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Nature of defect clusters in neutron-irradiated iron-based alloys deduced from small-angle neutron scattering

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Abstract

Small-angle neutron scattering (SANS) was applied to investigate Fe-based model alloys with intentionally varied Cu levels. The aims are to provide size distributions of scatterers and to interpret deviations of the measured ratio of magnetic and nuclear scattering cross-sections from the ratios calculated for pure vacancy clusters and pure Cu clusters. For the case of the low-Cu alloy the SANS results indicate the average scatterer to be an Fe–Cu–vacancy cluster of about 1 nm radius, the composition of which is constricted according to given inequalities. For the case of the Cu-enriched alloy the SANS results are consistent with Cu-rich clusters of about 1.5 nm radius containing 15% vacancies per bcc lattice site.

1. Introduction

The nature of the nanometre-sized features responsible for the neutron-irradiation-induced degradation of the mechanical properties of reactor pressure vessel (RPV) steels is still subject to debate. This is because, firstly, there is a variety of nanoscale features of different types [1] and the factors influencing their relative importance are complex, and, secondly, the conclusions drawn from experimental investigations using different methods are partly in conflict with one another [2,3]. It is therefore helpful to study model alloys, where the diversity of nanoscale features is constricted due to composition and where dominant effects can be identified or even isolated.

The present approach is based on Fe-based model alloys with low-carbon and low nickel contents, similar levels of the solution hardeners Si and Mn as in Russian-type VVER RPV steels and intentionally varied Cu levels. Preliminary interpretation of the ratio of magnetic and nuclear SANS cross-sections (*A*-ratio) indicated the dominance of vacancy-rich clusters and Cu-rich clusters in the case of the low-Cu and Cu-enriched model alloys, respectively [4]. However, a more detailed consideration of the measured cross-sections revealed significant deviations between measured *A*-ratio and the value calculated for pure vacancy clusters or pure bcc Cu clusters.

The aim of the present paper is to interpret these deviations in detail taking into account the composition of the model alloys as well as the magnetic character of the Fe atoms in the clusters [5,6]. Furthermore, the results of complementary studies of the same model alloys [7] as well as similar materials [8] using positron annihilation spectroscopy (PAS) are exploited in the discussion.

2. Experiments

2.1. Materials and specimens

The materials investigated are low-carbon Fe-based model alloys with low (alloy A) or intentionally increased (alloy B) Cu such as to cover the range of impurity Cu of

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Table 1
Composition of the model alloys (in wt%, balance Fe)

Material	C	Si	Mn	S	P	Ni	Cu
A	0.01	0.15	0.39	0.04	0.002	0.01	0.015
B	0.01	0.24	0.49	0.05	0.012	0.01	0.42

Table 2
Irradiation conditions applied to alloys A and B

Irradiation condition	Irradiation temperature/°C	Neutron fluence ($E > 0.5$ MeV)/ 10^{18} cm^{-2}	Neutron flux ($E > 0.5$ MeV)/ 10^{12} cm^{-2} s^{-1}
A1, B1	270	10 (≈0.01 dpa)	0.4
A2, B2	270	80 (≈0.08 dpa)	3.0

RPV steels of first and later generation RPVs. The composition of the model alloys is given in Table 1. The solute elements Mn and Si are similar as in VVER-type RPV steels, whereas Ni is essentially absent.

Charpy specimens of alloys A and B were irradiated with neutrons at surveillance positions of the VVER440 reactors ROVNO1 and KOLA3 over one reactor cycle. The irradiation conditions denoted 1 and 2, respectively, are specified in Table 2.

From the undeformed part of broken Charpy specimens several slices of about 1 mm thickness were machined for the purpose of SANS and hardness measurements in the unirradiated, as-irradiated or irradiated and annealed condition. Post-irradiation annealing was performed at 475 °C/100 h, i.e. about the condition of large-scale anneals performed for VVER440 RPVs.

2.2. Measurements

The SANS experiment was carried out at the BENSC V4 spectrometer at Hahn-Meitner-Institut (HMI) Berlin. The SANS instrument, V4, and the data processing software, BerSANS-PC, were described in detail in [9] and [10], respectively. Neutron wavelength was 0.6 nm. A two-dimensional position-sensitive detector was placed at distances of 1.1 m and 4 m from the specimen in order to cover a range of scattering vectors, Q, from about 0.1 to 3 nm^{-1}. Background correction and absolute calibration using a water standard were performed. A saturation magnetic field of 1.2 T oriented perpendicular to the neutron beam direction was applied to the specimens. This allows for the separation of the nuclear and magnetic contributions from the total scattered intensity. Part of the results was tentatively reported immediately after the SANS experiment [4]. Since then, analysis has been significantly refined and results from positron annihilation Doppler broadening and lifetime measurements for the same materials have become available [7].

A number of 10 Vickers hardness tests with load, $F = 98.1$ N, were performed for each material condition in order to derive estimates of the mean value and standard deviation of Vickers hardness, HV10.

3. Experimental results

The nuclear and magnetic scattering cross-sections measured for the model alloys A and B are summarized in Figs. 1 and 2.

For model alloy A an irradiation-induced increase of the scattering cross-section is observed at higher values of the scattering vector, Q. The Q value of first deviation from the unirradiated reference is equal for the magnetic cross-sections of conditions A1 and A2, $Q > 0.5$ nm^{-1}, but slightly differs for the nuclear cross-section. The excess scattering for the irradiated conditions is an increasing function of neutron fluence. For condition A1 this contribution completely disappears on average after annealing at 475 °C for 10 h. However, the relative scatter of the data obtained at high Q values/low intensities is large and no further data processing was performed for the annealed condition of alloy A1.

For model alloy B there is an irradiation-induced increase of the scattering cross-section starting at a slightly smaller Q value, $Q > 0.3$ nm^{-1}, than for alloy A. An increase of the neutron fluence from 0.01 dpa to 0.08 dpa does not further raise the scattering cross-section. In contrast with alloy A1 the irradiation-induced scattering cross-section of alloy B1 does not disappear after annealing but the Q range is shifted to lower values.

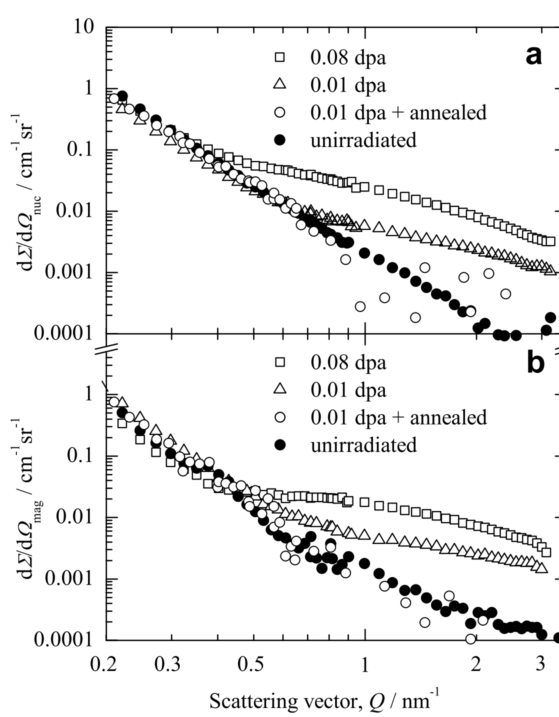

Fig. 1. Coherent nuclear (a) and magnetic (b) scattering cross-sections of iron alloy A for the unirradiated condition, two irradiated conditions distinguished by the respective neutron fluences in units of dpa and a post-irradiation annealed condition.