POSITRON ANNIHILATION SPECTROSCOPY STUDIES OF SANDBLASTED COPPER*

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The paper presents results of positron annihilation spectroscopy studies of the subsurface zone in technical purity copper samples exposed to sandblasting, compression and milling. Measurements of Doppler broadening of the annihilation line were used to obtain depth profiles of defects and allowed to determine the affected zone range in the samples after given mechanical treatments. The influence of air pressure and sandblasting time was studied and compared with results obtained for milling and compression.

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1. Introduction

Sandblasting is the process consisting in propelling abrasive material particles stream on a surface in order to expel rust or engrave it. It is commonly used to remove paint from mechanical parts or preparing surfaces prior to painting. The stream of particles introduces local plastic deformations in blasted material. Defects that appear in the crystal lattice beneath the treated surface due to this process may influence the properties of the material which are important for its applications. They can be detected by positron annihilation spectroscopy (PAS) technique. In the literature, one can find several papers on utilizing of PAS method in studies of different materials exposed to sandblasting [1–4].

PAS is a non-destructive, well-established tool that is capable of giving information about changes in micro-structure of the deformed subsurface region. Registered annihilation quanta carry information about the

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distribution of electrons' energies and their momentums at the annihilation site. Due to this, PAS can be used for studying the electron structure of metals and recognize regions, where the electron density is disturbed such as open-volume defects. PAS is explicitly sensitive to vacancies, vacancy clusters, microvoids and to some extended dislocations. A positron that enters a sample loses its kinetic energy and reaches the thermal energy in a few ps [5]. Then it diffuses in the crystal lattice and it can be trapped by defects [6]. This results in changes of measured positron annihilation characteristics.

This paper presents application of PAS for probing subsurface zone in technical purity copper samples after air-driven sandblasting. In addition, for the purpose of demonstrating sensitivity of PAS, one sample was compressed and another was subjected to cutting and PAS measurements were carried out.

2. Experiment description

2.1. Sample preparation

Six samples in the form of disks of 8 mm in diameter and 5 mm thick were cut from the refined copper rod with purity 99.9%.

In the first step, samples were polished with abrasive papers of various gradation. To prevent surface oxidation and to obtain samples in the same state with only residual defects, the samples were annealed at 600°C for one hour in vacuum of 10^{-5} Torr and then slowly cooled with the furnace to room temperature. One of the specimens was kept as a reference, bulk sample. The remaining samples were exposed to mechanical treatments.

Samples were sandblasted using Renfert Vario Basic Jet blaster. The process was performed with 125 μm in diameter glass balls which were blasted with 90° impact angle and with the distance of 1 cm between a nozzle and the sample. The influence of sandblasting parameters *i.e.* compression gas pressure and sandblasting time were studied. Surfaces of two samples were blasted for 1 minute under different pressure of 1 or 5 bar. The third specimen was blasted under 5 bar for 3 minutes. Figure 1 shows optical microscope images of the sample surface before and after blasting under 5 bar. A change in surface topology can be observed due to increased roughness caused by sandblasting. The fourth sample was only cut off from the rod using lathe tool with rotation speed of 1000 rev/min. The last of the copper specimens was pressed using hydraulic press with pressure of 15 MPa and its thickness reduction was 72%.

2.2. Experiment details

The PAS measurements were performed at the Joint Institute for Nuclear Research in Dubna using an encapsulated isotope ^{22}Na with activity of 15 μCi. The positron source was sealed in a small, 5 mm in diameter

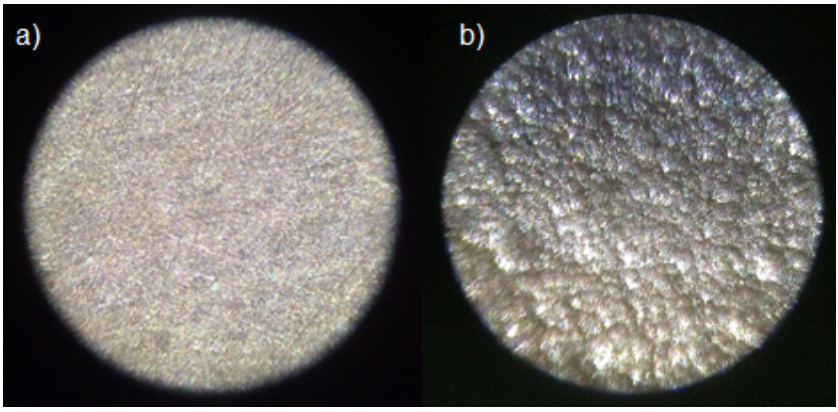

Fig. 1. An optical microscope image of the reference copper sample (a) and the one sandblasted under 5 bar (b) (magnification is equal to 180).

capsule made of copper. Positrons were emitted through a 5 μm thick titanium window placed on the top of the capsule. The positron source with the sample placed on its top was placed directly in front of the high purity germanium (HPGe) detector. Positrons were implanted both to the sample and the capsule. Any registered change in PAS characteristics was caused by changes in the sample material. More precise description of the discussed measurement setup can be found in [7].

