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INVESTIGATION OF HELIUM IMPLANTED Fe–Cr ALLOYS BY MEANS OF X–RAY DIFFRACTION AND POSITRON ANNIHILATION SPECTROSCOPY

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X-ray diffraction (XRD) and positron annihilation spectroscopy (PAS) have been used for the characterization of the two binary alloys Fe-Cr with Cr content 2.36 and 8.39 wt%. The influence of ion implantation on these alloys was studied. Different implantation doses of helium, up to 0.5 C/cm^2, were used to simulate neutron-induced damage in a sub-surface region. To characterize the damage, a lattice parameter, coherent domain size, residual stress and a crystallographic texture have been studied by grazing incidence X-ray diffraction (GIXRD). It was found out that these parameters showed a similar dependence on the implantation dose as the positron lifetime determined by positron annihilation spectroscopy.

Keywords: grazing incidence, helium implantation, positron annihilation spectroscopy, radiation damage, x-ray diffraction

1 INTRODUCTION

A new generation of nuclear power plants requires more radiation resistant reactor materials. The most perspective materials for the new generation of fission reactors or thermonuclear fusion reactors are currently chromium ferritic/martensitic steels. In particular, these materials have to deal with high temperature and neutron flux. Important phenomenon is the hardening due to the effect of helium. Helium effectively stabilizes vacancy clusters, which means that less vacancies are available to recombine with interstitials, (*ie*helium ties up vacancies and reduces interstitial-vacancy recombination). Interstitial clusters can then grow into dislocation loops and increase the strength [1]. Chromium is a ferrite-stabilizing element that is generally added to the steels for an oxidation and corrosion resistance. The importance of this alloying element for the radiation-induced microstructural changes was discussed in the last decade [2, 3]. Currently, it is studied using different experimental techniques as well as by computer simulations [4-7].

This paper shows a study of dependence of selected structural parameters on the implantation dose in the iron-based alloys with high and low chromium content (8.39 wt% and 2.36 wt%) under the radiation treatment. GIXRD is a suitable technique for observation of the implantation-induced damage in the sub-surface region. This technique in combination with the positron annihilation spectroscopy gives much information about structural changes in specimens after radiation treatment.

2 EXPERIMENTS AND METHODS

2.1 Specimens and implantation

All specimens were prepared at the Slovak University of Technology in Bratislava (see Ref. [8] for detailed information about the specimens). The chemical composition of the studied alloys is listed in Table 1.

Table 1. Chemical composition of the studied alloys in %

Alloy	Cr	O	N	C
L251	2.36	0.035	0.012	0.008
L252	8.39	0.067	0.015	0.021

Alloy	P	Ni	Cu	V
L251	0.013	0.044	0.005	0.001
L252	0.012	0.070	0.010	0.002

These Fe-Cr alloys have been cut to the size of 10×10×0.5 mm, ground and polished to mirror-like surface before an exposure. The helium implantations at 250 keV and 4 different levels of the implantation dose (0.1, 0.2, 0.3, 0.4, 0.5 C/cm^2) have been performed at the linear electrostatic accelerator of the Slovak University of Technology

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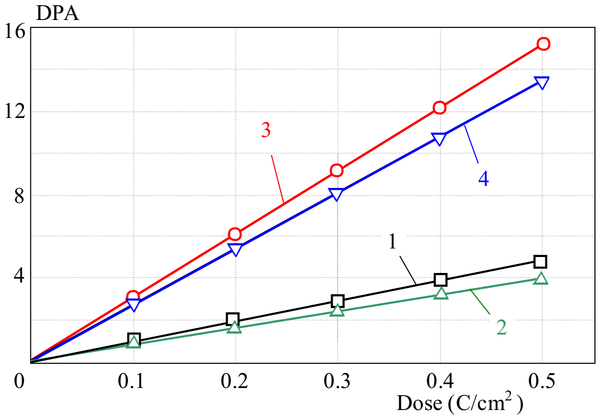

