

POSITRON ANNIHILATION SPECTROSCOPY

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Abstract

Positron annihilation spectroscopy is nowadays well recognised as a powerful tool of microstructure investigations of condensed matter. An overview of the method aimed for users from other fields is given and supplemented with several examples of applications.

Keywords

positron-electron annihilation, positron lifetime, Doppler broadening, angular correlation of annihilation radiation, electronic structure, positron trapping, defects in solids, free volume in polymers

1. Introduction

An overview of positron annihilation spectroscopy (PAS) and its applications to condensed matter studies and materials research is presented. The overview is aimed as an introduction for potential users of PAS in these areas. Detailed treatment of PAS method can be found in several books, e.g., in Proceedings [1, 2]. PAS applications to various kinds of materials (metals and alloys, semiconductors and macromolecular solids) are briefly discussed below and several examples are given.

2 Principles of PAS

Positron (e^+) is the antiparticle of electron (e^-). The electromagnetic interaction between electrons and positrons makes possible annihilation of $e^+ - e^-$ pairs in which the total energy of the annihilating pair may be transferred to quanta of the electromagnetic field (photons γ). Principal channel of this reaction is the two-photon annihilation,



The role of conservation of energy and momentum in process (1) is illustrated in Fig. 1. In the centre-of-mass frame of the $e^+ - e^-$ pair, the energies of the both annihilation photons are equal to the rest energy of the electron (positron), $E_0 = m_0c^2$, and the two photons are emitted in the strictly opposite directions. In the laboratory frame, in which positron is considered to be in the rest, energies of the two annihilation photons are shifted with respect to E_0 by $\Delta E \approx \pm cp_{-L}/2$ and the angle between emission directions of

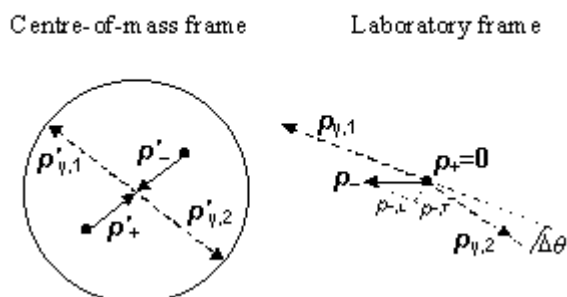


Figure 1. Kinematic diagram of two-photon annihilation of the $e^+ - e^-$ pair. In the laboratory frame (positron resides in the rest) momenta of electron (positron) are denoted as \mathbf{p}_- (\mathbf{p}_+) while momenta of the annihilation photons are $\mathbf{p}_{\alpha,i}$ ($i=1,2$). Subscripts L, T denote longitudinal and transversal components, respectively, related to emission of $\tilde{\alpha}_1$ -quantum. Corresponding quantities in centre-of-mass frame are marked with the apostrophe.

the two photons differs from π by $\Delta\theta \approx p_{-T}/m_0c$. In these expressions, non-relativistic approximation is used and symbols p_{-L} and p_{-T} denote longitudinal and transversal components of the electron momentum, respectively. The electron (positron) rest mass is designated as m_0 and c is the light velocity. Process (1) is characterised also with annihilation rate λ (positron lifetime $\tau = \lambda^{-1}$). Theoretical treatment reveals, see e.g. Ref. [1], that λ is proportional to the effective electron density n_e sampled by positron, viz. $\lambda \approx \pi r_c^2 c n_e$, where r_c stands for the classical electron radius¹.

To get a rough idea of magnitudes of quantities λ , ΔE and $\Delta\theta$ for $e^+ - e^-$ annihilation in condensed matter, one must insert realistic estimates of n_e and electron momentum p into the above expressions. Conduction electron densities in metals are typically of order of 10^{-29} m^{-3} [3]. Core electron momenta in atoms may be taken roughly as $\hbar/2r_a$, where atomic size is characterised by Bohr's radius r_a and \hbar is the Planck's constant. Then the following estimates of λ , ΔE and $\Delta\theta$ may be obtained: (i) Positron lifetimes $\tau \approx 600 \text{ ps}$ are expected in metals². (ii) Doppler shifts of annihilation photon energies amount $\Delta E \approx 1 \text{ keV}$. (iii) Angular correlation curves should exhibit widths of a few mrad.

