Form 24

DEVELOPMENT OF THE EXPERIMENTAL TECHNIQUES AND APPLIED RESEARCHES WITH SLOW MONOCHROMATIC POSITRON BEAMS

PROJECT SYMBOLIC NAME PROJECT-PAS

Theme 1126

DLNP: E. V. Akhmanova, A. G. Kobets, I. N. Meshkov, O. S. Orlov, A. Yu. Rudakov, K. Siemek, A. A. Sidorin, L. V. Soboleva, V. I. Hilinov, S. L. Yakovenko

LHEP JINR: V. V. Kobets

LNP JINR: M. Kulik

LNR JINR: V. A. Skuratov

Northern (Arctic) Federal University named after M.V. Lomonosov, Arkhangelsk: M. K. Eseev

Institute of Nuclear Physics PAN, Kraków, Poland: P. Horodek, E. Druzek, K. Skovron

Tomsk Polytechnic University: R. S. Laptev, A. M. Lider

Nuclear Technology Center, Ho Chi Minh City, Vietnam: Lu An Tuen

PROJECT GROUP LEADERS: A. G. Kobets, K. Siemek

PROJECT SCIENTIFIC LEADER I. N. Meshkov

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DATE OF THE LABORATORY'S STC **21.04.2020**

DATE OF PRESENTATION OF THE PHYSICAL BASIS AT THE LABORATORY WORKSHOP **20.03.2020**

Form 25

PROJECT APPROVAL SHEET

DEVELOPMENT OF THE EXPERIMENTAL TECHNIQUES AND APPLIED RESEARCHES WITH SLOW MONOCHROMATIC POSITRON BEAMS

PROJECT SYMBOLIC NAME: PAS PROJECT

Theme

PROJECT SUPERVISORS: A. G. Kobets, K. Siemek

PROJECT SCIENTIFIC SUPERVISOR I. N. Meshkov

APPROVED BY THE DIRECTOR OF JINR	_____ (Signature)	«____»_____20 (Date)
AGREED: JINR VICE-DIRECTOR	_____	«____»_____20
CHIEF SCIENTIFIC SECRETARY	_____	«____»_____20
CHIEF ENGINEER OF JINR	_____	«____»_____20
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LABORATORY DIRECTOR	_____	«____»_____20
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PROJECT SUPERVISORS	_____	«____»_____20
	_____	«____»_____20
PROJECT SCIENTIFIC SUPERVISOR	_____	«____»_____20
APPROVED THE PAC IN THE FIELD OF CONDENCED MATTER PHYSICS	_____	«____»_____20

Content

Abstract

To study the structure of various materials and defects that occur under various physical influences (aging, external loads, radiation exposure, etc.), high-precision methods are required that can distinguish inhomogeneities of the crystal structure at the nanometer level. One of these methods is positron annihilation spectroscopy (PAS). This method is sensitive to detecting various (so-called "open-volume") defects ranging in size from 0.1 to 1 nm with a minimum concentration of up to 10^{-7} cm^{-3}. The PAS method has 4 orders of magnitude better spatial resolution compared to a transmission electron microscope.

Applied research in the field of solid state by PAS methods and the development of experimental techniques using these methods are the goal of the project. To study defects in materials, the Doppler broadening of the annihilation line (DBAL) method is used, implemented both on an autonomous source and on a flow of slow monochromatic positrons. The (DBAL) spectrometer is made according to the standard scheme. The Positron Annihilation Lifetime Spectroscopy (PALS) method implemented on an stand-alone ^{22}Na source is also used. The PALS method on the flow of slow monochromatic positrons is being developed for the development of the experimental base. The team proposed an original version of this method, based on the formation of an ordered flow of slow positrons [1].

Introduction

There are three main classes of materials that have so far been studied by PAS. Historically, the first use of PAS was aimed at researching metals and alloys. Then the PAS methods began to investigate semiconductors. For these two areas, positron plays the role of a probe. The third area of application of PAS, which is currently developing intensively both experimentally and theoretically, is related to the study of polymers and other high-molecular materials containing large areas free of substance.

Applied research methods include, for example, the study of fatigue damage to materials that must bear a wide range of mechanical, radiation or thermal loads during service. Long-term aging and external loads, including irradiation, lead to the evolution of small defects in the material. This process is the driving force behind embrittlement. For example, radiation embrittlement is critical for high-pressure vessels in nuclear reactors. The use of common methods to study such problems (e.g. high-resolution electron microscopy) is not always appropriate or does not give the necessary accuracy. The various methods of positron annihilation spectroscopy, in these cases, may be more accurate and provide additional information about the processes taking place in the material [2].

