

STUDY OF THE DEFECT STRUCTURE'S EVOLUTION OF THE TITANIUM ALLOY VT1-0 AFTER THERMOHYDROGEN CYCLING BY POSITRON ANNIHILATION SPECTROSCOPY

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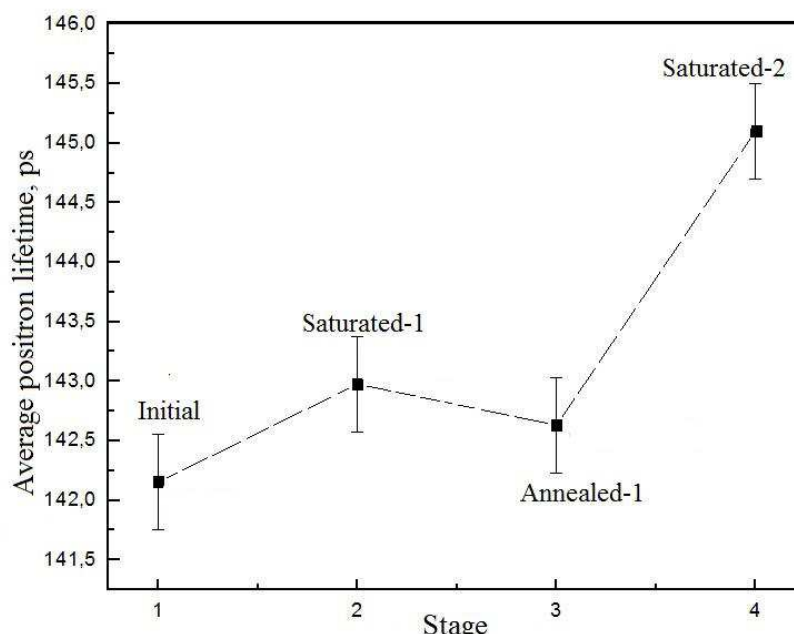
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Changes of the defect structure in titanium alloy after thermohydrogen cycling were studied by means of positron lifetime spectrometry and Doppler broadening spectrometry.

Titanium and its alloys having properties such as light weight, high specific strength, fracture toughness, corrosion resistance, have been widely used as a structural material. Using of materials in the chemical, nuclear, oil and gas industry, as well as the aviation industry is accompanied by interaction of materials with hydrogen. Under the influence of hydrogen take place the significant changes of the physical and mechanical properties of the metal and its alloys, which lead them to embrittlement and destruction [1,2].

The high-temperature annealing in a vacuum is the most reliable method for the extraction of hydrogen from the metal but the impact of this treatment (accumulation of hydrogen - thermal annealing) on the defect structure of titanium alloys is still open.



– the result obtained with [18])Fig. 2. The spatial distribution of energy release power density across the focused ion beam (10 keV Ar⁺ ion beam with the diameter of 50 nm is incident on Al-target)Figure 1.-

The purpose of this work is the studying of Defect structure's evolution of the titanium alloy VT1-0 after thermohydrogen cycling. As the methods of research we used methods of electron-positron annihilation (EPA), which have high sensitivity to determine the concentration and type of defects. Also the positron is the unique

probe to study the behavior of the proton in hydrogen-absorbing matters, for both particles has the same positive elementary charge. In perfect crystals, positrons delocalize in interstitial sites and annihilate with electrons there, since positrons are strongly repelled by ion cores due to Coulomb repulsion, as well as protons. Then, positron annihilation characteristics are determined by the local electronic structure at the interstitial sites, where protons also stay stable [3].

For research we used rectangular flat samples of titanium alloy VT1- 0 with dimensions of $20 \times 20 \times 1$ mm. Samples were mechanically ground, polished and annealed at 750°C for 60 minutes followed by slow cooling to room temperature in vacuum. The hydrogen saturation was conducted by the method of Siverst to 0.05 wt. % at 500°C at a pressure of 0.66 atm. Annealing and hydrogenation were carried out on the installation Gas Reaction Controller [4]. After every processing stage the samples were measured for positron lifetime and the Doppler shift of the annihilation line. [5]

The positron lifetime was measured with a spectrometer, described in detail in [5]. The essence of the method is shown in Fig. 3 and is based on measuring time interval between two events: birth and annihilation of the positron. For measurement of positron lifetime usually used the β^+ -source which also radiate secondary γ -quantum simultaneously with positron. This γ -quantum is a start signal on the timeline and it witnesses the birth of the positron. Stop signal is one of the two annihilation γ -quanta with energies of 511 keV. Relatively low activity sources of positrons allows to distinguish the Start and Stop pulses corresponding to one positron, because it is assumed that at each time point in the material is no more than one positrons.

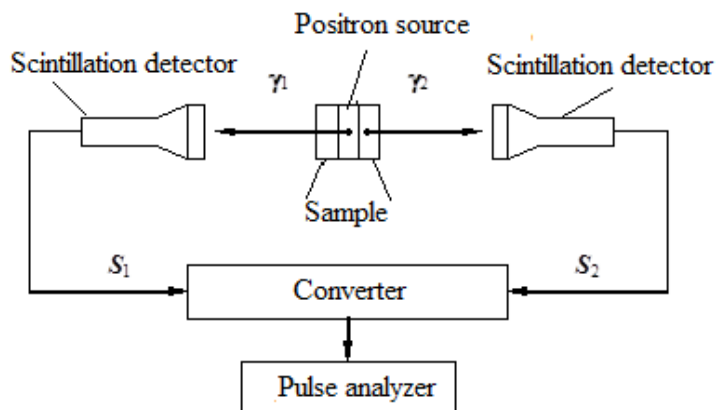


Figure 3.- The measuring principle of the positron lifetime in the material.

