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Comparative small angle neutron scattering (SANS) study of Eurofer97 steel neutron irradiated in mixed (HFR) and fast spectra (BOR60) reactors

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ABSTRACT

This contribution presents a comparative microstructural investigation, carried out by Small-Angle Neutron Scattering (SANS), of ferritic/martensitic steel Eurofer97 (0.12 C, 9 Cr, 0.2 V, 1.08 W wt%) neutron irradiated at two different neutron sources, the HFR-Petten (SPICE experiment) and the BOR60 reactor (ARBOR experiment). The investigated "SPICE" sample had been irradiated to 16 dpa at 250 °C, the investigated "ARBOR" one had been irradiated to 32 dpa at 330 °C. The SANS measurements were carried under a 1 T magnetic field to separate nuclear and magnetic SANS components; a reference, un-irradiated Eurofer sample was also measured to evaluate as accurately as possible the genuine effect of the irradiation on the microstructure. The detected increase in the respective SANS cross-sections of these two samples under irradiation is attributed primarily to the presence of micro-voids, for neutron contrast reasons; it is quite similar in the two samples, despite the higher irradiation dose and temperature of the "ARBOR" sample with respect to the "SPICE" one. This is tentatively correlated with the higher helium content produced under HFR irradiation, playing an important role to stabilize the micro-voids under irradiation. In fact, the size distributions obtained by transformation of the SANS data yield a micro-void volume fraction of 1.3% for the "SPICE" sample and of 0.6% for the "ARBOR" one.

1. Introduction

The behavior of Eurofer97 steel (0.12 C, 9 Cr, 0.2 V, 1.08 W wt%) under neutron irradiation has been extensively investigated, both by post-irradiation mechanical testing and by microstructural examinations, in a variety of irradiation conditions to predict as accurately as possible its performance in service; in fact this steel is the European reference for fusion applications. More specifically, it has been neutron irradiated at two different neutron sources, the High Flux Reactor (HFR) – Petten ("SPICE" irradiation experiment, ref. [1,2]) and the BOR60 Reactor ("ARBOR" irradiation experiment, ref. [3,4]). These two experiments were designed to investigate changes in Eurofer mechanical properties and microstructure under neutron irradiation, at $T \leq 450$ °C to a volume average dose of 16.3 dpa in the case of SPICE and at 330 °C up to 70 dpa in the case of ARBOR. Post-irradiation microstructural characterization has been carried out by transmission electron microscopy (TEM) [5–8] for ARBOR samples; TEM and small-angle neutron scattering (SANS) results on SPICE samples have also been published [9–13]. In this paper SANS results are compared, obtained on one ARBOR and one SPICE sample, already characterized by other techniques, in order to contribute in understanding the microstructural effects produced in Eurofer97 steel at different neutron sources. In general, quantitative comparisons between samples of a same steel irradiated by different neutron irradiation experiments (not necessarily at different neutron sources) are not straightforward, mostly because it is not possible to perfectly monitor and reproduce parameters such as the irradiation temperature, for instance, and consequent effects on the material. Additionally, in the present case the two investigated samples, available at the time when the SANS experiments had been scheduled, differ not only in irradiation temperature but much more in nominal irradiation dose. Therefore, the experimental results and suggested interpretation presented in this paper are not intended as conclusive ones but as a first step to contribute

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in understanding this complex matter, to be hopefully soon completed by further investigations of ARBOR and SPICE samples, irradiated to a same nominal dose level.

2. Material characterization

The following Eurofer97 samples were investigated by SANS:

Un-irradiated reference Eurofer 97-1 Heat E 83697 (980 °C 0.5 h/air + 760 °C 1.5 h/air)

"SPICE" Eurofer 97-1 E 83697 (YW01) 16.3 dpa 250 °C (1040 °C/0.5 h + 760 °C/1.5 h)

"ARBOR" Eurofer 97-1 E 83697 (E102K) 31.8 dpa 332 °C (980 °C/0.5 h + 760 °C/1.5 h).

The un-irradiated reference was machined as a platelet $1 \times 1 \times 0.1$ cm^3. The two irradiated samples were obtained by cutting, in the hot cells, from each of the corresponding KLST samples a slice $0.8 \times 0.4 \times 0.1$ cm^3; they were subsequently mounted each one on a Cd shielded sample-holder to allow safe transportation to the neutron source and manipulation during the SANS experiment (activity level 4.42×10^{10} Bq, contact dose rate in an Al capsule 14 mSv/h).

TEM characterization of precipitates in un-irradiated Eurofer97 can be found in ref. [14]. Post-irradiation characterization relating to the SPICE experiment is reported in ref. [1]. TEM studies of SPICE samples are still underway, TEM results on HFR irradiated Eurofer97 up to 8.3 dose level ("SUMO" experiment), focusing mainly on the characterization of dislocation loops and Burgers vectors orientation, are reported in ref. [13]. Concerning the ARBOR material, ref. [5] provides an assessment of neutron irradiation effects in Eurofer97. Ref. [5] also presents the results of TEM observations on ARBOR samples, showing that the volume density of the observed micro-voids and dislocation loops increases of almost one order of magnitude between 15 dpa and 32 dpa irradiation dose. New TEM results on these samples are presented at this conference [7] and shown in Fig. 1; they indicate a much smaller density of micro-voids compared to SPICE.

