PLEPS STUDY OF THERMAL ANNEALING INFLUENCE ON BINARY FE-11.62%CR ALLOYS

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1. Introduction

Lifetime of structural materials is one of the crucial factors for operation of nuclear power plants (NPP). Therefore, high expectations and requirements are put on these materials from the radiation, heat and mechanical resistance point of view. Even higher stresses are expected in new generations of nuclear power plants, such as Generation IV and fusion reactors. Therefore, investigation of new structural materials is among others focused on study of reduced activation ferritic/martensitic (RAFM) steels with good characteristics as lower activation, good resistance to volume swelling, good radiation, and heat resistance (up to 550 °C).

Our research is focused on study of radiation damage simulated by ion implantations and thermal treatment evaluation of RAFM steels in form of binary Fe-Cr model alloys. Due to the defect production by ions, there was applied an approach for restoration of initial physical and mechanical characteristics of structural materials in the form of thermal annealing, with goal to decrease size and amount of accumulated defects. Experimental analysis of material damage at microstructural level was performed by Pulsed Low Energy Positron System (PLEPS) [1] at the high intensity positron source NEPOMUC [2, 3] at the Munich research reactor FRM-II.

2. Experimental details and materials treatment

Detailed chemical composition of studied Fe-Cr alloy can be seen in the Table 1. Fabrication processes and treatments of the alloy can be found in [4]. “As-received” material was cut into desired dimensions, ground and polished to mirror-like surface before exposure to helium implantation.

Tab. 1: *Chemical composition of studied Fe-Cr alloy (wt%) [4].*

Alloy	Cr*	O*	N*	C*	Mn	P	Ni	Cu	V
Fe-11.62%Cr	11.62	0.031	0.024	0.028	0.03	0.05	0.09	0.01	0.002

* *measured after heat treatment during fabrication*

Accelerated helium ions were used to obtain cascade collisions in the microstructure of studied material without neutron activation. 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 [5] were performed at dose of 0.3 C/cm^2 (1.87x10^{18} cm^{-2}) corresponding to ~ 55 dpa. Maximum temperature during implantation did not overreach 100 °C.

Specimens were after PLEPS measurement at different ion implantation doses thermally treated with aim to understand influence of annealing on structure formation and defect behaviour. Thermal treatment was performed at „Universität des Bundeswehr“ in Neubiberg (Munich, Germany). Specimens were annealed in argon atmosphere (10 kPa) at temperatures of 400, 475, 525 and 600 ºC for 2 hours, then gradually cooled down (2 °C/min) and repeatedly measured after each temperature by PLEPS technique.

3. Results

Depth profiling of vacancy type defects was performed by PLEPS using positron energies between 2 keV and 18 keV corresponding to the mean penetration depth of 15 – 525 nm. Evaluation of Fe-11.62%Cr measured spectra was performed by PosWin code [6, 7]. The spectra were decomposed into three components and presented mean lifetimes (MLT) were calculated.

Interpretation of data achieved by PLEPS technique was significantly influenced by the Scanning Electron Microscopy (SEM) results performed at Institute of Materials Science (Faculty of Materials Science and Technology, STU). There was performed a diagonal cut under 12º angle for SEM technique (Fig.1) in implanted Fe-11.62%Cr specimen. The figure shows major damage in two regions. Considering the results achieved from SRIM [8] simulation (Fig.2), this two regions could be assigned to the maximum damage caused by He ions implantation. The first peak (Fig.2) in depth of ~300 nm is very significant and implies to damage from 100 keV and partially also from 250 keV helium ions. This can be recognized also from SEM figures where the area closer to the surface has more significant damage (100 keV He) than the one further from the surface influenced mainly by 250 keV He ions.

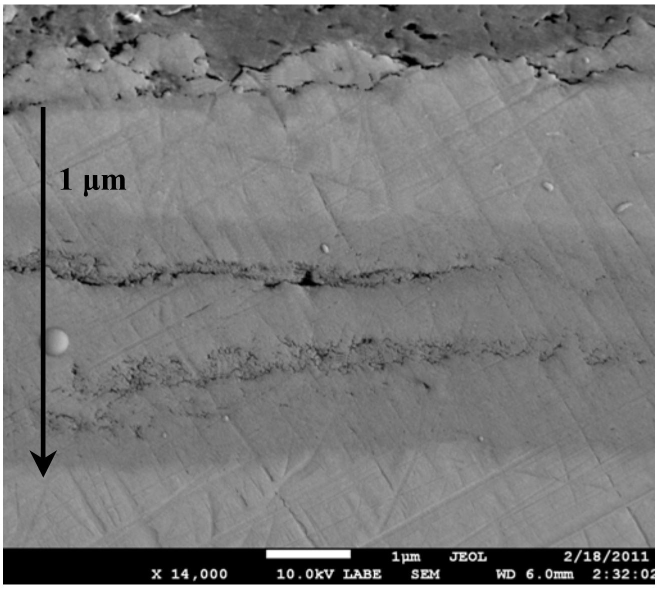

Fig.1: *Alloy Fe-11.62%Cr implanted at 0.3 C/cm^2 and annealed at 475 ºC.*

Annealing at temperatures between 0.3T_m-0.4T_m (melting temperature) can cause void and dislocation structure formation (the dislocation loops are unstable and grow into a dislocation networks) and diffusion is sufficient for the formation of precipitates [9]. This corresponds well with results achieved for annealing temperature of 475 ºC, as ions damaged area was multiplied and voids reached width of tens of nanometers.