Fig. 1 shows the dependence of the average lifetime of positrons from processing stage. The graph shows that the value of the average lifetime of positrons is practically unchanged after first hydrogenation and subsequent annealing. However, a sharp increase in the value of the average lifetime of positrons to $145,5 \pm 0,4$ ps is observed after re-hydrogenation. Average positron lifetime characterizes the cumulative impact of different types of defects on the electronic density of the material. More information about the defects after every processing stage gives multicomponent decomposition of the time spectrum.

Table 1.

Values of the two-component decomposition of time distribution

Processing stages			Positron lifetime components, ps		Intensity of components, %
			τ_1	τ_2	I_2
Initial			142,2	–	–
Saturated-1			142,8	301,8	0,10
Annealed-1			142,6	–	–
Saturated-2	144,4	656,6	0,16		

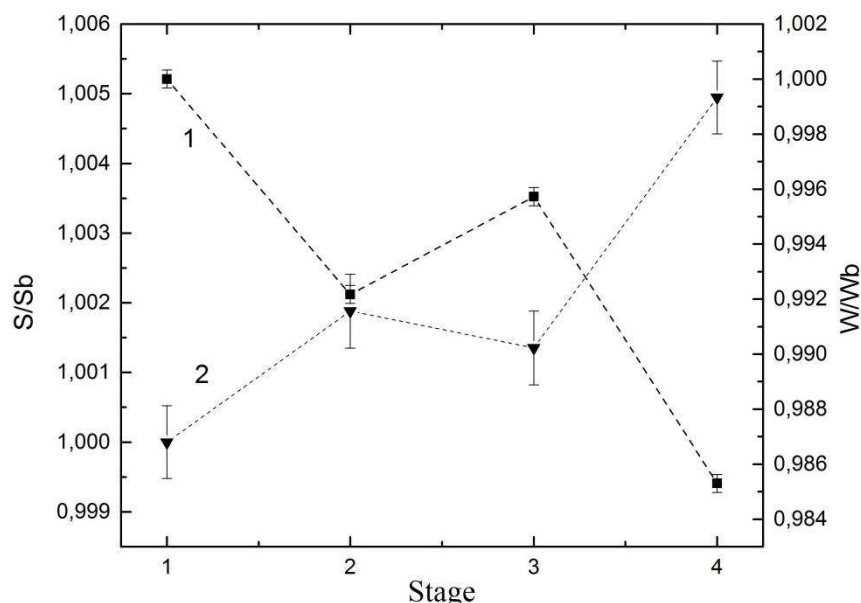


Figure 2. - Dependence S/Sb and W/Wb DUAL shape parameters from the processing stages. 1 - relative parameter S/Sb ; 2 relative parameter W/Wb .

The components and their intensities allow obtaining the qualitative and quantitative information about the type of defects and their concentration. For analyses we used two time components: short-lived component τ_1 and the long-lived component τ_2 and the corresponding intensities I_1 and I_2 . Component τ_1 is associated with the annihilation of positrons from the delocalized state in the lattice of the metal, and long-lived component τ_2 corresponds to the annihilation of positrons from a localized state in the vicinity of defects metal. Table 1 shows the results of the time spectra's processing. Hydrogenation leads to the appearance in the time spectra of long-lived component with a lifetime 301.8 ps, this value is much greater than the value of the positron lifetime in a single vacancy $\tau_{vac} = 220$ ps and corresponds to the annihilation of positrons trapped vacancy clusters consisting of four single vacancies [6]. After the second cycle of hydrogenation observed a significant increase of long-lived component, its value reaches 656.6 ps, which is probably due to the formation of micropores. Compared to the first cycle of hydrogenation the intensity of the component does not increase significantly.

Conservation of momentum during the annihilation process is the reason for the fact that the annihilation radiation contains information on the electron momentum distribution at the annihilation site. This can be used for the study of the electron structure in solids and for the investigation of defects.

In the analysis of Doppler broadening spectra of annihilation line the shape parameters S and W are used. S

parameter is defined in the low-energy and characterizes the probability of positron annihilation with the valence electrons. W parameter is defined in the field of high energy and characterizes the probability of positron annihilation with the core electrons [7]. For analysis S and W are commonly normalized to the values for a defect-free material S_b and W_b respectively. This approach allows more contrastly compare test samples with respect to the initial (zero-defect) sample.

Fig. 2 shows the dependence of relative parameters S/S_b and W/W_b of processing steps, and where S_b and W_b parameters of the original sample. The graph shows that when the sample is saturated with hydrogen the parameter S/S_b increases and W/W_b decreases. These changes of the annihilation characteristics are due to the formation of vacancy clusters. After annealing, one observed a decrease of the S_b parameter and an increase of the W parameter, which indicates the reduction of the number of defects in the sample. At this stage, it is seen that the defective structure of sample does not return to the initial state. Re-saturation causes a significant rise of the S parameter and drop of W parameter that corresponds to a sharp increase of number and sizes of defects in the sample. These results are in good agreement with the results of analysis by spectrometry average positron lifetime.

In the course of this work there was a study of the defect structure changes of titanium alloy VT1-0 for thermohydrogen cycling by EPA methods. The results analysis over spectrometry of positrons of average lifetime and Doppler broadening of the annihilation line showed that at saturation of titanium with hydrogen to 0.05 wt.%, the defects appear in the sample: after the first of hydrogenation the vacancy clusters are formed, which consist of four single vacancies, after the second cycle the micropores appear.

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