

# POSITRON ANNIHILATION SPECTROSCOPY AT LEPTA FACILITY

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## Abstract

Positron Annihilation Spectroscopy (PAS) is a sensitive method dedicated to detection of open-volume type of defects in materials. Nowadays, this technique is of a great interest due to the practical character of obtained results. New devices using monoenergetic positron beams are built. The poster presents progress in this field at LEPTA project at Joint Institute for Nuclear Research in Dubna, present and future directions of works.

## INTRODUCTION

Positron Annihilation Spectroscopy (PAS) is a great method for detection open-volume defects on the atomic level. It is applied in the field of solid body physics as well as in the material research. It gives interesting results as the independent method or the complementary technique for other methods such as Rutherford Back Scattering (RBS) or Mossbauer Spectroscopy.

PAS is based on the special properties of positron – electron annihilation process. Positron annihilates with its antiparticle – electron and as a result in 99.8 % cases two gamma quanta with energy of about 511 keV are emitted. [1] The annihilation process does not take place immediately, but positron spends some time in the matter on the thermalization and diffusion stages. [2] This time depends on the electron concentration in the structure. If the lattice includes defects the electron density is lower in comparison to a non-defected area and in this was positron life time will be longer. The positron life time means the time between positron emission eg. from the <sup>22</sup>Na isotope and annihilation. Because the electron momenta inside traps are also lower in comparison to the bulk, it also finds a reflect in the annihilation characteristics. In this way the energy of gamma quanta will be changed as a result of Doppler effect according to formula

$$E_{\gamma} \cong mc^2 + E_b \pm \frac{p_{\parallel}c}{2} \tag{1}$$

where  $E_b$  is the energy of positron-electron pair coupling and  $p_{\parallel}$  is a perpendicular component of the positron-electron pair's momentum. This effect is observed as the broadening of annihilation line 511 keV. The broadening always appears in this case but for positrons trapped in the defects will be smaller considering the lower electron momenta. It should be noticed that the momentum of thermalized positron can be negligible. Thus the registration 511 keV line gives information about momentum state of electrons taking part in annihilation process. It is the role of Doppler Broadening of Annihilation Gamma Line (DB) technique.

## DOPPLER BROADENING OF ANNIHILATION GAMMA LINE TECHNIQUE (DB)

The formula describing the changing of gamma energy can be also expressed as following

$$E_{\gamma} \cong mc^2 \pm \sqrt{\frac{1}{2}mc^2 E} \tag{2}$$

If the energy of an annihilating positron equals e.g. 7eV (Fermi energy for copper), then a change of energy of annihilating gamma quantum, according to the above formula, will equal 1,34 keV. Thus, the total broadening of annihilation line will equal 2,68 keV.

Observation of such broadening requires using detectors of a high energetic resolution. Currently available germanium detectors allow to take measurements with resolution equal to 1-2 keV around 511 keV energy.

Doppler broadening of annihilation line technique is used to detect concentrations of defects such as vacancies and their accumulations. A signal from annihilation of a trapped positron gives broadening of the 511 keV line accordingly smaller than the one that would occur in case of annihilation with nucleus electrons. In other words, less defected sample gives smaller broadening of the 511 keV line.

In practice, the broadening of 511 keV line is not calculated but the analysis is limited to evaluating of two characteristic parameters, the so-called S- and W-parameters (see Fig. 1). [3,4]

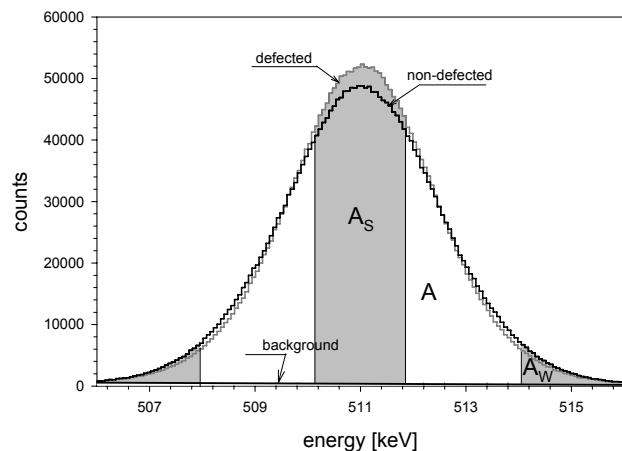


Figure 1: 511 keV lines for defected and nondefected samples.

