MICROSTRUCTURAL STUDIES OF STRONTIUM TITANATE CERAMIC PRESSED AT VARYING PRESSURE

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MICROSTRUCTURAL STUDIES OF STRONTIUM TITANATE CERAMIC PRESSED AT VARYING PRESSURE

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A dissertation submitted in fulfilment of the
requirements for the award of the degree of
Master of Science (Physics)

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JUNE 2014

I declare that this dissertation entitled "*Microstructural Studies of Strontium Titanate Ceramic Pressed at Varying Pressure*" is the result of my own research except as cited in references. The dissertation has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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This dissertation is dedicated to

my parents, my beloved husband (Akhmal Annas) and my dearest son (Amir Alhakim).

Thank you for being with me all along.

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ABSTRACT

The purpose of this study is to fabricate the Strontium Titanate ($SrTiO_3$) ceramics by using the High Energy Ball Milling Method (HEBM) for 9 hours at varying pressure between 60 MPa to 160 MPa at an interval 20 MPa. The samples were sintered at 1100 °C. The microstructures and morphology the samples were investigated by X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) respectively. Meanwhile, the densities and porosities of the ceramics were determined via Archimedes' method. The smallest crystallite size, 40.8 nm at 140 MPa and particle size, 0.58 μm were found at the pressure 160 MPa this is due to the decreased of large voids by reorganization of granules. The maximum density of the samples was found to be 4.99 gcm^{-3} at 140 MPa while the porosity was 22.35 % at 60 MPa.

ABSTRAK

Tujuan kajian ini adalah untuk membina seramik Strontium Titanate ($SrTiO_3$) dengan menggunakan proses *High Energy Ball Milling* (*HEBM*) selama 9 jam pada tekanan yang berbeza iaitu di antara 60 MPa hingga 160 MPa dengan peningkatan sebanyak 20 MPa. Semua sampel telah disinter pada suhu yang sama iaitu 1100°C. Mikrostruktur dan morfologi sampel telah dikaji dengan menggunakan analisis *X-Ray Diffraction* (XRD) and *Scanning Electron Microscopy* (SEM). Sementara itu, ketumpatan dan keporosan daripada seramik ditentukan melalui kaedah Archimedes. Saiz kristallit yang paling kecil, 40.8 nm pada 140 Mpa dan zarah yang paling kecil, 0.58 μm pada tekanan 140 MPa ini berlaku akibat penyusunan semula granul disebabkan tekanan yang dikenakan. Ketumpatan yang tinggi adalah 4.99 gcm^{-3} di 140 MPa dan peratusan maksimum keporosan yang tertinggi adalah pada 60 MPa iaitu sebanyak 22.35%.

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LIST OF ABBREVIATIONS

$SrTiO_3$	-	Strontium Titanate
HEBM	-	High Energy Ball Milling
FWHM	-	Full Wave Half Maximum
XRD	-	X-Ray Diffraction
SEM	-	Scanning Electron Microscopy

LIST OF SYMBOLS

$\%$	-	percentages
ε'	-	dielectric constant
ε''	-	dielectric loss
$<$	-	less than
$^{\circ}C$	-	degree Celsius
B	-	peak width
L	-	crystallite size
K	-	Scherrer constant
λ	-	lambda
θ	-	theta
ρ	-	density
W_1	-	weighed in air
W_2	-	weighed in toluene
$\rho_{toluene}$	-	density of toluene
p'	-	porosity
V_b	-	bulk volume
$S1$	-	sample 1 (60MPa)
$S2$	-	sample 2 (80MPa)
$S3$	-	sample 3 (100MPa)
$S4$	-	sample 4 (120MPa)
$S5$	-	sample 5 (140MPa)
$S6$	-	sample 6 (160MPa)
P	-	pressure
F	-	pressing force

A	-	Surface area

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CHAPTER 1

INTRODUCTION

1.1 Introduction

The term ceramics are often assumed to be nonmagnetic and different from metals where metals are ductile. Kingery *et al* (1976), defined ceramic as the art and science of making and using solid articles which have their essential component, and are composed in large part of inorganic non-metallic materials. This definition also applies to non-metallic magnetic materials, ferroelectrics, single crystals and glass ceramic besides of pottery, porcelain, refractories, clay products and cement.

Early in civilization, ceramics have been used and have its own roots in traditional aspects such as clay based ceramics and glasses. The widespread use of ceramics has led to a variety approaches to the subject. During the past few decades, ceramics uses in more advanced technological applications have been expanding. This phenomenon resulted in heightened demand for improvements in properties and reliability. Rahaman (2003), states that these improvements can only be achieved only through careful attention to the fabrication process. Since, the fabrication processes govern the microstructures manufacture with the desired properties.

Hence, ceramics processing approaches are alarmed with the understanding of fundamental issues and the application of the knowledge to the invention of microstructures that have functional properties.

This research is concerned primarily on the Strontium Titanate ceramics processing and the microstructures properties. Ceramics fabrication is a considerable attention since the route of processes will affect the properties of ceramics.

1.2 Background of Research

The size of particle gets into concern because the properties of the materials have change when the size changes, such as the constant lattice, chemical composition and topography. Particle size distribution is important since a controlled optimum particle size distribution is required to achieve maximum reproducible strength. By refer to York (1978), the degree of rearrangement is directly related to the fragility of the structure formed upon pouring, which is dependent on particle size, shape and surface texture, but there are many appliances where these criteria is not crucial. Refractories are a good example where most of them contain either large particles or high porosity (less dense) as the principal constituent in achieving the desired properties. The desired particle size distribution usually cannot be achieved simply by screening, classifying, or elutriating the raw materials. Hence, particle size reduction (comminution) step is required. The consequences of improper size analyses are reflected in poor product quality, high rejection rates and economic losses (Jillavenkatesa *et al.*, 2001).

