Positron annihilation spectroscopy studies of bronze exposed to sandblasting at different pressure

To cite this article: S Kurdyumov *et al* 2017 *J. Phys.: Conf. Ser.* **929** 012029

Positron annihilation spectroscopy studies of bronze exposed to sandblasting at different pressure

S Kurdyumov[1,2*], K Siemek[3,4] and P Horodek[3,4]

[1] Saint-Petersburg Peter the Great Polytechnic University, 29 Polytechnic Str. 195251 Saint-Petersburg, Russian Federation
[2] Department of Nanophotonics and Metamaterials, ITMO University, 197101 St.Petersburg, Russia
[3]Joint Institute for Nuclear Research, 6 Joliot Curie Str., 141980 Dubna, Moscow region, Russian Federation
[4]Institute of Nuclear Physics of the Polish Academy of Sciences, 152 Radzikowskiego Str., 31-342 Kraków, Poland
*corresponding author: Tel.:(+7)9112459937 e-mail: se.kurdjumov@gmail.com

Abstract. An application of Doppler broadening of annihilation line spectroscopy to samples of beryllium bronze DIN-CuBe2 exposed to sandblasting is presented in performed studies. It is familiar that sandblasting introduces open-volume defects. Samples were sandblasted under different pressure for 1 minute using 110 μm particles of Al_2O_3. For a non-defected sample the constant value of S-parameter was detected. In the cases of sandblasted samples, S-parameter decreased when the depth enhanced. In our studies the thicknesses of defected zones were determined (it was c.a. 30 μm for a sample blasted under pressure of 1 bar and 110 μm – for 5 bar), and it was also observed that if sandblasting pressure is higher the defected zone is larger.
Key words: positron annihilation spectroscopy, sandblasting, defects, bronze

1. Introduction

Positron Annihilation Spectroscopy (PAS) is a non-destructive technique of detecting open-volume defects in solids, such as vacancies, vacancy clusters, microvoids or dislocations. [1,2] It can be used in cases where other popular methods such as scanning electron microscopy (SEM) or X-ray diffraction are not applicable. [3]

In this paper studies of beryllium bronze DIN-CuBe2 exposed to sandblasting are presented. Sandblasting is a cold-working process especially used for removing rust with a stream of fast particles of abrasive powder. The stream hits the surface generating local plastic deformations. As a result, lattice defects appear and they can be detected by PAS techniques. The presence of defects has a direct impact on the properties of surface and subsurface regions. Thus, this study is important in application because the presence of defects influences on tribological and mechanical properties of material. [4,5]

There are few papers about PAS studies [3-6] of different materials exposed to sandblasting. Pure aluminum and aluminum alloy sandblasted with 0.5 mm in diameter silicon carbide particles under 6.5 bars were investigated by Dryzek. [6] Both materials had similar defected zone thickness of about 300 μm. Divacancies were found in pure aluminum while larger vacancy clusters were recognized in the aluminum alloy. Vacancies on the edge of dislocations in stainless steel sandblasted with 110 μm Al_2O_3 particles were detected. [3] This kind of defects appeared independent on pressure and time of blasting. Additionally, reported range of defects in beryllium bronze DIN-CuBe2 created by blasting with 250 μm particles of Al_2O_3 under 6 bar for 30 sec was c.a. 250 μm. [8] In our work the impact of pressure on

thickness of damaged layer and defect profile in the same kind of bronze was investigated using Doppler broadening (DB) spectroscopy.

2. Experimental technique of PAS

2.1. Samples preparation

Seven samples of beryllium bronze DIN-CuBe2 (Be – 2%, Co – 0.3%, Ni – 0.3%, Fe – 0.2% and Cu in 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 to get all samples in the same state.

