

# Experimental Report

17/02/2015

<b>Proposal:</b>	<b>5-32-793</b>	<b>Council:</b>	4/2014	
<b>Title:</b>	Spin correlations in the lightly doped semiconductor oxide Mn:ZnO			
<b>This proposal is a new proposal</b>				
<b>Research Area:</b>	Physics			
<b>Main proposer:</b>	LANCON Diane			
<b>Experimental Team:</b>	NILSEN Goran			
<b>Local Contact:</b>	NILSEN Goran WILDES Andrew			
<b>Samples:</b>	MnxZn1-xO with x=0, 0.015, 0.03			
<b>Instrument</b>	<b>Req. Days</b>	<b>All. Days</b>	<b>From</b>	<b>To</b>
D7	5	5	11/12/2014	16/12/2014
<b>Abstract:</b> Ferromagnetism in dilute magnetic semiconducting oxide materials has attracted both interest and controversy. Most studies thus far have focused on bulk magnetic properties, and the nature of correlations in these materials is therefore poorly understood. We propose here a polarized neutron scattering study on a sample prepared by a novel method to search for short ranged correlations in MnxZn1-xO.				

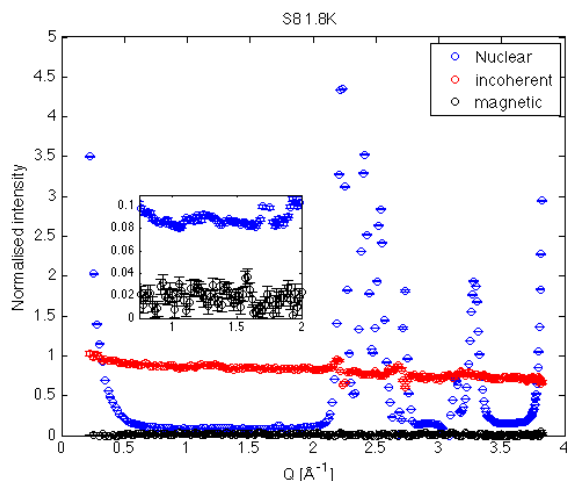
ZnO is a wide-gap semiconductor crystallizing in the wurtzite ( $\text{ZnS}_2$ ) structure, where each  $\text{Zn}^{2+}$  cation is coordinated tetrahedrally by four  $\text{O}^{2-}$  anions. Light doping by substitution of the zinc with another transition metal is reported to lead to room temperature ferromagnetism, which sparked interest from the field of spintronics.

This experiment aimed to study 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 the a solid state method or via the thermal decomposition of hydrozincite, showed phase segregation of the manganese into MnO nanoparticles.

During this D7 experiment, we measured 3 lightly doped samples of Mn:ZnO (1%,3%,5% nominal Mn doping), along with pure ZnO and a previous sample with actual Mn doping of 3.5% for reference. The masses for each sample were in the range 8-18g.

The spin incoherent cross section previously measured on DNS in similar samples 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 analyzers.

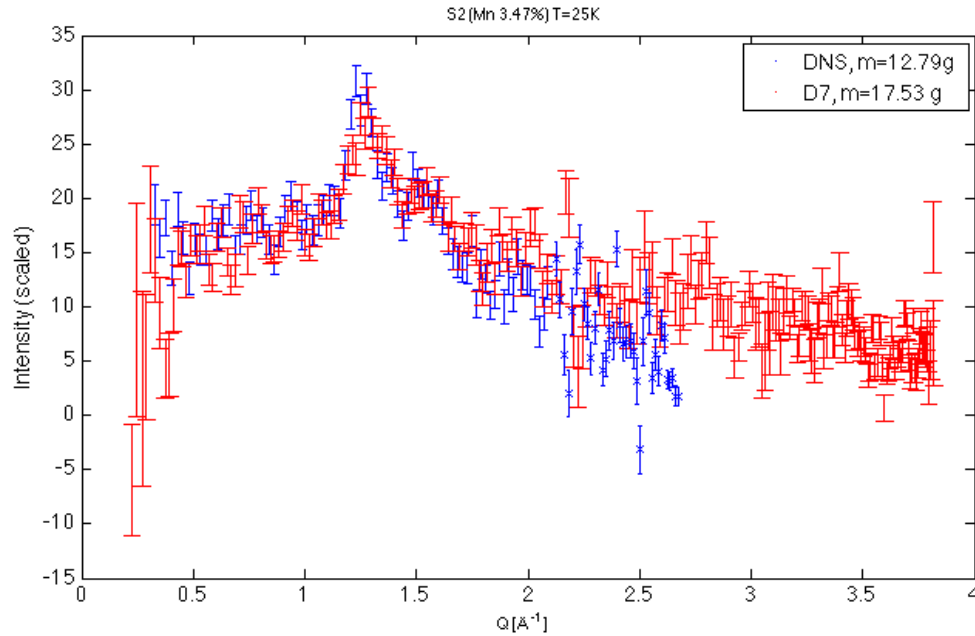
We performed 2 $\theta$  scans with 11 points, at an incident wavelength of 3.1 Å for a selection of temperatures between 1.8K and 200K. We measured for polarisation along X, Y, Z, X+Y and X-Y. Using this set-up, we were able to separate the magnetic, nuclear and spin-incoherent components of the scattering cross-section for all five samples, as shown on figure 1 for S8 (Mn doped at 5% nominal). Additional measurements with polarisation along X+Y and X-Y also allowed us to use the 10pts method for the separation of the cross-sections.



