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Ion implantations of oxide dispersion strengthened steels

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This paper is focused on a study of radiation damage and thermal stability of high chromium oxide dispersion strengthened steel MA 956 (20% Cr), which belongs to the most perspective structural materials for the newest generation of nuclear reactors – Generation IV. The radiation damage was simulated by the implantation of hydrogen ions up to the depth of about 5 μm, which was performed at a linear accelerator owned by Slovak University of Technology. The ODS steel MA 956 was available for study in as-received state after different thermal treatments as well as in ions implanted state. Energy of the hydrogen ions chosen for the implantation was 800 keV and the implantation fluence of 6.24×10^{17} ions/cm^2. The investigated specimens were measured by non-destructive technique Positron Annihilation Lifetime Spectroscopy in order to study the defect behavior after different thermal treatments in the as-received state and after the hydrogen ions implantation. Although, different resistance to defect production was observed in individual specimens of MA 956 during the irradiation, all implanted specimens contain larger defects than the ones in as-received state.

1. Introduction

Structural materials of nuclear power plants (NPPs), e.g. reactor pressure vessel steels, are exposed to high doses of irradiation, heat and mechanical stresses, which may reduce their lifetime during NPP operation [1–3]. Much higher radiation and thermal loads are expected in the newest generation of nuclear power plants, such as Generation IV (GEN IV) and fusion reactors, which will be operated at temperatures between 550 and 1000 °C and will be exposed to irradiation over 100 DPA (displacement per atom) during planned lifetime which is more than 60 years [4]. Consequently, the demands on their structural materials are much higher and so the research and development of these materials have to significantly progress in near future.

The advanced structural materials, as oxide dispersion strengthened (ODS) steels, are developed for application in cooling systems, reactor pressure vessel or fuel cladding of the GEN IV nuclear power plants. Application of these steels in fusion reactors is intended as a first wall material. The ODS steels fulfill demands on radiation, thermal, and mechanical resistance during operation of nuclear reactor. ODS steel MA 956, which was studied in this paper, has high thermal corrosion resistance based on alloying by chromium, aluminum, silicon, and on formation of dispersion of stable oxides in structure. This resistance is achieved by formation of protective grains from oxides as Y_2O_3. The oxide dispersions are intended to provide high temperature strength up to 650 °C [5].

Our work is focused on the study of radiation damage (simulated by ion implantations) evaluation of ODS steels with and without the thermal load applied before the irradiation. The experimental analysis of material damage at microstructural level was performed by conventional Positron Annihilation Lifetime Spectroscopy (PALS) at Institute of Nuclear and Physical Engineering, Slovak University of Technology.

2. Materials preparation and treatment

MA 956 is high chromium commercial ODS steel manufactured by company INCOLOY in USA. The chemical composition listed in Table 1 was analyzed by optical emission spectroscopy SPEKTROLAB (Type LAVWA18A) at Welding Research Institute in Bratislava.

The material was received in a form of blocks which were firstly cut to specimens suitable for PALS measurements with dimensions of $10 \times 10 \times 0.4$ (max 0.6) mm, then ground, and finally mirror-like polished. The zone affected by cutting usually goes to the depth of about 150 μm [6], but subsequent grinding removes the most affected subsurface. The powder used for polishing had particles with size of 0.5 μm. Therefore, the roughness of the surface was very fine. The measurements by atomic force microscopy [7] showed the maximum roughness up to 20 nm for the as-received specimens (Fig. 1).

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Table 1
Chemical composition of MA 956 ODS steel (wt.%).

C	Si	Mn	P	S	Cr	Mo	Ni	Al
0.072	0.04	0.12	0.008	0.003	19.49	0.1	0.07	3.399
Co	Cu	Nb	Ti	V	W	N	Y_2O_3	Fe
0.04	0.03	0.01	0.33	0.02	<0.01	0.038	0.48	75.75

Four different kinds of specimens (listed in Table 2) were investigated by positrons. Two specimens were prepared for comparison of two different as-received states; in longitudinal cut (ML) and in transversal cut (MT) of the specimen. It was for purpose of structural homogeneity study. Further two specimens were compound as combination of longitudinal cut and transversal cut (MLT) in each PALS specimen by reason of a structural inhomogeneity reduction during the irradiation and thermal experiments. These MLT specimens were further exposed to temperature of 475 °C during 100 and 500 h. Experiments at this temperature were also performed at binary Fe–Cr alloys with 12% of chromium content in our previous work [8]. Therefore, some behavior of microstructure in form of defects creation and accumulation was assumed in MLT specimens in comparison to the as-received specimens.