Fig. 1. DPA parameter of two binary alloys for depth of 0.3 and 0.6 μm

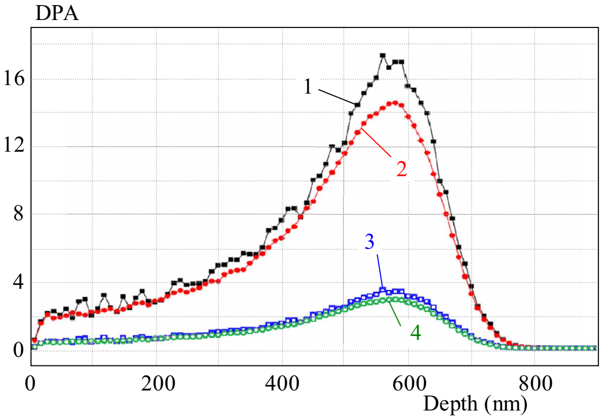

Fig. 2. DPA depth profiles from SRIM simulation, DPA/Dose: (1) — 2.36 wt % /0.5 C/cm^2, (2) — 8.39 wt % /0.5 C/cm^2, (3) — 2.36 wt % /0.1 C/cm^2, (4) — 8.39 wt % /0.1 C/cm^2

in Bratislava. The sample temperature was less than 343 K during implantation. Temperature of the sample holder was monitored by a thermocouple. The radiation damage in terms of the displacements per atom (DPA) simulated by the SRIM code [9] is presented in Fig. 1. The accelerator has a possibility to accelerate ions in a wide energy range from 10 keV to 900 keV. The high-energy He^{2+} ion implantation is performed in a dedicated vacuum chamber.

The high-energy implantation beam-line consists of an entrance collimator, a switching magnet (10 degree branch), exit collimating slits, a beam diagnostic block and the implantation chamber [10]. The beam, after passing the switching magnet, is shaped by the exit collimating slits that define the size of the beam on the sample. The helium implantation was chosen due to participation of alpha particles on the radiation damage induced by fission or fusion reactions [11, 12].

2.1 Analytical techniques

X-ray diffraction

XRD is a well-known non-destructive method for the evaluation of structural changes in materials. Diffraction measurements were performed in the parallel beam geometry with parabolic Goebel mirror in the primary beam at angle of incidence 2 deg, which corresponds to the penetration depth of approximately 0.3 μm. Bruker D8 DISCOVER diffractometer was used with X-ray tube equipped with a rotating Cu anode operating at 12 kW. The measurements of the diffractograms were performed in a way that the angle of incidence, α, was kept constant while the detector was moving along the 2Θ circle. All measurements have been performed in GIXRD setup at the Institute of Electrical Engineering, Slovak Academy of Sciences. For determination of the lattice parameter and the coherent domain size, the TOPAS software was used. The residual stress evaluation at different depths was obtained using the conventional GI-sin2ψ method [13,14].

Positron annihilation spectroscopy

The positron annihilation spectroscopy has been applied for the evaluation of the electron density by measuring the positron lifetime in the materials. These measurements have been carried out at the Slovak University of Technology in Bratislava. The used positron source, ^{22}Na, has a continuous spectrum of positrons up to energy of 545 keV. All positrons annihilate within 100 m from the surface. The measurements of the positron lifetime were performed in a standard positron annihilation lifetime spectrometry (PALS) equipment using a two-detector setup. All the lifetime spectra could be decomposed into three lifetime components with variances close to one. In our study, we used only the second lifetime component, τ_2, which describes positron annihilation in defects [15].

3 RESULTS AND DISCUSSION

In this paper, we are focusing on studying the influence of He-ion implantation on selected structural parameters of the steels with two different chromium-content levels. Helium-ion irradiation experiments can provide substitute for neutron irradiation [16]. Two Fe-Cr alloys with different Cr content have been investigated. The well-known effect of chromium on the corrosion resistance of the steels is more pronounced for the specimen with higher Cr content [17-19]. This was the reason why we focused on comparison of two alloys with different Cr content in our studies. GIXRD and PAS showed that the lattice parameter, the coherent domain size, and the residual macrostress show similar dependence on the implantation dose as the positron lifetime in defects. For the quantification of radiation damage, the DPA parameter calculated by SRIM can be used (Fig. 2).

