NON-DESTRUCTIVE STUDY OF NEW CONSTRUCTION MATERIALS FOR ADVANCED NUCLEAR REACTORS

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1. Introduction

Microstructure of new construction steels for advanced reactor systems with different type of structure: oxide dispersion strengthened steel – ODS Eurofer (20% Cr), ferritic-martensitic steel Eurofer 97 and austenitic steel NF 709 (See chemical composition in Tab. 1) were studied by positron annihilation lifetime spectroscopy. Samples were measured before and after helium ion implantation (He^+); therefore microstructure changes and radiation resistance to alpha particles of these steels were observed. Defect accumulation due to the radiation treatment was assumed in all investigated materials; therefore positron mean-lifetimes will increase up with notable change. The paper compares radiation damage of different type of structure and point out to the most radiation resistant structure / material from the investigated ones.

Tab. 1. Chemical composition of steels (in % wt.).

Steels	C	Mn	Ni	Cr	Mo	Ti	Ta	W	V	Si	Nb	N	Y_2O_3
ODS Eurofer	0.1	0.44	-	8.8	0.01	-	0.14	1.1	0.2	0.05	0.01	0.01	0.3
Eurofer	0.1	0.44	-	8.8	0.01	-	0.14	1.1	0.2	0.05	0.01	0.01	-
NF 709	0.06	1.0	25.0	20.3	1.05	1.5	-	-	.	-	0.26	-	-

2. Experimental Details

The samples were investigated by positron annihilation lifetime spectroscopy (PALS). The PALS [1] can determine concentration and size of vacancy-type defects in sample with very low concentration (from 0.1 to 500 ppm) [2]; therefore can describe area where microscopy techniques are not so sensitive. The measuring equipment [3] used in this work consists of two BaF_2 scintillation detectors connected in fast-fast mode. As positron source, ^{22}Na covered in Kapton foil (POSK 22) was used. The resulting spectrum was evaluated by program LifeTime9 [4]. The value of FWHM parameter was close to 240 ps. Fit Variant (reduction of chi-square) achieved value in range (1; 1.1).

Samples were loaded by radiation damage performed at a linear accelerator belonging to Slovak University of Technology. Helium ions (He^+) with a kinetic energy up to 500 keV were used. An implantation depth (See Fig.1) achieved approximately 1 μm according SRIM simulations [5]. The implantation level – surface charge was 0.16 C/cm^2 (~ 1x10^{17} ions cm^{-2}) and the radiation damage was around 45 dpa.

