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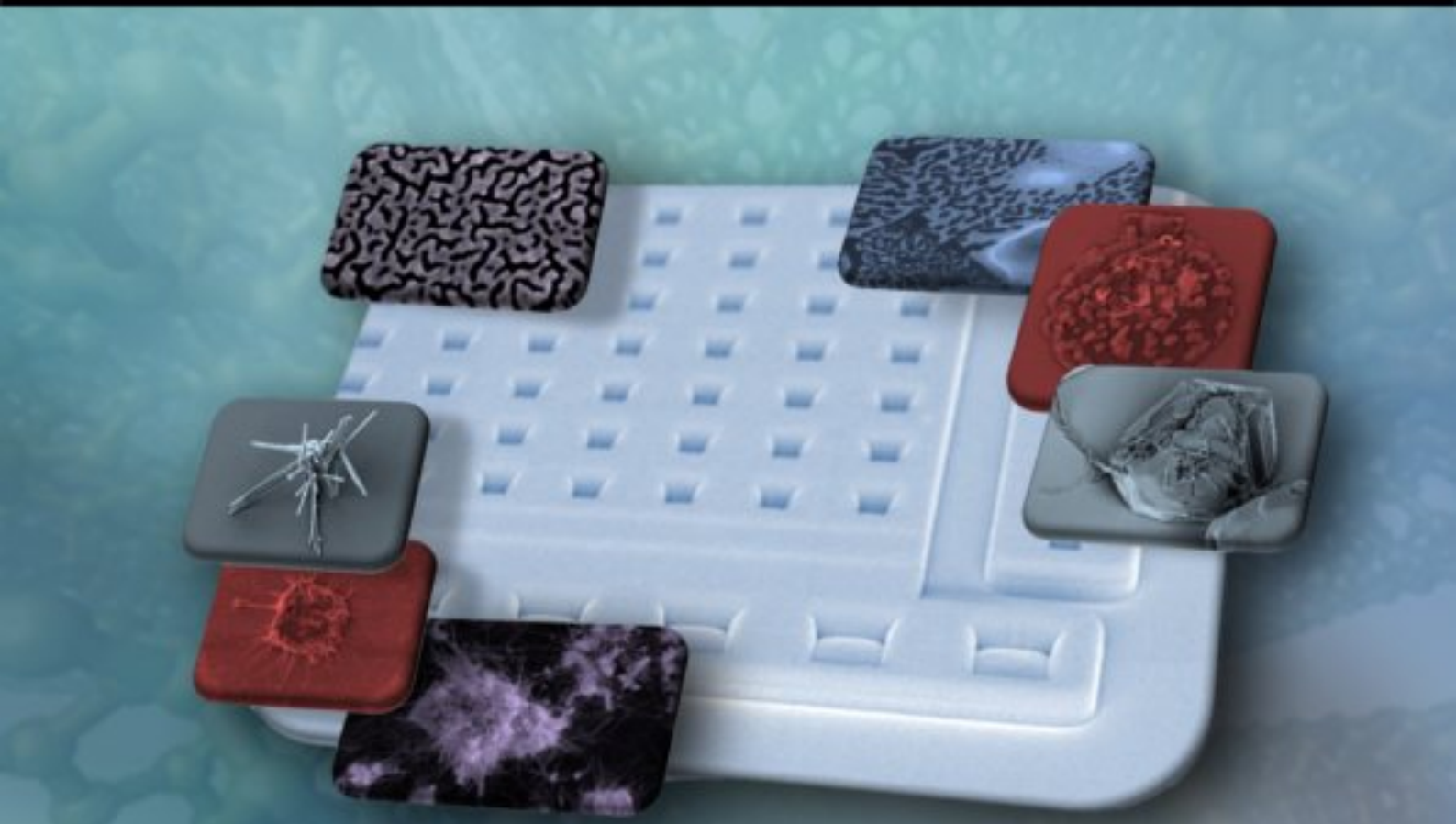

Nanomaterials and their Composites: from Fabrication to Applications

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Nanomaterials Characterization Using Nuclear Methods at IFIN-HH

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Abstract: The aim of this study is to analyze nanostructured materials using Positron Annihilation Spectroscopy (PAS), Nuclear Reaction Analysis (NRA) and Rutherford Backscattering Spectrometry (RBS) nuclear techniques. Three different types of nanomaterials: polymer nanofibers (polyurethane doped with silver nanoparticles), carbon nanowalls (CNW) and InN thin films on yttria-stabilized-zirconia (YSZ) substrate were investigated using these methods. PAS was demonstrated on polymer nanofibers providing information on vacancy defects, RBS was applied on InN thin films providing information regarding the thickness and the stoichiometry of the films. Preliminary NRA results are also presented, showing the possibility of detecting and measuring the hydrogen content in CNW.

Keywords: Carbon nanowalls, Indium nitride, Polymer nanofibers, Positron annihilation spectroscopy, Ion beam analysis.

