Angle resolved photoelectron spectroscopy as the method for investigation of electronic structure of graphene

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Photoelectron spectroscopy (PES) is one of the modern methods for investigation of occupied electronic states of solids. The photoeffect is the basic effect of this method. Electron in an occupied state can be excited to unoccupied state by photon. If the energy of photon $\hbar\omega$ is larger than a work function of solid some electrons can leave a solid and then can be registered. Using of the synchrotron radiation is prevailing last time. Ultrarelativistic charged particle motion in the storage ring leads to the synchrotron radiation which allows achieving of monochromatic radiation with a high energy resolution and a high intensity during registration of photoelectron energy distribution [1,2].

Angle resolved photoelectron spectroscopy (ARPES) is widespread method for measurement of dispersion dependences and symmetry of energy bands of solid. Basically this method has a conservation of parallel to the surface component of quasimomentum of photoelectron when overcoming the potential barrier. We can investigate features of the electronic structure of graphene when measuring dispersion dependences of electronic states in different directions of surface Brillouin zone. In the present time the only feasible route towards large-scale production of graphene is epitaxial growth on a substrate. The presence of the substrate will influence on the electronic properties of graphene layer. And the electronic structure of such graphene will differ from the one of ideal freestanding graphene with such distinctive features as linear dispersion dependences of π-states of graphene near the K point of the Brillouin zone of graphene and a location of Dirac point (the crossing point of cones of occupied and unoccupied electronic states) at the Fermi level.

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Diagnostics of the structure of thin films of polymerized C_{60} formed via electron-beam dispersion method

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Polymerized fullerene C_{60} forms a whole new promising class of carbon materials. Structure of polymerized C_{60} varies from dumb-bell- to peanut-shaped polymers which in turn can form dimers, linear chains, two-dimensional or even three-dimensional networks. The physical-chemical properties of C_{60} polymers vary in a wide range respectively. However, the diagnostics of the exact polymer composition and structure of such materials is often quite a complicated experimental task. Unlike C_{60} monomer, C_{60} polymers hardly can be dissolved. The polymer insolubility provides a quick and convenient technique to check the polymerization. However, on the other hand, it also makes measurement of the polymer composition even more complicated. Therefore, thorough analysis and comparison of the results obtained using different measurement techniques is necessary.

In the presented report, polymer composition and structure of thin C_{60} polymer films, deposited via electron-beam dispersion of pristine fullerite C_{60} in vacuum [1], have been studied using Raman and FTIR spectroscopy, laser desorption/ionization (LDI) and matrix assisted laser desorption/ionization (MALDI) mass spectrometry, X-ray photoelectron spectroscopy.

We have used a precise Lorentzian lineshape analysis to decompose Raman spectra of the deposited coatings into components characteristic of dumb-bell-shaped fullerene dimers, 1D and 2D polymers and thus, to analyse the polymer composition of the coatings. The results have been also proved by FTIR spectra analysis. However, we have also shown that estimations of the polymer composition based on the vibrational spectra clearly overestimate the content of fullerene dimers due to end C_{60} molecules in polymer chains and clusters.

It has been shown that LDI mass spectrometry allows for detection of polymer clusters in the deposited coatings. However, C_{60} polymers readily dissociate to monomers upon desorption/ionization process. Therefore, a soft mass spectrometry technique, MALDI was used to obtain information about the polymer composition of the coatings. Polymer clusters with the size of up to 7 monomer units have been found in the mass spectra. The results of the mass spectrometry measurement are compared to estimations based upon vibrational spectra as well as to XPS data. Peculiarities of each experimental technique in the measurement of thin C_{60} polymer film composition are discussed.

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Raman studies of epitaxial multi-graphene films grown on a 6H-SiC substrates.

