INVESTIGATION OF NEW MATERIALS AND DETECTORS USING THE ELECTROSTATIC ACCELERATOR AND NEUTRON GENERATOR BEAMS

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1. Introduction

Existing at PNPI low energy accelerators – a Van de Graaf electrostatic accelerator with the energy of 1.6 MeV and a neutron generator with 14 MeV monoenergetic neutrons [1–3] – were used for a wide program of fundamental research (nuclear physics, physics of solids, films and surface, semiconductors) as well as for technical and applied investigations (new materials, nuclear detectors). The nuclear physics techniques of material research are used at these accelerators. They include a Rutherford backscattering spectrometry, a nuclear reaction analysis, a proton induced *X*-ray emission and a neutron-activation analysis on fast neutrons. These techniques are nondestructive, high sensitive and precise.

The main results of investigations of solids carried out at these accelerators for past 20 years are stated in this article. They include:

- research of high temperature superconductivity materials $YBa_2Cu_3O_{7-x}$ (films, crystals, ceramics),
- investigation of fullerene materials (starlike fullerene C_{60} containing polystyrene films, endohedral metallofullerenes $Gd@C_{2n}$ and $Dy@C_{2n}$),
- research of metal nanoclusters in dielectric matrices of hydrogenated amorphous carbon α-C:H<Cu> and α-C:H<Co>,
- investigation of lithium diffusion mechanism and intercalation processes in nanoporous carbon materials,
- aging investigations of straw drift-tubes, proposed for experiments on Large Hadron Collider (LHC) and Deutches Electronen Synchrotron (DESY).

2. High temperature superconductivity materials

In 1986 Bednorz and Muller reported compound oxides with very superconducting transition temperatures. This discovery offered the possibility of operating superconducting devices at liquid-nitrogen temperatures. A significant step in this direction was the preparation of superconducting thin films with compositions close $YBa_2Cu_3O_{7-\delta}$, having complete transition temperature T_c above 85° K. Superconducting properties of these materials depend on its elemental composition and structure.

Evaporation of high T_c-materials (films, ceramics, and crystals) is a well developed manufacturing technique. Still, due to strong interaction of elements being evaporated with a substrate and atmosphere some deviations in stoichiometric composition and crystal structure can occur and superconducting properties are not realized. Hence, the study of content and depth distribution of elements was very actually, especially, of oxygen content due to the largest mobility of these atoms in $YBa_2Cu_3O_{7-\delta}$.

The content of oxygen distributed throughout the ceramic has been determined using neutron-activation analysis on the neutron generator. The nuclear reaction $^{16}O(n,p)^{16}N$ and $^{17}O(n,p)^{17}N$ at neutron energy E_n = 14 MeV were used. The ^{16}N radionuclide activity has been determined using high-energy γ-rays (E_γ = 6.1 and 7.1 MeV) with the short half-life $T_{1/2}$ = 7.14 s. The radionuclide ^{17}N is easily detected due to the short half-life $T_{1/2}$ = 4.17 s and delayed neutron with E_n = 100 keV. Although the concentration of ^{17}O in natural oxygen is $3.7\cdot10^{-4}$ the reaction can be used due to the high neutron yield of $2\cdot10^{12}$ s^{-1} from the neutron generator and high sensitivity of the delayed neutron detector used. Finally, the method of reference material was used and reproducibility of results *e.g.* the uncertainty of measurements, is about 0.3% of oxygen weight, oxygen detection limit is 1 μg [4].

The analysis of oxygen in surface layers and films is based on the nuclear reactions $^{16}O(d,p)^{17}O$. The yield of the protons is a measure of oxygen content in a sample. Oxygen sensitivity was estimated to be

10^{15} atoms/cm^2 (thinner than one monolayer) [4]. To measure the ^{18}O concentration the $^{18}O(d,\alpha)^{16}N$ nuclear reaction is used.

For the determination of oxygen distribution with the depth in films up to 1 μm thickness the $^{16}O(d,\alpha)^{14}N$ nuclear reaction was used [5–7]. Here the α-particle energy gives the information on the depth where the reaction occurs and the number of α-particles gives the oxygen content on this depth of the film. The depth resolution of this method is about 10 nm [6]. Content of yttrium, barium and copper was measured using the PIXE and the RBS.