1. Introduction

In the last two decades the development of advanced materials with desired properties was one of the main goals of materials research. These properties are connected with their chemical and physical characteristics. Understanding the chemical and physical properties at nanometer scale and also designing new tools for characterization of nanomaterials requires the input of interdisciplinary teams

of physicists, chemists, materials scientists and engineers. Nowadays nearly every type of chemical and physical analysis is applied in nanomaterials studies. Nanostructured materials present a variety of obstacles including particle stability, environmental effects, specimen handling and contamination [1, 2, 3]. Also the presence of impurities on these structures is not well characterized and sometimes not adequately identified.

The use of ion beam methods offers the possibility of analyzing and modifying the properties of the near-surface regions of materials without seriously affecting the bulk.

In this study we report the characterization of three types of nanomaterials:
- polymer nanofibers (polyurethane doped with silver nanoparticles) - this type of material is of an increased interest in medical applications, because it might be used as antiseptic bandages for skin burns, taking into account the well-known antimicrobial effect of silver ions [4];
- carbon nanowalls (CNW) - the dimensional characteristics and the specific shape give opportunities to new applications, especially for field emission devices, in the fabrication of combustion cells, gas storage (hydrogen storage), as base material for catalytic nanoparticles [5];
- InN thin films on yttria-stabilized-zirconia (YSZ) substrate - InN thin films are very interesting structures its potential applications being very large – for THz emission devices, solar cells and light emitting diodes [6].

The nuclear methods considered in this paper allow the identification of the concentration and distribution of vacancy-type defects and the space distribution of electronic densities in the bulk - Positron Annihilation Spectroscopy (PAS) [7], the hydrogen concentration using Nuclear Reaction Analysis (NRA) [8, 9] and the composition of multistrate structures and stoichiometry of thin layers by Rutherford Backscattering Spectrometry (RBS) [10].

At Horia Hulubei National Institute of Physics and Nuclear Engineering (IFIN-HH) we have the possibility to analyze nanocompounds using Ion Beam Analysis (IBA) techniques (Fig. 1) [11]. IBA uses the beam interaction with the atomic shell and the nucleus of the target material. IBA methods have a series of common features: most of them are non-destructive, being compatible with nanomaterial analysis; they can be used for the identification of a large number of elements; the analysis can be done on very small quantities of sample material (mg) and they allow the analysis of small surfaces, as the beam diameter is about 0.5 mm.

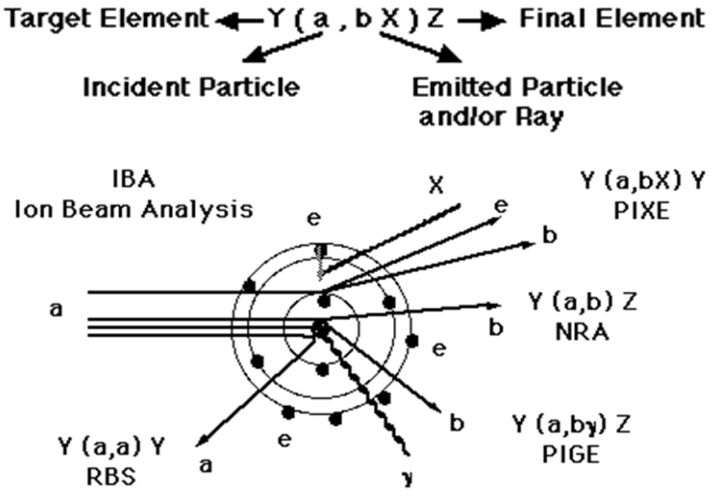


Fig. 1. IBA principle and techniques available at IFIN-HH.

The main IBA methods are:
- PIXE (Particle Induced X-ray Emission Analysis), which uses the atomic fluorescence (Y(a,bX)Y); the characteristic X-rays are analyzed and the method is easily adaptable for the detection of elements with Z>18 in various matrices [12];
- PIGE (Particle Induced Gamma-ray Emission Analysis) [13] based on (Y(a,bγ)Z) nuclear reactions, prompt-induced gamma rays are analyzed; PIGE is used for the detection of light elements as F or Na, which cannot be seen by PIXE;
- NRA is useful for the study of hydrogen impurities and depth profiling;
- RBS analysis is done with charged particles resulting from nuclear scattering (Y(a,a)Y). The efficiency of the method is high for Z>20 and for determining the concentration distribution of elements in depths of maximum 30 -50 μm in the sample.

The main limitation of RBS consists in the difficulties that occur in the investigation of a low atomic mass impurity within of a higher atomic mass substrate because the more weak impurity signal is superimposed on the strong substrate signal. NRA technique overcomes these difficulties.

For all IBA methods we should mention the relatively high cost of the beam time at the accelerator and the limited allocated beam-time per year for the same research team. Even if PAS technique is a non-invasive technique, some disadvantages must be pointed out like the long acquisition times (up to 24 hours per sample, due to the low activity of the radioactive source), and the necessity of using standards (being a relative method).