The use of PAS methods to detect defects in semiconductors, where atomic-sized defects are electrically active and play an important role in the electrical and optical properties of materials, has three advantages. First, PAS has a high sensitivity to vacancy defects, making their identification simple. Second, positron spectroscopy is strictly proven, and electro-positron annihilation can be calculated theoretically. Third, positron annihilation can be applied to any material, regardless of its structure and conductivity [3].

Nanoporous materials have a wide range of industrial applications, due to the extremely high surface area containing areas that can be used (e.g. in catalysis) to capture and separate molecules. Since the functional use of nanoporous materials is determined by their porosity (including the size and concentration of pores), the porous structure is specifically selected (formed) depending on their purpose, and numerous technologies such as molecular absorption (using N_2CO_2 and other molecules) and mercury metric have been developed as characteristic agents. However, most of these approaches have limitations. Imaging techniques, such as transmission electron microscopy, can detect information about the distribution of nanopores, but they require special sample preparation that can alter the distribution of pores. Thus, the methods available today to determine the distribution of pores in nanoengineering require significant improvement, while the PAS method can characterize nano-porosity, especially when the pores are closed and therefore unavailable, for example, for gas absorption methods [4].

Thus, research by PAS methods is a necessary tool for applied research in the field of solid-body physics, and their improvement will expand the range of tasks to be studied.

Research Status

Between 2017 and 2020. work on the project included research by PAS methods in solid-body physics and the development of an experimental base. PALS spectroscopy was realized by the standard scheme from the stand-alone source 22Na (Fig. 1). This isotope, 3,3 ps after positron emission, emits a gamma quant of 1274 keV energy, which is used as a spectrometer start signal to measure the lifetime. The stop signal is the registration of a gamma quant of 511 keV energy. The time difference between both signals gives the positron lifetime value in the sample. The positron source is covered with two thin foils (about 5 micrometers) and placed between two identical samples. This

"sandwich" is placed in front of two counters (scintillators BaF_2 and photo multipliers, Fig. 1) The PALS spectrometer uses Hamamatsu's PEM. A lifetime spectrometer APV8702 and a computer are used to analyze the signals for the match. Typically, several radiation measurement modules are required to analyze time, such as differential CFDs (Constant fraction discriminator), latency line, TAC (Time amplitude converter), SMA (Channel Multi Analyzer), etc. In APV8702, all of them are integrated. The result is a spectrum of positron lifetime, and subsequent analysis allows us to estimate the lifetime of the positron in the sample.

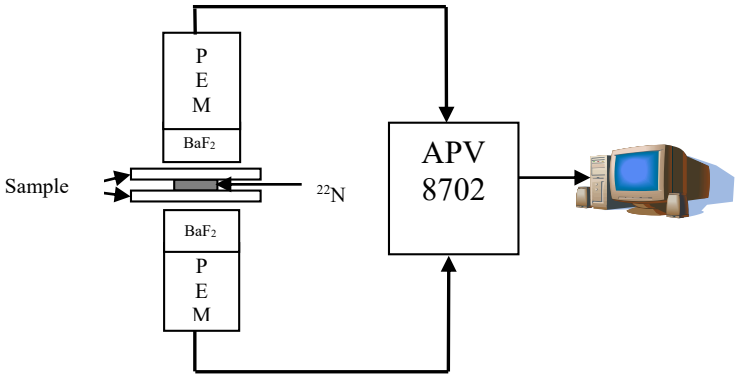

Fig.1. Scheme of a standard PALS spectrometer.

The DBAL spectrometer is made according to the standard scheme (Fig. 2). This spectrometer can work with both a stand-alone ^{22}Na source and a flow of monochromatic slow positrons. It consists of a high-voltage source, a HpGe detector, a preamplifier, a multichannel analyzer and a computer. The Ortec's HpGe detector is used to record the gamma quanta, which are born in positron annihilation. The detector has the following parameters: the relative registration efficiency of 1.33 MeV γ-photon (according to IEC 60973 standard) is 30%; photon energy resolution (full width at half magnitude) at 511 keV less than 1.25 keV; the detector energy range from 40 keV to 10 MeV. The detector is powered by a power supply fabricated according the NIM standard by the same company [5,6].

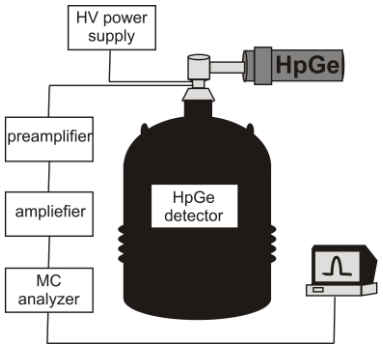

Fig. 2. Scheme of the DUBAL spectrometer.

