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Application of nuclear analytical spectroscopies and ion beams to the study of nanomaterials: cooperative projects between Vinatom and JINR (Dubna)

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Abstract: Due to the rapid scientific and technological development in the last decades, basic research in solid state physics, chemistry and material science has focused on objects and phenomena more and more confined in dimensions and time-scale, and well visible for the general publicity by introducing the terms “nanophysics, nanoscience, nanomaterials, etc.”, often featured in the media. Researchers therefore keep searching for better and better investigative techniques. Various nuclear analytical spectroscopies, such as Positron annihilation lifetime (PAL), Doppler broadening of positron annihilation energy (DB), Electron momentum distribution (EMD), Slow positron beam (SPB), Neutron diffractions (ND), Rutherford backscattering (RBS), etc., have proved themselves as useful tools for microscopic analysis of different material’s structure ranging from angstrom (Å) to nanometer (nm) scales. Besides, ion beams generated from accelerators (electron, ^{1}H, ^{2}He, ^{40}Ar, ^{86}Kr, ^{109}Ag, ^{123}Xe, ^{184}W, etc.) have also become very effective tools for modifying the micro structure of nanomaterials. These methods have been intensively utilized by our group at Vinatom with external collaborations from JINR (Dubna) in order to study the in-depth structure of different nanomaterials. This report introduces our research and collaborative activities, facilities and some recent highlighted results.

Keywords: *Positron annihilation spectroscopies, slow positron beam, ion beams, nanophysics, nanoscience, nanomaterials.*

I. INTRODUCTION

Controlling the reciprocal influences between porosity, crystalline structure, lattice defect, surface property, molecular transport and reaction of functional materials are keys for technological developments. Nanostructured materials, such as optical, photocatalytic, cracking catalytic, etc. samples, contain a complex matrix of pore, defect, and/or doping structure, which plays a crucial role to their applications in environmental treatment, industrial catalysis, energy storage, etc. In the study of these nanostructured materials, the

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applicable limitation of conventional methods at the atomic scale leads an urgent need of finding alternative methods, which are highly sensitive in this size scale [1]. During the last decades, nuclear analytical spectroscopies, such as Positron annihilation lifetime (PAL), Doppler broadening of positron annihilation energy (DB), Electron momentum distribution (EMD), Slow positron beam (SPB), Neutron diffraction (ND), Rutherford backscattering (RBS), etc., have been known as useful tools for the microscopic analysis of structures of nanomaterials ranging from angstrom (Å) to nanometer (nm) scales [2-4]. Recently, ion beams generated from accelerators or implanters (electron, ^{1}H, ^{2}He, ^{40}Ar, ^{86}Kr, ^{109}Ag, ^{123}Xe, ^{184}W, etc.) have been also applied as very effective tools for modifying the microstructure of nanomaterials [5-7]. Such nuclear techniques have gradually become irreplaceable tools in the field of material science in the next few decades.

At Vinatom, the research of nanostructured materials has been carried out since 2005 and accelerated recently through several cooperations between the Center for Nuclear Technology (CNT, Vinatom), Institute of Fundamental and Applied Sciences (IFAS, Duy Tan University), Joint Institute of Nuclear Research (JINR, Dubna, Russia), and Henryk Niewodniczanski Institute of Nuclear Physics (HNINP, Poland) [8-25]. By combining the PAL and DB techniques with the Val de Graaff 5 MV accelerator, we have investigated the effect of proton irradiation on the structure of 4A zeolite [12]. This research provided important information on the structural phase transition and the destructive dose of this zeolite under the influence of irradiation [13]. In a later study, which based also on PALS combined with XRD and SEM techniques, we have found, for the first time, the simultaneous existence of defects and mesopores in the ZSM-5 zeolite [9]. This finding revealed important information on the synthesis and modification of zeolite structure in the petrochemical catalysis. Following this research topic, a project (grant number DTCB:14/19 TTHN, term 2019-2020) supported by Ministry of Science and Technology (MOST) was performed by our group and successfully completed in 2020 [23]. In this project, we had obtained very promising results related to an ability to improve the catalytic cracking efficiency of the ZSM-5 zeolite irradiated by the 10 MeV electron beam at the fluence of 10^{13} – 10^{14} e/cm^2. For instance, the catalytic cracking efficiency of the irradiated ZSM-5 was found to increase by 12% for the C3 product (propylene) and by 10% for the total C3/C4 products (LPG) as compared to the unirradiated sample [23]. These findings have been reported at the *International conference on application of nuclear spectroscopies and related techniques in materials science* (Dec. 30, 2019), which was co-organized by CNT and IFAS with participation of international experts from JINR and HNINP. Based on the success of the first project, MOST has decided to support us another project (grant number DTCB: 15/21/TTHN, term 2021-2022), which is a second phase of the first project. In this second phase, we are going to deeply investigate the catalytic cracking efficiency together with the stability of our irradiated ZSM-5 materials. Moreover, we have recently extended our research areas, by collaborating with a group at University of Science and Technology of Hanoi (USTH), to nanomaterials used for photocatalytic applications. This project, which has been submitted and gone through the preliminarily review by Vinatom, aims to modify the structure of the $BiVO_4$ nano-photocatalytic membranes using implantations with ^{1}H, ^{2}He,

