APPLICATION OF ENCAPSULATED ^{22}Na ISOTOPE TO DOPPLER SPECTROSCOPY BASED ON POSITRONS EMITTED DIRECTLY FROM THE SOURCE*

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In the paper, we present results of examination of encapsulated ^{22}Na positron source with Doppler spectroscopy. For this reason, a special geometry of experiment is proposed. Using the suggested scheme, measurements of S- and W-parameters of deformed and non-deformed samples of copper and bronze DIN-CuBe2 were conducted. In the case of non-defected specimens, the constant value of S-parameters was observed. For samples exposed to sandblasting, decreasing of S-parameter with increasing depth was detected. The thickness of damaged region for copper was about 250 μm and, in comparison to bronze, it was twice as long. The pressed copper sample was characterized by a constant level of defect concentration in the total investigated volume.

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1. Introduction

Positron Annihilation Spectroscopy (PAS) is a successful method for detection of open volume defects and, due to this fact, it is useful for many aspects of solid state physics [1, 2], surface engineering [3], material science [4–6] *etc.* Positrons can be produced during pair creation from energetic photons, however, β^+ decay of nuclei is a common source of positrons as well. The former is a very effective but expensive method and possible to be realized only at huge facilities. For this reason, PAS experiments are commonly provided using β^+ isotopes.

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The most popular isotope is ^{22}Na having a long half-life close to 2.6 years, β^+ fraction 90% and maximum β^+ energy equal to 0.545 MeV. Additionally, it is widely used in the positron lifetime measurements due to emission of 1.274 MeV gamma quantum after positron production [7]. In the Doppler spectroscopy, a very popular isotope is ^{68}Ge/^{68}Ga. Its half-life is 0.8 year and the maximal energy of emitted positrons is 1.9 MeV [8]. In measurements, both mentioned isotopes are enveloped into two thin foils *e.g.* 7 μm kapton and are placed between two identical samples. The activity of these isotopes is usually not higher than 0.2 mCi. Studies with slow positrons, produced in small accelerators, are commonly provided using ^{22}Na with intensities of a few dozen of mCi. This type of emitter is closed in a special capsule with the titanium window through which positrons are emitted only in one direction [9].

According to the above discussion, application of PAS techniques requires positron sources convenient for the given type of investigations. In this study, we suggest to apply encapsulated ^{22}Na positron source to Doppler spectroscopy involving positrons emitted directly from the emitter (fast positrons). Such sources are used for accelerators called slow positron beams [10–12]. Recently, commercial companies offer small size spectrometers allowing to carry experiments out of the laboratory. The type of the source investigated in this paper would be highly appreciated in this case.

In this paper, we present studies of the defect depth distribution in the subsurface regions of copper and bronze samples exposed to sandblasting. The encapsulated ^{22}Na positron source, in the form dedicated for slow positron beam, but with low activity was used in Doppler spectroscopy. The aim of this work was the examination of this type of source in the conventional PAS experiments.

2. Application of the source in PAS experiment

The picture of the positron source used in measurements is depicted in Fig. 1. Isotope of ^{22}Na with activity *ca.* 15 μCi is hidden in a special copper capsule. Positrons are emitted through 4 mm in diameter and 5 μm thick titanium window. In the accelerator, they are injected to the moderator and after moderation accelerated to the desired energy to hit a sample. Positrons emitted in the opposite direction are useless.

The idea of experiment with the encapsulated positron source is presented in Fig. 2. The source is placed in a special holder with the window directed to the top. The studied sample covers the titanium window. Positrons pass through the window and are implanted into the sample at the depth of about dozen micrometers. One of the annihilation photons is detected in the HpGe detector. The lead shield located between the detector

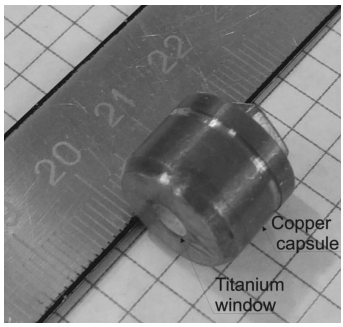

Fig. 1. Encapsulated ^{22}Na positron source used in the measurements.

and the source absorbs the annihilation photons coming from the annihilation in the capsule. The thickness of the lead shield is about 5 cm. This ensures that mainly annihilation photons from sample are detected by the HpGe detector. However, some background from annihilation photons in the copper capsule is also present.

