesis of diamonds for electronic applications were determined.



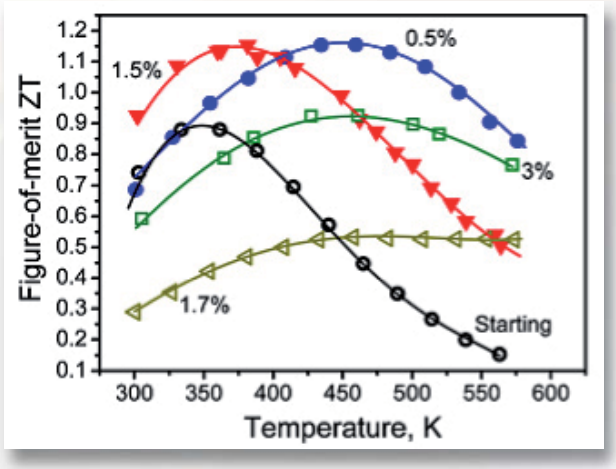


Relationship between the impurity composition of the semiconductor diamond and the boron content in the growth mixture

3. Method of mechanical polishing, cleaning and follow-up quality control of diamond plates for smooth and clean surface suitable for electronic applications was developed.
4. Field transistor model with Schottky gate based on synthetic single crystal diamond was designed.
5. The composite nanostructured thermoelectric materials based on a new type of alloys Bi-Sb-Te with fullerene C_{60} were developed and studied together with the Laboratory of Functional Nanomaterials. Increasing of the thermoelectrics quality factor ZT by 30-70% over a wide temperature range was obtained. The effect of «resonance» concentration of C_{60} fullerene, with the capture up to 6 electrons to the fullerene molecule, was discovered.



Dependence of concentration of free charge carriers (holes) on fullerene C_{60} concentration in the $Bi_{0.5}Sb_{1.5}Te_3$- C_{60} alloy



Dependence of the thermoelectrics quality factor ZT of $Bi_{0.5}Sb_{1.5}Te_3$- C_{60} alloys on temperature at various concentrations of C_{60}

6. Methods of measuring of thermal conductivity and heat capacity of synthetic single crystal diamond in the temperature range of 2 - 400 K was mastered.

Studied structures in the holders for studying the heat capacity and thermal conductivity of diamond

Advanced developments on the basis of synthetic diamond

1. Solar-blind ultraviolet detectors (wavelength range less then 250 nm), including matrix.
2. Dose X-ray and gamma rays detectors, including matrix.
3. Spectrometric detectors of ionizing radiation with high energy resolution.
4. Countable type detectors for measuring of flux of ionizing radiation.
5. Wide range tempreture sensors from 77 to 800 K.
6. Acoustoelectric filters, delay lines and microwave resonators.
7. Power and high speed Schottky diodes.
8. Power and RF FETs.
9. Single and broadband microwave generators .
10. Elements of RAM.
11. independent power supply with a long service life.

Laboratory of Physical Properties of Nanostructures
Group of Development and Research of Diamond-based Acoustoelectronic Devices

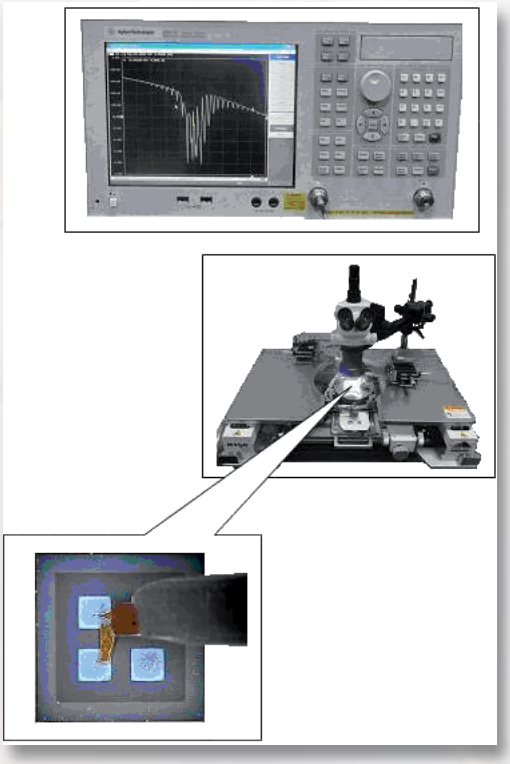
Group Head Professor, Dr. **B. Sorokin**

Acoustoelectronic devices based on bulk (BAW) and surface (SAW) acoustic waves are widely used in creation of acoustic resonators, generators, effective sensors, etc. The group objectives are the development of experimental devices, acoustoelectronic structures, conducting experiments to study the physical properties of crystals and layered structures, modeling propagation of acoustic waves in a crystal, including the conditions of the final external influences.

Research areas

- Development of experimental samples high-overtone bulk acoustic resonators (HBAR), working at 1 – 20 GHz;
- Development of SAW devices based on "IDT/AIN/diamond" layered structure;
- Development of highly sensitive sensors based on acoustoelectronic piezoelectric structures for gas analysis, deposition of ultrathin films, etc.;
- Development of technology for $Sc_xAl_{1-x}N$ thin piezoelectric films deposition in order to use them in the microwave piezoelectric transducers;
- Study of the acoustic properties of the piezoelectric materials and layered structures and their pressure, temperature, etc. dependences;
- Theoretical and experimental research of microwave attenuation in layered structures, such as "Me1/AIN/Me2/diamond".

Experimental devices



E5071C Vector network analyzer, M-150 workstation, and HBAR testing device

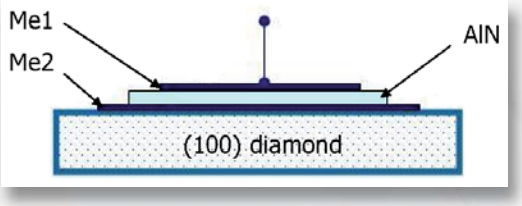
Methods

Long pulse method (RAM-5000 installation) is used for measuring acoustic waves velocities in the range of 20-200 MHz with an accuracy of 0.1%. Thin plate samples with a thickness at least 1 mm are used. Acoustic waves are excited by the piezoelectric transducers of X- and Y-cuts of quartz, and 36° and 163° Y-cuts lithium niobate.

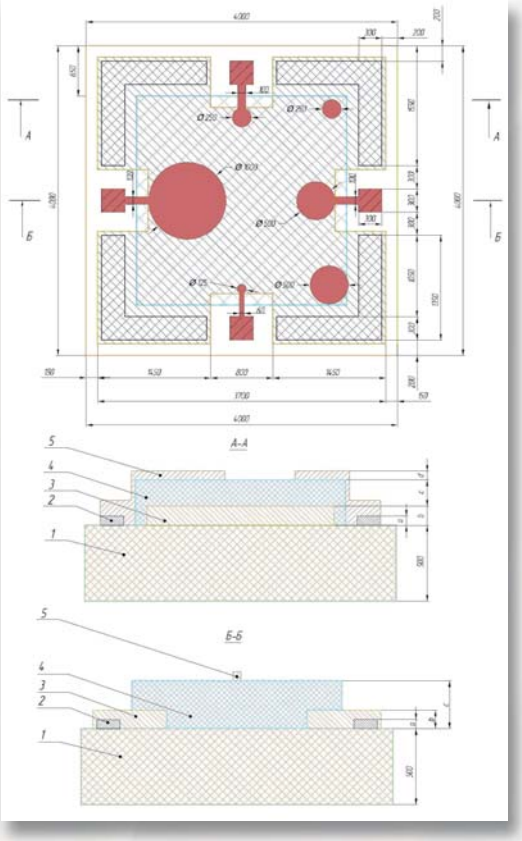
Pulse echo method (AVRK-2-B video pulses generator, DPO71254B oscilloscope) used for accurate measurements of the BAW velocities, and their changes due to external influences (pressure, temperature, electric field) in single crystals.

Measurements of AFC, PFC, and other acoustoelectronic devices characteristics are studied by E5071C vector network analyzer and M150 workstation, working frequency 300 MHz - 20 GHz, temperature range 20 - 300 °C.

Temperature study of acoustic properties of experimental samples produced by Quantum Design installation in the temperature range 4 – 400 °K.



