LASER PEENED AUSTENITIC STAINLESS STEEL STUDIED BY POSITRON ANNIHILATION SPECTROSCOPY*

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The paper presents preliminary results of positron annihilation spectroscopy studies of the subsurface zone created by Laser Shock Peening (LSP) in medical grade AISI 316L stainless steel. The positron lifetime measurements and variable energy positron beam were used to analyse LSP samples.

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1. Introduction

Laser shock peening (LSP) is a proven surface modification technique used to enhance properties of metallic components. The laser pulses heat and ionize the target surface or ablative layer (*e.g.*, black paint or adhesive tape) turning it into rapidly expanding plasma which generates a high-pressure shockwave in the target material. This process results in plastic deformation, heating-induced changes in the microstructure, and compressive residual stresses. All that leads to changes in the material properties (*e.g.*, hardness or corrosion resistance) [1].

Plastic deformation of the subsurface zone induces crystal lattice defects which can be detected by Positron Annihilation Spectroscopy (PAS) techniques. PAS is sensitive to the electron structure of the material and recognizes regions where the electron density is lowered such as open-volume defects [2–4].

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The aim of this research is to determine the defect depth profile in the subsurface zone induced by LSP in AISI 316L stainless steel which is widely used in biomedical applications.

2. Experiment details

The polished samples of AISI 316L stainless steel were annealed for 1 h at 1000°C in flow of nitrogen. The LSP was carried out on the sample surface (covered with 50 μm thick layer of black paint) in the points grid (0.64×0.64 mm^2) using the Q-switched Nd YAG laser system operating with the following parameters: wavelength: 1064 nm, frequency: 10 Hz, pulse length: 10 ns, energy: 0.55 J, spot diameter: 2.7 mm.

The Positron Annihilation Lifetime Spectroscopy (PALS) measurements were performed with the ^{22}Na isotope using the fast–fast spectrometer based on BaF_2 scintillators with the time resolution of 260 ps. The analysis of the obtained spectra with more than 10^6 counts was made with LT program [5].

To obtain the depth profiles, the LSP-treated samples were sequentially etched in glyceregia etchant and PALS measurements were carried out. It was established that etching does not introduce new defects that may affect positron characteristics. To investigate the 1 μm thick layer close to the LSP-treated surface, the Variable Energy Positron Beam (VEP) was used at the Joint Institute of Nuclear Research in Dubna [6]. Positrons with incident energy range between 0.01 eV and 32 keV were implanted into the samples studied. Doppler broadening (DB) spectra were measured at the room temperature using HPGe detector with 1.2 keV energy resolution at 511 keV. From the DB spectra, the so-called S-shape parameter was obtained [4].

The X-ray diffraction (XRD) patterns were registered using the X'Pert PRO Materials Research Diffractometer by PANalytical. The copper radiation was used for these measurements performed on the LSP surface at the Bragg–Brentano geometry over the 2 theta range of 20–120°, step 0.02°. The High Score Plus v. 305 software by PANalytical B.V. was used for the XRD patterns analysis.

3. Results and discussion

Clear peaks from austenite (γ) are shown in the XRD patterns of the LSP and reference samples. Additional small peaks of deformation induced martensite (α') can be seen for the LSP sample (Fig. 1).

The Williamson–Hall [7] analysis shows that LSP significantly reduces the size of the crystallites from 2735 ± 194 nm for the reference to 24 ± 13 nm for the LSP sample.

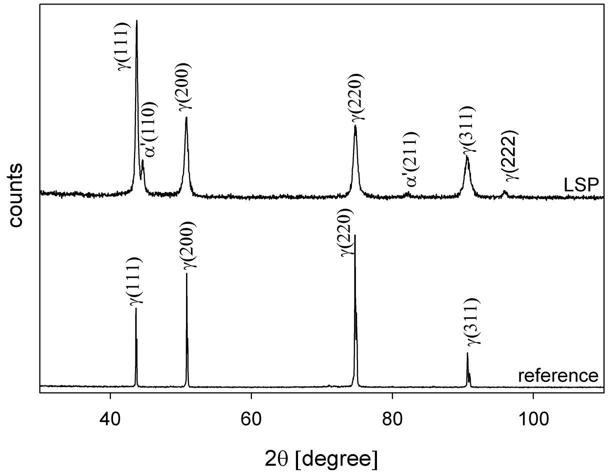

Fig. 1. X-ray patterns of AISI 316L samples.