Conventional sources of positrons for PAS are artificial radioisotopes emitting β^+ -radiation. Energy spectra of positrons emitted by such radioisotopes are continuous ranging from zero to an end-point energy which is typically of order of $0.1 - 1 \text{ MeV}$. Stopping profiles of

- 1 The n_e represents the overlap integral of the electron and positron densities
- 2 This guess is significantly higher than the experimental observations of positron lifetimes in metals. The reason is in neglecting enhancement of the local electron density at the positron site caused by Coulomb attraction between the positron and mobile conduction electrons of the host. The enhancement factors in metals amount typically to 4–5 leading thus to values of $\tau \approx 100 - 200 \text{ ps}$, in agreement with experiments.

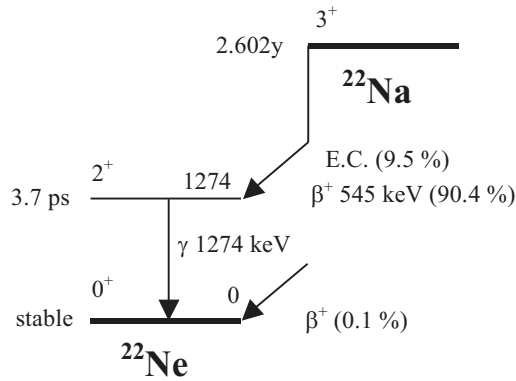


Figure 2. Decay scheme of ^{22}Na

β^+ -particles in solids decrease exponentially with increasing penetration depth z , $P(z) \sim \exp(-z/z_0)$, where typical mean penetration depths z_0 are of order of 10 – 100 μm [3]. Thus positrons from radioisotope sources probe volume properties of matter. The most frequently used among them is ^{22}Na radioisotope. Its decay scheme is shown in Figure 2. End-point energy of ^{22}Na is at 545 keV. An important feature of the ^{22}Na decay is simultaneous emission (within a few ps) of β^+ - and γ -radiation ($E_\gamma = 1274$ keV, see Fig. 2).

Recent progress in development of positron beams has made possible to utilise the variable energy positron beams as positron sources in PAS studies [4]. Positron beam energies range typically from 10 eV to 100 keV. The mean stopping depths of such positrons vary with energy from 1 nm to a few μm which illustrates potential of positron beams for PAS studies of near-surface regions in condensed matter.

Before annihilation, positrons penetrating through matter undergo various processes influencing the state from which the positron annihilates with an environmental electron. When the energetic positrons ($E_{\text{kin}} \approx 0.1\text{--}1$ MeV) are implanted into a condensed medium, they rapidly lose their energy. At highest positron energies, the main mechanism of energy losses is ionisation, i.e., the positron excites core electrons in collisions with the host atoms. At lower energies the electron-hole excitations take over. When the positron energy has degraded to a fraction of eV, the scattering off phonons dominates. Eventually, positrons reach thermal equilibrium with the medium, maintained due to phonon emission and absorption. During thermalization, the initial kinetic energy of positrons drops below 0.1 eV, i.e., by almost six orders of magnitude. Despite of such a colossal change in energy, positron thermalisation times t_{th} are typically as short as a few picoseconds [3], i.e., much shorter compared to the above estimated positron lifetimes.

Thermalised positrons are characterised with thermal wavelength $\Lambda_+ \approx 50(300 \text{ K}/T)^{1/2} \times 10^{-10}$ m [3] which implies that such a positron moves through the medium as a quantum-mechanical wave. After reaching thermal equilibrium with the host, the positron state develops as a diffusion process. In metals at room temperature, the positron mean free path l_+ is typically of the order of 10 nm whereas total diffusion length before annihilation L_+ is of the order of 100 nm [5]. During diffusion the positron

interacts with its surroundings and eventually annihilates with an environmental electron. In homogeneous defect-free media all positrons annihilate with the same rate λ_b which is a characteristic of the given material.