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Raman spectroscopy has historically been used to probe structural and electronic characteristics of graphite materials, providing useful information on the defects (D-band), in-plane vibration of sp^2 carbon atoms (G-band), as well as the stacking orders (2D-band) [1]. This method fit also for quality examination of new carbon material – graphene. Raman spectra of graphite (graphene) may contain three strongest lines. Line G at ~1582 cm^{-1} derives from the doubly degenerate phonon mode of E_{2g} symmetry from the Brillouin zone center. Line D at ~1352 cm^{-1} appears in samples with high concentrations of structural defects and is associated with phonons close to the K point of Brillouin zone boundary. Line $2D$ (~2710 cm^{-1}) originates from resonant light scattering involving two phonons of equal energy but with oppositely directed wave vectors, and provides information on the stacking order of graphite (graphene) layers.

The micro-Raman studies were performed in back-scattering geometry $y(xx)\bar{y}$ on a Horiba Jobin-Yvon T64000 spectrometer by means of a confocal microscope at room temperature. The spectra were excited with an Ar+ laser (λ = 514.5 nm). To exclude local heating effects, which could result in a shift of phonon lines, the laser radiation power on the sample was <1 mW (diameter of the laser beam was 4 μm).

For investigations multi-graphene layers on 6H-SiC substrates were used. Graphene films were grown on silicone carbide by sublimation method [2]. We used samples grown in the temperature range of 1300-1600°C to examine the quality of multi-graphene films from growth temperature.

Raman spectra of samples grown at low temperatures (1300-1400°C) exhibit strong line D, which indicate the presence of defects, whereas spectra of samples grown at high temperatures (1500-1600°C) don't exhibit D-line.

Thus Raman investigations show that the multi-graphene films grown at low temperatures are much more defective than the films grown at high temperatures.

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Infrared absorption studies of surface functional groups of chemically modified nanodiamonds

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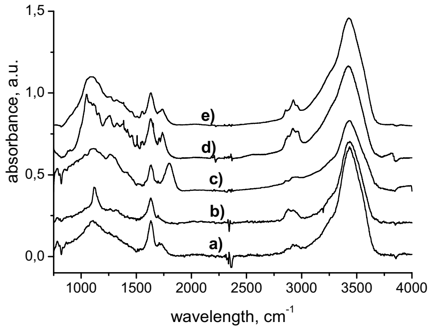
Efficient chemical modification of detonation nanodiamond (DND) surface are able to occur in suspension consist of separate DND particles. Production such suspension require either disaggregate solute (breaking bonds between particles) and prevent particles coagulation that happens when particles have certain charge due dissociation of surface functional groups.

"Z+" and "Z-" samples annealed at 400°C in hydrogen atmosphere and in air respectively. Surface of other samples are decorated with separate metallic ions Cu^{2+}, Co^{2+}, Ce^{3+}, Ce^{4+} or Eu^{3+} alternatively. Concentration of ions varies between 0.1 wt.% and 2.5 wt.%. All the samples compared with initial purified DND and with each other.

Obtained spectra reveal that "Z+" sample surface contain no carbonyl groups, but a lot of C-H groups, instead. Opposite, "Z-" sample has a lot of carbonyl groups in place of C-H groups. Apparently, ultrasonic treatment in water solution leads, that all samples contain significant and nearly equal amount of hydroxyl groups.

The metallic ions modified samples did not show any significant difference of its' surface visible at IR spectra. Furthermore, it is impossible to distinguish samples with different concentrations of the ions. The only one sample (modified with Cu^{2+}) showed visible difference, but this sample had been prepared from different kind of initial DND then others were.

Altogether, the only one functional group (except hydroxyl) predominate at the surfaces of each "Z+" and "Z-" samples, causing their high disperse ability in water solution. Analyzing IR spectra, neither concentration nor type metallic ions at DND particles surface can be established but certain information about initial DND and methods of purifying and modification applied to the sample can be obtained.



DND spectra of a) initial purified DND b) Z+ c) Z− d) Cu^{2+} modified DND e) Co^{2+} modified DND.