3. Experimental technique

General information on the SANS technique can be found in refs. [15,16] and in previous works on fusion steels [9–12]. Defining the modulus of the scattering vector $Q = 4\pi \sin\theta/\lambda$ (where 2θ is the full scattering angle and λ the neutron wavelength), in the case of magnetic samples the total SANS cross-section $d\Sigma(Q)/d\Omega$ (where Ω stands for the solid angle) can be written as the sum of two terms, a nuclear and a magnetic one

$$d\Sigma(Q)/d\Omega = d\Sigma(Q)/d\Omega_{nucl} + d\Sigma(Q)/d\Omega_{mag}\sin^2\alpha \quad (1)$$

where α is the azimuthal angle on the detector plane. Applying an external magnetic field to saturate the sample magnetization in the sample, the ratio $R(Q)$ of the SANS components perpendicular and parallel to this field

$$R(Q) = \frac{d\Sigma(Q)/d\Omega_{nucl} + d\Sigma(Q)/d\Omega_{mag}}{d\Sigma(Q)/d\Omega_{nucl}} = 1 + (\Delta\rho)^2_{mag}/(\Delta\rho)^2_{nucl} \quad (2)$$

is related to the composition of the microstructural inhomogeneities and its dependence on Q implies that defects of different size or composition are present in the investigated sample, $(\Delta\rho)^2$ being the "contrast" or square difference in neutron scattering length density (nuclear and magnetic respectively) between the observed nuclear and magnetic inhomogeneities and the matrix [15,16]. In the case of Eurofer97, assuming that the carbide precipitate composition is $Cr_{14}Fe_8W_{0.7}V_{0.3}C_6$ [14] a contrast value of 2.13 10^{20} cm^{-4} is found, while for the case of micro-voids the contrast is equal to the scattering length density of Eurofer97 itself, that is 5.51 10^{21} cm^{-4}, more than one order of magnitude larger.

Assuming that the investigated material is a diluted system of inhomogeneities, the SANS nuclear and magnetic cross-sections can each one be written as

$$d\Sigma(Q)/d\Omega = (\Delta\rho)^2 \int_0^\infty dR\, N(R)\, V^2(R)\, |F(Q,R)|^2 \quad (3)$$

where $N(R)dR$ is the number per unit volume of defects with a size between R and $R+dR$, V their volume and $|F(Q, R)|^2$ their form factor (assumed spherical in this case) and $(\Delta\rho)^2$ is the nuclear or magnetic "contrast". The volume distribution function is defined as:

$$D(R) = N(R)R^3 \quad (4)$$

$N(R)$ was determined, by transformation of Eq. (3), using the method described in [17] and more recently discussed in [18–20]. This code assumes that the size distribution function can be described by a set of cubic B-spline functions, with equispaced knots in log R scale (to account for the simultaneous presence of defects sizes differing in order of magnitude) and the constraint that the distribution is positive or null. The number of splines is determined by the R-range where the size distribution is to be explored

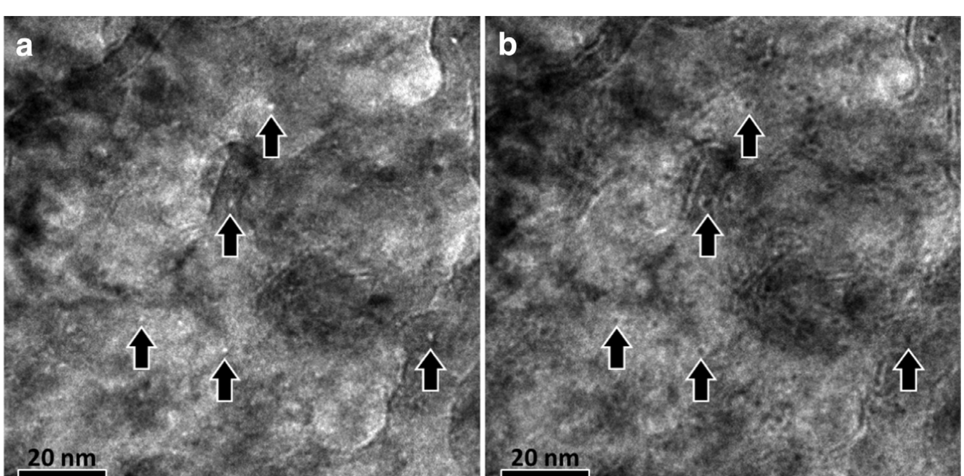

Fig. 1. TEM micrograph for Eurofer97/1 irradiated at 332 °C to 32 dpa ("ARBOR") in a) underfocus (−500 nm) and b) overfocus (+500 nm) condition. Some voids are marked by arrows [7].