THE PHYSICAL PROPERTIES AND DIRECT CURRENT CONDUCTIVITY OF TITANIUM (IV) DIOXIDE CERAMIC DOPED LITHIUM (II) NICKEL DIOXIDE

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This thesis is dedicated to my

Mum (Siti Haliyah Bt Musa), Dad (Abd Rahman Bin Abu Bakar),

my brothers (Mohd Fairuz, Mohd Fazlihisham, Mohd Firdaus and Mohd Farid),

my sisters (Noor Fizatul Akmal, Siti Faezah,

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ABSTRACT

A series of ceramic based on $x Li_2O$-(50-x)NiO–50TiO_2 ($0 \leq x \leq 50$ mol%) system have successfully been fabricated using solid state reaction method at various sintering temperature from 700°C to 1100°C. The occurrence of crystalline phase and the analysis of surface morphology are determined using X-Ray Diffraction (XRD) method and Scanning Electron Microscopy (SEM) respectively. Meanwhile, the ceramic density is determined using the Archimedes method and the hardness using Vickers's hardness test with 9.807 N of load. Brunauer-Emmett-Teller (BET) method is used to determine the porosity of the ceramic via adsorption and desorption of the nitrogen through the ceramic. The dc conductivity of the samples at room temperature is measured using four point probe method. The X-Ray Diffraction (XRD) result shows that the major phase occurrence is Li_2TiO_3 while $NiTiO_3$ and Li_2NiO_2 occur as the minor phases. The morphology of ceramic shows that the sample sintered at 1100°C exhibits the largest grain size which is due to the increasing of solid state neck formation between the grains. It is found that the density is in the range of 3.18 gcm^{-3} to 5.10 gcm^{-3}, increasing with the increasing of sintering temperature and NiO content. However, the hardness is observed in the range of 0.3707 GPa to 1.1673 GPa depending on the sintering temperature and Li_2O content. The porosity of the ceramic is found in the range 2.5250 m^2g^{-1} to 5.4405 m^2g^{-1} also depending on the sintering temperature and the contents of Li_2O. Meanwhile, the dc conductivity of the ceramic is found to be between 2.254×10^{-3} Sm^{-1} to 9.126×10^{-3} Sm^{-1}, increasing with the increase of sintering temperature and Li_2O contents.

ABSTRAK

Satu siri seramik berdasarkan sistem xLi$_2$O-(50-x)NiO–50TiO$_2$ ($0 \leq x \leq 50$ mol%) telah berjaya dihasilkan melalui kaedah tindak balas keadaan pepejal pada suhu pensinteran dalam julat 700°C ke 1100°C. Pembentukan fasa kristal dan analisis morfologi permukaan masing-masing ditentukan menggunakan pembelauan sinar-X (XRD) dan mikroskopi pengimbasan electron (SEM). Disamping itu, ketumpatan seramik diukur dengan menggunakan kaedah Archimedes dan kekerasan seramik ditentukan dengan menggunakan ujian kekerasan Vicker's menggunakan 9.807 N bebanan. Kaedah Brunauer Emmett Teller (BET) digunakan untuk menentukan keliangan seramik melalui penjerapan dan penyahjerapan gas nitrogen. Kekonduksian arus terus (AT) seramik pada suhu bilik diukur menggunakan kaedah penduga empat titik. Keputusan XRD menunjukkan pembentukan fasa utama ialah Li_2TiO_3 manakala $NiTiO_3$ dan Li_2NiO_2 sebagai fasa minor. Morfologi seramik menunjukkan bahawa seramik yang disinter pada suhu 1100°C mempunyai saiz butiran yang besar di sebabkan oleh pertumbuhan leher keadaan pepejal antara butiran. Didapati bahawa ketumpatan adalah dalam julat 3.18 gcm^{-3} hingga 5.10 gcm^{-3} bertambah dengan penambahan suhu pensinteran dan komposisi NiO. Walaubagaimanapun, kekerasan seramik didapati pada julat 0.3707 GPa sehingga 1.1673 GPa bergantung kepada suhu pensinteran dan komposisi Li_2O. Keliangan seramik ialah dalam julat 2.5250 m^{2}g^{-1} hingga 5.4405 m^{2}g^{-1} bergantung pada suhu pensinteran dan komposisi Li_2O. Manakala kekonduksian arus terus (AT) seramik bertambah dengan pertambahan suhu pensinteran dan komposisi Li_2O dalam julat 2.254x10^{-3} Sm^{-1} hingga 9.126x10^{-3} Sm^{-1}.

TABLE OF CONTENTS

LIST OF TABLES

(μm) of ceramic at difference Li_2O level.

