Experimental report

29/05/2020

Proposal: 5-31-2623 **Council:** 10/2018

Title: Crystal and magnetic structures ofthe high pressure CaMnMSbO6 (M = Cr, Fe) double double perovskites.

Research area: Materials

This proposal is a new proposal

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Samples: CaMnCrSbO6
CaMnFeSbO6

Instrument	Requested days	Allocated days	From	To
D1B	0	0		
D20	2	2	08/07/2019	10/07/2019

Abstract:

Perovskites, with the general formula ABO3, are of great interest due to their large variety of electronic and magnetic properties. Their composition can be modified to induce different cation orders into double perovskites (DPv), of general formula AA'B2O6 or A2BB'O6, and eventually doubly ordered perovskites (AA'BB'O6). Using high pressure and high temperature (HPHT) synthesis, we recently found a new type of perovskite derivative combining columnar order of the A-site cations with rock-salt order at the B sites into a so-called double double perovskite (DDPv). This structure crystallises with P42/n space group and a few cationic combinations have been checked to stabilise it under HPHT conditions.
Here we present two new DDPvs CaMnCrSbO6 and CaMnFeSbO6. They show ferrimagnetic behaviours with a single (Cr, TN = 49 K) and two successive (Fe, TN1 = 55 K, TN2 = 21 K) transitions respectively. Their accurate cation order is intended to be determined from room temperature NPD and their magnetic structures will be characterised at 1.5 K. The thermal evolution of their magnetic structures will also be studied.

Crystal and magnetic structures of the high pressure $CaMnMSbO_6$ (M = Cr, Fe) double double perovskites.

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Perovskites, with the general formula ABO_3, are of great interest due to their large variety of electronic and magnetic properties. Their composition can be modified to induce different cation orders into double perovskites (DPv), of general formula $AA'B_2O_6$ or $A_2BB'O_6$, and eventually doubly ordered perovskites ($AA'BB'O_6$) [1]. Using high pressure and high temperature (HPHT) synthesis, we recently found a new type of perovskite derivative combining columnar order of the A-site cations with rocksalt order at the B sites into a so-called double double perovskite (DDPv) [2] This structure crystallises with $P4_2/n$ space group and different cationic combinations have been checked to stabilise it under HPHT conditions, for instance $MnRMnSbO_6$ and $CaMnMReO_6$ [3].

In this experiment, two new high pressure $CaMnFeSbO_6$ and $CaMnCrSbO_6$ DDPv oxides were proposed. The later had been preliminary measured on D20 during experiment 5-31-2602 as a backup sample using take off angle 118˚ and λ = 1.87 Å. 3 h scans were collected at 1.5, 65 and 300 K, allowing for the complete nuclear and magnetic structural characterisations. All magnetic peaks are indexed with propagation vector k = [0 0 0] into a collinear Ferrimagnetic structure with FM Mn^{2+} and Cr^{3+} sublattices aligned into opposed directions along the y axis, as shown in Fig 1.

$CaMnFeSbO_6$ was measured using the more standard λ = 2.41 Å for magnetic structural determination. 2 h scans were collected at 3, 35 and 70 K and short scans were taken every ~1 K between 3 and 35 K. An additional high resolution pattern was collected at 300 K using the 90˚ take off angle and λ = 1.54 Å. The quasicubic cell parameters of this compound did not allow to unequivocally determine the direction of the magnetic moments from the low temperature data using 2.41 Å. Consequently, an additional 1.5 h scan was collected at 3 K during the related experiment 5-31-2690 using the 90˚ take off angle and λ = 1.54 Å. The results of these data allowed the complete characterisation of the sample, which magnetic structure, depicted in Fig. 2, shows Mn^{2+} and Fe^{3+} FM sublattices aligned antiparallel along the z axis.

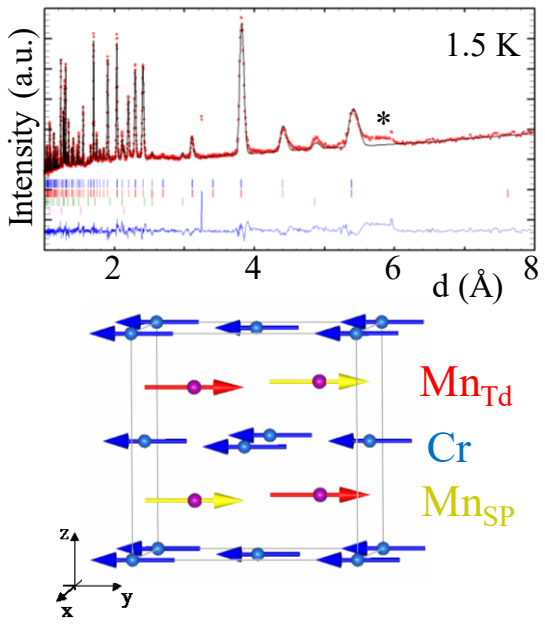

Fig. 1. Magnetic structure of the $CaMnCrSbO_6$ DDPv as refined from 1.5 K NPD data.

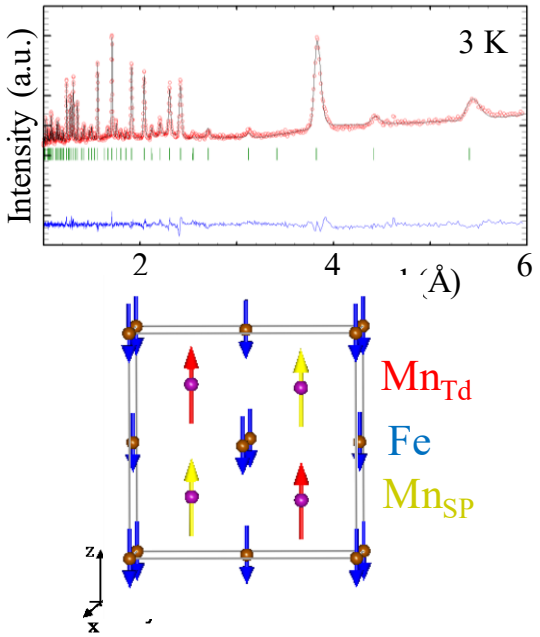

Fig. 2. Magnetic structure of the $CaMnFeSbO_6$ DDPv as refined from 3 K NPD data.

[1] G. King, P.M.Woodward. *Journal of Materials Chemistry*, 2010, **20**, 5785.
[2] E. Solana-Madruga et.al. *Angew. Chem. Int. Ed*. 2016, **55**, 9340.
[3] G. M. McNally et al. *Chem. Mater.* 2017, **29**, 8870−8874.