



**INFLUENCE OF HEATING TEMPERATURE ON Nd-Sr-Mn-O
MANGANITES SYNTHESISED VIA THERMAL TREATMENT AND
SOL-GEL METHOD**

By

HON XIAO TONG

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in
Fulfilment of the Requirements for the Degree of Master of Science**

December 2023

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Chair : Lim Kean Pah, PhD
Faculty : Science

Mixed-valence manganites have garnered significant interest owing to their intriguing colossal magnetoresistance effect. In the realm of manganites, the primary challenge is achieving remarkable magnetoresistance (MR) values even with weak magnetic fields, rendering these materials applicable for the utilisation of magnetic sensors near room temperatures. The selection of preparation methods and synthesis routes is crucial as it can alter the grain size formation and/or distribution, subsequently affecting the magnetic and electrical behaviour of manganite materials. In this project, a newly developed approach known as the thermal treatment method (TT) was employed to synthesise $\text{Nd}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$ (NSMO) manganites. Given the novelty of this approach, optimisation and fine-tuning of heat treatment parameters are imperative to achieve the utmost desirable physical properties. The first part of this investigation focuses on assessing the impact of calcination temperatures on the NSMO samples. Higher crystallinity was achieved when NSMO was calcined at 500 °C and 900 °C. However, an unfavorable morphology was observed in sample calcined at 900 °C and sintered at 1200 °C. Besides, the NSMO samples exhibited distinct magnetic phase transition when calcined at 500 °C. Regarding magneto-transport behaviour, the intrinsic MR is more predominant in the sample calcined at 500 °C and sintered at 1200 °C. Thus, it can be deduced that 500 °C is the optimum calcination temperature. The next part of this thesis aims to explore the grain size effect on the NSMO samples synthesised via thermal treatment and sol-gel (SG) methods at various sintering temperatures (700 °C to 1200 °C). While the sol-gel method yielded a single NSMO phase, minor phases were detected in the NSMO samples prepared using the thermal treatment method at a lower sintering temperature. XRD result indicated larger crystallite size formation and better crystallinity in SG, but SEM analysis revealed a greater variation of grain size in TT. Additionally, TT exhibited a broader magnetic phase transition due to the existence of nonferromagnetic clusters resulting from the detected minor phases. In both series of samples, the presence of MR peaks suggests the prevalence of intrinsic MR, with peak intensity increasing as the

sintering temperature rises. Interestingly, contrary to prior research, the intrinsic MR is more dominant in SG samples with smaller grain sizes, which can be attributed to the microstructure change and morphology variations. In conclusion, this study highlights the significance of structural and microstructural formation resulting from various synthesis methods in governing the physical properties of manganite samples. This study stands out as the pioneer in preparing Nd-based manganites using the thermal treatment method. It offers the advantages of being cost-effective and environmentally friendly, while also featuring a simplified preparation method for manganites.



Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

PENGARUH SUHU PEMANASAN PADA MANGANIT Nd-Sr-Mn-O YANG DISINTESISKAN MELALUI KAEDAH RAWATAN TERMA DAN SOL-GEL

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Manganit valensi campuran telah menarik perhatian yang ketara kerana ia mempamerkan kesan magnetorintangan kolosal yang menarik. Dalam bidang manganit, cabaran utama ialah mencapai nilai magnetorintangan (MR) yang luar biasa walaupun dengan medan magnet yang lemah, menjadikan bahan ini boleh digunakan dalam aplikasi sensor medan magnet berhampiran dengan suhu bilik. Pemilihan kaedah penyeliaan dan laluan sintesis adalah penting kerana ia boleh mengubah pembentukan dan/atau taburan saiz butiran, seterusnya menjejaskan tingkah laku magnet dan elektrik bahan manganit. Dalam projek ini, pendekatan baru yang dikenali sebagai kaedah rawatan terma (TT) telah digunakan untuk mensintesis manganit $\text{Nd}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$ (NSMO). Memandangkan kebaruan pendekatan ini, pengoptimuman dan penalaan halus parameter rawatan haba adalah penting untuk mencapai sifat fizikal yang paling diingini. Bahagian pertama penyiasatan ini memberi tumpuan kepada kesan suhu pengkalsinan pada sampel NSMO. Kehabluran yang lebih tinggi telah dicapai apabila NSMO dikalsin pada 500 °C dan 900 °C. Walau bagaimanapun, morfologi yang kurang baik telah diperhatikan dalam sampel yang dikalsin pada 900 °C dan disinter pada 1200 °C. Selain itu, sampel NSMO mempamerkan peralihan fasa magnet yang berbeza apabila dikalsin pada 500 °C. Mengenai tingkah laku magneto-pengangkutan, MR intrinsik didapati lebih pradominan dalam sampel yang dikalsin pada 500 °C dan disinter pada 1200 °C. Oleh itu, boleh disimpulkan bahawa 500 °C ialah suhu pengkalsinan yang optimum. Bahagian seterusnya tesis ini bertujuan untuk meneroka kesan saiz butiran pada sampel NSMO yang disintesis melalui kaedah rawatan terma dan kaedah sol-gel (SG) pada suhu pensinteran yang berbeza (700 °C hingga 1200 °C). Kaedah sol-gel telah menghasilkan fasa tunggal NSMO, manakala fasa minor pula dikesan dalam sampel NSMO yang disediakan oleh kaedah rawatan terma apabila disinter pada suhu yang lebih rendah. Keputusan XRD menunjukkan pembentukan saiz hablur yang lebih besar dan keabluran yang lebih baik dalam SG, tetapi analisis SEM mendedahkan variasi saiz butiran yang lebih besar dalam TT. Selain itu, TT mempamerkan peralihan fasa magnet yang lebih lebar kerana kewujudan kelompok bukan feromagnet yang terhasil daripada fasa minor yang dikesan. Dalam kedua-dua siri sampel, kehadiran puncak MR menunjukkan kelaziman MR intrinsik, dengan keamatan puncak meningkat apabila suhu pensinteran

