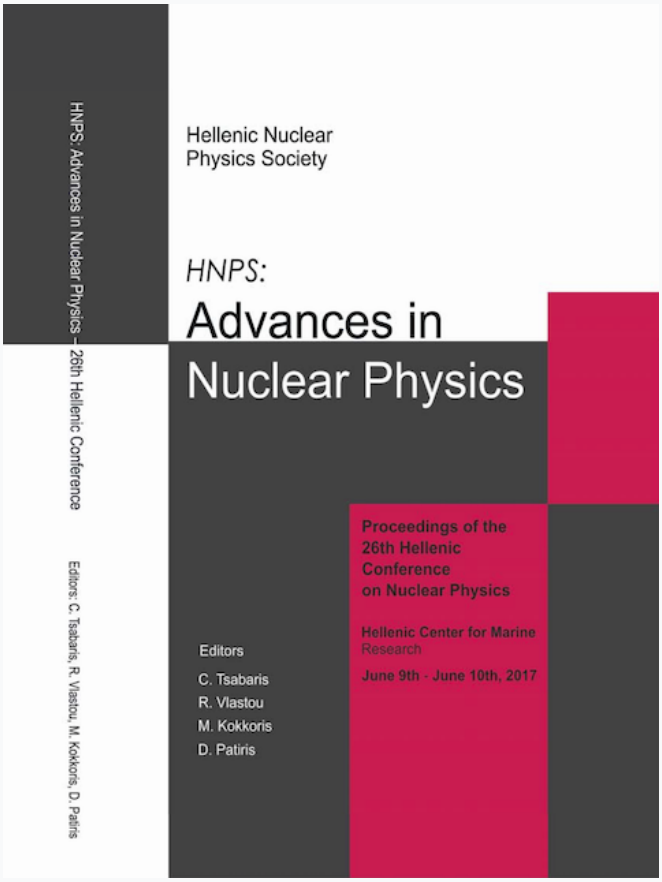
HNPS Advances in Nuclear Physics

Vol 25 (2017)

HNPS2017



Resistivity Recovery Stages of Proton Irradiated EUROFER97 Steel

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doi: 10.12681/hnps.1986

Resistivity Recovery Stages of Proton Irradiated EUROFER97 Steel

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Abstract

Irradiation defects were created in EUROFER97 steel, after 5 MeV proton irradiation at the cryogenic temperature of 45 K. Evolution of the defect concentration during recovery annealing was studied by *in-situ* electrical resistivity measurements. EUROFER97 is a Reduced Activation Ferritic Martensitic (RAFM) steel which is considered to be the primary choice as a structural material for future fusion power plants. The resistivity recovery spectra at different levels of irradiation dose indicate the presence of interactions between radiation defects and EUROFER97 solute atoms.
Keywords: proton irradiation; resistivity recovery; EUROFER97;

1. Introduction

EUROFER97 steel, based on the FeCr system, is considered to be the most promising structural material for the future fusion power plant. According to previous studies [1,2] it exhibits lower activation levels than the conventional Cr-Mo steels, while its good mechanical and physical properties are retained. However, the use of EUROFER97 during long time service at high temperatures (thermal ageing) and under low temperature irradiation (300–650 K) can significantly affect its mechanical properties (thermal creep, irradiation embrittlement). Point defects play an important role in the response of materials to irradiation so it is necessary to study their properties also in EUROFER97.

In the present work, we investigate by electrical resistivity measurements the recovery of radiation defects produced in EUROFER97 during 5 MeV proton irradiation at low-temperature. The recovery is mainly due to migration, interaction and annihilation of point defects. The presence of different solute atoms in EUROFER97 like W, V, Ta, C and mostly Cr affects the migration of interstitial and vacancy defects. The influence of dose levels is revealed by comparing the resistivity recovery of low and medium level dose.

2. Experimental Details

As starting material for this study a rectangular bar of EUROFER97 (Heat E83698) was obtained from the Karlsruhe Institute of Technology. The alloy has the following

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chemical composition in wt.%: 0.11C, 8.9Cr, 0.42Mn, 0.19V, 1.10W, 0.14Ta, balance Fe [1]. A 25 × 10 × 0.5 mm plate was cut from the starting material by a diamond raw. Foils of 100 μm thickness were obtained by cold rolling. Subsequently, the final thickness of 50 μm was obtained by electro-chemical thinning in a solution prepared by mixing 40 ml H_2SO_4 (96%) and 40 ml H_3PO_4 (85%). A platinum mesh was used as cathode and a DC voltage of 2.2 V was applied. The temperature of the solution was 70^{o}C and the mean thickness reduction rate was about 0.9 μm/min. After the thinning process the specimens exhibit a bright polished surface. Rectangular specimens 15 mm long and 2 mm wide were cut from the foils and annealed for 1 h at 700^{o}C under oil-free vacuum of 10^{-6} mbar in order to relieve stresses created during cold working. Current and potential leads of pure Fe were spot-welded on the specimens for performing the electrical resistivity measurements according to the standard DC four-probe method. Current polarity reversal was used to eliminate thermal voltages.

