JOINT INSTITUTE FOR NUCLEAR RESEARCH
The Dzhelepov Laboratory of Nuclear Problems

FINAL REPORT ON THE SUMMER STUDENT PROGRAM

Positron Annihilation Spectroscopy Studies of Bronze Exposed to Sandblasting at Different Pressure

Supervisor:
P. Horodek, PhD

Student:
S. Kurdyumov, Russia
Saint-Petersburg Polytechnic University

Participation period:
July 10 – 30
August 14 – September 03

Dubna, 2016

0. Abstract

Sandblasting is used to modify the surface of materials. It is known that sandblasting process generates high-density dislocations which after annealing lead to nanocrystalization [1,2]. In performed studies we investigate the impact of sandblasting pressure on surface and subsurface changes of beryllium bronze using Doppler broadening of annihilation line spectroscopy (PAS) and optical microscopy. Samples of beryllium bronze DIN-CuBe2 were blasted for 1 minute using 110 µm particles of Al_2O_3 at different pressure. In the case of a non-defected sample, the constant value of S-parameter was observed. For samples exposed to sandblasting, decreasing S-parameter with increasing depth was detected. The range of changes in subsurface zones was determined. It was observed that higher pressure causes enlargement of defected area and roughness of the surface.

Contents

1. Introduction

Positron Annihilation Spectroscopy (PAS) is a non-destructive technique of studying open-volume defects in solids with the size smaller than 10 nm and to approximate concentration on the level up to 10^{-7}. [3]

Positron (e^+) is an antiparticle of electron (e^-). It means that it has the same properties as electron but the opposite charge. Positron and electron annihilate generating gamma-quanta. In the most cases only two photons are observed (99.6%). Other annihilation channels are also possible but with much smaller probability. [4]

Standard positron sources for PAS are synthetic radioisotopes emitting β^+ radiation. These are e.g. ^{22}Na, ^{48}V and ^{68}Ge. Positrons emitted directly from ^{22}Na isotope are commonly used in the field of PAS. These positrons have continuous energy spectrum from 0 to some maximal energy specific for a given source, e.g. 545 keV for ^{22}Na. Stopping profiles of β^+-particles in solids decrease exponentially with increasing penetration depth z,

$$P(z) \sim exp(-z/z_0) \quad (1)$$

where z_0 is a usual mean penetration depth, which is equal to several dozen micrometers in metals and about 1 mm in polymers [1].

There are 3 experimental techniques within PAS: angular correlation of positron annihilation radiation (ACAR), positron annihilation lifetime spectroscopy (PALS) and observation of the Doppler broadening (DB) of annihilation line. In our studies the last method was used. This technique is based on detection of annihilation quanta with energy of c.a. 511 keV.

In annihilation process energy and momentum are conserved. In the center-of-mass frame of the pair, both annihilation photons have the same energy equal to the rest energy of the electron (positron)

$$E_0 = m_0c^2 \quad (2)$$

where m_0 is the rest mass of electron and c is the speed of light. The photons are emitted in the strictly opposite directions. In the laboratory frame energies of the annihilation photons are shifted by Doppler effect with respect to E_0 by $\Delta E \approx \pm cp_{-,L}$. Symbols $p_{-,L}$ and $p_{-,T}$ denote longitudinal and transversal components of the electron momentum. The annihilation line is always broadened because of the Doppler effect, however, the momenta of electrons at vacancies are lower so the annihilation line is less broadened in the case of annihilation in vacancy type defects. [4]

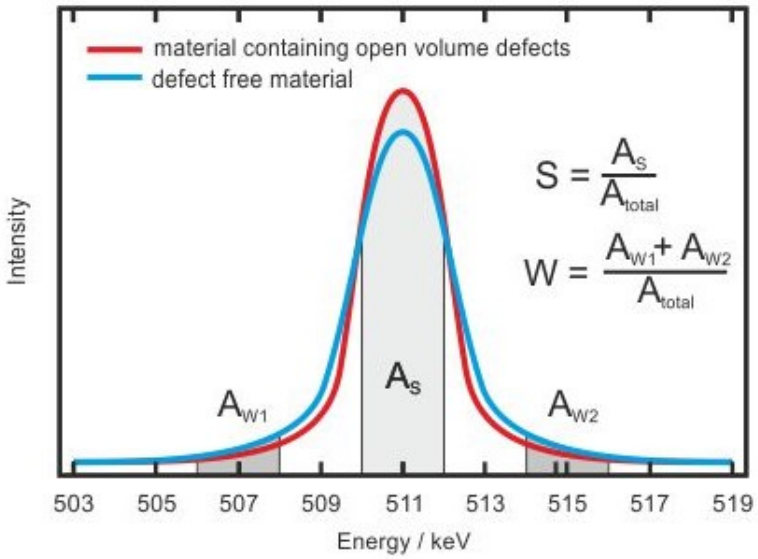

Fig.1. The definition of S and W parameters [5]

