

# Experimental report

13/02/2018

**Proposal:** 1-01-152

**Council:** 4/2016

**Title:** Precipitate formation under proton irradiation in the next generation of reactor pressure vessel steels

**Research area:** Materials

**This proposal is a new proposal**

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**Samples:** steel samples (x34)

Instrument	Requested days	Allocated days	From	To
D11	3	0		
D33	2	2	14/11/2016	16/11/2016
D22	2	0		

**Abstract:**

We propose a small-angle neutron study of the precipitation character of proton-irradiated low-Cu steel alloys under varying composition and thermal aging conditions. These low-Cu alloys are of crucial importance for the next generation of reactor pressure vessel steels.

# Precipitate formation in third generation reactor pressure vessel steels

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## Abstract

Small-angle neutron scattering was employed to investigate reactor pressure vessel steels with varying composition with the main focus given to the levels of Cu. The study of RPV steels containing different levels of Cu is important to establish the nature of the precipitates that may be formed in low-Cu alloys and what bearing these will have on macroscopic properties e.g. embrittlement. Our preliminary results from heat-treated samples indicate that non-trivial microstructural features are present within the samples prior to any irradiation. The features are suspected to be carbides and grain boundaries discerned as  $q^{-4}$  behaviour from the scattering profiles.

## Introduction

Reactor Pressure Vessels (RPVs) are thick-walled steel vessels that provide the primary containment of the reactor core in a nuclear power plant. Fast neutrons generated by the reactor irradiate the RPV over its lifetime, resulting in embrittlement that is usually attributed to the formation of defects and clusters within the lattice of the material [1]. Copper is one of the major factors contributing to the embrittlement of RPVs, as observed in steels with Cu in excess of 0.1 wt.%. In low-Cu RPV steels (wt.% < 0.1) Ni, Si and Mn phases are thought to be among the main precipitation features.

Numerous experimental techniques are capable of examining irradiation damage in such materials, however to date there is no single technique able to give a complete description of the generated defects [2, 3]. By using small-angle neutron scattering (SANS) one may probe the *distribution* of irradiation-induced precipitates via the difference in neutron contrast compared to the matrix and hence in scattered SANS intensity from the precipitates [1, 4].

## Experimental Details

### Materials

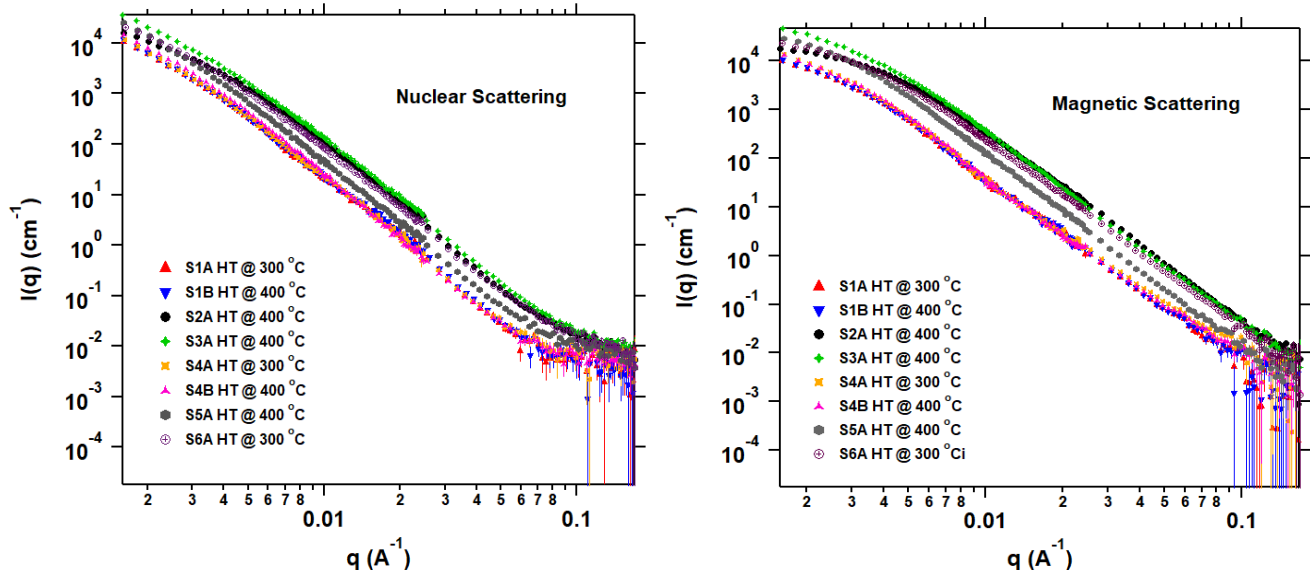
The materials investigated were high- and low-Cu (copper compositions ranging from 0.05 to 0.3 wt.%) RPV steel alloys. Steels of six different compositions were used and for clarity they were labelled S1 to S6 respectively. The samples were heat treated at 300°C (condition A) and 400°C (condition B) for 2.5 h.

### SANS Setup & Analysis

SANS measurements were performed on D33 with sample-to-detector distances of 3 to 12 m, and corresponding collimation, selected to measure scattering vectors  $q$  ranging between 0.0016 and 0.2 Å<sup>-1</sup>. Neutron wavelengths of 6 Å and 12 Å were used. Correction measurements for electronic noise and background scattering were performed. A saturating magnetic field  $\mu_0 H = 1$  T was applied perpendicular to the neutron beam to facilitate separation of nuclear and magnetic contribution to the scattering. Square Cd apertures were positioned in front of each sample defining the sample area measured. Raw-data treatment and analysis was performed using the software GRASP and the NCNR analysis package. Systematic checks for multiple scattering were also performed for all measurements.

## Preliminary Results

The resulting nuclear and magnetic differential scattering cross sections for selected samples are plotted as a function of  $q$  in Fig. 1. The analysis of the samples is still ongoing but first observations denote that the scattering intensity of all samples follow a  $q^{-4}$  dependence (Porod law) that is typical of grain boundaries. Preliminary fitting processes using a log-normal sphere model distribution also uncover the presence of scattering features in the 10 to 100 nm range. A detailed analysis and comparison with the literature will make possible the identification of these features; initial conclusions are that they are likely to be carbides, that typically form during the austenisation of steel alloys.



**Figure 1** (left) Nuclear and (right) magnetic differential scattering cross sections as a function of scattering vector  $q$  for high- and low-Cu RPV steel alloys heat treated at 300°C (condition A) and 400°C (condition B) for 2.5 h.

## References

1. R. Boothby, J. Hyde, H. Swan, D. Parfitt, K. Wilford and P. Lindner, SANS examination of irradiated RPV steel welds during in-situ annealing, *Journal of Nuclear Materials* 461, 45–50, (2015).
2. J. Jiang, Y. Wu, X. Liu, R. Wang, Y. Nagai, K. Inoue, Y. Shimizu and T. Toyama, Microstructural evolution of RPV steels under proton and ion irradiation, *Journal of Nuclear Materials* 458, 326–334 (2015).
3. R. Ngayam-Happy, C. Becquart, C. Domain and L. Malerba, Formation and evolution of MnNi clusters in neutron irradiated dilute Fe, *Journal of Nuclear Materials* 426, 198-207, (2012).
4. A.Wagner, F. Bergner, A. Ulbricht and C. Dewhurst, Small-angle neutron scattering of low-Cu RPV steels neutron-irradiated at 255°C and post-irradiation annealed at 290°C, *Journal of Nuclear Materials* 441, 487–492 (2013).