2. Experimental Part

2.1. Samples

Polymer nanofibers (polyurethane doped with silver nanoparticles) were prepared at "Alexandru Ioan Cuza" University-Iasi, using sol-gel technology based on electrochemical deposition of thin layers and electrospinning method [14, 15]. The samples thicknesses were in the range of 500–800 μm.

Carbon nanowalls were obtained at National Institute for Laser, Plasma and Radiation Physics-Magurele by using Radiofrequency Plasma-Beam-Enhanced Chemical Vapor Deposition [16].

InN thin films deposited on yttria-stabilized-zirconia (YSZ) substrate were obtained at National Institute for Optoelectronics-Magurele by reactive magnetron sputtering method [17].

2.2. Experimental Methods

2.2.1. Positron Annihilation Spectroscopy

The positron, immediately after generation, interacts with an electron, producing 2 gamma photons, with 0,511 MeV energy each ($e^+ + e^- \rightarrow 2\ \gamma$). PAS is useful in the study of single-vacancy-type or negatively charged cluster defects in condensed matter (Fig. 2). Passing through matter, positrons lose fast their energy (in picoseconds). The annihilation at eV energies is announced by the 511 keV photons, whose energy, momentum and time of emission can be precisely measured [18]. Since the annihilating electron possesses momentum, the relative angle between the two photons is slightly less than π, and the energies are Doppler-shifted. Due to their positive charge, positrons are strongly repelled by the positive ions in the solid. Free volumes like vacancies are attraction centers – they can capture positrons increasing as such their lifetime, and giving a measurable change in the annihilation

characteristics. Positron lifetime is measured with a laboratory setup as the time difference (Δt) between the 1.27 MeV (the prompt gamma ray of ^{22}Na source) and the annihilation 0.511 MeV gamma quanta. The technique using this phenomenon is PALS (Positron Annihilation Lifetime Spectroscopy). The measurement of the deviation angle (Θ) of the two 511 keV gammas, is used in ACAR (Angular Correlation of Annihilation Radiation) technique, and the measurement of the 511 keV line broadening (ΔE) is used in DBS (Doppler Broadening Spectroscopy). Electron momentum in propagation direction of 511 keV γ-ray leads to Doppler broadening of annihilation line which can be detected by conventional energy-dispersive spectrometers with HPGe detectors and standard nuclear electronics. These methods are schematically presented in Fig. 3.

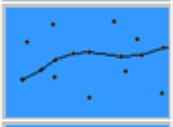
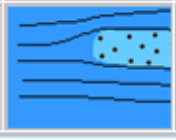
Type of defects		Dimension	Material
Vacancies		0.1 nm	Metals
Dislocations		1 nm - 10 μm	Metals
Holes		0.1 nm - 1 μm	Metals
Cavities		0.1 nm - 10 μm	Polymers

Fig. 2. Type of defects which can be detected with PAS.

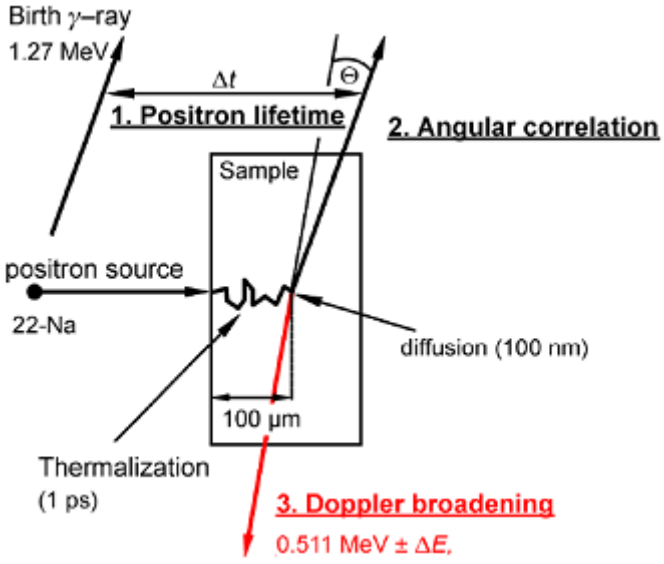


Fig. 3. Schematics of the PAS techniques: PALS, ACAR, DBS.

PAS proved to be a useful tool in finding out some information about the electron density and concentration of defects which can be found in one material. Moreover, the technique reported here - Coincidence Doppler Broadening Spectroscopy (CDBS) - has the advantage of using two HPGe detectors acquiring coincidence data, which makes the background radiation significantly reduced in

the high range of the longitudinal momentum electron-positron pair that annihilates. The annihilations corresponding to fast electrons (core electrons) make CDBS a suitable method to determine the concentration of free volume within the polymers [19].

The experimental device used in CDBS measurements is a classical one and it is shown schematically in Fig. 4 [20]. Two HPGe detectors working in "back-to-back" geometry are measuring the energy of each gamma quanta emitted in the annihilation process. Two identical samples form a sandwich with the positron source (^{22}Na) positioned between the two detectors. Detectors resolution is 1.2 keV for the 511 keV annihilation gamma ray which allows the study of Doppler broadening.