Due to the Coulomb repulsion by the positive-ion cores, positron in a condensed medium preferably resides in the inter-atomic space. At open-volume defects (monovacancies, larger vacancy clusters, dislocations etc.), the potential sensed by the positron is lowered due to reduction in the Coulomb repulsion. As a result, a localised positron state at the defect can have a lower energy than the state of delocalised (free) positron. The transition from the delocalised state to the localised one is called positron trapping. Positron binding energies E_b to defects like, e.g., monovacancies are typically of a few eV, see e.g. Ref. [6]. Thermal detrapping is impossible from such deep traps and positron remains trapped until annihilation. If a positron trap is shallow enough ($E_b < 0.1$ eV), phonon-assisted detrapping occurs. As local electron density at the defect site is lowered compared to that of the unperturbed regions, lifetime τ_t of the trapped positrons is correspondingly longer than $\tau_b = \lambda_b^{-1}$. Positron trapping is characterised by trapping rate κ which is proportional to defect concentration c_t in the sample, $\kappa = \nu c_t$. Trapping coefficient ν , together with annihilation rate $\lambda_t = \tau_t^{-1}$, are specific for given kind of defects.

As a result of positron trapping, additional exponential components occur in measured positron lifetime spectra. Their appearance can be explained by the trapping models which give the rate equations for the positrons annihilating from the delocalised state and from the localised states. Examples of such models and their use in the quantitative analysis of PAS data can be found in Refs. [5, 7, 8].

In many insulators, the positron can form a bound state with a host electron, positronium (Ps), which is an analogue of the hydrogen atom [3]. The binding energy of Ps atom is 6.1 eV and its radius is 0.106 nm. Ps atom can exist in the singlet (parapositronium, pPs) or the triplet (orthopositronium, oPs) state. While the two-photon self-annihilation of pPs is an allowed process, the two-photon annihilation of oPs can proceed only via the pick-off reaction with an electron of the host. Due to exchange interaction of its electron with environmental electrons, Ps atom is repelled to an unoccupied space in the material.

Basic principles which PAS investigations of condensed matter rely upon can now be summarised as follows:

When positrons are implanted into a condensed medium, they get quickly in thermal equilibrium with their surroundings. Being thermalised, positrons interact with their environment and eventually annihilate with electrons of the medium. Measurable parameters of annihilation reaction reflect the structure of the medium. There are two ways how the dependencies of the PAS parameters on the medium structure arise:

- Through the state of the electron participating in the annihilation reaction. This introduces dependence on the local electronic structure of the host at the annihilation site.

- Through the state of the positron itself at the moment of annihilation: the free positron, positron captured at a trap, different states of the Ps atom in the medium. This state is a result of medium-dependent positron interactions prior to annihilation.

The processes which positrons undergo in matter before they annihilate with environmental electrons proceed with characteristic time and length scales. Considering the conventional PAS studies in metals, characteristic lengths can be ordered in the following way:

$$\Lambda_+ \approx 5 \text{ nm} \leq l_+ \approx 10 \text{ nm} < L_+ \approx 100 \text{ nm} < z_0.$$

Similar ordering of characteristic lengths appears also for semiconductors. For interpretation of PAS data it is important to compare length scales of positron processes in matter with lengths characterising microstructure of materials like, e.g., grain size, average inter-defect distance, 'size' of sample inhomogeneities etc.

Characteristic times of annihilation process are typically much longer than t_{th}^3 and can be ordered according to the state from which positron annihilates. This is illustrated in Table 1.

3. Experimental Techniques of PAS

PAS involves several experimental techniques which originally came from subatomic physics, namely from nuclear spectroscopy. This is why progress in PAS is closely related with achievements in nuclear experimental methods.