Cryogenic source of slow monochromatic positrons (CSSMP) allows us to create a low-energy positrons beam satisfying the requirements of PAS methods. An additional possibility of adjusting the energy of positrons appears when the sample is "suspended" under the negative potential accelerating the positrons before they bombard the sample, which allows the monoenergetic positrons of the given energy to penetrate the sample up to chosen depth. Slowing down and monochromatizing the flow of positrons opens the possibility to trace changes in the structure of the material in thin layers.

The isotope ^{22}Na is used as a source of positrons. The half-life of the isotope is 2.6 years making it suitable for long-term experiments (Table 1). This isotope gives a relatively high output of positrons of 90.5%. In addition, the technology of its manufacture

has been well developed, which ultimately affects the price of the isotope [7].

Table 1. Characteristics of positron-active radionuclides used in researches

Isotope	Half-life	Maximum positrons energy, MeV	Percentage of decays, %
^{22}Na	2.603 *year*	0.545	90.50
^{44}Ti	47.3 *year*	1.467	98
^{48}V	15.974 *day*	0.696	50.7
^{55}Co	17.54 *h*	1.50	60
^{56}Co	78.76 *day*	0.421	1.03
^{57}Ni	36 *h*	0.85	50.0
^{58}Co	0.79 *day*	0.475	15.05
^{64}Cu	12.703 *h*	0.652	17.9
^{65}Zn	243.9 *day*	0.329	1.45
^{68}Ge	288 *day*	1.899	87.52
^{90}Nb	14.6 *h*	1.50	54

High-energy positrons emitted at decay of isotope ^{22}Na penetrate through the solid state moderator, where they lose their energy on ionization and (below the ionization threshold) through the creation of a pair of electron-hole and the exciton excitation of phonons slowing down to thermal speeds. Solid neon has been chosen as an efficient moderator. In 2015-2016 we have developed, manufactured and commissioned a new cryogenic source. It uses Sumitomo helium cryocooler to cool the emitter and surrounding heat screen. This source allows us to receive a flow of slow positrons at least 10^6 e^+/sec.

In 2018-2019 a specialized channel of slow positrons (SCSP, Fig. 3) has been fabricated and mounted. The magnetic system of the channel consists of a bending solenoid 1, longitudinal magnetic field coils 2, solenoid system for positron ordering 3, and transverse magnetic field coils correcting the positron trajectory (not shown in figure. 3).

The channel vacuum chamber is placed inside the magnetic system that ends with an experimental station 4. The holder with the samples is placed inside the experimental station through side flange 7 and attached to the remotely controlled manipulator 5. Up to seven samples can be placed at one vacuum volume opening. The experimental station has its own pumping system, which is separated from the main vacuum volume of the channel by vacuum valve 6.