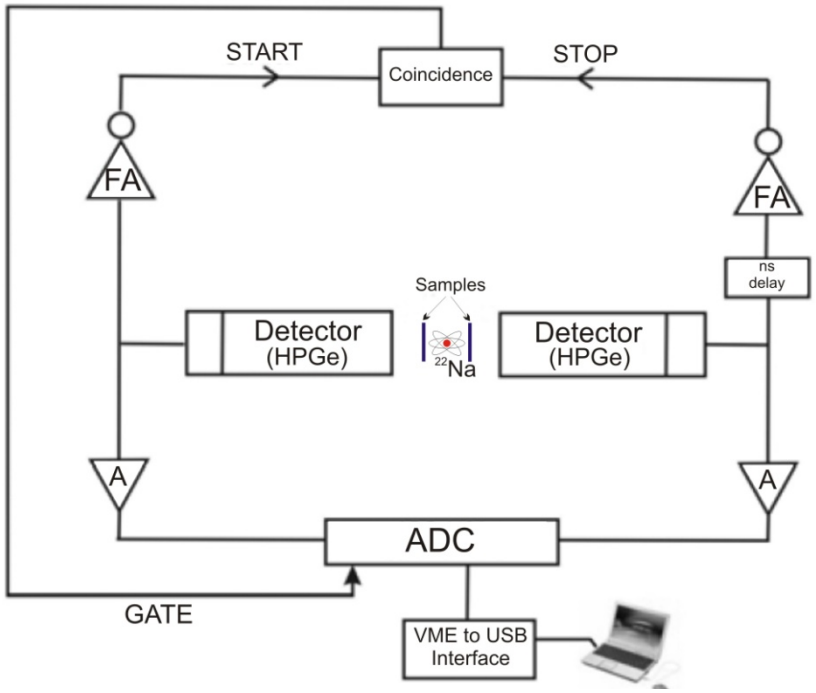


Fig. 4. Experimental set-up for CDBS measurements.

The acquisition system is based on a CAEN 32 Channel Multievent Peak Sensing ADC model V785. The ADC works in VME protocol and a dedicated electronic module (CAEN V1718) is used for interfacing it with a PC computer; the communication link is the USB 2.0 interface. The rest of the coincidence set-up is realized with the help of fast timing amplifiers (FA), two spectroscopic amplifiers (A, model Ortec 671), and a Time to Amplitude Converter (TAC) model Ortec 567 (Coincidence), which establish the coincidence window.

We studied the changes in nanocomposites structures due to gamma irradiation. Gamma-ray irradiations of polyurethane fibers were conducted at the SVST Co-60/B irradiator of the Multipurpose Irradiation Facility Center of IFIN-HH.

A set of three samples were analyzed, namely:

1. Polyurethane doped with silver nanoparticles, obtained by electrospining - this sample was used as a standard;
2. The same polyurethane membrane which was gamma irradiated - 50 kGy estimated absorbed dose;
3. The same polyurethane membrane which was gamma irradiated - 100 kGy estimated absorbed dose.

2.2.2. Nuclear Reaction Analysis

NRA consists in bombarding the sample which is being analyzed with a beam of heavy ions and then measuring the number of reactions with the hydrogen in the target. The reason of using nuclear reactions analysis is given by the natural way of measuring the depth profiles by measuring the nuclear reactions yields as a function of beam kinetic energy.

The three resonant reactions that have been used far more than others: those induced by ^{15}N [21], ^{19}F [22] and ^{7}Li [23]. Each of these reactions has its own advantages. For most applications, the ^{15}N reaction has the advantage of having the best combination of analytic characteristics (depth resolution and sensitivity). The ^{19}F reaction has the advantage that it can be conducted using natural F in the accelerator ion source. The ^{7}Li reaction has the advantage of allowing profiling to much greater depths in a sample than either of the above-mentioned reactions.

We used ^{19}F nuclear reaction technique for hydrogen profiling to gain information about the hydrogen distribution of the surface of CNWs. This technique is based on the ^{1}H (^{19}F, $\alpha\gamma$)^{16}O nuclear reaction.

A ^{19}F ion beam is focused on a hydrogen containing sample. If the energy of the ^{19}F ions is equal to the resonance energy (E_{res}=16.44 MeV) both nuclei fuse to the ^{20}Ne compound nucleus. This one decays immediately to an alpha particle and an excited ^{16}O isotope, whereas the oxygen emits a gamma quanta with a well-defined energy of E_γ = 6.13 MeV to reach its ground state. By measuring this photon one gets the proof for this nuclear reaction and thus a confirmation for hydrogen identification (Fig. 5) [24].

