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Reactor Pressure Vessel Task of Light Water Reactor Sustainability Program: Milestone M3LW-13OR0402012, Report on Small-Angle Neutron Scattering Experiments of Irradiated RPV Materials

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Light Water Reactor Sustainability

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1. INTRODUCTION

As stated in previous progress reports, the available embrittlement predictive models, e.g. [1], and our present understanding of radiation damage are not fully quantitative, and do not treat all potentially significant variables and issues, particularly considering extension of operation to 80y. A significant issue in this regard is that the RPV is generally considered to be an irreplaceable component. The major issues regarding irradiation effects are discussed in [2, 3] and have also been discussed in previous progress and milestone reports. As noted previously, of the many significant issues discussed, the issue considered to have the most impact on the current regulatory process is that associated with effects of neutron irradiation on RPV steels at high fluence, for long irradiation times, and as affected by neutron flux. It is clear that embrittlement of RPV steels is a critical issue that may limit LWR plant life extension. The primary objective of the LWRSP RPV task is to develop robust predictions of transition temperature shifts (TTS) at high fluence (ϕt) to at least 10^{20} n/cm^2 (>1 MeV) pertinent to plant operation of some pressurized water reactors (PWR) for 80 full power years. New and existing databases will be combined to support developing physically based models of TTS for high fluence-low flux ($\phi < 10^{11}$ n/cm^2-s) conditions, beyond the existing surveillance database, to neutron fluences of at least 1×10^{20} n/cm^2 (>1 MeV). A previous milestone report [4] described in detail the irradiation experiment underway at the Advanced Test Reactor (ATR) at Idaho National Laboratory (INL), designated ATR-2.

This report provides the status of the Reactor Pressure Vessel Task of Light Water Reactor Sustainability Program: Milestone M3LW-13OR0402012, "Report on Small-Angle Neutron Scattering Experiments of Irradiated RPV Materials." Background information is also discussed regarding current experimental activities involving the use of small-angle neutron scattering to characterize irradiation-induced microstructural features of various RPV materials, including some irradiated in high flux test reactors and some irradiated in commercial reactor surveillance programs.

2. BACKGROUND ON SMALL-ANGLE NEUTRON SCATTERING FOR LWRSP

2.1 BRIEF DESCRIPTION OF SMALL-ANGLE NEUTRON SCATTERING

Small angle neutron scattering (SANS) is a frequently used method for investigating fine-scale microstructural defects [5] and has been used in the study of neutron irradiation embrittlement of RPV steels [6-10]. The irradiation-induced precipitates have been identified as the dominant embrittling feature in most US RPV steels. SANS is an effective technique for studying the evolution of irradiation-induced damage and, in particular, precipitates in RPV steels both because of their small size, which typically evolve at sizes below the resolution limits of other common characterization techniques, such as conventional electron microscopy, but also because the precipitates scatter neutrons through nuclear and magnetic contrast mechanisms.

The SANS intensity in terms of the cross section $d\Sigma/d\Omega$ of precipitates in ferromagnetic steels is composed of nuclear ($d\Sigma_{nuc}/d\Omega$) and magnetic ($d\Sigma_{mag}/d\Omega$) scattering contributions [11]

$$\frac{d\Sigma}{d\Omega}(Q) = N_p V_p \left[(\Delta\eta_{\text{nuc}})^2 F_{\text{nuc}}^2 + \sin^2\alpha \cdot \left(\Delta\eta_{\text{mag}}\right)^2 F_{\text{mag}}^2\right] \quad (1)$$

where Q is the scattering vector, $4\pi\sin(\theta)/\lambda$, with θ the Bragg scattering angle, and λ is the neutron wavelength, $d\Sigma/d\Omega(Q)$ is the scattering cross section in absolute units, N_p is the number density of precipitates, V_p is the precipitate volume, $(\Delta\eta_{nuc})^2$, $(\Delta\eta_{mag})^2$ are the nuclear and magnetic scattering densities, F^2_{nuc}, F^2_{mag}, are the nuclear and magnetic scattering structure factors. The $\sin^2\alpha$ factor arises from the scattering interaction between the neutron and the atomic magnetization of the scattering materials

$$sin^2\alpha = 1 - (\check{\kappa} \cdot \check{\varepsilon})^2 \quad (2)$$

where κ is the unit magnetization vector, ε is the unit diffraction vector and α is the angle between the two unit vectors.