^{40}Ar, ^{86}Kr, ^{109}Ag, ^{123}Xe, and ^{184}W ions in order to enhance their photocatalytic performance [24]. In addition, the crystallization of MFI structured zeolites at high temperature will be studied in another project, which has been submitted to NAFOSTED. The aim of this project is to shorten the synthetic time and improve the production scale of MFI structured zeolites. To carry out these projects, in addition to the available equipment at CNT (PAL, DB, and EMD), several modern and advanced nuclear analytical equipment such as MT-25 electron accelerator, IC-100 cyclotron, SPB, RBS, HRTEM, etc., at JINR and HNINP will be utilized with the strong support from the membership of Vietnam Government at JINR [25] as well as the recent cooperation signed between Vinatom and JINR [26].

The goal of this report is to introduce our research group, facilities, and collaborative activities with domestic and international institutions, in particular JINR, Dubna. Some recent highlighted results obtained via those activities will be also reported.

II. LABORATORIES AND COLLABORATIVE STUDIES BETWEEN VINATOM AND JRNR

Fig. 1a illustrates the positron annihilation laboratory at CNT. The laboratory consists of PAL (the time resolution ~ 220 ps), DB (the energy resolution ~ 1.5 keV at the 570 keV peak of ^{207}Bi radioactive source) and EMD spectroscopies. These spectroscopies have used to study different nanomaterials such as carbon nanotubes [9,11], zeolites [10,12,13], adsorption materials [14,21], thermoelectric materials [16], hybrid nanocomposites [19], and optical materials [17]. Fig. 1b shows a SPB accelerator using Ne as a moderator to generate the SPB from a ^{22}Na radioactive source with low energy (from 1 to 40 keV) at the Dzhelepov Laboratory of Nuclear Problems (JINR) [15]. This is a special nuclear equipment used for researches of structural defects in thin films, membranes, and materials in the nuclear reactor [27]. We have used this equipment, with the agreement of JINR scientists, to investigate the structure of solar cell, thermoelectric, and zeolite materials [15].

Also in JINR, there are two advanced facilities, RBS (Fig. 1c) and ND (Fig. 1d), managed by the Frank Laboratory of Neutron Physics (FLNP). The RBS used a helium beam from the van de Graaff accelerator, whereas the ND utilized the thermal neutron beam from the IBR-2 high-flux pulsed 450 nuclear reactors. We have used the RBS to evaluate the depth distribution of different elements in the near surface layers of the W-Si solar cell material, while the ND was employed to study the nucleation and crystallization pathways of MnO_2 nanomaterial with alpha structure at room and annealed temperatures. Based on the ND measurements, we have obtained novel results on the evolution of crystalline structure in MnO_2 nanomaterial which have not been observed so far. Results of this study have been completed and a manuscript has been submitted to an internationally high-impact journal. The heavy-ion (U-400 and IC-100) accelerators (Fig. 1e) were equipped at the Flerov Laboratory of Nuclear Reactions in JINR. This equipment will be used in combination with SPB and RBS to modify the structures of zeolite [25] and $BiVO_4$ nano-photocatalytic membranes using ^{1}H, ^{2}He, ^{40}Ar, ^{86}Kr, ^{109}Ag, ^{123}Xe, and ^{184}W ions [28].

Figure 1. a) Positron annihilation laboratory at CNT (Vinatom). From b) to e) Nuclear analytical facilities at JINR (Dubna): slow positron beam accelerator (b), Rutherford back scattering spectroscopy (c), neutron sources from IBR-2 reactor used for neutron diffraction measurement (d), and ion beam accelerators (e). Figures b-e) are taken from [26].

