STUDY OF OXIDE DISPERSION STRENGTHENED STEELS FOR NUCLEAR APPLICATIONS

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1. Introduction

Structural materials of nuclear power plants (NPP), 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 - 1 000 °C and will be exposed to irradiation over 100 DPA during planned lifetime which ismore than 60 years [4]. Consequently, the demands on their structural materials are much higher and so the research and development of these materials has to have significant 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. 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 (Y_2O_3) in structure.

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 is listed in the Table 1.

Tab. 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

The material was received in a form of blocks which were firstly cut to specimens suitable for PALS measurements with dimensions of 10x10x0.4 (max 0.6) mm, then ground and finally mirror-like polished.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 (MLT100)and 500 (MLT500) hours.Radiation damage of ODS steels (without neutron activation) was afterwards simulated in all investigated specimens by hydrogen ion implantations with energy of 800 keV and the fluence of $6.24x10^{17}$ ions/cm^2 at linear accelerator at Slovak University of Technology in Bratislava [5,6]. For the determination of the detailed damage profiles, the SRIM (Stopping and Range of Ions in Matter) code was used. Damage profiles of hydrogen ions implantation of MA 956 specimens showed that the main damage peakappeared in the depth of about 5 μm.

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).Final PALS data were evaluated by LT 10 [7] software with application of diffusion model for positron trapping [8], which is normally used for evaluation of ODS steels measurements. Results achieved from this software were expressed by positron lifetimes in defects, which define the size of the defect. Therefore, increase in positron lifetime means 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 Figure 1. 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 hours) 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 but its defects slightly grew after the long-term annealing.

The hydrogen ion implantation caused increase in positron lifetimes for the implanted specimens as was assumed. Most significant increase was observed in implanted specimens after thermal treatment at 475 °C (MLT100-impl, MLT500-impl). With the values at level of about 275 ps, we can assume that the size of the defects increased from 4 to the 5-vacancy clusters. Intensities of positron lifetimes annihilating in the defects did not change significantly within the all specimens before and after the ion implantation. These changes were from about 48% to 52 %. Interesting results were achieved for the ion implanted MT, MLT100 and MLT500 specimens. The intensities of defects are at the same level of about 48%. Therefore, we can assume that after the ion implantation was the amount of the defects stable but their size increased for the thermal treated specimens at 475 °C. In case of the thermally treated and the implanted specimens, this effect was observable also in comparison to the as-received and the thermally treated specimens (MLT100, MLT500).

Figure 2 shows the Average positron lifetime of specimens before and after the ion implantation. In case of the as-received and the thermally treated specimens, there are some small differences between the specimens which could be caused by the thermal treatment. In the case of the as-received specimens could be these differences influenced by inhomogeneity of microstructure.Average positron lifetime of all implanted specimens reached level of about 205 ps with small deviations for the individual specimens.

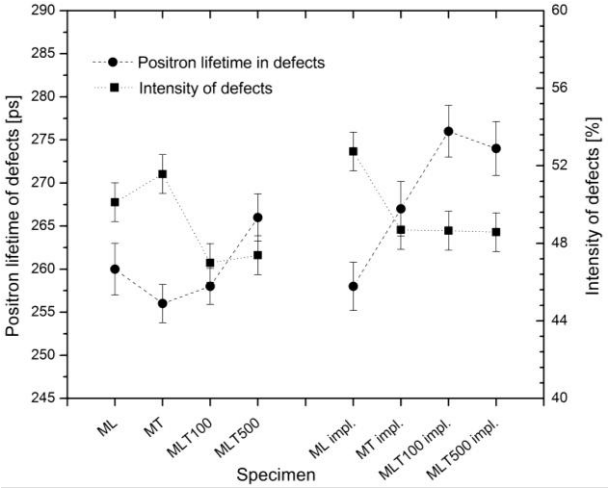

Fig. 1: Positron lifetimes of defects and their intensities.

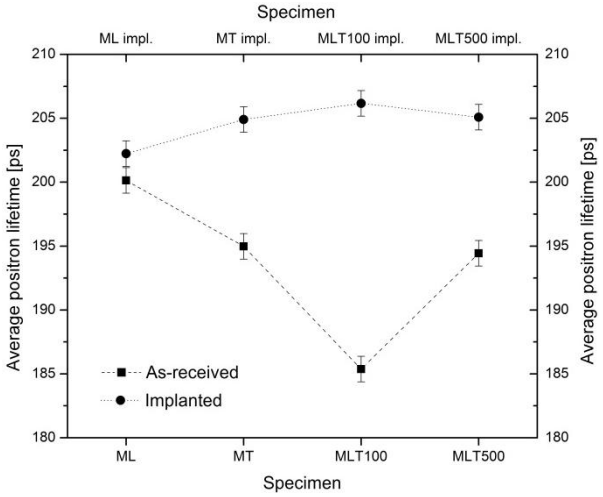

Fig.2: Average positron lifetime of specimens before and after hydrogen ions implantations.

