

Experimental Report

16/04/2014

Proposal:	5-32-790	Council:	10/2012	
Title:	Small angle scattering for the analysis of magnetic surface states with variation in material anisotropy			
This proposal is a new proposal				
Research Area:	Physics			
Main proposer:	PAUL Donald Mckenzie			
Experimental Team:	PAUL Donald Mckenzie COOK Robert			
Local Contact:	DEWHURST Charles			
Samples:	Alumina Co Ni			
Instrument	Req. Days	All. Days	From	To
D33	7	4	04/07/2013	08/07/2013
Abstract: We propose a SANS experiment using polarisation analysis to investigate the effect of magnetic anisotropy on the surface structure of several nano-ferromagnetic systems. This will be done in order to correct current magnetic analysis techniques for determining particle volumes in order to make them viable within an industrial setting. Nickel and Cobalt nano-crystallites on an alumina support were prepared at the Johnson-Matthey, Sonning Common site. In order to confirm the surfaces magnetic configuration we will require the use of the polarised ³ He spin filter on the d22 instrument, a spin flipper and incident polarised neutrons. Analysis of any material or particle size dependence on the surface configuration will allow for a more general application of magnetic analysis techniques within the chemical industries.				

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 particle¹. Such a structure will be dependent on the surface chemistry of the NP - herein we measure several alumina supported Ni NPs to contrast with previous work carried out on colloidal NPs (Experiment 6-52-309).

The alumina/ Ni systems were investigated using the D33 instrument employing spin polarization via the use of an RF flipper, with data collected in pairs of spin channels (+/-). Sample environment was controlled using an orange cryostat (2 to 300 K) and an electromagnet ($I_{Max} = 45$ A, $H_{Max} \approx 0.9$ T) with $\lambda = 6.0$ Å.

The scattering of the beam with and without the sample was observed while varying sample thickness until $1/e$ (≈ 0.36) transmission was achieved (see figure 1). Note that the beam without sample (black) shows a slight hump at $q \approx 10^{-5} \text{ Å}^{-1}$. This feature is likely a result of the cryostat and hence analysis of the sample scattering should ignore $q < 2 \times 10^{-2} \text{ Å}^{-1}$.

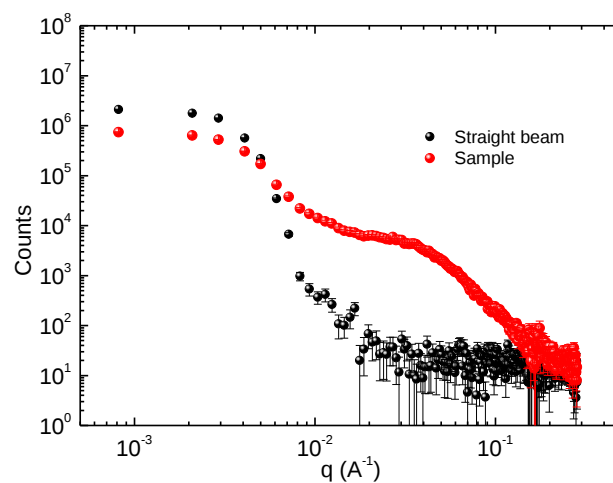


Figure 1 - 1D projection of scattering with (red) and without (black) the supported Ni sample. Transmission ≈ 0.36 .

The resultant 2D detector images are shown in figure 2. The nuclear scattering (left) shows a marked q dependence in the region $0.02 < q < 0.1 \text{ Å}^{-1}$ with no dependence on rotation, θ . The magnetic scattering shows a strong anisotropic behaviour consistent with a system near saturation.

The presence of alumina complicates determining the morphology of the Ni deposits - with either a spherical or hemi-spherical structure being suggested. As a result the analysis will first focus on comparison of the Guinier region.