Irradiations were performed in the dedicated materials irradiation facility IR^2 at the TANDEM accelerator of NCSR "Demokritos" [3], which offers the capability for *in-situ* measurement of the electrical resistivity during and after irradiation. The specimens where irradiated with 5 MeV protons. During the irradiation, the sample temperature was kept at 45 K by a closed-cycle helium refrigerator coupled to the accelerator beam line. At specific time intervals the proton beam was interrupted and the sample was cooled down to 20 K where the resistivity was measured. Data for the samples and irradiation conditions are given in Table 1, where Φ_0 is the total dose and $\Delta\rho_0$ is the total irradiation induced resistivity increase.

Table 1. Irradiation conditions

	Φ_0 (cm^{-2})	Flux (cm^{-2}/s)	$\Delta\rho_0$ (μΩ-cm)
Low Dose	1.55×10^{15}	1.91×10^{11}	0.234
Medium Dose	4.37×10^{15}	1.74×10^{11}	0.650

After the total proton dose was delivered, recovery annealing up to 760 K was performed *in-situ* without removing the sample from the irradiation chamber. At the end of each annealing step, the sample was rapidly quenched to the base temperature of 20 K and the residual resistivity was measured. Its recovery value was monitored as a function of annealing temperature.

3. Results and Discussion

The increase of the electrical resistivity due to the proton irradiation of EUROFER97 alloys is shown in Fig. 1 as a function of dose. The observed almost linear increase indicates that there is a constant accumulation of radiation defects. This is due to the significantly reduced mobility of defects at the irradiation temperature of 50 K which results in a low likelihood for annihilation. By fitting a linear expression to the data we obtain the slope which is 1.51 and 1.48×10^{-22} Ω-cm^3 for low and medium dose, respectively. It is noted that the difference between the slopes is lower than 2 %, which is within experimental resolution.

For a proper estimation of the amount of damage generated by proton irradiation a simulation was performed by mean of the SRIM code. A displacement threshold energy of $T_d = 40$ eV has been used, which is the value typically used for Fe. The simulation results showed that the protons penetrate through the 50 μm thick target and the value of the damage cross-section was found equal to $\sigma_D \approx 1.4 \times 10^{-20}$ cm^2.

The resistivity increase as a function of dose can be written as

$$\Delta\rho = \rho_F c_F = \rho_F \sigma_F \Phi,$$

where c_F is the concentration of generated Frenkel pair defects, ρ_F is the resistivity per unit concentration of Frenkel pairs and σ_D is the damage cross section. Thus, the defect concentration in the specimens at the end of the irradiation is estimated to be $c_F \approx 20$ and 60 ppm for low and medium dose, respectively. Dividing the slope of the resistivity damage curves by σ_D we obtain the value of ρ_F, which is 1.1×10^{-2} Ω-cm. Compared to the Frenkel pair resistivity of pure Fe $\rho_F = 3 \times 10^{-3}$ Ω-cm the value obtained here is 4 times higher. This is in agreement with previous results [4] who observed an up to 5-fold increase of ρ_F in proton irradiated Fe-Cr-C with Cr concentration of 10 at.%.

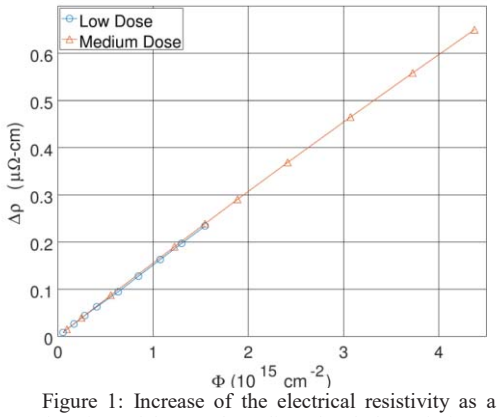

Figure 1: Increase of the electrical resistivity as a function of low and medium level dose during 5 MeV proton irradiation.