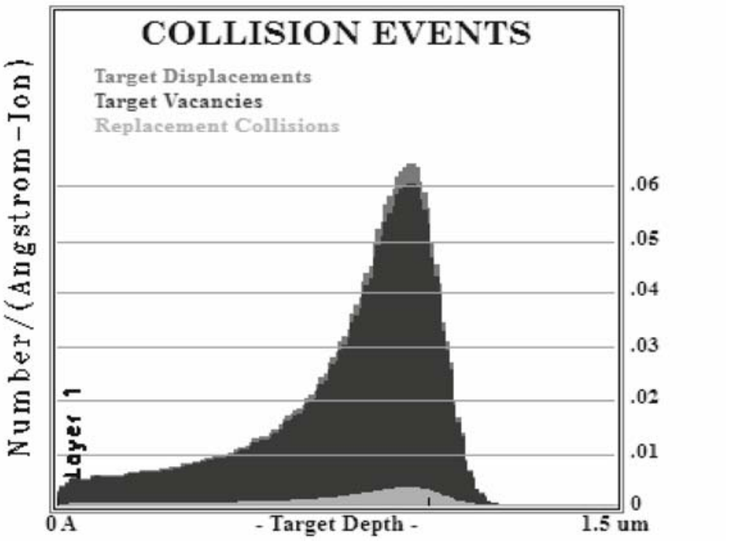


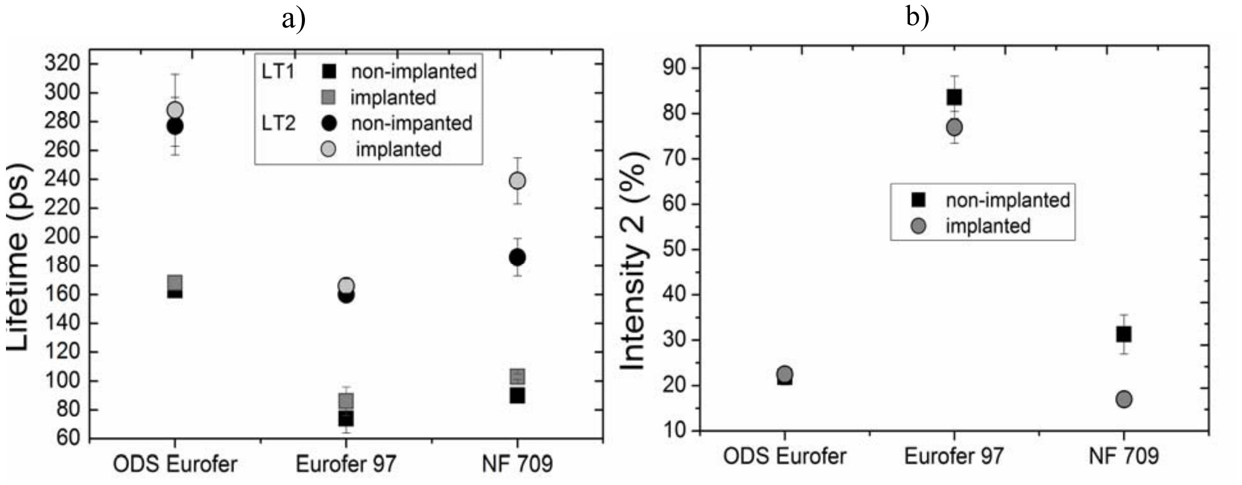
Fig.1: Simulation of implantation depth profile by SRIM.

3. Results & Discussion

PALS spectra were decomposed into three components according to the Standard trapping model [6]. The shortest lifetime (LT1) of Eurofer 97 and NF 709 achieved values up to 110 ps, which describes positron annihilation in bulk, eventually values reduced due to presence of defects especially for Eurofer 97. The LT1 of ODS Eurofer was much higher (160 ps) which also indicates presence of dislocations or mono-vacancies.

The second positron lifetime (LT2) found within the range from 160 to 300 ps characterizes vacancy type defects and is dependent on the size of three dimensional vacancy clusters Vn consisting n vacancies. This lifetime can be also affected in ODS Eurofer by positron annihilation in Y_2O_3 nano-particles (theoretical value 240 ps [7]).

Values of LT2 are presented and compared each other in Fig.2a. In this graph, the differences of microstructure are demonstrated via the presence of vacancy defects. Also evident increase of lifetimes due to implantation (Fig. 2a) was found for all steels as was assumed. The lifetime defining the bulk (LT1) and also defects (LT2) grew due to the effect of ion-atom collisions and atom knocking-out.


a)

b)

Fig.2: PALS results for non-implanted and implanted samples.

The highest defect size was observed for steel ODS Eurofer, which contains probably clusters with 5 vacancies (V5) in pre-dominance. After implantation the defect size remained stable – unchanged. Three vacancy clusters (V3) were observed in the implanted sample of NF 709. This steel demonstrated the most increase of LT2 due to implantation; the non-implanted sample had only mono-vacancies (V1) or dislocations. The lowest defect growth, almost negligible change of LT2, was recorded for Eurofer 97, which contained mono-vacancies (V3) and dislocations before and also after the implantation.

The intensities of positron annihilation in defects (I2) shown in figure 2b demonstrated a value decrease for all steels after implantation except of steel ODS Eurofer. This indicated that during implantation the defect amount was lessened. Defects merged to the larger ones as LT2 values have already presented.