Diagnostics of nitrogen-doped carbon prepared by polyaniline pyrolysis

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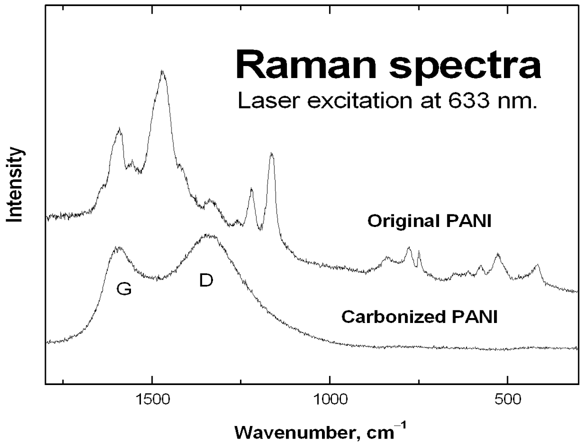
Nitrogen is the best dopant of carbonaceous materials due to its strong electron donating properties and atomic size being similar to that of carbon. Nirogen-doped carbon (NdC) demonstrates improve electron field emission properties because of reduced work function, save as the best support for the heterogeneous catalysis showing synergetic catalytic effect, hardly increased specific capacitance to cations and can be promising material for battery and supercapacitors.

Now the new NdC with high nitrogen content were prepared by pyrolysis of conducting polyaniline (PANI) and its composites and studied by the methods of electron microscopy, Raman and FTIR-spectroscopy, conductometry, thermogravimetric- and BET-analysis. Thermodestruction of PANI proceed at the range of 400-900°C at inert atmosphere and lead to chains cyclizatons were C=N (sp^{2}C-N) and C–N (sp^{3}C-N) bonds between nitrogen and carbon obtained. The atomic concentration of nitrogen was estimated by chemical elemental analysis and depending on pyrolysis conditions changes from 6 to 15%. The conductivity and specific surface area of NdC reach 10^{-4} S cm^{-1} and 350 m^2 g^{-1}. The morphology of prepared NdC can be very different and changes from 3D and 1D to complex hierarchical structures, since the morphology of polymer-precursor did not destroyed during the pyrolysis.



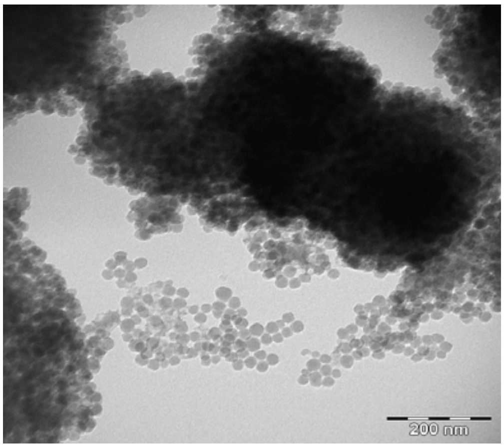


Figure. Raman spectra of PANI and carbonized PANI, transmission electron microscopy of carbonized PANI of 3D structures.

EPR and optical diagnostics of nanodiamonds

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Nitrogen is the main impurity in diamonds, which largely determines their properties. Nitrogen creates various paramagnetic centers in diamonds and exists as individual atoms and clusters. Recently, a great interest has been inspired by studies of nitrogen-vacancy centers (NV defects) in diamonds, for which the magnetic resonance on single defect was successfully observed at the room temperature. However, ND doping processes, formation and structure of intrinsic and impurity defects differ from those in bulk diamonds. In particular, the theoretical studies have shown that nitrogen impurities in ND seem to be metastable in contrast to bulk diamonds. The irradiation methods used to create the NV centers in diamonds/nanodiamonds are purely statistical and the effectiveness of creation of the NV centers in nanodiamond with the size less than 20 nm is still under the question.

Electron paramagnetic resonance (EPR) is one of the most informative and sensitive techniques for the diagnostics of defects in semiconductors. at the molecular level. Herein, we examine the defects in sintered nanodiamonds (ND) by EPR.

