POSITRON STUDY OF IMPLANTED ODS STEELS

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1. Abstract

This study was focused on commercial oxide-dispersion strengthened steels - MA 956 (20%Cr), PM 2000 (19%Cr) and ODM 751 (16%Cr) developed for construction of the Generation IV reactors. The ODS steels are investigated in order to compare their microstructure features and radiation resistance. Accumulation of vacancy defects due to alpha irradiation were observed by positron annihilation lifetime spectroscopy.

The alpha particles were simulated by helium ion implantation performed in a linear accelerator with a cascade 1 MeV voltage source at Slovak University of Technology. The lowest presence of open volume defects was found in PM 2000 before and also after the radiation treatment. The lowest defect accumulation during implantation (radiation resistance) was demonstrated in ODM 751; although this steel contained the largest defects before and also after irradiation (vacancy clusters V_5).

2. Introduction

Design and introduction of future nuclear reactor systems are strongly dependent on the choice of structural materials. These materials must withstand more exacting operating conditions, typical for the reactors within the international program Generation IV (Gen IV). In comparison to current commercial reactors, all construction materials (particularly for internal components) of the GEN IV reactors will be operated at elevated temperatures (~900^{o}C). The construction materials will be also loaded by higher radiation damage (up to ~150 dpa for internal structure) [1].

The knowledge about ageing-induced changes in term of microstructure of construction materials [2 - 4] is not sufficient for Gen IV reactors. Therefore, structural materials (those for the Gen IV included) have to be tested and the results of these tests need to provide the microstructural interpretation.

The presented paper is focused on a group of oxide-dispersion strengthened (ODS) alloys, which have been considered as candidate materials in most Gen IV concepts. Our experiments provide the microstructural characterization of chosen ODS steels using of non-destructive technique - positron annihilation lifetime spectroscopy (PALS).

3. Experiment

In this paper, commercial high chromium ODS steels are investigated. Although the presented ODS steels were primarily designed for application in space and for heat exchangers in thermal power plants, their derivates have been developed for nuclear applications.

Three following commercial materials were measured: MA 956 (20% Cr, product of Incoloy), PM 2000 (16% Cr, product of Plansee) and ODM 751 (16% Cr, product of Plansee). The chemical composition of investigated steels is listed in Table 1.

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

	C	Mn	Ni	Cr	Mo	Ti	Al	Si	Y_2O_3
MA 956	0.07	0.12	0.07	19.93	0.10	0.30	3.40	0.04	0.50
ODM 751	0.07	0.07	0.02	16.02	1.74	0.70	3.80	0.06	0.50
PM 2000	0.07	0.07	0.03	19.23	0.13	0.50	4.20	0.03	0.50

The 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. The mixtures were then consolidated using Hot Isostatic Pressing at 1150^{o}C under a pressure of 103MPa. The ODS alloys were supplied as rods in the recrystallized condition at 1050^{o}C during 2 hours for improved creep strength.

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 and damage after cutting and grinding. A zone affected by the mechanical process during preparation of the samples is up to ~ 1μm.

The positron annihilation lifetime spectroscopy (PALS) [5, 6] can determine concentration and size of vacancy-type defects in sample with very low concentration (from 0.1 to 500ppm) [6]; therefore it can describe the area where transmission electron microscopy (TEM) is not so sensitive. Positron lifetime is almost proportional to the size of three dimensional vacancy clusters V_n consisting n vacancies [7, 8]. A percentage of positrons (intensity) trapped and annihilated in the defects, can give information about defect concentration.

The measuring equipment used in this work consists of two BaF_2 scintillation detectors and two discriminators in fast-fast mode [9]. The measured spectra were evaluated by program LifeTime9 [10] according to the three-state positron trapping model [11]. The time resolution (FWHM) of the time spectrometer was close to 210 ps. Fit Variant (reduction of chi-square) achieved value in range (1; 1.1), which means that the goodness of the fit was sufficient and the relative deviation of the fit was below 0.1%.

Radiation damage was simulated by helium ion implantation performed with using a linear accelerator at Slovak University of Technology. The kinetic energy of helium ions was up to 500 keV and the implantation level achieved 0.1 C/cm^2 (10 dpa). The implantation profile was calculated in SRIM software and it is shown in Fig. 1. The Bragg peak is located approx. in a depth of 900 nm. The radiation damage ranges up to 1.15μm.

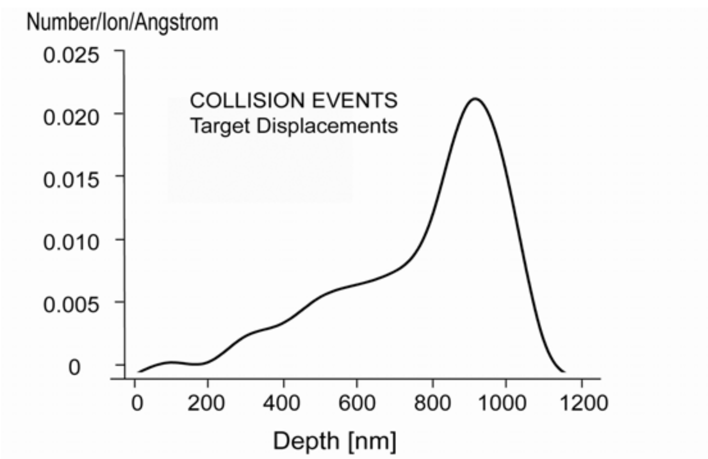

Fig. 1: *The implantation profile of helium ion implantation.*

4. Results and Discussion

PALS measurement was treated into three composite lifetimes (LT) according to the Standard trapping model [11]. The shortest lifetime (LT1) defines positron annihilation in almost defect free structure, called bulk. The bulk value for pure iron and also for steels is around 110ps. The LT1 observed in MA 956 and PM 2000 was reduced due to high presence of defects in the samples. Higher LT1 value was found for ODM 751. We assume that the bulk of ODM 751 was affected by very small defects like mono-vacancies (V_1) or dislocations, which are located in the samples along with some larger defects.