Yet, particle size analysis techniques are often applied inappropriately, primarily due to a lack of understanding of the underlying principles of size analysis, or due to confusion arising from claims of the analytical ability of size determination techniques and instruments. In accordance with Rahaman (2003), to improve the properties and reliability the fabrication process must do with full of attention and care. Knowledge of crystallite sizes and particle sizes of a powder is a prerequisite for most production and processing operations. In addition, regarding to Prasad, *et al* (2010) the size of crystallite and particle size in a smaller scale promote the sintering at a low temperature and give a fully dense material.

The High Energy Ball Milling (HEBM) is the method to reduce the particle size. It produces a broad particle size distribution rather than a narrow particle size range and a very active powder that is easier to dense in later process steps. Contamination is a snag in milling. As the particle size is being decreased, the mill walls and media are also wearing. According to Nurhashimah (2012), 0.1% contamination per hour was reported when milling Al_2O_3 powder with porcelain or SiO_2 media while porcelain cylinders picked up nearly 6% contamination in 72 hours of milling Si_3N_4 powder in a porcelain-lined. In order to control the contamination, mill lining and the media must be selected carefully. Besides that, a very hard grinding media can also reduce the contamination because they become more slowly (Richerson, 1982).

Selecting raw materials are essential to form ceramics. In this research, $SrTiO_3$ manufactured powders were selected as investigated material. Owing to the high melting points of 2060^0C, the fabrication of ceramics includes a heat treatment (sintering) step in which the powders were formed into required shape is converted into solid. Sintering has its origins in the early civilization together with the development of ceramics. Nowadays, the technologies are established that can widespread use of ceramics productions.

Consequently, different ceramics generally behave in the same way at room temperature. The pressure initially causes elastic deformation, this is a reversible change in shape and the sample recovers its original form when the pressure is reduced. In this research, the parameter such porosity, density and size of particle will examine due to the pressing pressure. However, in such a case the reliability of the absolute measurement can be affected by the number of particles that are counted, the representative nature of the particles included in the analysis, the shape of the particles, the state of dispersion and the sample preparation technique followed. Herein, if the pressure is increased further, the ceramics suddenly shatter into the finest splinters (Brunner.D, 2003).

Atomic structure, fabrication, microstructure, and properties of polycrystalline ceramics are related to each other. Fabrication process is responsible for the microstructures production in order to meet the application's needs. Information obtained from structural and characterization of surface material can help researcher to find the best use of the material in the industry. For this purpose, the researcher need to investigate which technique is suitable and excellent to use for measurement and characterization. In addition, to get a good result, researcher should handle the investigation procedures with care.

1.3 Problem Statement

Based on the previous study, determination of crystallite size and particle size of powders is a critical step in almost all ceramic processing techniques. Particle size has a significant effect on the mechanical strength, density, electrical and thermal properties of the finished object. In this research, the pressing pressure being analyses to identify whether the pressure affect the size of crystallite sizes and particle sizes. The density and porosity also play an important role in ceramic processing. A high density of sample is important as it leads to a desirable microstructure consisting of a large number of small crystals. Due to its high specific surface area, small crystallite and small particle sizes powder has high sinterability, allowing lower temperature manufacture of high density or small grain size ceramic pieces with improved mechanical properties.

1.4 Research Objectives

Objectives are very important for a research as guidance for researcher to plan stages of processing. Therefore, it is important to investigate the following objectives:

1. To fabricate the $SrTiO_3$ ceramic pressed at varying pressure.
2. To determine the microstructures of the ceramic powders using X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM).
3. To determine the porosity of the $SrTiO_3$ ceramic using Archimedes' method.
4. To determine the particle size of the ceramic powders using X-Ray Diffraction (XRD).

1.5 Research Significant

This research of structural measurement of ceramic will give valuable information about the ceramic. Consequently, this research is conducted in order to gain structural measurement of $SrTiO_3$ that gives precious information about the ceramic itself. By this, students, ceramists, and researchers can refer to the information to understand better on the $SrTiO_3$ behaviors and afterwards they can identify and fabricate ceramic. In addition, this research also gives valuable information for industry as we know that the ceramic use is spreading everyday and the technology are useable in the manufacturing, designing, and fabricating new technologies to apply in human needs.

For instant, gas sensor becomes important tool since there are such chemical factories that used it. Therefore, to improve the quality of the ceramic material, we need to examine how to make improvements to the process of fabrication done on ceramics.

Ceramic uses are widely spread around the world gives arise in advanced ceramics applications. Due to this factor, ceramists and researchers attempt to produce ceramics appliances. To design and fabricate good appliances, improvement such as new findings of materials and technology are required. Therefore, appliances from $SrTiO_3$ are hopefully can gives benefits for everyone since it was discovered to have potential for many applications.

1.6 Research Scope

The scope of the research is mainly on $SrTiO_3$ ceramic samples that will be prepared by the high energy milling machine for 9 hours at $1100^{o}C$ sintering temperature. After that, the samples were pressed at six different pressures. All the samples will investigate by SEM and XRD in order to identify the morphology of the samples. Investigation of structural characteristic of samples will be done in laboratory at Fakulti Mekanikal in Universiti Teknologi Malaysia.

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