



# Structural, magnetic and magnetocaloric properties of vanadium-doped manganites $\text{La}_{0.65}\text{Ca}_{0.35}\text{Mn}_{1-x}\text{V}_x\text{O}_3$ ( $0 \leq x \leq 0.5$ )



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## ARTICLE INFO

### Article history:

Received 12 July 2016

Received in revised form 5 December 2016

Accepted 27 January 2017

Available online 4 February 2017

### Keywords:

Solid state reaction

Manganites

Magnetocaloric effect

Magnetic properties

## ABSTRACT

Structural, magnetic and magnetocaloric properties of polycrystalline  $\text{La}_{0.65}\text{Ca}_{0.35}\text{Mn}_{1-x}\text{V}_x\text{O}_3$  have been investigated. Our samples were prepared using the solid state reaction method at high temperatures. X-ray diffraction analysis show that all samples crystallized in the orthorhombic structure with Pbnm space group with presence of  $\text{LaVO}_4$  as secondary phase. The SEM micrographs reveal micrometric grains with bimodal size distribution. Magnetic Measurements indicate that our samples exhibit a PM–FM transition, with a Curie temperature ( $T_C$ ) decreasing from 262 K for  $x = 0$  to 208 K for  $x = 0.5$ ; in addition to the presence of the Griffiths phase in the paramagnetic region. Using the isothermal magnetization versus magnetic applied field, we have deduced that the occurred magnetic transition is of second order. Moreover, we have calculated the magnetic entropy change, which reaches 5.25 J/kg K for  $\text{La}_{0.65}\text{Ca}_{0.35}\text{Mn}_{0.5}\text{V}_{0.5}\text{O}_3$  compound under 5T. This value is associated to a relative cooling power (RCP) of 207 J/kg.

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## 1. Introduction

Magnetic refrigeration is an efficient and a friendly refrigeration technology that can replace the common vapor compression–expansion cooling cycles of different refrigeration devices [1,2]. This technology is of great interest because of its good social effect and economical benefits and the possibility to develop high efficiency devices. It is based essentially on the magnetocaloric effect (MCE), which was firstly discovered by Warburg in 188 [3]. The MCE is characterized by the isothermal magnetic entropy change or the adiabatic temperature change emanating from the application or the removal of a magnetic field to or from a system with a magnetic degree of freedom.

Different materials were used for magnetic refrigeration application [4–6]. Recently, manganites have attracted considerable attention thanks to their specific structural peculiarities, charge and orbital ordering, magnetic phase transition, colossal magnetoresistance (CMR) effect and magnetocaloric effect (MCE) [7–9]. MCE properties are usually explained by the double exchange (DE) interaction between ( $\text{Mn}^{3+}$ ) and ( $\text{Mn}^{4+}$ ) ions [10]. Such interactions are determined by intrinsic parameters as average cationic