III. SOME RECENT HIGHLIGHT RESULTS

A. A hybrid model for determining the pore size from the *o*-Ps lifetime

In a recent study [18], we have proposed a model, called the hybrid (HYB) model, which can be used to estimate the free-volume size of porous materials based on the experimental ortho-positronium (*o*-Ps) lifetime data. The HYB model was derived by combining the semi-classical (SE) physics model (for the pore size $R > 1$ nm) with the conventional quantum model by Tao-Eldrup (TE) (limited to $R < 1$ nm only) [29]. By connecting two regions of small ($R < 1$ nm) and large pores ($R > 1$ nm) and by introducing the *o*-Ps diffusion probability parameter (D), our HYB model has provided a better agreement with the experimental *o*-Ps lifetime data than the most up-to-date rectangular TE (RTE) [30] and Tokyo models [31] (Fig. 2a). In particular, our model is able to, for the first time, describe reasonably well the defined experimental *o*-Ps lifetimes in the pores with both spherical and channel geometries (Fig. 2b). This is, therefore, considered as the most updated model for determining the pore size in the universal range of 0.2 − 400 nm for most of porous materials.

B. Removal of oxygen vacancy, vacancy cluster and phase interactions in a PVA/ZnO nanocomposite

In a later study [19], the influence of post-annealed process at 400 °C on the removal of oxygen vacancies due to their diffusion to the near-surface region of ZnO nanocrystals was detected by using our PAL measurements (Fig. 3a). In this study, we have found that the vacancy cluster size of ZnO nanoparticles (NPs) contains approximately 6 Zircon and 6 Oxygen atoms (V_{12}), while the size of nano-voids calculated from the o-Ps lifetimes using our Hybrid model [18] was about 0.5 – 0.57 nm. This finding is very interesting because this is the first experimental measurement that indicated a simultaneous existence of weak and strong interactions between ZnO NPs and polymer chains of PVA. These interactions, which are controlled by a competition between the Van der Waals force and the number of defects in ZnO NPs, could affect the thermal conductivity of ZnO/PVA nanocomposites (Fig. 3b). We also found a largely broad distribution of pore radius from 0.07 – 0.26 nm for the mixed shape NPs (Z2 sample) and narrower ones of 0.17 and 0.185 nm for the spherical-like NPs (Z1 sample) and hexagonal rod-like NPs (Z3 sample), respectively (Fig. 3c). As a result, we have proposed in this study a relationship between the morphology, intrinsic defects, and interfacial characteristics of nanofillers and resistive switching characteristics of nanocomposites based memory devices as illustrated in Fig. 3d [19].

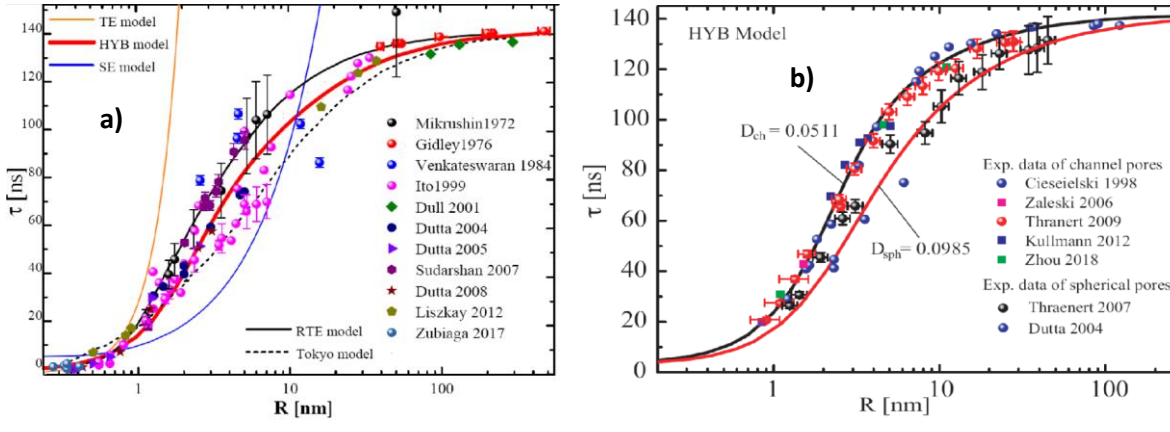

Figure 2. a) The *o*-Ps lifetimes obtained within the TE, SE, Tokyo, RTE, and HYB models versus the experimental data for different porous materials. b) The *o*-Ps lifetimes obtained within the HYB models versus experimental data for the materials with spherical and channel pores. Adapted from [18].

