SYNTHESES AND CHARACTERIZATIONS OF CULLET-CLAY-TITANIUM NITRIDE CERAMICS

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DEDICATION

Dedicated to my mother and father who taught me that the best kind of knowledge is to have learned for its own sake. Also dedicated to my beloved husband, who taught me that even the largest task can be accomplished and for always been support by my side. To me, to prove you are one of a kind.

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ABSTRACT

A series of ceramic based on composition of (60-x) cullet-40 clay-(x) TiN (0 ≤ x ≤ 6 mol %) has successfully been made by dry pressing technique followed by sintering at 1000 ^{o}C. The occurrence of crystalline phase is determined using X-Ray Diffraction (XRD) technique. The actual composition of ceramic is analyzed using Energy Dispersive of X-Ray Analysis (EDAX). Their surface morphology is observed under Field Emission Scanning Electron Microscope (FESEM) and the existence of molecular vibrational structure has been determined using Fourier Transform Infrared Spectroscopy (FTIR). Meanwhile, the physical properties are measured in terms of their density by using Archimedes method and porosity by using Brunauer-Emmett-Teller (BET) method. The mechanical properties are determined by means of Young's Modulus and Vickers Hardne ceramics hardness, respectively. The corrosion rate of the ceramics is characterized by using pH level technique. The DC conductivity of the samples at room temperature is measured using four-point probe method. It is found that the ceramic contain mostly of Silica and the phases are dominated by two major phases namely quartz and tridymite with TiN as minor phase. It is also found that the structural morphology is in the form of dendritic growth over the ceramic matrix. Several broad absorption peaks are observed using FTIR and it is found that the ceramic is dominated by the existence of Si-O peak around 1080 cm^{-1}. It is found that the density of the ceramic samples is in range of 1.941-3.019 gcm^{-3} while the porosity is in the range of 10.43-14.59 m^{2}g^{-1} and analysis shows that samples with higher TiN content exhibit optimum value. The mechanical strength is observed in the range of 195.4-522.6 MPa and the hardness is in the range of 113-181 GPa, depending on composition. The corrosion rate of the samples is found to increase with increasing pH level and the reaction mostly occurred after 10 days of test. The DC conductivity is in the order of 10^{-3} Sm^{-1}, which generally indicates that this material is semiconductor in nature.

ABSTRAK

Satu siri seramik berdasarkan komposisi (60-*x*) kaca cullet-40 tanah liat Kaolin-(*x*) TiN ($0 \le x \le 60$ mol %) telah berjaya dihasilkan dengan kering diikuti dengan persinteran pada suhu 1000°C. Kewujudan fasa kristal ditentukan dengan menggunakan teknik Pembelauan Sinar-X (XRD). Kandungan komposisi sebenar seramik dianalisis menggunakan Analisis Penyebaran Tenaga Sinar-X (EDAX). Morfologi permukaan pula diperhatikan di bawah Mikroskop Elektron Pengimbas Kesan Medan (FESEM) dan keberadaan struktur getaran molekul telah ditentukan menggunakan Spektroskopi Inframerah Transformasi Fourier (FTIR). Sementara itu, sifat fizikal diukur dari segi ketumpatannya menggunakan kaedah Archimedes dan sifat porositi dengan menggunakan kaedah Brunauer-Emmett-Teller (BET). Sifat mekanikal seramik telah ditentukan dengan menggunakan ujian Modulus Young dan ujian kekerasan Vickers masing-masing untuk kekuatan seramik dan kekerasan seramik. Kadar kakisan seramik dicirikan dengan menggunakan teknik aras pH. Kekonduksian AT sampel diukur pada suhu bilik menggunakan kaedah penduga empat titik. Didapati bahawa sampel seramik kebanyakkannya mengandungi Silika dan didominasikan oleh dua fasa utama iaitu Kuartz dan Tridymite dengan TiN sebagai fasa minor. Didapati juga struktur morfologi adalah dalam bentuk pertumbuhan dendrit pada matriks seramik. Beberapa puncak penyerapan yang lebar telah dicerap menggunakan FTIR dan didapati bahawa seramik didominasi oleh kewujudan puncak Si-O disekitar 1080cm^{-1}. Didapati bahawa ketumpatan seramik adalah dalam julat 1.941-3.019 gcm^{-3}, sementara keporosan berada dalam julat 10.43-14.59 m^{2}g^{-1} ini menunjukkan analisis sampel dengan kandungan TiN yang tinggi menghasilkan nilai yang optimum. Kekuatan seramik berada dalam julat 195.4-522.6 MPa dan kekerasan dalam julat 119.7-180.8 GPa, bergantung kepada komposisi. Kadar kakisan sampel pula meningkat dengan peningkatan pH dan tindak balas didapati berlaku selepas 10 hari ujian dijalankan. Kekonduksian AT didapati dalam julat x10^{-3} Sm^{-1}, dan ini secara amnya menunjukkan bahawa seramik ini adalah bersifat semikonduktor.