The deposition of various oxygen containing including high T_c superconducting films on various substrates is a technique that has been well mastered. However to reduce the influence of the substance on the film and stabilize the properties of the film, oxygen-containing substrates used (MgO, $SrTIO_3$), butter layers are created between the superconducting film and substrate, and protective coatings are deposited. The $Y_2BaCuO_5/YBa_2Cu_3O_x/Y_2BaCuO_5$ – structures with thickness of 30 nm / 100 nm / 30 nm on the MgO substrate were investigated [5]. The samples were prepared by laser sputtering. For superconducting film like $YBa_2Cu_3O_{7-\delta}$ the most mobile component is oxygen. The element composition and concentration profiles of oxygen in this structure were measured. The interaction between MgO-substrate and films was investigated too. The oxygen depth profile in this structure was homogeneous. It is shown that the Y_2BaCuO_5 ("green" phase) was good isolating film for $YBa_2Cu_3O_{7-\delta}$. For the structure the width ΔT_c was equal to 88.5–85.5 K.

Problem in the technology of high-T_c superconducting crystals is to obtain long specimens with a high critical current. We investigated Y-Ba-Cu-O rod-shape specimen produced Chokhralsky method [8]. The synthesis of these crystals needs to be done using the local control elemental composition and the properties of the medium at the microscopic level.

The nuclear microanalysis technique permits to measure the element composition in original ceramic regions, crystalline part and intermediate layers of this specimen. Using the small angle neutron scattering from the interior regions of the rod-shape specimen we can determine the inhomogeneities of material at the scale $\sim 10^1 - 10^2$ nm from neutron scattering with momentum transfer $q = 0.005–0.08$ A^{-1}. The phase composition of crystal part of this specimen was $YBa_2Cu_3O_{6.98}$(81 %) + $BaCuO_2$ (19 %). It was shown that there is the channeling of protons obtained in nuclear reaction $^{16}O(d,p)^{17}O$ for the crystal only. The neutron cross section shows a fractal behaviour: $d\sigma/d\Omega \sim q^{-(6-d)}$, where d is the fractal dimension of the interior surface in the specimen. The ceramic region have the magnitude of $D \approx 2$ that corresponds to well defined grain boundaries, as far as in intermediate layers $D \approx 2.4$ and in crystalline part the dimension achieved a high value $D \approx 2.6–2.8$. This increasing characterized the synthesis process, i.e. the evolution of the material from an original ceramics ($D \approx 2$ for the grain surface) to a crystal ($D \approx 3$ for a quite continuous medium). These methods should be effective for the determination of the conditions of perfect high-T_c crystals grown.

Crystals of $YBa_2Cu_3O_{7-\delta}$ several centimeters long with a critical current density $j_c = 10^4–10^5$ A/cm^2 at a magnet field $B = 0$ T and a temperature 77 K were grown and investigated.

These investigations were carried out in collaboration with A.F.Ioffe Physical-Technical Institute of RAS (St. Petersburg, Russia) and carried out as part of the Federal Program "High-Temperature Superconductivity".

3. Investigation of starlike fullerene containing polystyrene films

The discovery of the third allotropic carbon-fullerene (other then diamond and graphite) gave impetus to the development of a new investigation direction in polymer chemistry. Fullerene possesses a number of unique chemical properties, which, in particular, made it possible synthesize star like polymers whose structural element represents a macromolecule with a core in the form a C_{60} molecule. Fullerene containing polystyrenes films were synthesized, in which the number of polymer chains attached to the C_{60} molecule can reach six [9]. Fullerene-containing polystyrene molecules are linked *via* the $-Si(CH_3)_2$-group or $CH–C_6H_4–CH_2$-group. Molecular masses of chains were from 2000 up 7000. The C_{60} concentration in a molecule was from 0.5 up 7 % (molecule). These films are characterized by good adhesion with silicon, galillium arsenic, and glass, which were used as substrates. After a lapse of one year, the polymer films 100–200 nm thick did not peel from the given substrates and withstood repeated temperature changes from 300 to 77 K.

