UNIVERSITI PUTRA MALAYSIA

MICROSTRUCTURE AND DIELECTRIC PROPERTIES OF $La_{1-x}(Ba,Ca)_x Mn_{0.40}Ti_{0.60}O_3$ ($0.30 \le x \le 0.50$) CERAMIC SYSTEMS

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By

AALIYAWANI EZZERIN BINTI SININ

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

October 2015

DEDICATION

I dedicate this thesis to my family especially my beloved husband, daughter and both my father and mother.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

MICROSTRUCTURE AND DIELECTRIC PROPERTIES OF $La_{1-x}(Ba,Ca)_x Mn_{0.40}Ti_{0.60}O_3$ ($0.30 \leq x \leq 0.50$) CERAMIC SYSTEMS

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October 2015

Chair: Walter Charles Primus, PhD
Faculty: Agriculture and Food Science (Bintulu)

Composites of $La_{1-x}A_xMn_{0.40}Ti_{0.60}O_3$ with A = Ba and Ca where $0.30 \leq x \leq 0.50$ were prepared by the conventional solid-state reaction method. The samples were calcined twice at 800 °C for 12 hours and 1000 °C for 12 hours in powder form. Powder samples were pressed into pellets and sintered at 1200 °C for 12 hours. The sintered composites were characterized in terms of microstructure, crystal structure and dielectric properties at frequency range of 0.01 Hz – 100 kHz at room temperature to 200 °C using the scanning electron microscope (SEM), the X-ray diffraction method (XRD), and a low frequency LCR analyzer respectively.

The surface morphology of the samples shows rod-like and rounded grains for both series of samples. $La_{1-x}Ba_xMn_{0.40}Ti_{0.60}O_3$ has rounded grains with diameter approximately 1.675 μm and rod-like grains with length approximately 5.280 μm. For $La_{1-x}Ca_xMn_{0.40}Ti_{0.60}O_3$, the spherical shaped grains are 0.558 μm in diameter and rod-like grains with length of 1.083 μm. XRD analysis shows that both $La_{1-x}Ba_xMn_{0.4}Ti_{0.6}O_3$ and $La_{1-x}Ca_xMn_{0.4}Ti_{0.6}O_3$ have cubic, $Pm\bar{3}m$ structure with a few impurity peaks. Increasing the amount of barium and calcium ions at lanthanum site has increased the lattice parameter from 3.9404 Å to 3.9553 Å and volume ranging from 61.18 $Å^3$ to 61.88 $Å^3$ for $La_{1-x}Ba_xMn_{0.40}Ti_{0.60}O_3$ and from 3.9370 Å to 3.9579 Å for lattice parameter and volume, from 61.02 $Å^3$ to 62.00 $Å^3$ for $La_{1-x}Ca_xMn_{0.40}Ti_{0.60}O_3$ samples.

Dielectric properties measurement shows that the dielectric permittivity depends strongly on the applied frequency and temperature. The real part of dielectric permittivity, ε′ increases with the decreasing frequency and increases with the increasing temperature due to space-charge polarization and thermally activated electron hopping. A graph of log permittivity, ε′ versus temperature, T (K) was plot at 1 Hz, 1 kHz, and 10 kHz showing that the dielectric permittivity is more stable at all temperatures at higher frequencies that are 1 kHz and 10 kHz which is in the range of 1000 to 10000 for all samples. At 1 Hz, the dielectric permittivity for both barium and calcium series increase slightly from 100000 to 1000000 as the sample is heated. Analysis on the real part of capacitance, C′ and imaginary capacitance, C″ data under the function of frequency using the universal capacitor response reveal that the samples are dominated by diffusion in series with a quasi d.c process at room temperature and a quasi d.c process from a temperature of 313.15 K and above for $La_{1-x}Ba_xMn_{0.40}Ti_{0.60}O_3$. For $La_{1-x}Ca_xMn_{0.40}Ti_{0.60}O_3$, only quasi d. c. process takes place for all temperatures.

The characteristic frequency, f_c is found to be temperature dependence, the value of the fractional correlation, p is close to 1 indicates that the electrons motion are highly correlated at intercluster region while the correlation coefficients for specific intracluster, n decreases as temperature increases. The activation energy for both series has a value ranging from 0.30 eV to 0.50 eV for $La_{1-x}Ba_xMn_{0.40}Ti_{0.60}O_3$ and from 0.10 eV to 0.20 eV for $La_{1-x}Ca_xMn_{0.40}Ti_{0.60}O_3$.

