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POSITRON LIFETIME STUDIES OF THE SUBSURFACE ZONES CREATED UNDER DRY AND LUBRICATION CONDITIONS

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We present the measurement of the positron lifetime spectra in the subsurface zones in copper samples with surfaces damaged in dry and lubrication conditions. We observe that close to the damaged surface the single vacancies were present, but deeper in the sample treated in the dry condition the vacancy clusters were detected. A zone with dislocations was located deeper than the zone with the vacancies and their clusters.

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1. Introduction

Positron annihilation (PA) as a method for studies of open volume defects provides unique information about defects which can appear upon plastic deformation. This is due to the fact that during this process various types of defects like dislocations and vacancies are created in high concentration. Additionally, positrons represent a selective, nondestructive probe for such defects. In the literature many experimental results concerning plastic deformation were reported [1]. However, there are several theoretical papers in which authors calculated positron annihilation characteristics for positrons localised in certain types of defects [2]. For several years the PA method has been conveniently used also for the study of the subsurface zone created during friction and wear processes [3, 4]. This zone is located close to the damaged surface and, as it was shown, its thickness ranged from 50 μm up to 400 μm, depending on conditions, and exceeds the average positron implantation depth. This allows us to use conventional techniques where positrons are emitted from a radioactive source.

In our former studies we have detected the subsurface zone created upon dry sliding of two metals. On the other hand, in practical applications lubrication is used for a reduction of friction and wear. The presence of a lubricant between the surfaces of the tribo-elements causes that the shearing takes place inside the lubricant and its hydrodynamic properties mainly determine the drags of the motion. When the external load is not compensated completely by the hydrodynamic pressure inside the lubricant layer, the surfaces are covered by lubricant thin films of molecular thickness and a considerable interaction of surface roughnesses must occur. This situation leads to increased friction and wear.

In this paper we intend to show that PA method is sensitive enough to differentiate between the subsurface zone created under dry and lubrication conditions.

2. The sample preparation and measurement procedure

In our studies we have used the technical purity copper samples in the form of plates with dimensions 2 cm $\times$ 1.5 cm and thickness 2 mm. Prior to the experiment, all samples were annealed at 950°C for 24 h in vacuum (10^{-5} mbar) and slowly cooled to room temperature during two weeks. This procedure allowed removing almost all defects from the raw material. The measurement of the positron lifetime in the annealed samples has shown the value of $\tau = (117 \pm 1)$ ps, which indicated that positrons were annihilating in the defect-free sample. After this, the samples were mounted into the pin-on-disc tester and their surface was damaged by the motion of a small steel (100 Cr6/martensite, hardened) ball having 4 mm in diameter. The ball was pressed by a load perpendicularly to the surface of the samples. The load was equal to 150 G. Eight traces, 1 mm in distance, were made on the surface of each sample in order to enlarge the treated area. In all cases the speed of the ball was kept constant (7 cm/s). Each trace was done within 30 min. In our studies we damaged the copper surface under the dry condition in air and the next samples were treated in the presence of a lubricant. As a lubricant we have the good quality gear oil Hipol 15F GL-5 SAE 85W/90. The samples and the ball were immersed in the oil during tribotests. After the tribotest and before positron lifetime measurements the samples were cleaned with methanol and detergents.

We scanned the subsurface zone of the samples up to the depth 350 μm by sequential measurements of the positron lifetime spectrum after stepwise etching in nitric acid of *ca*. 20 μm thick layers from the surface of the samples. This had to be done because the positrons emitted from the radioactive source are implanted up to the depth of about 28 μm in copper. This technique was also applied in our former studies [3].

The lifetime spectra were measured using a conventional *fast-fast* lifetime spectrometer with NE111 plastic scintillators with a time resolution (FWHM) of 300 ps for the ^{22}Na energy window. Data were analysed using the computer program called LT [5]. In the analysis of the positron lifetime spectra the background and the source corrections were taken into account.

3. The discussion of the experimental results

For the sample with the surface slid without any lubricant the spectrum contained two lifetime components when the depth was smaller than 108 μm. But deeper in the sample only one lifetime component was sufficient to get the chi square close to unity. In the case of the sample with the surface slid with