Since uncontrollable background impurities remaining in starlike polymers after the synthesis could considerably affect their physical properties, we investigated the elemental composition of the films, by nuclear physics techniques (PIXE, RBS, NRA). Lithium, bromide, oxygen, and chlorine were found. Their concentrations were ~ 10^{20} cm^{-3} for lithium, bromide, and chlorine and ~ 10^{21} cm^{-3} for oxygen. Therefore, it can be concluded that elements involved in the synthesis of starlike fullerene-containing polystyrene and partly remaining in the prepared compound and can substantially affect its properties.

Physical properties of the films were investigated using ellipsometric, photoluminescence, and electrical conductivity techniques. Results obtained it this study indicate that fullerene forming covalent bonds with polystyrene considerably affect these properties despite its low concentration compared to the polymer Actually, according to the ellipsometric investigations, the fullerene containing polystyrene film has a nonzero extinction coefficient. The complex refractive index of the films is equal to $1.7 - i(0.05–0.10)$. It is found that a maximum in photoluminescence spectrum of the fullerene containing polystyrene film is shifted toward the high-energy range as compared to that of the C_{60} film. The energy shift ΔE is directly proportional to the number N of polymer chains chemically bonded to the fullerene molecule and can be described by the empirical formula ΔE [eV] = 0.04 N. The ΔE does not depend on molecular mass of chains. The electrical conductivity of the fullerene-containing polystyrene film is four or five orders of magnitude higher than that of pure polystyrene and correlated with an increase in the fullerene concentration in the films.

These investigations were carried out in collaboration with A.F.Ioffe Physical-Technical Institute of RAS (St-Petersburg, Russia) and supported by the State Scientific and Technical Program "Topical Directions Physics of Condensed Matter", the Direction "Fullerenes and Atomic Clusters" (project No. 98076 "Polymer-2") and Russian Foundation for Basic Research (project No. 98-02-3327).

4. Investigation of the production and enrichment condition of endohedral metallofullerenes $Gd@C_{2n}$ and $Dy@C_{2n}$

Endohedral metallofullerenes $Me@C_{2n}$ are novel forms of fullerene-related materials which can contain various types of metal atoms in the various size of fullerene cages C_{2n}. The yield of the endohedral metallofullerenes amounts typically 1–2 % of the extractable part of the soot produced by arc-evaporation of metal oxyde-graphite composite rods. The main part of the extractable fullerenes is hollow fullerenes. For isolation of individual metallofullerenes, commonly a preparatory high performance liquid chromatography is used. The RBS and PIXE methods were used for real-time elemental analysis during their enrichment and extraction from a fullerene mixture by the method of selective multistep extraction. We investigated the optimal conditions for production of endohedral metallofullerenes $Gd@C_{2n}$ and $Dy@C_{2n}$ [10]. It is shown that there is a distinct maximum of the yield of $Me@C_{2n}$ at helium pressure around 250 Torr inside the evaporation chamber. The yield at the maximum amounts 15% wt of the extractable fullerenes compared to 2.6 % at 45 Torr and 5.7 % at 450 Torr.

For the samples containing $Gd@C_{2n}$ and $Dy@C_{2n}$ the measured atomic ratios Gd/C and Dy/C are $(6.5 \pm 0.3)\cdot10^{-3}$ and $(7.3 \pm 0.3)\cdot10^{-3}$ and concentrations of the endohedral metallofullerenes converted to $Ln@C_{82}$ main component amount ~57 % wt and ~63 % wt of fullerene mixture respectively [11].

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5. Metal nanoclusters in dielectric matrices

Fabrication of systems of metal nanoclusters in dielectric matrices and investigation of their properties reflect the general interest of modern condensed-matter physics in limited-dimensionality structures. The modification of hydrogenated amorphous carbon (α-C:H) by various elements (metals) remains a significantly less advanced field of investigations, although at present, progressively greater attention is paid to this problem, in particular, to the introduction of metallic nanoclusters into the carbon matrix with the purpose of creating new types of electronic and magnetic media. Nanoclusters system based on amorphous carbon appear to be promising media for information recording and storage, especially if the clusters are formed from magnetic metals.