Therefore, the implanted specimens seem to have similar presence of defects in total, independently of the cut direction or the thermal treatment.

Based on the results achieved from the LT 10 software, we were able to calculate also the concentration of the defects. The defect concentration was calculated from positron data [9, 10]. The calculated concentrations of defects (Fig.3) for the as-received specimens differed from about 0.9 to 1.1×10^{23} m^{-3}.

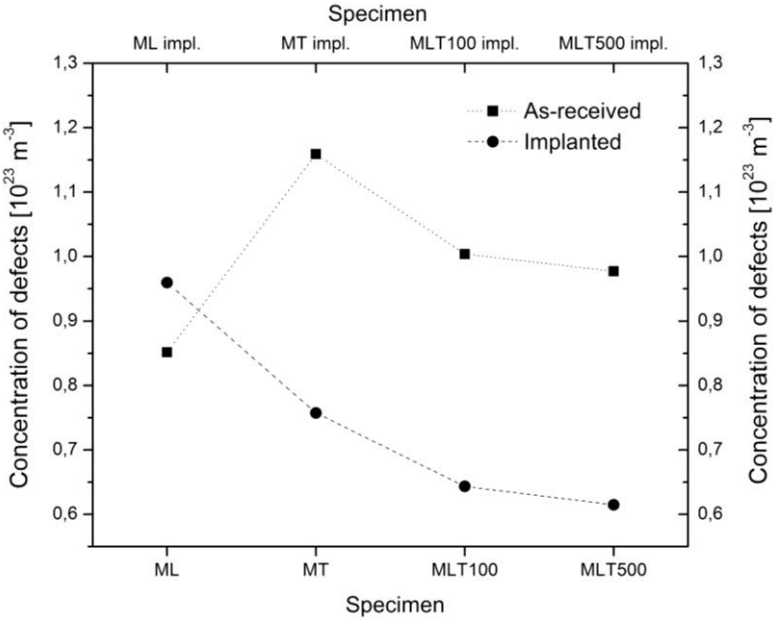

Fig. 3: *Concentration of defects for as-received and hydrogen ions implanted specimens.*

In case of the thermally treated specimens, there was observed some decrease of the concentration, probably due to the longer annealing time. Significant change was observed in the implanted specimens. The concentration of the defects decreased in comparison to the as-received and the thermally treated specimens. The influence of the increasing annealing time from 100 to 500 hours was also followed by the decrease of the concentration of defects. The

main reason of this decrease is the increase of positron lifetime in defects after the ion implantation as well as after the thermal treatment in case of the as-received specimens. It means that the defects grew and the bigger vacancy clusters were formed. Differences of the as-received specimens from the longitudinal (ML) and the transversal (MT) cuts can be caused by material inhomogeneity.

4. Conclusion

In this paper, the oxide-dispersion-strengthened (ODS) steels were studied as one of the promising structural materials for future fission and fusion reactors applications. Specimens of ODS steel MA 956 were investigated by Positron Annihilation Lifetime Spectroscopy at different states.

According to the results of positron lifetimes describing vacancy defects, we can assume that the hydrogen ions implantation caused increase of a size of the defects from the clusters of about 4 vacancies to clusters of the size of 5-6 vacancies. The duration of the thermal treatment also acts role in increasing of the defects size in case of the as-received as well as the implanted specimens. The longer annealing during 500 hours had strong degrading effect for both series of the specimens - before and also after the implantation. However, the annealing during shorter time of 100 hours demonstrated small structure relaxation for the non-implanted specimens. Intensity of these defects did not change significantly in dependence on the annealing time, especially for the implanted specimens. Thus, the amount/concentration of defects did not change after different time of thermal treatment (100 and 500 hours). In this case, only the size of defects increased. Calculated results of defects concentration showed its decreasing character for the as-received as well as the implanted specimens with the increase of annealing time. This is due to a defects aggregation into bigger clusters supported by higher temperature and caused by hydrogen ions implantation. Inhomogeneity of microstructure, created by longitudinal and transversal cutting schemes could also introduce some deviations into the specimens that appeared the same. These results are preliminary and will be followed by further work.

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