SYNTHESIS AND CHARACTERIZATION OF SODIUM BISMUTH TITANATE ($Na_{0.5}Bi_{0.5}TiO_3$) CERAMIC BY SOL-GEL METHOD AT DIFFERENT SINTERING TEMPERATURE

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Thesis submitted in fulfillment of the requirements
for the award of the degree of
Bachelor of Applied Science (Honor) Material Technology

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SUPERVISORS' DECLARATION

I hereby declare that I have checked the thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Bachelor of Applied Science (Honor)Material Technology.

Signature
Name of Supervisor : SITI AISAH BINTI HARUN
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STUDENT'S DECLARATION

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

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DEDICATION

Dedicated to the strength of my life especially my family and friends.Thank you my parents,who have raised me to be the person I am today.You have been with me every step of the way,through good times and bad.Thank you for all the unconditional love,guidance,and support that you give me,helping me to succeed and instilling in me the confidence that I am capable of doing anything I put my mind to.Thank you for everything.

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LIST OF SYMBOLS

~	-	approximately
λ	-	wavelength
θ	-	Bragg angle
^{o}C	-	degree celcius
g	-	grams
h	-	hour
g/cm^{3}	-	gram per centimetre cube
cm^{-1}	-	reciprocal centimeter
nm	-	nanometer
(Å)	-	constant lattice
α ,β ,γ	-	angles
kgf	-	kilogram force

LIST OF ABBREVIATIONS

PZT	-	Lead Zirconate Titanate
PMN	-	Lead Magnesium Niobate
PbO	-	Lead Oxide
BNT	-	Bismuth Sodium Titanate
SAW	-	Surface Acoustic Wave
$BaTiO_3$	-	Barium Titanate
CH_3COONa	-	Sodium Acetate
$Bi(CH_3COO)_3$	-	Bismuth (III) Acetate
$Ti(OC_4H_9)_4$	-	tetra-n-butyl titanate
XRD	-	X-ray Diffractometer
SEM	-	Scanning Electron Microscopy
FTIR	-	Fourier Transform Infrared Spectroscopy
FESEM	-	Field Emission Scanning Electron Microscopy
TGA	-	Thermogravimetric Analysis
Tc	-	Curie Temperature

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ABSTRACT

Sodium Bismuth Titanate is a prominent candidate for a lead-free piezoelectric material. In this research, $Na_{0.5}Bi_{0.5}TiO_3$ was synthesized at different sintering temperature by sol-gel method in which the solutions were prepared by using sodium acetate CH_3COONa, bismuth (III) acetate $Bi(CH_3COO)_3$, tetra-n-butyl titanate $Ti(OC_4H_9)_4$, as starting materials. Acetic acid glacial and 2-methoxyethanol as solvents and acetylacetone as a reagent to stabilize tetra-n-butyl titanate. The obtained BNT powder was analyzed using XRD, FESEM, FTIR. The mechanical properties of BNT was determined by hardness testing to investigate the strength of the samples. Pellets have been formed and sintered at 940 ^oC, 960 ^oC, 980 ^oC, 1000 ^oC for 5 hours. The XRD analysis confirmed that the perovskite structure was obtained at each temperature.The effect of slightly different sintering temperature on the structural was studied in detail. FESEM analysis of pellets showed the well-developed grains having larger size at higher sintering temperature. Hardness analysis that was examined by using Rockwell hardness testing shows that hardness measurement increased as the sintering temperature increased due to the amount of strain or defect content in the compact powders.