meningkat. Menariknya, bertentangan dengan penyelidikan terdahulu, MR intrinsik lebih dominan dalam sampel SG dengan saiz butiran yang lebih kecil, yang boleh dikaitkan dengan perubahan mikrostruktur dalam variasi morfologi. Kesimpulannya, kajian ini mengetengahkan kepentingan pembentukan struktur dan mikrostruktur hasil daripada kaedah sintesis yang berbeza dalam mengawal sifat fizikal sampel manganit. Kajian ini menonjol sebagai perintis dalam penyediaan manganit berasaskan Nd menggunakan kaedah rawatan terma. Ia menawarkan kelebihan seperti kos efektif dan mesra alam, sementara memaparkan kaedah penyediaan manganit yang ringkas.



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LIST OF ABBREVIATIONS

AFI	Antiferromagnetic insulating
AFM	Antiferromagnetic metallic
BSE	Backscattered electrons
CP	Co-precipitation
CMR	Colossal magnetoresistance
DE	Double exchange
E_A	Activation energy
FESEM	Field emission scanning electron microscopy
FI	Ferromagnetic insulating
FM	Ferromagnetic
FWHM	Full width at half maximum
GMR	Giant magnetoresistance
H	Magnetic field
H_C	Coercivity
HMS	Hall effect measurement system
ICSD	Inorganic Crystal Structure Database
J-T	Jahn-Teller
LFMR	Low field magnetoresistance
LSMO	Lanthanum strontium manganese oxide
M	Magnetisation
MR	Magnetoresistance
MR (%)	Magnetoresistance value
M_s	Saturation magnetisation

NSMO	$\text{Nd}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$
PI	Paramagnetic insulating
PM	Paramagnetic
PVP	Polyvinylpyrrolidone
R	Resistance
R^2	Squared linear correlation coefficient
SE	Secondary electrons
SEM	Scanning electron microscopy
SG	Sol-gel
SPH	Small polaron hopping
SS	Solid-state
T	Temperature
T_C	Curie temperature
T_{MI}	Metal-insulator transition temperature
T_S	Sintering temperature
TT	Thermal treatment
VRH	Variable range hopping
VSM	Vibrating sample magnetometer
XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction
θ_D	Debye's temperature
ρ	Resistivity
ρ_{peak}	Peak resistivity
χ^2	Goodness of fit

CHAPTER 1

INTRODUCTION

This chapter begins with an overview of manganite research, then addresses the problem statement, and explores the research gaps. Furthermore, the objectives of this work are clearly outlined.

1.1 Background

The rich history of manganite studies can be traced back to 1950 when Jonker and Van Santen (1950) initiated their pioneering investigations by introducing alkaline earth metals such as SrMnO_3 , into the structure of LaMnO_3 . Their groundbreaking research discovered that manganites evinced marvellous traits by experiencing metal-insulator transition as well as ferromagnetism in the presence of mixed valency of manganese ions in the system. Building on these seminal findings, Volger (1954) embarked on further comprehensive experimental investigations on ferromagnetic manganese oxide with perovskite structures. His study revealed the emergence of the negative magnetoresistance (MR) effect in $\text{La}_{0.8}\text{Sr}_{0.2}\text{MnO}_3$ near its Curie temperature, T_C . The 1990s witnessed a resurgence of interest in mixed-valence manganite, culminating in the advancement of Chahara et al. (1993) and von Helmolt et al. (1993) by developing high-quality thin films with giant magnetoresistance (GMR). However, the true breakthrough occurred when Jin et al. (1994) made a remarkable discovery by unveiling an impressively strong negative isotropic MR effect, famously known as colossal magnetoresistance (CMR) in La-Ca-Mn-O thin films. This CMR effect surpassed the typical GMR effect observed in such films by more than three orders of magnitude.

