Experimental Report

Proposal: **5-32-791** **Council:** 10/2012

Title: Small angle scattering for the analysis of magnetic surface defects in metallic nanoparticles

This proposal is continuation of: 5-42-309

Researh Area: Physics

Main proposer: **PAUL Donald Mckenzie**

Experimental Team: PAUL Donald Mckenzie
COOK Robert

Local Contact: DEWHURST Charles

Samples: Ni
Oleyl amine
Trioctyl phosphine

Instrument	Req. Days	All. Days	From	To
D33	5	3	21/05/2013	24/05/2013

Abstract:

We propose continuation to our previous SANS experiment using polarisation analysis to investigate the magnetic structure of several Ni nano particle systems. This will be done in order to establish standards against which magnetometry techniques may be calibrated and to investigate the magnetic dead layer associated with nano particles. Nickel nano particle samples were synthesised via an organically stabilised method at the Johnson-Matthey Sonning Common site. In order to obtain accurate magnetic volumes we will require the use of the polarised 3He spin filter on the d22 instrument, a spin flipper and incident polarised neutrons. Analysis of the magnetic structure will allow for verification of magnetometry based particle sizing and hence the expansion of the technique into industrial applications.

SANS for the analysis of magnetic surface defects in metallic nanoparticles

Previous SANS experiments have suggested that magnetite nanoparticles (NPs) comprise a ferromagnetic core surrounded by a shell of canted moments, resulting in a reduced magnetic volume of the particle[1]. We have previously attempted to measure the difference in magnetic and nuclear scattering form factors (experiment 6-52-309) but the high concentration of spheres introduced a structural factor. In order to avoid this the NPs were imbedded into a commercially available fluorinated grease (Fomblin) to reduce the inter-particle scattering.

The supported systems were investigated using the D33 instrument employing spin polarization and analysis via the use of an RF flipper and polarised ^{3}He gas chamber. The Ni samples were prepared as a colloidal system, employing a surfactant stabiliser, and suspended in hexane before being dispersed via evaporation into the Fomblin.

Data from all 4 spin channels (++, +-, --, +-) were collected in the region 2 to 50 K and at an applied current of 45 A (~ 0.9 T) with λ = 6.0 Å in all cases. NP magnetic systems of a low loading behave like atomic paramagnets with an increased moment and susceptibility. Within this temperature range 0.9 T would be sufficient to saturate the NP moment.

In order to maximise the Q range of the ^{3}He analysis the cryostat - cell distance was placed at the minimum obtainable distance, ≈ 1 m, and Teflon was used as a high scattering material in order to map the detection limits. Figure 1 (left) is the resultant scattering - with a marked decrease in scattering at Q ≈ 0.085 A^{-1}.

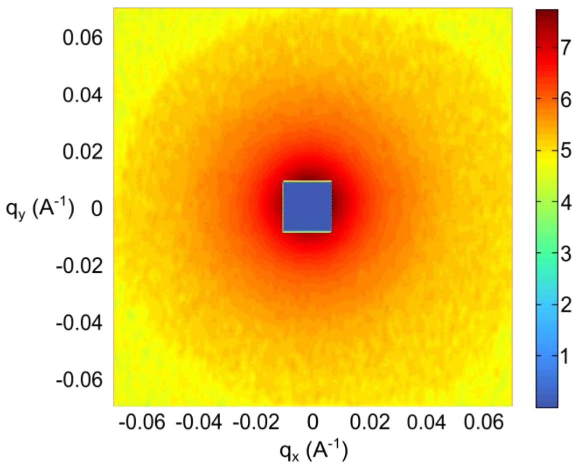

Figure 1 - 2D nuclear scattering for Teflon. Note the sharp boundary for the Teflon scattering caused by the edges of the ^{3}He cell.

The NPs being used have been found to be spherical when viewed with TEM and the spherical form factor reaches its first minima at Q = 4.49/r, where r is the sphere radius. Hence the form factor can only be resolved currently for particles with R > 52 A. The sample reported here has r ≈ 125 A according to TEM.

The 2D scattering for the fomblin embedded NPs at 3 K and ≈0.9 T is shown in Figure 2 (left - nuclear, right - magnetic). Note that the non-spin flip scattering is ≈ 2 orders of magnitude greater than the spin flip scattering.