The SANS analysis of precipitates in RPV steels is based on two major assumptions. First, the precipitate is assumed to be a uniform sphere of radius R. Second, the precipitate is assumed to have no magnetic scattering so magnetic scattering arises from magnetization of the iron matrix alone. The third assumption is that the precipitate size distribution is a logarithmic normal distribution.

2.2 THE SANS FACILITY AT THE HIGH-FLUX ISOTOPE REACTOR

A series of upgrades have been undertaken at the High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory, including the installation of a supercritical hydrogen moderator ($T \approx 20$ K, hereinafter referred to as the cold source), which has boosted the flux of long-wavelength neutrons by over two orders of magnitude. Four cold-neutron guides (CG1–4) were installed on the cold source, allowing for the development of new neutron scattering instrumentation, including two SANS instruments. The current suite of instruments at HFIR is described by Selby & Smith [12], and is shown schematically in Fig. 1.

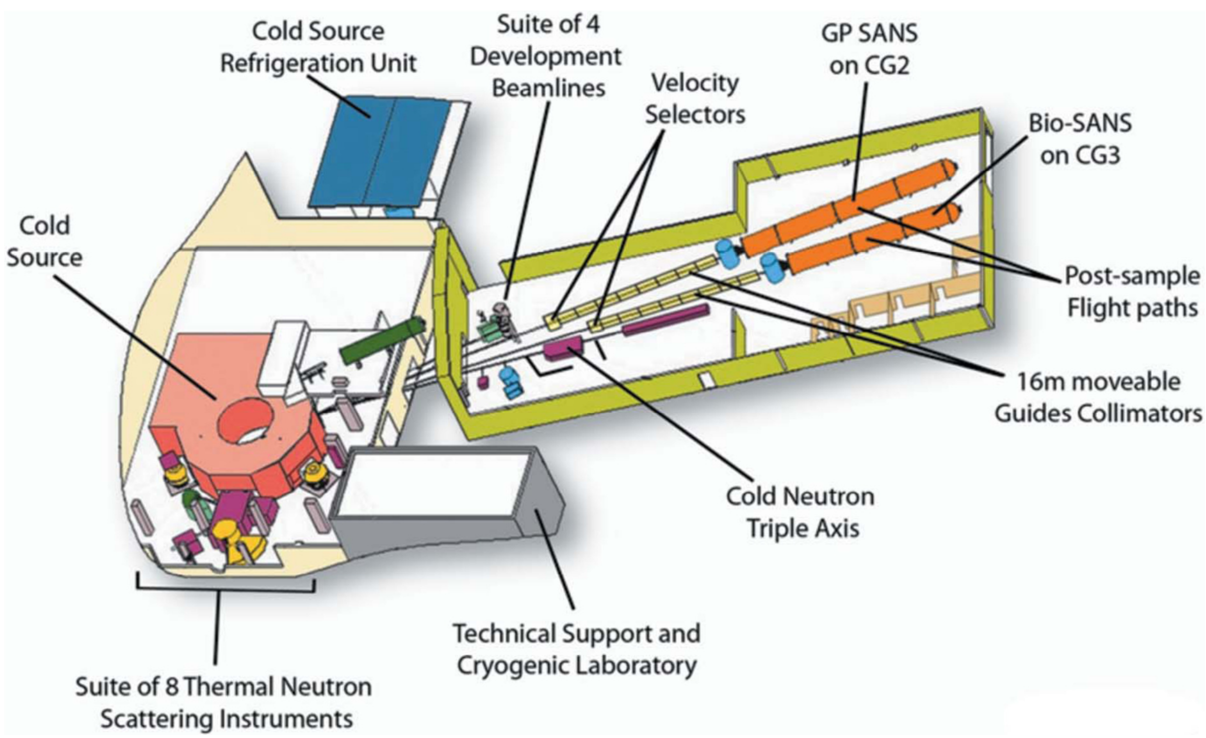


Figure 1. Instrument layout in the HB4 guidehall.

In order to take advantage of the new capabilities, a 40 m-long SANS instrument has been constructed, which utilizes a mechanical velocity selector, pinhole collimation and a high-count-rate ($>10^5$ Hz) large-area (1 m^2) two-dimensional position-sensitive detector. The incident wavelength (λ), resolution (Δλ/λ), incident collimation and sample-to-detector distance are independently variable under computer control. The detector can be moved up to 45 cm off-axis to increase the overall Q range [$0.001 < Q = (4\pi/\lambda)\sin\theta < 1$ Å^{-1}, where 2θ is the angle of scatter]. The details of design and characteristics of this instrument are described in [13].