Second positron lifetimes (LT2) within the range 190 and 240ps can describe the size of vacancy clusters V_n consisting n vacancies. The last lifetime (>500ps) expressed annihilation in the air, which was not fully removed during process of source component correction. The LT3 is not further listed because it is neglected value without any physical meaning. The results are shown in Tab. 1.

Tab. 1. *Positron lifetimes – LT (ps) and intensities –I (%) for non-implanted and implanted samples.*

Samples		**LT1 (ps)**	**I1 (%)**	**LT2 (%)**	**I2 (%)**
MA 956	non-implanted	79 ± 3	43.9 ± 1.1	229 ± 4	54.3 ± 1.1
	implanted	87 ± 2	46.7 ± 1.1	251 ± 5	51.2 ± 1.1
PM 2000	non-implanted	60 ± 4	39.8 ± 1.4	199 ± 5	58.1 ± 1.4
	implanted	75 ± 3	42.5 ± 1.4	225 ± 5	55.1 ± 1.6
ODM 751	non-implanted	116 ± 3	61.3 ± 1.8	288 ± 11	37.9 ± 1.8
	implanted	109 ± 2	56.9 ± 0.4	227 ±78	42.2 ± 0.4

The highest positron lifetime characterizing vacancy defects (LT2) was found in non-implanted ODM 751 with LT2=288±11ps. Therefore, this non-implanted sample contains bigger vacancy cluster consist of 5 vacancies (V_5). Positrons annihilated in these defects with intensity (I2) approx. 38%. After implantation, the LT2 probably describe the same defects but the measured value is more inaccurate (absolute deviation is 78ps). The intensity I2 increased, which demonstrate accumulation of larger defects in the sample due to implantation of helium ions.

The sample of MA 956 contains in pre-dominance three-vacancies (V_3) before implantation and four-vacancies (V_4) after implantation. The defect size grew during the implantation in this sample. Expected increase of I2 did not occur. The value of I2 even decreased, which was probably due to rapid merging of defects. Smaller defects like mono-vacancies (V_1) were able to merge to the larger ones. Therefore intensity I2 drooped down.

The similar effect was observed for the I2 of PM 2000. The LT2 for non-implanted sample achieved 199±5ps belonging to di-vacancies (V_2) and after implantation PM 2000 contains three-vacancies (V_3) in pre-dominance, where positrons annihilated with lifetime ~ 225±5ps.

Positron mean lifetime (MLT) was calculated from all three lifetimes and their intensities according the Eq.(1) for each sample. The MLT values are presented in Fig. 2.

$$MLT = \sum_{i=1}^{3} LT_i \cdot I_i \qquad (1)$$

The highest MLT for the non-implanted samples belongs to ODM 751. This material shows more extensive presence of defects in compare to other investigated samples. According to the MLT increase (See Fig. 2), the least significant damage due to helium ions was observed also for ODM 751. The difference in MLT is close to the absolute deviation of MLT (2ps). The highest increase of MLT was registered for PM 2000.

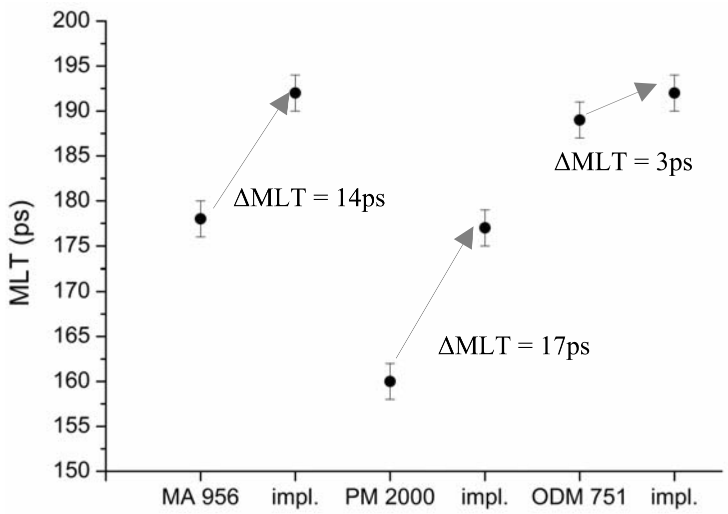

Fig. 2: *Mean lifetime (MLT) for non-implanted and implanted samples.*

5. Conclusion

Positron annihilation lifetime spectroscopy was applied in the microstructural study of the commercial oxide dispersion strengthened steels – MA 956, PM 2000 and ODM 751. Differences in microstructure of the investigated materials were indicated due to different presence of vacancy defects, which can be a result of manufacturing and post-manufacturing thermal treatment. The highest presence of defects was found in ODM 751 as well as the biggest defects. ODM 751 was followed by MA 956 and the lowest defect concentration was in PM 2000.

The investigated samples were also observed for purpose of defect accumulation during helium ion implantation. The highest radiation resistance was visible in ODM 751 and the lowest in PM 2000.

6. Acknowledgements

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