In the measured spectrum, presented in Fig. 1, two main regions can be distinguished: a low-momentum region near the peak position which reflects annihilation with valence electrons and a high-momentum region near the tails of the spectrum which demonstrates annihilation with high-momentum electrons. [6]

Usually two integral parameters (S and W) are used to probe the mentioned regions of the spectrum. The first one, called S-parameter, is calculated as the ratio of the area under the central part of the annihilation line to the total area under the line

$$S = \frac{A_S}{A_{total}} \tag{3}$$

The value of this parameter is sensitive to the concentration of the open-volume defects: as there are more defects the S-parameter's value is greater.

The second parameter, called W- parameter, is defined as the ratio of the integral of the wing part of annihilation line to the total integral of the entire annihilation line.

$$W = \frac{A_{w1} + A_{w2}}{A_{total}} \tag{4}$$

Both W and S parameters provide information about the presence of one or more kinds of defects. [7]

The aim of this work was to find out defect concentration profiles and thicknesses of defected zones in samples of beryllium bronze DIN-CuBe2 exposed to sandblasting with different pressure from 1 bar to 5 bars with the step 1 bar using the DB technique.

2. Experimental details

2.1. Samples preparation

Samples of beryllium bronze DIN-CuBe2 (Be – 2%, Co – 0.3%, Ni – 0.3%, Fe – 0.2% and Cu is the rest), in the shape of cylinder 6 mm high and 1 cm in diameter were studied. Firstly, they were annealed at 800^{o}C for two hours in vacuum conditions of 10^{-5} Torr. Then they were cooled down to the room temperature in a closed furnace. This procedure allowed to remove defects and get samples in the same state.

After that, 5 samples out of 7 were sandblasted for 1 minute using 110 μm particles of Al_2O_3 at different pressure ranging from 1 to 5 bars with the step of 1 bar. One sample was left as a reference, non-defected specimen. Sandblasting is a cold-working abrasion of surface with a stream of abrasive powder sprayed by the air flow. The stream hits the surface, removes corrosion, and shapes a clean surface. In this way, the local plastic deformations below the deformed layer and residual stresses in this layer are generated. Thus, structural defects appear and they should be visible by PAS. In Fig.2 surfaces of sample before and after sandblasting are shown.

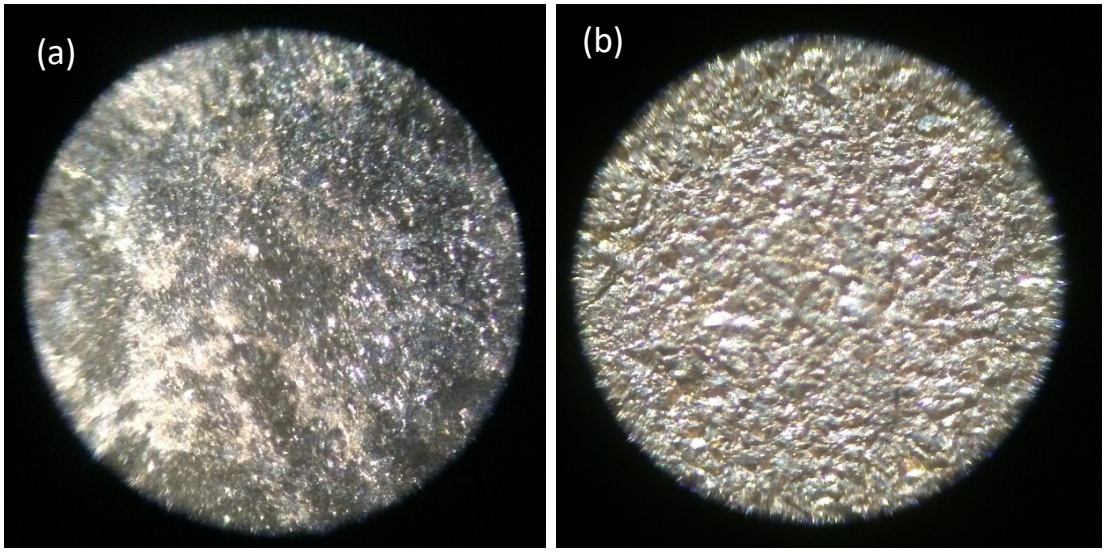

Fig.2. A sample of beryllium bronze DIN CuBe2 before (a) and after (b) sandblasting

One of the samples was not sandblasted. It was left for studying the defect distribution from depth exposed to pressing. Thus, the thickness was reduced up to 35%.

