

Dose effects in He implanted Eurofer97 steel

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Abstract. Reduced Activation Ferritic/Martensitic steels are being extensively studied because of their foreseen application in fusion reactors. To reproduce neutron irradiation conditions, Eurofer97 samples were implanted at room temperature with helium ions at energies of 500 keV and 2 MeV and doses of 1×10^{15} - 10^{17} He /cm². The implantation induced defects were characterized by positron beam Doppler Broadening (DB). The samples were annealed in the range 300 – 1500 K, in 100 K steps. As the temperature increases, the annealing of vacancies and vacancy clusters is noticed and followed by the coalescence of He_nV_m clusters. At temperatures around 1200 K HeV pairs dissociate and bubbles are formed. Above 1300 K the helium release from bubbles is observed. The *S-W* graphs reveals that the samples have similar positron traps up to 1200 K. At 1200 K helium bubbles are noticed and the *S,W* pair shows a clearly distinct behaviour from the *S,W* values of vacancy type defects. As the temperature increases and helium is release, the *S,W* pairs shift towards the *S,W*'s of vacancies.

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 caused by 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 alterations 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 [3]. To mimic these neutron irradiations, in this work Eurofer97 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 present work is the continuation of a previous study



[4] in which the behaviour of He in tempered Eurofer97 steel was studied and helium release mechanism was suggested.

1. Experimental

The samples used in this study were cut from the Eurofer97 batch, produced by Böhler, Austria, with a nominal composition of Fe-9Cr-1W-0.2V-0.1Ta-0.1C (wt.%). The dimensions were $1.2 \times 1.2 \times 0.05 \text{ cm}^3$. In order to anneal the defects introduced by cutting and to obtain a tempered martensitic microstructure, the samples were first annealed for 30 min at 1253 K, and then tempered for 90 min at 1033 K, both followed by cooling in argon ambient.

Low dose ($1 \times 10^{15} - 1 \times 10^{16} \text{ He/cm}^2$) 500 keV and 2 MeV room temperature helium implantations were performed at CEMHTI-CNRS Orléans, France. In the case of the 2 MeV energy implanted samples, the dose range was extended to $1 \times 10^{17} \text{ He/cm}^2$. Details regarding the implantations can be found elsewhere [4]. The beam DB measurements were performed up to positron implantation energies of 25 keV, corresponding to a maximum probing depth in steel of approximately $1 \mu\text{m}$.

2. Results and discussion

Figure 1 shows the DB S -parameter as a function of the positron implantation energy (bottom axis) and positron mean implantation depth (top axis) for the reference and the implanted samples. For all implantations a higher S -parameter reflects a higher implantation dose. The lines through the data are the fits obtained with the VEPFIT program [5]. In case of the 2 MeV implantation, one layer defining the S -parameter plus a layer representing the bulk was sufficient to obtain a good fit. For the 500 keV

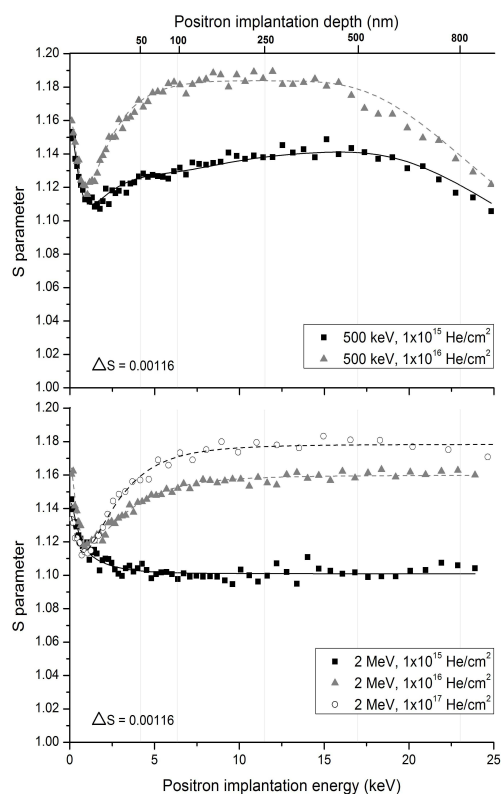


Figure 1. Normalized S parameter vs. positron implantation energy (bottom axis) and depth (top axis). Data fitted with VEPFIT.