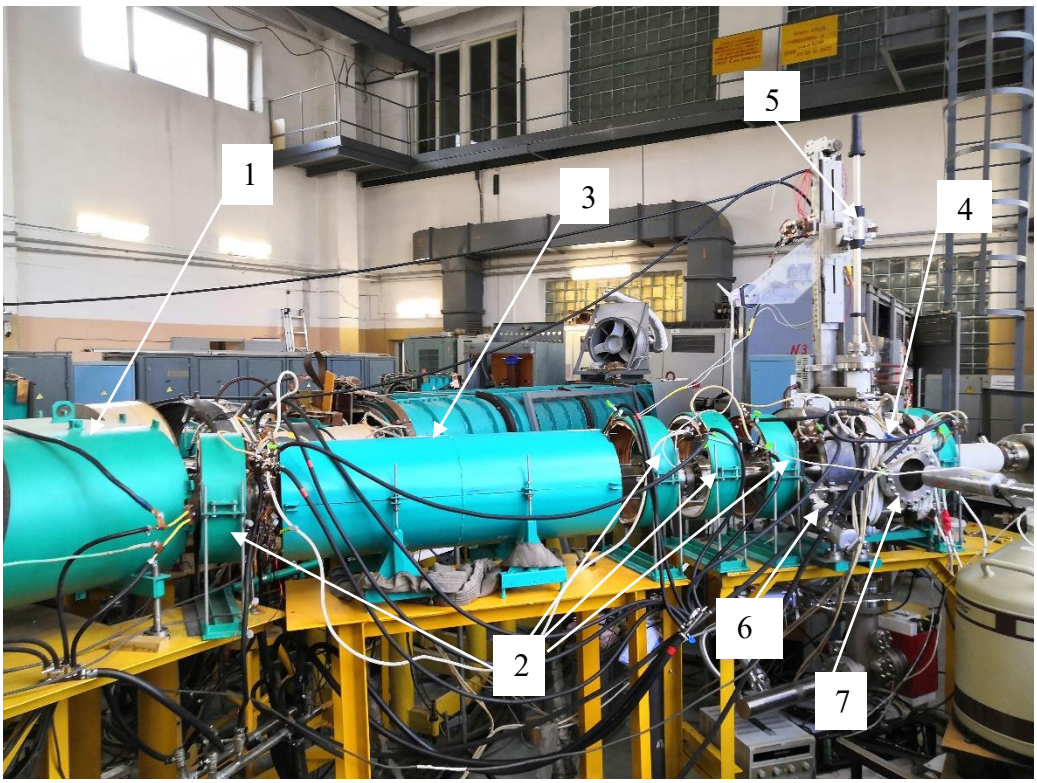

Fig. 3. Specialized channel of slow positrons.

The positron flow ordering system is part of the created of positron lifetime spectrometer (PALS) [1]. Between 2017 and 2018. an original way of ordering positrons has been proposed and its implementation has begun (Figure 4). The monochromatic positron beam to be formed passes the section of high-frequency (radio frequency - U_{RF}) voltage of a special form. Then the positrons accelerate in a static electric field of the high-voltage acceleration (U_A) gap and hit the target placed at U_{target} potential.

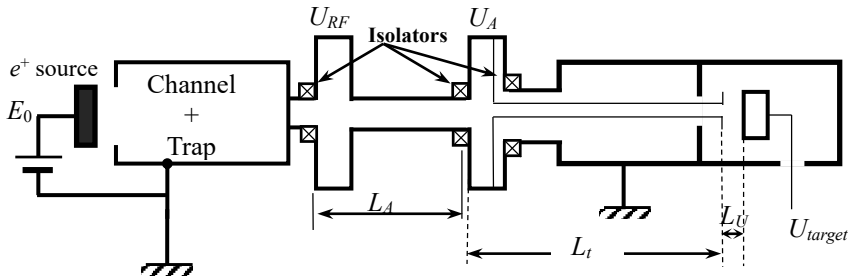

Fig. 4. Scheme of ordering the flow of positrons (explanations in the text).

The RF signal is formed according to the following scheme.

Assume that the positrons fly to the high-frequency cavity (U_{RF}) at the moment of time t_{inj} (injection) on average at intervals of $T_0 = (\dot{N}_{e+})^{-1}$ and with energy $E_0 + \delta E$, $\delta E \sim \pm 1$ eV. The speed of the positron at the exit of the RF-cavity is

$$v_{RF}(\delta E, t_{inj}) = \sqrt{\frac{2}{m} \cdot (E_0 + \delta E + eU_{RF}(t_{inj}))}, 0 \le t_{inj} \le T_0. \quad (1)$$

The positron time of flight from the U_{RF} to the U_a section is $\Delta t_A = \frac{L_A}{v_{RF}}$. The positron speed after passing the acceleration section is equal to v_A :

$$\frac{mv_A^2}{2} = \frac{mv_{RF}^2}{2} + eU_A ,$$

$$v_A(\delta E, t_{inj}) = \sqrt{v_{RF}^2 + \frac{2}{m} e U_A}\,. \quad (2)$$

At this speed, the positron flies a section of the channel length L_t to the experimental chamber entrance, where the test sample is located, placed under the negative potential of U_{target}. The flight time of the positron from the RF resonator entrance to the chamber acceleration gap is equal to

$$\Delta t_{target} = t_{inj} + \Delta t_A + \frac{L_t}{v_A(E, t_{inj})} = t_{inj} + \frac{L_A}{v_{RF}} + \frac{L_t}{v_A}. \quad (3)$$

Then we find the $U_{RF}(t)$ function from the condition of equality of the values of Δt_{target} for positrons with energy E_0 (i.e. $\delta E = 0$) for any value of t_{inj} in the interval

$$0 \le t_{inj} \le T_0, \quad (4)$$

i.e.

$$\Delta t_{target}(t_j = 0) = \Delta t_{target}(t_{inj} \le T_0). \quad (5)$$

In this case, all particles, regardless of t_{inj}, arrive at the target simultaneously. We have the equation for the function $U_{RF}(t_{inj})$:

$$\frac{L_A}{v_{RF}(0,0)} + \frac{L_t}{v_A(0,0)} + t_{inj} = \frac{L_A}{v_{RF}(0,t_{inj})} + \frac{L_t}{v_A(0,t_{inj})}. \quad (6)$$

This equation can be solved by the method of successive approximations.