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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
FESEM	-	Field Emission Scanning Electron Microscopy
FTIR	-	Fourier Transform Infrared Spectroscopy
Li_2O	-	Lithium (II) Oxide
MRI	-	Magnetic Resonance Imaging
NiO	-	Nickel Oxide
NMR	-	Nuclear Magnetic Resonance
SEM	-	Scanning Electron Microscopy
TiN	-	Titanium Nitride
TiO	-	Titanium Oxide
TiO_2	-	Titanium (IV) Dioxide
UV	-	Ultraviolet
XRD	-	X-Ray Diffraction

LIST OF SYMBOLS

A	-	Area of a material
$\AA$	-	Angstroms
β	-	Full Width Half Maximum
e	-	Electron
F	-	Force
h	-	Planck constant
H_v	-	Hardness
I	-	Current
L	-	Probe distance from the edge
Ls	-	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
Rs	-	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)
Ws	-	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

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CHAPTER 1

INTRODUCTION

1.1 Problem Background

Ceramic industries is one of the compatible industries that can be generated to promote conservation of solid waste as it comprises a variety of compounds and could be prepared through various methods (Junkes et al., 2011). There are many types of waste such as cullet glass, incinerated ash, sludge ash, dust wood, and others that had potential used in manufacturing of ceramic products. The advantages of using waste in ceramic processing are the ability of the ceramic to be radical with variation of waste composition. In addition, the mechanical and chemical properties of the ceramic from waste material are comparable with the commercial products (Isa, 2011).

There are a huge concern regarding to the increasing amount of the industrial wastes such as plastic, glasses, and grogs. Regarding to Global Environment Centre (GEC), currently over 23 000 tonnes of waste produced each day in Malaysia. The amount of waste produced continues to increase due to the increasing population and development. Additionally, the huge increment of solid waste in Malaysia is due to exponential growth rate of population, accelerated urbanization and development of industry and technology (Zahari et al. 2010). The total of solid waste generated in Malaysia is estimated about 9.0 million tonnes per year and would be increasing up to 15.6 million tonnes per year by 2020 (Agamuthu et al. 2011). As reported, the disposal of these wastes is some of the issues that have received a lot of attention and a high demand for the safety of the environment (Loryuenyong et al. 2009).

Recycling is a one technique used to reduce such wastes which is not only benefits to the environment but also to the economy. Furthermore, in Malaysia only less than 5% of the waste is being recycled. Therefore, to reducing waste disposal, the invention of new materials from recycle waste is very important since it can convert the wasted materials to valuable products. Generally, ceramic materials are brittle, hard, strong in compression, and weak in shearing and tension. The properties of any ceramics substances are based on its crystalline structure and chemical composition. The demanding uses of ceramics on advanced technological applications introduce high improvement in the based on requirement of ceramics.