2.2. Application of the source in PAS experiment

Encapsulated isotope of ^{22}Na with activity about 15 μCi was used in this experiment. Positrons were emitted through 4 mm in diameter and 5 mm thick titanium window. The scheme of this experiment is presented in Fig 2 [6]. The source was placed in a special holder

with the window looking at the top. The investigated sample was put on the top of this source. One of two annihilation photons was detected in the HpGe detector.

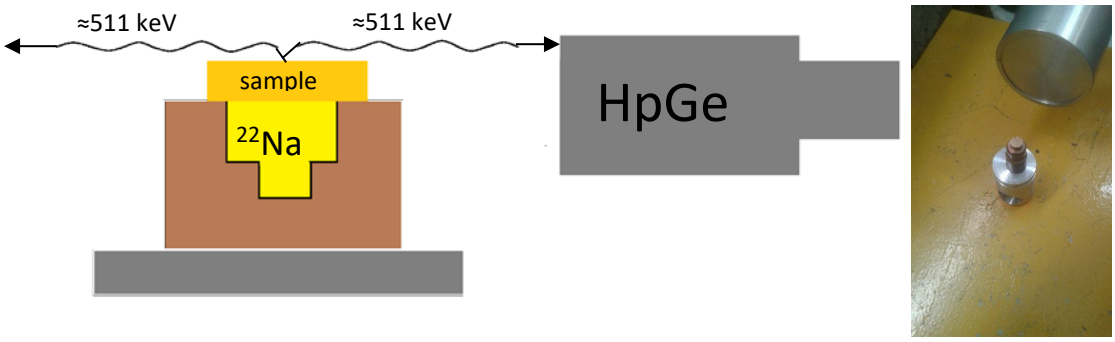

Fig. 3. The geometry of experiment with an encapsulated positron source

The energy of positrons is limited so the mean implantation depth is limited too. It was calculated by the formula

$$\bar{z} = \frac{1}{29.3Z^{0.15}\rho}, \tag{5}$$

where $\bar{z}$ is the mean implantation depth [8], Z is the atomic number of the dominating element in the alloy (for bronze this element is copper and its atomic number is 29), and ρ = 8.93 g/cm^3 stands for density of the bronze. The estimated average implantation depth is equal to 23 μm. The implantation depth is limited, so to obtain the of S parameter on depth the sample was sequentially etched in nitrid acid. Etching does not cause defects so this technique could be used in this experiment.

A standard DB spectrometer was used to observe the annihilation line. Its most important part is a high-purity germanium (HpGe) detector. Only one annihilation quantum was registered by this detector. In Fig.4 the spectrometer is shown.

Fig.4. The photo of spectrometer

The energy resolution of applied detector (FWHM) is equal to 1.2 keV at 511 keV. The spectra were analysed by SP-16K program [9]. The interface of this program is presented in Fig.5.