HBAR's schematic diagram

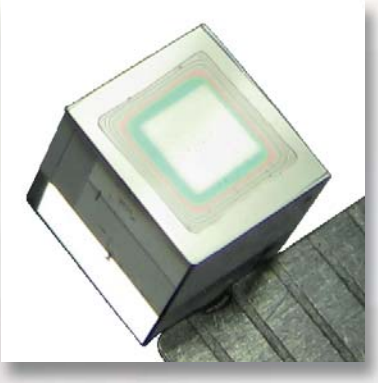


HBAR's documentation design development in AutoCad

Results

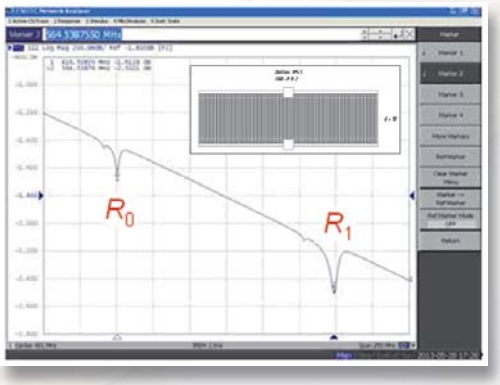
- The second-order elastic constants of IIa type synthetic diamond single crystal were obtained. From measurements of pressure dependences of BAWs velocities for the first time the diamond's third-order elastic constants were obtained.
- Experimental samples of HBAR with a structure "Me1/AlN/Me2/diamond" were created. The record high resonant frequencies up to 20 GHz and quality parameter $Q \times f \leq 10^5$ GHz were obtained. Based on effective data samples pressure and temperature sensors were created

  Structures characteristics:
  - Diamond substrate roughness $R_a < 15$ nm;
  - The accuracy of orientation of the crystallographic faces less than 10';
  - Piezoelectric AlN film thickness from 0.5 to 5.5 μm;
  - Metal films thickness from 100 to 300 nm;
  - Operation temperature from -100 to +600 °C;
  - Operation in the range of uniaxial pressure from 0 to 10 GPa;
  - Operating frequencies: 300 MHz - 20 GHz.
- Samples of BAW pressure sensors operable at pressures up to 10 GPa were created.
- Experimental samples of SAW resonators and delay lines working on frequencies 400 - 1600 MHz were developed.
- Experimental samples of SAW pressure sensors with a diamond substrate were developed. Efficient at pressures up to 150 MPa.
- SAW and BAW pressure sensors with layered piezoelectric structures based on diamond single crystal have competitive characteristics and can be used for measurements of high and ultrahigh pressures.



HBAR with a diamond single crystal as a substrate

HBAR with a diamond single crystal as a substrate with complex electrodes geometry



SAW resonator with a diamond single crystal as a substrate. 418 MHz resonance – SAW Rayleigh wave (R_0), 566 MHz – SAW Sezava wave (R_1)

DEPARTMENT OF FUNCTIONAL AND CONSTRUCTIONAL NANOMATERIALS

Department Head Dr. **S. Perfilov**

Laboratory of Functional Nanomaterials

Laboratory Head Dr. **M. Popov**

Laboratory of Synthesis of Novel Superhard Materials

Laboratory Head Dr. **A. Pozdnyakov**

Laboratory of Physical and Mechanical Measurements

Laboratory Head Dr. **N. Lvova**

Fields or research

- Nanofragmentation and modification of bulk materials by carbon nanoclusters for improvement of their physical, mechanical and functional properties.
- Structure study of carbon nanoclusters interactions and phase transitions during consolidation and sintering.
- Industrial applications of carbon nanoclusters.
- High pressure - high temperature experimental study of nanosized materials.
- Bulk ceramic samples sintering, phase transitions and chemical reactions under high pressures.
- Works on synthesis and application of superhard materials (diamond and cubic boron nitride).
- Research of transport, calorimetric and mechanical properties of nanostructured compact materials.
- Development of synthesis procedures for nanostructured, superactive, metastable materials.
- Development of equipment for the production of metal–carbon clusters at high pressure and temperature, and under intense shear deformation.

We develop and improve the techniques required for cluster production in metal–nanocarbon systems (fullerenes, onions, graphenes, nanotubes, and nanodiamonds). We solve problems related to creating homogeneous mixtures of nanoparticles of various compositions, the production of nanoparticles covered by graphene coatings (onions), the production of clusters from fullerene molecules and metals, and aggregation of the resulting composites.

Double-layer plates of cubic boron nitride - hard alloy.

Cubic boron nitride is the second-hardest natural material.

One of the main methods of cBN application is the use of double-walled plates comprising cBN and a hard alloy. The cBN layer consists of a hard framework of grains, yielding high hardness, wear resistance, and thermal conductivity. The hard alloy substrate has high durability for bending and effectively supports the working layer of the instrument during treatment.

Technological processes developed at FSBI TISNCM for the production of double-walled plates enable the manufacture of products with diameters up to 14 mm and heights up to 16 mm.

Laboratory of Functional Nanomaterials

Laboratory Head Dr. **M. Popov**

Research areas

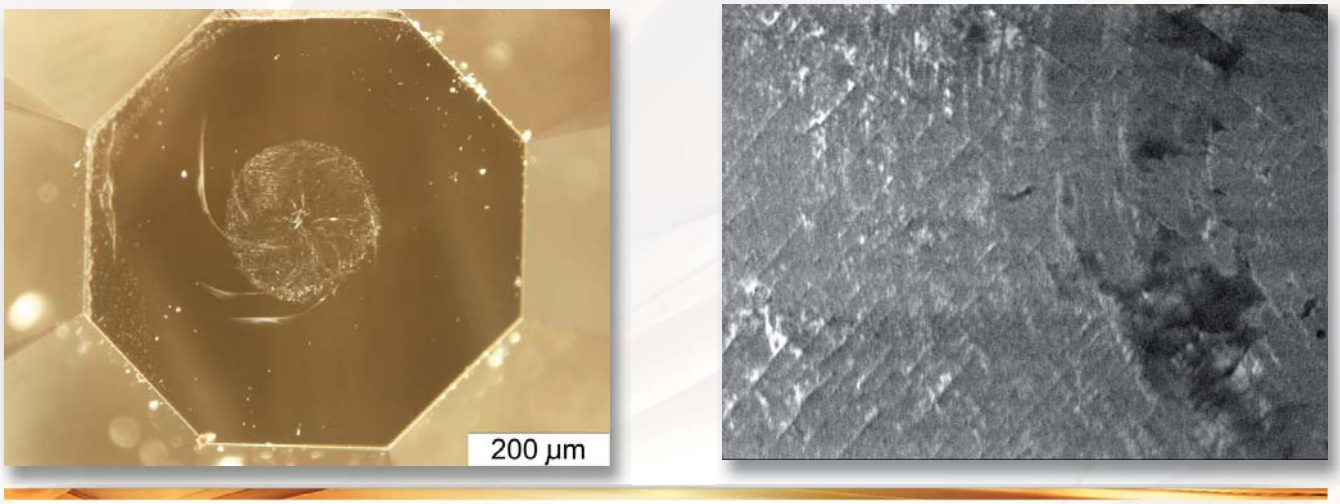
- New carbon materials, including nanostructured and modified by carbon nanoclusters materials;
- Material science, solid state physics, high pressure physics, mechanics of solids, superhard materials and ceramics;
- Raman spectroscopy of carbon nanocluster-based materials and composites;
- Transport properties of solids.

Results

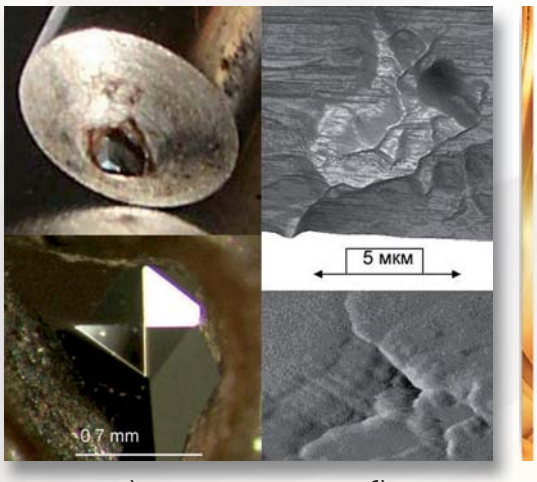
- Discovery of synthesis of ultrahard fullerite with a catalytic 3D polymerization reaction of C_{60};
- A conception development of nanostructured and modified by fullerene C_{60} materials which properties are determined by C_{60} concentration, grain size of nanocrystals and bonding between one and C_{60};
- A development of materials with essentially perfected transport and mechanical properties;
- Developed materials and synthesis procedures are designed for industry.