1st approximation:

$$\frac{L_A}{v_{RF}} \gg \frac{L_t}{v_A}. \quad (7)$$

From (6) find

$$\frac{L_A}{v_{RF}^{(1)}(0,0)} = t_{inj} + \frac{L_A}{v_{RF}^{(1)}(0,t_{inj})}. \quad (8)$$

Taking $U_{RF}(0) = 0$, we get from (8) (for $\delta E = 0$!)

$$U_{RF}^{(1)}(t_{inj}) = \left[\frac{1}{\left(1 - \sqrt{\frac{2E_0}{m}}\frac{t_{inj}}{L_A}\right)^2} - 1\right] \cdot E_0, \quad (9)$$

$$v_A^{(1)}(0, t_{inj}) = \sqrt{\frac{2}{m} \cdot (E_0 + e U_{RF}^{(1)}(t_{inj}) + e U_A)}. \quad (10)$$

2nd approximation:

Equation (6) is represented as

$$\frac{L_A}{v_{RF}^{(2)}(0,0)} = t_{inj} + \frac{L_A}{v_{RF}^{(2)}(0,t_{inj})} + \delta t_2, \quad (11)$$

where

$$\delta t_2 = L_t\left(\frac{1}{v_A^{(1)}(0,t_{inj})} - \frac{1}{v_A^{(1)}(0,0)}\right). \quad (12)$$

From here we find

$$U_{RF}^{(2)}(t_{inj}) = \left[\frac{1}{\left(1 - \sqrt{\frac{2E_0}{m}}\frac{t_{inj}+\delta t_2}{L_A}\right)^2} - 1\right] \cdot \frac{E_0}{e}\,, \quad (13)$$

$$v_{RF}^{(2)} = \sqrt{\frac{2}{m} \cdot (E_0 + \delta E + e U_{RF}^{(2)})},$$

$$v_A^{(2)} = \sqrt{\frac{2}{m} \cdot (E_0 + \delta E + e U_{RF}^{(2)}(t_{inj}) + e U_A)}\,.$$

This gives us the time of flight (TOF) of a positron with energy $E_0 + \delta E$

$$t_{target}(\delta E, t_{inj}) = t_{inj} + \frac{L_t}{v_A^{(2)}(\delta E, t_{inj})} + \frac{L_A}{v_{RF}^{(2)}(\delta E, t_{inj})}. \quad (14)$$

We are interested in the TOF difference between the arrival at the target of positrons with $\delta E \neq 0$ and $\delta E = 0$

$$\delta t_{target}(\delta E, t_{inj}) = t_{target}(\delta E, t_{inj}) - t_{target}(0, t_{inj}). \quad (15)$$

U_{RF} is formed using a set of three to four Fourier harmonics of $U_{RF}(t)$ (13). Preliminary analysis showed that three harmonic signals are sufficient. The ordering of the positron flow occurs as a result of the passage of the accelerating gap by particles. To this gap three harmonics of the RF-voltage is applied, the sum of which creates the voltage of a special form (13). By attaching to the high-voltage phase, one can know exactly when the position enters the sample.

Three harmonic signals (100, 200, 300 MHz) with a certain amplitude and phase are used to form the voltage of a special form (Fig. 5, a), which are applied to two accelerating gaps. For this purpose, the special RF cavity system was developed and manufactured (Fig.5, b).

