Experimental report

11/09/2019

Proposal: **5-31-2613**

Council: 4/2018

Title: Investigation of the exotic magnetic ground state in cycled Na07CoO2

Research area: Physics

This proposal is a new proposal

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Samples: NaCo0p7O2

Instrument	Requested days	Allocated days	From	To
D20	5	2	12/10/2018	13/10/2018

Abstract:

NaxCoO2 (NCO) is famous for its exotic phase-diagram that changes dramatically with the number of conduction electrons on the transition metal oxides layer. Most of the reported studies were based on samples growth by the solid-state reaction method, which is known for producing samples with inhomogeneous Na content. It has become evident that the potential landscape from ordered Na-vacancies plays an important role for the ground state of this material. Lately, the NCO compound has gone through somewhat of a revival, driven by the energy storage community and industry. This has resulted in the realization of electrochemically cycled (in a battery cell) NCO samples. We propose a powder neutron diffraction study of homogenous and well-ordered Na0.7CoO2 samples grown by the electrochemical method. In detail, this sample started out as an “as grown” (non-cycled) x = 0.7 sample and has then been subjected to a full battery charge/discharge cycle by an electrochemical process, returning it to x = 0.7 composition. Muon spin rotation (μ+SR) measurement on this sample revealed a magnetic phase below T = 20 K, which was not existing in the solid-state sample.

Investigation of the exotic magnetic ground state in cycled Na07CoO2

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1. Introduction

NCO is well known for being a very sensitive sample where a long series of irreproducible experimental results have been presented by many different research groups. Most of the reported studies were based on samples growth by the solid-state reaction method, which is known for producing samples with inhomogeneous Na content. Lately, an increase of interest in NCO, driven by the energy storage community and industry, has resulted in the realization of electrochemically cycled (in a battery cell) NCO samples. This process uses stable x = 0.7 samples as starting material and give access to the full range of Na-contents (x) with a reproducible and robust Na- order via the well-controlled Na-ion intercalation process.

The general goal of this proposed experiment is to investigate cycled NCO samples with x = 0.7. In detail, this sample started out as an "as grown" (non-cycled) x = 0.7 sample and has then been subjected to a full battery charge/discharge cycle by an electrochemical process, returning it to x = 0.7 composition. Through our previous muon spin spectroscopy (muSR) study, the cycled NCO was shown to order at lower temperature (the none-cycled NCO does not). Since obtaining the detailed magnetic order is difficult with muSR, our goal with this proposal was to: Reveal the detailed spin structure of the cycled and clearly magnetically ordered x = 0.7 sample.

2. Experiment

The sample is known to be air sensitive and was thus sealed in a Vanadium can inside a He Glovebox. The available sample mass (m ≈ 300 mg) was considerably small and a relatively long exposure times were needed. Using a CCR, a temperature range of 5 – 300 K was available and several neutron diffraction patterns were collected as a function of temperature. The collected patterns were analyzed during the experiment using the software package Fullprof. Unfortunately, the reactor went down during the last day and there was no time to collect data at higher temperature.

3. Results

The neutron diffraction pattern at base temperature revealed no aditional or changes in peaks in comparison to the pattern above T_N = 22 K (apart from thermal displacements/contraction). Regardless, the crystal structure and the atomic positions, specifically the Na positions (not visible with X-rays), were obtained and will be a key information inorder to undertsand the underlying mechanism behind the magnetic order. In order to dicern any sample decomposition during the travel from Sweden and France, the same batch was quickly measured with muSR and a clear magnetic order was visible.

4. Conclusion

The main goal of the experiment was to unveal the detailed spin structure of the magnetic order at low temperature. Our neutron diffraction experiment revealed no magnetic signal. We belive the resons to be a too weak magnetic moment (not a problem in muSR). The size of the magnetic moment for the sample is around the limit of what is considered in Neutron diffraction. Indeed, sample degredation during the transport from Sweden to France needs to be considered as well. However, this deems unlikly considering the observed neutron diffraction pattern (crystallographic) were as expected, and both batches provode similar results.