Positron-lifetime measurements (PAS/LT). Principle of the PAS/LT measurements with ^{22}Na positron source is given in Figure 3. The positron is implanted into the sample almost simultaneously with the birth γ -ray 1274 keV. Lifetimes of individual positrons, t , can be measured as time differences between emission of the birth γ -quantum

Table 1. Characteristic time scales of various positron annihilation processes in matter

Positron state	Type of process	Charact. lifetime
'free' e^+	2γ	0.1 – 0.4 ns
'trapped' e^+	2γ	0.2 – 0.5 ns
pPs	2γ , self-annihilation	0.1 ns
	2γ , pick-off process	> 1 ns
oPs	3γ , self-annihilation	140 ns
	2γ , pick-off process	> 1 ns

and one of the annihilation photons. PAS/LT measurement then consists in measuring the spectrum of delayed coincidences $\gamma_{1274} - \gamma_{511}$ keV. In Figure 3, the simplest configuration of a lifetime spectrometer, referred to as the fast-fast one, is also shown. Detectors D1 and D2 (BaF_2 or plastic scintillators) detect the birth and annihilation photons, respectively. Fast signals related to moments of emission of corresponding γ -quanta are generated by constant-fraction differential discriminators (CFDDs). These signals are lead to the start and stop inputs of a time-to-amplitude converter (TAC). Coarse energy selection is provided by the CFDDs, too. The fast coincidence unit (FCC) produces gate signal for the TAC provided that the coincidence event of photons with proper energies occurred. Amplitude of the TAC output signal, which is proportional to time delay t between the birth and the annihilation photons, is digitalised by an analog-to-digital converter (ADC) and the event is then stored at corresponding address of a histogramming memory (HM). Thus the spectrum of positron lifetimes is obtained as the histogram of counts in dependence on t , $N(t)$.

PAS/LT measurements are also feasible with pulsed positron beams. In this case start signal is generated by positron-beam pulse (pulses of length as short as ≈ 100 ps are necessary). One of several such facilities has been recently built up at TU München [9].

Spectrum of positron lifetimes is a sum of exponential components

$$N(t) = \sum_{i=1}^n I_i \lambda_i \exp(-\lambda_i t) \quad (2)$$

Annihilation rates $\lambda_i = \tau_i^{-1}$ and positron fractions I_i can be obtained using standard computer procedures [10 - 12]. Number of components n is equal to number of different states which positrons can annihilate from, e.g., in the case of k types of defects $n = k+1$. Generally n should be regarded as an unknown parameter, too. In practise only up to three components can typically be resolved in spectra without involving any constraints on parameters. On the other hand, for complex systems with many different annihilation sites it is often more adequate to consider even continuous distributions of λ . Standard computer programs are available also for this case [13 - 15].

The experimentally obtained spectra differ from the analytical description (2) by convolution with time resolution function $R(t)$ which is the response of the spectrometer to prompt coincidences. The $R(t)$ function resembles Gaussian like shape and is characterised by the full width at half maximum (fwhm). The fwhm and count rate are crucial characteristics of each PAS/LT spectrometer which determine quality of experimental data. The time resolution may be improved using the fast-slow coincidence scheme in which a more precise energy selection of coincidence events than that inherent to the scheme of Figure 3 is accomplished. see, e.g., Ref. [16]. This may result, however, in considerable reduction of

3 Exclusions of this rule, however, may occur in insulators, where energy losses due to collisions of host electrons are not as effective as in metals or semiconductors.