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

MIKROSTRUKTUR DAN PENCIRIAN DIELEKTRIK SISTEM SERAMIK $La_{1-x}(Ba,Ca)_x Mn_{0.40}Ti_{0.60}O_3$ ($0.30 \leq x \leq 0.50$)

Oleh

AALIYAWANI EZZERIN BINTI SININ

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Komposit $La_{1-x}A_xMn_{0.40}Ti_{0.60}O_3$ dengan A = Ba dan Ca di mana $0.30 \leq x \leq 0.50$ telah disediakan menggunakan kaedah tindak balas keadaan pepejal yang konvensional. Sampel dikalsin sebanyak dua kali pada suhu 800 °C selama 12 jam dan 1000 °C untuk 12 jam dalam bentuk serbuk. Sampel serbuk di mampatkan menjadi pelet dan disinter pada suhu 1200 °C selama 12 jam. Komposit yang disinter dicirikan dari segi mikrostruktur, struktur, struktur kristal, dan sifat dielektrik menggunakan mikroskop imbasan electron (SEM), teknik pembelauan sinar-X (XRD), dan penganalisi LCR berfrekuensi rendah masing-masing.

Morfologi permukaan menunjukkan butiran sampel berbentuk rod dan berbentuk bulat bagi kedua-dua siri sampel. $La_{1-x}Ba_xMn_{0.40}Ti_{0.60}O_3$ mempunyai butiran berbentuk sfera yang licin dengan diameter 1.675 μm dan panjang zarah rod dalam 5.280 μm. Bagi $La_{1-x}Ca_xMn_{0.40}Ti_{0.60}O_3$, butiran berbentuk sfera mempunyai diameter 0.558 µm dan butiran seperti rod mempunyai panjang 1.083 μm Analisis XRD menunjukkan kedua-dua $La_{1-x}Ba_xMn_{0.4}Ti_{0.6}O_3$ dan $La_{1-x}Ca_xMn_{0.4}Ti_{0.6}O_3$ mempunyai struktur kubik, $Pm\bar{3}m$ dengan beberapa puncak bendasing. Penambahan amaun ion barium dan calcium di tapak lanthanum telah meningkatkan parameter kekisi dari 3.9404 Å ke 3.9553 Å dan isipadu, dalam julat dari 61.18 $Å^3$ ke 61.88 $Å^3$ untuk $La_{1-x}Ba_xMn_{0.40}Ti_{0.60}O_3$ dan dari 3.9370 Å ke 3.9579 Å untuk parameter kekisi dan isipadu, dari 61.02 $Å^3$ ke 62.00 $Å^3$ untuk sampel $La_{1-x}Ca_xMn_{0.40}Ti_{0.60}O_3$.

Pengukuran ciri-ciri dielektrik menunjukkan bahawa ketelusan dielektrik dipengaruhi oleh frekuensi dan suhu yang dikenakan. Bahagian nyata ketelusan dielektrik, ε' meningkat dengan penurunan frekuensi dan meningkat dengan peningkatan suhu disebabkan oleh pengutuban caj ruang serta loncatan elektron yang diaktifkan oleh haba. Graf log ketelusan dielektrik, ε' lawan suhu, T (K) pada frekuensi pada 1 Hz, 1 kHz, dan 10 kHz. Graf tersebut menunjukkan ketelusan dielektrik lebih stabil pada frekuensi1 kHz, dan 10 kHz yang berada dalam julat 1000 ke 10000 untuk semua sampel. Pada frekuensi 1 Hz, ketelusan dielektrik untuk kedua-dua siri barium and calcium meningkat sedikit dari 100000 ke 1000000 apabila sampel dipanaskan. Analisis bahagian nyata kapasitans, C' dan kapasitans tidak nyata, C'' dalam fungsi frekuensi menggunakan respon kapasitor universal menunjukkan sampel didominasi oleh proses resapan yang bersiri dengan proses quasi d.c pada suhu bilik dan hanya proses quasi d.c pada suhu 313.15 K ke atas untuk sampel siri $La_{1-x}Ba_xMn_{0.40}Ti_{0.60}O_3$. Bagi sampel $La_{1-x}Ca_xMn_{0.40}Ti_{0.60}O_3$ hanya proses quasi d.c yang berlaku bagu kesemua

suhu. Frekuensi pencirian, f_c didapati bersandar kepada suhu, nilai nisbah korelasi, p menghampiri 1 menunjukkan pergerakan elektron adalah berkolerasi tinggi dalam interkluster tertentu dan pemalar kolerasi untuk intraklustur tertentu, n menurun apabila suhu meningkat. Tenaga pengaktifan bagi kedua-dua siri berada dalam julat daripada 0.30 eV ke 0.50 eV untuk $La_{1-x}Ba_xMn_{0.40}Ti_{0.60}O_3$ dan dari 0.10 eV ke 0.20 eV untuk $La_{1-x}Ca_xMn_{0.40}Ti_{0.60}O_3$.

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I certify that a Thesis Examination Committee has met on 28 October 2015 to conduct the final examination of Aaliyawani Ezzerin binti Sinin on her thesis entitled "Microstructure and Dielectric Properties of $La_{1-x}(Ba,Ca)_x Mn_{0.40}Ti_{0.60}O_3$ ($0.30 \le x \le 0.50$) Ceramic Systems" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Degree of Masters of Science.

