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Helium implanted Eurofer97 characterized by positron beam Doppler broadening and Thermal Desorption Spectroscopy

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ABSTRACT

Reduced Activation Ferritic/Martensitic steels are being extensively studied because of their foreseen application in fusion and Generation IV fission reactors. To produce irradiation induced defects, Eurofer97 samples were implanted with helium at energies of 500 keV and 2 MeV and doses of 1×10^{15}–10^{16} He/cm^2, creating atomic displacements in the range 0.07–0.08 dpa. The implantation induced defects were characterized by positron beam Doppler Broadening (DB) and Thermal Desorption Spectroscopy (TDS). Results show that up to ~600 K peaks that can be attributed to He desorption from overpressured He_nV_m ($n > m$) clusters and vacancy assisted mechanism in the case of helium in the substitutional position. The temperature range 600–1200 K is related to the formation of larger clusters He_nV_m ($n < m$). The dissociation of the HeV and the phase transition attributed to a sharp peak in the TDS spectra at 1200 K. Above this temperature, the release of helium from bubbles is observed.

1. Introduction

Eurofer97 is a Reduced Activation Ferritic/Martensitic (RAFM) steel used as reference structural material for future fusion reactors. Eurofer97 steel is known to possess a high resistance against swelling from gaseous transmutation products (hydrogen, helium), has attractive mechanical properties and shows reduced activation behavior in a fusion neutron spectrum [1]. The effects of neutron irradiation and the accumulation of defects in this material are extensively studied as Eurofer97 will be used for constructing test blanket modules in the ITER fusion reactor [1,2]. As a consequence of the neutron irradiation, displacement damage will be created together with the production of hydrogen and helium [1,2]. This leads to changes in the microstructure and ultimately to changes in the material's mechanical properties. The work described here is part of a larger project in which Thermal Desorption Spectroscopy (TDS) and electron microscopy studies will be performed on Eurofer97 neutron irradiated in the High Flux Reactor at NRG. To mimic these neutron irradiations, samples are implanted with 500 keV and 2 MeV He^+ ions. The defect structures after implantation and annealing treatments are characterized with the positron beam Doppler Broadening technique (DB). The thermal behavior of the implanted He is studied in parallel by TDS.

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2. Experimental

2.1. Sample preparation

The samples used in this study were cut from the European Union Eurofer97 batch, produced by Böhler, Austria, with a nominal composition of Fe–9Cr–1 W–0.2 V–0.1Ta–0.1C (wt.%). Samples of $1.2 \times 1.2 \times 0.05$ cm^3 were cut so that after implantation nine small squares of $0.2 \times 0.2 \times 0.05$ cm^3 (for TDS) could be detached from the larger central piece (for DB) with the dimension of $1 \times 1 \times 0.5$ cm^3. To remove the surface damage introduced by cutting, the samples were annealed at 1253 K for 30 min. After this step, the samples were tempered at 1033 K for 90 min to regain the tempered martensitic structure. Both steps were followed by quenching. Before every annealing step the samples were electro-polished to remove possible surface contamination. This step was also performed after the annealing/tempering.

Low dose (1×10^{15}–1×10^{16} He/cm^2) room temperature helium implantations were performed at CEMHTI-CNRS Orléans, France. For the 500 keV and 2 MeV implantations the beam current was 270–280 nA and 120–212 nA, respectively, with implantation times ranging from 1 up to 15 h. TRIM [3] calculations for the dose of 1×10^{15} He/cm^2 are shown in Fig. 1. It is noticed that the defect production (dpa) has a maximum value of 0.08 and 0.07 dpa at a depth of 1 and 3 μm for 500 keV and 2 MeV, respectively. The maxima in the He distributions are slightly deeper, with a FWHM (Full

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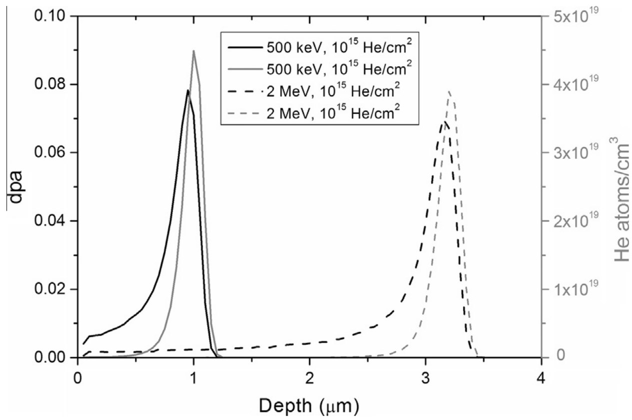

Fig. 1. TRIM simulation obtained for a dose of 1×10^{15} He/cm^2 in Eurofer97. Displacement per atom (black) and ion range (grey) as a function of depth. Ions with 500 keV energy in full line and with 2 MeV energy in dashed line.

Width at Half Maximum) of 0.2 μm. For the dose of 1×10^{16} He/cm^2 similar defect and He distributions are obtained with 10 times higher dpa levels.