Amorphous hydrogenated carbon films (α-C:H<Cu> and α-C:H<Co>) having metal concentrations of several to tens of atomic percent and belonging to nanoclusters mesoscopic systems were investigated [12–14]. The films were prepared by magnetron co-sputtering of the graphite and metal targets in the argon-hydrogen plasma (80% Ar + 20% H_2) on silicon substrates. Thickness of films was from 100 nm to 2200 nm. The concentration of metal introduced into the carbon films was varied by changing the metal-to-graphite target-area ratio Σ = S[Me] / S[C].

The relative atomic concentrations of carbon, copper, and oxygen were determined using the RBS and the NRA (reactions $^{16}O(d,p)^{17}O$ and $^{12}C(d,p)^{13}C$). The ratio of atomic concentrations [Cu]/[C] changes from 0.17 to 0.76 for thin films and from 0.90 to 1.3 for thick films. The ratio of atomic concentration [O]/[C] changes from 0.35 to 0.55 for thin films and from 0.8 to 0.95 for thick films. Two basic conclusions can be made from these measurements. First, the actual ratio of the atomic concentrations [Cu]/[C] exceeds that of the target areas by a factor of 6–8, owing to the difference in copper and graphite sputtering rates. Second, a high oxygen content is characteristic of the films. Its presumed origin is the residual atmosphere in the grown chamber. Similarly to copper, oxygen is introduced into thin and thick films in different concentrations under the same technological conditions. The oxygen concentration grows with increasing copper concentration in both types of samples. For films up to several micrometers thick the mass thickness were also determined using the RBS. They change from 1.8 g/cm^3 for the α-C:H-films to 3.81 g/cm^3 for the α-C:H<Cu>-films, where [Cu]/[C] was equal to 1.3. The value of about 1.7 is typical of amorphous hydrogenated carbon. The values of about 3.6 g/cm^3 for films with copper are directly due to the copper contribution (the bulk copper density is 8.9 g/cm^3).

Scanning electron microscopy was used to obtain information on the surface morphology and the internal microstructure of the films. The in-depth non-uniformity of the films was observed. The presence of uniform layer near by a substrate with the thickness of 100 nm, the formation of non-uniformities, their widening with moving from the substrate to the free surface, the emergence of the internal inhomogeneities into the surface with the formation of a "granulated" relief was observed. The average size of the granules increases from 150 nm near a substrate to 300 nm on the film surface. The in-depth non-uniformity of the film can be explained taking into account large internal stresses characteristic of the amorphous carbon films.

The conductivity data demonstrate an anisotropy associated with the geometry of the α–C:H<Cu>-film growth process as well. Conducting channels of the "punctire" type across the film are observed in a wide range of copper concentrations (in the range from 0 to ~ 30 at. % as estimated), while the in-plane conductivity is low. The whole set of conductivity data for copper concentrations up to 50 at. % (approximately) can be interpreted in terms of the insular-matrix model ($\sigma = 10^{-10}$–10^{-8} Ω^{-1} cm^{-1}) with inclusions of two different conducting phases with the conductivity of $\sigma = 10^{-5}$–10^{-3} and 10^{-5}–10^{-6} Ω^{-1} cm^{-1}. The first of these is assumed to be composed of graphite-like nanoclusters, including those intercalated with copper atoms, whereas the second phase is formed by copper nanoclusters.

Thus, structure, composition and electrical properties of the films, prepared by magnetron sputtering are consistent with the interpretation of α-C:H<Cu> as a mesoscopic medium.

These studies were carried out in collaboration with A.F.Ioffe Physicotechnical Institute of RAS (St-Petersburg, Russia). This work was supported by the State Program "Integration" (project No 75) and the Russian Foundation for Basic Research (projects No 00-02-16994 and 00-02-17004).

6. Investigation of lithium diffusion and intercalation processes in nanoporous carbon materials

Lithium can be inserted reversibly within most carbonaceous materials. Therefore, in the last decade, much research efforts have been focused on the search for suitable carbon materials as an alternative anode(s) for lithium rechargeable batteries. The basic requirement to these materials is a high quantity of reversible lithium ion insertion. The process of lithium insertion in nanoporous carbons was studied [15–17]

The samples of nanoporous carbon used in this study were obtained by a high temperature chlorinating process from an intermediate product prepared on the base carbide powders (SiC, TiC, Mo_2C). These materials have high total porosity is up to 70% and nanoporosity is about 50%. Their specific surface area is up to 1300 sm^2/g.