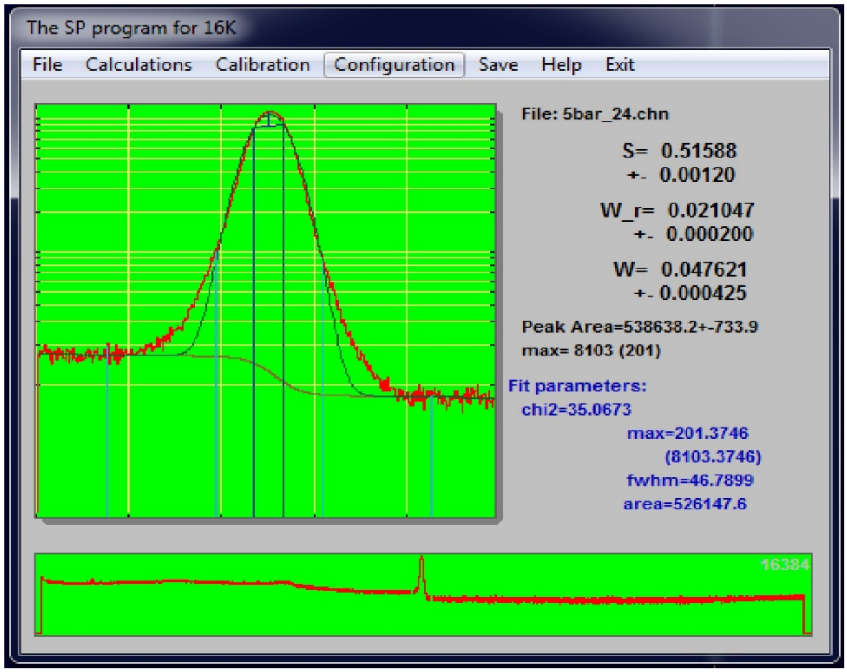

Fig.5. Interface of SP-16K program and measured spectrum

3. Results

In Fig. 2 there is the plot of S-parameter versus compressed depth. This measurement is accessory and it was done to illustrate sensitivity of PAS technique. S-parameter increases while thickness reduction is less than 15%. After that a plateau appears. In this region the increment of S-parameter is comparable with its error. Plateau reflects the thickness reduction in which PAS method cannot be used. Thus, bounds of PAS applicability were demonstrated.

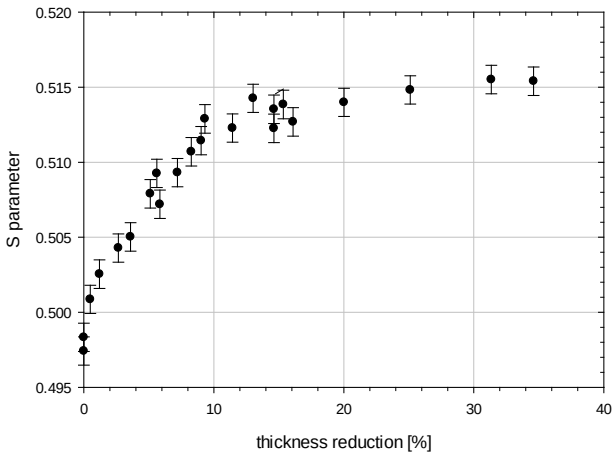

Fig.2. S-parameter in dependency on thickness reduction for a sample of beryllium bronze DIN-CuBe2 exposed to compressing under the load of up to 15 kN.

In Fig. 3a the dependency of the S-parameter on depth is depicted. For the reference sample, S-parameter values obtained in different depths are constant within the measurement accuracy (marked area). These results are expected because the reference sample contains only residual defects. [6]

In the depth equal to 0 for all sandblasted samples S-parameter is larger than for the reference sample because there are induced defects in those samples. Then, for samples sandblasted with pressure from 1 to 4 bars, S-parameter decreases linearly with the growth of the depth. This means that the defect concentration decreases with the increasing depth.

There is depth, special for each sample, in which the value of S-parameter reaches its value for non-defected sample. This depth is the thickness of defected zone. It depends on the pressure applied during sandblasting. For sample, sandblasted with pressure of 1 bar, this depth is equal to 25 μm, for 2 bars – 50 μm, for 3 bars – 85 μm and for 4 bars – 100 μm.

Results differ for the sample sandblasted with pressure of 5 bars. At the beginning, the value of S-parameter is almost the same as for 4 bars. It is approximately a constant line until the etched depth is less than 50 μm. This means that in the depth of up to 50 μm there are so many defects that PAS method is not sensitive enough to determine defect concentration (like it was shown on Fig.2). Only after 50 μm the decreasing of the S-parameter is observed. In this sample the estimated thickness of defected zone is 110 μm.

