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Study of structural inhomogeneity of commercial oxide-dispersion-strengthened steels

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Abstract. Oxide-dispersion-strengthened steels, candidate materials for construction of new generation of nuclear reactors, can have inhomogeneous structure and anisotropic mechanical properties due to their fabrication, especially by process of mechanical alloying. This paper examines two similar commercial oxide-dispersion-strengthened steels MA 956 and PM 2000 in two different orientations of samples in order to study inhomogeneity of defect presence using positron annihilation techniques. Positron data showed small or negligible differences between samples of individual steels (up to 4 % for average lifetime). Moreover, structural divergence between two orientations (transversal cut and longitudinal cut) of the same steel was also not apparent. However, higher presence of defects and also bigger defects were found for both orientations of MA 956 samples. The structure of PM 2000 probably contains fewer defects with smaller size, though their occurrence in surface and subsurface layers up to 1.6 μm (from Doppler broadening spectroscopy) seem to be less homogeneous than in MA 956.

1. Introduction

Oxide-dispersion-strengthened (ODS) steels belong to perspective candidate materials for new nuclear reactors within the frame of the international program Generation IV and also for fusion reactors. The ODS steels have appropriate mechanical properties and good resistance to corrosion and thermal strains. However, these steels are fabricated by process of mechanical alloying which brings inhomogeneity of their structure and anisotropy of their properties [1]. Methods of homogenization and anisotropy suppression are still studied in many international laboratories, e.g. by addition of variant alloying elements [2, 3] or by different post-manufacture thermo-mechanical treatment (i.e. warm rolling, annealing) [4].

Anisotropy of mechanical properties is not acceptable in construction materials of nuclear facilities. Therefore recent R&D is focused on improvement of technology processes which includes also optimization of milling time and intensity of base material and Y_2O_3 powder or optimization of grain size [5]. As well as laboratory produced steels, also commercial fabricated steels have to be tested in order to eliminate application of inhomogeneous and anisotropic material in construction of the facilities. Mechanical properties are dependent on microstructure, i.e. lattice, structural defects, etc. For that reason, this paper deals with structural study of two commercial ODS steels with almost identical chemical composition and the same process of production but in two different companies.

The emphasis was put on observation of structural defects in samples of two different orientations (transversal and longitudinal cuts) for each investigated material. This is for purpose to compare homogeneity of defect presence in samples depending on their location in extruded steels. Defect presence was observed by positron techniques - Doppler broadening spectroscopy at Aalto University in Finland and positron annihilation lifetime spectroscopy at Institute of Nuclear and Physical Engineering in Bratislava.

2. Experiment

Two following commercial high chromium ferritic ODS steels were measured: MA 956 (products of Incoloy) and PM 2000 (product of Plansee). 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	V	Y_2O_3
MA 956	0.07	0.12	0.07	19.49	0.10	0.33	3.40	0.03	0.04	0.01	0.04	0.02	0.50
PM 2000	0.07	0.07	0.03	19.07	0.13	0.49	4.23	0.01	0.03	0.01	0.01	0.01	0.50

These investigated ODS steels were produced by mechanical alloying, i.e. matrix materials were milled and mixed together with yttria particles to form solid solutions with a uniform dispersion of oxide nano-particles, and the mixtures were then consolidated using HEx at 1150°C under a pressure of 100 MPa. Investigated samples were prepared from as-received material (cylinder shape) by cutting steel sheets one after another into suitable pieces.

Two directions of cuts – longitudinal and transversal ones were prepared (as is shown in Figure 1) for purpose of studying a potential inhomogeneity of defect presence in differently orientated samples, which can support properties anisotropy. Investigation of the longitudinal direction can bring a view of defect presence from inner to outer sides of the received cylinder for the extrusion at the same moment. The transversal direction can show a dependence of defect presence on the extrusion time, as each sample was formed at different moment. 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.

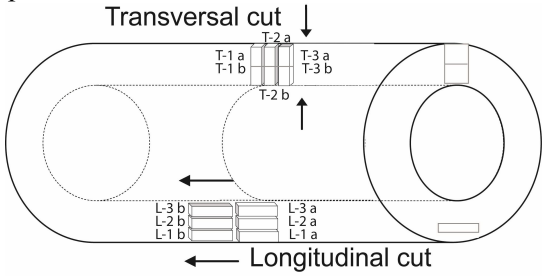

Figure 1. Cutting scheme of two kinds of samples - longitudinal and transversal cuts.

The samples were observed by two different positron annihilation methods describing the presence of vacancy type defects in structure: positron annihilation lifetime spectroscopy (PALS) and positron Doppler broadening spectroscopy (DBS).

The PALS [6] was measured in fast-fast mode [7] with the FWHM parameter up to 240 ps. The variance of fit (reduced chi-square) achieved value in range (0.99; 1.14). The PALS can determine the defect size (lifetime) and defect concentration (intensity) up to the depth ~ 120 μm.

