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Journal of Nuclear Materials 258–263 (1998) 1291–1294

journal of nuclear materials

Microstructural characterisation of F82H-mod. steel using small-angle neutron scattering

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Abstract

Small-angle neutron scattering (SANS) measurements have been carried out on samples of F82H-mod. martensitic steel submitted to different treatments: (a) hardening, (b) tempering and (c) ageing. Size distribution functions referable to the precipitates, formed during the different treatments are obtained both for nuclear and magnetic SANS components. The results, discussed making reference to transmission electron microscopy (TEM) observations and microhardness measurements, show that while samples of groups (a) and (c) exhibit a stable microstructure, marked differences are detected varying the tempering temperature, especially at 550°C (where a fine precipitation is observed) and for temperatures higher than 850°C (formation of delta ferrite).

1. Introduction

This paper presents the results of small-angle neutron scattering (SANS) measurements carried out on F82H-mod. steel as a complement to other microstructural characterisations. General information on SANS and its applications in physical metallurgy may be found in Refs. [1,2], while Refs. [3,4] report more specific applications to steels of interest for nuclear applications, including fusion reactors. The usefulness of SANS in this domain is well demonstrated by the work carried out on MANET steel, to characterise the growth of He-bubbles [5–10] and to investigate microstructural evolution under tempering [11–13].

In the following sections, the investigated metallurgical treatments are listed and the corresponding SANS results including nuclear and magnetic size distribution are presented. The metallurgical interpretation of these results is discussed making reference to results obtained by transmission electron microscopy (TEM) and microhardness [14].

2. Material characterisation

The investigated samples were obtained from reference F82H-mod. steel (8.0 Cr, 0.10 C, 0.16 Mn, 0.16 V, 2.0 W, 0.02 Ta wt%) submitted to standard metallurgical treatment: 1040°C 37′ + 750°C 1 h. These samples were platelets of approximately 1 cm^2 in surface and 1 mm in thickness. The following heat treatments were investigated:

(a) *hardening* by austenitisation at 950°C, 1050, 1150°C, 30′ and by interrupted cooling from 1040°C 30′ followed by holding at 500°C 5 h, 24 h, 100 h, 500 h, 2000 h, 10000 h;
(b) 1040°C 30′ + *tempering* 2 h at 550°C, 650°C, 800°C, 850°C, 875°C, 900°C;
(c) 1040°C 30′ + *ageing* at 550°C (up to 2000 h) and 700°C (up to 1000 h).

A sample solely submitted to the reference recommended metallurgical treatment, 1040°C 37′ + 750°C 1 h, was also investigated. TEM observations [14] have shown that oriented, platelike shaped precipitates of M_2X phase (X:C or N) are present after austenitising and during holding at 500°C. The same phase is probably responsible for the secondary hardening of F82H-mod. steel when tempered at 550°C [14,15]. $M_{23}C_6$ phase is formed under tempering or ageing in the range from

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0022-3115/98/$19.00
PII: S0022-3115(98)00196-2

600°C to the beginning of reaustenitisation (835°C). These precipitates are predominantly intergranular and grow with increasing tempering time or temperature up to sizes in 1000 Å range.

3. Experimental method

Reference is made to bibliography [1,2] for a general presentation of SANS. Experimental data more specifically pertaining to SANS measurements on martensitic steels have been presented in Refs. [10,13]. The SANS measurements were carried out using the PAXY instrument at Laboratoire Léon-Brillouin (CEA-CNRS, Saclay). Sample-to-detector distances of 2 and 3 m and a wavelength of 6 Å were used. This gave a Q-interval ranging from 0.02 to 0.160 Å^{-1}, which corresponds to particle sizes ranging from 20 to 200 Å approximately; the effect of scattering centres with size outside this interval is estimated by the adopted transformation method, as discussed in Refs. [10,11,16] and shown below. A horizontal magnetic field was applied perpendicular to the incoming neutron beam in order to fully align the magnetic moments in the sample. Thus only nuclear scattering occurs in the horizontal plane, while nuclear and magnetic scattering occur in the vertical one. In fact, in the case of magnetic samples, the total SANS cross-section $\mathrm{d}\Sigma/\mathrm{d}\Omega$ can be written as the sum of two terms:

$$\frac{\mathrm{d}\Sigma}{\mathrm{d}\Omega}(Q) = \left(\frac{\mathrm{d}\Sigma}{\mathrm{d}\Omega}(Q)\right)_{\text{Nucl.}} + \left(\frac{\mathrm{d}\Sigma}{\mathrm{d}\Omega}(Q)\right)_{\text{Magn.}} \sin^2\Phi, \qquad (1)$$

where Φ is the angle on the detector plane. The ratio of the "vertical" to the "horizontal" SANS components:

$$R(Q) = \frac{\left(\frac{\mathrm{d}\Sigma}{\mathrm{d}\Omega}(Q)\right)_{\text{Nucl.}} + \left(\frac{\mathrm{d}\Sigma}{\mathrm{d}\Omega}(Q)\right)_{\text{Magn.}}}{\left(\frac{\mathrm{d}\Sigma}{\mathrm{d}\Omega}(Q)\right)_{\text{Nucl.}}} = 1 + \frac{\left(\Delta\rho_{\text{Magn.}}\right)^2}{\left(\Delta\rho_{\text{Nucl.}}\right)^2} \qquad (2)$$

is related to the nuclear and magnetic scattering inhomogeneities, differences between scattering centres and matrix ($(\Delta\rho_{\text{Nucl.}})^2$ and $(\Delta\rho_{\text{Magn.}})^2$ respectively). The SANS nuclear and magnetic cross-sections can be written as

$$\frac{\mathrm{d}\Sigma}{\mathrm{d}\Omega}(Q) = (\Delta\rho)^2 \int_0^{+\infty} N(R)V^2(R)|F(Q,R)|^2\,\mathrm{d}R, \qquad (3)$$

where $N(R)\mathrm{d}R$ is the number per unit volume of centres with a typical size between R and $R + \mathrm{d}R$, V their volume and $|F(Q,R)|^2$ their form factor. $N(R)$ was determined by transformation of Eq. (3), using the method described in Ref. [16]. 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. The logarithmic representation of $N(R)$ is quite suited for the case of technical alloys, where different kinds of microstructural inhomogeneities with sizes differing in order of magnitude are often simultaneously present. The fitting procedure yields an 80% confidence region on the obtained size distribution. For the present case, these error bands, which are not presented in Section 4, are few percents for the nuclear distributions and 20–30% for the magnetic ones. The differences found between size distribution functions obtained using spherical or plate-like form factors were smaller than such bands.

4. SANS result and discussion

(*a*) *Hardened samples:* No difference in SANS cross-section is detectable varying the austenitisation temperature. Important differences have been detected in grain size [14,15], but such large features cannot be appreciated by the SANS technique. The comparison with the sample submitted to standard treatment shows that 1 h at 750°C relieves fine scale microstructural inhomogeneities which are present immediately after quenching from austenitisation temperature. More specifically the corresponding size distribution functions of the tempered sample show negligible differences between nuclear and magnetic distributions (with a low value of the R ratio, of about 2) while the austenitised sample exhibits a noticeable density in the magnetic distribution (Fig. 1) around 100 Å (and a much higher and Q-dependent value of the R ratio). This is consistent with a dissolution of M_2X precipitates at 750°C. For the samples with interrupted cooling and long hold times identical SANS cross-section are found for all the investigated times for both the nuclear and the magnetic component. This result agrees with the indication of