The instrument-control and data-acquisition systems have borrowed much of the technology already in use on the other instruments at HFIR. User interaction with the instrument is through the spectrometer and instrument control environment (*SpICE*) software, developed at ORNL [14]. *SpICE* is built on *LabVIEW* and provides a simple command-driven interface for performing all necessary functions of the instrument. *SpICE* can also run user-written macros, and scripting capabilities are provided through the Python programming language. The user interacts through a graphical user interface, GUI, developed specifically for the SANS instruments at HFIR, providing an easy-to-use interface with integrated real-time feedback. The raw data are saved in XML files containing pertinent instrument-configuration information, and a template style sheet is available for viewing this information in a tabular format using a web browser.

Data reduction is implemented in *IgorPro* (Wavemetrics Inc., USA). The absolute cross section, [$d\Sigma/d\Omega(Q)$], is defined [15] as the number of neutrons scattered per second (neutrons s^{-1}) into unit solid angle divided by the incident neutron flux (neutrons cm^{-2} s^{-1}) and thus has the dimensions of area (cm^2). On normalizing to unit sample volume, $d\Sigma/d\Omega(Q)$ has units of cm^{-1}. From this definition, the relationship between the cross section and the measured count rate in a detector element of area Δa and counting efficiency γ situated normal to the scattered beam at a distance r from the sample is given by

$$\frac{d\Sigma}{d\Omega}(Q) = \frac{I(Q)r^2}{\gamma I_o \Delta a A t T} \quad (3)$$

where I_o is the intensity (counts s^{-1} cm^{-2}) on a sample of area A and thickness t and hence irradiated volume At. The measured transmission, T, accounts for the attenuation of the beam on passing through the sample. For SANS, it is assumed that the attenuation factor is the same for all scattered neutrons and this approximation is reasonable for $\theta < 10^o$. Similarly, equation (3) assumes that the solid angle subtended by a detector element is independent of 2θ, and this approximation again holds for small angles where cos(2θ) is close to unity. For higher-Q measurements (e.g. $Q > 0.5$ Å^{-1}), geometric corrections are applied after subtracting the 'blocked-beam' (i.e. the count rate in the absence of a neutron beam) and parasitic backgrounds (i.e. the count rate with a beam but no sample), and normalizing all data sets to the same total number of beam monitor counts (to correct for differences in counting time and variations in the beam intensity during the measurements). The counts in each pixel are then divided by $\cos^3(2\theta)$, to correct for the small variation in solid angle subtended by a pixel because of the planar geometry of the detector [16]. Similarly, small corrections are applied to the sample transmission to account for the differences in path length through the material with scattering angle [17]. The net scattering from the sample is further corrected for nonuniformities in the detector efficiency by dividing, pixel by pixel, by the measured scattering from an isotropic scatterer such as water or a hydrogen-rich polymer such as polystyrene or polymethyl methacrylate [18]. The reduced data can be converted into absolute units (cm^{-1}) through the use of a suite of absolute calibration samples developed for the facility [19], or by use of attenuated direct-beam measurements performed without a sample.

The latest improvement to this SANS instrument was completed in October 2012 by commissioning a powerful magnet to perform neutron scattering in a saturated magnetic field. This upgrade allows studying ultra-fine particles (like irradiation-induced precipitates) in ferromagnetic materials (e.g., RPV steels). The HV-7V Vertical Electromagnet by Walker LDJ Scientific was pursued by joint efforts of the LWRSP Materials Pathway manager Dr. Jeremy Busby and purchased by University of Tennessee Governor's Chair Professor Brian Wirth. Actual adaptation of this magnet to the SANS instrument configuration was performed by a team led by the instrument leader Dr. Ken

Littrell. Figure 2 shows the magnet set-up in the SANS instrument configuration. The magnet is placed in front of the neutron detector.