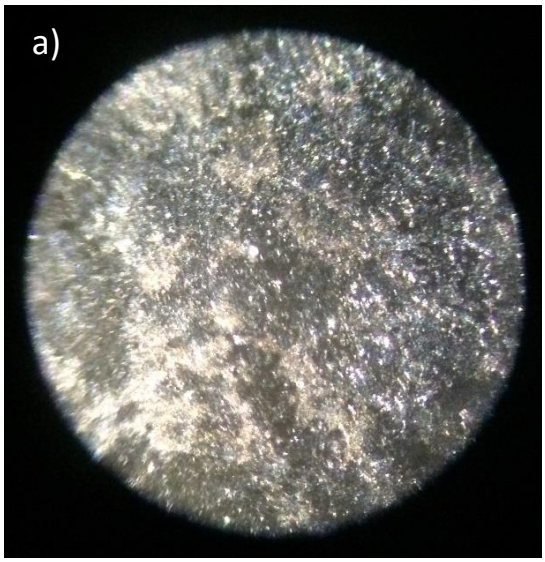

Figure 1a. An optical microscope image (magnification is equal to 180) of beryllium bronze DIN CuBe2 sample before sandblasting

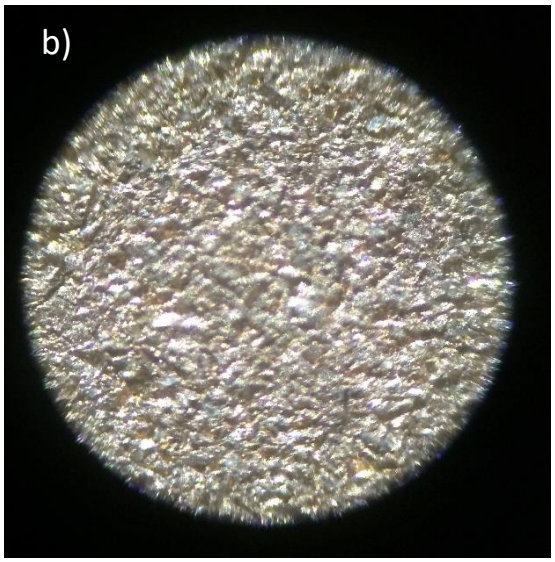

Figure 1b. An optical microscope image (magnification is equal to 180) of beryllium bronze DIN CuBe2 sample after sandblasting under 5 bar

One sample was left as a reference, non-defected specimen. Another 5 samples were sandblasted for 1 minute using 110 μm particles of Al_2O_3 at different pressure ranging from 1 to 5 bar with the step of 1 bar. In Fig.1 surfaces of sample before and after sandblasting are shown. Some differences in surface topography are visible because sandblasting increases roughness on the surface.

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. 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 μm thick titanium window. The source was placed in a special holder with the window directed towards the top. The investigated sample was put on the top of this source. One of two annihilation photons was detected in the HpGe detector. Using LYS-1 code the number of positron which annihilate in source could be estimated [7]. Around 48% of all emitted positrons annihilated in studied sample. The experimental procedure was similar to the one reported in Ref. [9].

The energy of positrons and, consequently, the implantation depth are limited. The average implantation depth can be calculated from the formula [9]

$$\bar{z} = \frac{1}{29.3Z^{0.15}\rho}, \quad (1)$$

where $\bar{z}$ is the mean positron implantation depth in [cm], 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 $\rho = 8.93$ g/cm^3 stands for density of bronze. The estimated average implantation depth is equal to 23 μm. About 63% positrons annihilate in the layer of such thickness from the enter surface, thus main information about positron annihilation comes only from this depth. In order to get information from deeper regions we applied the etching technique. The sample was sequentially etched in nitric acid to remove a layer of about 10 μm thick and after the measurement was performed. Etching does not damage or introduced additional defects, which could disturb the initial state. [10, 11]
The energy resolution of applied detector (FWHM) was equal to 1.20 keV at 511 keV. The spectra were analyzed by SP-16K program. [12] This program allows to obtain two characteristic parameters: S-parameter and W-parameter, which are defined as the ratio of area under the central part of the annihilation line to the whole area under the line and the ratio of area under the wing parts of the annihilation line to the whole area, respectively. According the trapping model S-parameter can be directly linked with the defect concentration, which localize positrons. Briefly, its value increases when concentration increases [8], however the dependency is not linear and can be also depend in the size the defect. The occurrence of different type of defects is reflected in the value of the W-parameter. [13] The energy windows for calculation of S parameter was $|E_\gamma - 511 \text{ keV}| < 1.38$ keV while for W parameter was determined at the energy range between 515 keV and 519 keV.