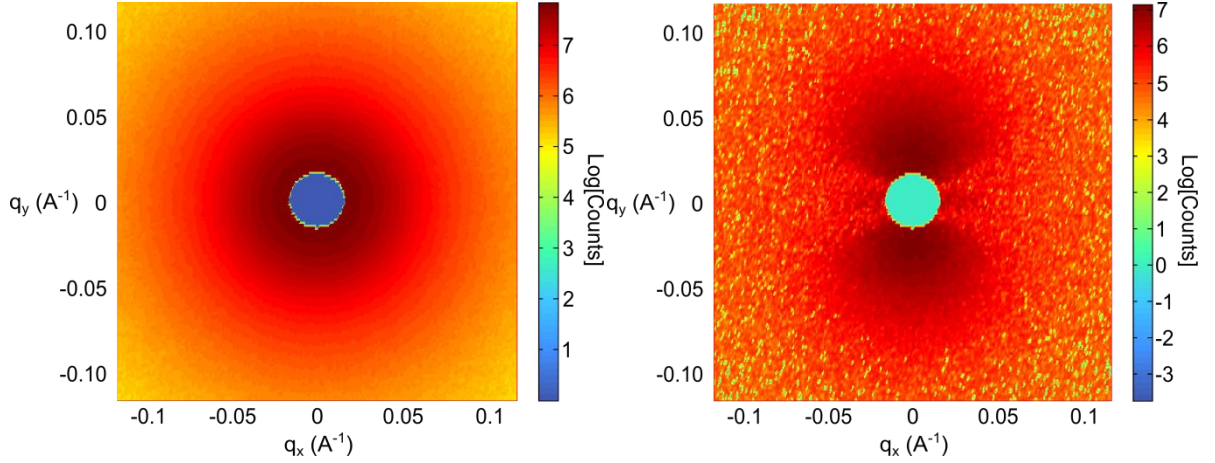


Figure 2 - 2D detector images for alumina supported Ni crystallites for the nuclear ($I^+ + I^-$, left) and magnetic ($I^+ - I^-$, right) channels taken at $H \approx 0.9$ T, $T = 5$ K.

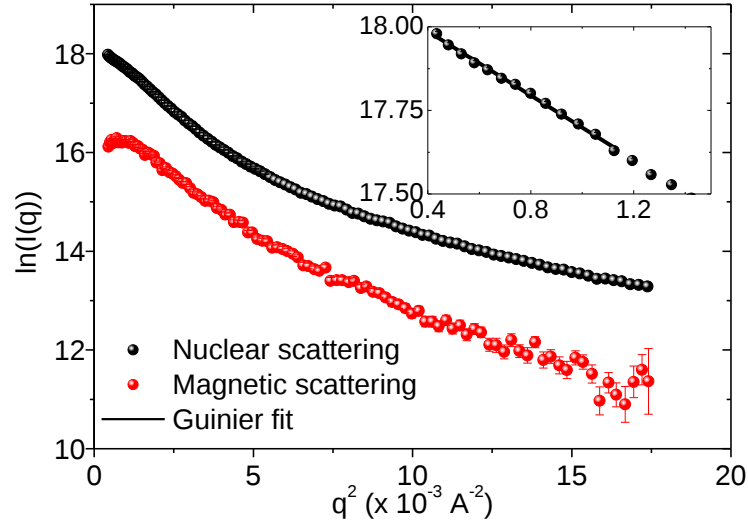


Figure 3. Low q Guinier plots for the nuclear (black) and magnetic (red) scattering. The nuclear term is consistent with a radius of gyration, $R_g = 38.0(3)$ Å, and $I_0 = 7.85(5)$. The magnetic scattering is non-linear at low q in the guinier plot - suggesting a strong structure factor component.

The nuclear scattering is consistent with an average particle size $r_{sphere} = 49.0(5)$ Å or $r_{hemisphere} = 45.3(3)$ Å, assuming spherical and hemispherical morphology respectively. Comparisons to TEM and XRD will serve to better understand the morphology. The magnetic scattering suggests the presence of a more complex magnetic structure that requires further investigation.

¹ Krycka, K. L. et al., Physica B, 404 (2009) 2561.

² Krycka, K. L., et al., Phys. Rev. Lett. 104 (2010) 207203.