A catalytic synthesis of ultrahard fullerite

Pressure of ultrahard fullerite synthesis is decreased by a factor of 2 in the presence of a catalyst. Thus, pressure decreases from 18 to 6-7 GPa under conditions of large plastic deformation at room temperature; upon temperature increasing the synthesis pressure further decreases.



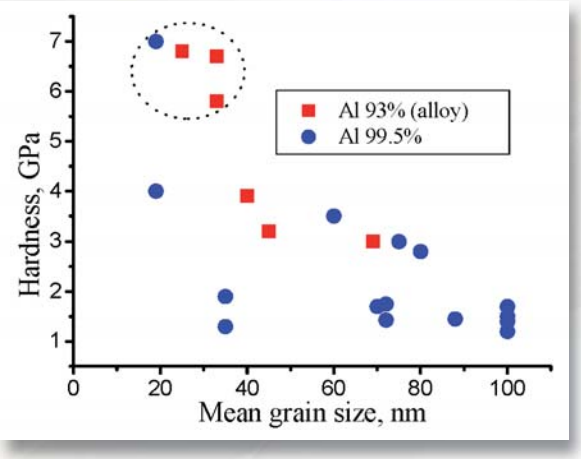
Diamond anvils deformed during synthesis of superhard fullerite: (a) general view of the anvil, (b) × 3000



a) б)

a) Photos of Vickers indenter made from ultrahard fullerite
b) Indentations images produced by this indenter on (111) face of nitrogen-free (nitrogen concentration 0.3 ppm) diamond under indenter load 3N (up image) and 2 N (down image).

Nanostructured and modified by fullerene C_{60} metals



A theoretical shear-strength limit for nanostructured and modified by C_{60} aluminum is achieved.

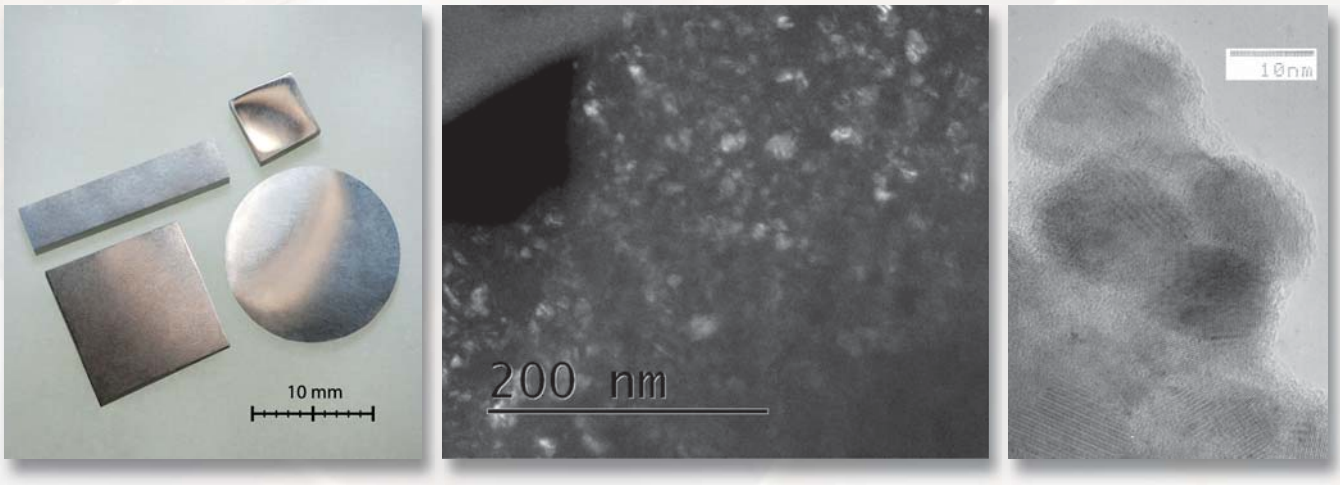
Example:

The dependence of hardness of nanostructured and modified by C_{60} aluminum upon grain size. Maximal hardness H of Al-C_{60} is 7 GPa which appropriates to yield stress $\tau^* \approx 0.33\ H \approx 2$ GPa.

The theoretical shear-strength limit of aluminum is $\tau^*_{theor} \approx G/(10\text{-}15) \approx 2$ GPa also (shear module of aluminum G = 27 GPa)

Nanostructured and modified by carbon nanoclusters thermoelectric materials based on Bi_2Te_3 and Si-Ge

- Synthesized nanocomposites consists of nanocrystals of thermoelectric material covered by layers of C_{60} molecules or other carbon nanoclusters show high figure of merit (ZT) values.
- The carbon layers in the composites prevent recrystallization of nanocrystals during sintering.
- Discovery of a modulation of electron transport properties (Seebeck coefficient S, electrical conductivity σ, mobility μ) by quantum confinement effect.
- Figure of merit $ZT = S^2\sigma T/k$ increases by 20% as a result of independent variation of the parameters S, σ, k in the nanocomposites.



(a) - Samples of sintered nanostructured thermoelectric based on Si-Ge;

(b) - TEM image of nanostructured Si-Ge alloy with mean grain size 10-20 nm;

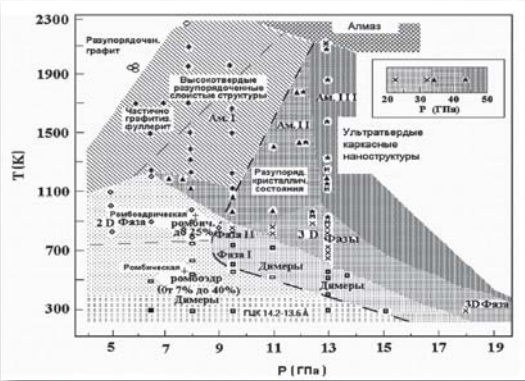
(c) - nanocrystals Bi-Sb-Te covered by C_{60}.

Laboratory of Synthesis of Novel Superhard Materials

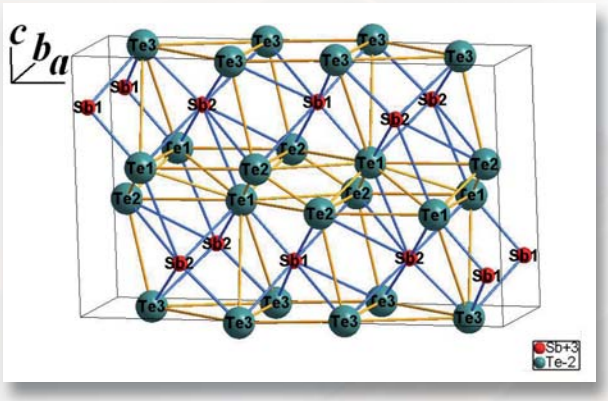
Laboratory Head Dr. **A. Pozdnyakov**

Research areas:

- Development and improvement of high-pressure equipment.
- Improvement of HPHT methods of synthesis of superhard and ultrahard materials with specified data and study of their properties.
- Development of methods and technologies for production of novel nanostructured functional and structural materials with improved properties.



P, T -fields for the synthesis of fullerite C_{60}



The structure of the Sb_2Te_3 metastable phase. Te1-Te2 layers are pulled together in the middle of the unit cell

Results:

Employees of the laboratory together with other subdivisions of FSBI TISNCM carried out a pioneer work on the preparation and study of the structure and properties of superhard and ultrahard phases of C_{60} and C_{70}.

New metastable phases of antimony telluride and bismuth, the most important thermoelectrics near room temperature, were synthesized by rapid cooling after the high pressure (4 GPa) and temperature (600-700° C). By using X-Ray powder diffraction the crystal structure of obtained phases was defined, in which the layered structure is preserved, but alternation of layers is changed. Under the influence of high pressure atomic layers approach each other and metallic bond is formed between the Te- Te layers. New phases have superconducting properties.

