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Micro-structural effects of irradiation temperature and helium content in neutron irradiated B-alloyed Eurofer97-1 steel

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ABSTRACT

The micro-structural effects of different neutron irradiation temperatures and helium contents, for 16 dpa dose, have been investigated by means of small-angle neutron scattering (SANS) in B-alloyed ferritic/martensitic steel Eurofer97-1 (0.12 C, 9 Cr, 0.2 V, 1.08 W wt%, B concentrations up to 1000 ppm); due to B transmutations, fusion relevant He/dpa values are expected to be produced under neutron irradiation. SANS measurements have been carried out on a sample irradiated at 350 °C, with estimated helium content of 5600 appm, and compared to previous SANS results, obtained on two other irradiated samples of this same B-alloyed steel. These new measurements confirm that for such high helium contents the SANS cross-section increases in order of magnitude and the magnetic SANS component is strongly reduced, compared to lower helium content (400 appm). Such effects are attributed to increase in helium bubbles density and to the presence of micro-cavities, produced after dissolution of large B-carbides. The SANS data analysis procedure has been improved, also thanks to the additional information provided by the new measurements, and more accurate helium bubble size distributions have been obtained for all the investigated samples. For 5600 appm helium content, bubble volume fractions are found of 0.025 for the sample irradiated at 350 °C and of 0.041 for the previously investigated sample irradiated at 400 °C, significantly increasing with the irradiation temperature. These values are approximately one order of magnitude larger than the value of 0.003 previously found for the sample with 400 appm helium. The size distributions are compared with electron microscopy observations of these same samples. It appears that the occurrence of complex micro-structural changes in irradiated Eurofer97-1 steel should be taken in due account when considering its application under high He/dpa ratio values.

1. Introduction

Within the frame of the "SPICE" European irradiation programme [1], the standard composition of the well-known ferritic/martensitic steel Eurofer97-1 has been modified introducing natural B contents variable between 10 ppm and 1000 ppm: in this way, under fission neutron irradiation fusion relevant helium contents and He/dpa ratios are expected to be produced by B activation. Such B-alloyed Eurofer97-1 heats have been neutron irradiated at the High Flux Reactor of the JRC-Petten, to a dose level of 16 dpa (displacement per atom) at temperatures of 250 °C, 300 °C, 350 °C and 450 °C. Reference is made to [1–7] for results of post-irradiation mechanical testing and observations by transmission electron microscopy (TEM). Results obtained on some of these sample by means of small-angle neutron scattering (SANS) are presented in Refs. [8–12]. More specifically, the results presented in Ref. [10] have shown that an Eurofer97-1 sample, with estimated helium content of 5600 appm after irradiation at 400 °C, exhibits a SANS cross section more than one order of magnitude larger compared to a sample of the same steel, irradiated at 450 °C, with estimated helium content of 400 appm helium. Moreover, for that sample containing 5600 appm helium, a strong reduction of the magnetic SANS component is observed. These SANS effects have been tentatively attributed to complex micro-structural modifications in the investigated steel. Namely, it has been suggested that for 5600 helium content the bubble distribution increases both in size and volume fraction; furthermore, the observed reduction in neutron magnetic scattering length density of the Eurofer97-1 matrix has been associated to the presence of micro-cavities as large as 10 μm, originated by the dissolution of B-carbides due to transmutation under irradiation [10].

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In order to confirm and further investigate the origin of this simultaneous increase in SANS cross-section and decrease in magnetic SANS component, new SANS measurements have been carried out on another Eurofer97-1 sample containing 5600 appm helium and irradiated at 350 °C. These new SANS results have provided additional experimental information, useful also to improve the previously adopted SANS data analysis procedure. In this way, the uncertainties associated to the fitting have been reduced and more accurate size distributions have been obtained for all the investigated irradiation conditions, namely: three different irradiation temperatures and two helium contents, for same dose level of 16 dpa. As discussed in the previous papers [10,11] and here below (Section 3), the micro-structural effects of neutron irradiation in such B-alloyed are very complex: in fact, in addition to the effects relating to the dissolution of large B-carbides, a simultaneous occurrence of micro-voids and helium bubbles is to be expected, hardly distinguishable from each other both by SANS and by TEM. Therefore, the suggested interpretation of the SANS results and related analysis is not intended as a conclusive one, but as a new contribution to progress in understanding the micro-structural radiation damage effects in Eurofer97-1 under conditions close to those expected in a fusion reactor.

2. Material characterization

The standard chemical composition of Eurofer97-1 is the following: 0.12 C, 9 Cr, 0.2 V, 1.08 W Fe bal wt%. It has been modified introducing, by mechanical alloying, ^{10}B contents of 0.0083 mass-% (“ADS3” heat) and of 0.1120 mass-% (“ADS4” heat): under neutron irradiation, they correspond to estimated helium concentrations of 400 appm and 5600 appm respectively. The irradiated samples prepared for the SANS measurements were platelets, approximately 4×9 mm^2 in surface and 1 mm thick; they were cut from the KLST specimens prepared for post-irradiation mechanical testing [1]. The reference, un-irradiated samples were approximately 1 cm^2 in surface and 1 mm thick; they had been submitted to the standard treatment 1040 °C 30′ + 760 °C 1.5 h, like the irradiated ones. The micro-structural effect of ageing at 450 °C, for a duration equivalent to the irradiation time (770 days) has been checked experimentally, finding no differences between the SANS cross-sections of the un-aged and aged sample [12].