Lithium insertion in the samples was carried out by vacuum evaporation and subsequent diffusion at the temperatures ranging from 30°C to 200°C. The nuclear reactions $^{7}Li(p,\alpha)^{4}He$ and $^{6}Li(d,\alpha)^{4}He$ were applied to measure the depth concentration profiles of lithium. Using these results the diffusion coefficients of lithium D_{Li} in nanoporous carbon were determined.

The comparison diffusion processes in the different nanoporous carbides enables to conclude that the main mechanism of diffusion is lithium diffusion alone pore walls. As a result of lithium diffusion process investigations in different samples with and without preliminary annealing it is stated that value of lithium diffusion coefficient depends on the size of pores and their physical state. Values of D_{Li} in the above-mentioned temperatures vary for different samples in the range 10^{-9}–10^{-7} cm^2/s. The value of lithium diffusion coefficient depends on the diffusion process duration. It is elucidated that such behaviour of $<D_{Li}>$ reflects the different processes that occur in series during the entire insertion process: Li diffusion over pore walls, accumulation of lithium in pores giving rise to Li clusters and the forming of intercalation phases.

The investigation of phase composition of the lithiated samples was performed by means of the *X*-ray diffraction measurements. Intercalation phases LiC_6, LiC_{12}, LiC_{24} and LiC_{40} were observed. Besides Li_2C_2 and Li_2CO_3 phases were register in some cases. It was stated that the correlation between the diffusion rate and the evaporation rate is responsible for the phase composition.

Using these results the conditions for producing of lithium containing samples without lithium carbide and carbonate were found. The samples up to a thickness of about 0.6 mm uniformly impregnated by lithium and containing intercalation phases only were fabricated.

The studies were carried out on nanoporous carbon samples developed and produced in collaboration with Skeleton Technologies Group (Stockholm, Sweden).

7. Aging investigation of the straw drift-tubes

The high energy physics detectors proposed for the Large Hadron Collider (LHC) demand a higher performance than what has been available from conventional gas detectors. It is anticipated that the radiation level near the beam of a storage ring collider will result in an integrated charge level of 10-13 C/cm after about 10 years of operation. This is at least an order of magnitude more than have been achieved in previous experiments. Thus, the study of aging effects for these new detectors is especially important and timely. So, in order to perform the aging investigation of gas-filled detectors and obtain experimental data in a reasonable time, a current at least 10 times more than the one expected in the real experiment has to be used.

Usually, aging effects are the result of a surface degradation of both the anode and cathode electrodes that occurs in the form of “deposits”. This “deposit” consists of gold, tungsten and atoms having in a gas mixture. For the detection and quantitative evaluation of oxygen, carbon and other light elements, which were coming from the gas avalanche and penetrated in to the wire gold coating, the $^{16}O(d,p)^{17}O$ and $^{12}C(d,p)^{13}C$ nuclear reactions were applied [18].

The results of investigations for different points along the anode gold-coated tungsten wire with a diameter of 35 μm from straw drift-tubes are presented in Fig.1. The gas mixture is $Xe+10\%CO_2+20\%CF_4$. The aging test of detector was carried out using an intensive 2 Ci ^{90}Sr β-source. The applied high voltage was chosen so as to provide a gas gain of 2.5×10^4 in the centre of irradiated zone of the wire. At this gain, the straw-tube had a rate of charge accumulation of 0.08 C/cm per day and a total accumulated charge of 3.6 C/cm. To monitor the gas gain, the 5.9 keV peak position of the pulse-height distribution from a ^{55}Fe source was measured.

It was determined that these deposits are distributed both in the center of the irradiation area and along the wire, corresponding to the gas flow direction. We see that the peak concentration of oxygen collected in the center of the irradiation zone increased more than 20 times in compare with the non-irradiated wire. It is correlated with the increase of wire diameter from 35.3 μm to 37.3 μm and the decrease of the measured gas gain in the irradiated zone [19]. The amount of oxygen in the centre of irradiated zone is 1.7×10^{18} at/cm^2. The amount of oxygen measured in the wire near the gas outlet end is large then the one in the gas inlet end.

