UNIVERSITI PUTRA MALAYSIA

MICROSTRUCTURAL AND ELECTRICAL BEHAVIOR OF RECYCLED SODA LIME SILICA GLASS DOPED ZNO-COO BASED VARISTOR CERAMICS

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By

NUR QURATUL AINI BINTI ISMAIL

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

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September 2020

Chair : Nor Kamilah Sa'at, PhD
Faculty : Science

The need for green dopant to fabricate a reliable electronic component to protect electrical circuits from overvoltage has gained considerable attention in varistor ceramics field. It is worth to reveal a new potential of recycle Soda Lime Silica (SLS) glass as a dopant electrical enhancer in Cobalt oxide doped Zinc oxide based varistor ceramics. The objectives of the study are; to synthesis ZnO-CoO varistor ceramics at different SLS glass concentration from 0.5 to 2.0 mol% by solid state method sintered at 1100 ˚C for 2 hours; secondly to study the effect of varied SLS glass concentration using different milling method (wet and dry); and thirdly to study the effect of sintering time varies from 60 to 180 minutes on microstructure and electrical properties of ceramics by dry milling method. The raw materials were weighed to their mol percentage and then subjected to a 24 h ball milled process, powder pre-sintered at 800 ˚C for 2 h, pressed by 4 tonne pressure to produce pellet form of 10 mm diameter with 1 mm thickness and sintered. The characterization is divided into structural, morphology and electrical measurement. The structural and morphology of varistor ceramics samples were examined with X-ray diffraction (XRD) and Scanning electron microscopy (SEM) with Energy dispersive X-Ray (EDX). The XRD analysis shows the presence of main phase of ZnO on (0 0 2) plane and secondary phases of Zn_2SiO_4 as reaction between SiO_2 in SLS glass with ZnO. The SEM with EDX results shows the microstructure well-formed grains and its boundaries and the presence of all the elements used. It was found that Zn_2SiO_4 inhibited the grain growth of the ZnO sample thus increase the number of grain boundaries. The density also increases with the increase of doping concentration. This due to the heavier atomic mass of ZnO (65.390) compared to other atomic mass of Si (28.086), Ca (40.078) and Na (22.989). Dry milling shows a good microstructure as the average grain size (*D*) decrease from 26.12

to 21.60 µm compared to wet milling as the *D* decrease from 25.01 to 22.46 µm. Moreover, dry milling also exhibits good electrical properties as the nonlinear coefficient α increase from 5.52 to 6.97 compared to wet milling as the nonlinear coefficient increase from 2.82 to 4.38 as increases the SLS glass concentration. Besides, the knee of the *J-E* curve of dry milling shifted to higher electric field indicating the breakdown voltage (E_b) increase from 120.4 to 261.1 V/cm attributed the decrease of leakage current (J_L) from 5.40 to 4.87 µA/cm^2. Further study of 2.0 mol% SLS glass doped ZnO-CoO based varistor ceramics was prolonged the sintering time. Samples sintered at 120 minutes shows the smallest grain size of 21.60 µm and largest grain size of 28.80 µm sintered at 180 minutes. Meanwhile, samples sintered at 150 minutes exhibited excellent nonlinear electrical properties since it has high nonlinear coefficient, α = 7.36, lowest in J_L = 4.32 µA/cm^2 and high in E_b = 278.6 V/cm. In a conclusion, the growth of Zn_2SiO_4 phase in 2.0 mol% SLS glass doped ZnO-CoO based varistor ceramics by dry milling through solid state method sintered at 1100 ˚C for 150 minutes produced the best microstructure with grain size of 22.92 µm, and density of 5.45 g/cm^3 and the best nonlinear electrical properties with high α = 7.36, lowest J_L= 4.32 µA/cm^2 and high E_b = 278.6 V/cm.

