Experimental report

09/05/2022

Proposal: **EASY-926** **Council:** 10/2020

Title: Structural, Magnetic and Orbital Complexities in the Frustrated Spinel, ZnV_2O_4

Research area: Physics

This proposal is a new proposal

Main proposer: **Jennifer GRAHAM**

Experimental team: Jennifer GRAHAM

Local contacts: Andrew WILDES

Samples: ZnV_2O_4

Instrument	Requested days	Allocated days	From	To
D7	72	72	14/06/2021	17/06/2021

Abstract:

We have prepared two powder samples of ZnV_2O_4, one via a traditional high-temperature sintering method and the other via a novel rapid microwave-assisted route. We find that in the microwave sample, no structural transition occurs and the sample remains in a cubic phase down to 2 K. A single cusp in magnetic susceptibility at 11 K could be indicative of a spin glass phase. The sintered sample undergoes a structural transition at 40 K to a tetragonal structure. This is followed by an a magnetic transition at 13 K. We have performed a diffuse scattering study on the sintered sample on D7 (Exp. 5-32-897), where we find no indication of a long-range ordered magnetic ground state down to 2 K. Analysis using RMC methods shows that correlations between pairs of moments are weak and fall to zero within 15 A. We now wish to perform a similar study on the microwave sample in order to compare the magnetic ground states. This will allow us to complete our study on how synthesis affects the chemical structure and therefore the magnetic ground state.

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Due to only having 2 g of the microwave sample available for the experiment, only one measurement at 1.5 K was collected. The NC, NSI and magnetic contributions are shown in Fig. 1a) where the NSI is flat, indicating the separation is good. However, closer inspection of the magnetic contribution in Fig. 2a) shows that the magnetic contribution is very flat, despite counting for three days. As we only had a small sample, with a small signal ($\mathbf{S} = 1$) there can be an error with the background subtraction leading to very flat magnetic cross-sections, as is the case here. To fix this, the background subtraction was removed from the data reduction, and the resultant NC, NSI and magnetic contributions are shown in Fig. 1b). Now the NSI is not flat, and there is very large scattering at low Q in the NC, but the magnetic cross-section in Fig. 2b) is showing a feature. This is okay to do, as the calibration and background measurements do not include any magnetic contributions but does leave the NSI and NC cross-sections unusable. The magnetic feature is not as defined as the sintered sample, but does roughly follow the same shape, and peaks at the same point. Physically this suggests a more strongly frustrated magnetic ground state, which is inkeeping with our explorations into the average structure, in which we found from diffraction measurements that the chemical structure remained cubic down to 4 K and the spin glass type transition in the susceptibility. The NC in the background subtracted data set (Fig. 1a)) is similar to the NC of the sintered sample with small, additional peaks present due to the VO_2 impurity in this sample.

The diffuse magnetic data for the microwave sample were fit using `SPINVERT`. Like the sintered sample, a cubic $Fd\bar{3}m$ setting was used, weight = 10 and moments allowed complete rotational degrees of freedom. However, a smaller box size of $4 \times 4 \times 4$ unit cells was used as the feature was less defined. The resultant fit is shown in Fig. 3 and describes the data well. The derived magnetic moment from the `SPINVERT` scale is $\mu_{\text{eff}} = 0.54\ \mu_{\text{B}}$ per V^{3+} ion, which is significantly reduced from the expected moment of $\mu_{\text{eff}} = 2\ \mu_{\text{B}}$ per V^{3+} ion. This μ_{eff} is also reduced from the sintered effective moment at the same temperature. However, this experimental effective moment is in agreement with previous neutron scattering measurements