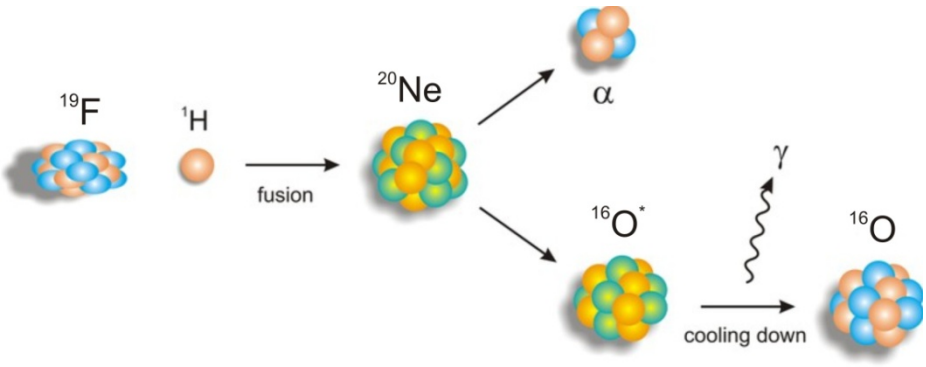


Fig. 5. Illustration of the nuclear reaction for hydrogen detection with ^{19}F ions.

A schematic representation of the ^{19}F resonant nuclear reaction profiling techniques is shown in Fig.6.

Fig. 6. Hydrogen depth profiling principle using (^{19}F, $\alpha\gamma$)^{16}O nuclear reaction.

This reaction has a large cross-section at the resonance energy (16.44 MeV in the laboratory frame), respectively 500 mb. If the ^{19}F beam energy of the bombarding beam it is at an energy above the resonant energy and the number of characteristic gamma rays produced in the target is counted using a detector one can obtain information about the concentration of hydrogen in that sample. When the sample is bombarded with ^{19}F ions above the resonant energy, there are negligible nuclear reactions with hydrogen from the sample surface because the energy is above the resonance. Because the ^{19}F ions penetrate the target, they give off energy and reach the resonance energy at a certain depth in the sample, where the cross-section is greater and the number of gammas increases considerably. The yield of gamma radiation is proportional to hydrogen concentration at this depth. In conclusion, by measuring the yield as a function of beam energy, the hydrogen concentration as function of depth is determined.

The detection of gamma rays was done with a Canberra HPGe detector with 50 % efficiency. For determining the hydrogen profile, a reference sample - Diamond Like Carbon (DLC) with known hydrogen concentration of 30 % in the bulk - was used. We made preliminary measurements on CNW samples.

2.2.3. Rutherford Backscattering Spectrometry

Analysis of surfaces is one of the most studied areas in both science and technology. RBS is one of the most powerful tools used for this purpose. This technique involves the use of an accelerated ion beam, usually H or He ions with energies of a 2 - 4.5 MeV. Samples are irradiated with a He beam. Most ions penetrate inside the sample up to a maximum depth of 10 μm, due to the loss of kinetic energy. During irradiation some ions collide with atoms of the samples and a part of them undergo elastic Coulomb scattering (Rutherford scattering) and another part of the ions would be backscattered.

The energy of backscattered ions from the target depends on the mass of atoms which have collided, allowing elemental analysis of the region near the surface to a depth of several microns, by measuring the energy spectrum of backscattered ions. Fig. 7 schematically illustrates an experimental model of arrangement and a spectrum obtained for a thin film composed of two elements, deposited on a substrate composed of elements with small Z mass number. Ions scattered by each element form one peak. Number of atoms in the film can be deduced from the peak's height and its width gives information on film thickness [25].

The main advantages of RBS are: the technique is nondestructive; it allows quantitative measurements; the depth resolution is about 10 nm, which can be improved by using special equipment; the time required for a measurement is typical short; it can detect concentrations of the order of 100 ppm for heavy elements; the detection limit decreases for lighter elements.

We analyzed one sample of InN deposited onto YSZ. The sample was irradiated with a He^{2+} ion beam with the energy of 4.5 MeV obtained from the 9 MV tandem accelerator of IFIN-HH. Backscattered particle detection was done by using an ORTEC detector BU 012-450-500 (Ultra ion-implanted detector), with resolution of 12 keV at 5.486 MeV of ^{241}Am line for α particles. The detector active area is 450 mm^2. Signal Processing from ORTEC detector was performed using electronic devices (preamplifier, Tennelec amplifier TC 243). After obtaining one-dimensional amplitude spectra from the detector, information retrieval was performed off-line using the SIMNRA software [26]. Film thickness value was calculated using 3.18×10^{22} atoms cm^{-3} (6.81 g/cm^3) for the volume density of InN.