Laboratory has obtained:

- a superhard composite (RF invention patent number 2491987 priority 17.11.2011, published on 10.09.2013) based on boron and carbon, which may be used as pressure vessels, the cutting tools with high wear resistance and tool elements for drilling.
- carbon-nitrogen material (RF invention patent number 2485947, priority of 03.11.2011, published on 20.06.2013), which can be used in production of damping elements, dampers, friction pairs and wear-resisting part of micromachines.

Invention patents applied:

Method for obtaining superhard composite material (Number 2012152827/05 from 07.12.2012) based on a fullerene which can be used to make tools for mining, stone processing and metalworking industry.

Method of producing ultra-hard material (Number 2013143992 from 01.10.2013) based on boron and carbon, which may be used in production of cutting tools, drilling tools, and for other applications where high-wearing feature is required.

Laboratory of Physical and Mechanical Measurements

Laboratory Head Dr. **N. Lvova**

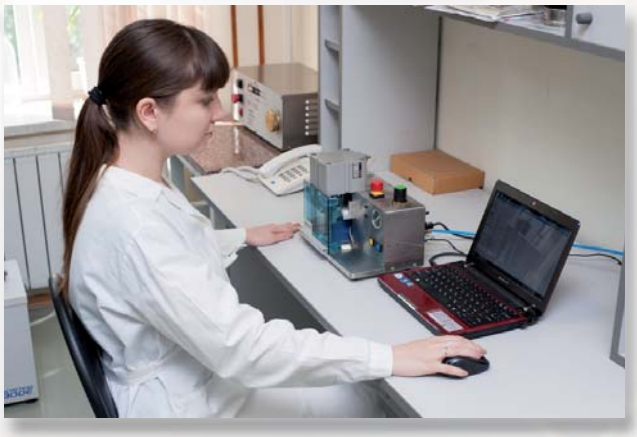
Measuring of properties: static and dynamic strength, morphology, thermal stability of powder materials. Certification of superhard materials: microgrit and micropowder diamonds (natural and synthetic) and cubic boron nitride in accordance with existing standards.

1. DiaTest-S facility (Vollstadt Diamant company), for measuring the strength of powders of superhard materials from 150 micron and higher under static loading.

   A nominal number of loads: 0 ... 2000 N;

   Maximum load: 2300 N.
2. DiaHeat facility for heat treatment of materials in an inert atmosphere.

   Maximum temperature of 1150 °C.
3. ERS-10/5 Electromagnetic roll separators for dry separation of feebly magnetic materials into magnetic and non-magnetic components.
4. DDA-33 facility for measuring of the static strength of superhard materials powders with grain soze from 40 microns to 150 microns.
5. Re.TEK.s.a.s., Diamond Comparative Friability Tester for measuring the dynamic strength index of diamond microgrit powders.
6. Retch, EIT Screening separators. Set of screens for screening and screen analysis of superhard materials powders in accordance with GOST 9206-80, ISO 565, FEPA.
7. SVLM Modernized laboratory vibrational separator (vibrating table) for separating particles of superhard materials into fractions depending on their shape.
8. UAS2M facility for measuring of the abrasive ability of superhard materials micropowder in accordance with GOST 9206-80.
9. Measuring of the density by hydrostatic weighing.
   Laboratory analytical electronic scales (Kern, Germany) with attachment (Sartorius, Germany), model 770-60, Readability 0.01 mg, maximum permissible error of 0.1 mg, accuracy class according to GOST 24104-88 -extra fine fit.



DiaTest-C facility (Vollstadt Diamant)

Electronic scales (Kern) with attachment (Sartorius)

DEPARTMENT OF PHYSICAL AND MECHANICAL PROPERTIES RESEARCH

Department Head Dr. **A. Useinov**

Field of work

- Development of the novel approaches for measuring physical and mechanical properties of materials at the nanometer scale.
- Design and development of new devices and associated software and methods, enabling study of material properties at different scales ranging from nanometer to macroscopic.
- Development of metrological base for ensuring the uniformity of measurements of physical and mechanical values on different scales.
- Development of metrology systems and regulatory basis for ensuring the uniformity of measurements to certify the composition, structure and properties of structural and functional nanomaterials, mechanical and tribological properties of nanomaterials and nanotechnology products.
- Comprehensive study of physical and mechanical properties of broad range of materials: from superhard to polymer, the study of the mechanical properties of thin films and coatings. Measurements of local electric surface properties of the samples.

Methods

- Modelling of physical processes by finite element method
- Profilometry
- Atomic-force microscopy
- Measurement of mechanical properties
- Nanotribology

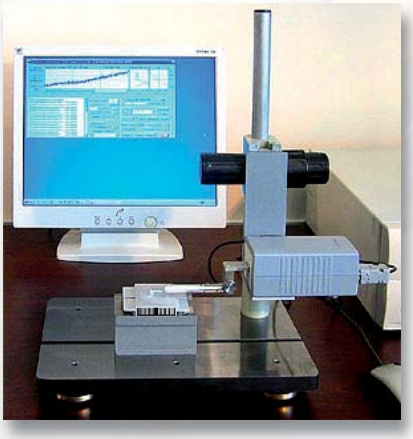
Studied properties

- Surface topography, roughness.
- Hardness measurement over the residual indent image
- Scratch hardness
- Hardness and elastic modulus measurements by instrumented nanoindentation in accordance with the standards ISO 14577 and GOST R 8.747-2011
- Map of mechanical irregularities on sample surface.
- Tomograms of hardness and elastic modulus.
- Adhesion of thin films
- Microconstruction stiffness
- Mechanical nanolithography
- Fracture toughness
- Wear resistance
- Coefficient of friction

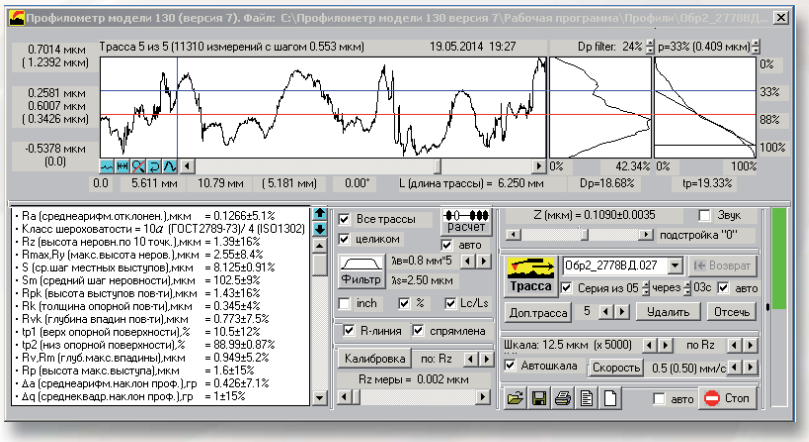
Equipment

- **Contact profilometer «Profi 130», Russia**

Profiling and post-processing with the calculations of the roughness parameters according to GOST 2789-73 and ISO 3274 is using in profilometer model 130 (ZAVOD PROTON-MIET).



Профилометр модели 130

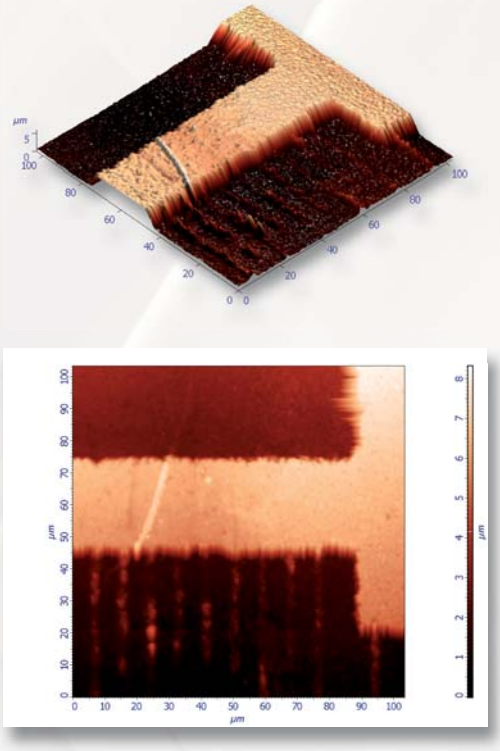


Панель управления, профиль рельефа,параметры шероховатости

- **Modular SPM (AFM, STM) system «NTEGRA Prima», Russia**

This device is a Nanolaboratory, that allows us to use up to 40 measuring methods, in particular measuring of the electric surface potential by Kelvin Probe Force Microscopy. The main and most commonly used technique is to scan the surface topography, which can be carried out in the contact or semicontact mode. Scan size is from 5x5nm to 100x100mkm.