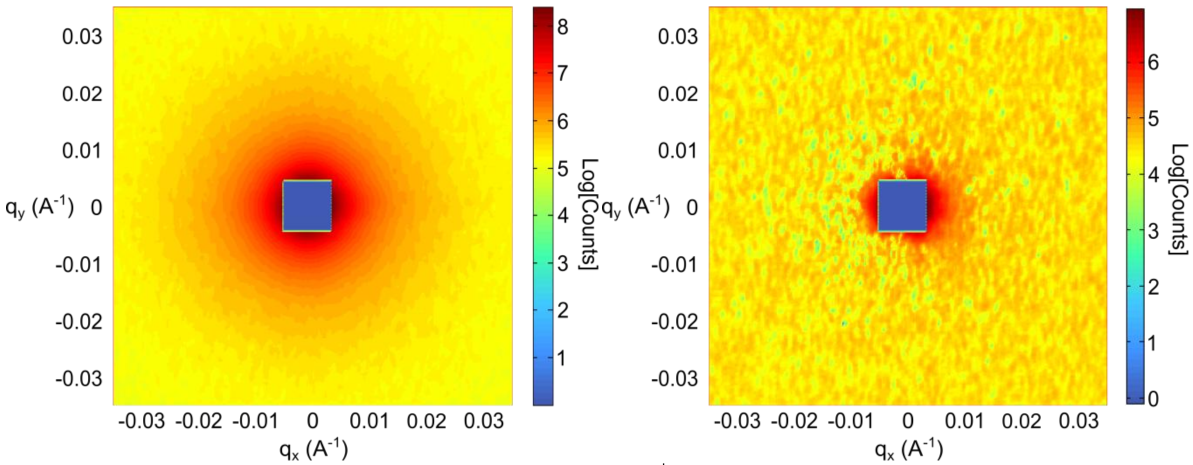

Figure 2. Nuclear (I^{--}, left) and magnetic (I^{-+}, right) scattering for the fomblin mounted NP materials corrected for leakage from the other spin channels.

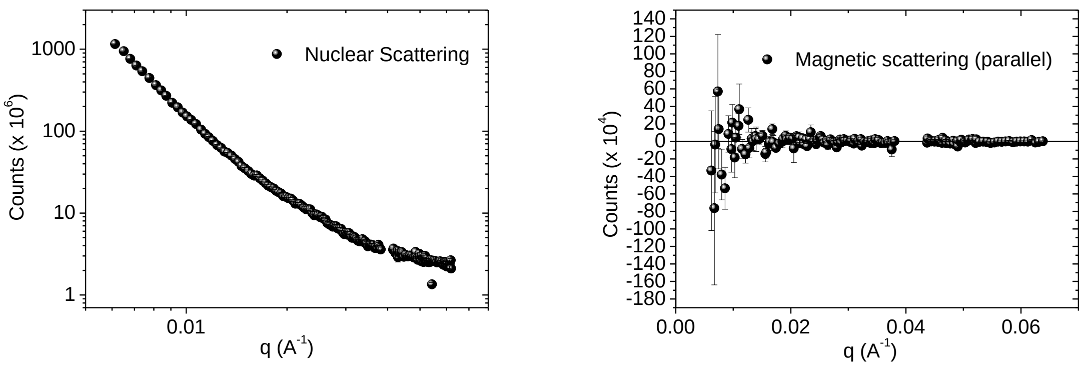

Figure 3. Nuclear (left) and magnetic (right) scattering from fomblin mounted Ni NPs.

Using the same spin selection rules as in experiment 6-52-309[2] the nuclear and magnetic (parallel to field) scattering are extracted from the 2D data sets (figure 3). No magnetic component is seen in this region parallel to the applied field and the nuclear scattering is inconsistent with a spherical form factor (r ≈ 12.5 nm). The nuclear scattering is likely purely from the fomblin. It would appear that Fomblin grease provides too large a background signal to be of use at the low loadings required to avoid a structural factor.

[1] Krycka, K. L. et al., Physica B, 404 (2009) 2561.
[2] Krycka, K. L., et al., Phys. Rev. Lett. 104 (2010) 207203.