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A polarised SUSANS facility to study magnetic systems

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Abstract. Using a right-angled magnetic air prism, we have achieved a separation of ∼10 arcsec between ∼2 arcsec wide up- and down-spin peaks of 5.4 Å neutrons. The polarised neutron option has thus been introduced into the SUSANS instrument. Strongly spin-dependent SUSANS spectra have been observed over $\pm 1.3 \times 10^{-4}$ Å^{-1} range for several magnetic alloy samples. Spatial pair-distribution functions for the up- and down-spins as well as the nuclear and magnetic scattering length density distributions in the micrometer domain, have been deduced from these spectra.

Keywords. Small-angle neutron scattering; polarised neutrons; magnetic agglomerates.

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Ultra-small angle neutron scattering (USANS) measurements require a neutron beam with an extremely sharp angular profile. Bonse and Hart [1] proposed multiple Bragg reflections from a channel-cut single crystal to obtain a nearly rectangular angular profile for the beam. The Bonse–Hart proposal was first realised in its totality with triple–triple Darwin reflections from optimally designed [2] monochromator and analyser crystals to achieve the sharpest angular profile [3] for a neutron beam. This has paved the way for super ultra-small angle neutron scattering (SUSANS) studies. Investigations of samples with micrometer-sized magnetic agglomerates require SUSANS measurements with polarised neutrons. We present here the first polarised SUSANS experiment.

The experiment was carried out at the V12b double crystal diffractometer set up in the neutron guide hall of the 10 MW reactor at the Hahn-Meitner-Institut in Berlin, Germany. Neutrons of 5.4 Å wavelength were subjected to 7 Ewald reflections each at the monochromator and analyser channel-cut silicon single crystals. In each crystal, a silicon prism was inserted after the third reflection [4] to deflect neutrons by about 4 arcsec. Due to this shift between the triple and subsequent four-fold reflection patterns within each channel-cut crystal, the width of the rocking curve for the unpolarised beam, obtained by rotating the analyser crystal, reduced to nearly 2 arcsec (figure 1). Between the monochromator and analyser, the horizontal neutron beam traversed a vertical magnetic field of 3.7 kOe in a 2 cm high air gap between 2 cm × 20 cm rectangular poles of a C-shaped permanent magnet, at a small angle to the diagonal of the rectangle. Neutrons were thus deflected by the 'magnetic air prism' [5] of 90° apex angle. The magnet was fabricated by attaching eight rare earth permanent magnet slabs (2 cm wide, 5 cm long and 1.25 cm high, maximum energy product $BH_{\max} > 30$ MGOe) each, just above the upper pole piece and just below the lower pole piece, within a magnet-grade soft iron 'C'. The neutron angle of incidence to the magnet was optimised to achieve a separation of about 10 arcsec between the up- and down-spin neutron peaks [6] and a count rate of about 10 neutrons/s at each peak position (figure 1) for a 10 mm wide and 20 mm high beam. A sample could be inserted in a holder, in a 1.1 kOe vertical magnetic field produced by a pair of ferrite magnets, placed between the magnetic prism and analyser. The SUSANS instrument was thus equipped with the polarised neutron option.

The neutron polarisation attained here is ideal, since unlike other polarisers, a magnetic prism separates the two polarisations with 100% efficiency and the spin-flip probability for either state during its passage to the sample through *air* in the guide field is insignificantly small. Hence the polarisation P and flipping efficiency ε can both [7] be safely equated to unity. We further have an advantage of recording SUSANS spectra for both the spin states side by side in a single rocking curve. This enables a direct comparison between the up- and down-spin spectra with no need for separate normalisations or for a spin-flip operation.

We illustrate the capability of the instrument with polarised SUSANS spectra of an as-cast $Fe_{73}Al_5Ga_2P_8C_5B_4Si_3$ ribbon sample (figure 2). The rocking curve recorded without a sample, representing the instrument resolution, is also shown for comparison. The sample has broadened the up-spin peak (left) considerably, but has an insignificant effect on the down-spin peak.

