PLEPS STUDY OF THE CHROMIUM INFLUENCE IN RAFM STEELS

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1. Introduction

Current nuclear power plants (NPP) require radiation, heat and mechanical resistance of their structural materials with ability to stay operational during NPP planned lifetime. [1-2]. Much higher radiation damage is expected in new generations of nuclear power plants, such as Generation IV and fusion reactors. Investigation of perspective structural materials for new generations of nuclear power plants is, among others, focused on study of reduced activation ferritic/martensitic (RAFM). Their microstructures and alloying elements, for example chromium, provide them with high swelling, radiation and thermal (up to 550 °C) resistance [3].

Our work is focused on the study of radiation damage (simulated by ion implantations) evaluation of RAFM steels represented by binary Fe-Cr model alloys. The influence of different content of chromium on the defects size and amount was studied. The experimental analysis of material damage at microstructural level was performed by conventional positron lifetime measurements (PALS) at Institute of Nuclear and Physical Engineering at Slovak University of Technology and by the Pulsed Low Energy Positron System (PLEPS) [4] at the high intensity positron source NEPOMUC [5] at the Munich research reactor FRM-II.

2. Materials treatment

The detailed chemical composition of studied Fe-Cr alloy can be seen in the table 1. The fabrication processes and treatments of the alloy can be found in [6]. "As-received" material was cut into desired dimensions, ground and polished to mirror-like surface before an exposure to helium implantation.

Tab. 1: *Chemical composition of studied Fe-Cr alloys (wt%) [6].*

Alloy	Cr*	O*	N*	C*	Mn	P	Ni	Cu	V
L251	2.36	0.035	0.012	0.008	0.009	0.013	0.044	0.005	0.001
L259	4.62	0.066	0.013	0.02	0.02	0.011	0.06	0.01	0.001
L252	8.39	0.067	0.015	0.021	0.03	0.012	0.07	0.01	0.002
L253	11.62	0.031	0.024	0.028	0.03	0.05	0.09	0.01	0.002

Accelerated helium ions were used to obtain cascade collisions in the microstructure of the studied material without neutron activation. The helium implantation was performed in two steps with ions energy of 250keV and 100keV, respectively. Implantations at the linear accelerator of the Slovak University of Technology in Bratislava [7] were performed at dose of 0.1 C/cm2 (6.24x1019 cm-2) corresponding to ~ 19 dpa. The maximum temperature during implantation did not exceed 100 °C. The ion energies were chosen to ensure the possibility of application of non-destructive techniques, sensitive in near surface areas (PAS, SEM, etc.). Applicability of these energies was also simulated by SRIM code (Stopping and Range of Ions

in Matter) and the maximum damage peaks were calculated for the depths of about 300 nm and 550 nm corresponding to the energies of 100 keV and 250 keV helium ions, respectively.

3. Results

The depth profiling of vacancy type defects was performed by PLEPS technique on ions implanted Fe-Cr alloys with different chromium content using positron energies in a range from 2 keV to 18 keV corresponding to the positron penetration depth of about 15 – 525 nm. The evaluation of Fe-11.62%Cr measured spectra was performed by PosWin code [8]. Mean positron lifetimes (MLT) were calculated from the three partial components of the lifetime spectra, usually assigned as; $\tau 1$- positron annihilation in bulk, $\tau 2$- positron annihilation in defects (vacancies, vacancy clusters) and $\tau 3$- positron annihilation in large defects (voids). As the MLT parameter can give the overall information about the microstructural defects only its data are shown in the paper.