At the core of manganite studies lies the perovskite structure, defined by a cubic crystal lattice where oxygen atoms surround manganese ions in octahedral arrangements (Pavone et al., 2014). This structure holds the key to understanding the complex electronic and magnetic properties of manganites. Within this crystal framework, some influential theories come to the forefront, including the double exchange (DE) mechanism and the Jahn-Teller distortion (J-T) effect (Shimomura et al., 1999). These theories have unlocked the secrets behind CMR and the diverse electronic behaviours, leading them to find their way into the practical world, revolutionising fields such as magnetic sensors, magnetic data storage, and spintronic devices (Xia et al., 2020).

Manganites exhibit a fascinating interplay between intrinsic and extrinsic MR effects. The former originates from the grain properties while the latter emerges from the spin-dependent scattering or spin-polarised tunnelling near grain boundaries (Panwar et al., 2007). Thus, the magneto-transport properties are closely associated with the microstructural behaviour of the sample, which can be influenced by the specific preparation methods utilised (Raddaoui et al., 2021). This marks the significance of the synthesis route in shaping the physical properties of these materials.

In summary, manganite perovskites have garnered worldwide attention thanks to their captivating electronic and magnetic attributes. These materials hold the promise to bring about significant advancements across various fields, rendering them an exceptionally fascinating area of study within the science community.

1.2 Problem Statement

Extensive research efforts have been committed to the study of CMR materials. This dedication is motivated not only by their diverse physical characteristics but also by their promising roles in applications. Thus, the key challenge revolved around achieving significant MR values under low magnetic fields, particularly at or near room temperature (Acharya et al., 2017). While lanthanum-based manganites have been extensively studied in the past, recent research has shifted its focus towards exploring different types of perovskite manganites. This brought neodymium-based manganites into the spotlight as a primary area of interest in current studies. Among the perovskite manganites investigated thus far, the neodymium-based manganite system with narrow bandwidth stands out for displaying intriguing phenomena distinct from those observed in lanthanum-based manganite systems (Bhargav et al., 2022).

The physical traits of manganites are intricately shaped by the preparation methods and processing conditions. Among these methods, the solid-state reaction method stands out for its simplicity (Amin et al., 2022). It provides a quick route to synthesise perovskite materials with high purity and excellent crystallinity (Sujiono et al., 2018). However, it does have its drawbacks, including the requirement for extremely high temperatures and prolonged heating duration. Additionally, it is necessary to go through multiple rounds of milling and heating cycles to achieve the desired phase and chemical compositions. Thus, researchers have embraced the wet chemical approach, specifically the sol-gel method, to tackle this shortcoming (M'nassri et al., 2015). Moreover, this wet chemical approach permits better stoichiometry, shorter heating time, and smaller particle size (Brinker & Scherer, 2013; Danks et al., 2016; Zhao et al., 2017). In this project, a newly developed synthesis method, known as the thermal treatment method, is employed for NSMO preparation. Since this is a novel approach, optimisation and fine-tuning of heat treatment parameters are essential to attain the best possible physical properties.

Previous studies proposed that physical properties are strongly governed by controlling both the grain size and distribution. This can be accomplished by adjusting the calcination and sintering temperatures. The sintering behaviour of manganite samples was discovered to be highly sensitive to the calcination conditions. Calcination serves as the initial stage where the precursor material undergoes controlled heating at temperatures typically below the melting point of the desired product in order to facilitate the decomposition of nitrates and the formation of the oxide (Jiang, 2012). On the other hand, sintering involves the elevated heating of the materials at significantly high temperatures (typically near the melting point of the desired compound) resulting in diffusion between the particles and forming a compact structure (Bhattacharya & Basak, 2016; Lim et al., 2009). These crucial steps can induce alterations in microstructure as well as a reduction in grain size, subsequently influencing their physical behaviour.

1.3 Objectives

Building on the existing manganite research background and addressing its research gap, it is crucial to emphasise that the physical properties of manganites are strongly dependent on the preparation methods as well as the microstructure modification. Thus, the objectives of this project are delineated as follows:

1. To determine the effect of calcination and sintering temperatures on the NSMO samples synthesised via the thermal treatment method.
2. To investigate the influence of sintering temperatures on the structural, microstructural, magnetic, electrical and magneto-transport properties of NSMO samples prepared by thermal treatment and sol-gel methods.
3. To evaluate the grain size effect on the physical properties of NSMO samples prepared by thermal treatment and sol-gel methods.

1.4 Thesis Content

The thesis starts with Chapter 1 by providing an introduction to the research project. Subsequently, Chapter 2 offers an extensive literature review encompassing prior work on perovskite manganites and the analysis theory underpinning this project. Moving forward, Chapter 3 covers the methodology of sample preparation, the material and equipment used, as well as the characterisation techniques employed. This is followed by the result and discussion in Chapter 4, while Chapter 5 concludes the research with recommendations for future investigations.

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