Experimental report

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Proposal: 1-04-196 **Council:** 4/2020

Title: SANS experiments to study irradiation embrittlement in reactor pressure vessel steels

Research area: Materials

This proposal is a new proposal

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Samples: reactor pressure vessel steel

Instrument	Requested days	Allocated days	From	To
D11	2	2	28/09/2021	30/09/2021

Abstract:

This work aims to understand the formation of solute clusters and precipitate evolution in reactor pressure vessel steel as a function of irradiation fluence and temperature. We propose to investigate the evolution of precipitates using small angle neutron scattering (SANS) on a series of irradiated aged samples. The information obtained from SANS, would be used to understand the embrittlement mechanism in reactor pressure vessel steels and to study the correlation existing between the NDE signals (electric, micro-magnetic and ultrasonic) and material damage (embrittlement) using a range of characterisation methods on materials exhibiting different radiation damage levels. The tool developed as a part of this project will play a critical role in evaluating the residual lifetime of in-service Nuclear Power Plant (NPP) components.

SANS experiments to study irradiation embrittlement in reactor pressure vessel steels

1. Introduction

An extensive irradiation program including post-irradiation testing and non-destructive radiation damage evaluation was undertaken within the EU H2020 project – NOMAD aimed at developing and validating a non-destructive evaluation (NDE) tool for the local and volumetric characterisation of the embrittlement in operational reactor pressure vessels (RPVs) for improving safety of Generation II and III reactors. This require deeper understanding of irradiation induced RPV material degradation through comprehensive microstructural examination and correlation with mechanical properties including tensile and charpy impact properties. Both eastern and western grade RPV steel grade samples were irradiated at SCK-CEN, Belgium. The irradiation has resulted in significant changes in the mechanical properties of the materials. SANS experiment was done in D11 instrument at ILL to characterise the nano-precipitates evolved during the irradiation.

2. Experimental

A total of 9 samples made of steel grades A508Cl3, 22NiMoCr37, JRQ and 15Kh2NMFA have been extracted from various locations of RPV steel and one set is chosen as reference sample as shown in Table 1 (baseline). Another set irradiated under the test programme CHIVAS with activity ranging from 1200 to 3500 uSv/hr at temperatures ranging from 150-310°C. The irradiation fluence and temperature conditions of the samples are shown in Table 2.

Table 1: Reference samples for the proposed experiment

Material	Specimen ID	Thickness (mm)	Condition
A508	A5	0.5	Baseline (unirradiated)
	B54_0.5	0.5	Baseline (unirradiated)
	B54_0.3	0.3	Baseline (unirradiated)
	C1_0.5	0.5	Baseline (unirradiated)
	C1_0.3	0.3	Baseline (unirradiated)
	D21	0.5	Baseline (unirradiated)
	E6	0.3	Baseline (unirradiated)
22NiMoCr37	R865	0.5	Baseline (unirradiated)
JRQ	R165	0.5	Baseline (unirradiated)
A508-B	R176	0.5	Baseline (unirradiated)

Table 2: Irradiation activity and dose rate of samples for the proposed experiment

Sample ID	Activity (Bq)	Dose rate (µSv/hr)
B13	3.49E+07	1500
E4	4.85E+07	3500
A1	3.59E+07	1400
C9	3.59E+07	1300
B1	3.49E+07	3200
B5	3.49E+07	unknown
B15	3.49E+07	unknown
B7	3.49E+07	1700
D2	3.59E+07	unknown