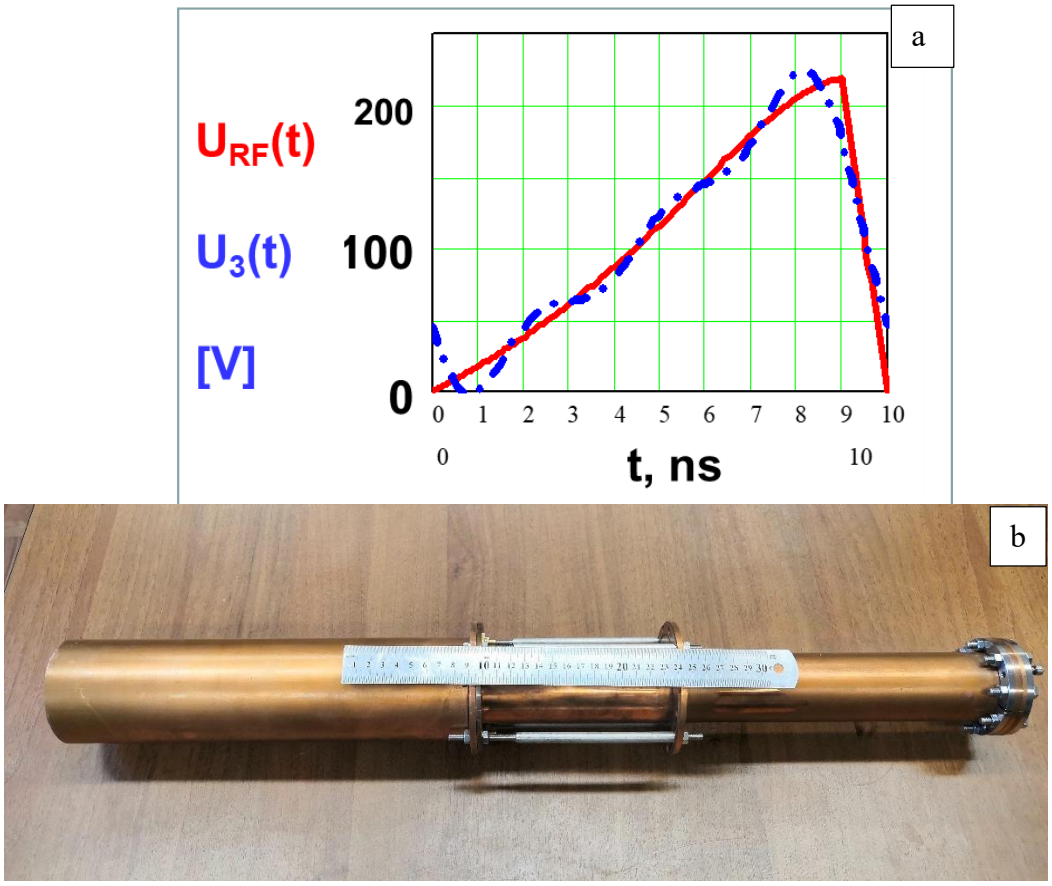

Fig. 5. (a) The $U_{RF}(t)$ function (13) and the $U_3(t)$ function formed by 3 harmonics

(b) Cavity system for ordering monochromatic positron flow.

The cavity system is assembled, tuned to three harmonics and prepared for installation inside the vacuum chamber.

A device for reactive ion etching has been developed to make it possible to study thin-film samples. It is based on an commercial ion source from PREVAC Cº. The system is located under the experimental station (Fig. 6).

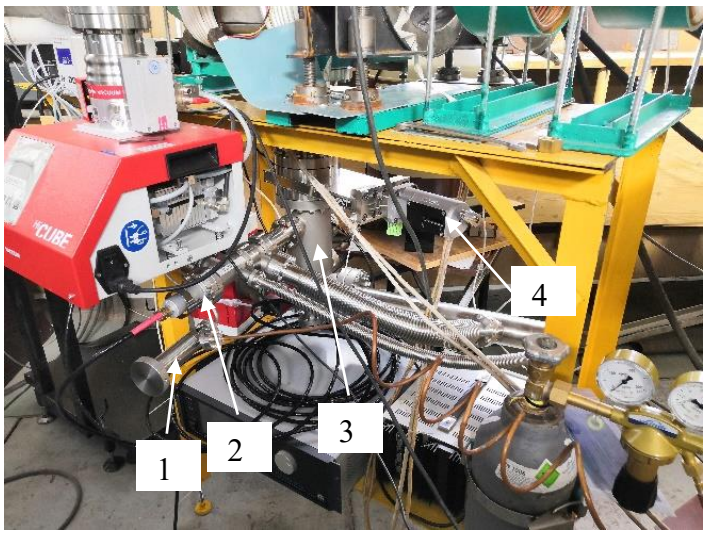

Fig. 6. Reactive ion etching device (description in the text).

For etching, argon ions are used, which is injected through the leak valve 1 into the ionization chamber of the ion source 2. Etching is carried out in the chamber *3* when the sample holder is lowered after opening the gate 4 located under the experimental station. In standby mode, the vacuum in the etching chamber is maintained by a magnetic pump. When the ion source is operating with the gate 4 open, a turbomolecular station of experimental volume is used for pumping. This system allows us to study multi-layer surface with layer thicknesses from units to tens of nanometers. Studies of this type are conducted using a positron flow with a fixed energy and scanning the sample by depth is carried out by removing the atomic layers by ion etching.

The goals and objectives of the project 2021-2023 years.

The main goals of the project are the following:

1) study the defect formation in materials as a result of various physical influences;

2) expanding the existing research base.

Research by PAS methods can be continued with existing measuring equipment.

The priority area of research at this time is the study of defect formation in materials used in nuclear energetics. These materials include various types of ceramics and refractory metals. In the preparation of samples for the study, it is necessary to carry out a procedure of "zeroing" defects in samples arising from mechanical processing. The essence of the procedure is to heat the prepared sample to a temperature equal to about 2/3 of the melting point of the material. Heating has to be done in a vacuum. The problem of high-temperature vacuum heating can be solved by heating samples with an electron beam,. The available technical capabilities allow this method of heating to be implemented. The first attempts of high-temperature annealing on the stand "Recuperator" (SEC DLNP) were performed.

