Nucl. Sci. and Tech., Vol.14, No. 3 (2024), pp. 41-50

Nuclear Science and Technology

Journal homepage: http://jnst.vn/index.php/nst

Preliminary analysis of structural defects in thin BiVO4 layer using the slow-positron-beam based facilities at JINR, Dubna

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Abstract: The present paper introduces the slow-positron beam system at the Joint Institute for Nuclear Research (JINR). Preliminarily studies on thin films using the combined analyses of the variable energy Doppler broadening (VEDB) and variable-energy electron-momentum distribution (VEEMD) measurements at JINR are also reported. These studies provide a unique tool for the in-depth investigations of the structural defects in nanomaterial as thin films, from material's surface to various depths (in the range from a few nm ups to 1 μm). Application of that method to BiVO4 thin film implanted with P+ ions (200 keV) reveals that the structural defects in the thin film achieve the highest concentration in the depth range of 40 – 200 nm (VEDB analysis), whereas the introduction of P+ ions into the thin film should reduce the positron annihilation probability with high-momentum core electrons. These results open the possibility to use advanced analytical techniques for in-depth study of nanomaterial in JINR, performed, in particular, by Vietnamese scientists.

Keywords: *Slow-positron beam, variable-energy Doppler broadening (VEDB), variable-energy electron-momentum distribution (VEEMD), JINR, BiVO4 thin film, positron annihilation spectroscopy (PAS).*

https://doi.org/10.53747/nst.v14i3.470

Received 08 May 2024, accepted 22 October 2024

I. INTRODUCTION

The analysis of structural defects in materials is one of the major but challenging topics in materials science due to its essential role in improving the materials properties and related applications [1]. Among the known-methods, positron annihilation spectroscopy (PAS), including positron annihilation lifetime (PAL), Doppler broadening (DB), angular correlation of annihilation radiation (ACAR) and electron momentum distribution (EMD), has been recognized as a unique tool for characterizing those structural defects at the atomic scale [2-6]. This arises from the fact that, PAS can provide the in-depth information on the structural defects in materials such as the defect size, type and concentration of defects with a remarkable sensitivity of about 10-7 [7].

Positron is an antiparticle of electron, which can be generated from the beta β+ decays of some radioisotopes. After entering the materials, the positrons quickly lose their kinetic energy to be in thermal equilibrium with the material environment [8]. After that thermalization, positrons diffuse in the material and annihilate with electrons in different structures, including perfect lattice, atomic-scale defects (monovacancy, vacancy cluster), nano-pores or large voids, etc. Each positron-electron annihilation generates two gamma photons of equal energy (511 keV) but moving in opposite directions [6]. In the structural defects, nano-pores, and large voids, under a convenient condition, a positron also can combine with an electron to form a so-called positronium (Ps), which has two states depending on the spins of positron and electron, namely para-positronium (p-Ps) with a very short lifetime of ~0.125 ns (antiparallel spins) and ortho-positronium (o-Ps) with a maximum lifetime in vacuum of ~142 ns (parallel spins). The o-Ps lifetime depends on the structural defects and the size of pores and voids through the pick-off annihilation and can be measured by PAL experiment [9]. In addition, the re-arrangement of the electron configurations at the defective and/or doping sites can lead to the change of total electron momentum in the annihilation energy distribution of positron-electron pairs [10]. Such changes can be practically investigated with DB, ACAR and EMD analyses [11]. Details on the experimental PAS measurements can be found in our recent publications [12-17].

Although PAS has been widely employed in materials science, conventional PAS measurements using 22Na source with the maximum positron energy of 544 keV is not effective for the thin-film materials with layered structure ranging from nanometers to micrometers because of the too high penetration of high-energy positron in materials. Thus, investigating the distribution of defects in the thin-film materials requires the control of the incident energy of positron beam in material layers [18]. In such the case, the use of low-energy positron beams with various energy levels can provide an effective tool for studying thethin-film materials. This idea can be practically implemented by slowing down positrons emitted from the 22Na source to a near-zero energy level and then accelerating them to various energy levels to implant into the layers of thin-film material [19]. The slow-positron beam (SPB) with a wide energy range from several tens eV to several tens of keV [20] is suitable for the depth investigations of structural defects from the surface to the depth of a few nm to 1 µm [18].