Results achieved by the PLEPS (figure 1) technique, in case of as-received specimens showed, based on the positron lifetimes reaching levels of about 190 ps, presence of vacancy type defects with the size of about 1-2 vacancies for alloys with 5-12 % of chromium. Specimens with 2.5 % of chromium had the defects slightly bigger and could, according to the literature [9], indicate presence of small clusters with 3 vacancies. These defects, in as-received specimens, can be present in microstructure from the fabrication process or preparation of alloys for measurements. Top layers of microstructure (<100 nm) were not taken into account because of the probable surface annihilation of positrons and because of the thin oxide layer which could also increase the positron lifetimes to the depth of about 100 nm.
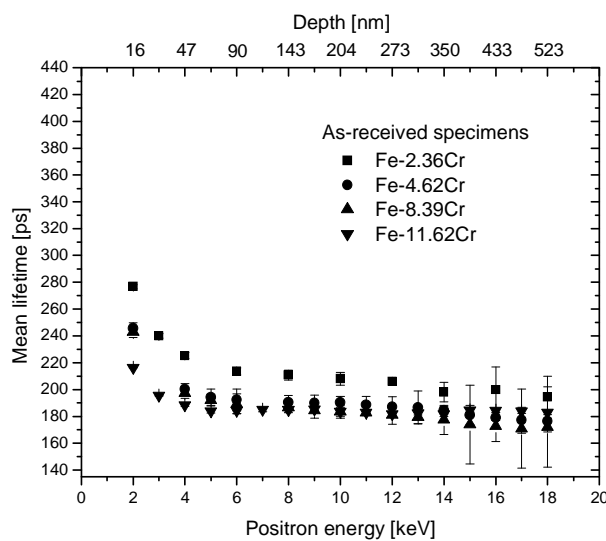

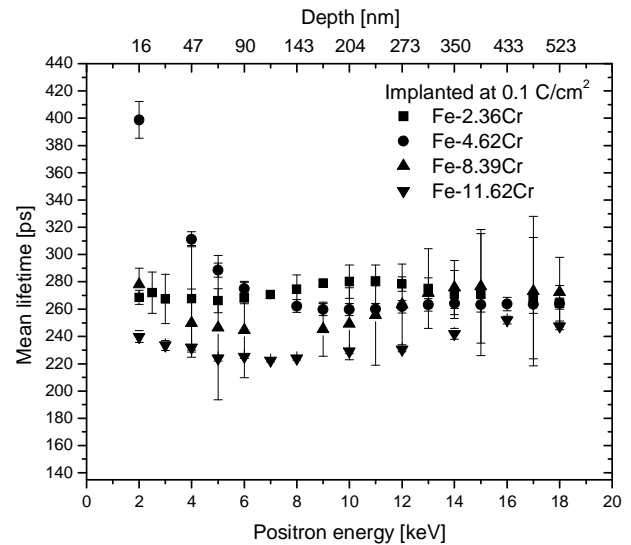Fig. 1: *Mean positron lifetimes of as-received Fe-Cr alloys measured by PLEPS.*

Fig. 2: *Mean positron lifetimes of ions implanted Fe-Cr alloys measured by PLEPS.*

The ion implantation at 0.1 C/cm2 caused a significant increase of positron lifetimes in comparison to as-received state of specimens. The mean positron lifetimes were of 40-80 ps higher than in the case of as-received specimens. According to the figure 2 the lowest values were observed for the specimens with 11.62% of chromium and with decreasing content of the chromium content increased also the positron lifetime to maximum of 280 ps. This positron lifetime increase corresponds to the increased size of the vacancy type defects to clusters of about 8 vacancies [9].

The lowest values of mean lifetimes (~ 220 ps) correspond to the clusters of about 3-4 vacancies. This is, in comparison to the as-received specimens, a small increase of positron

lifetime and positive influence of chromium on the smaller defects formation is observed. The maximum damage peaks from helium ions were not recognized in measured PLEPS spectra probably due to the very high level of damage introduced into the material that was discussed in our previous work [10].

The partial study of these steels was also performed by the conventional positron lifetime measurement (PALS) but the results achieved by this technique were unsatisfactory as the mean positron lifetimes in comparison to PLEPS results were lower of about 60-130 ps (figure 3). Such enormous decrease of MLTs cannot be explained as techniques deviations and it has to be assigned to the ability of PLEPS technique to see the increased amount of defects in specific depths thanks to the monoenergetic positrons. It means that the PALS technique is not so sensitive close to the surface and therefore the final results can be influenced by an undamaged bulk.
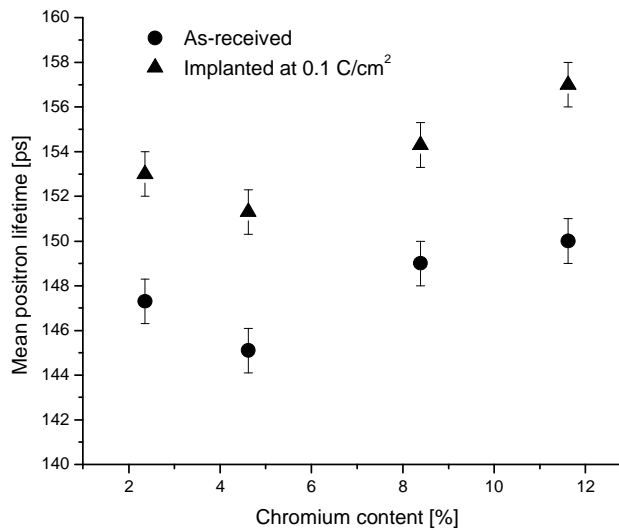