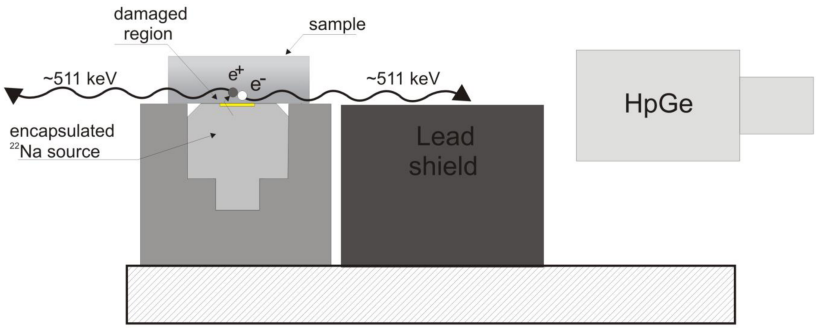

Fig. 2. The geometry of experiment with an encapsulated positron source.

A positron implanted into the matter reduces its initial energy to the thermal energies and after a random walk of about 100 ps annihilates with an electron, mainly a valence one. Two annihilation photons created in this process are emitted in the opposite directions. Their energies depend on the momentum of the annihilating positron–electron pair, but in practice on the momentum of the electron. This is a Doppler effect. Due to the positive charge, a randomly walking positron can be trapped at open volume defects, which contain mainly low momentum electrons. It will be reflected in the Doppler broadened annihilation line as an increase of the contribution in its low momentum region close to 511 keV. The valence or core electrons have much higher momenta, thus, they contribute to the 511 keV line but in the other part. The energy of the annihilation photons can be expressed in an approximate form as follows

$$E_{1,2} \simeq m_0 c^2 \pm cp/2\,, \tag{1}$$

where m_0 is the electron mass at rest, c is the speed of light, and p is the projection of the electron momentum on the direction of the annihilation photon emission.

The annihilation line is observed using spectrometer whose main part is a HpGe detector. The energy resolution (FWHM) of applied detector is 1.2 keV at 511 keV. This is a crucial parameter because a typical Doppler shift is about 2–3 keV, according to Eq. (1). Each measured spectrum contains 2.5×10^5 counts in the region of annihilation line. The spectra were analysed by SP program [13]. This code allows to obtain two characteristic for this method factors, namely S- and W-parameters. The former represents the fraction of positron annihilation with low momentum electrons. It is defined as the ratio of area below the central part of 511 line to the total space under this line. Value of this parameter arises with the increase of defect concentration. It has been demonstrated by many authors [14, 15] that this parameter is extremely sensitive to the presence of open volume defects, such as vacancies, their clusters, dislocation lines, and jogs at dislocation lines, where positrons can be trapped and annihilate. The latter, characterizes annihilation of positrons with high momenta electrons. W-parameter is given as the ratio to the area below the wing part of annihilation line to the whole region under the line. It informs about chemical environment of a defect. In the literature, one can find useful mathematical models which describe this dependency [16].

3. The experimental details

For the examination of an encapsulated source in PAS application, three discs of pure copper with size of 20×5 mm^2 and two beryllium bronze DIN-CuBe2 with size of 10×5 mm^2 were used. Surfaces of all of them were polished and then cleaned in an ultrasound cleaner in acetone for 1 hour at 50°C. After that, they were annealed for 2 hours at 800°C in the vacuum conditions of 10^{-5} Torr and slowly cooled down in the closed furnace to the room temperature. One of copper and bronze discs were sandblasted using Renfert Vario Vasic Jet blaster for 30 sec with 250 μm particles of Al_2O_3 under the pressure of 6 bars perpendicularly to the surface. This treatment induces the defects at the surface and in region adjusted to it. The second sample of copper was pressed with 5 kN load. In this way, the thickness was reduced for about 60 μm (1.2%). The press also introduces defects due to the plastic deformation. All surface treatments processes mentioned above produced defects in specimens which should be detected in PAS measurements.

In order to recognize the defect depth distribution below the surface, we applied the following procedure. The value of the S-parameter was measured in the setup presented in Fig. 2, however, the surface exposed to sandblasting was located at the titanium window, through which positrons are emitted. They were implanted directly into the sample. After the measurement, the sample was etched in the nitrite acid and as a result about 20 μm thick layer was removed. Then, again, the sample was placed on the window and the measurement of the annihilation line was performed. The sequenced etching allowed us to find out how the S-parameter changes with the thickness of the etched layer, *i.e.*, depth from the surface exposed to sandblasting.

