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Positron study of alpha particles effect on oxide-dispersion-strengthened steels

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Abstract. Microstructure of oxide-dispersion-strengthened steels with different chromium content – MA 956 (20% Cr), ODM 751 (16% Cr) and MA 957 (14% Cr) were studied by positron annihilation lifetime spectroscopy. Samples were measured before and after helium ion implantation (He^{2+}); therefore microstructure changes and radiation resistance to alpha particles of these steels were observed. Defect accumulation due to the radiation treatment was proven in all investigated materials. After ion implantation, mean lifetimes increased of about ~ 10 ps, which indicates significant change in microstructure. According to calculations of a defect volume from defect concentration and pre-dominant size of defects, ODM 751 is the most resistant steel in comparison to other investigated materials.

1. Introduction

Oxide-dispersion-strengthened alloys belong to perspective candidate materials for new reactors within the frame of the international program Generation IV. Therefore, it is necessary to ensure their integrity during their projected lifetimes. Construction steels applied for the advanced nuclear facilities could be exposed to high temperatures (~ 900^{o}C) and high irradiation levels (~ 150 dpa) [1], which significantly deteriorate their mechanical properties (hardness, embrittlement and toughness) [2].

This paper deals with a study of microstructure of oxide-dispersion-strengthened steels with different chromium content – MA 956 (20% Cr), ODM 751 (16% Cr) and MA 957 (14% Cr). A radiation resistance to alpha irradiation, which transmits high energies in a short-range area, was investigated for samples of the studied steels via positron annihilation lifetime spectroscopy.

The effect of alpha particles was simulated by helium ion implantation with a kinetic energy up to 500 keV performed by using a linear accelerator at Slovak University of Technology. Radiation damage around 45 dpa was applied. Two states of the samples, before and after the implantation, were measured and results were compared. Defect accumulation due to the radiation treatment was proven in all investigated materials.

2. Experiment

Three following commercial high chromium ferritic ODS steels were measured: MA 956 and MA 957 (products of Incoloy) [3] as well as ODM 751 (product of Dour Metal) [4]. The chemical composition of investigated steels (See table 1) was observed by optical emission spectroscopy at Institute of Materials, Slovak University of Technology.

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

Steels	C	Mn	Ni	Cr	Mo	Ti	Al	Cu	Si	Nb	N	Y_2O_3
MA 956	0.07	0.12	0.07	19.97	0.10	0.30	3.40	0.03	0.04	0.01	0.04	0.50
ODM 751	0.07	0.07	0.02	16.20	1.74	0.66	3.80	0.02	0.06	0.01	0.03	0.50
MA 957	0.03	0.09	0.13	14.40	0.03	1.00	0.03	-	0.04	-		0.30

These investigated ODS alloys were produced by mechanical alloying, i.e. matrix materials were milled and mixed together with yttrium particles to form solid solutions with a uniform dispersion of oxide nano-particles, and the mixtures were then consolidated using Hot Isostatic Pressing (HIP) at 1150°C under a pressure of 103MPa. The recrystallization heat treatment resulted in a coarse columnar grain structure. Samples of investigated steels were prepared from as-received material by cutting the steel sheets into suitable pieces. After cutting, the sample surfaces were polished in order to remove surface impurities. Mechanical treatment of samples affects surface and subsurface layers, although samples are polished almost into a mirror level.

The samples were investigated by positron annihilation lifetime spectroscopy (PALS). The PALS [5] can determine concentration and size of vacancy-type defects in sample with very low concentration (from 0.1 to 500 ppm) [6]; therefore can describe area where microscopy techniques are not so sensitive. The measuring equipment [7] 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 [8]. 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^{2+}) with a kinetic energy up to 500 keV were used. An implantation depth achieved approximately 1 μm according SRIM simulations [3]. The implantation level – surface charge was 0.16 C/cm^2 (~ 1x10^{17} ions cm^{-2}) and the radiation damage was around 45 dpa. During the ion implantation, a defect accumulation occurred as result of atom knocking-out due to slowing down of ions. Therefore increase of defect concentration or defect size was assumed in the investigated samples [9].