The DB of the annihilation line was used in our investigations. This method consists in characterizing the obtained annihilation spectrum by the so-called shape parameters. The most commonly used parameter is the S-parameter which is defined as the ratio of the area under the fixed central part of the annihilation line to the area under the whole annihilation line. This parameter indicates the fraction of positrons which annihilate with low-momentum electrons which are present in open-volume defects. The S-parameter is very sensitive to changes of the density of open-volume defects, which traps positrons. In brief, value of the S-parameter increases with the number of open-volume defects. This dependency is well-described with the help of the positron trapping model [8].

The DB of the annihilation line was measured using HPGe spectrometer with energy resolution FWHM = 1.20 keV at 511 keV. Positron annihilation measurements were performed at room temperature. Each of the acquired spectra was analyzed using SP16K program [9]. This program allows to extract S- and W-parameters after fitting Gaussian curve to measured points with simultaneous subtraction of the background. The energy range used for computation is constant within the entire measurement series. The energy window used for determining the value of the S-parameter was 511 ± 1.38 keV.

Positrons emitted from radioactive ^{22}Na has continuous energy spectrum with maximum energy equal to 545 keV. Thus, positrons probe a certain layer of material. Positrons are implanted at the depth of several dozens of micrometers. The average depth that is penetrated by positrons depends on the density of the medium ρ and slightly on its atomic number Z. For positrons emitted from ^{22}Na source, the average implantation depth $\bar{z}$ can be calculated from the formula [10]

$$\bar{z} = \frac{1}{29.3\, Z^{0.15} \rho}\ [\mathrm{cm}]\,.$$

In the case of pure copper, the estimated average implantation depth is equal to 23 μm. The estimated implantation range is several times smaller than the layer affected by *e.g.* sandblasting. Thus, if the defect profile extends up to a few hundreds of micrometers, it is possible to observe the changes of the S-parameter as a function of the depth from the surface. In order to obtain in-depth profile of positron annihilation characteristics, a thin, around 20 μm, layers of the sample were removed by etching. All of the specimens were sequentially etched in the nitric acid solution in distilled water in order to remove further layers and PAS measurements were carried out. In this way, deeper layers were revealed. After each step, the thickness of the sample was measured using a digital micro-screw with ± 1 μm accuracy.

It was confirmed that sequential removing of layers by chemical etching makes it possible to obtain an in-depth profile of positron annihilation characteristics. Furthermore, chemical etching does not introduce new defects into sample material which could distort the initial values of positron annihilation characteristics [11].

3. Results

Figure 2 presents dependencies of the S-parameter on the depth for the copper sample that was pressed under 15 MPa and the second one cut by lathe machining. This measurements were made in order to demonstrate sensitivity of the PAS method.

In general, samples subjected to different types of cold-working processes exhibit different defect profiles. For the milled sample, the S-parameter and thereby defect concentration decreases with the etched depth and, finally, reaches values of the S-parameter measured for the defect-free, reference sample at the depth of *ca.* 300 μm. The profile of the S-parameter obtained for the pressed specimen differs from that received for milling. Obtained values of the S-parameter are constant in the whole depth of the sample.

Taking into account the sample internal structure, the differences in the profiles are caused by different outcome of the mentioned above cold-working processes.

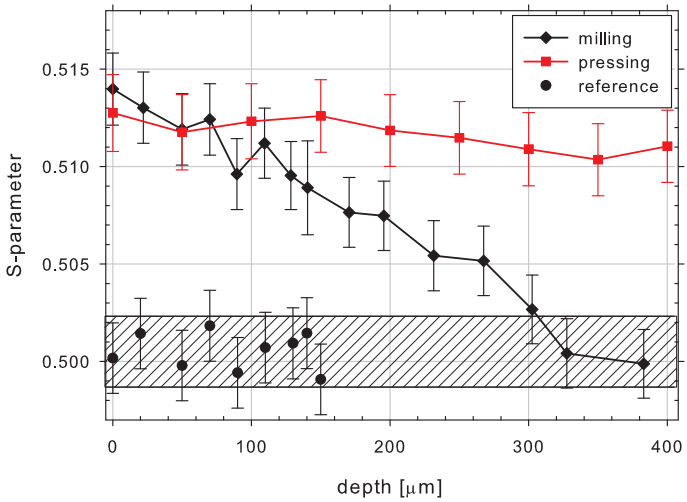

Fig. 2. The dependence of the S-parameter on the depth for the samples subjected to cutting (rotation speed 1000 rev/min, using tool steel lathe, without cooling) and compression (reduction of thickness by 72%). The dashed horizontal area corresponds to the value of the S-parameter measured for the reference, defect-free sample.

