Experimental report

20/03/2024

Proposal: 5-32-941 **Council:** 10/2022

Title: Spin Disorder vs. Spin Canting in Iron Oxide Nanoparticles

Research area: Chemistry

This proposal is a new proposal

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Samples: Fe2O3 (on silicon substrate)
Fe2O3 (in toluene-d8 dispersion)

Instrument	Requested days	Allocated days	From	To
D33	5	3	02/06/2023	05/06/2023

Abstract:

We propose to investigate the interparticle interaction effects on the magnetic surface structure of iron oxide nanoparticles with magnetic SANS. Using a set of samples with systematically varied interparticle interactions we are interested in analyzing the dependence of surface disorder on interparticle coupling. Full polarization analysis allows to distinguish between disorder and correlated spin canting and will facilitate quantitative analysis of the nanoparticle spin structure. This work will be the basis for assessing whether dipolar interactions in nanoparticle assemblies are a determining driving force to create correlated spin canting effects.

Experimental report: Spin Disorder vs. Spin Canting in Iron Oxide Nanoparticles

The aim of this experiment was to investigate the interparticle interaction effects on the magnetic surface structure of iron oxide nanoparticles using full polarization analysis SANS (POLARIS).

Three different samples of nanoparticles with increasing interparticle interactions were studied. Nanoparticles embedded within a deuterated polystyrene matrix were created with the purpose of having stationary, non-interacting nanoparticles. The remaining two samples of self-assembled nanoparticles on silicon wafers (mesocrystals) represent samples with decreased interparticle distance and thus increased interactions. POLARIS was measured at 2.8/2.9 and 0.01 T with the field direction perpendicular to the neutron beam. Using the detector distance of 5.1 m and the corresponding collimations of 5.3 m at the D33 afforded a scattering range between $6 \cdot 10^{-3}$ and $7.7 \cdot 10^{-2}$ Å^{-1}.

In the process of the data reduction, it was announced that there was an error with the programmed detector distance. The data reduction was repeated using an offset of the detector distance in GRASP Variant of 12 cm and re-evaluated again.

Initial SANS measurements of the nanoparticles immobilized in deuterated polystyrene still indicate a certain degree of interparticle interactions, with a small correlation peak at low q (below $q = 3.2 \cdot 10^{-2}$ Å^{-1}). Nonetheless, the corresponding interaction distance is weaker than for the mesocrystals, which exhibit in radial averages the expected correlation reflection of the mesocrystalline superstructure at $3.6 \cdot 10^{-2}$ Å^{-1} and at $4.4 \cdot 10^{-2}$ Å^{-1}.

The obtained I^{++} and I^{--} (NSF) data was evaluated by performing azimuthal averaging around the position of the mesocrystal reflection and exhibited maxima/minima every 90°C (parallel to the field), corresponding to the expected a $\sin^4(\alpha)$ oscillations of a longitudinal magnetization component. The I^{+-} and I^{-+} (SF) data showed a similar $\sin^2(\alpha)\cos^2(\alpha)$ behavior, however with no apparent $1+\cos^4(\alpha)$ transversal magnetization contribution in the case of mesocrystals at 2.8 T (Fig. 1).

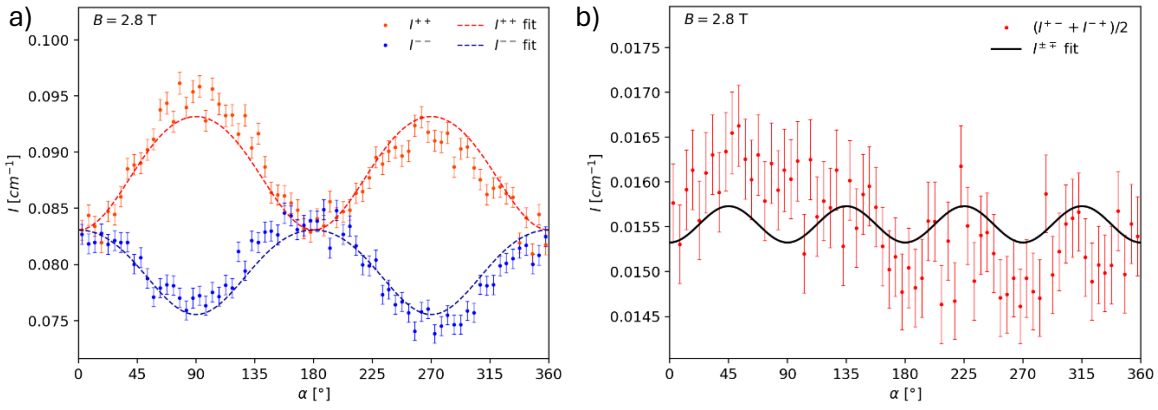

Fig. 1: Azimuthal intensity integrations of NSF (a) and SF (b) spin channel data around the position of the mesocrystal reflection (Q = 0.035-0.050 Å^{-1}). The nuclear scattering amplitude F_N^2 and the Fourier transforms of the longitudinal ($|\tilde{M}_Z|^2$) as well as transversal ($|\tilde{M}_\perp|^2$) magnetizations were determined from combined fits of the non-spin-flip (I^{++} and I^{--}) and spin-flip (I^{+-} and I^{-+}) scattering data.

Further analysis will reveal the existence of a transversal magnetization component suggesting ordered spin-canting at the nanoparticle surface. In addition, the field- and nanoparticle interaction-dependent trends in the magnetically ordered particle volume will be evaluated and compared to macroscopic magnetization measurements.