Our studies have shown that single nitrogen atoms occupy the stable position in nanodiamond lattice and can be observed in detonation ND sintered under different conditions. Under peculiar sintering conditions it is possible to observe the effect of self-organization of ND into micron size arrays, which is confirmed by orientation dependencies observed in the EPR spectra.

We have also detected very intense EPR spectra corresponding to NV centers in diamonds. Observation of these spectra with and without illumination of the samples allows us to conclude that NV centers can be fabricated in ND without any post or prior irradiation. The formation of NV centers is governed only by high pressure high temperature sintering of detonation ND. The EPR data are confirmed by measurements of photoluminescence (PL) spectra. To determine the best sintering conditions we have performed the series of PL in different types of sintered nanodiamonds and irradiated diamonds.

This work has been supported by the Ministry of Education and Science of the Russian Federation under Contracts No. 14.740.11.0048.

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Determination of the diamond content in the detonation products of explosive

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Currently, the ultradispersed diamond (UDD) powders synthesized in the shock waves from explosives are widely used. A separation of UDD from the synthesis product is quite complex and time-consuming process. Purification of metal impurities and removing non-diamond carbon are the main stages of this process. A test of the purification degree of UDD and identification of impurities, especially impurities of carbon, which forms the amorphous structures, are no less complex.

In this paper, the studies of diamond content in the UDD synthesis product purified from metallic impurities are presented. Relations of sp^2 and sp^3 - hybridized carbon in the samples with various concentrations of diamond were determined by Raman spectroscopy.

Initially, the synthesis product powder was purified from metal impurities. Then removing non-diamond carbon was carried out by treating the powder in the strong liquid-phase oxidants. In the first stage, the diamond content in powder was determined from the decrease in the mass of powder after the treatment.

The obtained data correlate with the number of sp^2 and sp^3 – hybridized carbon in the product.

The relative error in determining the content of diamond in the product by reduce its mass during treatment is less than 5%.

Analysis of two-level organization of detonation nanodiamond clusters by SANS

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The influence of model selection for data processing of small-angle neutron scattering (SANS) by fractal clusters of detonation nanodiamonds in powders [1] and aqueous dispersions [2] is discussed. In addition to previous work we focus our attention on the analysis of the scattering, corresponding to the level of nanocrystallites, taking into account the polydispersity. It was shown that the model of the diffusion surface corresponding to the graphene shell of the nanocrystallites agrees with the contrast variation data in the aqueous dispersions (mixtures H2O/D2O) against the average characteristics, and explains the differences in average grain size determined by small-angle neutron scattering and X-ray diffraction (broadening of the peaks). However, direct determination of the form and parameters of the distribution function of crystallite size encounters difficulties due to the irregular structure of the surface. In particular, determined structural parameters of the particles strongly depend on the type of model distribution function and the residual background.

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Investigation of triplet fullerene C_{70} lineshape EPR under continuous light illumination: zero field splitting parameters distribution

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Continuous wave (CW) and pulse electron paramagnetic resonance (EPR) spectra of triplet state of fullerene C_{70} in various glassy matrices were obtained at temperatures 5K÷260K. At temperatures below 30K the emission region appeared at EPR spectra because populations of $^3C_{70}$ spin sublevels are non-equilibrium immediately after photoexcitation. At low temperatures spin-lattice relaxation time of $^3C_{70}$ becomes comparable with triplet $^3C_{70}$ lifetime. At temperatures higher 50K EPR spectra contained only absorptive part.

Transversal relaxation rate was obtained by pulse EPR methods. It does not substantially depend on EPR spectrum position. So, exchange process within EPR spectrum due to rotations or pseudorotations of $^3C_{70}$ molecule around its long symmetry axis has not appreciably influence the EPR lineshape below 77K.