During compressing, compressive stresses appear in the whole volume of the sample. They cause creation and movement of dislocations. Dislocations lines are accompanied by large number of vacancies and the range of their movement extends in the sample volume [12]. Therefore, a significant, constant value of S-parameter can be observed in the whole sample volume. On the other hand, during cutting, mainly shear stresses appear which can as well cause movement of the dislocations in the region beneath the cutting surface.

Figure 3 shows results of DB measurements for the reference sample and specimens sandblasted under different pressures. The values of the S-parameter are presented as a function of the depth. The hatched region represents values obtained for the reference sample. Similarly to Fig. 2, the constant values of S-parameter were observed for the reference sample because chemical etching does not introduce additional defects in the interior of the sample.

For the sandblasted samples, the values of the S-parameter close to the surface are higher than for the reference sample. This means that there is formed a subsurface zone with defects introduced by sandblasting. After

sandblasting, higher stresses and plastically deformed region are expected near the surface similar to cutting. However, the unusual increase in the S-parameter in a layer at some depth close to the surface is observed.

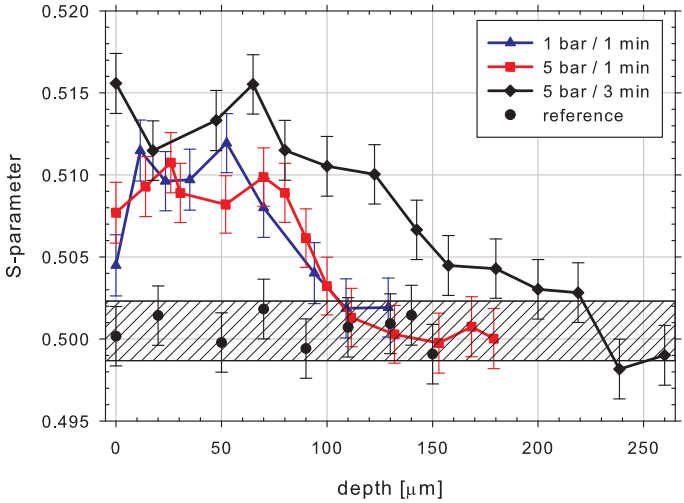

Fig. 3. The S-parameter as the function of the depth from the surface for samples of copper exposed to sandblasting with 125 μm glass particles for different pressure and time. The hatched region represents the values obtained for the reference sample.

It is clearly visible for the samples that were sandblasted for 1 minute. The value of the S-parameter increases starting from the surface to a depth of *ca.* 50–80 μm. Then, beyond this specific depth, the value of the S-parameter decreases almost linearly as the depth increases. Hence, the number of defects is decreasing with the depth from the sandblasted surface. For the sample sandblasted under pressure of 5 bar for 5 min, the maximum of the S-parameter value is also visible at the depth of 70 μm.

For all the sandblasted samples, the S-parameter values are decreasing to the values received for the reference sample which means the end of the subsurface zone induced by blasting. The size of the subsurface zone depends mainly on the time of blasting. For the sample that was sandblasted under pressure of 5 bar for 3 min, the depth of the subsurface zone is the largest and equal to *ca.* 240 μm. For the specimens that were sandblasted for 1 min under the pressure of 1 bar or 5 bar, the depth of the subsurface zone is similar and equal to *ca.* 110 μm. Similarity of these two profiles can be explained in the following way: The higher pressure should cause an increase of the defect concentration below the surface. However, the higher pressure causes also higher erosion of the surface. In this way, the most deformed part of the sample is removed, and similar profiles are generated.

The layer with the higher values of the S-parameter close to the surface of the samples may be caused by the presence of glass particles that were used in sandblasting. It is likely that they may have been embedded in the sample material. A similar phenomenon has been observed by Horodek *et al.* [13] in samples of pure copper that were blasted using aluminum oxide particles of size of 250 μm. Positrons implanted into the sample can annihilate both in copper and in the particles used for sandblasting which could get inside the sample material. It should be noticed that the S-parameter values are derived from all possible annihilation states from the sample volume. This means that the increase in the S-parameter value can come from the positron annihilation in glass particles. However, Kurdyumov *et al.* [14] who studied samples of beryllium bronze sandblasted using 110 μm particles of Al_2O_3 did not report abrasive particles deposition inside the samples.

4. Conclusions

Sandblasting of copper samples results in creation of the subsurface zone whose properties differ from those of the bulk material. During this process, dislocations and point defects such as vacancies were created which was observed in the S-parameter changes. PAS measurements accompanied by etching of the sample layers allowed us to determine the range of the subsurface zone created in the specimens. It can be noticed that for the sandblasting parameters studied, the size of the created subsurface zone depends mostly on the sandblasting time. The differences between sandblasting pressure were negligible. For the specimens that were sandblasted for 1 min under the pressure of 1 bar or 5 bar, it was *ca.* 110 μm. For the sample that was sandblasted under the pressure of 5 bar for 3 min the depth of the subsurface zone was equal to 240 μm. An increase in the S-parameter value just beneath the surface may be caused by the presence of glass particles retained in the samples material.

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