Ceramic are crystalline and therefore the definition of ceramic is often classified to inorganic crystalline materials as opposed to the non-crystalline glasses. Ceramics are generally made by taking mixtures of clay, earthen elements, powders and water then shaping them into desired forms. They are normally crystalline in nature and a compound formed among metallic and non-metallic elements such as titanium and oxygen (TiO_2), aluminium and oxygen (alumina-Al_2O_3), calcium and oxygen (calcia-CaO) and carbon and nitrogen (carbon nitride- C_3N_4), etc.

Sand is natural resources or an earthen element which highly contained with 70%-90% of silica (Safwan, 2011). If all glass industries are simultaneously using this material, the environment could be harm. One way to avoid from vanishing the environment are by recycling. Cullet waste glass which means the broken bottle is the component of the municipal solid waste material. Cullet is used in glass container manufacturing industry since the earliest days of glass making for the purpose of assisting melting and saving raw material consumption (Owen-Broadway, 1994). Additionally, it has been stated that for every 60% of cullet used, 30% of fuel energy may be saved (Sahar et al., 2003).

Furthermore, cullet is used in many kinds of manufacturing activities for example glass reforming or melting additives for ceramics as it has low softening temperature. Several reasons that cullet has to be recycled are most importantly are the main component for glass production is silica. New ceramic materials can be producing by adding a recycle glass which is cullet as reported by researcher (Luz, 2007).

Throughout history, ceramic have been the material of choice because of their high temperature stability and strength as they had played an important role in the emergence of aerospace industry (Sanders et al., 2000; Naslain et al., 2004) and also for key component in heat engine (Katz, 1985). Moreover, regular application of ceramic is based upon resistance to wear and chemical corrosion for example include seal and valves (Haugen et al., 1995; Jalili et al., 2003), in pumping parts (Tanka et al., 2006; Martinez et al., 2011), bearings element (Swab, 1995; Chunfu et al., 2007) and others application. Regarding on these features, numerous researcher projects have been conducted on ceramics (Julien, 1990; Traversa, 1995; Whittingham *et al.*, 1997; Gopukumar *et al.*, 2003; Hwu *et al.*, 2005; Mazaheri *et al.*, 2009; Sharifah, 2011).

Typically, people viewpoint that metals are the element which is good conductors while ceramic are good insulators. Indeed, ceramics can become excellent electrical conductor by two modes of charge transfer like in electrons and ions (Richerson, 2006). There are existent ceramic materials with improved properties such as high in strength, ductility, heat resistance, corrosion resistance, wear resistance and others (Kuwahara et al., 2001). Many research growths for a new material and for surface modification process are actively carried out especially in ceramics field which focused on oxide ceramics, though there are many useful nitride ceramics for example TiN, Si_3N_4, AlN, cubic-BN and C_3N_4 (Kuwahara et al., 2001).

Titanium nitride (TiN) is a ceramic material which is a crystalline inorganic compound. From titanium-nitrogen compound, it is forming a simple cubic crystalline structure. TiN has identified with its high hardness, high wear resistance, high corrosion resistance and high specific strength (Kuwahara et al., 2001). As due to their unique properties, it is considerable material that high interest in both materials science and industrial applications. Additionally, TiN has good intrinsic properties such as high hardness of about 18-21GPa (Holleck *et al.*, 1995; Lengauer *et al.*, 1995), and also high electronic conductivity and low coefficient of friction

(Pivkina, 1996). These properties appear characteristic for covalently bonded materials and simultaneously appear good thermal and electrical conductivities as known otherwise from metals (Kotz et al., 2014). Though, TiN is limited to use as thin film layer for all purposes and there is little study to create TiN as mixing or doped agent in ceramic compound.

