Applied Surface Science xxx (2014) xxx–xxx

Contents lists available at ScienceDirect

Applied Surface Science

journal homepage: www.elsevier.com/locate/apsusc

Surface study of radiation damaged oxide dispersion strengthened steels

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ARTICLE INFO

Article history:
Received 9 January 2014
Received in revised form 26 February 2014
Accepted 26 February 2014
Available online xxx

Keywords:
RAFM steel
ODS steel
Positron annihilation
Helium ion implantation

ABSTRACT

In this paper, a structural analysis of Eurofer and ODS Eurofer was performed in terms of their radiation resistance. The radiation damage was simulated by helium ion implantation with kinetic energy of ions up to 500 keV. The implanted ions reached the depth of about 1.2 μm and the implantation dose was ~1×10^{18} ions/cm^2. The radiation damage (~45 dpa) was investigated by suitable positron annihilation technique–Doppler broadening spectroscopy which showed visible accumulation of defects due to the implantation. According our results, ODS Eurofer seems to be more radiation resistant than classic Eurofer.

1. Introduction

Reduced activation ferritic–martensitic (RAFM) steel – Eurofer and its oxide-dispersion-strengthened (ODS) form – ODS Eurofer are studied as the best materials for application in fusion reactors due to their good mechanical properties, high thermal stress capacity, compatibility with gas coolant and good resistance to radiation damage [1]. Even, ODS steels have improved strength at higher temperatures and better resistance to creep rupture in comparison to classic steels with the same chemical composition [2,3].

However, ODS steels have not yet been tested in radiation environment for longer time and the complex microstructural analyses of ODS alloys are also still missing. On that account, we focused on the post-irradiation behavior of ODS Eurofer and classic Eurofer mostly in term of microstructural study. The radiation damage was simulated by He^{2+} implantation during which the vacancy defects accumulated in the structure as a result of the helium nuclei–atom collisions [4]. Therefore, resistance to accumulation of radiation defect can be observed and the investigated structures can be compared.

2. Investigated samples

Two different structures of Eurofer were investigated: classic Eurofer and ODS Eurofer. The chemical composition of investigated steels (see Table 1) was observed by optical emission spectroscopy at the Institute of Materials at Slovak University of Technology.

RAFM structure of Eurofer was formed during austenitization at 980 °C for 1.5 h and cooling velocity achieved 5 °C/min. Then it was tempered at 760 °C during 30 min and cooled in air for material relaxation. ODS steel was produced from Eurofer 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 mixture was then consolidated using Hot Isostatic Press (HIP) at 1150 °C under a pressure of 103 MPa.

The samples of the investigated steels were prepared from as-received material by cutting the steel sheets into suitable pieces. In order to remove surface impurities, the sample surfaces were polished after the milling cutting. The treatment of samples can affect surface and subsurface layers up to 150 μm [5]. The roughness of the surface after grinding and polishing determined by atomic force microscopy [6] was up to 100 nm.

3. Experimental treatment and methods

The samples were loaded by radiation damage performed at a linear accelerator belonging to the Institute of Nuclear and Physical Engineering, Slovak University of Technology. Helium ions (He^{2+}) with a kinetic energy up to 500 keV were implanted into the samples. The implantation temperature achieved maximum of 62 °C; therefore it has no effect on the structure. The implantation depth was up to 1.2 μm according to SRIM calculation [7]. The implantation level was ~1×10^{18} ions cm^{-2} and the maximum

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http://dx.doi.org/10.1016/j.apsusc.2014.02.146
0169-4332/

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

Steels	C	Mn	Cr	Mo	Cu	Si	Nb	N	W	Ta	Y_2O_3	Fe
Eurofer	0.10	0.44	8.80	0.003	–	0.05	0.002	–	1.10	0.14	–	Balance
ODS Eurofer	0.10	0.44	8.80	0.003	–	0.05	0.002	–	1.10	0.14	0.30	Balance
Fe–9Cr (reference sample)	0.02	0.03	8.40	–	0.001	–	–	0.02	–	–	–	Balance

radiation damage, calculated from the average number of vacancies in 1 cm^2 of material obtained by SRIM, was around 45 dpa for the damaged zone.

During ion implantation, point defects accumulate into the structure as a result of the atom knocking-on by the helium nuclei [8]. Therefore, accumulation of small vacancy defects, which are typical for ferritic structure, was assumed in the investigated RAFM and ODS steels.

The samples were investigated by positron Doppler broadening spectroscopy (DBS). The DBS technique [9] with a conventional setup was applied during the measurement and DBS spectra were recorded by one HPGe detector with Gaussian resolution function of 1.24 keV. The energy windows for calculation of the S parameter is $|E\gamma - 511\ \text{keV}| < 0.83\ \text{keV}$ and for W parameter is $3\ \text{keV} < |E\gamma - 511\ \text{keV}| < 7.6\ \text{keV}$. Positrons used in the DBS measurements are acquired from a slow positron beam with ^{22}Na source and tungsten moderator [10]. The monoenergetic beam can achieve energies ranging from 0.5 to 36 keV. This technique can be used to study defect profiles as function of positron implantation depth in samples up to 1.6 μm. Higher positron energy causes deeper penetration of positrons into the sample which is defined by Markhov profile [11].