The expansion of the experimental base will be carried out in several directions:

1) Improving the DBAL spectrometer by introducing into the measurement scheme the registration of the coincidence of two annihilation gamma quanta. In a standard measurement scheme (one detector), the peak-to-background ratio is usually 30 to 1. This ratio can be improved by more than two orders of magnitude when using the method of coincidence of two gamma quanta registration (Fig. 7). This technique can be used to measure the contribution of the nucleus and the valence electron recoil into the Doppler broadening spectrum. Measuring the distribution of pulses of the electrons of the nucleus, characteristic of each element, one can identify the chemical elements around the place of the positron annihilation and give at least a qualitative description of the sample

structure and microscopic location of the atoms surrounding the vacancy. This method is widely used to study polymers, semiconductors, alloy additives, etc.

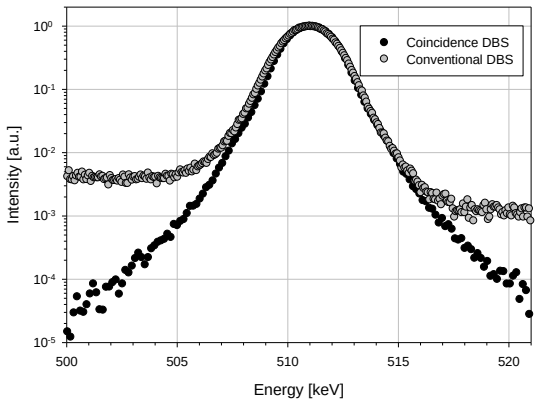

Fig. 7. Comparison of the spectrum obtained by conventional and coincidence methods for the same sample of aluminum alloy Al88. 8 Pb11. 2%.

2) Completion of the positron ordering system and commissioning of the PALS spectrometer with a monochromatic positron beam.

PALS spectroscopy is a unique tool for analysis of structural defects and voids in materials. The lifetime of a positron is related to the size of the void in which the positron is captured and weakly depends on the material structure surrounding the void. However, quantity identification of positron lifetime is difficult when study thin films or layered structures with a submicron layer thickness that are of high technological significance. The isotropic radiation and high energy of conventional positron emitters gives a depth of penetration of the millimeter scale. Currently developed devices overcome this limitation by using monochromatic positron beams, which makes it possible to conduct studies of thin films structure dependence on the depth at a scale from nanometers to micrometers. There are only a few such installations in the world.

3) Development of the ion etching technique with the created etching system and its application for the study of thin-film multilayer materials.

The commissioned ion source allows irradiating samples with argon ions with an energy up to 5 keV and studying the distribution of defects with the best depth resolution. The currently operated positron beam provides the use of positrons with energies up to 40 Kev. After implantation in the material, the distribution of positrons can be described by the Mach profile. An approximate profile of positron implantation with energies of 5, 15 and 25 Kev is shown in Fig. 8. An increase in energy gives a wider spread of positrons. Therefore, the standard beam measurement method allows one to examine thin films located on the surface of the sample.

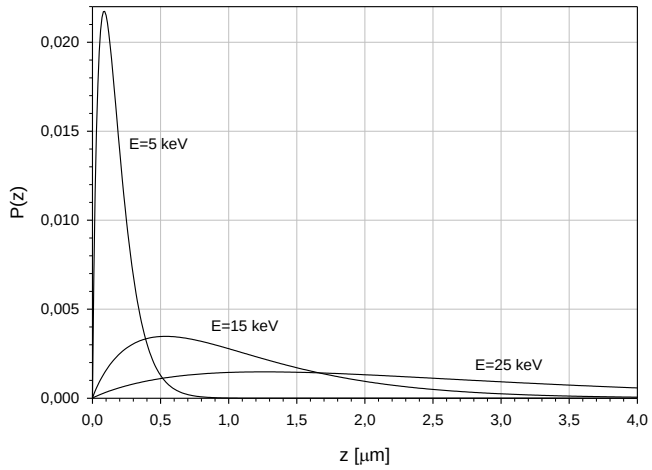

Fig. 8. Implantation profile of positrons with energies of 5, 15 and 25 keV.

If the sample surface consists of several films with a total thickness of more than a few hundred nanometers, then only the top of them can be studied. The use of an ion source makes it possible to spray the target by ion etching and keep low positron energy. Studies have shown that when the surface atoms of the sample are gradually removed by argon ions, the defective structure of the sample area under study does not change if the positron energy is chosen high enough to exclude reverse positron diffusion to the damaged surface. The results of measurements using the standard method and using ion etching for samples of CIGSe solar cells are presented in Fig. 9.