S parameter defines proportion of annihilation of positrons with low-momentum electrons. It is closely related to concentration of defects in a material. It is

defined as ratio of surface area under the central part of the 511 keV line to total surface area under this line. Areas are usually selected so that their ratio approximated 0.5. Bigger S parameter value means bigger concentration of defects.

The second parameter, the so called W parameter is defined as a ratio of surface area under the wing part of 511 keV line to total surface area. It is related to annihilation of positrons with high-momentum electrons and it provides information about chemical environment of the defect. Value of this parameter is also selected arbitrarily – it tends to be smaller than 0.01.

Both S and W parameters are calculated after the background reduction. Calculations are made by special computer software. As stated above, the analysis of results of Doppler broadening of annihilation line is based on calculating S and W parameters, changes of which provide information about changes in concentration of defects.

## SLOW POSITRON BEAM AT LEPTA FACILITY

The PAS can be realized in two ways. As the standard experiments when positrons emitted directly from the source eg.  $^{22}\text{Na}$  are used or using the so called slow positron beam. In the first case positrons have continuous energy spectrum from zero to some maximal energy characterized for chosen isotope eg. 545 keV for  $^{22}\text{Na}$  and 1.89 MeV for  $^{68}\text{Ge}$ . In this way the mean implantation depth is a few dozen micrometers in dependency on the kind of material. Changes in the structure near the surface are invisible. The second solution allows slowing-down positron to the energy of about 50 eV and accelerating to the chosen energies - in practice not higher than 40 keV. It is possible to use special moderators based on the W foil or frozen Ne gas. This approach makes the detection of defects in the range up to a few micrometers under the surface possible.

Since the year 2000 at JINR in Dubna project LEPTA has been realized. Its main aim is to acquire a positronium atom in flight [5]. A positron beam (see Fig. 2) constructed for this aim can also be used for PAS.

The idea of producing the beam is following. Positrons emitted from  $^{22}\text{Na}$  go through a moderator, which is a condensed Ne source. As a result of elastic scatterings on gas particles some electrons lose their energy. Those two types of particles are separated by the use of

perpendicular magnetic fields. Fast positrons are stopped at diaphragm, while the slow ones are slaloming and are then formed into a beam and accelerated by negative potential to needed energies. Currently available beam's parameters are given in Table 1.

Table 1: Parameters of Positron Beam at LEPTA Facility

Feature	Value
activity of $^{22}\text{Na}$ isotope	25 mCi
moderator	frozen Ne (7K)
longitudinal magnetic field	100 Gs
vacuum conditions	$10^{-9}$ Torr
intensity	$3 \times 10^5$ e <sup>+</sup> /s
energy range	50 eV ÷ 35 keV
diameter of the flux	3 mm

## EXAMPLE OF APPLICATION

The problem of formation of oxide films on surfaces of stainless steel 304 AISI annealed for 2, 3 and 8 hours at 800 °C in vacuum of  $6 \times 10^{-6}$  mbar, air and in flow  $\text{N}_2$  of  $0.35 \text{ cm}^3/\text{s}$  atmospheres was studied using slow positron beam.

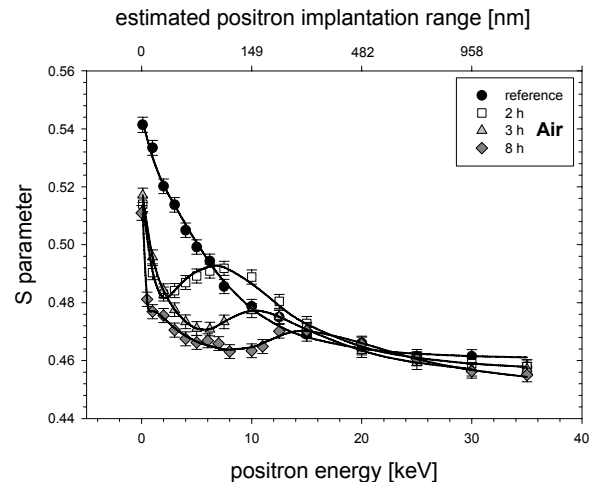


Figure 3: S parameter on dependency the positron implantation energy for studied samples. The solid lines represent the best fits of model function using VEPFIT code. [6]