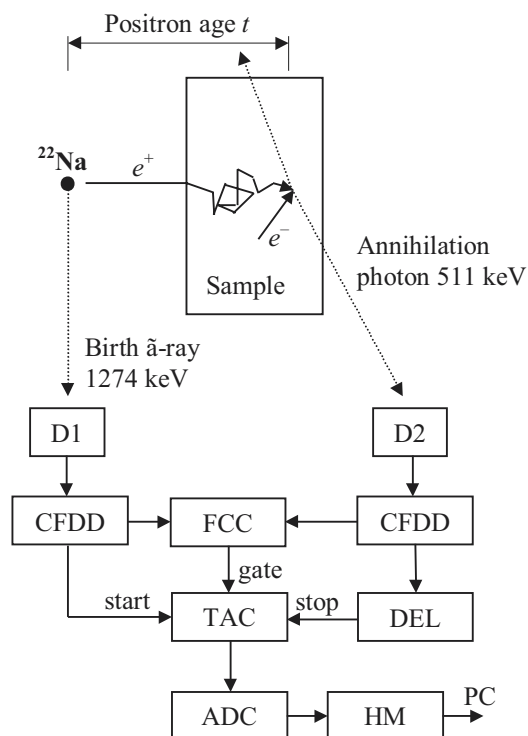


Figure 3. Principle of PAS/LT method with ^{22}Na positron source and a simple fast-fast PAS/LT spectrometer: D1, D2 – detectors, CFDD – constant-fraction differential discriminator, DEL – delay line, FCC – fast coincidence unit, TAC – time-to-amplitude converter, ADC – analog-to-digital converter, HM – histogramming memory, PC – computer.

coincidence count rate. In practise one have to choose a compromise between time resolution and count rate, depending on the nature of the problem studied. The best time resolution reported so far in real PAS experiments is 150 ps fwhm for ^{22}Na which was achieved at a reasonably high count rate of 80 s^{-1} using the spectrometer built in Prague [16]. Under such conditions components with lifetimes as short as 40 ps and intensities as weak as 10 % could be resolved if $\sim 10^7$ coincidence events were accumulated in the spectrum, i.e., 24 hour measurements [16]. Capability of resolving such components is especially important for defect studies in metals, because knowledge of free-positron lifetime and fraction is necessary for obtaining full information about positron trapping process, see e.g. Ref. [8].

Doppler broadening of annihilation line (PAS/DB) can be measured with a standard γ -ray spectrometer equipped with the HPGe detector. Energy resolution of such devices $\Delta E_\gamma \approx 1.2 \text{ keV}$ at 511 keV is now being normally achieved. Observed line shapes are usually characterised with shape parameters S and W . The former parameter is experimentally determined as the relative area of the central part of the line shape while the latter one expresses the relative contributions of the peak tails to the total peak area. Thus the S -parameter is higher if relative contribution of lower-momentum electrons to positron annihilation is enhanced while the W -parameter becomes greater if contribution of the core electrons with higher momenta tends to increase. For instance, relative increase in fraction of posi-

trons trapped at the open-volume defects can be markedly reflected by increase in the observed S -values. An useful approach is to present experimental data in term of S - W plot which allow to draw some conclusions about evolution of defects participating in positron trapping, see e.g. Ref. [17] for details.

A substantial problem that severely limits capability of PAS/DB method in its simplest configuration described above is high background. This is especially expressed for the measurements of the high-momentum parts of the annihilation peak. The background can be drastically reduced by use of the coincidence technique which registers both annihilation photons. The coincident γ -quantum is almost anti-collinear to the primarily registered annihilation photon and it serves to suppress Compton background near 511 keV energy which arises from the 1274 keV γ -ray of ^{22}Na . The best results can be obtained with the coincidence system with two Ge detectors. The two-dimensional spectrum is recorded where the axes represent the energy scales of the respective detectors. The improved peak-to-background ratio of 10^{-5} and better energy resolution were obtained in this way using the spectrometer [18].

Angular correlation of annihilation photons (PAS/AC).

The origin of angular correlation of annihilation radiation can be seen in Fig. 1. The two annihilation photons are emitted simultaneously. Thus $\Delta\theta$ as a function of the transversal electron momentum component can be measured in coincidence arrangement with position-sensitive detectors. A simple position sensitive detection can be realised in one dimension by mechanical movement of a long scintillation detector with lead slits allowing for angular resolution [19]. The momentum distribution can also be recorded in two dimensions using a two-dimensional detector arrays [19]. The sample-to-detector distance amounts typically to several metres so that γ -quanta from only a small solid angle are detected. Hence much stronger positron sources compared with conventional PAS/LT and PAS/DB techniques are required. On the other hand, angular resolution can be adjusted in the range of 0.2 to 5 mrad [18]. This corresponds to the energy resolution of PAS/DB measurement in the range of 0.05 to 1.3 keV. Thus PAS/AC technique provides essentially the same kind of information as PAS/DB, however, the momentum resolution of the method is much better.