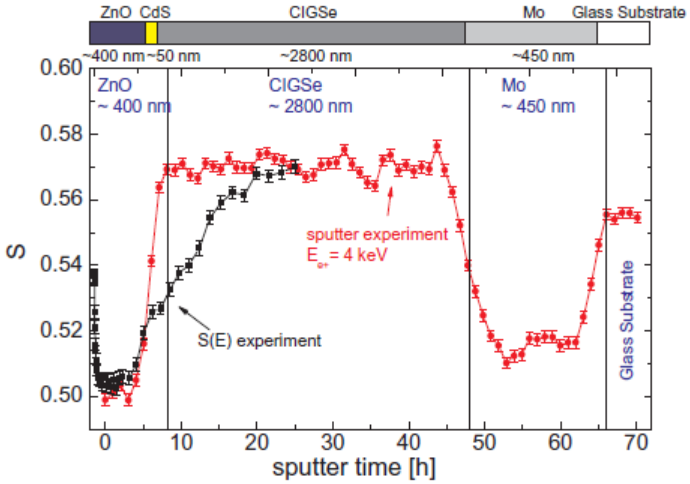

Fig. 9. Defect profiles obtained using the standard DBAL PAS method (black) and ion etching (red).

A brief SWOT analysis.

The strengths of the project.

The group's experimental capabilities are already able to compete with many of the centers and laboratories that are engaged in research with PAS method. Due to collaboration within JINR Laboratories, the group has access to various accelerators for sample preparation, as well as various research equipment for additional analysis.

Weaknesses of the project.

There is no strong theorist in the field of solid-state physics in the team or in the collaborating groups who can set research tasks and explain the various effects obtained, for example, the long-range effect.

Opportunities.

Access to research equipment of SEC DLNP for scientific groups from the Member States of JINR is possible and welcome.

The equipment can be used for personnel training in the field of positron spectroscopy, vacuum and accelerator technology, including training of students and advanced training of engineers and researchers.

Threats.

The research uses expensive measuring, vacuum, and cryogenic equipment. The cost of individual components is commensurate with the project's annual budget. There are risks of a long interruption in the operation of individual spectrometers when the equipment fails.

Not fully funded.

Human resources assessment.

Table 2. DLNP staff involved in the project.

№	Full name		FTE
1	Meshkov I. N.	Theory, setting up and carring out an experiment, result analysis	0,25
2	Kobets A. G.	Setting up, preparing, carring out an experiment, result analysis	0,85
4	Sidorin A. A.	Carring out an experiment, result analysys	0,85
5	Siemek K.	Setting up, preparing, carring out an experiment, result analysis	1,0
6	Akhmanova E. V	Numerical simulation of the positron dynamics	0,7
7	Yakovenko S. L.	Experiment setup, the discussion of the results	0,1
8	Orlov O. Yu.	Carring out an experiment, result analysis	0,85
9	Hilinov V. I.	Design, manufacturing, mounting of the PAS equipment	0,85
10	Soboleva L. V.	Documentation, preparation and formation of reports	0,85

Conclusion

The implemantation of the program presented in this project will bring the complex to a qualitatively new level and create new opportunities for experimental studies by the PAS method with directed flows of monochromatic positrons.

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Appendix 1

Form 26

Proposed schedule and necessary resources for the project

"DEVELOPMENT OF THE EXPERIMENTAL TECHNIQUES AND APPLIED RESEARCHES WITH SLOW MONOCHROMATIC POSITRON BEAMS"

Names of costs, resources, and funding sources			Cost (thousands of dollars). Resource requirements	Proposal of the laboratory for the distribution of funding and resources		
				1 year	2 year	3 year
Costs		1. Measuring equipment 2. RF components 3. Vacuum equipment 4. Materials	70 17 18 15	20 7 8 5	25 5 5 5	25 5 5 5
Funding source	Budgetary funds	Budget costs, including foreign exchange funds	120	40	40	40

PROJECT SUPERVISORS A. G. Kobets,

K. Siemek

Appendix 2

Form 29

Cost estimates for the project "DEVELOPMENT OF THE EXPERIMENTAL TECHNIQUES AND APPLIED RESEARCHES WITH SLOW MONOCHROMATIC POSITRON BEAMS"

NN пп	Name of cost items	Full cost	1 year	2 year	3 year and so on
	Direct Project Costs				
1.	Materials	**15** thousands of dollars	5	5	5
2.	Equipment	**105** thousands of dollars.	35	35	35
3.	Travel expenses, including	**30** thousands of dollars			
	а) to non-ruble zone countries		8	8	8
	б) to cities of the ruble zone countries		2	2	2
	Total direct expenses:	**150**	50	50	50