LIST OF FIGURES

LIST OF SYMBOLS

A	-	Area of a material
Å	-	Angstroms
β	-	Full Width Half Maximum
e	-	Electron
F	-	Force
h	-	Planck constant
H_v	-	Hardness
I	-	Current
L	-	Probe distance from the edge
L_s	-	Sample length
l	-	Length of the material
n	-	Negative
p	-	Positive
P	-	Load
P'	-	Porosity
P_0	-	Partial pressure of oxygen
p	-	Partial pressure
R	-	Electrical resistance
R_s	-	Sheet resistance
R'	-	Particle size
S	-	Specific surface area
s	-	Probe spacing
s_1	-	Probe spacing between probe (1) and probe (2)
s_2	-	Probe spacing between probe (2) and probe

		(3)
s_3	-	Probe spacing between probe (3) and probe (4)
T	-	Temperature
t	-	Sample thickness
V	-	Voltage
V_{in}	-	Input voltage
V_2	-	Voltage at probe 2
V_3	-	Voltage at probe 3
V_{23}	-	Voltage between probe (2) and probe (3)
W_s	-	Sample width
w	-	Width of a material
W_1	-	Weight in air
W_2	-	Weight in toluene
x	-	Probe position from the edge of the sample
$< d >$	-	Crystallite Size
σ	-	Electrical conductivity
ρ	-	Electrical resistivity
ρ	-	Density
λ	-	Wavelength of X-Ray radiation
π	-	Pi = 3.14159
θ	-	Diffraction angle

LIST OF ABBREVIATIONS

Al_2O_3	-	Alumina
BET	-	Brunauer-Emmett-Teller
BJH	-	Barrett-Joyner-Halenda
DC	-	Direct Current
EDAX	-	Energy Dispersive X-Ray Analysis
Fe_3O_4	-	Magnetite
Li_2O	-	Lithium (II) Oxide
MRI	-	Magnetic Resonance Imaging
NiO	-	Nickel Oxide
NMR	-	Nuclear Magnetic Resonance
ReO_3	-	Rhenium Trioxide
SEM	-	Scanning Electron Microscopy
TiO	-	Titanium Oxide
TiO_2	-	Titanium (IV) Dioxide
UV	-	Ultraviolet
VO	-	Vanadium Oxide
XRD	-	X-Ray Diffraction

LIST OF APPENDICES

CHAPTER I

INTRODUCTION

1.1 Background

Ceramic can be defined as an inorganic, non-metallic solid prepared by the reaction of heat, application of pressure and subsequent cooling. Ceramic materials may have a crystalline structure, partial crystalline structure or amorphous (example: a glass). However, ceramic most commonly are crystalline; therefore the definition of ceramic is often classified to inorganic crystalline materials as opposed to the non-crystalline glasses.

Most people have the stereotypical view that metals are good conductors and ceramics are good insulators. Actually, ceramics can become excellent electrical conductor by two modes of charge transfer i.e. electrons and ions (Richerson, 2006). Throughout history, ceramics have been the material of choice because of their high temperature stability and strength. Thus, they have played an important role in the emergence of our aerospace industry (Sanders *et al.* (2000) and Naslain *et al.* (2004)) and for key component in heat engines (Katz, 1985). In addition, many applications of ceramics are based upon resistance to wear and chemical corrosion. Examples include seals and valves (Haugen *et al.* (1995), Jalili *et al.* (2003) and Sonsino, (2003)), pump parts (Tanaka *et al.* (2006) and Martínez *et al.* (2011)), bearings (Swab, (1995) and Chunfu *et al.* (2007)) and others. Based on these features, many researches projects have been conducted on ceramics (Julien, (1990), Leigh, (1990) Traversa, (1995), Whittingham *et al.* (1997), Gopukumar *et al.* (2003), Hwu *et al.* (2005) and Mazaheri *et al.* (2009)).

Over the past few decades, much attention has been given toward the development of conducting ceramic for the efficient electronic device especially Titanium dioxide (TiO_2) ceramic. TiO_2 has been well known for its wide range of applications in gas sensor, electrochemical batteries, water purification, wastewater treatment, solar cells and others (Imanaka *et al*. (1995), Anukunprasent *et al.* (2005), Hussain *et al*. (2009) and Qureshi *et al.* (2009)). The other reasons for greater attention given to TiO_2 as compared to other materials such as Zinc oxide (ZnO) and Cadmium Sulfide (CdS) are their chemical and physical stabilities are better, lower production cost and higher life time. The efficiency of TiO_2 based ceramic material mostly depends on the particle size and pore morphology of TiO_2 (Subramaniam *et al.*, 2006).

Increasing awareness of environmental factors and limited energy resources have led to a profound evolution in the way the generation and supply of energy are viewed. Although the fossil and nuclear sources remain the most important energy providers for the future, flexible technological solutions which involve alternatives means of energy supply and storage need to develop immediately. Therefore the improvement of alternative energy resource such as batteries should be more environmental friendly (Moulson and Herbert, 1990). Beside that the energy provided are very optimum. Many researches have been conducted due to this matter (Kennedy *et al.* (2000), Bernardes *et al.* (2004), Morford *et al.* (2000), Fu *et al.* (2006), Iwahori *et al.* (2000), Georén and Lindbergh, (2003), Marcos, (2007) and Fang, (2007)).

The aims of the present study are to investigate of the structural evolution, the mechanical changing and the electrical behaviour of titanium dioxide (TiO_2) when doped with lithium (II) nickel dioxide (Li_2NiO_2) ceramic.