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

PERILAKU MIKROSTRUKTUR DAN ELEKTRIKAL KACA SODA LIME SILIKA TERPAKAI DOP ZnO-CoO BERASASKAN SERAMIK VARISTOR

Oleh

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Keperluan untuk dopan hijau untuk menghasilkan komponen elektronik yang boleh dipercayai untuk melindungi litar elektrik daripada arus berlebihan telah mendapat perhatian yang cukup dalam medan seramik varistor. Adalah penting untuk mendedahkan potensi baru kaca terpakai Soda Lime Silika (SLS) sebagai penguat elektrik dopan dalam kobalt oksida yang dop berdasarkan zink oksida seramik varistor. Objektif kajian ini adalah; untuk sintesis ZnO-CoO seramik varistor pada kaca SLS yang berbeza kepekatan dari 0.5 hingga 2.0 mol% dengan kaedah keadaan pepejal yang disinter pada 1100 ˚C selama 2 jam; kedua untuk mengkaji kesan kepekatan kaca SLS yang berbagai dengan menggunakan kaedah milling yang berbeza (basah dan kering); dan ketiga untuk mengkaji kesan masa sintering yang berlainan dari 60 hingga 180 minit pada struktur mikro dan sifat elektrik seramik oleh milling kering. Bahan bahan ditimbang dengan peratusan mol mereka dan kemudian menjalani proses milling selama 24 jam, serbuk di pre-sinter pada 800 ˚C selama 2 jam, dengan tekanan 4 tan untuk menghasilkan bentuk pelet ukur lilit 10 mm dengan ketebalan 1 mm dan sintered. Pencirian dibahagikan kepada struktural morfologi dan pengukuran elektrik. Struktural dan morfologi sampel seramik varistor diselidiki melalui difraksi sinar X (X-Ray) dan mikroskop elektron pengimbasan (SEM) dengan penyebaran tenaga X-Ray (EDX). Analisis XRD menunjukkan kehadiran fasa utama ZnO pada pelan (002) dan fasa kedua Zn_2SiO_4 sebagai tindak balas antara SiO_2 dalam kaca SLS dengan ZnO. Keputusan SEM dan EDX menunjukkan biji biji mikro terbentuk dengan baik bersama sempadannya dan kehadiran semua elemen yang digunakan. Didapati bahawa Zn_2SiO_4 menghalang pertumbuhan bijian sampel ZnO lalu ia meningkatkan bilangan sempadan bijian. Kajian menunjukkan peningkatan ketumpatan dengan peningkatan kepekatan doping. Ini adalah disebabkan oleh jisim atom ZnO yang lebih berat (65.390) berbanding jisim atom yang lain seperti Si (28.086), Ca

(40.078) dan Na (22.989). Milling kering menunjukkan struktur mikro yang baik kerana purata saiz butiran (*D*) berkurang dari 26.12 kepada 21.60 µm berbanding milling basah apabila *D* berkurang dari 25.01 kepada 22.46 µm. Malahan, milling kering juga mempamerkan sifat elektrik yang baik kerana pekali taklinear α meningkat daripada 5.52 sehingga 6.97 berbanding milling basah kerana pekali taklinear meningkat dari 2.82 sehingga 4.38 apabila kepekatan kaca SLS meningkat. Selain itu, lengkung *J-E* milling kering beralih ke medan elektrik yang lebih tinggi yang menunjukkan peningkatan voltan pecahan (E_b) daripada 120.3 sehingga 261.1 V/cm disebabkan penurunan kebocoran arus (J_L) dari 5.40 sehingga 4.87 µA/cm^2. Kajian lebih lanjut mengenai 2.0 mol% kaca SLS yang didop seramik varistor berasaskan ZnO-CoO telah memanjangkan masa sintering. Sampel yang disinter pada 120 minit menunjukkan saiz bijian terkecil 21.60 µm dan saiz bijian terbesar 28.80 µm disinter pada 180 minit. Sementara itu, sampel yang disinter pada 150 minit mempamerkan sifat elektrik taklinear yang cemerlang kerana ia mempunyai pekali taklinear yang tinggi α = 7.36, J_L = 4.32 µA /cm^2 yang paling rendah dan E_b = 278.6 V/cm yang tinggi. Kesimpulannya, pertumbuhan fasa Zn_2SiO_4 dalam 2.0 mol% kaca SLS didop seramik varistor berasaskan ZnO-CoO berdasarkan milling kering melalui kaedah pepejal yang disinter pada 1100 ˚C selama 150 minit menghasilkan struktur mikro yang terbaik dengan saiz butiran 22.92 µm, dengan ketumpatan 5.45 g/cm^3 dan sifat elektrik taklinear terbaik α = 7.36 yang tinggi, J_L= 4.32 µA/cm^2 terendah dan E_b = 278.6 V/cm tertinggi.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