The DBS [8] with a conventional setup of the measurement was recorded by one HPGe detector with Gaussian resolution function of 1.24 keV. The results are shown in S parameters which is calculated from an energy window of $|E\gamma - 511\ \text{keV}| < 0.83$ keV. Positrons used in the DBS measurements are acquired from a slow positron beam [9]. The monoenergetic beam applied energies ranging from 5 to 36 keV during the measurement. This technique and the equipment can be used for a

study of the defect profile as a function of a positron implantation depth in samples up to 1.6 μm. The S parameter increases with growth of defect presence (defect size or defect concentration).

3. Results and Discussion

Three samples of each orientation were observed by two positron annihilation techniques. Data of positron annihilation lifetime spectroscopy were treated by spectrum decomposition into two components according to Diffusion trapping model [10] in software LT10 [11]. The shortest lifetime (τ_f) achieved values between 125 and 140 ps which describes positron annihilation in free state - defect free material (bulk) together with small defects, probably dislocations. The bulk values according to the calculation [12] are around 80 ps. Second positron lifetime (τ_D) found in range between 228 and 258 ps describes positrons annihilated in defects with size probably three and four-vacancies in predominance together with annihilation in yttria particles (theoretical lifetime - 240 ps [13]). A paper published by Krsjak et al. in 2010 [12] describes that approximately 10% of τ_D forms annihilation in yttria in our studied materials.

PALS results (Figure 2) show that samples of both investigated steels have small divergences in lifetime of defects (τ_D), although the lifetimes indicate presence of the same defects for the individual steels. The average values of τ_D for MA 956 are 244 ± 6 ps for longitudinal orientations and 256 ± 4 ps for transversal cuts. MA 956 has probably four-vacancies in predominance. The values τ_D for PM 2000 were 230 ± 5 ps for longitudinal cuts and 240 ± 2 ps for transversal ones; therefore PM 2000 contains mostly three-vacancies.

Intensities of defects (I_D), percentage of positrons annihilated with τ_D, have a light dispersion (Figure 2). The average values I_D for MA 956 are 57.7 ± 1.5 % for longitudinal cuts and 53.6 ± 2 % for transversal ones. The values I_D for PM 2000 are 57.1 ± 2 % for longitudinal cuts and 52.1 ± 1.0 % for transversal ones.

The PALS results for individual steels are similar and the data dispersion can be only formed due to the data decomposition. The Average lifetime (τ_{AV}) calculated by LT10 program is presented in Figure 3a. This value is more accurate in comparison to the individual lifetimes, because it is calculated from the measured spectrum without the data decomposition. The average lifetime for MA 956 is 176 ± 2 ps and for PM 2000 163 ± 3 ps. The error of the measurement equipment achieves ± 2 ps in common. Therefore, the average lifetimes lead to assumption that homogeneity of the investigated steels (up to depth of 120 μm) was attained enough during the process of manufacture.

Further, the Average lifetime demonstrated with high probability that steel MA 956 has higher defect presence than PM 2000. It is indicated by all investigated samples in both sample orientations. The minimum difference of τ_{AV} is 6 ps which is sufficient for our statement in term of the measurement error.

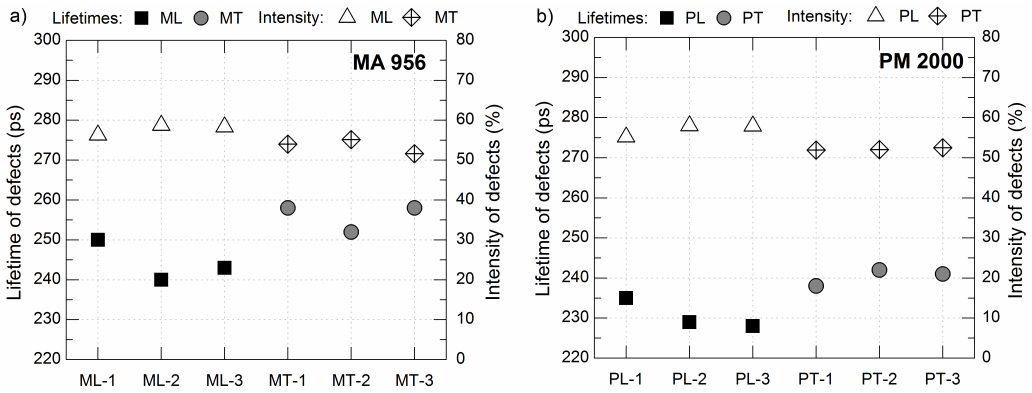

Figure 2. Positron lifetimes and intensities describing defects in MA 956 (a) and in PM 2000 (b).