The investigation of other anode wires with the diameters from 25 to 50 μm for different gas mixtures containing Ar, Xe, CO_2, CF_4 and $C_2H_2F_4$ molecules in different concentrations were used too [20–23]

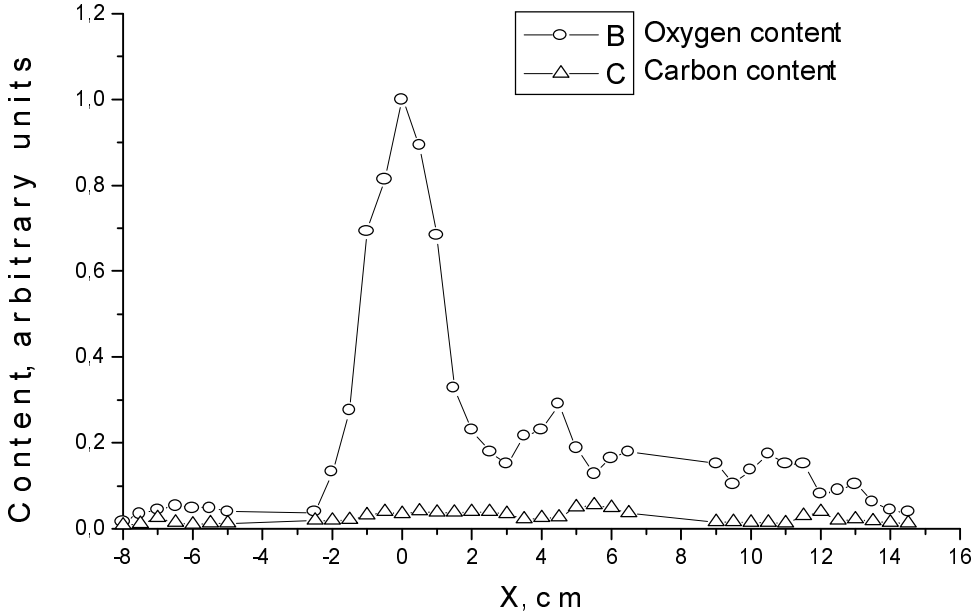

Fig. 1. Distribution of oxygen and carbon along the wire after its irradiation. The amount of oxygen in the center of an irradiated zone is $1.7 \cdot 10^{18}$ at / cm^2. The irradiation dose is 3.6 C/cm. The gas mixture is Xe + 10%CO_2 + 20%CF_4

A comparison of the wire surface structures and the deposits distributed along the wire lead us conclude that the nature of the forces causing the wire aging is the swelling of anode wire. Phenomenon of anode wire swelling is really exists. An analysis of these results shows, that oxygen plays a very important role in this anode wire aging process and greatly speeds up the aging of the anode wires. The penetration of oxygen into the gold coating of the wire and its reaction with tungsten finally causes an increase in the wire volume i.e. the wire swells.

These studies are carried out in collaboration with the PNPI tracing detector department.

8. Conclusion

After the 20 years operation, the PNPI electrostatic accelerators continues to be one of the most active facilities in investigations of materials by the nuclear physics methods. The simultaneous usage of these methods (a proton induced X-ray emission, a Rutherford backscattering spectrometry and a nuclear reaction analysis) in the single installation allows to obtain unique, detail and precise information on a composition, a structure and properties of investigated sample from one experiment. These techniques permit to investigate thin films, near surface region of solids, thin wires one its destruction.

Using these techniques the conditions for producing of different new materials were found and its some properties were investigated. They are the high temperature superconductivity films and crystals $YBa_2Cu_3O_{7-x}$, starlike fullerene C_{60} containing polystyrene films, endohedral metallofullerenes $Dy@C_{2n}$ and $Gd@C_{2n}$, a lithium containing nanoporous carbon. A number of samples of these materials were fabricated.

The investigation of amorphous hydrogenated carbon films (α-C:H<Cu> and α-C:H<Co>), having metal concentrations of several to tens of atomic percent, shown that structure, composition and electrical properties of the ones are consistent with the interpretation of α-C:H<Me> as a mesoscopic medium.

Aging investigations of the anode wires of the straw drift-tubes at a high accumulated doses of radiation that oxygen plays a very important role in this process and greatly speeds up the aging of these gas filled detectors.

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