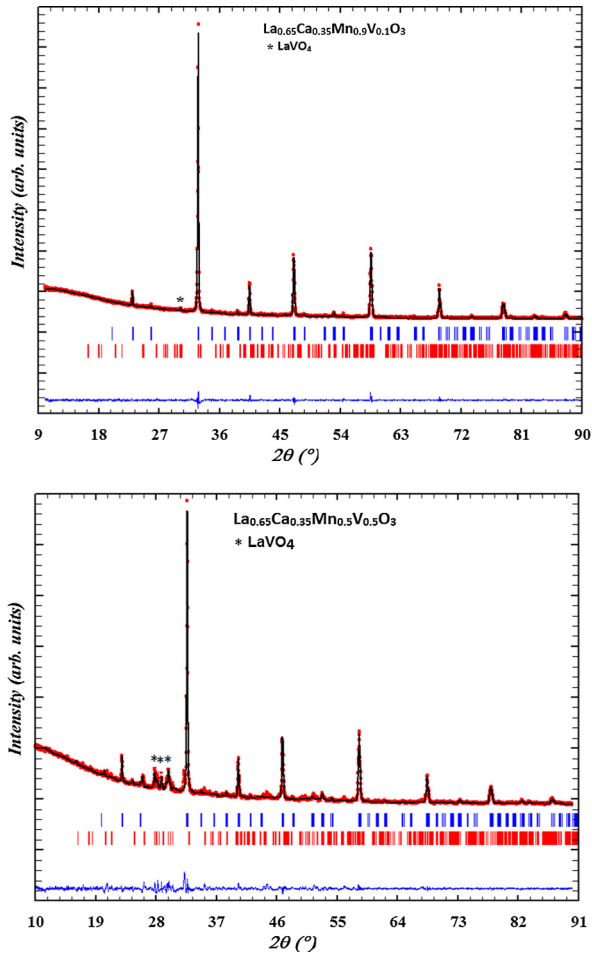
size [11], cationic disorder, doping level and oxygen stoichiometry [12]. The structural, magnetic and magnetocaloric properties of different manganites are sensible to many factors such as A and B-site doping. The average size of the A and B-site cations is an important parameter that can influence the different properties of manganites.

In our research group, we have largely studied the magnetocaloric effect (MCE) in manganites especially the  $\text{La}_{0.65}\text{Ca}_{0.35}\text{MnO}_3$  system and significant values were observed, especially with a partial calcium substitution by monovalent element [13–16].

In this paper, we report a magnetic and magnetocaloric study of  $\text{La}_{0.65}\text{Ca}_{0.35}\text{MnO}_3$  perovskite doped with vanadium in B site. Recently, V-doped manganites have been well studied. Gencer et al. [17] have reported the structural and the magnetic properties of  $\text{La}_{0.67}\text{Ca}_{0.33}\text{Mn}_{1-x}\text{V}_x\text{O}_3$  samples. Zhao and al. [18] have also demonstrated the existence of  $\text{LaVO}_4$  secondary phase, which crystallizes in a monoclinic structure with  $P2_1/n$  space group. They also noticed the presence of a multi-phase compound when sintered in the air since V does not substitute Mn easily to form  $\text{La}_{0.67}\text{Ca}_{0.33}\text{Mn}_{1-x}\text{V}_x\text{O}_3$  compound. The reported Curie temperature is found to decrease with V content. Moreover, Mansouri et al. [19] demonstrated that only 5% of vanadium can destroy the charge order present in  $\text{La}_{0.5}\text{Ca}_{0.5}\text{MnO}_3$  system.

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**Fig. 1.** X-ray diffraction patterns at 300 K including the observed and calculated profiles as well as the difference profile for  $\text{La}_{0.65}\text{Ca}_{0.35}\text{Mn}_{1-x}\text{V}_x\text{O}_3$  ( $x = 0.1$ ;  $x = 0.5$ ).

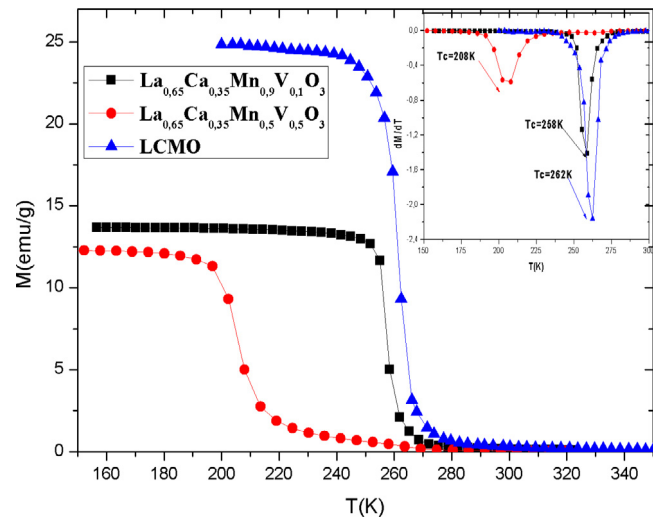
**Table 1**  
Cell parameters and unit cell volume for  $\text{La}_{0.65}\text{Ca}_{0.35}\text{Mn}_{1-x}\text{V}_x\text{O}_3$ .

