Slow positron beam studies of zirconium exposed to dry sliding

To cite this article: Jerzy Dryzek and Pawel Horodek 2017 *J. Phys.: Conf. Ser.* **791** 012020

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Abstract. Slow positron beam studies of Zr samples exposed to dry sliding are reported. The observed increase of the annihilation line shape parameter indicates generation of vacancy clusters although an increase of the positron diffusion length suggests recovery process indicated by sliding. The increase of the number of positronium created on the worn surface in comparison to the non damaged surface was noticed.

1. Introduction

Technological treatments of surface by ion implantation [1], laser treatment [2], blasting [3], polishing [2], cutting [4], sliding, and etc. induce elastic and plastic deformation which stands behind the crystalline defects generated in the subsurface region. These defects are distributed like strain in a certain volume of the samples adjoining the surface and finally affect physical properties like hardness and performance of products. Thus the knowledge about defect distribution can be significant from practical point of view. Sliding contact accompanies many technological processes. The defect depth distribution induced in such a case is not well recognized especially at the depth close to the worn surface. One can expect that for instance a flow of heat accompanying friction can significantly affect defectstructure causing recovery the zone adjoining the surface. Another process is the mass transport at the worn surface between contra parts in sliding contact.

The experimental technique which is suitable for studies of defects close to the worn surface is the slow positron beam technique. The application of slow positron beam to study of subsurface defects and other phenomena is a natural extension of conventional bulk spectroscopy employing beta decay positrons. In this case the low energetic positrons are injected at the depth of about 1 μm and after thermalization they annihilate with electrons. Open value defects like, vacancies, their clusters and dislocation with jogs can localize positrons. The annihilation with low energy electrons which occupy such defects is reflected in the annihilation radiation parameters like Doppler broadening of annihilation line shape parameter or mean positron lifetime. This is a convenient method for observation of defects and changes of their concentration.

The constant defect concentration at the depth less than 350 nm in gold exposed to dry sliding was observed [5]. Additionally the concentration of vacancies increases with the increase of the applied load. This indicates that the zone at such a depth is uniform. However, for copper a maximum at a depth between 300 and 500 nm in defect concentration was resolved using the VEPFIT code [6]. In other cases the defect concentration at this depth remains constant. This indicates that sliding causes close to the surface only the deep plastic deformation uniformly distributed. To confirm this we intend to perform similar studies for pure Zr. This metal has poor tribological properties thus one can expect new features which are not present for other studied metals commonly applied in tribojunctions.

Zr has only niche applications. Its excellent resistance for corrosion allows its using as an alloying element in materials that are exposed to aggressive environments, for instance alloys for surgical instruments, chemical and biomedical industries. Because of low neutron-capture cross section of Zr nuclei, zircaloys are used for cladding of nuclear reactor fuels.

In the studies the variable energy positron beam (VEP) is applied to studies of Zr exposed to dry sliding.

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2. Sample preparation

In our studies we used Zr purity of 99.2% purchased from Goodfellow. Samples had a shape of discs of 10 mm in diameter and 3 mm thick each, their surfaces were polished. Before treatments they were annealed in the flow of N_2 gas at a temperature of 550 $^{\circ}$C for 1 hr, and then slowly cooled to room temperature. After annealing, the samples were etched in the etching paste TS-K 2000 delivered by Pelox to reduce their thickness by 10 µm and clean their surface. After this procedure only residual defects were present. In fact, for virgin samples the measured positron lifetime spectra contained only one lifetime component equal to 162±1 ps. This corresponds well with the data reported in the literature as the bulk lifetime for Zr [7] .

One of a virgin sample of Zr was located in a tribotester and the base surface of the sample was sliding against the rotating disc with the load of 25 N. This disc of diameter of 50 mm was made of the martensitic steel (steel SW18 hardness about 670 HV0.1). The speed of the disc relative to the surface of the sample was about 5 cm/s. The treatment was performed in air during a certain time, no oxidation was observed.

3. Measurements details

Doppler broadening (DB) spectra of studied samples were measured at room temperature using VEP at JINR. Positrons formed in the 5 mm diameter beam with intensity of 10^5 e+ /s were implanted with incident energy range between 50 eV and 36 keV. Annihilation gamma quanta were registered with high purity germanium (HPGe) detector whose energy resolution was equal to 1.20 keV at 511 keV.

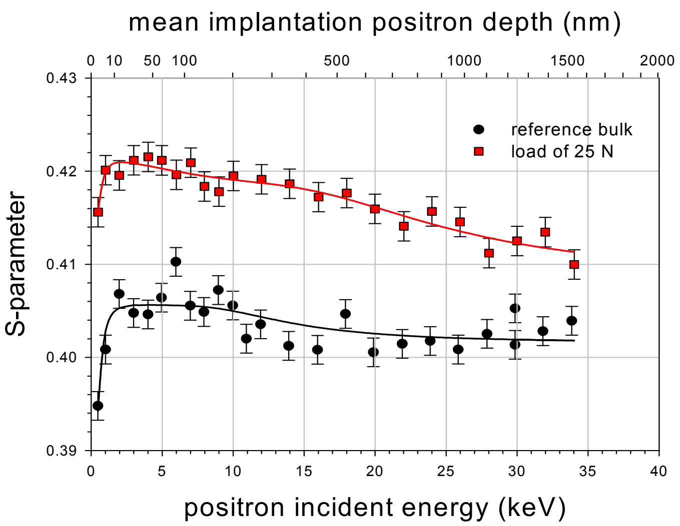

Figure 1. The dependency of the S-parameter as the function of the positron incident energy in VEP experiment measured for reference (closed circles) and exposed to the dry sliding (closed squares) samples. The solid lines represents the best fits using VEPFIT code.