Due to the rapid technological development during the last four decades, SPB has been intensively applied in the studies of thin-film, layered-structure and irradiated bulk materials [18,19,21–25], in particular at Dzhelepov Laboratory of Nuclear Problems (DLNP), the Joint Institute for Nuclear Research (JINR), where the variable-energy Doppler broadening (VEDB) spectroscopy using SPB has been utilized for investigating the structural defects in ion-implanted materials. For example, Horodek et al used VEDB measurements at DLNP to investigate the defected layers of the bulk iron and gold irradiated with the heavy swift-ion irradiation of Xe, Kr [26]. In this study, the positron diffusion lengths were determined to evaluate the damage level of investigated materials. In a recent study by Siemek et al, the swift heavy 167 MeV Xe26+ ion beam together with VEDB analysis have been used to study the effect of irradiated doses as well as the role of grain size on the formation of structural defects in titanium [27]. Those studies have provided a better understanding on the structural defects under different irradiation conditions. However, the combination of VEDB analysis with the various-energy electron-momentum distribution (VEEMD) one, which is necessary for comprehensive studies of thin films and nanomaterials, has still been not yet developed at JINR.

In this paper, we introduce the SPB facility at DLNP, JINR. Detail descriptions of this equipment and its operation are presented. In addition, the combined VEDB and VEEMD experiments and analyses recently developed and utilized recently by Vietnamese scientists are also reported.

II. THE SLOW-POSITRON BEAM FACILITY AT JINR

A. Positron injection

Since 2000, the low-energy positron toroidal accumulator (LEPTA) at DLNP had been develope [28]. LEPTA contains a compact-positron storage ring equipped with an electron cooling system and a positron injector [29,30]. The primary aim of LEPTA was to generate the direct flux of o-Ps for the foundation physical studies [28]. The low-energy positron beam developed later by LEPTA has become an ideal tool for PAS measurements at JINR, now known as the PAS facility. The production of the positron beam is based on the following steps. In the first step, positrons are emitted from the β+ decay of the 22Na source with an activity of ~30 mCi. emitted positrons are then directed to pass through a system of frozen neon gases, which acts as a moderator to slow down positrons to a thermal energy through the elastic scatterings (Figure 1). A cryogenic source, designed for Doppler measurements, is included in a special holder with the liquid helium and neon gas streams. The 22Na source is placed in a vacuum chamber maintained at a base pressure of 4 × 10-9 Torr. The liquid helium is used to ensure a low temperature of about 7 K for the system. This way the positron flux intensity can be controlled at ~3 × 105 e+/s, while the average positron energy remains at ~1.5 eV. The slow and fast positrons are then separated using a system of 100 Gs magnetic field [30].

In the second step, after passing through the moderator, a monoenergetic-positron beam of energy ~50 eV is guided to the transport channel. A small diameter diaphragm has been placed in the transport channel to prevent the neon gas (from the source area) from entering the positron trap. This process increases the positron survival time, which depends on the composition of the residual gas before being implanted in materials. In addition, the transport channel is also used for separating the energetic positrons from the moderator (Figure 2) [30]. The main parameters of the SPB system are shown in **Table 1**.

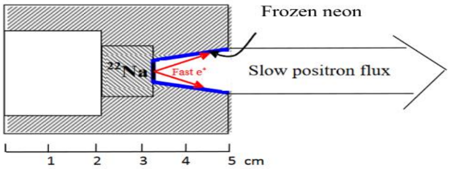

Fig. 1. Illustration of the moderation of fast positrons (fast e+) to the slow positrons by using frozen neon gas at DLNP [31].