Probe Nanolaboratory «NTEGRA Prima»



AlN structure on the diamond

- **Scanning nano-hardness tester NanoScan-3D, Russia**

The equipment of "NanoScan" series implements a number of methods for the quantitative and qualitative characterization of samples. The NanoScan 3D facility is capable of measuring hardness and elastic modulus of samples by using methods of nano-and microindentation, sclerometry (measurement of hardness) and force spectroscopy (measurement of the modulus of elasticity). Measurements of coating thickness qualitative characterization of adhesion strength and wear resistance are also carried out. Measuring of the coefficient of friction is also possible. All the devices are developed by TISNCM, which make it possible to customize them according to customer's requirements or features of the measuring samples.

Scanning nano-hardness tester NanoScan-3D

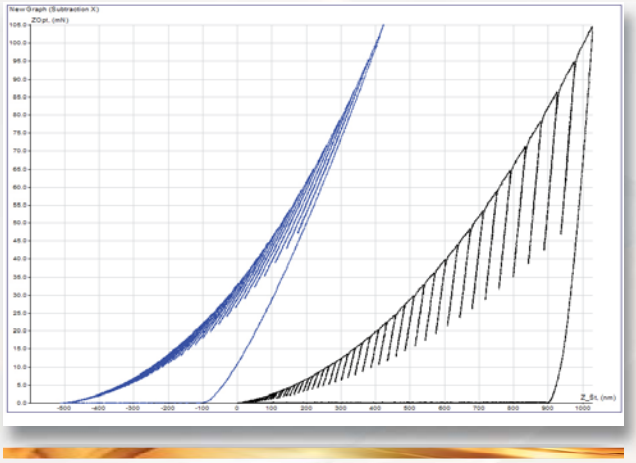
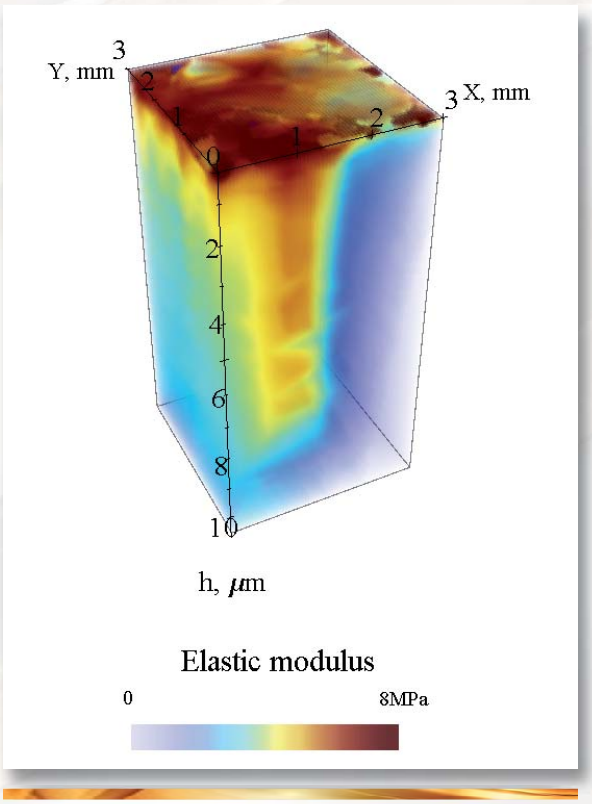


Diagram: power, loading - depth for multicycle pricking, blue curve - fused quartz, blackcurve - steel.

Research areas

- Nanophase and composite materials;
- Superdispersed hard alloys;
- New superhard materials;
- Nanoconstructional materials;
- Semiconductor technology;
- Automotive industry;
- Engineering applications;
- Medical applications;
- Diamonds and diamond powders;
- Data storage device;
- Micro-and nanoelectromechanical systems (MEMS and NEMS);
- Thin films and coatings;
- Protective and wear-resistant coatings.



Elastic modulus tomogram

DEPARTMENT OF NEW CHEMICAL TECHNOLOGIES AND NANOMATERIALS

Department Head Dr. **V. Mordkovich**

R&D directions

The department was founded in 2009 as a division for R&D in chemical methods of nanostructures fabrication and nanomaterial applications. In particular, the research areas include carbon nanotubes and nanothreads, functional and construction composites based on carbon-polymer, ceramic-carbon or metal-carbon components.

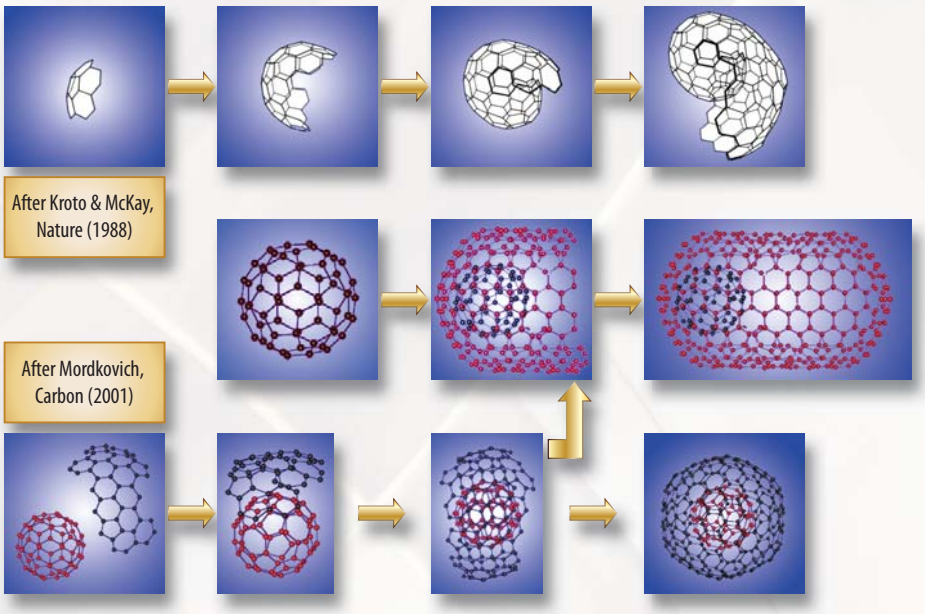
INFRA GTL pilot facility developed in TISNCM in the Department of New Chemical Technologies and Nanomaterials and commissioned by Gazprom.

Methods

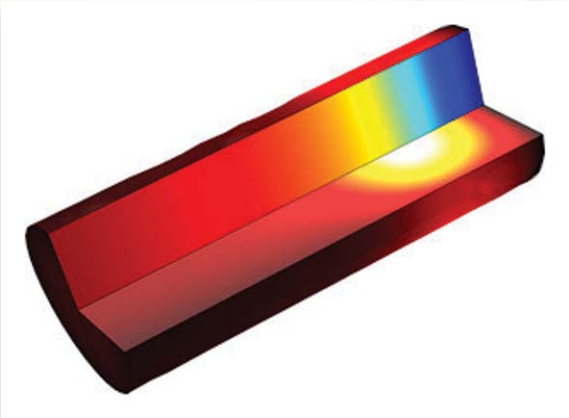
The department of new chemical technologies and nanomaterials is carrying out both experimental works and mathematical modeling of processes. An extensive set of experimental installations is currently under construction, in particular:

- nanostructure fabrication by chemical methods, in particular CVD;
- investigation of catalytic activity of nanostructures in technologically important processes;
- characterization of thermal properties, surface and adsorption properties of the synthesized nanostructures;
- fabrication of composite materials on the basis of the synthesized nanostructures.