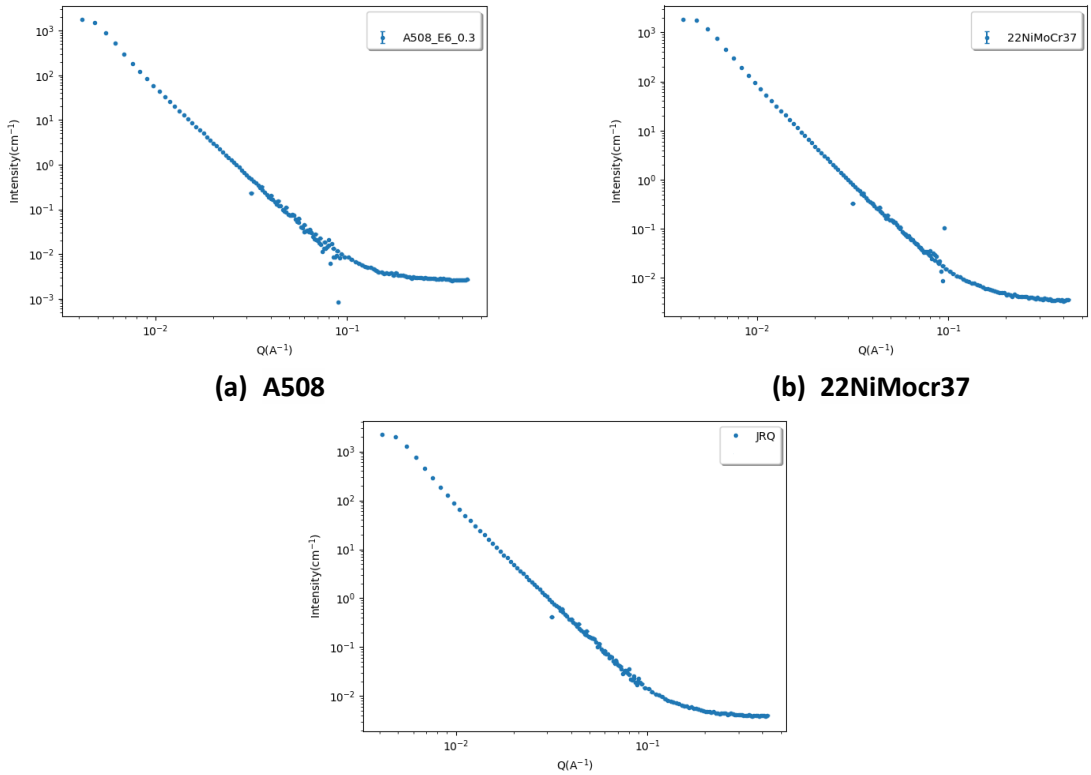
The experiment was conducted on the D11 instrument. Two different sample to detector distances (1.7m and 13.5m) were used in order to explore a wide Q range (0.004Å^{-1} to 0.42 Å^{-1}). Each sample was be mounted in the chamber applying a magnetic field of 1T in the horizontal direction parallel to the sample surface.

All the references samples were measured at two detector distances, with and without a magnetic field. The irradiated samples could not be measured since the container of the active specimen was found to be radioactive and the experiment team and Instrument scientists decided not to use these active samples for SANS measurement because of safety concerns.

The reduced data was analysed for model independent relations using Kratky and Guinier model. A shape-dependent approach was also be used to fit the curves to the data in order to deconvolute the size and volume fraction by using SASView software program.

3. Results

The successful measurement of reference samples showed no scattering as expected. The Intensity was unchanged over the measured Q range of 0.004Å^{-1} to 0.42 Å^{-1} as shown in Figure 1.



(a) A508 **(b) 22NiMocr37**

(c) JRQ

Figure 1. Scattered intensity (I) vs Q of reference samples of different grade RPV steels (a) A508 (Western grade) (b) 22NiMocr37 (Western grade) (c) JRQ (Japanese grade)

The absence of any scattering in the broad Q range measured during the experiment confirms that there are no nano-precipitates present in the reference material in the range of 1nm to 150nm diameter. The inability to measure the irradiated active sample has been a setback. Even though the measured reference sample data is used to confirm absence of any nano-precipitates in the material. Similar grade of samples were also investigated using NDE methods based on electric, micro-magnetic and ultrasonic techniques. A critical comparison with the NDE data can be used to build consensus among the different NDE methods. The experiment data obtained will be used to compare with other characterisation methods planned for these set of samples.