CW EPR lineshape of $^3C_{70}$ at 77K was simulated supposing the equilibrium population of $^3C_{70}$ spin sublevels. The zero field splitting parameters D and E were revealed to have probability distributions. EPR lineshape of $^3C_{70}$ was simulated successfully assuming Gaussian probability distribution of D value, whereas E value probability distribution function was different. It had a property: at $E = 0$ the probability value is 0. At work [1] EPR lineshape of $^3C_{70}$ was simulated assuming rectangular probability density of E value.

With temperature increasing higher 100K the rapid rising of the transversal relaxation rate of $^3C_{70}$ was obtained from pulse EPR data. The phenomenon can be explained by $^3C_{70}$ molecule motion due to glass matrix softening. CW EPR lineshape narrowing with temperature increasing confirmed the suggestion about $^3C_{70}$ motions.

D and E values distribution can be explained by dependence of molecular $^3C_{70}$ Jahn-Teller distortion on local surrounding of each molecule. We can conclude that $^3C_{70}$ symmetry is not higher than D_{2h}.

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Intercalation of Cu underneath a graphene layer on Ni(111) and Co(0001) substrates studied with a synchrotron radiation

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The electronic structure of graphene on a metallic substrate is similar to that of free-standing graphene, but there are essential differences, which depend on a structure and a material of the substrate and are determined by the character of the graphene-substrate interaction. One of the main tasks is to study the properties of graphene on a wide range of substrates, as the solution of this problem provides an opportunity to purposefully affect on the characteristics of this material. An efficient approach to the solution of this task is based on a phenomenon of intercalation of various atoms underneath graphene films on metals.

Thick monocrystalline films of Co(0001) and Ni(111) were deposited on a preliminarily cleaned surface of tungsten crystal W(110) under the ultra-high vacuum conditions. Graphene films of a good quality were prepared on Co(0001) and Ni(111) films in a process of propylene cracking [1,2]. Intercalation of copper atoms underneath a graphene layer took place after the thermal annealing of pre-deposited copper layers [3].

The electronic structure of a core C1s level and a valence band of a graphene layer on the metallic substrates before and after the copper intercalation has been studied with X-ray photoelectron spectroscopy and angle-resolved ultra-violet photoelectron spectroscopy. X-ray absorption fine structure near the carbon K-edge has been measured for studying unoccupied graphene states. Synchrotron radiation with a linear polarization has been used to reveal a contribution of π^* and σ^* states [4].

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The problem of nanodiamond visualization in biopharmaceutical research

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The problem of carbon nanoparticles diagnostics in biopharmaceutical research becomes more and more actual during the last years. But the complexity of control whether such particles are present and in what state *in vitro* and *in vivo* precludes us from using the standard physic-chemical methods. Therefore the search for new methods and techniques of carbon nanoparticles visualization and of widening their functions via development of new original approaches might be extremely important. This is very important for the research in nanotoxicology and biomedical applications including pharmacology research and drug delivery. The applications of nanodiamonds (ND) as nanocarriers in drug delivery systems necessitated finding the effective methods of their visualization. For the diagnostics of ND *in vitro* we used TEM. This allowed us to determine the average size of ND particles (5 nm) and the presence there of carbon atom layers (shells) with the diamond core in the center. It was possible to estimate the thickness and structural peculiarities of the ND shells depending on their chemical modification by HRTEM. In order to determine the chemical composition of ND surface and the presence to sp^2/sp^3 carbon atoms in near-surface layers of ND particles the XPS method was used. Raman spectra showed that ND have characteristic band of 1332 cm^{-1}. But creation of C_{diam}–N– bonds on the surface results in high fluorescence which is so higher than the peak of diamond phase that totally masks it. The further fluorescence research of ND with grafted drugs by Raman spectra might give us a powerful way to visualize such complexes. The radiochemical methods of carbon nanoparticles visualization are one of the most fast and accurate. We received ND with ^{3}H-label firmly fixed by covalent bond C_{diam}–^{3}H that allowed us to study their biodistribution *in vivo* in rats. The presence of heavy atoms in the grafted layer on the ND surface opens new diagnostic possibilities to determine ND *ex vivo* и *in vivo* by mass-spectrometry. Thus the long-term biodistribution of ND in rabbits was studied. The devised complex of physic-chemical methods of ND visualization allows us to effectively find ND in model systems, biological liquids, tissues and organs.