Age-momentum correlation (AMOC). This technique combines PAS/LT and PAS/DB using the same annihilation event [20]. One of the two annihilation photons provides the stop signal for the positron lifetime while the second photon is used for the measurements of the Doppler broadening of the annihilation line. The triple coincidences of both the annihilation photons with the start signal are registered. The Ge detector for measurement of the annihilation photon energy in coincidence must be arranged opposite to the scintillator detector for the stop signal. Two dimensional spectra are stored with the positron age and Doppler shifts being represented along the co-ordinate axes. Thus dependence of the electron

momentum distributions on the positron lifetime can be measured. For example, Doppler curves for oPs events with a very long lifetimes can be separated out of the annihilation events related to pPs or positrons that did not formed Ps.

4. Examples of PAS Applications

There are three main classes of materials which have so far been studied by means of PAS. Historically earliest PAS applications were devoted to studies of metals and alloys. Then PAS investigations were extended towards semiconductors. In both these fields positron plays a role of the main probe and formation of Ps is usually of less significance. The third area of PAS applications, which is nowadays intensively developing both in the experimental as well as theoretical planes, is concerned with studies of polymers and the other macromolecular substances containing large fraction of unoccupied space. Contrary to the above two kinds of materials, Ps is often formed with a high yield and becomes a principal probe in these systems.

Metals and alloys. It has been already noted in Section 2 that Coulomb attraction between positron and conduction electrons in metals effectively increases local electron density at positron and thus reduces positron lifetime. Observed lifetimes of positrons in defect-free metals, τ_b , lie typically in 100 to 200 ps region [21]. There are only several cases when positron lifetimes exceed 300 ps, e.g., in alkali metals. Theoretical calculations of positron states in matter and PAS parameters are feasible, including Coulomb screening factors [6]. These are in excellent agreement with experimental data.

Positron trapping at monovacancies, dislocations and vacancy clusters in metals has been extensively studied by means of PAS [5]. Lifetimes of positrons trapped at monovacancies, τ_{1V} , are usually observed to be $\tau_{1V} \approx 1.6 \tau_b$ [21]. Specific trapping rates at monovacancies are expected to be $v_{1V} \sim 10^{14} - 10^{15} \text{ s}^{-1}$ [6]. Thus the range of sensitivity of PAS to detect vacancy concentrations lies between $10^{-7} - 10^{-4}$. Vacancies represent a deep traps for positrons while dislocations are shallow traps. It is however generally accepted [7, 22] that once positron is trapped at the dislocation it quickly diffuses along the dislocation line and is subsequently trapped at point-like defects associated with the dislocation. This assumption may serve as an explanation why observed lifetimes of positrons trapped at dislocation are close to those of monovacancies [5].

Extensive PAS/LT and PAS/DB study of the reactor pressure vessel (RPV) steels has been performed by Prague positron group with the aim to contribute to knowledge of the neutron irradiation-induced embrittlement of the steels during operation of the nuclear reactors. Firstly, detailed microstructure characterisation of non-irradiated specimens was made [23] which revealed the dislocations are the main positron traps in these materials. The ratio of the screw to edge dislocations could be deduced in these experiments. Then RPV specimens irradiated by fast neutrons at various fluences and fluxes were investigated [24]. To suppress unwanted contribution of ^{60}Co to positron lifetime spectra measured with the irradiated

samples, a three-detector setup has been constructed [25] and for the first time used in real measurements. Small vacancy clusters were identified in [24] as a product of irradiation. Their size of ≈ 5 vacancies was not changed while their concentration slightly increased with increasing neutron fluence.