Members of the Thesis Examination Committee were as follows:

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LIST OF SYMBOLS AND ABBREVIATIONS

λ	Wavelength
θ	Diffraction angle
ε	Permittivity
ε_o	Permittivity of free space
ε_r	Dielectric relative permittivity
χ	Dielectric susceptibility
α	Polarization
ε_∞	Permittivity at high frequency
ε'	Real part of dielectric permittivity
ε''	Imaginary part of dielectric permittivity
τ	Relaxation time
Z	Impedance
Y	Admittance
C^*	Complex capacitance
C'	Real part of capacitance
C''	Imaginary part of capacitance
f_c	Characteristic frequency
SEM	Scanning Electron Microscope
XRD	X-ray Diffraction
LF-IA	Low Frequency Impedance Analyzer
G	Conductance
σ	Conductivity
μ	Micro
ρ	Density
a	Lattice parameter
Å	Angstrom
ln	Natural logarithm
E_a	Activation energy
k	Stefan Boltzmann constant

CHAPTER 1

INTRODUCTION

1.1 Research Background

Ceramics are made from compounds of a metal and a non-metal which is inorganic and are form by the action of heat and subsequent cooling. Ceramic materials are very unique in which it can possess different properties such as magnetic, superconductive and insulating. Commonly, ceramic materials are almost insulators.

Insulators are materials which does not conduct electricity thus has no free moving charges. Most insulators are dielectric materials, dielectrics are electric insulators but can be influenced and polarized by an external electric field. The charges do not move freely but actually shift a bit from their average equilibrium position causing dielectric polarization. Dielectric materials such as barium titanate and calcium titanate are known as ferroelectrics. In particular, ferroelectric ceramics which was born in the early 1940s was the discovery of the ferroelectric phenomenon of ferroelectricity as the source of unusually high dielectric constant in ceramic barium titanate capacitors (Haertling, 1999).

Ferroelectric shows spontaneous polarization below the Curie temperature, ferroelectric domains and has a ferroelectric hysteresis loop. The spontaneous polarization is the value of dipole moment per unit volume or the value of charge per unit area on the surface perpendicular to the axis of spontaneous polarization and is temperature dependent. Ferroelectric crystals possess regions having uniform polarization known as ferroelectric domains where all the electric dipoles are aligned in the same direction. These spontaneous polarizations can be switched whenever an external field is applied and the polarization reversal can be observed by obtaining the ferroelectric hysteresis loop. All ferroelectric material has a transition temperature known as the Curie point, (T_C). At temperatures higher than the Curie temperature, the crystals do not exhibit ferroelectricity, and are ferroelectrics at situations below the Curie temperature. When decreasing the temperature through the Curie point, the ferroelectric material undergoes a phase change from a non-ferroelectric to a ferroelectric phase.

The simplest type of ferroelectric crystal (and the one most widely studied) is the perovskite structure (Ashcroft and Mermin, 1987). When doping or substituting with other elements to these materials, the dielectric properties changes or shifts where the phase changing temperature can be shifted to a higher value in order to contribute in electrical application. However, to date, no material that possess high dielectric properties with magnetic properties.

In this research, ceramic compound of $La_{1-x}Ba_xMn_{0.40}Ti_{0.60}O_3$ and $La_{1-x}Ca_xMn_{0.40}Ti_{0.60}O_3$ ($0.30 \leq x \leq 0.50$) was fabricated in order to study the substitution effect on dielectric properties and crystal structure of the ceramics produced.

In this dissertation, Chapter 2 is the literature review related to the research. In Chapter 3, the theory applied in the measurement of this research and data analysis was listed out and elaborated. Chapter 4 explains the methodology used is fabricating the samples and also including the experimental process, tools, and machines involved in this research.

The results and discussion was placed in Chapter 5. In this chapter, all the graphs and analysis of samples was combined and explained and elaborated including both the microstructure and dielectric analysis. The conclusion of this research thesis report is stated in Chapter 6.

1.2 Objective

In this research, samples containing mixed valence titano-manganite $La_{1-x}Ba_xMn_{0.4}Ti_{0.6}O_3$ and $La_{1-x}Ca_xMn_{0.4}Ti_{0.6}O_3$ ($0.30 \leq x \leq 0.50$) were synthesized using solid state method and physical and dielectric properties were determined. Three objectives have been highlighted and they are:

1. to fabricate $La_{1-x}Ba_xMn_{0.4}Ti_{0.6}O_3$ and $La_{1-x}Ca_xMn_{0.4}Ti_{0.6}O_3$ ($0.30 \leq x \leq 0.50$),
2. to study the effect of different concentration on the microstructure of the samples, and
3. to study the dielectric properties of the samples using the LCR meter (LF-IA) for frequency range of 0.01 Hz – 100 kHz between room temperature to 200 °C and determine the equivalent circuit modelling.
4. To determine the activation energy of the samples from the modelling data.

In order to achieve the objectives, several types of measurements were performed using SEM and XRD for microstructure, and LCR meter for dielectric properties. The dielectric response of the samples under frequency and temperature variation were discussed thoroughly.

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