Transient charging phenomena in graphite

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In carbon research, as well as in general research, graphite being the most stable carbon allotrope is one of the most studied materials. In scanning probe microscopy, graphite is used as calibration grates and atomic-smooth hydrophobic substrates. Surprisingly, very few studies are made on electrostatic force microscopy. Here, we demonstrate the ability of this technique to detect hidden defects (buried underneath) and follow the spatial charge redistribution under the action of electric field.

Graphite is a good electric conductor along its planes, and an insulator only perpendicular to the planes. Therefore, the electric field applied to the plane should not cause any potential fluctuations. And yet, they exist.

Under applied voltage, dendriform quickly changing charged areas were formed limited in their spatial expansion by the surface steps (see Fig. 1). Changing the tip polarity inverted the dark/bright images (Fig. 1 b and c). The charging phenomena were observed only in the pyrolytic graphite samples with the lowest mosaic spread, i. e. highest quality.

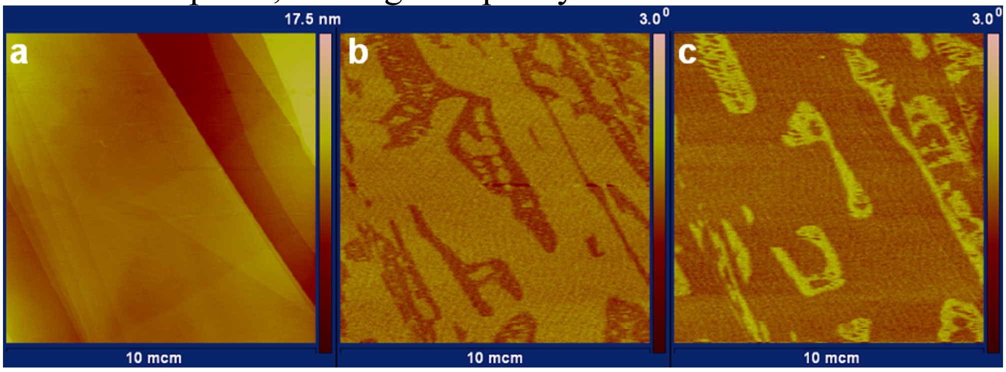

Figure 1. EFM images of HOPG graphite: a – topography, b, c – phase images taken at +2V and -2V, respectively.

The measurements performed in nitrogen atmosphere proved that observed fluctuations do not belong to effects caused by surface contamination such as adsorbed water. Besides, ideal graphite screens poorly, and applied electric field penetrates ~100 nm. Thus, the potential information comes not only from the surface, but charges within the bulk might group themselves at or close to surface steps or buried plane edges and also contribute to the signal.

Further studies on the charging phenomenon of HOPG graphite are needed to understand the origin of this effect.

Electrical conductivity and optical transparency measurements of thin carbon films

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The outstanding electrical1, mechanical and chemical properties of graphene make it attractive for applications in flexible electronics. [1]

Experiments were performed at the lowered pressure in the flow-type reactor. The system involved the horizontal tube furnace, the temperature control systems, gas flow controllers and forvacuum pump. Cu foil was used as a wafer. Thin carbon films were obtained by pyrolysis of ethanol vapor.

Then we measured the transmittance of graphene transferred to a polyethylene terephthalate (PET) substrate using an ultraviolet-visible spectrometer. The sheet resistance was measured using the four-point probe method.

The films display appreciable electrical conductivity and good transparency (92-95%). Because the transmittance of an individual graphene layer is 2,3% [2], this transmittance value indicates that the average number of graphene layers is two to four. These results were verified by atomic-force microscopy measurements.

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