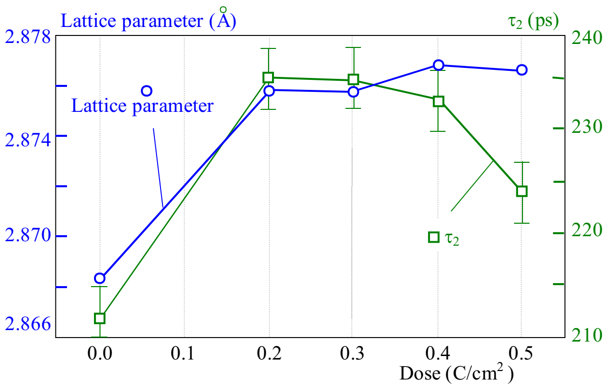

Fig. 3. The lattice parameter and positron lifetime in defects as a function of implantation dose in the sample with 2.36 wt% Cr

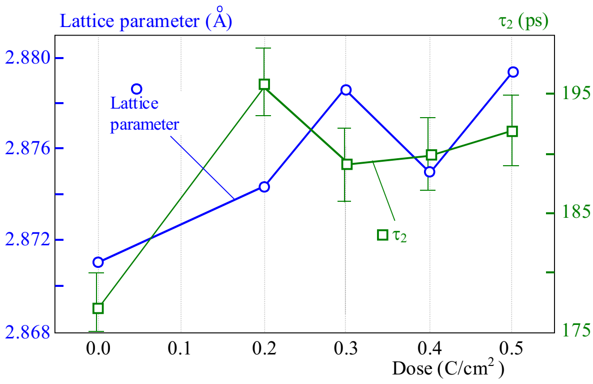

Fig. 4. The lattice parameter and positron lifetime in defects as a function of implantation dose in the sample with 8.39 wt% Cr

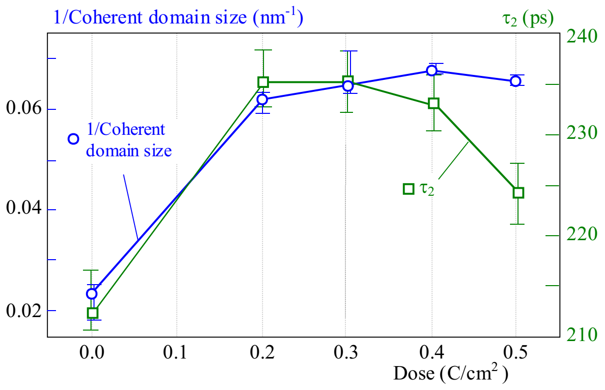

Fig. 5. The 1/coherent domain size and positron lifetime in defects as a function of implantation dose in the sample with 2.36 wt% Cr

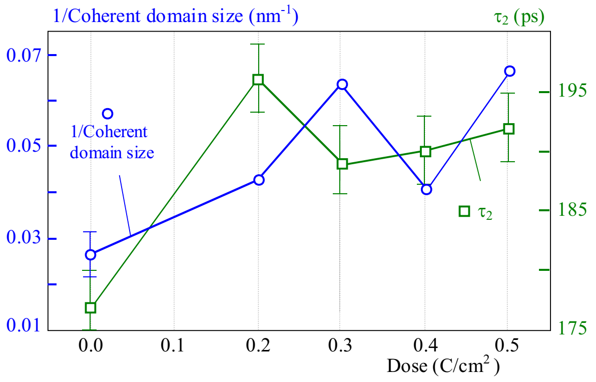

Fig. 6. The 1/coherent domain size and positron lifetime in defects as a function of implantation dose in the sample with 8.39 wt% Cr