The vacuum chamber of the positron source and the transport channel are vertically offset from each other. A special superposition of the longitudinal electric magnet field and an additional transverse magnetic field allows only the slowed positrons to enter the trap (Fig. 2). Therefore, the slowed positrons will have "slalom" trajectory that only low-energy positrons can travel to the sample holder. The sample holder is designed to be moveable and on the potential of 0 – 30 kV, allowing it to carry simultaneously several samples for measurements [30].

Table 1. Parameters of the variable-energy positron beam [20]

Feature	Value
Radioactivity of ^{22}Na	30 mCi
Magnetic field	100 Gs
Vacuum condition	4×10^{-9} Torr
Intensity	3×10^{5} e^{+}/s
Energy range	50 eV÷30 keV
Diameter of the beam	3 mm

*1 Gauss (Gs) = 10-4 Tesla (T)

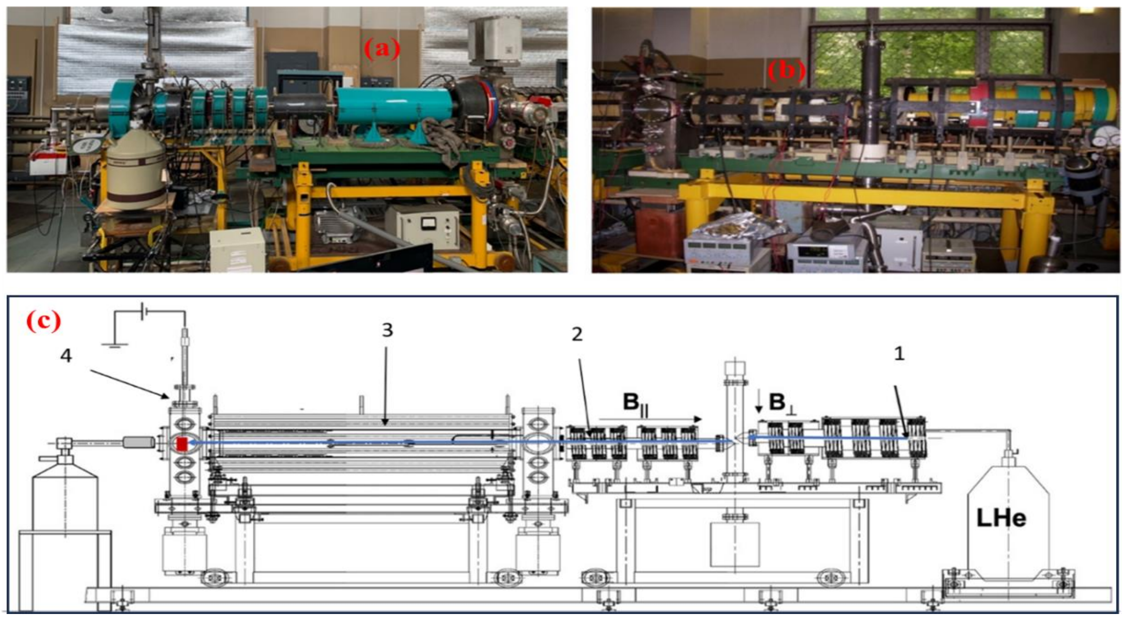

Fig. 2. The slow-positron beam system at DLNP. (a): The accelerating and controlling systems for the positron beam energy before entering the studied sample. (b): The system containing the 22Na source, moderator, and transport channel. (c): The detail components of the slow-positron beam system, including positron source (1), transport channel (2) positron trap (3) and sample holder (4) [20]