This measurement needs to be combined with complementary techniques in order to characterise the distributions over shapes, sizes and orientations of nuclear

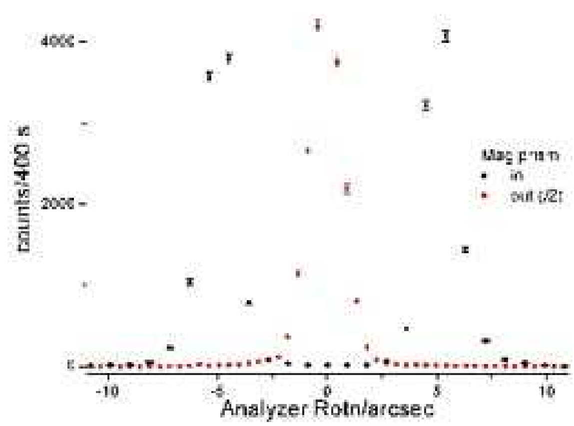

Figure 1. Up–down spin splitting with a magnetic prism.

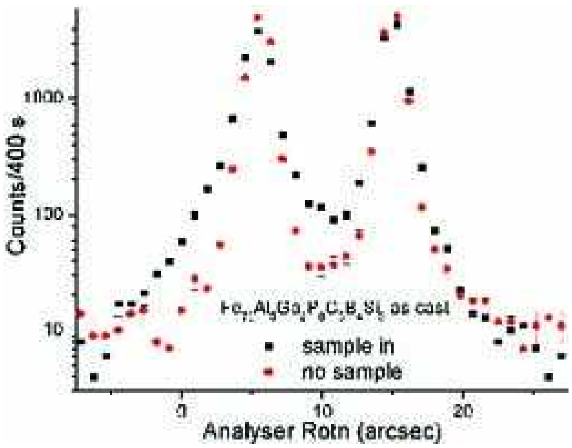

Figure 2. Polarised SUSANS for a magnetic sample.

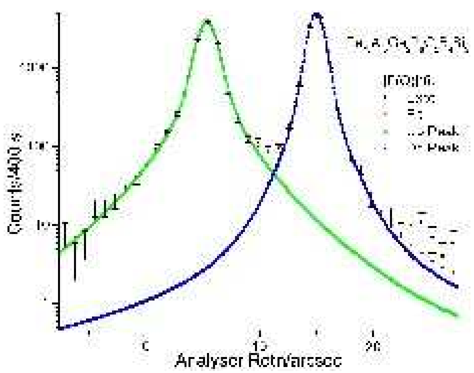

Figure 3. Fitted up- and down-spin SUSANS spectra.

and magnetic scattering structures in the sample. Proper Fourier transforms of scattering length distributions taking all these variations into account should then be used to analyse the polarised SUSANS spectra. However, a flavour of the information obtainable can be provided even by simplistically assuming identical spherically symmetric nuclear and magnetic structures. The up- and down-spin SUSANS spectra corrected for the instrument resolution yield squares of the respective Fourier transforms, whose spherically symmetric inverse Fourier transforms help visualize the averaged spatial distributions, as outlined below.

We begin with parameterised spherically symmetric scattering length distributions $\rho_{\mathrm{u}}(\mathbf{r})$ and $\rho_{\mathrm{d}}(\mathbf{r})$ of identical, spherically symmetric 'particles' for up- and down-spin neutrons respectively. Each Fourier transform $F(\mathbf{Q})$ equals the volume integral [8] of the respective $\rho(\mathbf{r})\exp(-i\mathbf{Q}\cdot\mathbf{r})$, $\mathbf{Q}$ denoting the wave vector transfer. The sum of convolutions of $|F_{\mathrm{u}}(\mathbf{Q})|^2$ and $|F_{\mathrm{d}}(\mathbf{Q})|^2$ with the respective fits to the instrument resolution, peaked at parameterised Q-centres, is least-square fitted (figure 3) to the sample SUSANS spectrum to extract $\rho_{\mathrm{u}}(\mathbf{r})$ and $\rho_{\mathrm{d}}(\mathbf{r})$. The