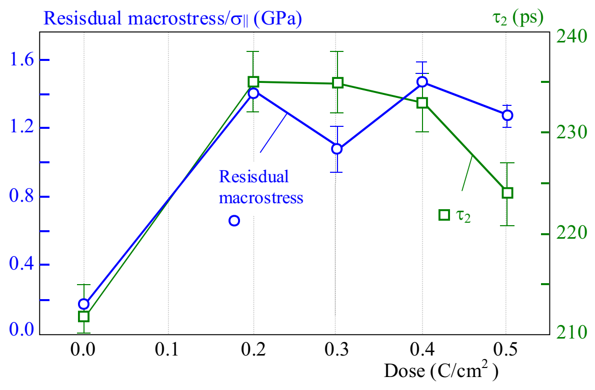

Fig. 7. Residual macrostress and positron lifetime in defects as a function of implantation dose in the sample with 2.36 wt% Cr

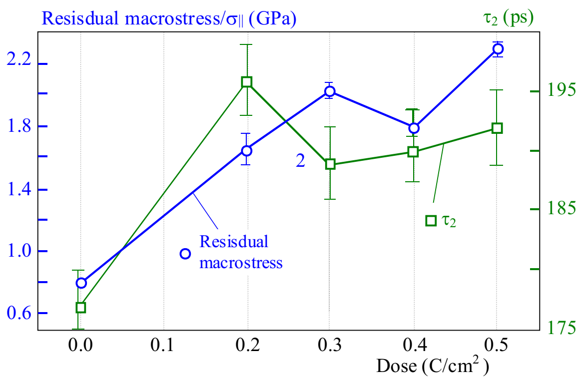

Fig. 8. Residual macrstress and positron lifetime in defects as a function of implantation dose in the sample with 8.39 wt% Cr

Based on analysis of the X-ray diffraction data by the TOPAS software, the phase transitions have not been observed. Contrary, changes of lattice parameter and inverse coherent domain size due to helium implantation have been found. As it can be seen in Figs. 3-6, the lattice parameter and inverse coherent domain size increase with the implantation dose. Similar behavior can also be seen from the PAS results describing the creation of a small vacancy cluster, the size of which saturates at a certain level of the implantation dose. Determination of residual stress was done by the GI-sin2? method. This method is used very often to determine residual stresses in depth because it is a non-destructive method. As it can be seen in Fig.7 and Fig.8, there is a rapid increase of residual stresses with increasing implantation dose.

The crystallographic texture was characterized by pole figure (110). The angle distribution of X-ray diffraction response after correction on the defocusing effect is pre-