Radiation damage of ODS steels (without neutron activation) was afterward simulated in all investigated specimens by hydrogen ion implantations at energy of 800 keV and the fluence of 6.24×10^{17} ions/cm^2 at linear accelerator at Slovak University of Technology in Bratislava [9,10]. The temperature during the implantation did not exceed 100 °C; therefore an accumulation of new vacancy defects was assumed without additional processes of structure relaxation as the defects could more easily diffuse and recombine from the temperature effect. For determination of the detailed damage profiles, the SRIM (Stopping and Range of Ions in Matter) code has been used. Fig. 2 shows damage profiles of hydrogen ions implantation of MA 956 specimens. The main damage peak appeared in the depth of about 5 µm. Displacement per atom calculated for these steels is at level of about 0.4 for whole volume of the specimen.

3. Experimental results

Specimens of MA 956 in the as-received, the thermally treated and the ion implanted state were measured by Positron Annihilation Lifetime Spectroscopy (PALS) in a fast–fast mode [11] with the FWHM parameter close to 200 ps. The Variance of fit (reduced chi-square) achieved value in range of ⟨1; 1.1⟩. The PALS can determine the defect size and the defect concentration up to the depth ~120 µm, which is effective mostly for

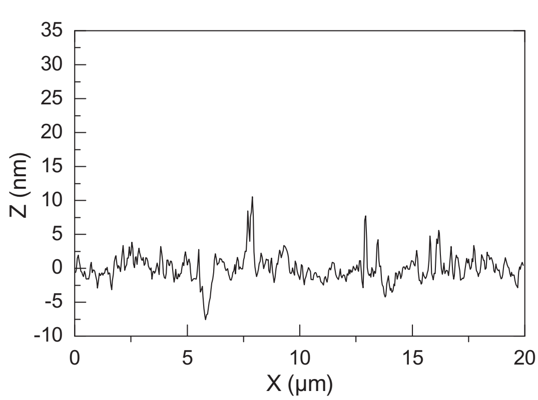

Fig. 1. AFM view on coarsen profile of as-received specimens.

Table 2
Treatments performed at specimens prior to ion implantations.

Specimen	Characterization	Temperature (°C)	Time (h)
ML	Longitudinal cut, recrystallized, as-received	–	–
MT	Transversal cut, recrystallized, as-received	–	–
MLT100	Longitudinal + transversal cut, recrystallized	475	100
MLT500	Longitudinal + transversal cut, recrystallized	475	500

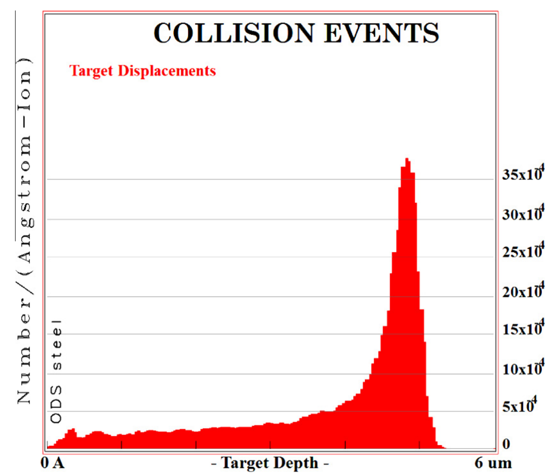

Fig. 2. Damage profile of ODS steel MA 956 implanted by 800 keV hydrogen ions with the fluence of 6.24×10^{17} ions/cm^2.

measurement of the as-received and the annealed specimens. The PALS measurement of the implanted specimen is less sensitive, although a significant change of the structure can be well visible by this technique.

Final PALS data were evaluated by LT 10 [12] software with application of diffusion model for positron trapping [13], which is normally used for evaluation of ODS steels measurements. Results achieved by this software were expressed by positron lifetimes in defects, which define the size of the defect. Therefore, increase in positron lifetime means the increase in the defect size. Another parameter (intensity of positron annihilation in the defects) from LT 10 analysis gives information about the amount of the defects in studied microstructure. Other parameters as the Average positron lifetime and concentration of the defects, stated in this paper, were calculated based on the data achieved from LT 10 analysis.

Positron lifetimes of defects in the investigated specimens are shown in Fig. 3. Lifetimes of the as-received and the thermally treated specimens reached level of about 260 ps which could indicate presence of vacancy clusters with the size of about 4 vacancies. The results from the as-received specimens showed small difference in the longitudinal specimen (ML) and the transversal one (MT). This is due to structural inhomogeneity as the specimens were cut in different directions and different area of the received block of material. The positron lifetime for MLT100 (longitudinal and transversal cuts annealed at 475 °C during 100 h) seems to be an average of MT and ML specimens, although the defects concentration proportional to positron intensity slightly decreased. This could indicate reduction of vacancy defects due to their recombination within structure lattice. A similar effect on defect concentration was observed in MLT500 (annealed during 500 h), but its defects slightly grew after the long-term annealing.