implantation two layers (with thicknesses of 250 and 800 nm) plus a layer for the bulk were needed. This is consistent with the TRIM results which show that for the 2 MeV implantation the defect production profile is flat over the positron probing range while for the 500 keV implantation the defect profile peaks at the end of the positron range. The He/V (He per vacancy) ratio in the first layer (250 nm thick) of the 500 keV sample is comparable to that of the 2 MeV sample over the whole region probed by the positrons.

The fitted positron diffusion lengths ranges between 60 nm for the reference sample and less than 15 nm after helium implantation, indicative for high positron trapping efficiency.

Figure 2 shows the fitted S -parameter (measured at room temperature) as a function of annealing temperature. The annealing was performed *in situ*, inside the DB setup. With increasing annealing temperature the thickness of the second layer of the 500 keV samples was decreased in order to obtain a good fit. Nonetheless, the sum of the thicknesses of the first and second layers was kept constant. Above 1300 K, one layer describing the implantation effects and a layer associated with the bulk was sufficient to achieve satisfactory results.

Below 600 K a drop in the S -parameter is observed that is associated with the increase in the mobility of the vacancies and dissociation of vacancies from vacancy clusters [6]. This effect is more pronounced in the first layer of the 500 keV samples in comparison with the second layer for each implantation dose, respectively.

As the temperature increases to 800 K, a further slower decrease in the S values is observed in the first layer of the 500 keV implanted sample whereas a stable S parameter is noticed in the second layer. This decrease can be interpreted as a decrease of the number of positron traps (e.g. continuation of vacancy annealing) in the first layer.

In the range 800 – 1300 K multiple helium release mechanisms take place [4]. Between 800 – 1000 K the S parameter increases significantly both in the second fitted layer of the 500 keV samples and in the high dose (1×10^{17} He/cm²) 2 MeV implanted samples. This increase can be explained by growth of vacancy-helium clusters through coalescence or by Ostwald ripening. In the case of the latter process ultimately the more stable He_nV_m ($n, m = 1$) pairs are created. For the first layer of the 500 keV as well as for the lower dose (10^{15} - 10^{16} He/cm²) 2 MeV samples, the relatively stable S shows that the initial defect density in combination with the presence of Helium is crucial for the above growth processes to take place. Also the smaller distance to the surface which acts as a sink for defects is believed to play a role.

As the annealing temperature increases from 1000K to 1300 K the S -parameter drops because of the dissociation of HeV pairs. Retrapping of helium in the larger clusters occurs, forming He bubbles [4]. In this temperature interval the phase transition of Eurofer97 at 1160 K takes place [7].

For temperatures above 1300 K the release of He bubbles is expected [8, 9] and this is noticed as a decrease in the S value of the 500 keV, 1×10^{16} He/cm² implanted sample.

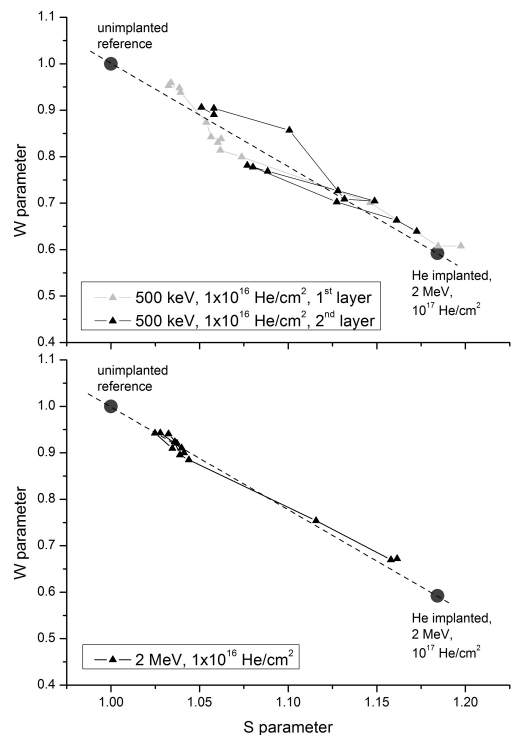


Figure 3. S vs. W parameter. Data fitted with VEPFIT.