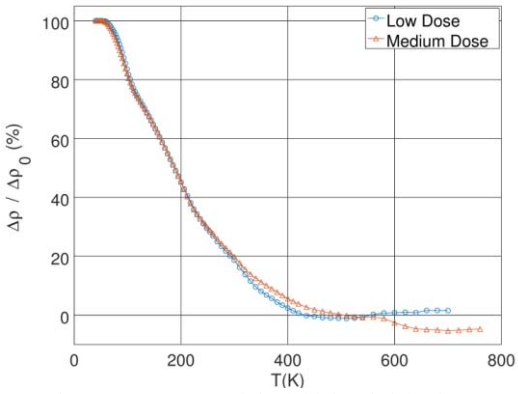

Figure 2: Recovery of the initial resistivity increase as a function of annealing temperature of proton irradiated EUROFER97.

Fig. 2 shows the resistivity recovery of EUROFER97 for low and medium dose as a function of annealing temperature *T*. The ratio $\Delta\rho(T)/\Delta\rho_0$, where $\Delta\rho_0$ is the total resistivity increase and $\Delta\rho(T)$ is the remaining resistivity after annealing at temperature *T*, reflects the fraction of radiation defects that survive after annealing at the given temperature. It is observed that the recovery proceeds similarly in the two experiments, however at the higher dose the recovery process slows down above about 250 K. Notably, in both curves the resistivity regains its pre-irradiation value at T=500 K. At higher temperatures, only at medium dose level irradiation, the resistivity of EUROFER97 became lower than its pre- irradiation value and this is probably due to solute atom re-ordering taking place during the migration of remaining radiation defects.

In Fig. 3 the recovery rate $-\Delta\rho_0^{-1}(d\Delta\rho(T)/dT)$ is presented as a function of annealing temperature. This graph assists in the identification of temperature regions of fast recovery that may be associated with different defect reactions. The peaks in fig. 3, show the recovery stages, labeled from I to IV according to previous work [5]. Stage I is centered at ≈100 K and is attributed to the correlated recombination of Frenkel pairs by the migration of self-interstitial atoms (SIA) to their corresponding near lattice vacancies. Stage II is observed at ≈210 K and is attributed to the release of SIA defects captured by alloy solute atoms (Cr, C, N) during stage I migration. Stage III is associated with vacancy migration and is observed at ≈320 K. The recovery is very similar in both dose levels. This means that the recovery processes are independent of the initial defect numbers. In terms of defect kinetics, the reaction processes are 1st order. Stage IV is observed only at medium dose level must be due to migration of EUROFER97 atoms and the formation of clusters. The atoms are

removed from the Fe matrix and their contribution to the resistivity of the steel is reduced below the initial one.

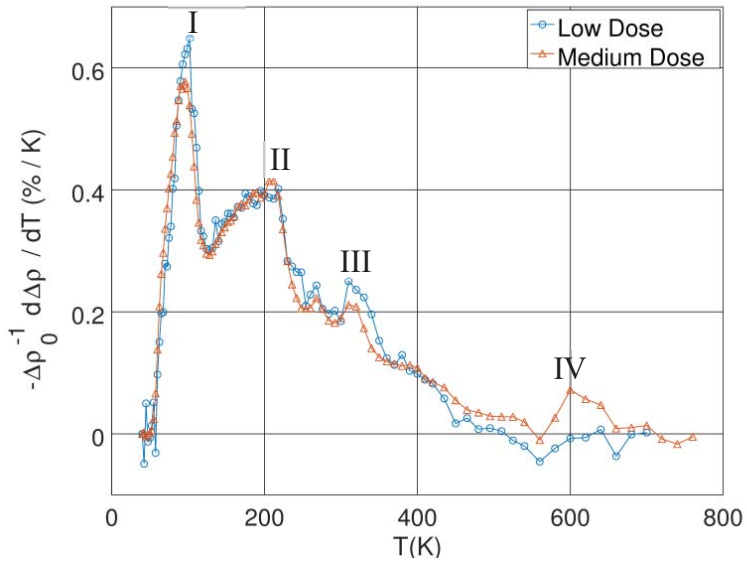

Figure 2: Resistivity recovery rate as function of annealing temperature in proton irradiated EUROFER97.

4. Conclusions

The resistivity recovery in EUROFER97 has been measured after 5 MeV proton irradiation at 45 K. A number of distinct peaks were observed which were identified as recovery stages I-IV and each has been linked to a different thermally activated mechanism. The results showed that there is significant interaction between EUROFER97 solute atoms and interstitial and vacancy defects. The dose levels employed in this work showed minor effects on the recovery but irradiation at even higher doses may be needed in order to reveal possible dose effects.

Acknowledgments

This work has been carried out within the framework of the EUROfusion Consortium and has received funding from the Euratom research and training programme 2014-2018 under grant agreement No 633053. The views and opinions expressed herein do not necessarily reflect those of the European Commission.

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