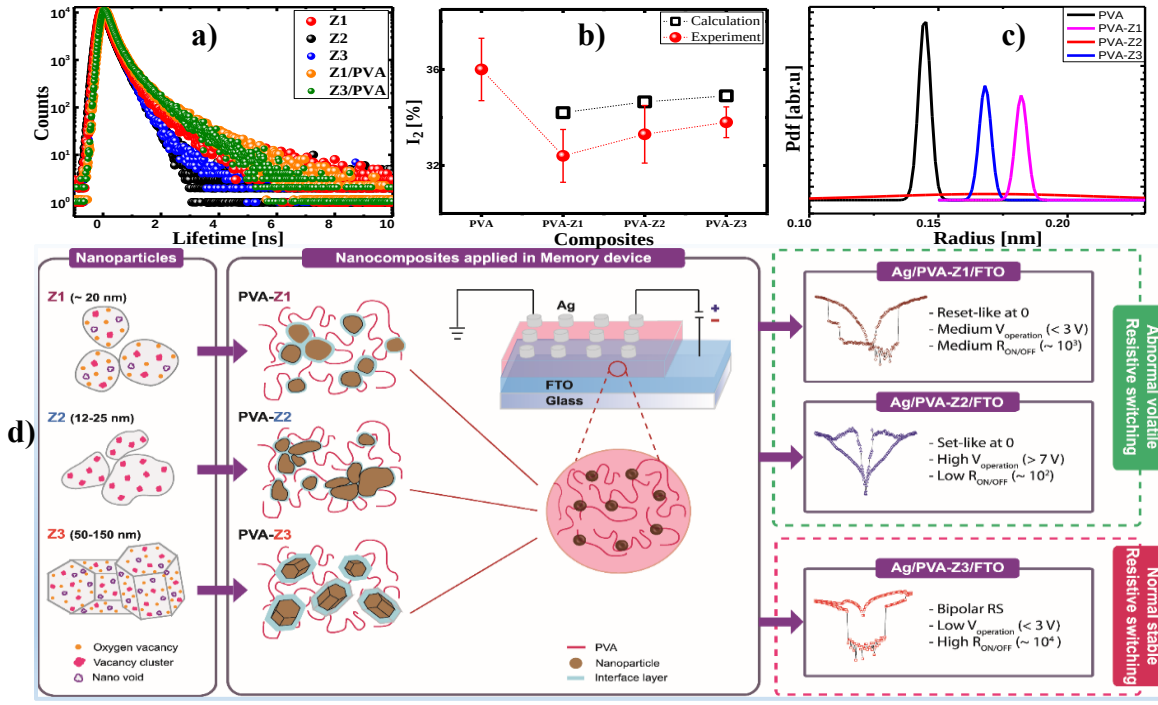

Figure 3. a) Positron lifetime spectra. b) Positron annihilation intensity in the interfaces of nancomposites. c) Pore size distribution in the studied samples. d) Schematic relationship between the morphologies, intrinsic defects, and interfacial characteristics of nanofillers and resistive switching characteristics of memory-device-based nanocomposites. Z1, Z2, and Z3 denote the ZnO samples having spherical-like, mixed shape, and hexagonal rod-like nanoparticles (NPs), respectively. Adapted from [19].

C. Vacancy, di-vacancy and micropore in the K_2SiF_6 doped mn^{4+} material for micro white light-emitting diodes

In another study [17], the positron annihilation spectroscopies have been applied to the K_2SiF_6 doped Mn^{4+} ($K_2SiF_6:Mn^{4+}$) nanocrystals, which have been widely used for the micro white light-emitting diodes (micro-WLEDs). The samples were synthesized via a feasible emulsification route [17]. The host K_2SiF_6 and $K_2SiF_6:Mn^{4+}$ (KSFM) samples prepared without using surfactants are, respectively, denoted as KSF and KSFM-WS, whereas, those prepared in the presence of Tween80 (Tw) surfactant and oleic acid (OA) are abbreviated as KSFM-TwOA. By using PAL measurements (Fig. 4a), we have found that there existed both Si^{4+} mono-vacancy and K^+ di-vacancy in the structure of KSF. The K^+ di-vacancy was formed due to the absence of two K^+ ions and the Mn^{4+} ions may prefer to occupy the K^+ di-vacancy site other than the Si^{4+} one in the KSFM-WS and KSFM-TwOA samples. In addition, the KSFM-WS exhibited no porous structure, while KSFM-TwOA contained some porous structures (size of ~0.62 nm) as indicated by PAL (τ_3 ~ 2.27 ns) in Fig. 4a and HRTEM image in Fig. 4b. Due to this porous structure, the bulk density of KSFM-TwOA material can be reduced, leading to an improvement of the electron mobility and photon emission. This porous structure also additionally contributed to the increase in the quantum yield of KSFM-TwOA sample. In particular, the porous KSFM-TwOA exhibited the low-thermal quenching, deep red emission and high emission quantum yield ~95% by 455 nm light excitation [17]. Hence, our PAL analysis has provided a deep understanding on the role of defects and porous structures of $K_2SiF_6:Mn^{4+}$ nanocrystals on the light emission quantum yield of micro-WLEDs (Fig. 4c).