TEM observations of these irradiated samples [3,6] show various morphological changes, particularly in the samples containing 5600 appm helium. In fact, under neutron irradiation the dissolution of B precipitates (BN, $M_{23}(C,B)_6$) leaves empty regions or micro-cavities as large as a few μm. Around such cavities subsequent halos are formed with high concentrations of helium bubbles or micro-voids, often characterized by bi-modal size distributions [3]. This effect, theoretically investigated for the more general case of transmutation-induced deposition profiles in steels [13], is shown in Fig. 1 referring to an Eurofer97-1 sample with 5600 appm helium after irradiation at 300 °C–16 dpa [14]. As discussed in Refs. [3,6], the TEM observed bubble distributions appear much more influenced by the ^{10}B content, therefore by the helium content, than by the irradiation temperature. It is noted that the TEM resolution power decreases strongly for sizes below 10 Å and that it is not easy to distinguish by this technique helium bubbles from micro-voids. The size histograms presented in Ref. [6] are compared with the SANS size distributions in Section 3 here below.

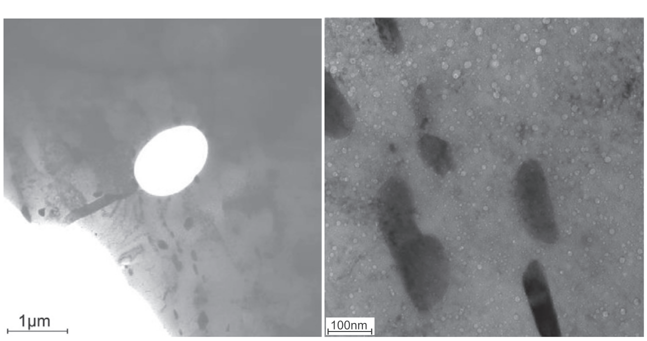

Fig. 1. Eurofer97-1 sample, containing 5600 appm helium after irradiation at 300 °C to 16 dpa; formation of halos containing bubbles/cavities around the hole [14].

3. Experimental technique and data analysis

The application of the SANS technique to the case of a magnetic steel has been presented in the previous works on this subject [8–12]; more general information on SANS can be found in Refs. [15,16]. The measurements have to be carried out applying to the investigated sample an external magnetic field of at least 1 T in order to saturate its magnetization. In this way, it is possible to measure separately the nuclear and magnetic components of the SANS cross-section, defined as follows:

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

where 2θ is the full scattering angle, λ the neutron wavelength, $Q = 4\pi\sin\theta/\lambda$ the modulus of the scattering vector, α the azimutal angle on the detector plane and Ω stands for the solid angle. The ratio of the SANS components measured in the directions perpendicular and parallel to the magnetic field is defined as follows:

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

it is related to the nuclear and magnetic square differences in neutron scattering length density between the defects and the matrix,$(\Delta\rho)^2_{nucl}$ and $(\Delta\rho)^2_{mag}$ respectively [15,16]; its dependence on Q implies that defects of different composition contribute in the measured total SANS cross-section. In Eurofer97-1, assuming for the carbides a composition $Cr_{14}Fe_8W_{0.7}V_{0.3}C_6$ [17] a nuclear contrast value of 2.13 10^{20} cm^{-4} is found for such precipitates; for micro-voids, the contrast is equal to the scattering length density of Eurofer97-1 itself, that is 5.51 10^{21} cm^{-4}, while for helium bubbles it is 4.88 10^{21} cm^{-4} [16]. Therefore, for comparable values of the corresponding volume fractions, both helium bubbles and micro-voids are expected to give rise to SANS effects one order of magnitude larger than precipitates, but on the other hand are quite difficult to be distinguished from one another.

The distributions of the scattering defects are obtained by inverse transformation of the experimental data. Namely, if their volume fraction is low and there is no inter-particle interference (dilute system), 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 $N(R)$ is the number per unit volume of defects with a size between R and $R + dR$, V their volume, $|F(Q, R)|^2$ their form factor (assumed spherical in this case) and $(\Delta\rho)^2$ is the nuclear or magnetic “contrast”.

The volume distribution function $D(R)$, average defect radius, $\langle R\rangle$, and volume fraction, f, are defined respectively as follows:

$$D(R) = N(R)R^3 \quad (5)$$

$$< R > = \int_0^\infty dR\, N(R)R / . \int_0^\infty dR\, N(R) \quad (6)$$

$$f = \int_0^\infty dR\, N(R)V(R) / .(\Delta\rho^2)\, V_{tot} \quad (7)$$

where V_{tot} is the total volume of the investigated sample.

$N(R)$ was determined by the mathematical method described in Ref. [18] and successfully utilized for several studies on technical steels, particularly those in Refs. [19,20]. This fitting procedure assumes no a-

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