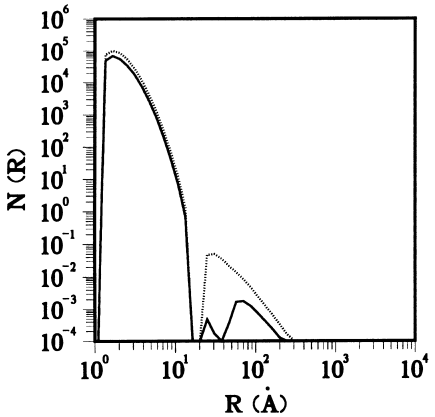

Fig. 1. Magnetic size distribution functions for F82H-mod. austenitised 30′ at 1150°C (dots) and at 1040°C then tempered 1 h at 750°C (line).

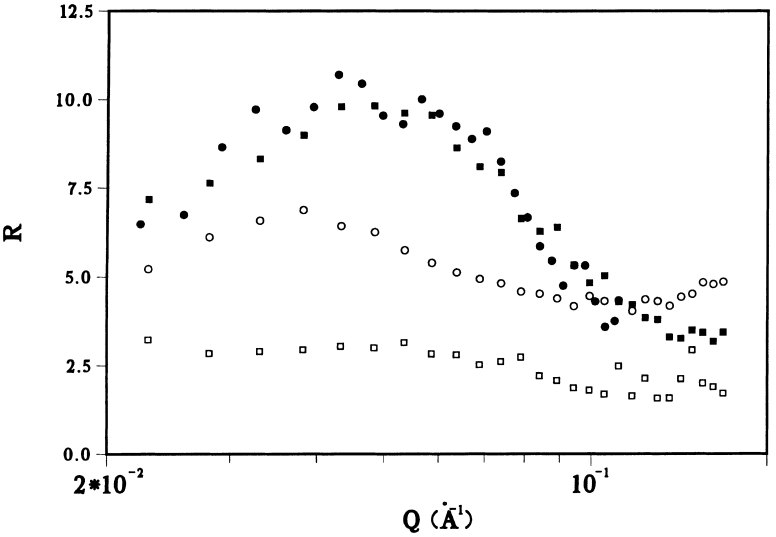

Fig. 2. $R(Q)$ ratio for F82H-mod. tempered 2 h at 550°C (empty circles), 1 h at 750°C (empty squares), 2 h at 875°C (full circles). The $R(Q)$ value for the sample austenitised at 1050°C 30′ is also reported for comparison (full squares).

other techniques that the microstructure produced by this treatment is quite stable at 500°C.

(*b*) *Tempered samples:* There are marked differences among the SANS data obtained for the samples tempered at different temperatures. Fig. 2 reports the $R(Q)$ ratio for the samples tempered at 550°C, 750°C and 850°C. The corresponding size distribution functions are reported in Fig. 3. The sample tempered at 550°C has the highest cross-section at Q-values corresponding to very tiny size (around 10 Å), which may be correlated to the M_2X phase precipitation, as reported in Section 2. Only M_2X precipitates larger than 50 Å could be detected by TEM, but their observation is very difficult

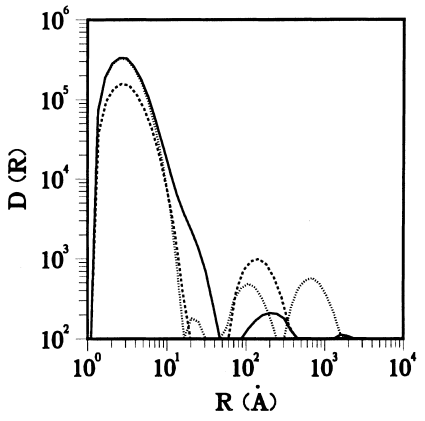

Fig. 3. Nuclear size distribution functions for F82H-mod. steel tempered 2 h at 550°C (line), 1 h at 750°C (dashes) and 2 h at 875°C (dots).