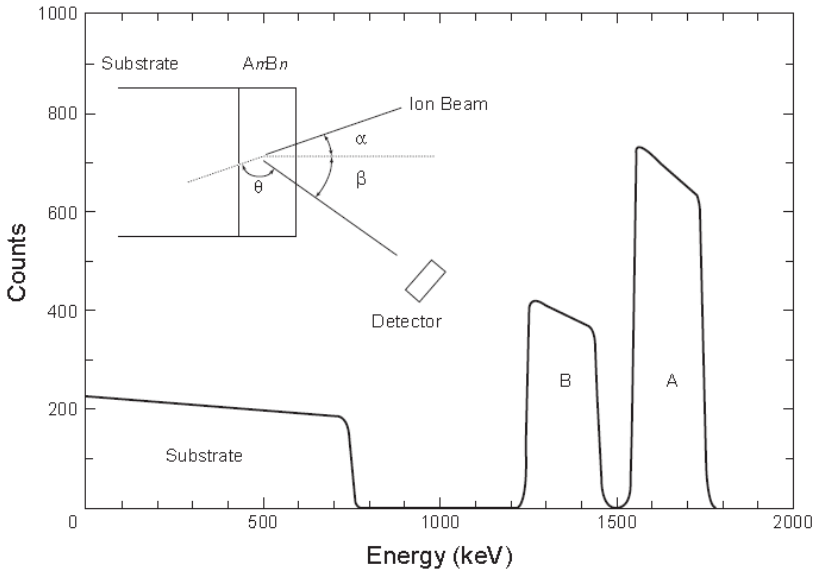


Fig. 7. RBS spectrum for a thin film composed of two elements (A_mB_n) deposited on a substrate made of low Z elements.

3. Results

3.1. Positron Annihilation Spectroscopy

Fig. 8 presents the comparison between the momentum density distributions for the two samples of irradiated polyurethane. The longitudinal momentum of the electron-positron pair is plotted versus the longitudinal momentum density.

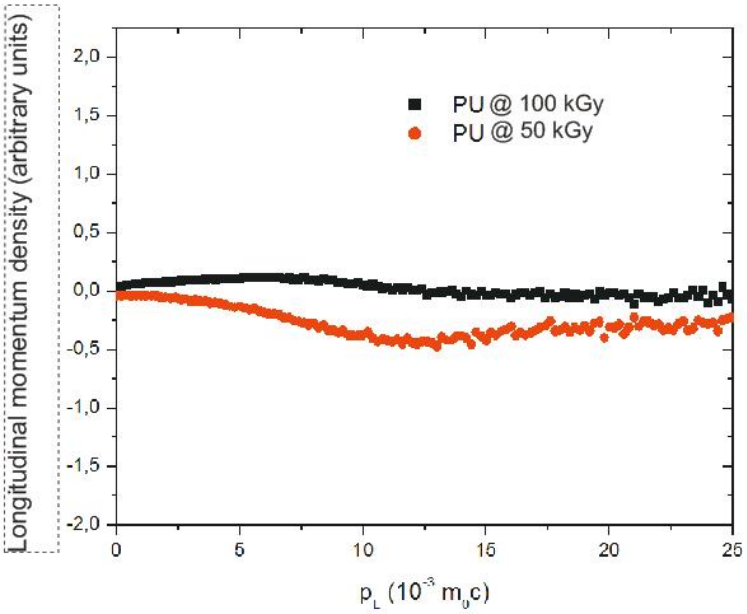


Fig. 8. Comparison between the momentum density distributions for the two samples of irradiated polyurethane.

Doses of 15-20 kGy are expected to produce changes in the distribution of the electron momentum. Possible interpretation of the modifications is that a certain fraction of the positrons injected into the

sample form Ps atoms (typically 20%), which can be in one of the two orbital states: ortho-Ps with long life time and para-Ps with a shorter life time. The rest of positrons not forming Ps within the material are gaps in the structure where the polymer will eventually annihilate with an electron of a neighbor atom. Also o-Ps annihilate with high probability with the valence electrons as it undergoes a "pick-off" process.

We conclude that Doppler broadening annihilation photon energy is similar for the case of free positron annihilation and o-Ps annihilation, because both are types of annihilation with valence electrons. Regarding p-Ps things are different, because the positron annihilates with the electron that is forming the Ps atom, which will lead to a narrowing of the momentum distribution. According to literature data, the p-Ps contribution should become significant for doses greater than 15 kGy [27].

For higher doses (100 kGy) we expect a decrease of the density of free volumes, but the volume of the defect becomes larger in size which means a larger uncertainty in position and a smaller one in momentum. CDBS using the W parameter analysis for polyurethane samples irradiated at different doses turned out to be an accurate method to describe the modifications induced by gamma ray irradiation. It was shown that silver nanostructures facilitate the increase of the annihilations with slow electrons from the conduction band. We proved that PAS can be a useful tool because it serves as a probe for the defects concentration modifications due to different external factors [28].

3.2. Nuclear Reaction Analysis

The presence of hydrogen in some materials has dramatic effects on their properties. Many of these phenomena are not properly understood because of the difficulties in applying a technique to measure the hydrogen concentration as a function of depth into the material. NRA offers the possibility of measuring depth profiles. Hydrogen is being used in the fabrication of CNW but its role in CNW growth is only partly understood [29].

Fig. 9 shows the 6.13 MeV gamma rays resulting from the (^{1}H (^{19}F, αγ)^{16}O) nuclear reaction acquired with an HPGe detector. The incident ion energy was 16.5 MeV $^{19}F^{4+}$, corresponding to a depth of 40 nm in the CNW. It can be seen that the region of high energy gamma rays is not affected by the background radiation.