ABSTRAK

Natrium Bismut Titanate calon utama untuk bahan piezoelektrik bebas plumbum. Dalam kajian ini, Na0.5Bi0.5TiO3 telah disintesis pada suhu pembakaran yang berbeza dengan kaedah cecair-gel di mana penyelesaian telah disediakan dengan menggunakan natrium asetat CH3COONa, bismut (III) asetat Bi (CH3COO) 3, tetra-n-butil titanat Ti (OC4H9) 4, sebagai bahan permulaan. Asid asetik glasier dan 2-methoxyethanol sebagai pelarut dan acetylacetone sebagai reagen untuk menstabilkan tetra-n-butil titanat. Serbuk BNT yang diperolehi dianalisis menggunakan XRD, FESEM, FTIR. Sifat mekanik BNT ditentukan dengan ujian kekerasan untuk menyiasat kekuatan sampel. Pelet telah dibentuk dan dibakar pada suhu 940 ^{0}C, 96 ^{0}C, 980 ^{0}C, dan 1000 ^{o}C selama 5 jam. Analisis XRD mengesahkan bahawa struktur perovskit telah diperolehi pada setiap suhu. Kesan perbezaan suhu yang sedikit terhadap struktur sampel telah dikaji secara terperinci. Analisis FESEM pelet menunjukkan bijirin yang maju mempunyai saiz yang lebih besar pada suhu pembakaran yang lebih tinggi. Analisis kekerasan yang telah diperiksa dengan menggunakan ujian kekerasan Rockwell menunjukkan bahawa kekuatan sampel meningkat jika suhu pembakaran meningkat kerana jumlah ketegangan atau kandungan kecacatan dalam serbuk padat mempengaruhi kekuatan sampel.

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

Method for producing solid materials from little particles that contain multi-step process generally starts with the mixing of raw materials is called sol-gel process. This process is a technique that is used for creation of both ceramics or glassy materials. In ceramics processing, the irregular molecule sizes and shapes in powder often lead to non-uniform pressing morphologies that can cause packing density variations in the compaction of powder. Abandoned flocculation of powders because of the attraction of van der Waals forces also can increase the homogeneities of micro structural. The applications for sol-gel products are large. One of the application of ceramics in industry is thin films, which can be created on a piece of substrate by coating include dipping or spinning. Decorative or protective coating,and the components of electro-optic can be utilized with metal, glass and other types of substances by applying these methods. Dense ceramics or glass articles can be formed by cast into a mold, drying and heat treatment that cannot be produced by other methods. Electrophoresis, inkjet printing, spraying and roll coating are the other examples of coating methods. In this proposed research, sol gel process become the main idea because of it's advantages which are lower temperature processing, the particle size of samples is small and ability in controlling morphology in synthesizing powders. The samples can also be sintered at low temperature and the most important thing is sol-gel process shows better homogeneous final product that is formed compared to method of traditional ceramic.

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1.2 PROBLEM STATEMENT

Most of ceramic materials made from lead-bearing compounds, for examples lead magnesium niobate (PMN) and lead zirconate titanate (PZT) (Aksel & Jones 2010). These compounds can effect environmental, health and social reasons. PZT based ceramics are environmentally burdened materials because of volatilization of toxic of lead oxide (PbO) during sintering at high temperature (Kim et al. 2003). Therefore researches now are in search to find good and environmentally friendly electroceramics with efficient ferroelectric properties that contain lead-free to reduce and ultimately eliminate the lead content of the materials.. BNT are expected to replace PZT among the lead-free piezoelectric ceramics due to the growing concern with environmental pollution. BNT also has received more attention compared to $BaTiO_3$ because $BaTiO_3$ has a relatively high sintering temperature during processing (Badapanda et al. 2013).

1.3 OBJECTIVES OF STUDY

Objectives of this research are:

1. To synthesis sodium bismuth titanate ceramics by using sol gel method at different sintering temperature.
2. To investigate the effect of temperature to the structural and mechanical properties of sodium bismuth titanate ceramics.

1.4 SCOPE OF STUDY

In this research, Sodium Bismuth Titanate (BNT) was synthesized and characterized by using the sol-gel technique starting with mixing the raw materials which were Sodium Acetate, Bismuth(III) acetate and Titanium(IV) butoxide. The mixture was then sintered at different sintering temperature which were 940 ^{0}C, 960 ^{0}C, 980 ^{0}C, and 1000 ^{0}C for five hours. The BNT ceramis were characterized by using X-Ray Diffractometer (XRD). The sintered pellets were observed under Field Emission Scanning Electron Microscopy Analysis Technique (FESEM) to measure the grain size

of the BNT. The samples also was characterized by FTIR to determine the functional group and the hardness testing to test the strength of ceramic

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