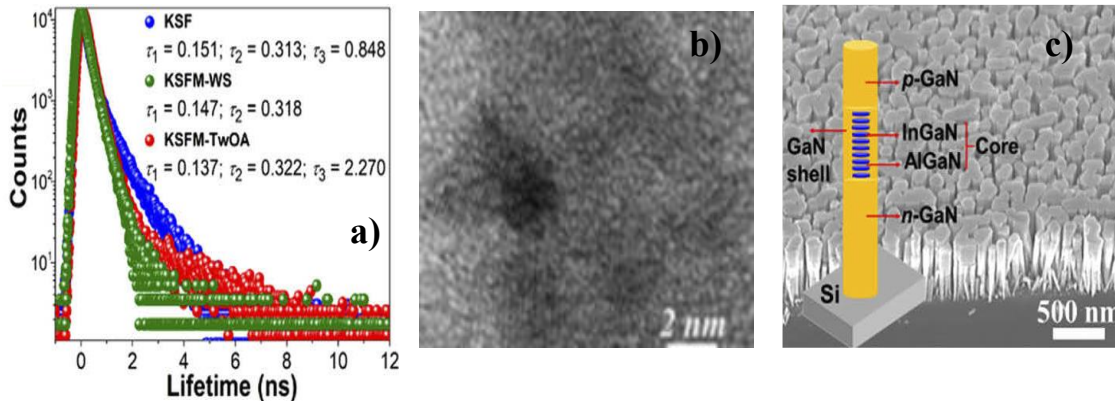

Figure 4. a) PAL spectra of the KSF, KSFM-WS and KSFM-TwOA nanocrystals. b) HRTEM images of KSFM-TwOA. c) A 45° tilted SEM image of the nanowires on silicon wafer. Inserted image shows the schematic structure of an InGaN/AlGaN core-shell single nanowire LED. Adapted from [17].

D. Nucleation and crystallization growth of α-MnO_2 nanomaterial under annealed temperatures

In a latest study, the nuclear analytical spectroscopies (ND, PAL, DB and EMD) combined with conventional characterization methods (XRD, FT-IR, BET-BHJ, and TPR-H_2) and theoretical *ab initio* calculations have been applied, for the first time, to systematically investigate the crystallization pathways, morphologies, and structural defects of α-MnO_2 nanomaterial synthesized under different annealed temperatures. Here, the ND were carried out in Dubna, while PAL, DB, and EMD were performed first in CNT and then re-examined in Dubna. Both ND and PAL analyses have indicated that the α-MnO_2 nucleation can be occurred even at room temperature, while the crystallization gradually developed with increasing annealed temperatures and become α-MnO_2 nanorods at above 500 ^oC. In addition, by combining the PAL analysis with theoretical *ab initio* calculations, we found the existence of either H^+ or K^+ ions in the tunnels [1x1] of α-MnO_2. Furthermore, the combined DB and EMD measurements suggested the simultaneous presence of Mn and O vacancies in α-MnO_2 crystals at low temperatures, while these O vacancies were removed at high temperatures, leaving only the Mn vacancies in the samples.

IV. CONCLUSION

This report has provided an overview of our research group on the application of nuclear analytical spectroscopies to the study of material structure at the nanoscales. Some collaborative activities between our group and domestic and international institutions and laboratories are introduced and highlighted results are also given. Those collaborations, in particular the cooperative works with JINR, Dubna, have proved to be very important for our past and present studies in the field of materials science. These results have significantly enhanced the role and position of Vietnamese scientists as well as Vinatom researchers at JINR. Many open questions on the in-depth structure of nanomaterials used for diverse applications are still needed to be explored. We, therefore, call for continuous supports from Vinatom and MOST to this research direction.

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