Experimental report

27/01/2016

Proposal:	**5-32-824**	**Council:**	4/2015
Title:	Spin correlations in lightly dopedMn:ZnO		
Research area:	Physics		

This proposal is a continuation of 5-32-793

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Samples: Mn:ZnO

Instrument	Requested days	Allocated days	From	To
D7	5	5	16/09/2015	21/09/2015

Abstract:

We propose here a continuation to our previous polarised neutron studies on the dilute magnetic semiconductor Mn:ZnO. These revealed rather different behaviour between solid-state and hydrothermally prepared samples; while the former was shown to contain MnO at all Mn concentrations, the latter indicated that some Mn was dissolved in the ZnO matrix. The low concentration (<3%) regime in the hydrothermally synthesised samples yielded only broad diffuse scattering consistent with either ferromagnetic correlations or paramagnetic spins. To resolve this ambiguity, we propose another experiment on D7, this time at a longer wavelength, allowing us to access lower Q.

ZnO is a wide-gap semiconductor crystallizing in the wurtzite (ZnS_2) structure, where each Zn^{2+} cation is coordinated tetrahedrally by four O^{2-} anions. Light doping by substitution of the zinc with another transition metal is reported to lead to room temperature ferromagnetism, which sparked interest from the field of spintronics.

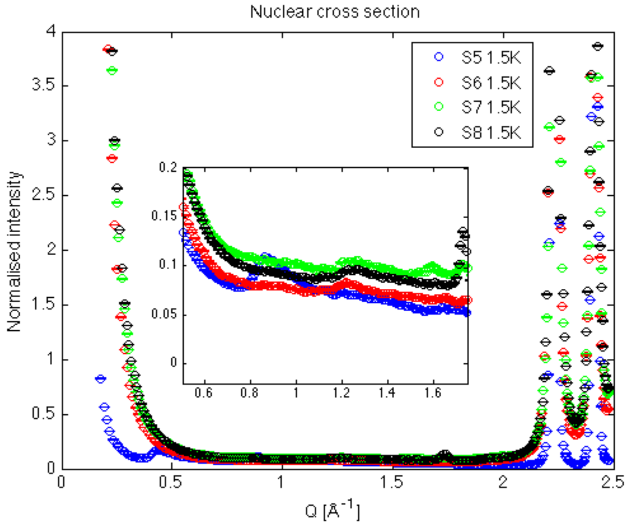
This experiment is a continuation of the study of the low temperature spin correlations in powder samples of Mn doped ZnO prepared by the thermal decomposition of hydrozincite in the composition range 0-3% Mn (actual). A previous experiment on DNS at FRMII with higher Mn concentration, manufactured via either a solid state method or via the thermal decomposition of hydrozincite, showed phase segregation of the manganese into MnO nanoparticles. A second experiment was then done on D7, measuring more lightly doped samples (1%,3%,5% nominal Mn doping, corresponding resp. to 0.59%,1.14% and 1.57% actual) and a higher doping as a reference at an incident wavelength of 3.1 Å. We observed possible indications of ferromagnetic short-ranged order but no evidence of MnO nanoparticles in the lightly doped samples. However, further investigation on D7 was needed to confirm and clarify the results, this time at a longer wavelength, allowing us to access lower Q.

During this D7 experiment, we measured 2 lightly doped samples of Mn:ZnO (1.57 and 1.14 actual%) along with pure ZnO, at a wavelength of 4.81 Å. The 0.59% doped sample was also measured at this wavelength during an internal beam time. The samples were about 12g each.

The spin incoherent cross section previously measured is large, which hints towards the adsorption of hydrogen, probably in the form of water on the sample. In order to limit this effect, the samples were baked at 100°C under vacuum for 4-12hrs and then immediately loaded into 20mm diameter Al cans. Calibrations were performed, including empty can, vanadium (annular), quartz (rod) and Cadmium (annular) measurements. This allowed subtraction of background and for calibrations of the detectors and analysers.