Polylayer structure growth mechanisms

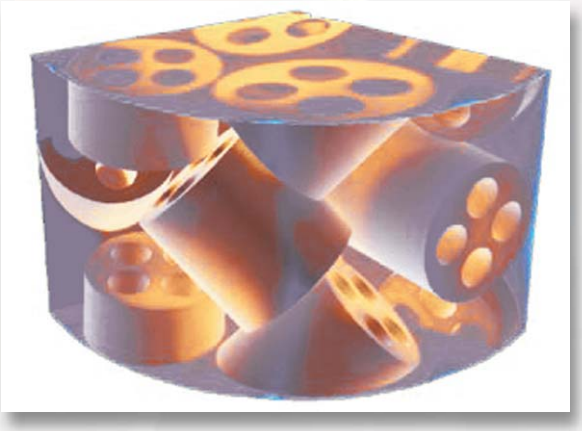


Modern possibilities of heterogeneous catalytic process modeling and main directions of modern development



Quasihomogeneous approach to porous environment modeling

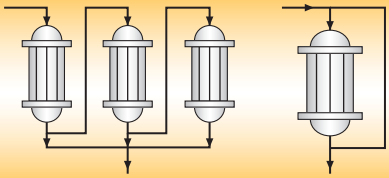
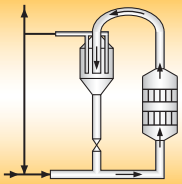
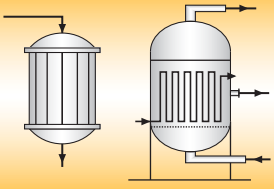
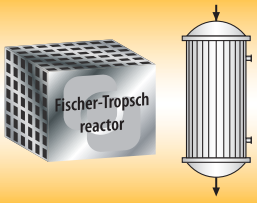
- Simplicity of description.
- Low calculating expenditure.
- Restricted use in the case of low ratio of characteristic size of the apparatus to the size of catalyst granule.
- Impossibility of local measurement of needed parameters description.
- Some effects are impossible to describe within the frames of the approach.



Direct modeling of porous environment

- Complicated description based on CPD of calculating package.
- Considerable calculation inputs.
- The cases of low ratio of characteristic size of the apparatus to the catalyst granule size and porous environment with structured granule packing are described well.
- Precise description of local changes of the needed parameters.

Development of Fisher-Tropsh synthesis technology

1-st generation	2-nd generation	3-rd generation	4-th generation
			
1930-50-e Rurchemie, "ARGE", Sasol	1950-80-e Kellogg, Sasol	1980-90-e Shell, Exxon, BP, Sasol	2000-e CompactGTL Oxford Catalysts, INFRA GTL (TISNCM)

Increasing of reactor power-to-weight ratio – the way to increase the economy of the process.

FSBI TISNCM SHARED-USE EQUIPMENT CENTER

Center Head Dr. **V. Prokhorov**

The year 2004 saw the establishment of the Shared-use Equipment Center "Research of Nanostructured, Carbon and Superhard Materials". The center is part of the Russian SUEC network.

The aim of SUEC is to provide access to scientific and technological equipment within the framework of the Federal Program "Development of Nanoindustry Infrastructure in 2008–2010."

SUEC combines the resources of the Department of Structural Research, Laboratory of Physical Properties of Nanostructures, and Laboratory of Synthesis of Novel Superhard Materials. SUEC houses and oversees modern equipment and original devices and techniques developed by TISNCM.

Applied and fundamental research is focused on superhard and carbon materials, including nanostructured materials. SUEC synthesizes constructional, functional, superhard, and nanostructured materials developed in the Institute or by external clients using an installation УСУ-01/А (pressure up to 13 GPa, temperature up to 2500 °C). Diamond shear anvils (DAC-24; pressure up to 100 GPa, temperature up to 1500 °C) are used for the synthesis, thermobaric treatment, and analysis of nanomaterials.

Technological equipment

URS-01/A facility for thermobaric treatment and synthesis of large single cristal diamonds, superhard and nanostructured materials and for nanocomposites processing.

Laser processing facility on the basis of Lumera picosecond laser for dimensional cutting, grinding and manufacture of superhard materials.

SUEC analytical equipment

Optical studies by absorption spectroscopy, photoluminescence and Raman spectroscopy

Raman scattering (RS) facility with the system of exciting radiation suppression notch filters. The facility was created on the basis of TRIAX series spectrometer produced by HORIBA Jobin Yvon Inc. STABILITE 2017 laser by Spectra-Physics company is a source of excitation light

The integrated optical system for optical spectral studies within the temperature range from 5 to 800 K

Cary spectrophotometer is used to solve a wide range of spectrophotometric tasks. It provides a spectrum with the removal rate of 24,000 Nm/min in the range of 190-1100 nm with a resolution of 1.5 nm. The device has a high sensitivity, stability of two-beam scheme, wide measuring range (200-800 nm)

Structural research by electron microscopy and X-ray diffraction

Multi-purpose X-ray topograph and diffractometry system based on 18kW X-ray source with rotating anode

High-Resolution Transmission Electron Microscope JEM-2010 with resolution up to 1.4 Å (Jeol, Japan)

X-ray powder diffractometer (TETA ARL X'TRA, Thermo Electro Corporation, USA/Switzerland)

A scanning probe microscope–nanoindenter ("NanoScan," developed by TISNCM) is used for research into the relief, structure, and mechanical properties of superhard materials with nanoscale resolution.

Microscope (Olympus BX-51, Japan)

High-frequency acoustic microscope (range 25–100 MHz)

Scanning probe microscope (Ntegra Prisma Basic, NT-MDT, Russia), device for mechanical tests (1958 U10-1, upgraded in 2008), plant for strength measurements (KERN model 770-60), and metallographic equipment (Struers) with a microscope (METAM PB-21) are also applied.

Research into the electrical properties of materials is conducted using equipment for EDS Hall measurements in magnetic fields up to 2T in the temperature range 10–800 K, and equipment for volt-ampere research (including low temperatures and weak magnetic fields).

A thermogravitometer (TAG-24, Setram, France) is used for thermal-physical research.

Since its establishment, TISNCM SUEC has worked with more than 20 scientific institutions in Russia and abroad.

PHYSICS AND CHEMISTRY OF NANOSTRUCTURES CHAIR IN THE MOSCOW INSTITUTE OF PHYSICS AND TECHNOLOGY

Chairholder Prof. **V. Blank**

The Chair was created at the Faculty of Molecular and Biological Physics, MIPT by the order of rector № 37-1 from 24.01.2008. TISNCM was appointed the basic organization of the Chair. The Chair provides students education in following directions:

Bachelor's degree		
Course		Speciality
03.03.01	Applied Mathematics and Physics	Chemical physics and properties of nanostructures
Master's degree		
Course		Speciality
03.04.01	Applied Mathematics and Physics	Chemical physics

Curriculum

Bachelor's degree

Fundamental subjects:

- Introduction to X-ray structure analysis;
- Introduction to the condensed-matter physics;
- Fundamentals of the scanning probe microscopy;
- Physical and chemical properties of nanomaterials;
- Quantum-chemical modeling of the structure and properties of solids;
- Electron microscopy.

Master's degree

Fundamental subjects:

- Structural forms of carbon;
- Solid State Physics;
- Nanoelectronic devices;
- Experimental methods for studying nanostructures;
- Physics and Chemistry of carbon nanostructures;
- Physical basis of the strength of superhard materials.

Schedule of specialist training

Scientific and research work:

- Year IV (bachelor's degree) - 540 hours.
- Years V and VI (master's degree) - 1512 hours.

Lecture attendance:

- Bachelor's degree - 306 hours.
- Master's degree - 330 hours.
- Bachelor's degree qualification paper defence in the 8th semester;
- Master's degree paper to be undertaken in the 12 semester.

RESEARCH AND EDUCATION CENTER "PHYSICS AND CHEMISTRY OF NANOSTRUCTURES"

The Research and Education Center (REC) titled Physics and Chemistry of Nanostructures was founded in 2008 in accordance with FSBI TISNUM administrative order № 36-o.

REC undertakes the fundamental and applied scientific research and innovation research in a wide range of directions in the field of nanotechnology and other priority areas, and develops the critical technologies to promote the high-level science and technology in the Russian Federation.

The REC scientific and technical research and innovative activity represents an integral part of the training the highly qualified specialists and teaching experts provided by the MIPT Physics and Chemistry of Nanostructures department. TISNCM is the governing institute for MIPT.

