Experimental report

15/02/2021

Proposal: 5-24-636 **Council:** 4/2019

Title: Insights into the molten salt synthesis of new honeycomb magnets

Research area: Chemistry

This proposal is a resubmission of 5-24-619

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Samples: li2ruo3
AgNO3
Li2MnO3

Instrument	Requested days	Allocated days	From	To
D20	3	1	23/09/2019	24/09/2019

Abstract:

Honeycomb magnets have recently returned to prominence due to the prediction and discovery of Kitaev physics. Nevertheless, the family of candidate materials is limited. This is due to the requirement for particular heavy transition metal oxidation states. 10 years ago, we discovered a molten salt route to exchange the interlayer cations in Li2MO3 materials. This was demonstrated by converting Li2RuO3 to Ag3LiRu2O6, with a surprising change in magnetic properties. However, we currently have no idea of the active species present in the molten salt, and no idea how to tune e.g. stacking disorder. Here we propose the first in-situ neutron powder diffraction during this process. This will yield insights into the above problems, and may even yield routes to completely delaminate this important materials.

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In this experiment, we investigated the synthesis of metastable mixed noble metal oxides. These honeycomb materials are of interest for their magnetic properties, due to the high level of spin-orbit coupling, which induced novel ground states.

Of particular interest is Ag3LiRu2O6, which is made by metathesis of Li2RuO3 in molten AgNO3. This chemistry was believed to take place above the melting point of AgNO3 (210 °C), and to take some hours to complete (when we originally made this material, it was held for 24 hours at high temperature).

We measured a sample of Li2RuO3:AgNO3 in a 1:10 ration. Although this was contained in a silica ampoule, and contains only 200 mg of Li2RuO3, all phases are clearly seen. We actually find that reaction occurs in the solid state, as soon as the beta-AgNO3 polymorph is formed. Once the melting point is exceeded, bulk reaction occurs in a just a few minutes. Full analysis of this data is in progress, and expected to lead to a publication soon.

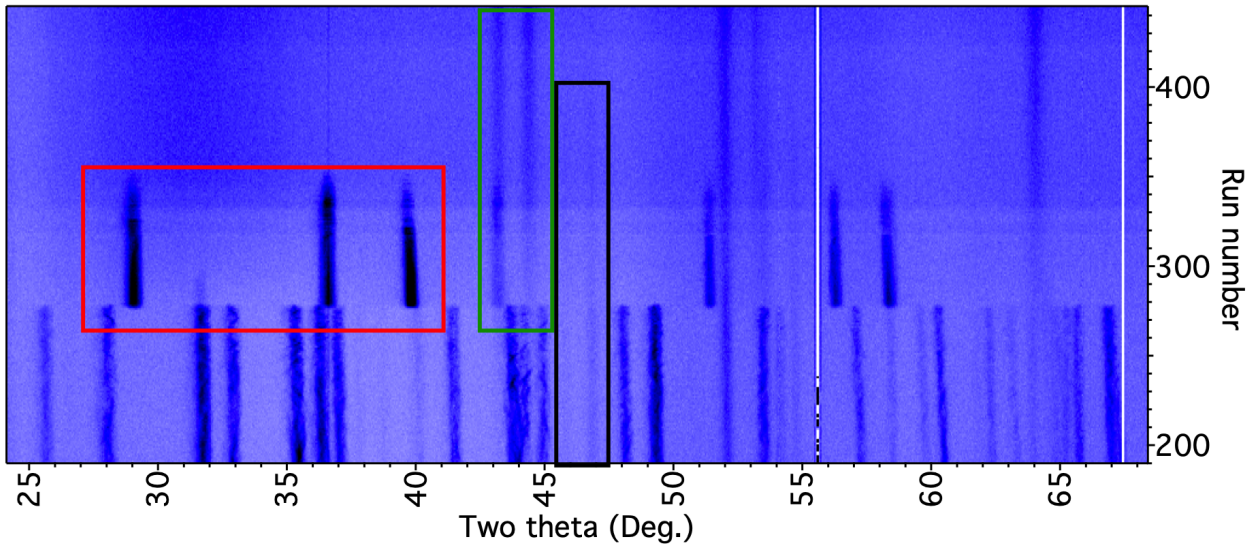

Fig. 1, temperature dependent data for a 1:10 mixture of Li2RuO3 and AgNO3. The red box shows beta-AgNO3, green is Ag3LiRu2O6, and black is Li2RuO3

In addition, we collected data for pure AgNO3 on warming, as shown in Fig. 2, we detect the alpha-beta phase transition, melt, and metastable phases on cooling. This work is the subject of a followup proposal on D2B.

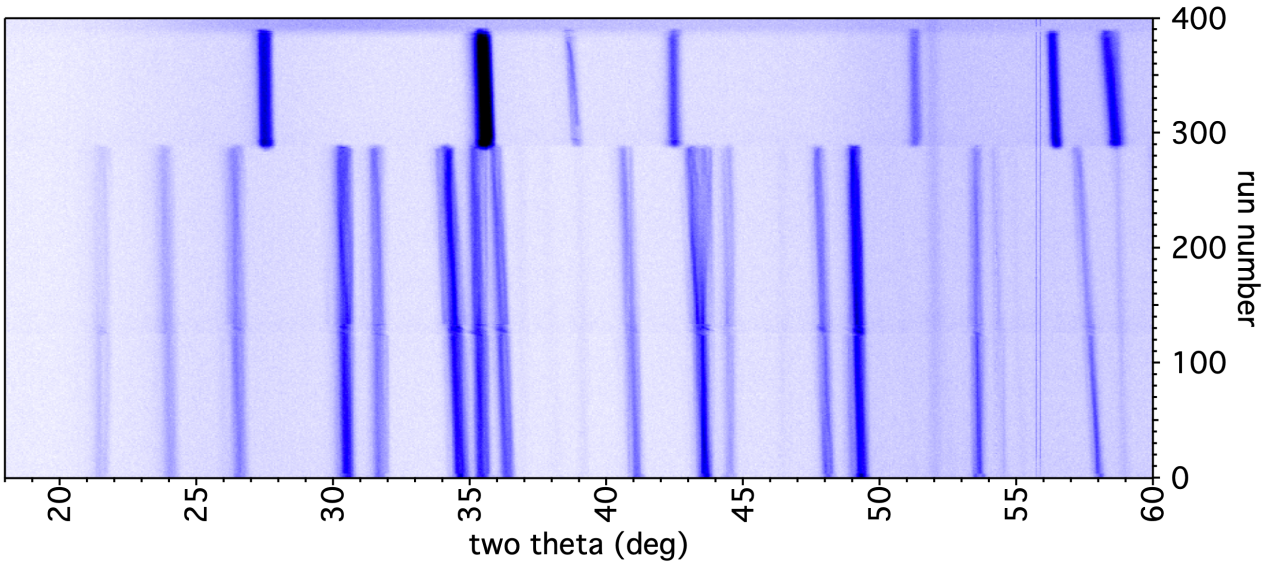

Fig. 2, temperature dependent data for pure AgNO3. The alpha-beta phase transition is clearly seen, as is the melting point.