2.2. Positron annihilation and VEPFIT analysis

Positron annihilation Doppler broadening (PADB) was used to monitor the formation and thermal behavior of the implantation induced defects. The annihilation of an electron with a positron produces two gammas, each with an energy around 511 keV. As a result of momentum conservation, the momentum of the electron positron pair results in a Doppler broadening of the measured 511 keV annihilation photo peak. This broadening is quantified by specific line shape parameters *S* and *W*. The *S* (sharpness) parameter is calculated as the ratio of the counts registered in a fixed central momentum window ($|p_{//}|$ <3.5×10^{-3} m_oc, where $p_{//}$ is the momentum of the electrons in the direction of the gamma emission, m_o is the electron rest mass and c is the speed of light) to the total number of counts in the photo peak. This choice of the momentum window makes the *S* parameter sensitive to annihilations with low momentum valence electrons. Similarly, the *W* (wing) parameter is obtained from the high momentum regions W_{left} and W_{right} ($10 \times 10^{-3}\, m_oc < |p_{//}| < 26 \times 10^{-3}\, m_oc$) and accounts for annihilations with high momentum core electrons. In general, for a positron trapped in an open volume defect (such as a dislocation, a vacancy or vacancy cluster) the probability of annihilations with core electrons is reduced compared with that for valence electrons resulting in a higher *S* parameter and lower *W* parameter value. Since for a defect free material the (material specific) *S (W)* parameter is the lowest (highest), all *S* and *W* values reported here are normalized with respect to these values of S_{ref} = 0.4532 and W_{ref} = 0.1038, respectively. Generally, the measured *S* parameter increases with increasing defect density or increasing defect size, with the *W* parameter showing the opposite behavior. In this study the PADB experiments were performed with the Delft Variable Energy Positron beam (VEP). Positrons emitted from a ^{22}Na source are after moderation to thermal energies and subsequent acceleration injected in the samples with a kinetic energy from 0.1 to 25 keV. The beam intensity is about 10^5 positrons per second and the beam diameter at target is 8 mm. The mean implantation depth of the positrons $\langle z\rangle$ scales with the implantation energy according to: $\langle z\rangle = A/\rho \cdot E^{1,62}$, with *A* a material independent parameter (4.0 μg cm^{-1} keV$^{-1,62}$), ρ the density (g/cm^3) and *E* the positron implantation energy (keV).

In Fe the implantation energy of 25 keV corresponds to an implantation depth of about 1 μm. In Fig. 2 the top horizontal axis

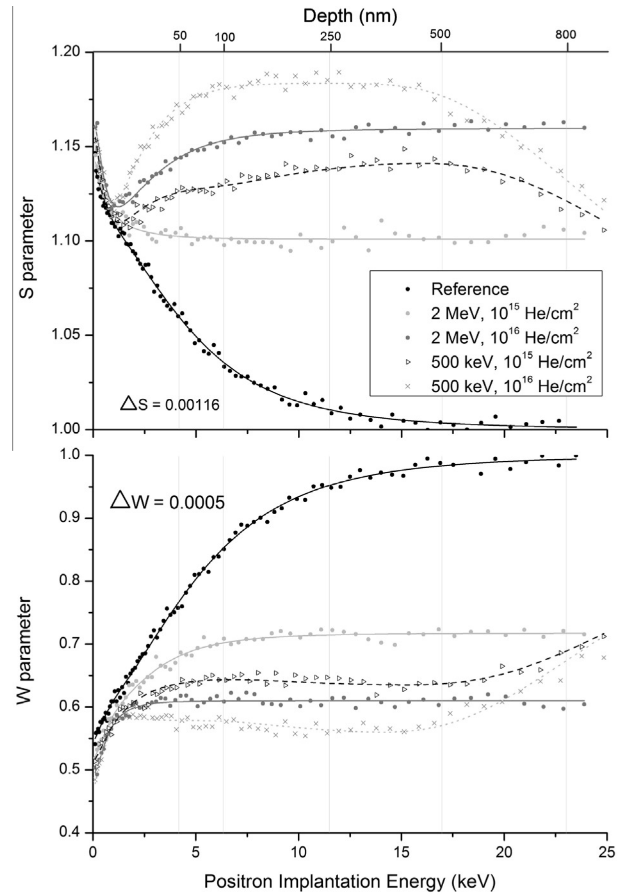

Fig. 2. Normalized *S* and *W* parameter as a function of positron depth (top axis) and positron implantation energy (bottom axis). The lines are obtained with VEPFIT.

represents the main implantation depth corresponding with the implantation energies shown at the bottom horizontal axis. The energy resolution of the detector setup is 1.2 keV at 511 keV. A detailed description of the experimental setup can be found in [4]. The Doppler broadening data were analyzed using the VEPFIT fitting and modeling program [5,6]. The program models and fits the data using a layered structure model. The implantation is modeled using a standard Makhovian implantation profile,

$$P(z,E) = \frac{2z}{z_o^2} e^{-\left(\frac{z}{z_o}\right)^2} \quad \text{with } z_o = < z > /\Gamma[(3/2)] \tag{1}$$

And $\langle z\rangle$ is the above defined mean implantation depth. The VEPFIT program describes the system as a stack of different positron trapping layers, taking positron inter-layer diffusion into account. To each layer a specific *S*-value is ascribed. The measured *S*-value for a given positron energy *E* is then given by

$$S(E) = f_{surf}(E)S_{surf} + \sum_{i=1}^{m} f_i(E)S_i \tag{2}$$

where f_i (*E*) is the fraction of positrons implanted at energy *E* which after thermalization and diffusion annihilate in layer *i* and f_{surf} is the fraction of positrons back-diffusing to the surface. A similar equation can be set up for *W* and by fitting the two sets of experimental data information about the layer specific *S* and *W* values, the positron diffusion length in the layers and the layer thicknesses can be obtained.

The *in situ* annealing DB experiments were performed for a number of selected annealing temperatures. Every temperature was reached by ramp annealing with a similar heating rate as