Recent applications of small-angle neutron scattering in the characterisation of irradiated steels for nuclear technologies

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SUMMARY

Study of helium bubble growth in α-implanted F82H-mod

Characterization of Eurofer-97 neutron irradiated at 250°C-300°C up to 16.3 dpa

Characterization of un-irradiated, thermally treated, and irradiated 9 Cr Eurofer-ODS
SANS INSTRUMENT D22 AT THE ILL-GRENoble
NUCLEAR AND MAGNETIC SANS

Nuclear and magnetic SANS cross-section

\[ \frac{d \Sigma(Q)}{d \Omega} = (\Delta \rho)^2 \int_0^\infty dR N(R) V^2(R) |F(Q, R)|^2 \]

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

Polarised SANS

\[ A_M \cdot A_N \propto \Delta \rho_m \cdot \Delta \rho_n \]

a) reference sample, b) irradiated sample
He bubbles in F82H-mod. steel implanted with $\alpha$-particles at RT then annealed 2 h at temperatures between 250° C and 975 °C

Coalescence of helium bubbles after annealing at 975 °C
(M. Klimiankou, FZK)
SANS contrast depends on bubble radius $R$

$$\Delta \rho (R) = \rho_{F_{82}H} - b_{He}\rho_{He}(R)$$

$$C_{He} = v_M \int \rho_{He}(R) V(R) N(R) dR$$

The dependence of the contrast on the bubble radius is taken into account in the fitting procedure but given the small value of $\rho_{He}$, very large changes of the He mass density would be necessary to lead to significantly different distributions. Assuming that the He concentration is equal to the nominal value (400 appm), the obtained variations on range typically from $-10\%$ at 2 Å to $+12\%$ at 100 Å. The resulting variations in $N(R)$ are generally of a few per cent, therefore well inside the statistical uncertainty band.
F82H-mod. steel as-implanted at 250°C then tempered at 825°C:
SANS cross-sections of implanted and reference samples
best-fit He bubble volume distributions $D(R)$ ($N(R) \times V(R)$) in Å⁻¹ in compared with the corresponding TEM histogram
Helium bubbles volume distribution in F82H-mod.

250 °C

825 °C

975 °C

The dashed area represents the 80% confidence band.
Best-fit helium bubble volume fraction, $\Delta V$, helium concentration, $C_{He}$, and radii obtained from SANS data. The $R$ and $\Delta V$ values in parentheses are those obtained from TEM.

<table>
<thead>
<tr>
<th>Tempering Temperature</th>
<th>$\Delta V$</th>
<th>$C_{He}$(appm)</th>
<th>$R_1$(Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>250 °C</td>
<td>0.0012</td>
<td>209.0</td>
<td>11.1 (7)</td>
</tr>
<tr>
<td>825 °C</td>
<td>0.0053</td>
<td>375.9</td>
<td>3.8 14.6 (17)</td>
</tr>
<tr>
<td>975 °C</td>
<td>0.0085</td>
<td>558.9</td>
<td>4.1 45.9 (46)</td>
</tr>
</tbody>
</table>
RESULTS ON 2.5 AND 8.4 dpa IRRADIATED EUROFER (ICFRM13 Proceedings)

Nuclear SANS cross-sections of the difference between Eurofer97 neutron irradiated at 250°C at 2.5 dpa and at 300°C at 8.4 dpa and their respective reference samples
Increase the dose the average radius remains nearly unchanged but a consistent increase is observed in the volume fraction of the observed defects, from 0.005 at 2.5 dpa to 0.011 at 8.4 dpa.

Volume distribution functions $D(R)$ (nm$^{-1}$) obtained from the nuclear SANS difference between Eurofer97 neutron irradiated at 300°C and their respective reference samples.
Eurofer-97 irradiated at 250°C 2.5 dpa (squares), at 300°C 8.4 dpa (full circles) and at 250°C 16.3 dpa (empty circles)

Eurofer-97 unirradiated references for irradiated at 250°C 2.5 dpa (squares), at 300°C 8.4 dpa (full circles) and at 250°C 16.3 dpa (empty circles)
Difference between irradiated Eurofer-97 and reference sample for irradiated at 250°C 2.5 dpa, at 300°C 8.4 dpa and at 250°C 16.3 dpa.
Polycrystalline 9Cr Eurofer-ODS with 0.5 wt% Y$_2$O$_3$

TEM observation of small and large Y$_2$O$_3$ particles

SANS characterization of the same material
R. Coppola et al., Physica b 350 (2004) e545
Microstructural stability of ODS alloys
Evolution of ODS particles after annealing of MA powder

MA ODS-Eurofer powder
as-milled

MA ODS-Eurofer powder
annealed 1100 °C 2h
Nuclear SANS cross-sections of 9Cr Eurofer-97 ODS powders reference mechanically alloyed (squares), mechanically alloyed plus annealed at 750°C (circles) and at 1100°C (triangles).
Volume distribution in A. U. \(D(R)\) volume per unit volume of \(Y_2O_3\) particles with a radius between \(R\) and \(R+dR\) for 9Cr Eurofer ODS mechanically alloyed plus annealed at 750°C and 1100°C; the hatched area represents the 80% confidence band.
Eurofer ODS irradiated at 250°C 16.3 (empty circles), as-milled nano-powder (full circles)
refining the determination of the size distributions, for modeling purposes, with special attention to the effect of background subtraction.

SANS measurements on B-doped Eurofer steel with up to 5000 He appm

in-situ high temperature measurements for kinetics studies in Eurofer-ODS

TEM observation of irradiated Eurofer already investigated by SANS
REFERENCES


The bcc lattice cell of MANET steel with an interstitial C atom in octahedral position; the six nearest neighbours can be Fe atoms or a number of Cr ones varying between 1 and 6 for the different thermal treatments.
quench from 1200°

quench from 1075 °C

The C-Cr elementary aggregates, giving rise to the magnetic anisotropy, dissolve for $T > 1180^\circ$C
Nuclear-magnetic interference term for MANET quenched from 1075°C (full dots moduli of the measured negative values, empty dots positive values), quenched from 1200°C (triangles), quenched from 1075°C then tempered 2 h at 700°C (squares).
R(Q) for MANET quenched from 1075°C (dots), quenched from 1200°C (crosses), quenched from 1075°C then tempered 2 h at 700°C (squares)
(a) nuclear SANS cross-section $N^2$ for reference (empty dots) and as-irradiated (full dots) MANET samples; (b) $R(Q)$ ratio for reference (empty dots) and as-irradiated (full dots) MANET samples; (c) nuclear-magnetic interference term for reference (empty dots) and as-irradiated at 250°C 0.8 dpa (full dots) MANET.
IRRADIATED MANET

As-irradiated: increase in N, R(Q) and NM with respect to reference

➔ small magnetic defects (α’ precipitates)

Irradiated and tempered: increase in N, no change in R(Q) and NM with respect to reference

➔ large non-magnetic defects (microvoids, He-bubbles)

Post irradiation tempering seems to promote the growth of large (1-10 nm) non-magnetic defects, such as He-bubbles or microvoids.

This effect has been observed in other irradiated steels (data analysis underway).