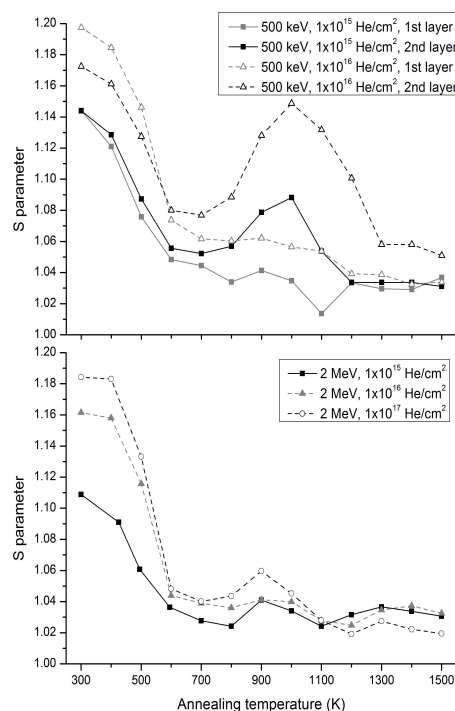


Figure 2. S parameter vs. annealing temperature. Data fitted with VEPFIT.

Figure 3 shows the S - W values of the near surface layers in the 1×10^{16} He/cm² samples. The dashed line connects the S - W values of an unimplanted Eurofer97 reference sample and the sample with the highest concentration of defects (2 MeV energy and a dose of 1×10^{17} He/cm²) and is therefore associated with Eurofer97 vacancy type defects. For temperatures up to 500 K the points of the second layer of the 500 keV sample are located very close to this line, revealing the vacancy type character of these defects. As the temperature increases to 800 K, the S - W points move down from this line due to an increase in the He/V ratio in the positron traps (over-pressurized He_nV_m clusters). In the temperature range 800 – 1200 K the S - W pairs shift back towards the vacancy type defects line. This behavior is in accordance with the idea of the Ostwald ripening as it produces He_nV_m ($n, m = 1$) pairs, sensed as vacancies. By coalescence bigger clusters may be formed as well but apparently with less probability. At 1200 K the S - W pairs appear above the line. This hints at the formation of larger helium filled voids, where due to the relatively low amount of He per vacancy the

helium is not sensed by positrons. At temperatures above 1300 K the bubbles may start to migrate and the remaining low concentration traps are again similar to vacancies.

Up to the temperature of 800 K, the S - W pairs the 2 MeV and the first layer of the 500 keV samples show a similar behavior as the ones of deeper layer of the 500 keV sample with S - W pairs ending up close to the line. However, the tendency to return into the direction of S - W characteristic for higher density of He_nV_m ($n, m = 1$) defects is not observed. This is explained by a lower overall initial defect concentration and a smaller He/V ratio. As the temperature further increases to 1500 K the location of the S - W pairs slightly moves into the direction of the reference defect free material, indicating that no major changes take place in this region.

3. Conclusions

He implantations have been performed in Eurofer97 steel with energies of 500 keV and 2 MeV and doses in the range of 1×10^{15} - 10^{17} He/cm². By annealing the samples up to 1500 K it is possible to observe an initial decrease of the S -parameter, associated with the annealing of vacancies and vacancy clusters, followed by a significant increase of this value due to growth of the helium-vacancy clusters. At temperatures in the range 1100 – 1200 K HeV pairs dissociate and retrapping by the bigger clusters occurs and helium filled bubbles are formed. Above 1300 K these bubbles anneal out by dissociation or migration, causing a drop in the S -parameter.

The S - W graph reveals that the positron traps of the samples implanted with a dose of 1×10^{16} He/cm² are of similar character up to a temperature of 800 K. The different behavior of S - W pairs above this temperature show that implantation conditions, such as dose and energy, are determining the thermal evolution of implantation (and radiation) induced defects.

Acknowledgements

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