1.2 Problem statement.

In 1973, Liang reported that the conductivity of the well-blended mixture of anhydrous LiI and active alumina (Al_2O_3) with high specific surfaces areas at operational temperature around 600°C is $5x10^{-7}$ Scm^{-1} increasing when the LiI is increased. He concluded that the conductivity depends on the existence of Lithium ion in the ceramic system.

Moreno *et al.* (1998) reported that the conductivity values of $Li_{0.25}La_{0.583}TiO_2$ are of about 8.4 x 10^{-4} Scm^{-1} at 25°C of operation temperature and of $1.74x10^{-3}$ Scm^{-1} at 50°C. On a similar work, Belous (1998) reported the conductivity is 1.85 x 10^{-4} Scm^{-1} at 125°C and 4.5 x 10^{-4} Scm^{-1}at 390°C for $Li_{0.5}La_{0.5}TiO_3$ and Leon *et al.* (1997) reported 1 x 10^{-3} Scm^{-1} at 60°C for the same composition. They concluded that the conductivity depends on the composition in the ceramic system.

Meanwhile, the conductivity of polycrystalline titanium dioxide TiO_2 (rutile) is increased from 5.50 x 10^{-3} Scm^{-1} at 1023 K to 1.22 x 10^{-1} Scm^{-1} at 1223 K corresponding to the increasing of operational temperature as reported by Kurotami *et al.* (2002). They concluded that the increasing of the mobility of the electron corresponding to the temperature of the ceramic. Evidently, research has shown that conducting ceramics exhibit high conductivity when the operational temperature of the system is above room temperature (25°C). Therefore, this ceramic are limited to be used for certain application only especially as batteries electrolyte.

The addition of additive lithium (II) oxide (Li_2O) and nickel oxide (NiO) at different sintering temperatures for preparation of TiO_2 ceramic has been used as a framework of the experiment. TiO_2 based ceramic has advantages in term of cost effectiveness, chemical stability, safety and environmental compatibility. Thus, these ceramics which are required for specific applications can be used as gas sensor, electrochemical batteries, water purification and wastewater treatment. (Hussain *et al.* 2009).

The addition of lithium (II) oxide (Li_2O) and nickel oxide (NiO) into the TiO_2 ceramic is expected to modify the chemical and physical properties of the system. Li_2O-NiO-TiO_2 ceramic is chemically and electrochemically stable to be used as the electrolyte and electrode material. These contribute to the battery performances which are the energy, power density, cycle life and safety of the batteries. The ceramic has the advantage of being strong enough to resist the high tension of battery construction. It is therefore appropriate to investigate the effect of Li_2O and NiO content to the chemical, physical and electrical properties of the Li_2O-NiO-TiO_2 ceramic.

1.3 Objectives of the research.

The objectives of this research are:

i) To prepare the Li_2O-NiO-TiO_2 ceramic at different sintering temperatures and different Li_2O and NiO compositions.

ii) To determine the crystal phase and surface analysis (morphology) of the Li_2O-NiO-TiO_2 ceramics.

iii) To determine the density and hardness of the Li_2O-NiO-TiO_2 ceramics.

iv) To determine the porosity of Li_2O-NiO-TiO_2 ceramics.

v) To determine the DC conductivity of Li_2O-NiO-TiO_2 ceramic at different compositions and sintering temperatures.

1.4 Scope of the research.

In order to achieve the above objective, the scopes of this research are:

i) Fabrication of Li_2O-NiO-TiO_2 ceramics using the solid state reaction technique for (50-x) mol % of $Li_2CO_3.10H_2O$, x mol % of $Ni(NO_3)_2.6H_2O$ and 50 mol % of TiO_2 ($0 \leq x \leq 50$ mol %). The sintering temperatures are in the range of 700°C to 1100°C.

ii) Identification of crystal phase and surface analysis (morphology) of the sample using X-Ray Diffraction (XRD) technique and Scanning Electron Microscopy (SEM) technique respectively.

iii) Measurement of density and hardness of the Li_2O-NiO-TiO_2 ceramics using Archimedes Principle and Vickers Hardness measurement technique respectively.

iv) Measurement of porosity using BET method.

v) Measurement of DC conductivity of the Li_2O-NiO-TiO_2 ceramics using the four point probe method at room temperature.

1.5 Thesis plan

This thesis is structured into several chapters which includes introduction, literature reviews, experimental procedure, results and discussion as well as conclusion.

Chapter I describes the framework of the research including the background study, problem statement, objectives and scopes of the research.

Chapter II reviews the theoretical part of the study. This would cover a general theory of ceramic especially electrical conductivity, structure of the titanium dioxide and theory related to the measurement that will be conducted.

Chapter III describes the experimental and measurement techniques which include sample preparation and the equipments used for both electrical and microstructural analysis. The parameter and physical measurement are defined.

Chapter IV reports the overall experimental results and the discussion that follows. This includes the results of Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), density, hardness, BET surface area and dc conductivity of the ceramic.

Finally, Chapter V reports the overall conclusion of the experiment that has been discussed in chapter IV; some of the suggestions for future experiments will also be inserted.

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