Fig. 3: *Mean positron lifetimes of as-received and implanted Fe-Cr alloys measured by PALS.*
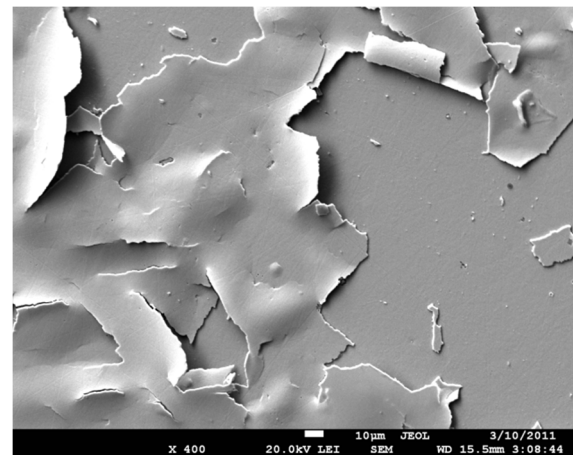

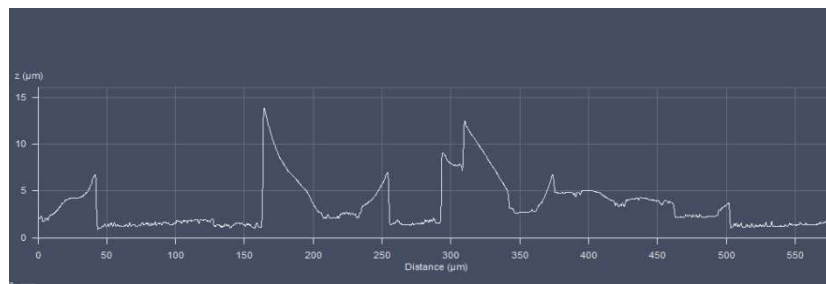Fig. 4: *SEM view of Fe-11.62%Cr surface implanted by He ions.*

Fig.5: *Profile extremes of implanted Fe-11.62%Cr alloy.*

The interference of the bulk structure to the final PALS results could be also indirectly influenced by blistering because of the exposed undamaged structure. Figure 4 shows Scanning Electron Microscopy (SEM) results of the great blisters on the surface of the Fe-11.62%Cr alloy after ions implantation and the undamaged structure under the blisters. Assumption about undamaged structure is based on the SEM figure and it is also supported by confocal microscope results (Fig.5), where the maximum blisters size is about 13 μm. Therefore, helium ions damage is not expected in that depth.

4. Conclusion

The investigation of ions implanted Fe-Cr alloys with a different content of chromium was performed in this work. The Pulsed Low Energy Positron System (PLEPS) and conventional positron lifetime technique (PALS) were applied for Fe-Cr steels investigation. Comparison of these techniques was also performed and the disadvantages of the PALS technique, in form of the inefficiency in the study of material layers close to the surface (up to 1 µm), were revealed. The blisters on the surface also influenced final results of the PALS technique.

Study of the as-received and helium ions implanted Fe-Cr alloys showed expected behavior and the mean positron lifetimes increased after a damage introduction by implantation, therefore the size of the defects increased. The increase in positron lifetime of 40-80 ps for implanted specimens in comparison to as received specimens, where the mono/di-vacancies were present, indicated presence of the vacancy clusters with the size of about 4-8 vacancies based on the chromium content. The implanted alloys with the highest content of chromium contained the smallest defects (4 vacancies). Therefore, the positive influence of the chromium content was observed and the formation of the bigger vacancy clusters, which were present in specimens with low chromium content (~2.5% Cr) was blocked.

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