Two components were detected in the measured PALS spectra for the LSP samples in the subsurface layer up to the depth of 13 μm (*i.e.*, τ_1 and τ_2 — see Fig. 2). Both components are longer than the reference bulk lifetime

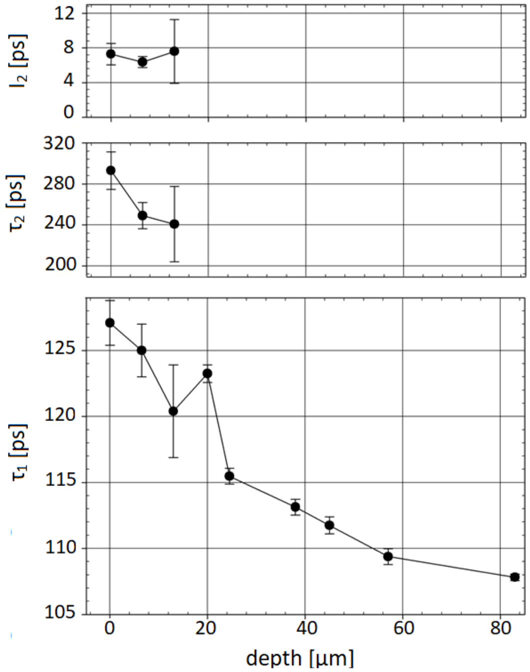

Fig. 2. Positron lifetime components τ_1 and τ_2 and intensity I_2 of the longer component *versus* depth.

value ($\tau_{\text{bulk}} = 106$ ps). The first lifetime τ_1 can be attributed to positron trapping by dislocations. The second lifetime ($\tau_2 = 240 \div 300$ ps) indicates the presence of clusters which consist of two to five vacancies. For the depth larger than 13 μm, only one component in the spectra was resolved. The mean positron lifetime decreases with the depth reaching the bulk value at about 83 μm which can be considered as the total depth of the LSP induced changes. This is much shorter than the range observed *e.g.* for cutting [8].

The values of the S parameter *versus* the energy of the incident positrons, obtained using VEP are presented in Fig. 3. The S parameter decreases with the positrons energy and then saturates. The level of saturation is higher for the LSP sample which points out a higher concentration of defects in comparison to the reference sample. The mean implantation depth z can be estimated using the formula: $z = \frac{AE^n}{\rho}$, where E is the positron energy in keV, $A = 2.62$ μg cm^{-2}keV^{-n}, $n = 1.692$ are Makhov's parameters for iron [9], and $\rho = 8$ g/cm^3 stands for density. VEPFIT code [10] was used for fitting the model function. The obtained positron diffusion length equals $L_{+\text{ref}} = 84 \pm 2$ nm for the reference sample and $L_+ = 60 \pm 1$ nm for the LSP sample.

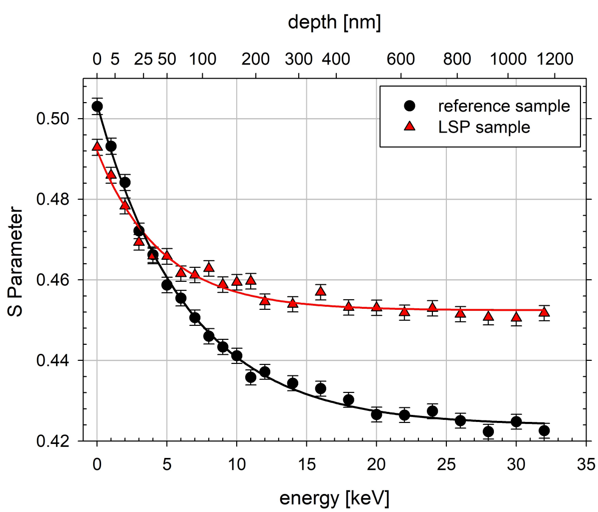

Fig. 3. The S parameter as a function of the incident positron energy as well as its implantation range.

Assuming that all positrons are trapped in defects and defect density is constant in the probed volume, it is possible to relate positron diffusion length L_+ obtained from VEP measurements with dislocation density σ using the formula [11]: $\sigma = \frac{1}{\mu\tau_{\text{bulk}}}\left(\left(\frac{L_{+\text{ref}}}{L_+}\right)^2 - 1\right)$, where μ is the specific trapping rate of positrons in dislocations. We used the value of this parameter determined for iron equals 0.51×10^{-4} m^2/s [12]. The approximated dislocation density for the LSP sample is of the order of 10^{14} m^{-2}.

4. Conclusions

The LSP induced the structure changes in the steel AISI 316L samples that extend to the depth of 83 μm. Near the surface, at the depth less than 13 μm, clusters consisting of two to five vacancies are found. The dislocation density near the LSP treated surface at the depth less than 1 μm estimated from the VEP results is of the order of 10^{14} m^{-2}.

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