4. Results and discussion

Obtained dependencies of the S-parameter as a function of the positron incident energy are depicted in Figure 1. This parameter is defined as the integral of the central part normalized to the total area of the measured annihilation line. It is well proved experimentally that this parameter is extremely sensitive to the presence of open volume defects. The values of the S-parameter for the sample exposed to dry sliding (closed squares) are placed above the results for the reference sample, (closed circles). However, the dependencies do not like results obtained for other metals like steel [8] or copper [6] where the continuous decrease of the S-parameter values takes place. Increase of the S-parameter is caused by the presence of a very thin, only few atoms layer, mainly oxides on the surface, which reduces the value of the S-parameter on the surface, as it observed for silicon [9].

For description of the dependencies VEPFIT code was used [10]. The Makhovian function was used as the positron implantation profile with the following parameters: *m*=1.720, *n*=1.667 and *A*=2.84 $\mu g/cm^2 keV^n$, which corresponds to the values for copper [11]. The mean positron implantation depth is depicted in the top axis in Figure 1 The both samples exhibit an increase of the S-parameter at low energies less than 4 keV, i.e., at the depth of c.a.10 nm, followed by a gradual decrease for higher energies. In the air due to the passivation process the surface of the Zr is covered by thin ZrO_2 monolayer which shield the interior. The S-parameter in this layer is lower than in the metal. The striking feature of the obtained results clearly visible in Figure 1 is the increase of the value of the S-parameter at the surface, from 0.4058(10) to 0.4215(6) caused by sliding. Thus sliding significantly changes the properties at the surface. However, the best description of the obtained dependencies requires assumption of the presence of two layers. The value of the S-parameter in the adjoined surface layer also increases, from 0.4055(5) to 0.4180(5) for the samples exposed to dry sliding. The thickness of this layer is about 400 nm for the reference sample and 1060 nm for the second sample and positron diffusion length obtained from the fitting procedure are as follows: 51(26) nm and 130(40)nm, respectively. This is a surprising results. On one point the rise of value of the S-parameter indicates the increases of the defects concentration but this is not accompanied of reduction of the positron diffusion length, as one should expect. This can be explained by recovery process induced by sliding, what can cause the generation of vacancy clusters for which the S-parameter has a large value. The value of the S-parameter deeper than 400 nm for reference sample and 1060 nm for the sample after sliding are about 0.4016(4) and 0.4085(8), and corresponding positron diffusion length are as follows: 3 nm and 44 nm, respectively. This indicates presence of open volume defects due to the significant deformation in the latter sample.

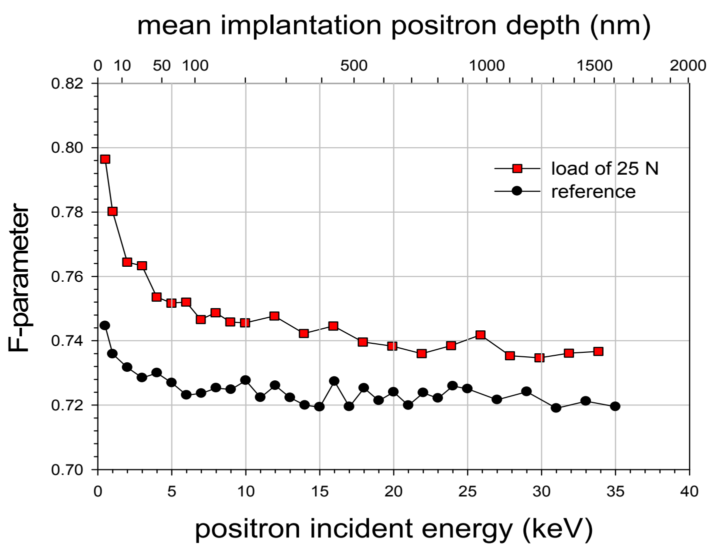

Figure 2. The dependency of the F-parameter as the function of the positron incident energy for the reference sample of Zr and the sample exposed to dry sliding.

It is well known that at the surface of metals positronium, i.e., the bound state between electron and positron can be created. Three quanta annihilation at of this state is reflected in the increase of the so called F-parameter, called “valley to peak” extracted from the energy spectrum of the measured photons. Energy spectrum of quanta from triplet annihilation is continuous from zero to about 511 keV, thus they contribute to the “valley” region in the front of 511 keV peak. This parameter is defined as number of counts in the energy range between 340 and 504 keV to the number of counts in the range from 504 to 518 keV. In Figure 2 the dependencies of this parameter as a function of the positron incident energy are depicted. One can notice the increase of this parameter for the sample exposed to dry sliding in comparison to the reference sample. This indicates that the positronium is created at the worn surface with higher probability than at the surface of the reference sample. This can be caused by the presence of surface defects and/or oxides. The reason can be that the worn surface is more rough after sliding affecting the amount of positronium creation.

5. Conclusions

Slow positron beam studies of pure Zr exposed to the dry sliding were performed. The increase of number of vacancy clusters in the region adjoin the worn surface was detected. The increase of number of positronium at the worn surface was also observed.

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