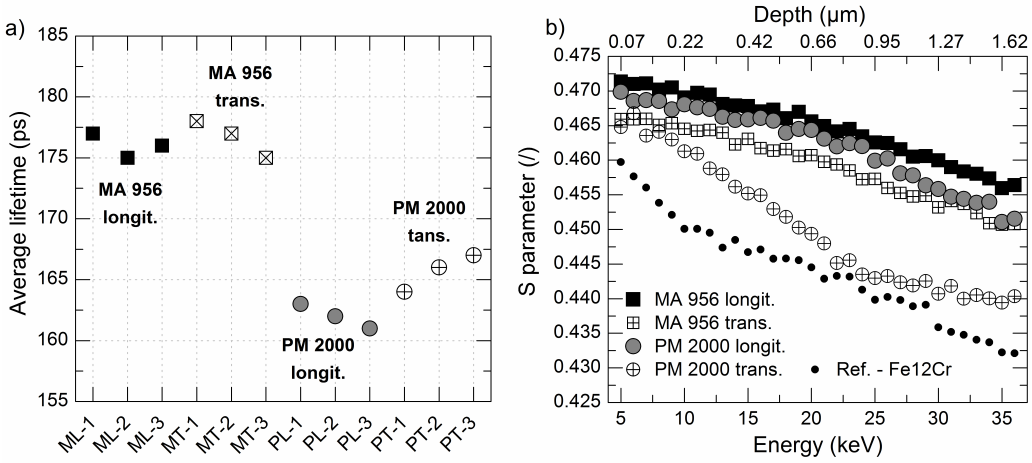

Figure 3. Average lifetime of MA 956 and PM 2000 (a). Dependency of S parameter and positron energy/implantation depth of investigated steels (b).

The investigated samples were later measured by DBS measurement. Results of DBS measurements are shown in Figure 3b - plot of S parameter versus the implantation energy/depth of positrons. The S parameter of PM 2000 in transversal cut demonstrated distinctive difference in comparison to the other samples, while these other samples (both orientations of MA 956 steel and PM 2000 sample in longitudinal cut) present only slight divergences to each other. PM 2000 (transversal cut) has the lowest S parameter and so the defect presence is the smallest there.

DBS measurement was completed by measurement of a reference sample - binary alloy Fe12Cr which was prepared by classical casting and then by the same process of cutting, grinding and polishing as the investigated steels. This sample was annealed after the mechanical treatment at 600°C during 2 hours for elimination of defect presence; therefore it can be considered as reference bulk structure. If the S parameter of the reference sample and the studied samples are confronted, it is found that both orientation of steels MA 956 and PM 2000 (even PM 2000 in transversal cut with minimum defects) contain defects over the equilibrium state described by Fe-12Cr. This is probably influence of mechanical alloying which creates strengthened structure with more lattice defects or it can be also an effect of yttria presence attracting positrons more than bulk as is published in [14].

It is not able to exactly determine the diffusion length (L^+) of positrons from our S parameters; therefore calculations of defect concentration performed according to [12], with using L^+ equal to 40 nm, can be only informative (Table 2). The calculations are based on concentration and size of yttria particles in MA 956 and PM 2000 found in [15], thus the concentration of vacancy clusters can be straight separated from the second component of PALS results (τ_D, I_D).

The average values of four-vacancy concentration for MA 956 are (1.43 ± 0.2) x 10^{23} m^{-3} in longitudinal cuts and (1.14 ± 0.06) x 10^{23} m^{-3} for transversal cuts. For steel PM 2000, the concentrations of thee-vacancies were found as (2.3 ± 0.03) x 10^{23} m^{-3} in longitudinal cuts and (1.47 ± 0.03) x 10^{23} m^{-3} for transversal cuts.

Table 2. Concentration of defects for both orientations of samples - MA 956 and PM 2000.

Sample	ML1	ML2	ML3	MT1	MT2	MT3	PL1	PL2	PL3	PT1	PT2	PT3
Concentration of defects (10^{23} m^{-3})	1.25	1.54	1.50	1.14	1.20	1.08	2.33	2.29	2.30	1.47	1.43	1.43

The difference of S parameters for individual sample orientations (Figure 3b) was visible for both studied steels, while PALS data observed similar presence of defects. The transversal cuts have smaller S parameter which means also lower presence of defects (probably lower concentration). This is in a good accordance with defect concentration calculated from PALS results. Though, it is

necessary to realize that these two techniques are not fully comparable due to observed depth of samples. They can rather complete each other.

4. Conclusion

In this paper, the structural inhomogeneity (especially a distribution of vacancy defects) was observed in two similar oxide-dispersion-strengthened steels. Two different orientations of samples (longitudinal and transversal cuts) for steels MA 956 and PM 2000 were studied in term of determining whether properties anisotropy can have connection with defect distribution in a sample. Results from positron annihilation lifetime spectroscopy showed existence of small or almost negligible differences in defect size or defect concentration for the individual samples prepared in both directions of cuts. This demonstrated sufficient homogeneity of the investigated steels concerning to vacancy defect presence up to 120 μm.

Though, the results of Doppler broadening spectroscopy observed visible differences mostly for different cuts of PM 2000. The variance was around 5 % of S parameter for PM 2000 and only around 1 % for MA 956. However, the Doppler broadening spectroscopy was performed only for one sample from each type of material and cut. Further, the measurement also describes shallow depth up to 1.6 μm. Thus, this difference could be formed during the process of sample preparation.

All samples of steel PM 2000 showed smaller presence of defects, probably defect concentration as was calculated form the lifetime spectroscopy, than the samples of MA 956. However, MA 956 introduced greater homogeneity in total.

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