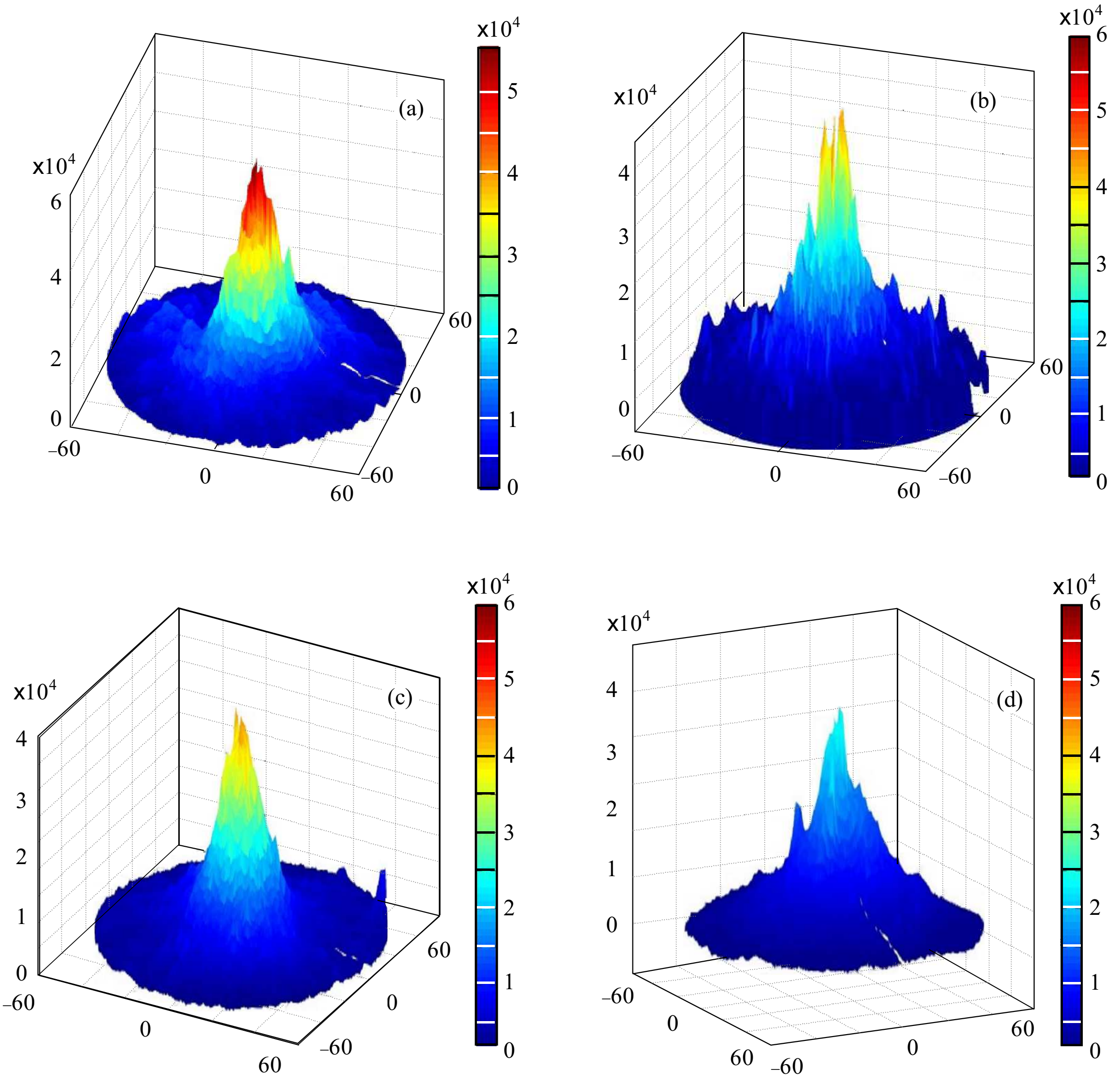

Fig. 9. Pole figure (110) of Fe-Cr alloys: (a) – non-implanted 2.36 wt% Cr, (b) – implanted 2.36 wt% Cr with maximum implantation dose, (c) – non-implanted 8.39 wt% Cr, (d) – implanted 8.39 wt% Cr with maximum implantation dose. The vertical axes: The X-ray diffraction response corrected on the defocusing effect, arbitrary units. The first horizontal axes: The polar angle, grad. The second horizontal axes: The azimuthal angle, grad.

sented for investigated specimens on Fig.9. The texture in the higher Cr content alloy after helium implantation is the most weak. For this aspect, the specimen with 8.39 wt% Cr has the most optimal crystallographic texture [20], because the isotropic state provides the improvement of mechanical properties of the loading steels. It corresponds to the choice of currently most perspective materials for advanced nuclear power systems.

5 CONCLUSION

In this work, two Fe-Cr alloys with the low and high chromium content under the different helium implantation doses were compared. XRD determination of the structural parameters has been performed after He-ion irradiation of the steel with 2.36 and 8.39 wt% Cr. The dose dependence of the lattice parameter and the coherent domain size is observed for both specimens. A similar dose dependence of the lattice parameter, the inverse coherent domain size and the positron lifetime in defects was found. The level of residual macrostress increases due to He implantation. The texture measurement after He implantation confirmed that the steel with 8.39 wt% Cr is an optimal material for advanced nuclear reactors. Our study showed that GIXRD in combination with PAS can be used to study sub-surface layers damaged by ion implantation.

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