4. Results and discussion

The DBS results are given in both line-shape parameters: S parameter which value usually grows with an increase of the presence of defects, while W parameter commonly decreases in that time [11]. The measured data of the investigated steels were supplemented by a reference sample ferritic–martensitic steel Fe–9Cr (Table 1) which was formed and treated into the sample by similar way as classic Eurofer. The reference sample was annealed for 2 h at 600 °C in argon atmosphere for purpose of obtaining a bulk structure with minimum defects and an ideal defect-free S and W parameters for this kind of iron structure. The comparison of the investigated steels and the reference sample is for purpose of demonstration of residual stress accumulated in the investigated samples during the process of manufacture.

If we compare the defect depth profile of investigated steels before the implantation and the reference Fe–9Cr, a visible difference is found. The S parameter of the investigated steels is higher than the bulk values of Fe–9Cr (Fig. 1), which can be due to higher presence of precipitates or presence of more alloying elements in the investigated steels than in reference sample. The steels have also higher presence of residual defects which were removed in Fe–9Cr by annealing.

The S parameters of the non-implanted steels indicated a slight peak in depth of 470 nm which can describe defects formed during the preparation of samples (milling cutting, grinding and polishing) as was published in [5]. The investigated steels have a similar behavior of S parameter in non-implanted state; therefore these steels are almost identical from the view of defect presence. The variance is shown only in a behavior of W parameter indicating different defect surrounding in the individual investigated steels which is probably the effect of additional yttria in ODS Eurofer.

After the implantation, the depth profiles of S parameter changed significantly for both investigated materials (Fig. 2). It describes distinctive defect accumulation during the implantation of helium ions as was also published by Carvaliho et al. [12] for Eurofer implanted by a similar way with lower dose (10^{15} ions cm^{-2}). Both studies present that Bragg peak is wider and its maximum is located in a smaller depth (~900 nm in our research and ~600 nm for [12]) than it was calculated by SRIM (~1000 nm). It is due to heterogeneity of structure and the presence of lattice defects in the studied materials which are not included in SRIM calculations. The difference between our work and [12] is probably in a different process of sample preparation for individual experiments.

The increase was higher for classic Eurofer in comparison to ODS Eurofer. The maximum change of S values after the implantation was ~0.37 (increase about 8%) for Eurofer and ~0.25 (5%) for ODS Eurofer.

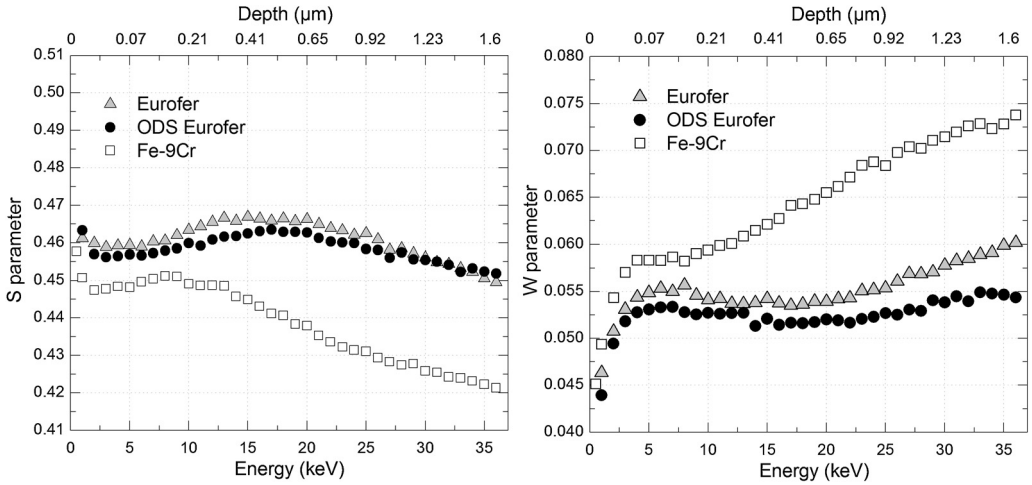

Fig. 1. Defect depth profile of non-implanted 9% Cr steels shown in line-shape S and W parameters as a function of positron depth (top axis) and positron energy (bottom axis).

Please cite this article in press as: J.Š. Veterníková, et al., Surface study of radiation damaged oxide dispersion strengthened steels, Appl. Surf. Sci. (2014), http://dx.doi.org/10.1016/j.apsusc.2014.02.146