x	0	0.1	0.5
Space group	P b n m	P b n m	P b n m
a (Å)	5.469(1)	5.455(3)	5.494(5)
b (Å)	5.432(1)	5.470(3)	5.477(2)
c (Å)	7.740(1)	7.706(8)	7.741(9)
V (Å <sup>3</sup> )	229.936	229.938	232.931
% LaVO <sub>4</sub>	0	3.05	8
$\chi^2$	1.5	2	3.1

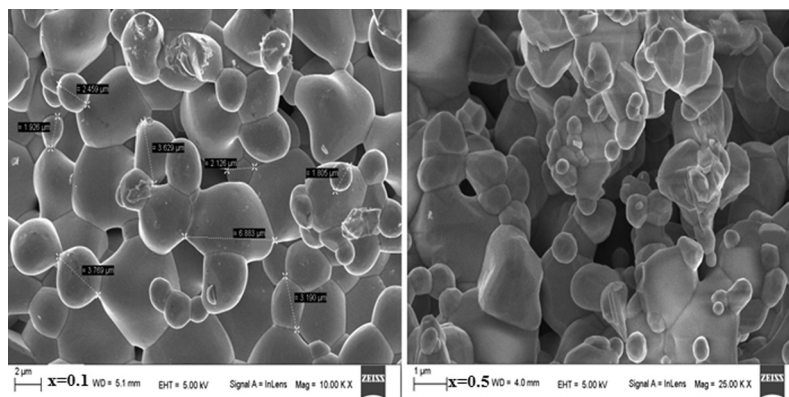
## 2. Experimental section

Our samples were prepared by the conventional solid state reaction method at high temperature. Stoichiometric amounts of high-purity powders;  $\text{La}_2\text{O}_3$ ,  $\text{CaCO}_3$ ,  $\text{MnO}_2$  and  $\text{V}_2\text{O}_3$  were mixed together during 30 min in the suitable proportions for the reaction. The mixture was then heated up to 800 °C for 24 h and 1000 °C for 18 h with intermediate grindings and repelling. Finally, the powder was pressed into pellets and sintered for 48 h at 1300 °C.

Phase Purity, homogeneity, and cell dimensions were determined using X-ray powder diffraction (XRD) with Cu  $K_\alpha$  radiation at room temperature. Structural studies were carried out using standard Rietveld technique [20]. The microstructural properties and the morphology of the samples were analyzed by scanning electron microscopy (SEM) via a Philips XL30 microscope which operates at 5 kV. Magnetization measurements versus temperature in the temperature range 10–300 K and versus magnetic applied field up to 5T were performed using a Vibrating Sample Magnetometer (VSM). Using the Maxwell relation, MCE was deduced from the magnetization measurements versus the magnetic applied field up to 5T at several temperatures.



**Fig. 3.** Temperature dependence of magnetization measured in a magnetic applied field of 0.05T for  $\text{La}_{0.65}\text{Ca}_{0.35}\text{Mn}_{1-x}\text{V}_x\text{O}_3$  with  $0 \leq x \leq 0.5$ . The Inset shows  $dM/dT$  curves versus temperature for  $\text{La}_{0.65}\text{Ca}_{0.35}\text{Mn}_{1-x}\text{V}_x\text{O}_3$  ( $x = 0$ ;  $x = 0.1$  and  $x = 0.5$ ).



**Fig. 2.** SEM images for  $\text{La}_{0.65}\text{Ca}_{0.35}\text{Mn}_{1-x}\text{V}_x\text{O}_3$  ( $x = 0.1$ ;  $x = 0.5$ ).

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