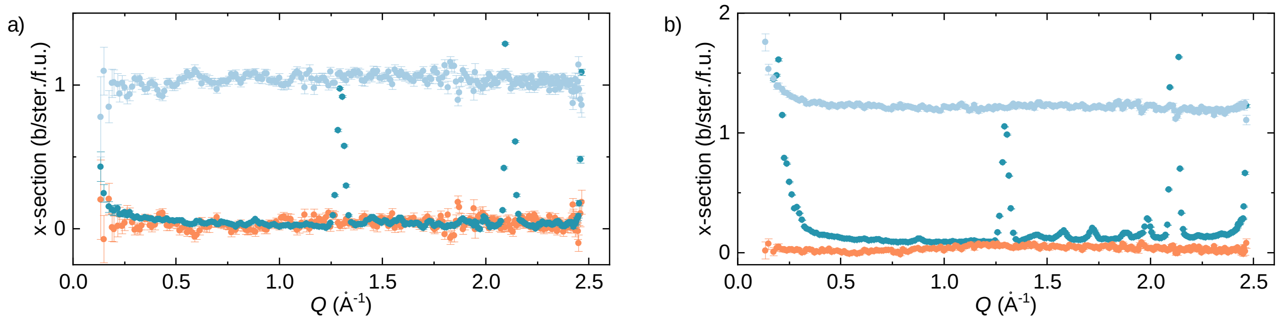

Figure 1: NC (dark blue), NSI (light blue) and magnetic (orange) cross-sections for the microwave sample at 1.5 K. a) With background subtraction and b) without background subtraction.

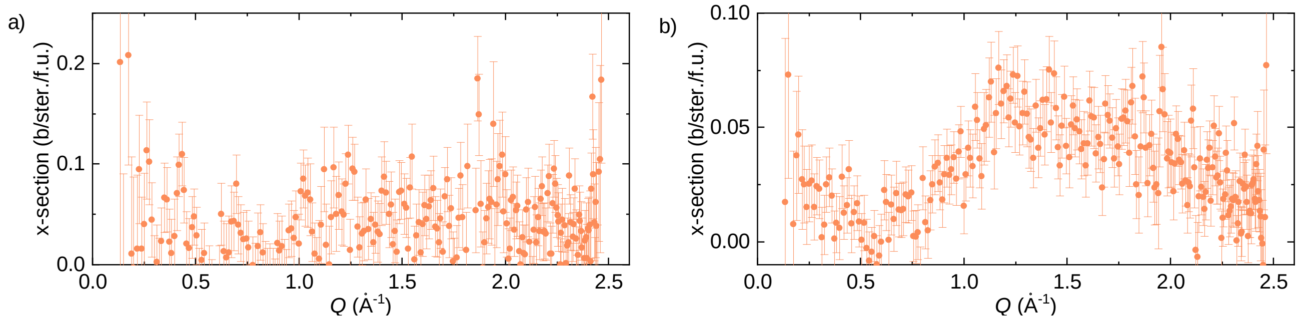

Figure 2: Diffuse magnetic cross-sections at 1.5 K for the microwave sample. a) With background subtraction and b) without background subtraction.

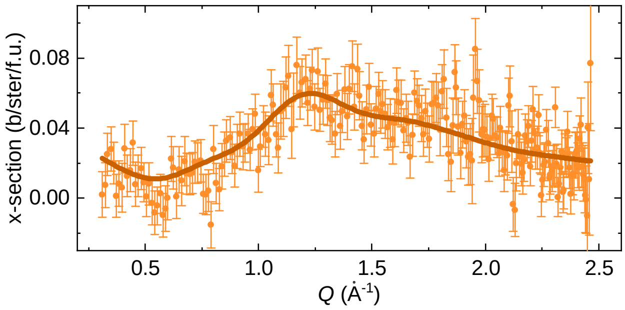

Figure 3: Magnetic diffuse scattering data for the microwave sample at 1.5 K. Data have been fit using the `SPINVERT` program, as shown by the solid line.

of $\mu_{\text{eff}} = 0.65\ \mu_{\text{B}}$ by Lee *et al.*. Some possible explanations for this underestimate of the effective moment could be significant spin-orbit couplings, a low spin state on the V^{3+} ions or a dimerised state.

We will calculate the spin correlations using `SPINCORREL` and compare their behaviour against our other ZnV_2O_4 sample.