**Figure 1 : Separation of magnetic, nuclear and spin incoherent for 5% Mn (nominal) sample on D7**

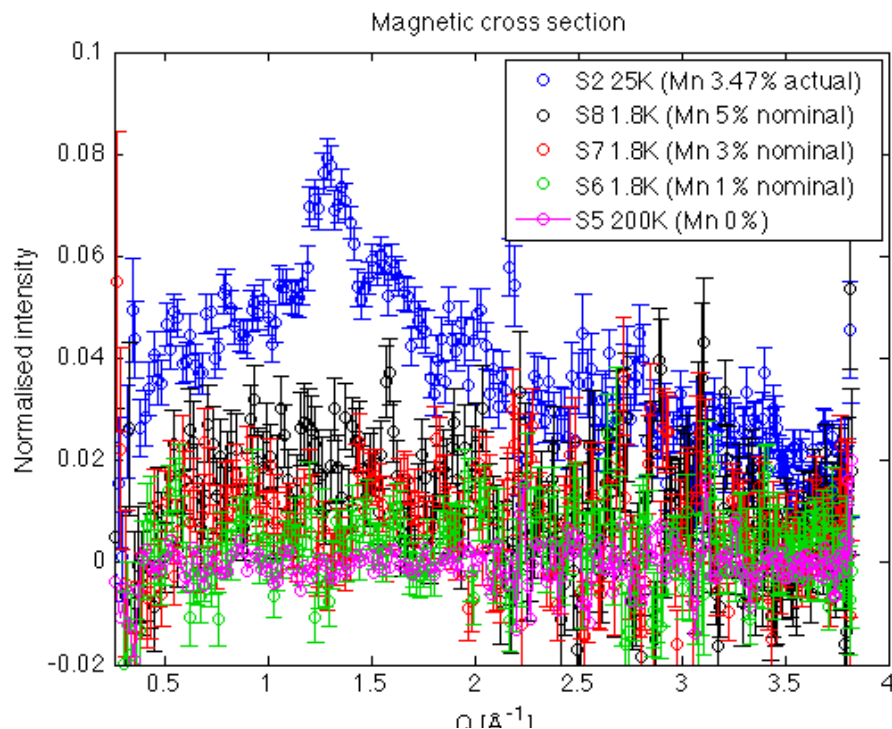
Sample 2 (3.47% actual Mn doping) had already been measured on DNS and was measured again during this D7 experiment, both as a reference for the more lightly doped samples and as a benchmark between the two instruments. Figure 2 shows measurements of the magnetic cross-

section for this sample on each instrument (counting time of 4hrs) and we observe in both cases the features identified as due to MnO nanoparticles.



**Figure 2 : Magnetic cross section for 3.47% Mn doping (actual) on both DNS and D7**

In the more lightly doped samples, we observed indications of ferromagnetic short-ranged order but no evidence of MnO nanoparticles, as shown in figure 3 which compares the magnetic cross section of all the samples measured during this D7 experiment.



**Figure 3 : Magnetic cross section of the Mn doped ZnO samples, with pure ZnO as a reference**