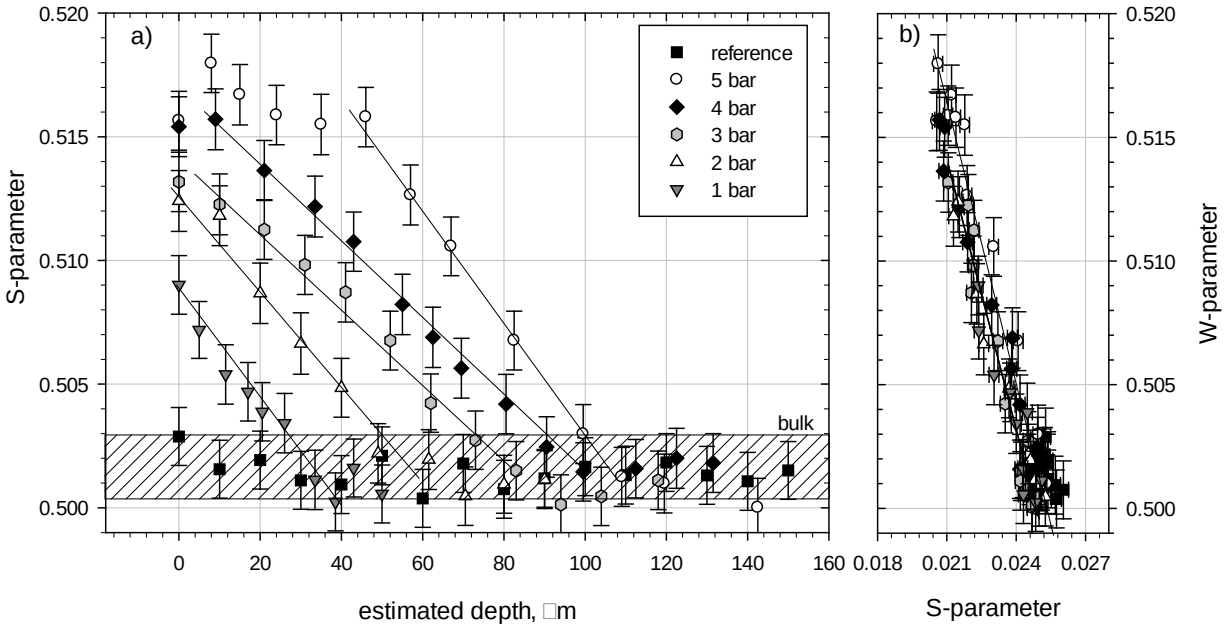

Fig. 3. S-parameter in dependency on estimated depth (a) and W-parameter versus S-parameter (b) for samples of beryllium bronze DIN-CuBe2 exposed to sandblasting for 1 minute with 250 μm particles of Al_2O_3 under different pressure ranging from 1 to 5 bars with step 1 bar. The marked region is referred to the well-annealed sample in which only residual defects present (black squares).

In the Fig. 3b S-parameter in dependency on W-parameter is shown for all studied samples. Points are situated along a straight line and this means that in all studied cases contain same type of defects is observed. Unfortunately, type of defects cannot be defined by using Doppler spectroscopy

4. Conclusion

The beryllium bronze DIN-CUBe2 exposed to sandblasting was investigated using PAS. The defect concentration decreased with the growth of depth, which was reflected in the plot of S-parameter versus depth. Furthermore, it was shown that increasing of defected area was caused by higher pressure. Also, thickness of defected zone in every sample was found. It was c.a. 30 μm for a sample blasted under pressure of 1 bar and 110 μm – for the sample blasted under 5 bars.

For the compressed sample the borders of PAS method's applicability were demonstrated. It was shown that material can be saturated with defects, *i.e.*, the defect concentration can be so high that PAS technique is unable to recognize it.

5. Acknowledgements

I would like to express my appreciation to Horodek P., PhD and PhD Siemek K., PhD, my practice supervisors and director of University Centre of Joint Institute for Nuclear Research, S. Z. Pakuliak and A.S. Zhemchugov, for giving me an opportunity to participate in Student Summer Program 2016.

I also wish to acknowledge the help provided by Kobets A.G, PhD during experiments.

In addition, I would like to express my gratitude to the organizers of Student Summer Program for help that they provided to the participants.

6. References

[1] X.S. Guan, Z.F. Dong, D.Y. Li 2005 Nanotechnology **16** 2963-2971

[2] X.Y. Wang, D.Y. Li 2003 Wear **255** 836-845

[3] P. Horodek, A.G. Kobets, I.N. Meshkov, A.A. Sidorin, O.S. Orlov 2015 Nukleonika **60** 725-728

[4] I. Prochazka, *Materials Structure*, vol.8, number 2 (2001)

[5] https://www.hzdr.de/db/!Publications?pSelInstitute=FWK&pNid=347

[6] P. Horodek, J. Dryzek 2016 Acta Phys. Pol. B Proc. Supp. **9** 1001-1007

[7] P.V. Kuznetsov, Yu.P. Mironov, et.al. 2014 AIP Conference Proceeding **1623** 327-330

[8] P. Horodek, J. Dryzek 2010 Nukleonika **55** 17-19P. Horodek, J. Dryzek, Nukleonika **55**, 19 (2010)

[9] http://www.ifj.edu.pl/~mdryzek/page_r18.html