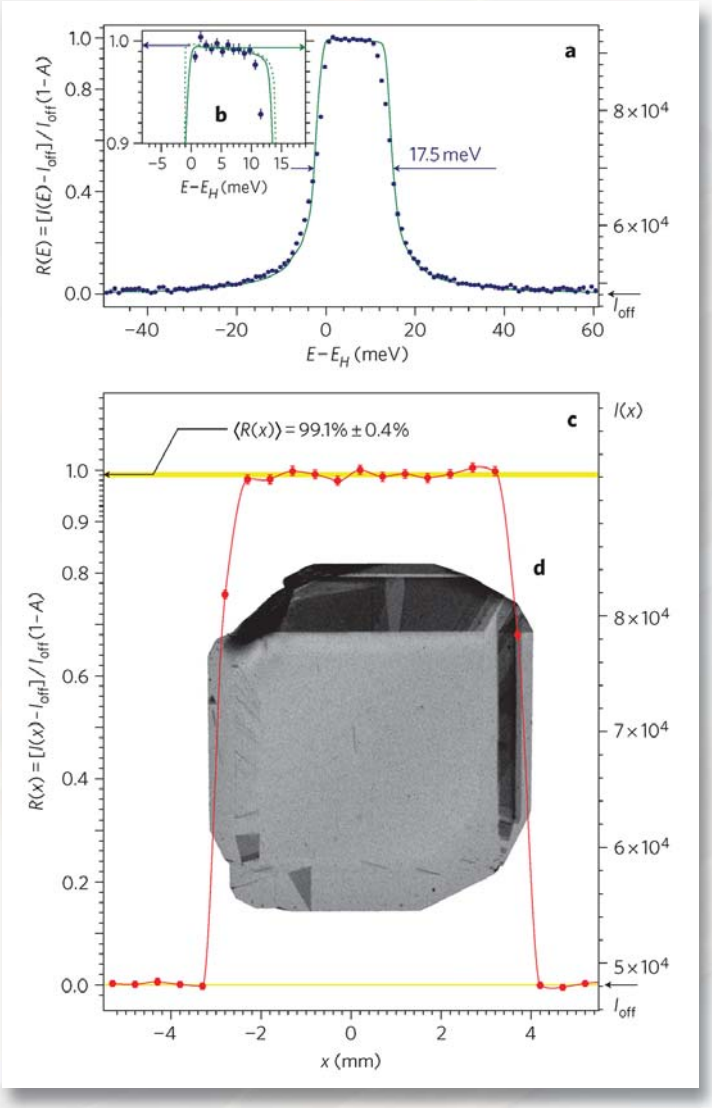
Main research themes

- Physical and mathematical modeling of the formation and behavior of nanosystems.
- Fundamental mechanisms of the nanostructure formation in solids at intensive outer influence.
- Properties and functions of nanostructures (supramolecules, nanocrystals, superlattices, and quantum points).
- Physical and chemical properties of nanomaterials.
- Methods of industrial processes modeling.
- Development and production of the synthetic-diamond-based electronic nanodevices: single electrons, optoelectronics, superconductors.
- Spin electronics, sensors, biosensors, quantum generators.
- High-accuracy methods and devices for the measurement of mechanical, electrical, and magnetic properties of nanosystems.
- High-accuracy measurement and treatment equipment for the nanotechnological processes.
- Mechanisms of diffusion-controlled processes in bulk nanostructured systems.
- Designing the new types of superhard nanocomposite films.
- Production of nanostructured alloys and nanocomposites with the unique mechanical properties.
- Development of production principles for the new homogenous and composite ceramic materials with the nanophase and nanocrystal structure.

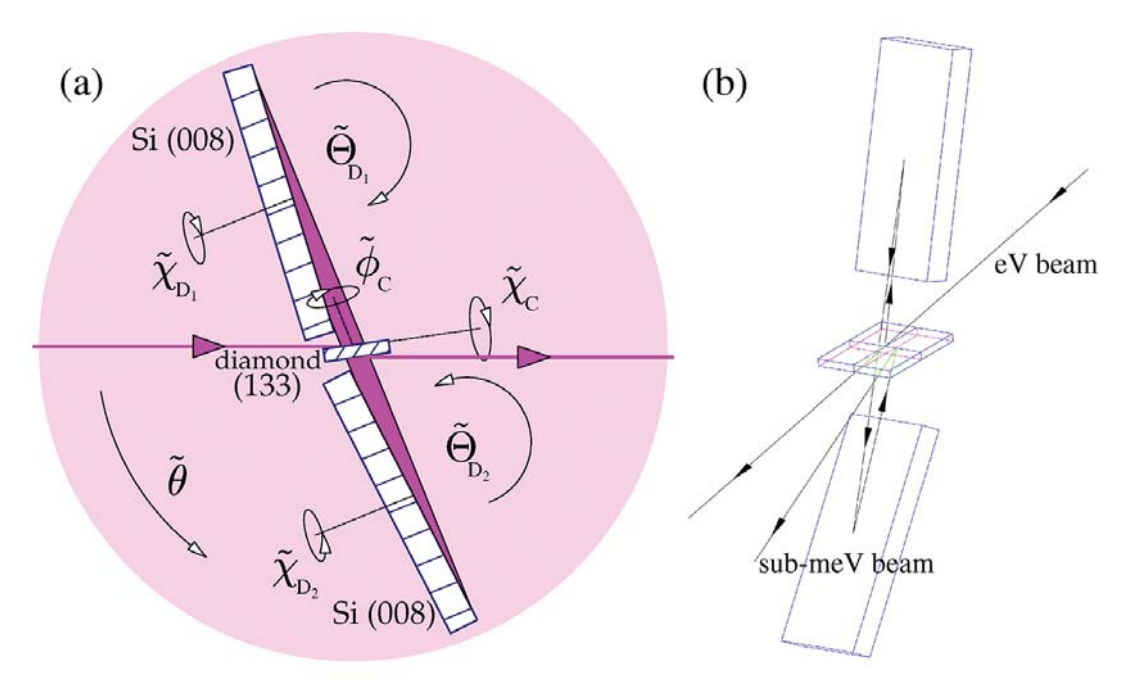
Single Crystal Diamonds for X-Ray optics

TISNCM together with the Argonne National Laboratory and the SLAC National Accelerator Laboratory (DoE) is working on the development of a new generation X-ray optic elements capable of operating in power synchrotron sources. Today TISNCM is a leader in the production of high quality diamonds. On the base of these diamonds the following unique results were obtained:

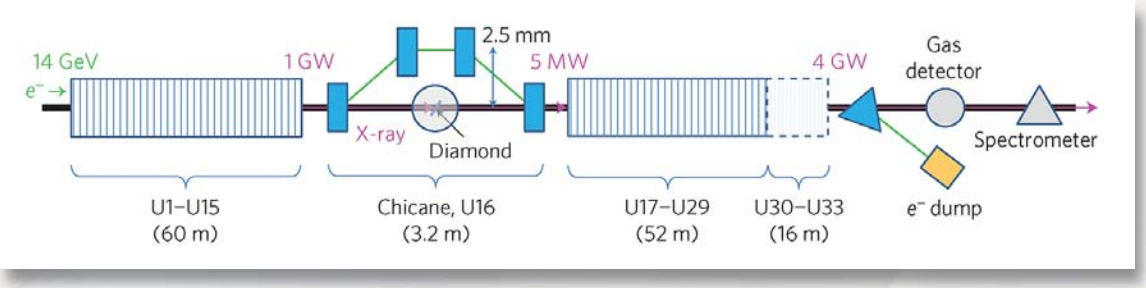
- Diamond plate entirely free of defects with a coefficient of reflection ≈ 99.2%, which is close to the theoretical limit were created. That fact opens the prospect of use of these plates as a resonator mirror to create oscillator type free electron laser.
- Thin diamond plates was used as monochromator set between the two undulators, in world's first experiments for generation of the coherent X-ray radiation in single-pass free-electron laser
- Hybrid monochromator «the diamond -silicon» with extremely small spectral width of 0.25 meV and with high spectral efficiency of ≈ 65 % for inelastic scattering spectroscopy with energy resolution <0.1meV was developed and created.
- Module entirely consisting of diamond components for use on beam channels of synchrotron sources simultaneously as a beam splitter and as a double-crystal spectrometer was created.