In Fig. 3 (a), S-parameter *versus* etched depth for samples of sandblasted (black circles) and pressed (white circles) copper are depicted. The grey circles represent a non-defected side of a sample for comparison. In this case, S-parameter values obtained in different depths are constant within the measurement accuracy (hatched area). These are the expected results, because this sample contains only residual defects which were not removed during long annealing. However, the dependency obtained for sandblasted sample is different. The S-parameter on the surface is much higher. At the depth of 20 μm slightly arises and then the S-parameter values decrease with the etched depth. As a result, a small plateau close to the surface is observed. Decreasing of S-parameter means the decreasing of defect concentration. The depth where the value of S-parameter for a defected sample achieves the value from the hatched area determines the thickness of a damaged

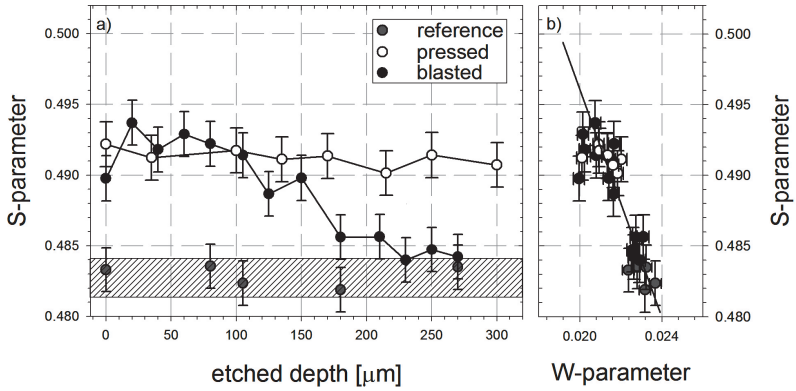

Fig. 3. S-parameter in dependency on etched depth (on the left) and *versus* W-parameter (on the right) for samples of copper exposed to pressing under the load of 5 kN (white circles) and blasting for 30 sec with 250 μm particles of Al_2O_3 under the pressure of 6 bars perpendicularly to the surface (black circles). Hatched region represents the mean value of S-parameter obtained from measurements of a well-annealed sample (grey circles).

subsurface zone. In this case, it is *ca.* 250 μm. The pressed sample is characterized by constant values of S-parameter in the whole investigated region below the surface, it means 300 μm. The unchanged level of defects concentration in pressed copper was reported in [17].

In Fig. 3 (b), S-parameter in dependency on W-parameter for discussed samples is shown. Points are oriented along the straight line. It points out the domination of the same kind of defect introduced to structures for both treatment processes. The recognition of its type is not possible using Doppler spectroscopy.

In Fig. 4 (a), S-parameter *versus* depth for bronze samples is presented. The discussion of this case is similar to the above analysis of respective samples. Similarly, for the reference sample, the measured S-parameter values in the whole depth range correspond to the bulk value, hatched region in Fig. 4. Sandblasting introduces lattice defects whose concentration decreases with the depth up to *ca.* 120 μm. It should be noticed that in comparison to copper it is twice as thick. The difference can be induced by the presence of alloying addition. The different size of surfaces could also have an impact on this situation. Another important similarity is a small plateau close to the surface, which appears *e.g.* in stainless steel exposed to sliding [18]. Decreasing of S-parameter in copper and bronze is linear within obtained measurement accuracy but the exponential decay cannot be definitively excluded.

In Fig. 4 (b) $W(S)$ points also lie in the straight line. It points out the presence of only one kind of defect in a studied sample.

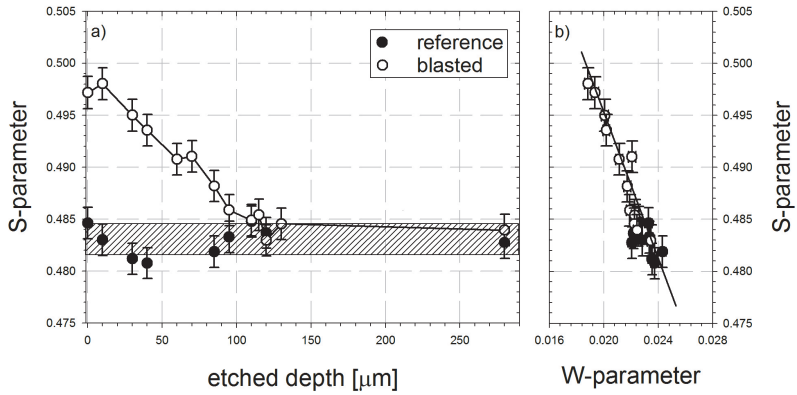

Fig. 4. S-parameter in dependency on etched depth (on the left) and *versus* W-parameter (on the right) for samples of bronze DIN-CuBe2 blasted for 30 sec with 250 μm particles of Al_2O_3 under the pressure of 6 bars perpendicularly to the surface (white circles). Hatched region represents the mean value of S-parameter obtained from measurements of well-annealed sample (black circles).

4. Summary

In this study, using encapsulated ^{22}Na isotope to the conventional PAS experiments with positrons emitted directly from the source was proposed. On the contrary to the conventional geometry of this kind of experiment, the exit of positrons is possible only through a special window. In this way, the registered signal is interrupted by annihilation of positrons inside the capsule. The set presented in this paper allows to eliminate undesirable influences. Obtained characteristics show that the source in this form can be successfully applied in this kind of experiment.

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