Results of positron study can be also treated into mean lifetime (MLT), which is not loaded by error formed during spectra decomposition into the lifetimes and intensities. The MLT has absolute deviation equal to 2 ps; therefore it introduces a quite accurate way for comparison of steels. The MLT of our investigated steels is presented in figure 3a, where also changes of defect volume for samples before and after implantation are displayed. There, we can observe influence of implantation on defect accumulation.

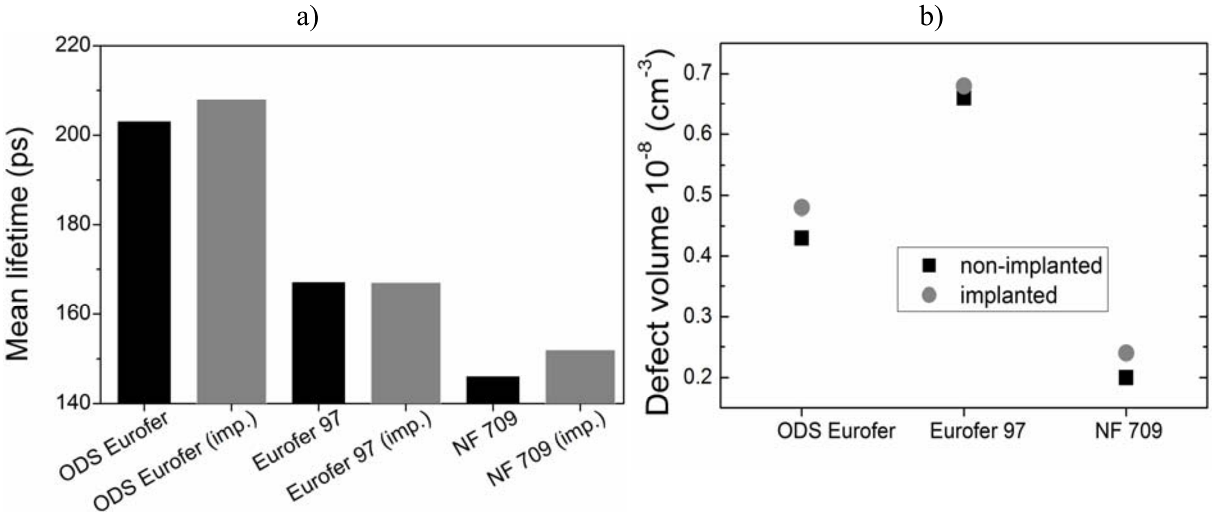


Fig.2: Positron mean-lifetime values for samples before and after implantation (a), the change of defect volume after implantation (b).

The negligible change of MLT value was identified for the samples of Eurofer 97. Enough high change due to implantation was proven for NF 709 (7 ± 2 ps) and ODS Eurofer (4 ± 2 ps). Eurofer 97 seems to be the most radiation resistant material in compare to other investigated steels.

With using of LT2 and I2 values, defect concentration was calculated according to equations published in [8, 9]. The defect concentration also decreased after implantation due to defect merging and the growth of the defect size. However defect volume calculated from the defect concentration and the defect size of pre-dominance defects increased after implantation, which is evident proof of accumulated damage by alpha particles. Significant change of vacancy defect volume due to implantation was found for ODS Eurofer and NF 709.

4. Conclusion

Three different commercial steels were investigated by positron annihilation spectroscopy. Steels differ in chemical composition (mostly in chromium and nickel content) and in the structure of iron. They showed different sensitivity to defect accumulation during He^+ ions (alpha particles) implantation. ODS material ODS Eurofer seems to be the most radiation affected; it contains combination of the biggest defects together with mono-vacancies and dislocations, however the concentration of big defects is low. Steel NF 709 indicated significant increase of defect volume due to defect accumulation, as was assumed for austenitic steel. The most radiation resistant material is ferritic-martensitic Eurofer 97.

This study can supplement complex investigation of candidate materials for GEN IV reactors and can be helpful for final selection of the most appropriate construction material for application in environment with alpha radiation.

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