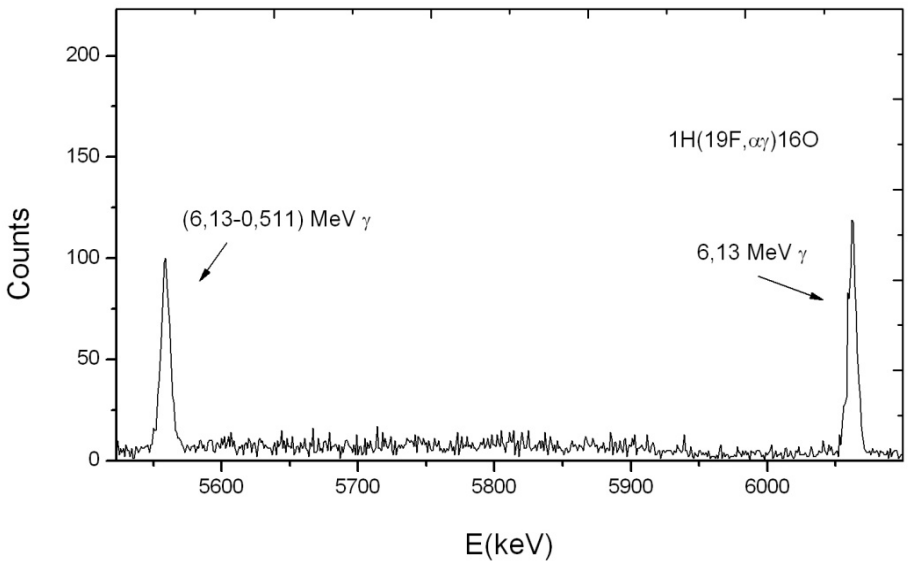


Fig. 9. NRA spectrum of CNW sample.

Fig. 10 presents the hydrogen profile for the CNW [30]. The level of hydrogen is high for the first 50 nm of the material, and then it decreases more than 50 %. From these preliminary results we see that hydrogen profiling is possible, and this is very useful in the design of structures of various morphologies [31].

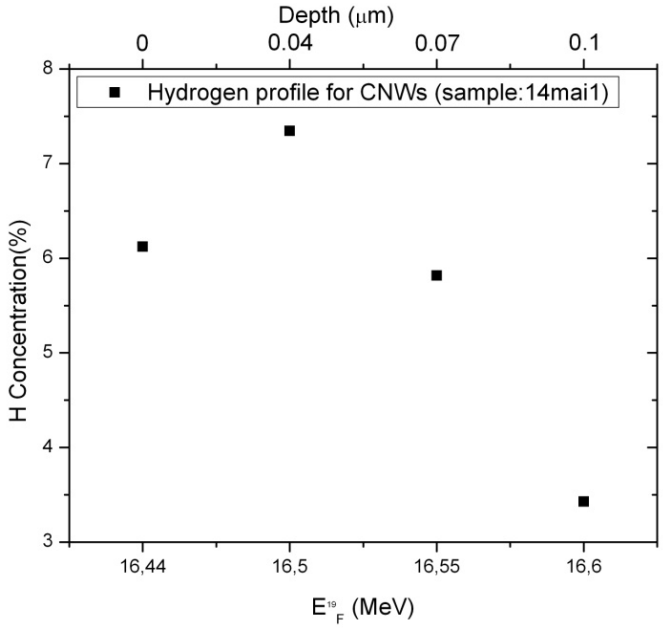


Fig. 10. Hydrogen profiling in CNW sample using NRA (^{1}H (^{19}F, αγ)^{16}O).

3.3. Rutherford Backscattering Spectrometry

Fig. 11 shows the simulation of a two-layer structure consisting of an InN layer with no oxygen content at surface (360 nm of $In_{0.38}N_{0.62}$) and a nitrogen-rich interfacial oxide layer (75 nm of $In_{0.2}O_{0.6}N_{0.2}$). This indicates that InN exhibits the ability to extract oxygen from a variety of materials. It was shown that the high levels of oxygen uptake from the substrate provide motivation for the use of buffer layers when growing InN layers on substrates of sapphire (Al_2O_3) [32]. This is valid also for YSZ which is a mixture of ZrO_2 and Y_2O_3. Obtaining ideal growth conditions for the semiconductor of InN is still a challenge for the fabricants. Our results regarding the composition of the thin films are very important because this affects the material properties, so one can obtain quality materials, by adjusting the growth conditions parameters (substrate temperature during deposition, electric power applied to discharge, etc).

4. Conclusions

Three nuclear techniques: PAS, RBS and NRA were used to analyze three types of nanomaterials: polymer/metal particle matrix type nanocompounds, carbonic nanostructures and InN thin films. PAS provided insights into vacancy defects of polymer nanofibers, RBS provided information regarding the InN thin film thickness and the stoichiometry, while NRA preliminary analysis showed the possibility of detecting and measuring hydrogen profile in CNWs. The application of these nuclear techniques can provide specific information contributing to the control of the processing of nanostructures in order to obtain the necessary purity and the desired properties.