B. VEDB and VEEMD measurements and analyses

The VEDB system requires a high-energy-resolution detector to measure the energy of annihilation photons (~511 keV). At PAS facility, a high-purity germanium (HPGe) detector (ORTEC) having the energy solution of 1.2 keV at the 511 keV annihilation peak has been set up and combined with the SPB system for measurements. In the VEDB measurement, based on the Doppler shift of the pair annihilation energy originated from the positron annihilation with electrons having different momenta, two characteristic S (shape) and W (wing) parameters are often used to evaluate the change of materials structure. For a certain DB spectrum obtained from the VEDB measurements (Figure 3), S parameter, which is determined from the ratio of the annihilation events of positron with low-momentum valence electrons (A) and the total pair annihilation events (A+B+C), is sensitive to the defect concentration [21]. W parameter, which is determined by the ratio of the positron annihilation events with high-momentum core electrons (C) and the total pair annihilation events, reflects the change of the chemical environment at the annihilation sites [10]. Moreover, in a series of materials doped under different conditions, the linear correlation plot between S and W parameters for all studied samples can provide important information on the possible presence of similar defective types in this sample series [23].

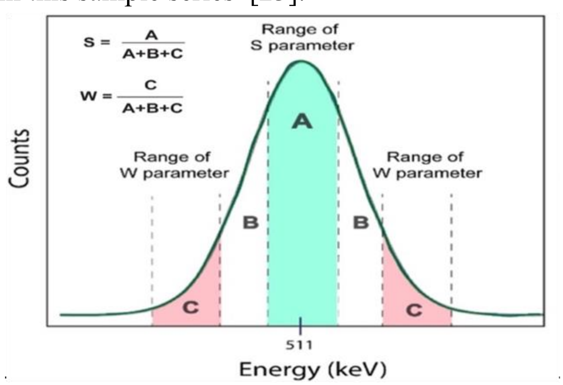

Fig. 3. Illustration of a DB spectrum and data analysis for two characteristic S and W parameters obtained from the VEDB measurements [6].

The low-background EMD analysis has been successfully performed at the positron annihilation laboratory, the Center for Nuclear Technology, VINATOM. This analysis, in combination with PAL and DB measurements, has provided the in-deep information of the structural defects and the doping site in several nanomaterials [13-17]. However, similar analysis using SPB at JINR has not yet been performed. Thus, we have used the VEDB data to construct the one-dimensional (1D) VEEMD spectra by using the energy-conservation relation, namely 2□E = c × PL, where □E is the Doppler shift, PL is the longitudinal electron momentum and c is the speed of light [24]. The VEEMD analysis is then combined with the VEDB one to investigate the defect types as well as the changes of chemical environment in each layer of thin-film materials. Such a combination of VEDB and VEEMD analyses certainly provides a comprehensive study on the change of structural defects at the atomic and nano-scales induced by doping process in thin-film materials.

B. Slow positron experiments for BiVO4 thin-film

The VEDB and VEEMD experiments and analyses have been performed for a bismuth vanadate thin film (BiVO4) having a thickness of ~800 nm. This material was synthesized by the electric-chemical deposition technique on a FTO target before being implanted with P+ ions with energy of 200 keV and irradiation dose of 1013 ions/cm2 at room temperature.

1. Calculation of positron implantation mean depth

To investigate the structural defects in the P+ implanted BiVO4, we first calculated the positron implantation depth in this material. The positron implantation profiles were known to be described by a Gaussian, which can be expressed by the so-called Makhovian profile [32,33]:

$$p(z) = \frac{mz^{m-1}}{z_0^m}\exp\left[-\left(\frac{z}{z_0}\right)^m\right] \quad (1)$$

where m and z_0 are, respectively, the shape and penetration parameters, whose values are adjusted to fit the experimental data. Here, z0 depends on the implanted positron energy (E) as

$$z_0 = \frac{AE^n}{\rho\Gamma(1+\frac{1}{m})} \quad (2)$$

where A and n are constants or Makhovian parameters; ρ is the material density; and Γ is the gamma function [34]. The mean depth of implanted positron (z_mean) can be estimated using the common formula [40,41]

z_mean=z_0 Γ(1+1/m)=A/ρ E^n .   (3)

To calculate z0 and zmean for BiVO4, the following parameters, A = 4.0 µg/cm2, n = 1.62, ρ = 6.25 g/cm3 and m = 2, are chosen [18]. Calculated results are shown Figure 4. It is seen in this figure that the mean depth increases with increasing positron energy. The mean depths calculated using equations (2) and (3) are not much different, especially at low-positron energies (below about 8 keV). Hence, equation (3) is used to calculate the mean positron depth [32,33].