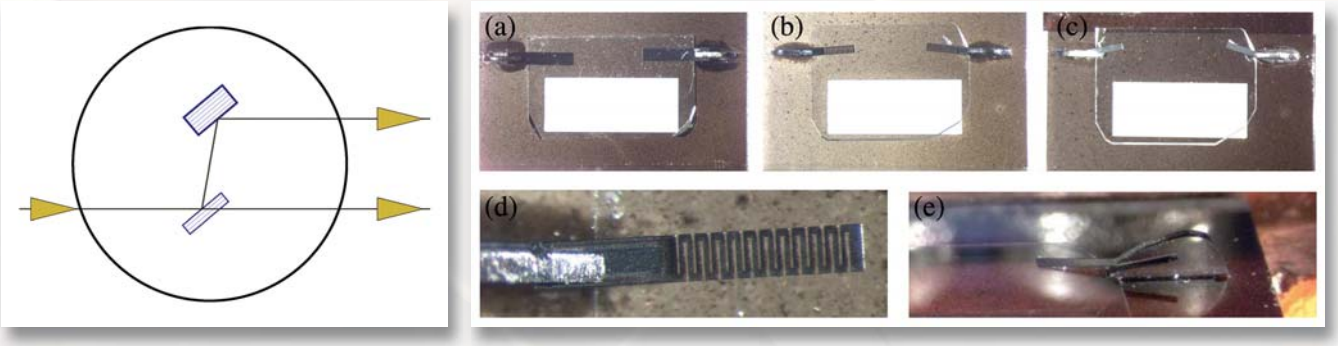
The reflection coefficient of the X-ray with energy of 13 keV of the synthetic diamond with minimum defects level produced by TISNCM



Optical scheme of a hybrid monochromator with a extremely low spectral sensitivity



Scheme of the Free Electron Laser with use of a single crystal synthetic diamond for monochromatic X-ray beam pulse shaping



Diamond components for the two-crystal spectrometer module and the beam splitter

JOINT LABORATORY TISNCM-SIEMENS "FUNCTIONAL AND CONSTRUCTIONAL NANOMATERIALS"

Laboratory Head Dr. **V. Prokhorov**

SIEMENS

Joint scientific research on constructional materials has been conducted since 2007.

An agreement between TISNCM and Siemens Aktiengesellschaft (Siemens AG) was signed on May 30th 2008, establishing the laboratory "Joint Laboratory TISNCM–SIEMENS "Functional and Constructional Materials."

The aim of the TISNCM–SIEMENS laboratory is to facilitate collaborative scientific research and to develop relationship between the organizations in research fields of mutual interest, including:

- cooperation in the field of nanocarbon and modified nanocarbon materials and other fields according to mutual agreement;
- training and support of engineers and scientists in the field of nanoconstructional materials.

Signing of the agreement.

CARBON SOCIETY - RUSSIAN PUBLIC ORGANIZATION OF SPECIALISTS IN THE FIELD OF CARBON AND CARBON MATERIALS

Russian Carbon Society was established on January 8th, 2004. By this time two international conferences ware already held on the carbon theme. In 2014, Professor V. Avdeev was elected as President of the Russian Carbon Society, and Professor V. Blank was elected as Secretary General of this organization. In 2004 – 2006, the 3rd, the 4th, and the 5th international conferences on the theme “Carbon: fundamental problems of science, material science, technology": were held on the basis of Moscow State University.

In 2009, Professor V. Blank was elected as the President of the Russian Carbon Society, and Secretary General of the Society became Prof. V. Avdeev. In the same year the 6th International Conference on traditional topics on the basis of TISNCM was held in Troitsk, Moscow region.

In 2010, the 7th International Conference "Carbon: fundamental problems of science, material science, technology" was held together with Vladimir State University in Suzdal, and in 2012 the 8th International Conference was held in Troitsk on the basis of TISNCM.

In 2011 and 2013 the Russian Carbon Society organized seminars on the theme: "The participation of young scientists in fundamental and applied studies to develop a new carbon and nano-carbon materials" for young scientists of Ural and central regions in Perm and Zelenograd (Andreevka) under the guidance of academician V. Antziferova and member of Russian Academy of Sciences - V. Kostikova.

Each conference includes plenary, sectional and poster presentations; round tables and discussions on topical issues of research, different cultural programs. Leading scientists from Russia and other countries participate in these conferences, and much attention is given to the participation of young scientists from Russia.

QUALITY MANAGEMENT SYSTEM (QMS)

In order to improve the quality of development and to enable the collaboration with the Ministry of Defense of the Russian Federation, it was decided in 2006 to establish a TISNCM Quality Management System (QMS) (Order # 2/1-o dated January 20, 2006). In December 2006, the commission of 22 Central Research and Test institute of the Ministry of Defense during the inspection of the QMS of TISNCM made the conclusion that the current QMS of TISNCM meets the requirements of the Government standard in the development of vacuum tubes, instruments, pressure and temperature measurement and quantum electronics, and recommended to the QMS Certification Department of 22 Central Research and Test institute to issue a decision of that TISNCM is able to provide conditions necessary to ensure the affordance of the State Defense Order.

Hereafter, the institute was constantly working on improving the QMS. As a result of this work in 2012, TISNCM received a Certificate # SVS.01.411.0051.12 dated July 13, 2012 in the system of voluntary certification "Voenelektronsert", certifying that QMS of the Institute meets the requirements of Government standards

In 2013, due to the expansion of the scope of development of the Institute, the final work on QMS documentation was carried out and the Certificate # BP 31.1.6483-2013 of Voluntary Certification System “Military Register” certifying that QMS of TISNCM extending to design and manufacture of the following products classes ETUC: 1010, 1015, 1020, 1025, 1030, 1035, 1310, 1315, 1320, meets the requirements of Government and other standards, valid from September 20,2013 to September 19, 2016, was received.

Currently, the effectiveness of the QMS of TISNCM confirmed by three Certificates of Accredited Russian Certification Center "Elektrosert" and by Voluntary Certification System “Military Register”.

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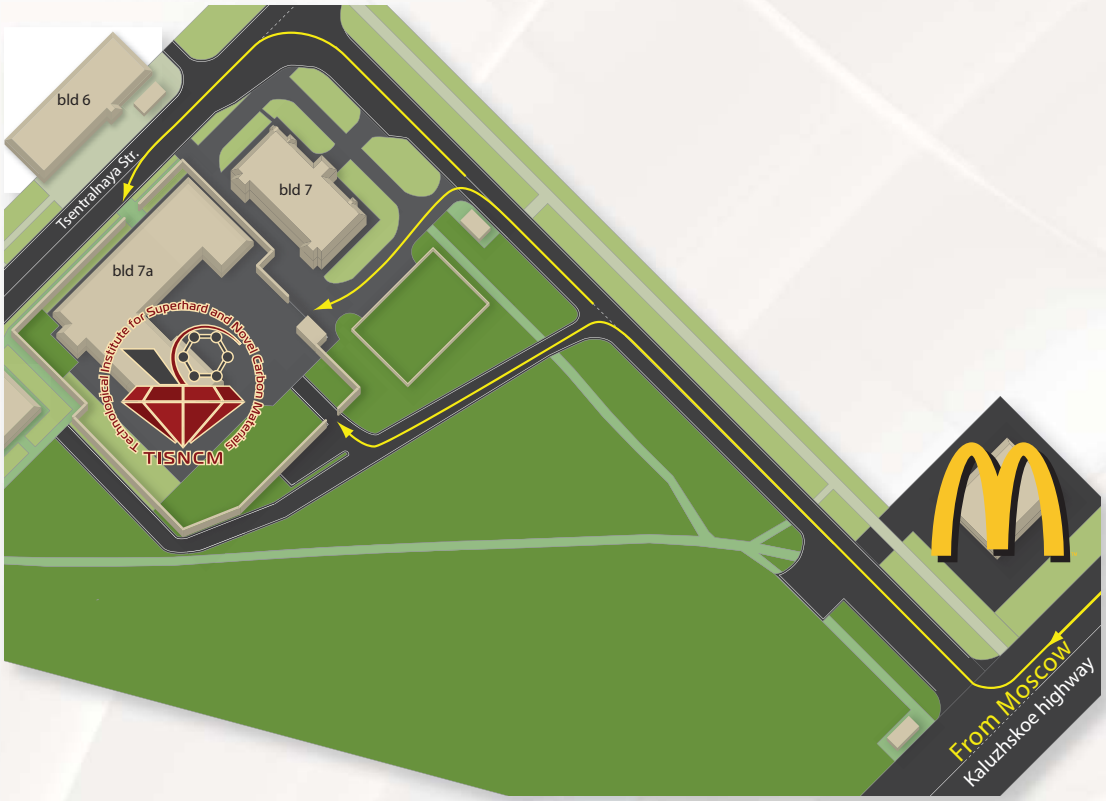

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