3. Results

The plot of S-parameter versus compressed depth is presented in Fig. 2. This measurement is accessory and it was done to illustrate the limits of PAS technique sensitivity to defect concentration induced by pressing. S-parameter increases while thickness reduction is less than 15%. After that a plateau appears, however, defect concentration still increases. In this region the increment of S-parameter is comparable with its error. So information about defect concentration cannot be distinguished using PAS. Thus, the limit of PAS applicability was demonstrated.

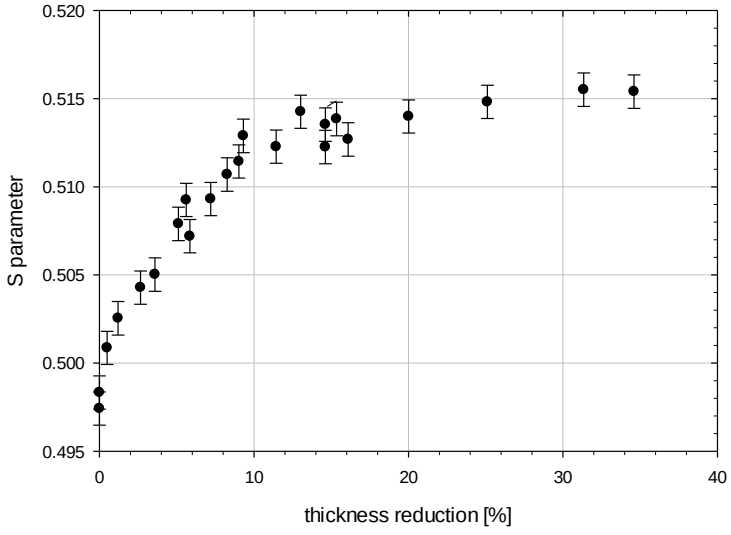

Figure 2. S-parameter in dependency on thickness reduction for a sample of beryllium bronze DIN-

CuBe2 exposed to compressing under the load of up to 15 MPa.

In Fig. 3a the dependency of the S-parameter on etched depth is depicted. For the reference sample (black squares), 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 and etching does not introduce defects, as mentioned above. [10,11]

