Experimental report

04/04/2022

Proposal: EASY-858 **Council:** 10/2020

Title: Crystal structure and magnetic ground state of novel Mn3MnTa2O9 high pressure triple perovskite.

Research area: Materials

This proposal is a new proposal

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Experimental team:

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Samples: Mn3MnTa2O9

Instrument	Requested days	Allocated days	From	To
D20	12	12	14/05/2021	15/05/2021

Abstract:

Ambient pressure Mn4Nb2O9 and Mn4Ta2O9 are well known multiferroic materials. The high pressure (HP) modification of Mn4Nb2O9 (Mn3MnNb2O9) crystallises with a triple perovskite (TPv) structure, with A sites occupied by Mn2+ and B sites by perfectly 1:2 ordered Mn:Nb, thus notable as the first A-site manganite with TPv structure.
Three transitions at TN = 52 K, TR = 29 K and TL = 4.2 K are observed for Mn3MnNb2O9 from magnetic susceptibility and heat capacity data. D20 NPD data revealed the spins align into a colinear AFM structure at TN, modulate below TR into a complex SDW with kR = [kx 0 kz] continuously evolving down to a locked phase with kL = [⅓ 0 -].
HP Mn3MnTa2O9 crystallises with isostructural TPv (S.G. Cc, a = 9.92(1)Å, b = 5.33(1)Å, c = 13.24(1)Å and β = 92.8(1)°). Magnetic susceptibility shows three transitions at 52.6 K, 30.9 K and 22.8 K, presumably alike those of the Nb analogue. Only the two first are observed from heat capacity, suggesting a different nature of the third one. A D20 long scan is requested on Mn3MnTa2O9 at 300 K using λ = 1.59 Å for accurate structural determination and a low temperature scan using λ = 2.41 Å to confirm the magnetic ground state.

Crystal structure and magnetic ground state of novel $Mn_3MnTa_2O_9$ high pressure triple perovskite.

Elena Solana Madruga

$Mn_4Nb_2O_9$ and $Mn_4Ta_2O_9$ are well known as multiferroic materials when prepared under ambient pressure conditions. A complex monoclinic distortion of the cubic perovskite structure had recently been observed for the high-pressure modification of $Mn_4Nb_2O_9$ (HP-$Mn_3MnNb_2O_9$) with *Cc* space group and $a = \sqrt{2}\sqrt{3}a_c$, $b = \sqrt{2}a_c$ and $c = 2\sqrt{3}a_c$ cell parameters. It was reported as the first A-site manganite with triple perovskite (TPv) structure and 1:2 (Mn:Nb) B-site order. A complex magnetic behaviour with three subsequent transitions was observed, where the spins align into a colinear AFM structure with propagation vector [0 0 0], modulate into a complex SDW with $k = [k_x\ 0\ k_z]$ propagation vector continuously evolving down to a locked phase with $k_L = [⅓\ 0\ -⅙]$ at low temperature.

In this experiment, the related HP-$Mn_3MnTa_2O_9$ was measured. High resolution NPD data were collected on D20 at 300 K (Fig. 1a) for accurate structural characterisation using the 90° take-off angle and $\lambda = 1.54$ Å in the $0^\circ < 2\theta < 150^\circ$ angular range with a 0.05˚ step size. Thermodiffraction studies were performed from D20 high intensity data collected at the take-off angle of 42° on ramping from 1.5 to 70 K using $\lambda = 2.41$ Å (Fig. 1b). Magnetic structures (Fig. 1c) were determined from 1.5 K and 40 K long scans collected under the same conditions.

The data collected from this EASY experiment enabled the structural characterisation, confirming the TPv structure with *Cc* space group (Fig. 1a bottom) and the determination of the complex ground magnetic structure and its thermal evolution, similar to that reported for the related niobate. The results were soon published on *J. Mater. Chem. C*, **2021**, 9, 14916, where the experiment's DOI (10.5291/ILL-DATA.EASY-858) is included in the acknowledgements section.

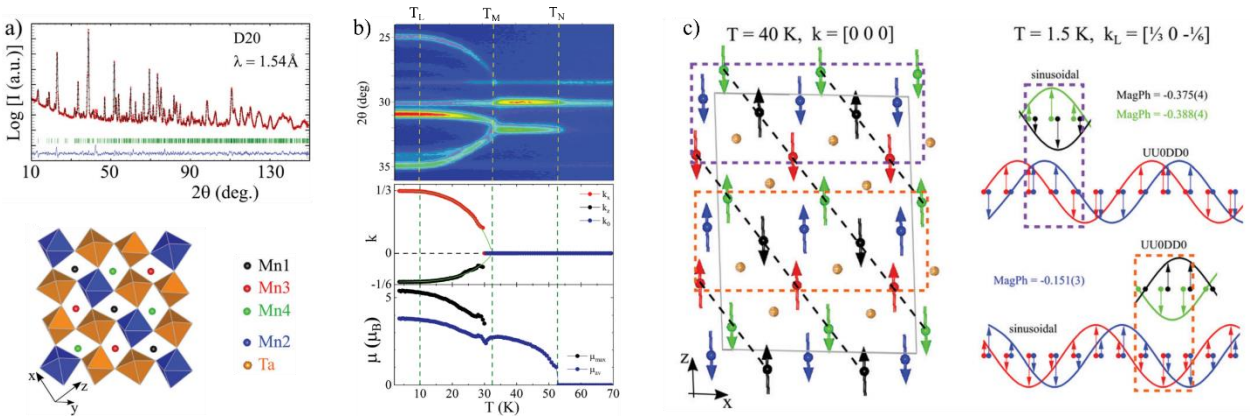

Figure 1. a) Rietveld fit against D20 high resolution NPD data collected at 300 K (top) and refined TPv structure of HP-$Mn_3MnTa_2O_9$ (bottom). b) Thermodiffraction data collected in high intensity mode selected in $24^\circ < 2\theta < 36^\circ$ range along with refined propagation vector and magnetic moment. c) Magnetic structures refined at 40 K and 1.5 K.