3. Results and Discussion

PALS spectra were decomposed into three components according to the Standard trapping model [10]. The shortest lifetime (LT1) of MA 956 and ODM 751 achieved values up to 110ps, which describes positron annihilation in bulk, eventually values reduced due to presence of defects especially for MA 956. The LT1 of MA 957 was much higher (170 ps) which also indicates presence of dislocations or mono-vacancies.

The second positron lifetime (LT2) found within the range from 225 to 300 ps characterizes vacancy type defects and is dependent on the size of three dimensional vacancy clusters V_n consisting n vacancies. This lifetime can be also affected by positron annihilation in Y_2O_3 nano-particles (theoretical value 240 ps [11]) or in similar precipitates or segregation.

Values of LT2 are presented and compared each other in figure 1. In this graph, the differences of microstructure are demonstrated via the presence of vacancy defects. Also evident increase of lifetimes due to implantation (See figure 1) was found for both groups of 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.

The highest defect size was observed for steel ODM 751, which contains probably clusters with 5 vacancies (V_5) in pre-dominance. After implantation the defect size remained stable – unchanged. Five or four vacancy clusters (V_4 – V_5) were observed in the implanted sample of MA 957. This steel demonstrated the most increase of LT2 due to implantation, the non-implanted sample had only three-vacancy clusters (V_3). Lower defect growth, but also evident change of LT2, was recorded for MA 956. MA 956 contained three-vacancies (V_3). Due to radiation treatment, defects start to enlarge and

LT2 increased. The implanted MA 956 has probably three-vacancies with some amount of four-vacancies (V_3- V_4).

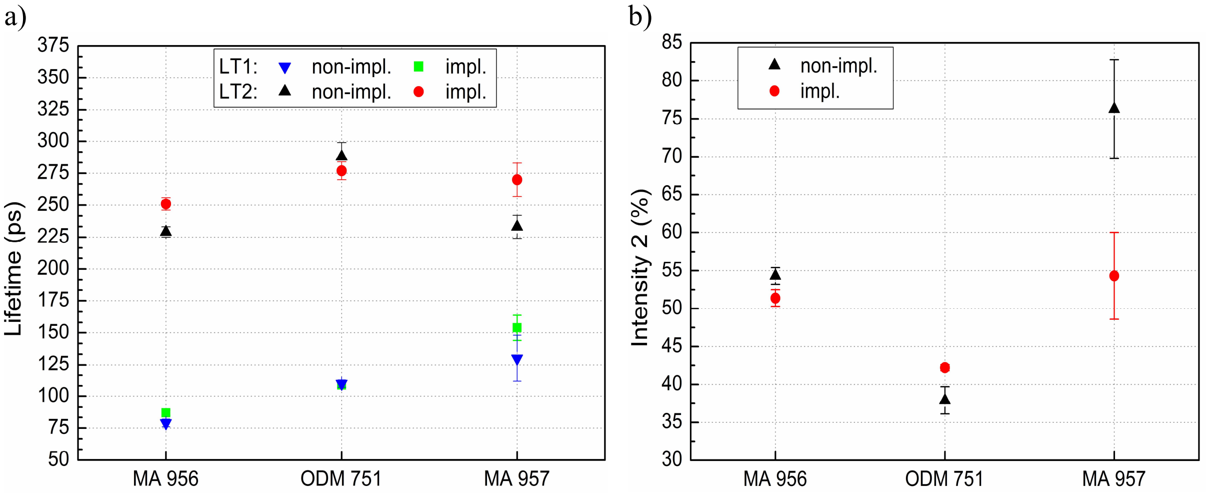

Figure 1. PALS results for non-implanted (non-impl.) and implanted (impl.) samples.