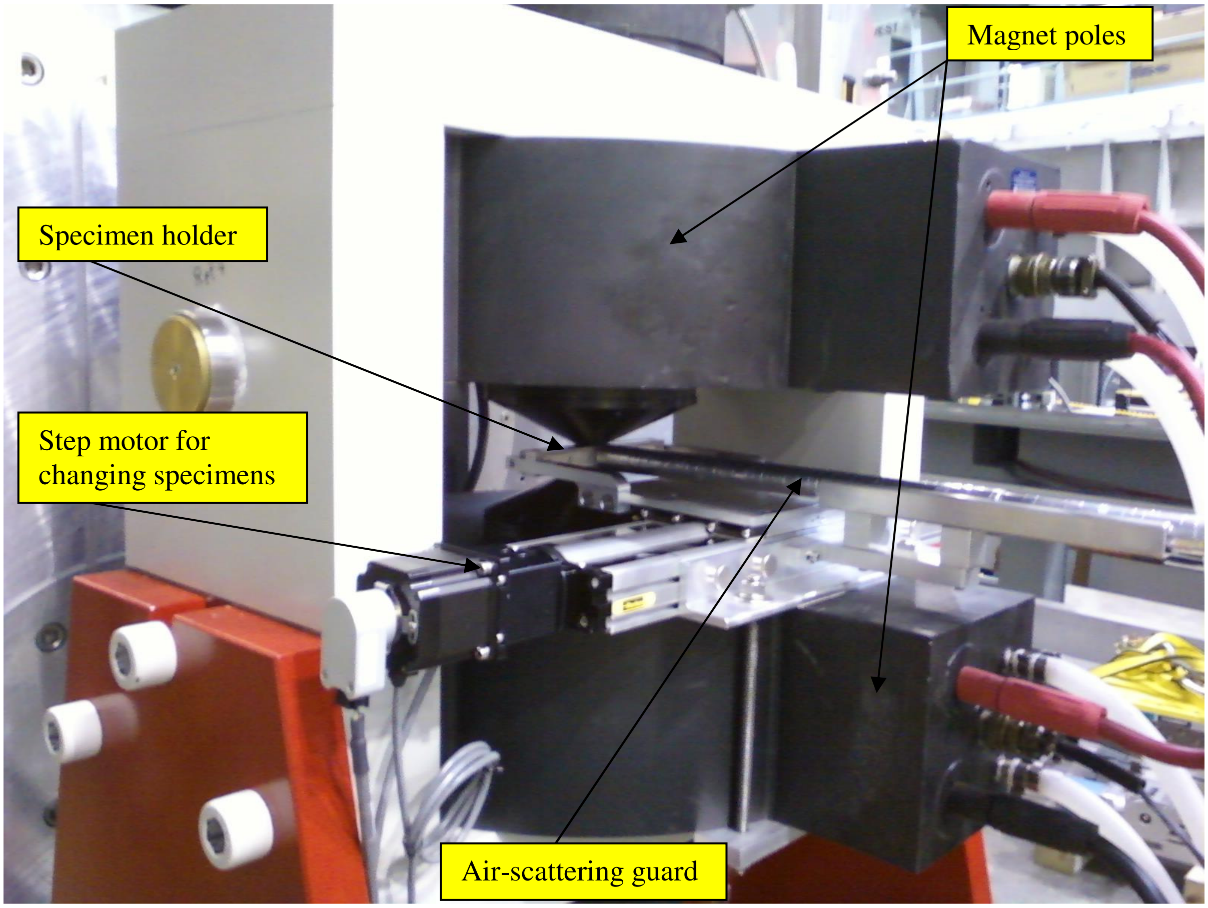


Figure 2. Photograph showing the magnet with holder for RPV samples on the SANS instrument.

In order to handle previously irradiated specimens, special holder was designed, see Fig. 3. The irradiated RVP steel specimens were cut from previously tested Charpy specimens. Thus each specimen is 10 mm square with thickness less than 1 mm. The thickness, t, of each specimen was measured individually prior to SANS measurements and used as per equation 3. The RPV specimens were inserted inside an aluminum space holder, see Fig. 3. Then, the holder with RPV specimens was placed inside a large holder between quartz glasses to prevent potential contamination of beam hall area. The neutron beam is not affected by the aluminum holder and quartz glasses.

Figure 3. Specimen holder with irradiated RPV steel specimens.

2.3. MATERIALS TO BE STUDIED

In [4], materials for five different projects were described and discussed; (1) Materials for the ATR-2 experiment, (2) Materials from the Zion reactor, (3) Materials from the Ringhals reactors, (4) Materials from the R. E. Ginna reactor, and (5) Materials from the Palisades reactor.