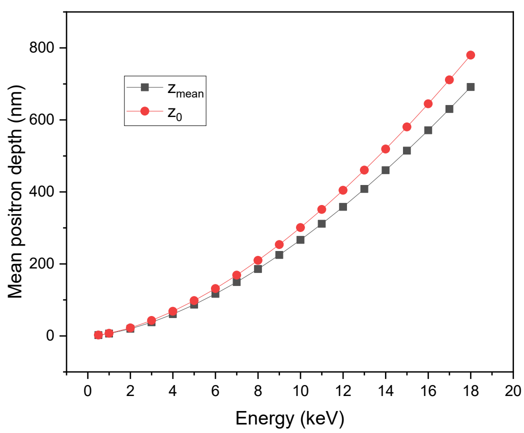

Fig. 4. The mean depths versus the implanted positron energy calculated using equations (2) and (3) for BiVO4 thin film.

2. Results of VEDB and VEEMD analyses

For the VEDB analysis, the energy windows used to calculate the values of S and W parameters were selected at 510.8 keV to 511.8 keV and 514.5 keV to 517.8 keV, respectively. In order to calculate the S and W parameter, the package SP program was used [35]. Obtained results are presented in Figure 5.

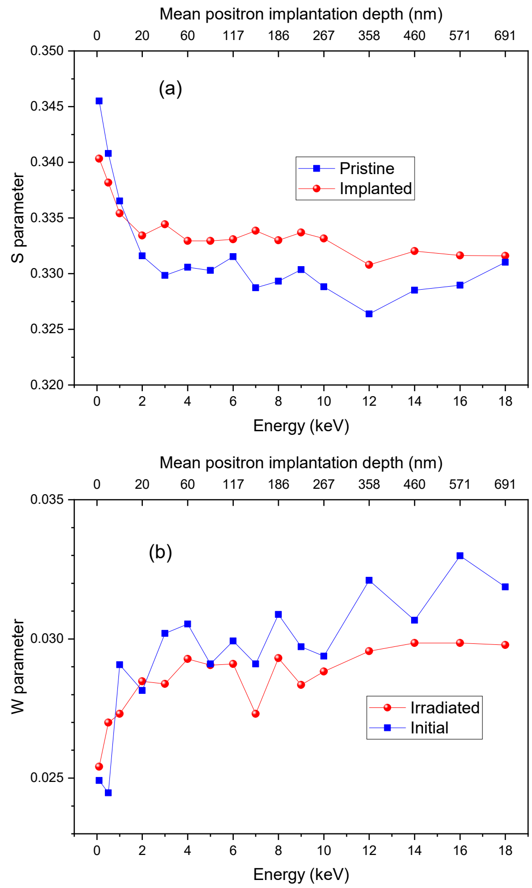

Fig. 5. The values of S (a) and W (b) parameters as functions of positron energy (E) and mean positron depth obtained within the VEDB analysis for pristine and implanted BiVO4 thin film.

From Figure 5a, one can see a clear difference between the S parameters obtained for un-irradiated (pristine) and irradiated (P+ implanted) samples. The deduction of S parameter obtained in the depth range of 0 – 30 nm for both samples is attributed to the back diffusion of positrons to the surfaces of BiVO4 [36]. In this depth range, the S value of the pristine is higher than that of the implanted sample, implying that the surface of P+ implanted BiVO4 had been modified to reduce the valence electron density or the defect concentration. In a deeper depth range (30 – 700 nm), the S value of the pristine becomes lower than that of the implanted sample. This confirms the formation of structural defects across the moving path of the implanted P+ ions. The highest defect concentration is found in the depth range of 40 – 200 nm for the P+ implanted BiVO4 as indicated in Figure 5a.