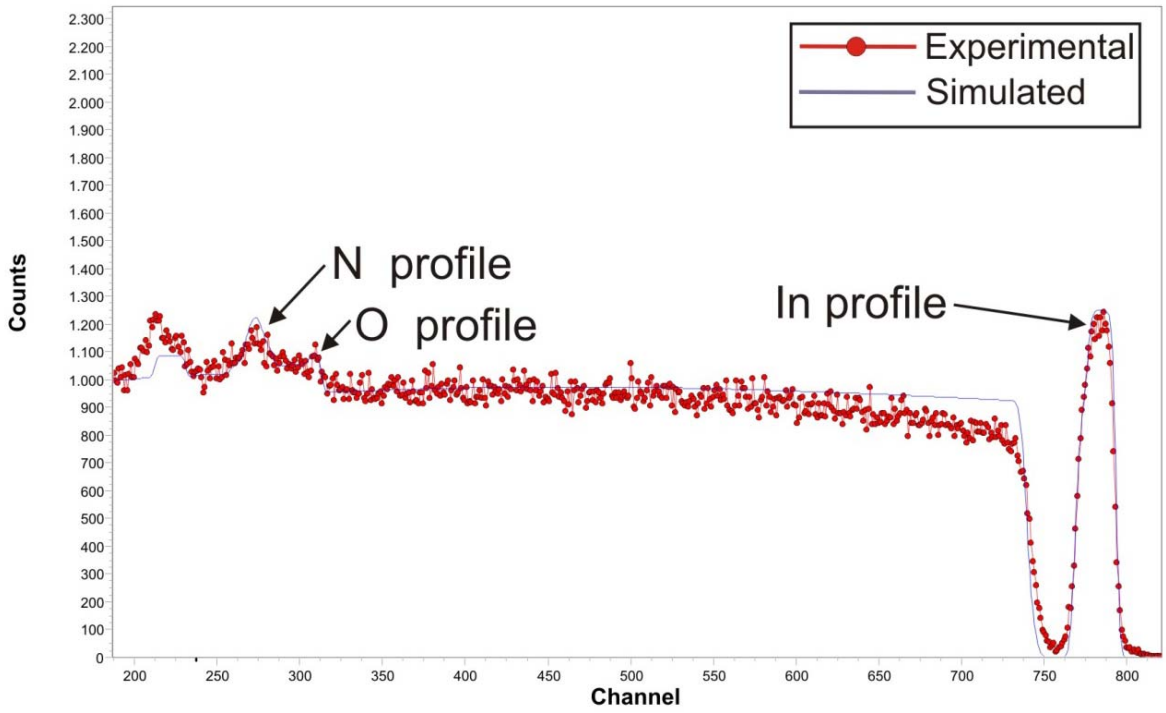


Fig. 11. RBS spectrum of an InN/YSZ sample.

For the future works we would like to apply other IBA methods available at IFIN-HH, such as PIXE and PIGE, for measuring the presence of any impurities in the materials. We also intend to develop a slow positron beam facility which will make possible the depth profiling analysis of defects.

Experimental observation and theoretical modeling of radiation-induced defects and domain evolution in nanostructured silicon carbide ceramics or other nanostructured materials are research subjects that our team suggests to the partners from COINAPO project.

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Sensors & Transducers Journal

Guide for Contributors

Aims and Scope

Sensors & Transducers Journal (ISSN 1726-5479) provides an advanced forum for the science and technology of physical, chemical sensors and biosensors. It publishes state-of-the-art reviews, regular research and application specific papers, short notes, letters to Editor and sensors related books reviews as well as academic, practical and commercial information of interest to its readership. Because of it is a peer reviewed international journal, papers rapidly published in *Sensors & Transducers Journal* will receive a very high publicity. The journal is published monthly as twelve issues per year by International Frequency Sensor Association (IFSA). In additional, some special sponsored and conference issues published annually. *Sensors & Transducers Journal* is indexed and abstracted very quickly by Chemical Abstracts, IndexCopernicus Journals Master List, Open J-Gate, Google Scholar, etc. Since 2011 the journal is covered and indexed (including a Scopus, Embase, Engineering Village and Reaxys) in Elsevier products.

Topics Covered

Contributions are invited on all aspects of research, development and application of the science and technology of sensors, transducers and sensor instrumentations. Topics include, but are not restricted to:

- Physical, chemical and biosensors;
- Digital, frequency, period, duty-cycle, time interval, PWM, pulse number output sensors and transducers;
- Theory, principles, effects, design, standardization and modeling;
- Smart sensors and systems;
- Sensor instrumentation;
- Virtual instruments;
- Sensors interfaces, buses and networks;
- Signal processing;
- Frequency (period, duty-cycle)-to-digital converters, ADC;
- Technologies and materials;
- Nanosensors;
- Microsystems;
- Applications.

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