Microstructure of ultra fine grained (UFG) copper has recently been investigated by means of the high-resolution PAS/LT technique in Prague, too [8]. Positrons were found to be trapped mainly at dislocations in distorted regions along grain boundaries and at microvoids (of size of ≈ 5 vacancies) distributed homogeneously throughout the grains. Recovery of UFG structure during annealing, involving abnormal grain growth followed by primary recrystallisation and further growth of the recrystallised grains at higher annealing temperatures, was observed, see Ref. [8] for detailed description.

The common feature of both the RPV steels and UFG materials is a strongly non-homogeneous spatial distribution of dislocations which represent the main positron traps. Due to this circumstances the conventional trapping model [5] had to be modified [7, 23] in order to obtain consistent description of positron trapping. This model was successfully applied to RPV steels [23]. Further improvement of the model leading to the diffusion trapping model was introduced [8] to obtain consistent description of experimental data on UFG copper.

Aluminium based alloys were also investigated by PAS/LT in Prague. As an example the contribution [26] can be regarded.

Semiconductors. In semiconductors Coulomb screening of positrons is not so effective as in metals [5, 6, 17]. This is why observed positron lifetimes for semiconductors are generally larger than those for metals, i.e., $\tau_b \sim 200 - 300$ ps. On the other hand, $\tau_{1V} \approx 1.2 \tau_b$ is typically found in semiconductors [5] which makes identification of monovacancies in semiconductors more difficult compared to metals. Important advantage of PAS of semiconductors is its sensitivity to the charge state of positron trap. Remarkably different temperature dependencies of trapping rates may be observed if the defect is charged positively, negatively or is neutral [6, 17].

Polymers. In polymers three components are typically resolved, the two lowest lifetimes being attributed to pPs, free and trapped positron annihilations [27]. The third one arises from the oPs annihilation via pick-off process and is characterised with lifetimes τ_{oPs} of a few ns. It is generally accepted that τ_{oPs} can serve as a measure of the free-volume hole size seen by Ps. The simple model of Ps in a spherical potential well of radius R leads to correlation between τ_{oPs} and R [28, 29],

$$\tau_{oPs} = 0.5 \times \left[1 - \frac{R}{R + \Delta R} + \frac{1}{2\pi} \sin 2\pi \frac{R}{R + \Delta R} \right]^{-1}, \quad (3)$$

where $\Delta R = 0.166$ nm is an empirical parameter and τ_{oPs} is given in ns. Despite of simplicity of the model assumptions, Eq. (3) seems to hold surprisingly well in the



region of R up to 1 nm and constitutes a base for numerous PAS applications to studies of the free volume and its changes in polymers, e.g., glass transitions [27], pressure dependencies [27], free-volume collapse [30-33] and blending [24]. On the other hand, interpretation of the oPS intensities as a measure of the hole concentration must be taken with certain caution because of possible influence of various factors on Ps formation, e.g., irradiation effects [34, 35], which have not yet been sufficiently understood.

5 Conclusions

PAS has demonstrated itself as a powerful tool of microstructural studies of condensed matter. Atomic-scale details of the microstructure like, e.g., electronic structure and small-sized defects can be investigated by PAS. The main advantages of PAS can be summarised as follows:

- Small-sized defects at low concentration can be detected by PAS which can otherwise be hardly investigated by traditional basic techniques like, e.g., TEM or X-ray diffraction.
- PAS can probe both the bulk properties of matter as well as near-surface regions and layered structures.
- Reliable theoretical calculations of PAS parameters are nowadays feasible in metals and semiconductors. These may substantially simplify unique interpretation of data measured by PAS.
- PAS is a non-destructive technique.

In many cases PAS becomes not only substantial complement of traditional methods (e.g., TEM or X-ray diffraction), but it is an equivalent and independent method of microstructural investigations of condensed matter.

Acknowledgement

This work was supported by Ministry of Education, Youth and Sports under contract No. 2134.

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