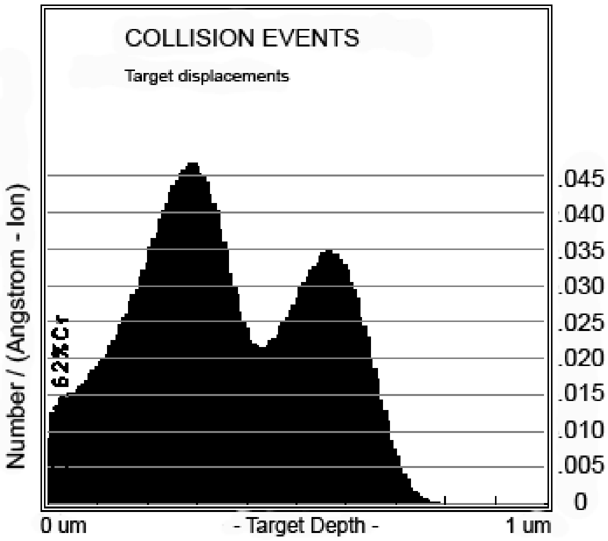

Fig.2: *SRIM simulation of damage by 100keV (left peak) and 250keV (right peak) He ions.*

Specimens implanted at level of 0.3 C/cm^2 registered significant decrease of mean positron lifetime (MLT) at temperature of 600 ºC (Fig.3). Analysis based on the MLT at this temperature, in depth of main damage caused by He ions, could be interpreted as extensive decrease of defects size to monovacancies or small vacancy clusters [10, 11]. Also the particular components reflecting positron lifetimes in bulk and defects, registered decrease and small clusters and the bulk with dislocations were recognized. Therefore, taking into account literature [9], regeneration of the microstructure at this temperature can be considered.

The growth of MLT from the depth of ~170 nm to the surface could be among others caused by the oxide layer, which was also measured by Energy Dispersive X-ray analysis.

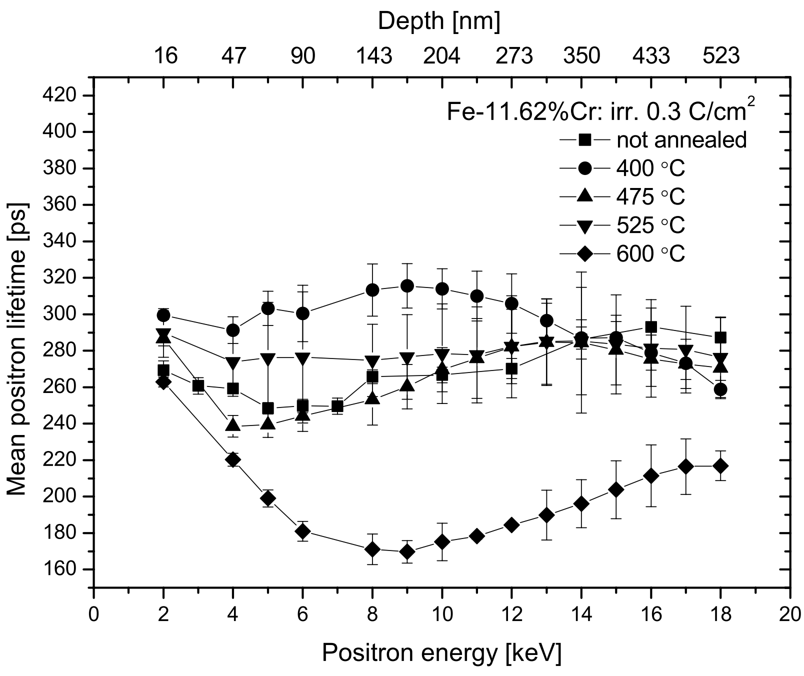

Fig.3: *Mean positron lifetimes of annealed Fe-11.62%Cr alloys.*

4. Conclusions

Investigation of irradiated Fe-11.62%Cr alloys after thermal annealing was performed in this work. Evaluation of the measured data of annealed Fe-11.62%Cr alloys by PLEPS technique was significantly influenced by Scanning Electron Microscopy (SEM) results of specimen annealed at 475 °C, which showed major damage in two helium influenced regions. Mean lifetimes describing damage of annealed (400-525 °C) specimens were at level of ~290 ps and in comparison to not annealed specimen, annealing effect was not observed. Only the temperature of 600 °C showed major decrease of mean positron lifetime. Therefore, considering the literature [9] about temperature influence on reduced activation ferritic/martensitic steels above $0.4T_m$ (melting temperature), we could say that such great voids in affected area (<1 μm) were continuously annealed out or at least their size decreased to monovacancies and small vacancy clusters not overreaching size of 5 vacancies.

Ion implantation damage occurred in specimens, was at very high level and with connection to the thermal annealing and oxide layer on the surface introduced many variables and created complicated system for the final evaluation of measured data. Therefore, even if the positron annihilation spectroscopy is very applicable technique for the vacancy type defects study, some limitations in form of problems to define too large defects (PLEPS) represented by MLT were recognized. Therefore, other destructive or non-destructive techniques have to be used to give us different point of view on the examined materials.

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