The purposes of the present study are focused on to create new ceramic compound. In this research, kaolin clay powder ($Al_2O_3.2SiO_2.2H2O$) as the starting material. The chemical reaction during sintering of kaolin is very complicated and depending on the composition of the raw materials, characteristics of the powder and fabrication condition (Safwan, 2011). Use of kaolin powder that consist of small amount of quartz and by adding cullet which act as fluxing agent (Braganca and Bregmann, 2005) also adding titanium nitride to increase the properties in term of the strength (Sahar et al., 2003) which is a new ceramic can be formed.

1.2 Problem Statement

Study of cullet-clay and cullet-incinerated paper are very important issues to be addressed especially in term of ceramic production point of view (John *et al.*, 1996; Liaw *et al.*, 1998; Tuccia *et al.*, 2004; Toya *et al.*, 2006; Maschio *et al. 2009;* Furlani et al. *2011;* Sahar *et al.*, 2011; Samah *et al.*, 2016). Titanium nitride has long been known to be used to harden the materials (Boljanovic, 2009). The combination of both materials wills hopefully producing a new ceramic material. Unfortunately, up to now there are lack of report on development of cullet-clay embedded with titanium nitride which is can be useful for industrial application such as in semiconductor which is growing rapidly. This research is intended to seek a new material with a better physical performance compare to other ceramic materials. Additionally, this research is expected to enhance the activity of recycling the waste glass and producing a new ceramic product.

1.3 Research Objectives

The objectives of the research are:

i. To synthesize the cullet-clay-titanium nitride ceramic by dry pressing technique.

ii. To identify the crystal phase and surface morphology of the ceramic sample as a function of TiN concentration.

iii. To determine the physical, mechanical and corrosion properties of the prepared ceramic sample.

iv. To determine the Nitride concentration dependent in DC conductivity of the produced ceramic sample.

1.4 Scope of Research

In order to achieve the above objective, the scope of the research is:

i. Preparation of cullet-clay-TiN ceramic using dry pressing technique with composition of (60-x) cullet – 40 clay – (x) TiN, where 0 ≤ x ≤ sintering temperature is 1000 oC.

ii. Identification of crystal phase and surface morphology of the sample using X-Ray Diffraction (XRD), Field Emission Scanning Electron Microscopy (FESEM) and Fourier Transform Infrared Spectroscopy (FTIR) technique respectively.

iii. Measurement of density and porosity of the ceramic samples using Archimedes Principle technique and BET method respectively. Measurement of strength by using Young's Modulus te dness by using Vickers Hardness technique. Characterization of the corrosion rate of the sample using pH level.

iv. Measurement of Direct Current (DC) conductivity of the ceramic sample using the four-point probe method at room temperature.

1.5 Research Significance

This research will produce a new ceramic material which is cullet-clay doped with titanium nitride. From this point, this research is intended towards the performance and improvement of this new ceramic material. Therefore, the result will give the information needed to the useful application.

Moreover, this research will also give alternative ways of converting some waste product into marketable and environmentally friendly materials. Indirectly, it will create awareness among the society to preserve the natural resources for development green technology.

1.6 Thesis Outline

This thesis is structured into several chapters which includes introduction, literature reviews, experimental procedure, result and discussion including conclusion.

In chapter 1 describes the framework of the research including the background of research, problem statement, objective, scopes of the research and research significance.

In chapter 2, reviews about the theoretical part of the study. This would cover a general knowledge and theory of ceramic especially the element of the ceramic contains which consists of kaolin clay, cullet and titanium nitride also the theory related to the measurement that will be conducted.

In chapter 3 illustrates the experimental and measurement technique that include preparation of the sample and the equipment used for the analysis. The parameter and physical measurement are defined.

Chapter 4 results the overall experiment results and the discussion. This include the results of identification of the crystal phase and surface morphology, characterization of the physical and mechanical properties and also dc conductivity of the ceramic.

Finally, in chapter 5, deliberation of conclusion of the experiment has been discussed and some suggestions for future experiments also been inserted.

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LIST OF PUBLICATIONS

Synthesis and Characterization of Cullet-Clay- Titanium NitrideCeramic

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