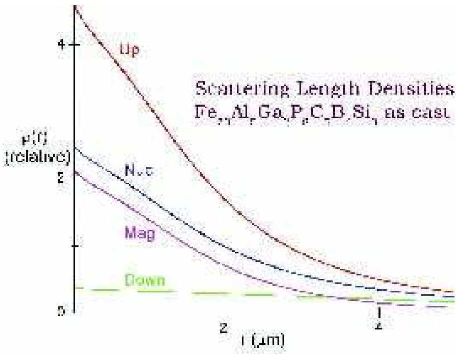

Figure 4. Deduce scattering length densities.

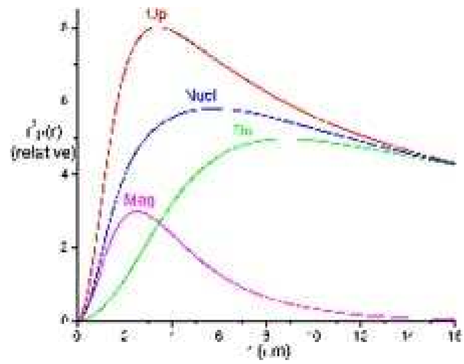

Figure 5. Scattering length shell densities.

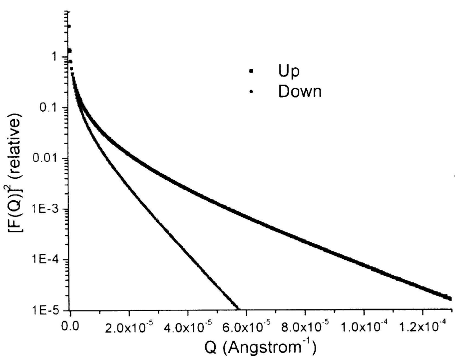

Figure 6. $|F_{\mathrm{u}}(\mathbf{Q})|^2$, $|F_{\mathrm{d}}(\mathbf{Q})|^2$ corrected for instrument resolution.

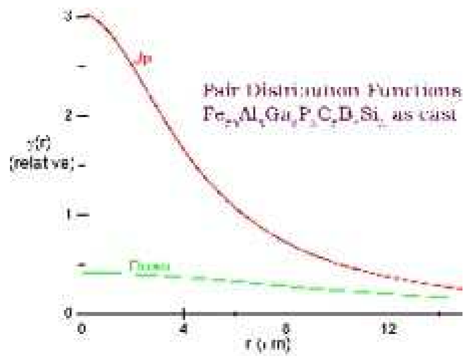

Figure 7. Inferred pair distribution function.

nuclear and magnetic scattering length density distributions $\rho_{\mathrm{N}}(\mathbf{r})$ and $\rho_{\mathrm{M}}(\mathbf{r})$ then equal half the sum and difference respectively between $\rho_{\mathrm{u}}(\mathbf{r})$ and $\rho_{\mathrm{d}}(\mathbf{r})$ (figure 4). The distributions extend up to a few micrometres, the up-spin distribution being narrower but stronger at $r = 0$ than that for the down-spin, as expected from the SUSANS spectra. The average diameter deduced for the scattering 'particle' is about 3 μm with the up- and 9 μm with the down-spin neutrons. Figure 5 displays the scattering length densities over a shell of radius r. The magnetic shell density is narrower and weaker than the nuclear shell density. The fitted $|F(\mathbf{Q})|^2$ distributions (figure 6) are inverse Fourier transformed [9] to obtain the respective pair distribution functions $\gamma(\mathbf{r})$ (figure 7), which are wider than the respective $\rho(\mathbf{r})$ curves.

To conclude, the first polarised SUSANS instrument described here spans $Q > 10^{-5}$ Å^{-1} range and can characterise micrometer-sized magnetic agglomerates in samples.

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