and smaller sizes such as those indicated by SANS measurements cannot be excluded. Completely different SANS curves are found for higher temperatures (650°C, 750°C, 800°C): for high Q's (small sizes) the intensity is much lower than in the sample tempered at 550°C and decreases with increasing tempering temperature. An increase in the high R's tail of the size distributions of Fig. 3 is also observed, especially for tempering at 750°C, which can be correlated with the growth and coalescence of $M_{23}C_6$ precipitates in this same temperature range. For tempering in the range where reaustenitisation and ferrite formation take place (850–900°C) a marked increase in the SANS intensity and in the $R(Q)$ ratio (Fig. 2) is observed. Also this effect can be correlated with the increase in hardness observed for these temperatures. Namely the fact that $R(Q)$ is similar to the ratio found for the hardened samples (see Fig. 2) can be correlated with the fact that after reaustenitisation a transformation into martensite takes place at room temperature.

(*c*) *Aged samples:* For the samples aged at 550°C between 2 h and 100 h or 2000 h there is a marked evolution which can easily be interpreted as a growth of large precipitates, as suggested by the size distribution functions. The occurrence of different kinds of precipitates is also suggested by difference between nuclear and magnetic size distribution functions. Practically no change is detected between 100 h and 2000 h. A comparison with the nuclear size distribution of the sample submitted to interrupted cooling and held 2000 h at 500°C, confirms that the same type of precipitate, namely M_2X, should be present both in the samples aged at 550°C and in those held at 500°C. For the

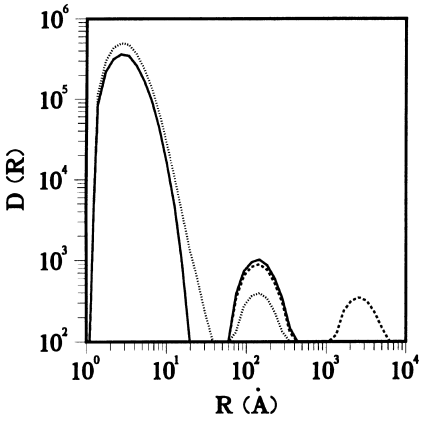

Fig. 4. Nuclear size distribution function for F82H-mod. steel aged at 550°C 2 h (dots) and 2000 h (dashes) and hold 2000 h at 500°C after interrupted cooling (line).

samples aged at 700°C only changes in intensity are detected and corresponding size distribution functions are quite similar to those reported in Fig. 4 for the sample tempered 1 h at 750°C. An increase in $M_{23}C_6$ precipitate volume fraction may explain this result.

5. Conclusions

SANS investigations carried out to study the microstructural effect of various thermal treatments on F82H-mod. steel confirm that the microstructure is quite stable in this steel varying the hardening conditions (austenitisation temperature, holding time at 500°C) and for long ageing times, at 550–700°C. Marked changes are detected varying the tempering temperature within the range 550–900°C, which correlate with the corresponding changes in microhardness. Namely indications of fine precipitation at 550°C are obtained, which correlate with the maximum in the hardness vs T curve arising from M_2X precipitation. A correlation with hardness behaviour is also found in the range 650–850°C (large $M_{23}C_6$ carbides precipitation) and for higher temperatures where reaustenisation and ferrite formation occur. These findings should now be checked comparing the experimentally determined value of $R(Q)$ with the one obtained from the chemical composition of the different precipitated phases. It is also mentioned that the present results are providing a basis to study the microstructural effect of irradiation in F82H-mod. Namely an experiment has recently been carried out at the High Flux Reactor of ILL-Grenoble, to investigate the growth of He-bubbles in samples implanted at 250°C and then annealed at the same temperatures investigated in the present study.

Acknowledgements

This work has been carried out in the frame of the "Long Term Programme" (subtask SM 6.4). We thank Mr. M. Schirra (FZK IMF I) for useful discussions and suggestions. Dr. A. Lapp's (LLB) assistance and collaboration during the SANS measurements is gratefully acknowledged. The support from HCM Program is also acknowledged.

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