The intensities of positron annihilation in defects (I2) shown in figure 1b demonstrated a value decrease for all steels after implantation except of steel ODM 751. This indicated that during implantation the defect amount was lessened. Defects merged to the larger ones as LT2 values have already presented. Only steel ODM 751 has higher I2 value after implantation (ΔI2= 4.3 %), which means that accumulation of new defects occurred along with the weak merging of defects in this material.

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 2a, where also changes of MLT between samples before and after implantation are displayed. There, we can observe influence of implantation on defect accumulation.

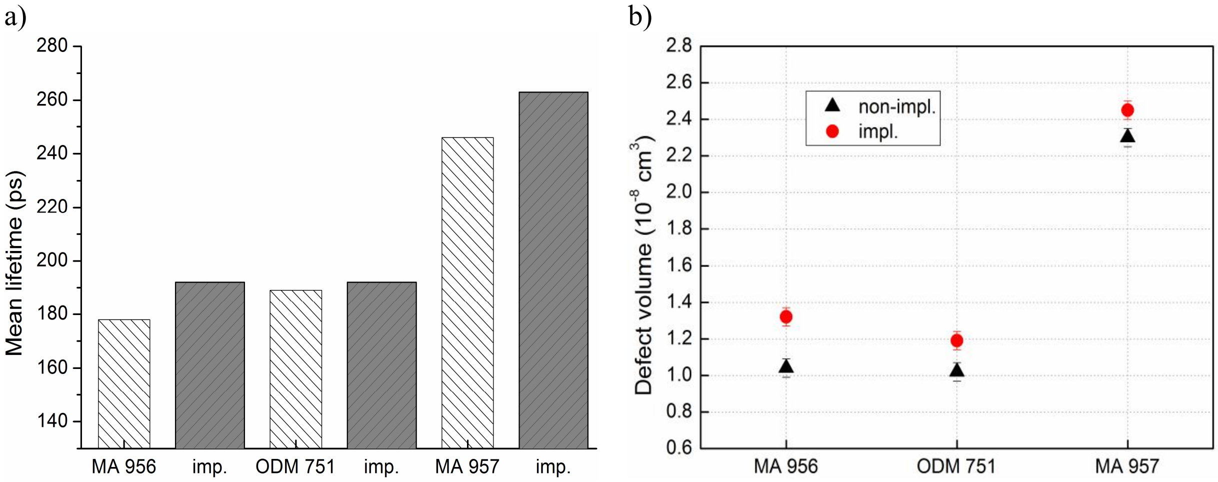

Figure 2. Positron mean lifetime values for samples before and after implantation (a).The change of defect volume after implantation (b).

The smallest MLT change was identified for the samples of ODM 751 (3 ± 2 ps). Enough high change due to implantation was proven for MA 956 (14 ± 2 ps) and MA 957 (17 ± 2 ps). ODM 751 seems to be the most radiation resistant material in compare to other investigated steels, although it contains the biggest defects.

With the use of LT2 and I2 values, defect concentration was calculated according to equations published in [12, 13]. 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. The highest change of vacancy defect volume due to implantation was found for MA 956. MA 957 is typical by the highest volume of vacancy defects, but it is necessary to realize that dislocation or mono-vacancies found in this material were not included in the volume, which were calculated only from LT2 and I2 values.

4. Conclusion

Three different commercial oxide-dispersion strengthened steels were investigated by positron annihilation spectroscopy. Steels differ in chemical composition, mostly in chromium and aluminum content. They were formed by the same process of manufacture, but their sensitivity to defect accumulation during He^{2+} ions (alpha particles) implantation is not similar. Material ODM 751 seems to be the most resistant, but it contains the biggest defects with the lowest concentration. Steel MA 956 indicated the highest radiation damage due to defect accumulation shown in the change of defect volume.

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.

Acknowledgement

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