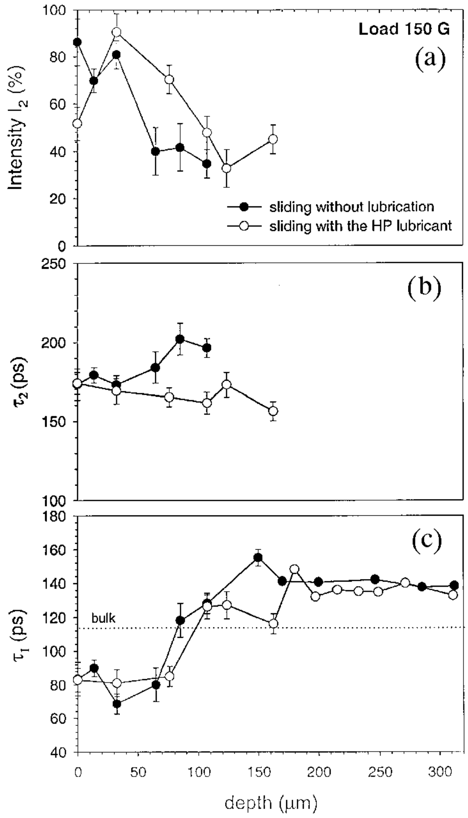

Fig. 1. The dependencies of the first (τ_1) and second (τ_2) lifetime component and the intensity I_2 of the second lifetime component on the depth (thickness of the etched layer). The measurements were performed for the well annealed copper samples with the surfaces damaged in the tribotest under dry conditions and with lubrication conditions. The pin was loaded with the load 150 G for both samples. The dashed line presents the value of the bulk positron lifetime obtained for the well annealed sample.

the lubricant, two lifetime components were present at the depths smaller than 162 μm.

The results of the deconvolution procedure were summarised in Fig. 1. The second lifetime component at the depths smaller than 33 μm for both samples is close to 174 ps, which can be associated with positron annihilation at a single vacancy [6]. Such vacancies were induced by the motion of dislocations with jogs. The drugging of jogs on the dislocation with dominating screw character produces strings of vacancies (or interstitial atoms) [7] which may disintegrate into string-like clusters. Formation of such clusters occurring only for the sample with the surface was damaged in the dry condition.

At the depth above 69 μm the second lifetime component increases visibly up to 202 ps, which suggests that the vacancy clusters consist of two or three. We observed a similar effect in our previous studies [8] when graphite rod damaged the surface of the copper sample. It is interesting that this effect was not observed for the sample with the surface damaged upon the lubrication condition. The intensity of the second lifetime component for this sample was higher than for the sample slid in the dry condition. The explanation could be that in the second case single vacancies start to aggregate and this is the reason of an increase in the second lifetime component. The aggregation could be caused, e.g., by an increase in the temperature which could be higher during dry sliding compared to sliding with the lubricant.

The first lifetime component at depths below 85 μm is smaller than the bulk value, which is well understood within the standard trapping model. If we assume that the trapping efficiency of positrons at a single vacancy is equal to 5×10^{14} 1/s [6], then from τ_1 or I_2 we can evaluate the vacancy concentration *ca.* 9×10^{-6} in this region of the sample. It is interesting that above 180 μm the first lifetime component does not drop to the bulk value (117$\pm$1 ps) for both samples. The lifetime in this region was slightly above 130 ps, which can be associated with positron localisation at the dislocation lines. Thus in this region the production of vacancies and their clusters was stopped. Probably the density of dislocations close to the surface is much higher than deeper in the sample. The concentration of dislocation lines influenced the creation of jogs at their lines. Jogs appear when two dislocation lines cross each other [7]. In the region above 180 μm the dislocation concentration was too small for the creation of jogs and consequently vacancies.

In conclusion, we can point out that positron lifetime measurement distinguishes the significant differences in the defect structure of the subsurface zones created under the dry or lubrication condition. In the sample with the surface damaged under the lubrication condition we have observed a less efficient process of vacancy aggregation. The depth range of the region where vacancies or their clusters exist is smaller for the sample with the surface damaged under the dry condition than for the sample treated with the lubricant. Beneath the zone with vacancies we have detected a zone where mainly the dislocations are present.

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References

[1] T. Wider, S. Hansen, U. Holzwarth, K. Maier, *Phys. Rev. B* **57,** 5126 (1998).

[2] H. Häkkinen, S. Mäkinen, M. Manninen, *Phys. Rev. B* **41,** 12441 (1990).

[3] J. Dryzek, E. Dryzek, T. Stegemann, B. Cleff, *Tribology Lett.* **3,** 269 (1997).

[4] J. Dryzek, E. Dryzek, B. Cleff, *Appl. Surf. Sci.* **116,** 236 (1997).

[5] J. Kansy, *Nucl. Instrum. Methods Phys. Res. A* **374,** 235 (1996).

[6] H.-E. Schaefer, *Phys. Status Solidi A* **102,** 47 (1987).

[7] P.B. Hirsch, D.H. Warrington, *Philos. Mag.* **6,** 735 (1961).

[8] J. Dryzek, A. Polak, *Tribology Lett.* **7,** 57 (1999).