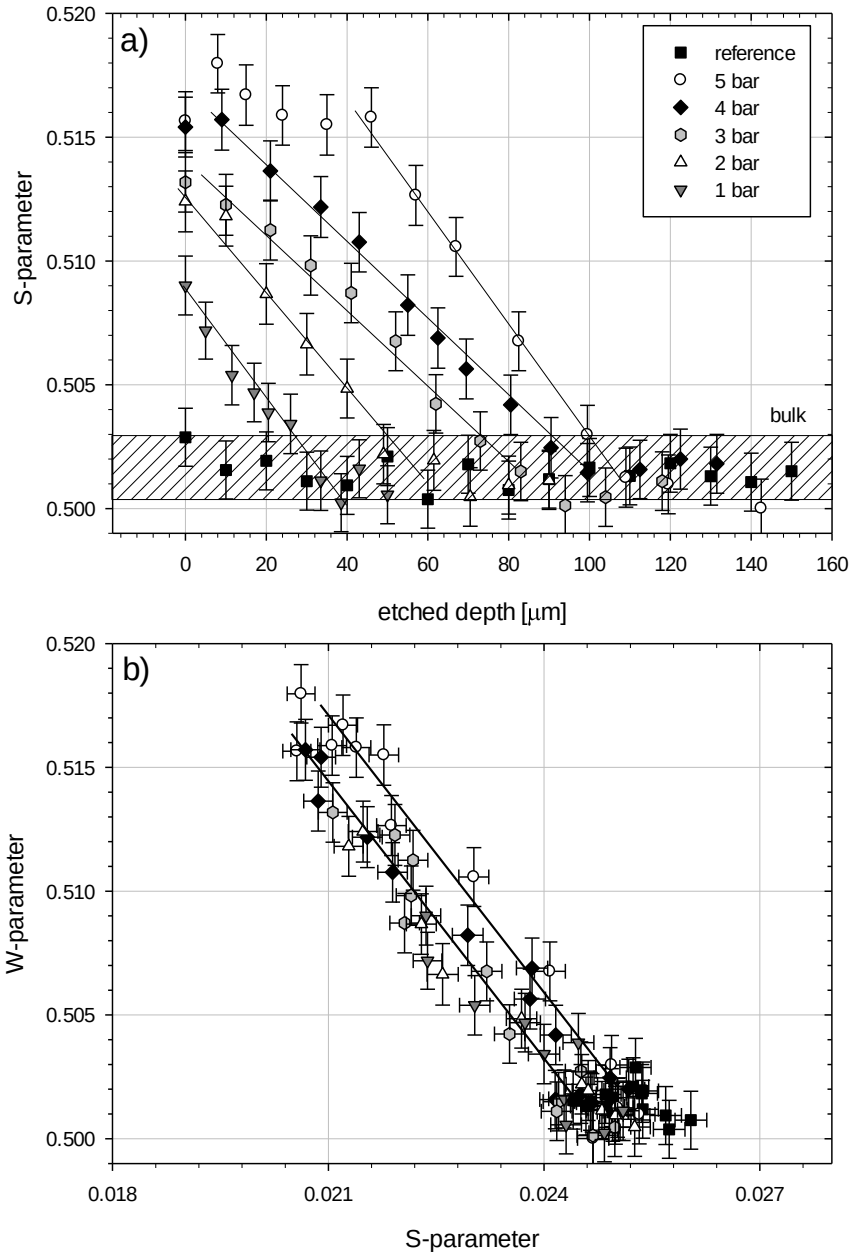

Figure 3. S-parameter in dependency on etched depth (a) and W-parameter versus S-parameter (b) for samples of beryllium bronze DIN-CuBe2 exposed to sandblasting for 1 minute with 250 μm particles of

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).

The value of S-parameter at the surface for all sandblasted samples is larger than for the reference sample because there are induced by defects introduced by this process. Then, for samples sandblasted with pressure from 1 to 4 bar, 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 reference sample. This depth is the total thickness of damage zone in the sandblasting process. 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 under pressure of 5 bars. At the beginning, the value of S-parameter is almost the same as for 4 bars and it is similar to value of maximum S-parameter for the compressed sample. This is the value of S-parameter which reflects to the limiting concentration of defects from which PAS technique does not give information about defect concentration. It is approximately a constant line until the etched depth is less than 50 μm. This means that at the depth of up to 50 μm there are so many defects that all positrons are trapped in the defects and S-parameter saturates. Only after 50 μm the decreasing of the S-parameter is observed. In this sample the estimated thickness of defected zone is 110 μm.

In Fig. 3b S-parameter in dependency on W-parameter is shown for all studied samples. Points representing pressures in the range 1-4 bar are situated along a straight line and it means that in cases the same type of defects is observed. The second slope between surface and bulk appears for sample blasted at 5 bar. It can point out the creation of another kind of defect. Unfortunately, type of defect cannot be defined using Doppler spectroscopy. Using positron lifetime spectroscopy can help to identify them.

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 damage zone in each sample was determined. 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 defect concentration can be so high that PAS technique is unable to determine it precisely.

5.Acknowledgements

This work was done in the frames of Students' Summer Program 2016 in JINR, Dubna.

References

[1] I. Makkonen, E. Korhonen, V. Prozheeva, F. Tuomisto 2016 J. Phys. Condens. Matter **28** 1-7

[2] I. Prochazka 2001 Materials Structure **8** 55-60
[3] P. Horodek, M. Eseev, A. Kobets 2015 Nukleonika **60** 721-724
[4] X.S. Guan, Z.F. Dong, D.Y. Li 2005 Nanotechnology **16** 2963-2971
[5] X.Y. Wang, D.Y. Li 2003 Wear **255** 836-845
[6] E. Dryzek 2003 J. Mater. Sci. **38** 3755–3763
[7] K.Siemek, J.Dryzek, Acta Phys. Polon. A, **125** (2014) 833-836
[8] P. Horodek, J. Dryzek 2016 Acta Phys. Pol. B Proc. Supp. **9** 1001-1007
[9] P. Horodek, J. Dryzek 2010 Nukleonika **55** 17-19
[10] J. Dryzek, Tribol. Lett. 2014 **54** 229-236
[11] J. Dryzek, Tribol. Lett. 2011 **42** 9-15
[12] http://www.ifj.edu.pl/~mdryzek/page_r18.html
[13] P.V. Kuznetsov, Yu.P. Mironov, et.al. 2014 AIP Conference Proceeding **1623** 327-330