The LWRS Program, in cooperation with EPRI, the NRC, and Westinghouse, is engaged in discussions with Zion Solutions, Inc., owner of the Zion Nuclear Plant, Units 1 and 2. These two reactors have been shut down since 1998, having operated for only about 15 effective full power years. A number of specific recommendations have been made by the RPV task of the LWRS Program relative to information provided by Zion Solutions, Inc. These recommendations are to obtain specific identified tested and untested surveillance specimens from both Zion 1 and Zion 2 for examination, and to obtain a large section of Zion 2 for direct examination of the RPV.

A detailed description of the materials from the Ringhals Reactors was presented in [20]; all the surveillance specimens are from low-copper high-nickel weld metals in Ringhals Units 3 and 4, both pressurized water reactors. These specimens have been used to prepare samples for atom probe tomography (APT) and SANS to characterize the microstructure relative to irradiation-induced precipitates and other defects, and some early results are discussed in [21]. Materials from the R. E. Ginna reactor and the Palisades Reactor were described in [22]. The intent of the RPV task is to examine all of those materials with APT and SANS with a view towards enabling comparisons of irradiation-induced microstructures from high flux test reactor experiments and lower flux commercial reactor surveillance programs.

In addition to the materials mentioned above, the LWRS Program, in cooperation with the U.S. Nuclear Regulatory Commission and the SCK-CEN in Belgium, is performing both APT and SANS experiments with five RPV materials irradiated in the BR-2 reactor at relatively high neutron flux and to relatively high neutron fluences. The materials are a Palisades Reactor Weld (PW), a beltline weld from the Midland Reactor (MBW), a plate from the Heavy-Section Steel Technology (HSST) Program, HSST Plate 02 (HSST-02), a weld from the Heavy-Section Steel Irradiation (HSSI) Program (73W), and a plate of A533 grade B class 1 steel designated JRQ and used as a reference material by the International Atomic Energy Agency (IAEA) (JRQ). The chemical compositions of the five RPV steels are given in Table 1.

Table 1. Chemical composition of five RPV materials irradiated in BR-2 FRISCO-R.

Material	C	Mn	Si	S	P	Cr	V	Cu	Mo	Ni	W	Al
PW	0.11	1.25	0.18	0.017	0.014	0.04	0.003	0.20	0.55	1.2	-	-
MBW	0.09	1.607	0.622	0.009	0.017	0.12	0.005	0.256	0.41	0.574	<0.01	0.015
HSST-02	0.23	1.55	0.20	0.014	0.009	0.04	0.003	0.14	0.53	0.67	<0.01	0.019
73W	0.10	1.56	0.45	0.005	0.005	0.25	0.003	0.31	0.58	0.60	<0.01	0.005
JRQ	0.18	1.42	0.24	0.004	0.017	0.12	0.002	0.14	0.51	0.84	<0.01	0.014

The irradiation campaign, denominated FRISCO-R (Fusion and Reactor Materials Irradiation SCK•CEN/ORNL – RPV steels), was performed during the last three cycles of the Belgian Reactor 2 (BR2) in the period July/December 2005 [23]. All specimens, with the exception mentioned below, have been irradiated in the in-pile section 3 (IPS-3) of BR2 during cycles 04/2005 and 05/2005, at an equivalent fission flux of approximately 2×10^{13} n/(cm^2·s), E > 1 MeV. This part of the experiment is the lower flux irradiation, while samples from the Palisades Weld were also irradiated in the in-pile section 2 (IPS-2) of BR2 during cycle 03/2005, at an equivalent fission flux of approximately 5 × 1013 n/(cm^2·s), E > 1 MeV, the higher flux irradiation. The lower flux irradiation has been conducted between Oct 12 and Dec 20, 2005 at a water temperature between 295 and 300 °C in the K311 channel (IPS-3) of the CALLISTO rig in the BR2 reactor. In order to achieve uniform irradiation conditions (fluence and flux) in the radial direction, the rig has been rotated by 180° between the first and the second cycle. The higher flux irradiation has been conducted between July 29 and Aug 26, 2005 at the same water temperature (295-300 °C) in the D180 channel (IPS-2) of the CALLISTO rig. For both irradiations, the parameters relative to the coolant have been chosen in conformity with the technical specification of PWR primary water chemistry:

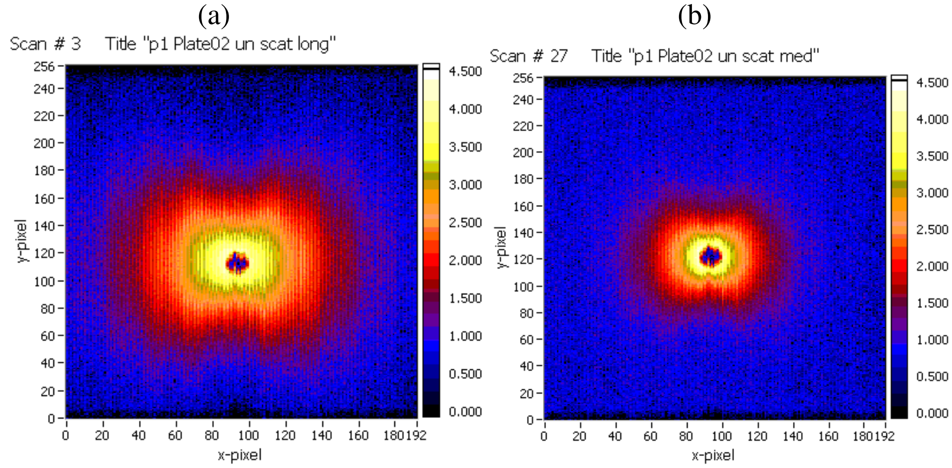
- Temperature 295-300 °C
- Boron (boric acid) ± 550 ppm
- Lithium (lithium hydroxide) 1.8 ppm ≤ [Li] ≤ 2.2 ppm
- pH 7.00 ≤ pH25°C ≤ 7.08 or 7.26 ≤ pH300°C ≤ 7.34
- Dissolved hydrogen 25 ccSTP/kg ≤ [H2] ≤ 35 ccSTP/kg

The specimens were in direct contact with the water.

3. PRELIMINARY RESULTS FOR MATERIALS IRRADIATED IN BR-2

Prior to performance of scattering measurements and analyses on the actual materials, a series of tests to determine good magnet function were performed. Figures 4 (a)-(c) show examples of the data measured for HSST Plate 02 at extremes of the q-range. The butterfly patterns evident in the long and medium settings are proof of the magnet's function. The straight shadows top and bottom on the short configuration data are the shadows of the magnet coil assembly—the straightness shows how well-centered the sample is between the coils. To minimize risk of contamination, as stated earlier, the samples are measured in preloaded sample cassettes with quartz windows.

(a) (b)



(c)

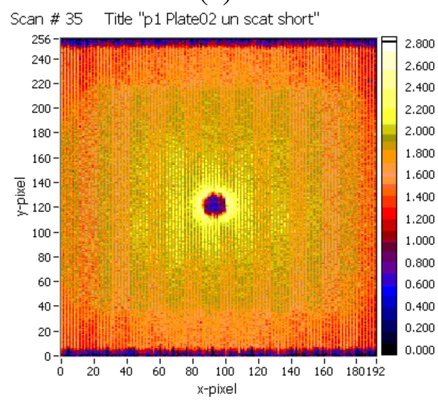


Figure 4. Images from SANS measurements at the extremes of the q-range using HSST Plate 02, showing (a) long setting, (b) medium setting, and (c) short setting.

Verification of the magnet performance now allows for complete analyses of the SANS experiments on the materials described above.

4. SUMMARY AND CONCLUSIONS

This report provides descriptions of the SANS technique and ORNL SANS instrument to study irradiation-induced microstructural changes in RPV steels. The latest improvement to this SANS instrument was completed in October 2012 by commissioning a powerful magnet to perform neutron scattering in a saturated magnetic field. This upgrade allows studying ultra-fine particles (like irradiation-induced precipitates) in ferromagnetic materials (e.g., RPV steels). The report provides descriptions of the materials that are selected for SANS characterization within the Light-Water Reactor Sustainability Program. The main reasons for selection of these materials are for RPV surveillance application and for representative materials that were irradiated to high fluences. The SANS measurements will be compared with atom tomography results to provide better understanding of microstructural processes in RPV steels under high fluence irradiation that are representative of extended life conditions. Experimental verification of the magnet performance now allows for complete analyses of the SANS experiments on the selected materials.

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