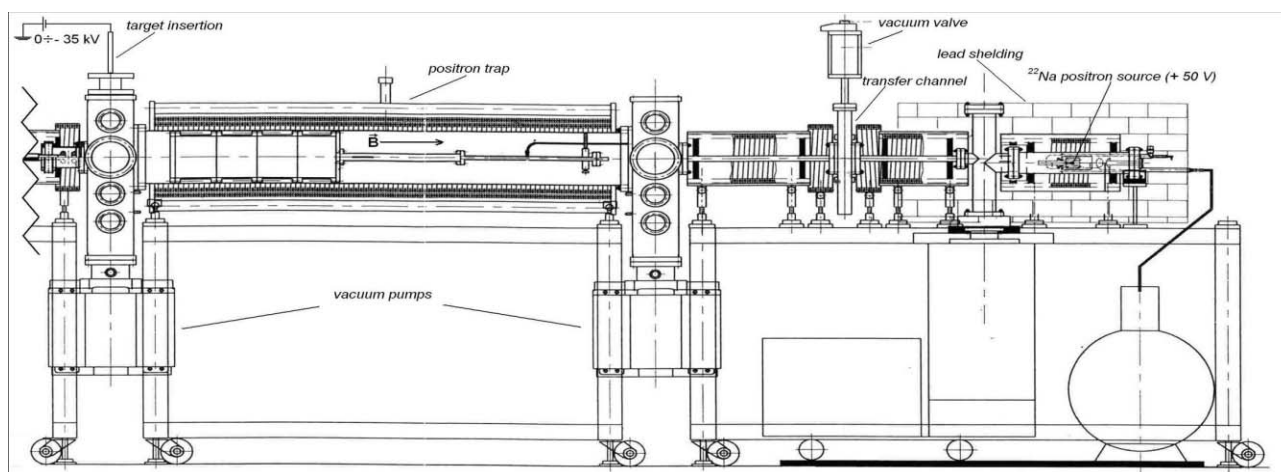


Figure 2: The scheme of slow positron beam at lepta facility.

The S- parameter profiles in dependency on the positron implantation are presented in Fig. 3. The creation of multilayer systems of oxides on the surface after annealing in air and N<sub>2</sub> conditions was confirmed. The fitting procedure [6] of obtained S parameter profiles allowed to find the thicknesses of oxide layers. The S-parameter profiles in dependency on the positron implantation are presented in Fig. 3.

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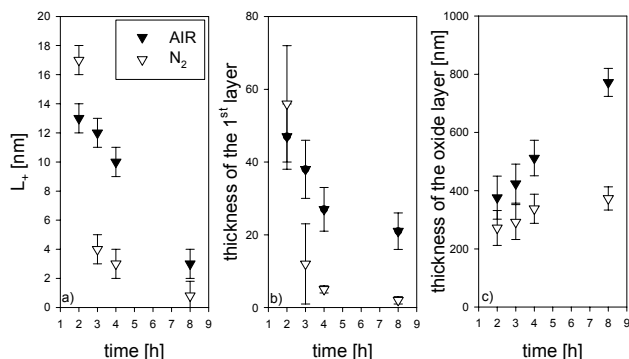


Figure 4: The time-dependencies of parameters obtained from fitting profiles presented in Fig. 3

The information about formation of duplex oxide on the surface, where  $\alpha$ -F<sub>2</sub>O<sub>3</sub> is created in the outermost layer before the inner spinel rich in chromium up to temperature of 850 °C is given in the literature [7]. There is no information about the thickness of these layers. The time-dependencies of parameters obtained from fitting profiles presented in Fig. 3 are presented in Fig. 4.

In the case of sample annealed in vacuum the positron diffusion length in the 4.2±0.9 nm thick oxide layer was 8±1nm, while in the bulk it was 94±3 nm. The thickness of the oxide layer depends on the kind and time of annealing atmosphere. The shield atmosphere of N<sub>2</sub> in flow does not protect the material before oxidation.

## CONCLUSIONS

The standard version of DB spectrometer works at LEPTA facility at JINR in Dubna. The first results confirm the beam works in an appropriate way. The research is going to concern mostly materials engineering, both in metals and semi-conductors. The main research is going to include studies on the influence of surface treatment processes on the defecting of the surface layer in materials which are commonly used in industry, such as stainless steel. Furthermore, plans are made to conduct research oriented around thin layers and layers created by ion implantation. In further perspective there are plans to develop the equipment to automatize the measurements and using Surko trap for bunching signal in the aim of building pulsed positron beam for life time measurements.

## APPENDIX

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