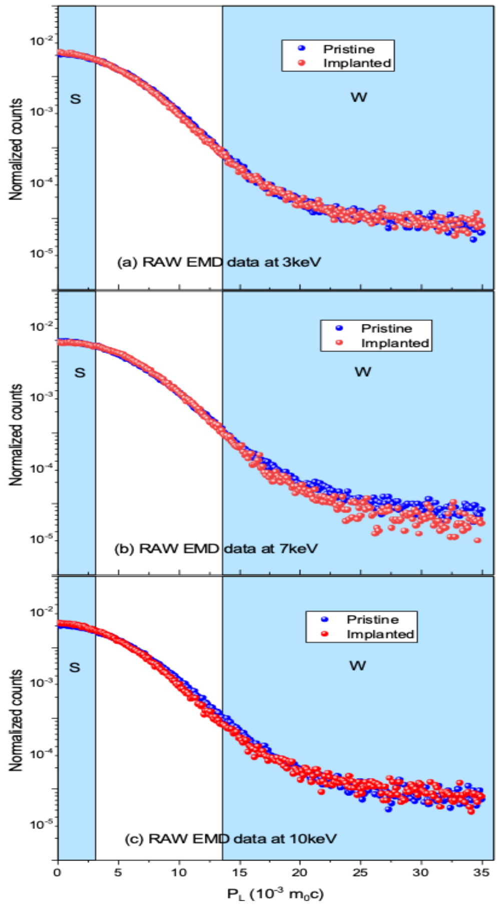

Fig. 6. The VEEMD data obtained from the VEDB measurements for the pristine and implanted BiVO4 thin films at different positron energies of 3 keV (a), 7 keV (b) and 10 keV (c).

Figure 5b depicts the variation of W parameter with the positron energy and mean depth, which associates to the change of chemical environment in the implanted sample. The W parameter of the implanted sample is lower than the pristine one, indicating the reduction of positron annihilation probability with high-momentum core electron in this implanted sample. This is reasonable because the insert of P+ ions in the thin-film structure should cause the increase of the valence electron density [10,37]. The increase of W parameter in the depth range of 180 – 750 nm for implanted sample indicates that implanted P+ ions should mainly locate in this material layer.

To insight into the change of electron configurations caused by the ion implantation, the VEEMD analysis is conducted and results are shown in Figure 6. In this figure, the shape of the EMD curves for implanted sample at 3, 7 and 10 keV is narrower than that for the pristine one. This result explains the low-positron annihilation probability with high-momentum electrons in the implanted sample. However, those preliminary VEEMD results are insufficient to provide the in-depth information on the defect types due to the lack of a defect-free reference, which is conventionally required in EMD analysis. Such measurements and analyses will be performed and reported in our forthcoming study.

III. CONCLUSION

This work introduces the slow-positron beam system at the PAS facility of JINR. The preliminary studies using the combined VEDB and VEEMD experiments and analyses are also reported for a P+ implanted BiVO4 thin film. This combination provides the in-depth investigations on the structural defects from the surface to the depths of few nm to 1 μm in the implanted sample. For instance, the structural defects in studied sample achieve the highest concentration in the depth of 40 – 200 nm as indicated by the VEDB analysis. In addition, the insert of P+ ions into the thin-film structure should lower the positron annihilation probability with the high-momentum core electrons as indicated by the VEEM analysis. However, due to the lack of defect-free reference sample, deeper VEEMD analysis for the structural defects has still been limited. Further experiments and analyses are on-going. This work opens an advanced research direction for in-depth analysis of structural defects in thin films and other nanomaterials at JINR, which can be mastered by Vietnamese scientists.

ACKNOWLEDGEMENT

This work was supported by Ministry of Science and Technology, Vietnam under grant number: ĐTCB.12/2023/TTHN. The authors would like to sincerely thank the Vietnam Atomic Energy Institute and Duy Tan University for very strong supports to this work.

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