We performed 2ϑ scans with 11 points, at an incident wavelength of of 4.81 Å for temperatures between 1.8K and 200K. The measuring time for each was around 40 hours. We measured for polarisation along X, Y, Z, X+Y and X-Y. We were able to separate the magnetic, nuclear and spin-incoherent components of the scattering cross-section for all samples. The following figures show the nuclear, incoherent and magnetic cross sections for all the samples measured at 4.8 Å (lightly doped and pure ZnO).

Figure 1 : Nuclear cross section for the lightly doped samples S6 S7 S8 and pure ZnO S5



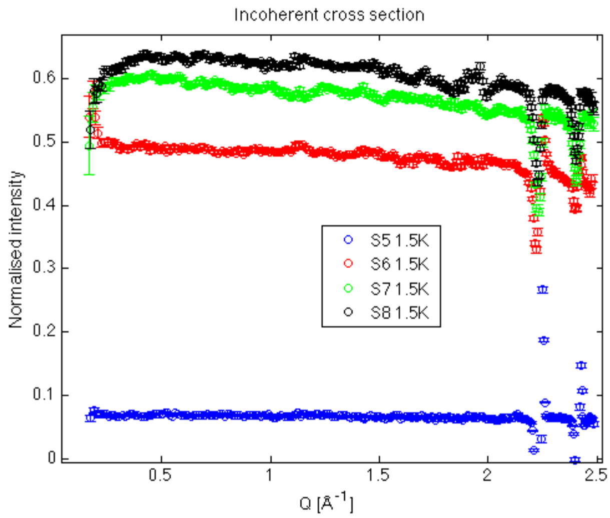


Figure 2 : Incoherent cross section for the lightly doped samples S6 S7 S8 and pure ZnO S5

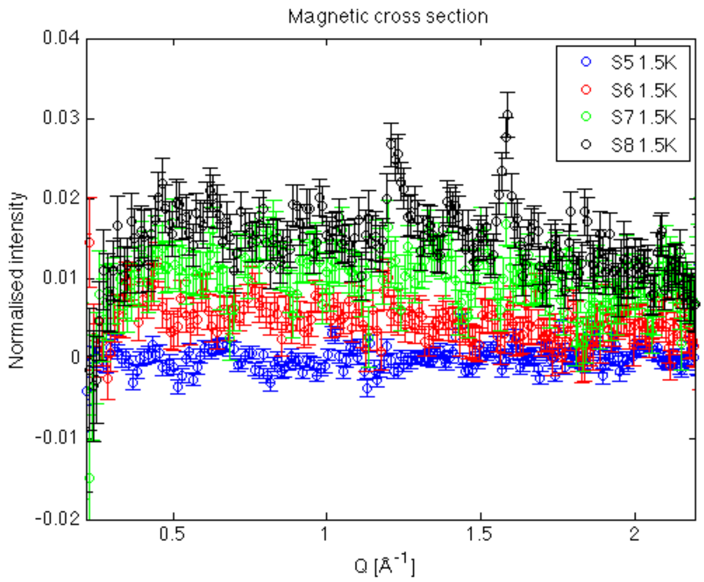


Figure 3 : Magnetic cross section for the lightly doped samples S6 S7 S8 and pure ZnO S5

The nuclear and incoherent cross section are coherent with previous results. In addition, we obtain new information on the magnetic cross section of the lightly doped samples, as we observe only paramagnetic behaviour for S6 (0.59% doping) and S7 (1.14% doping) which can be fitted with the Mn^{2+} form factor. S8 (1.57%) also shows no indication of ferromagnetism, but has two very narrow and weak peaks at Q points corresponding to MnO nanoparticles. Contrary to the conclusion from previous data at shorter wavelength, it is now clear that there is no indication of ferromagnetism in the more lightly doped samples in the Q range accessed.