UPM	Universiti Putra Malaysia
VDR	voltage dependent resistor
I-V	current-voltage
SiC	silicon carbide
MOV	metal oxide varistor
ZnO	zinc oxide
CoO	cobalt oxide
SiO2	silicon oxide
SLS	soda lime silica
Zn2SiO4	willemite
DSB	double schottky barrier
FWHM	full widths half maximum
PVA	polyvinyl alcohol
XRD	x-ray diffraction
SEM	scanning electron microscopy
EDX	energy dispersive X-ray
α	nonlinear coefficient
Eb	breakdown voltage
JL	leakage current
ΦB	barrier height
D	average grain size
pavg	average density
J-E	current density-electric field

CHAPTER 1

INTRODUCTION

1.1 Varistor

Varistors are electronic components that are used for electrical and electronic circuit protection. Varistor has a non-linear or non-ohmic current-voltage (I-V) characteristic which alike to diode. The nonlinearity of current-voltage makes the varistor suitable to provide protection to electrical and electronic circuits. The word varistor come from the combination of word variable and resistor which describe how it works in a circuit. The resistance of variable resistor can be manually varied among its average values, while the varistor will change its resistance value depending on the voltage across it making it a voltage-dependent resistor.

There are 2 varistor types which are silicon carbide (SiC) varistor and metal oxide varistor (MOV). SiC has been the most popular type of varistor before the MOV comes onto the market. SiC is made up from SiC particles bonded together with ceramic binder which electrical resistance decreases with an increase in the applied voltage (Takeshi & Michio 1968; Harris, 1995). MOV is a varistor made up from semiconductor material from the group II-VI. The combination is zinc (Zn) from the group II and O from the group VI. MOV replace the SiC because of the high nonlinear behavior of current-voltage (I-V) characteristics and providing improved the circuit protection (Dlamini et al., 2020).

1.2 Research Background

Recent years many technologies for electrical and electronics devices have been produced and using in daily life. Protection is needed to prevent electronic devices from damaged. In spite of that matter, many researches regarding surge protector have been conducted. In the early 1970's, metal oxide varistor was initially developed by Matsuoka in Japan (Matsuoka, 1971). The study indicated the properties of zinc oxide doped with alkaline earth metal oxide exhibited nonlinear behavior in current-voltage properties. Then, many researchers focused on the improvement of non ohmic properties.

ZnO varistor consist of zinc oxide (ZnO) as base ceramic and doped with several additives. Metal oxide additives such Bi_2O_3, Pr_2O_3, and Sb_2O_3 control the growth of crystals and the density of materials at temperatures above 1100 °C (Skidan, 2003). Varistor acts as an open circuit until the applied voltage passes the breakdown voltage, causing the varistor become a low resistance current path. Most of previous studies have been reviewed in Chapter 2.

In this study, the ZnO varistor based ceramics prepared using standard solid state methods were fabricated by using metal oxide CoO to serve as a Schottky barrier in the ZnO grains. Previous study use metal oxide such as Bi_2O_3, Sb_2O_3, CoO and rare-earth such as Pr_6O_{11}, CeO_2, as additives for ZnO varistor. Previous study shows glass additives affected nonlinear coefficient and breakdown voltage (Lin et al., 1993). Several studies have confirmed the effectiveness of borosilicate glass, boron oxide (B_2O_3) and pure SiO_2 as additives in the varistor system (Wu & Shyu; 1989; and Lu et al., 2020). However, there is lack of research use SLS glass as additives in varistor. The uses of soda lime silica (SLS) glass as additives for ZnO have improved electrical and thermal stability of the ZnO varistors. SLS glass contains majority composition about 74% of SiO_2 which act as grain growth inhibitor and also increase the nonlinearity of ZnO varistor.

1.3 Justification of Study

Although other researchers studied the production of varistors for electrical and electronic applications, there was a lack of study of the use of glass as additives. The use of recycled SLS glass can reduce costs and offering new green technology for electronic devices particularly ceramic based ZnO varistor.

1.4 Zinc Oxide

Zinc Oxide (ZnO) is a white powder, inorganic compound that is insoluble in water. The combination is of Zn from the group II and O from group VI. The native doping of this combination is n-type due to electrons as a majority carrier compared to hole carrier. The ZnO semiconductor does have many characteristics, such as high mobility of electrons, wide band gap, good transparency and strong room-temperature luminescence. The band gap of ZnO at low temperatures and room temperature are 3.44 eV and 3.37 eV. These properties allow ZnO to be used widely in optoelectronic applications.

1.5 Additives

1.5.1 CoO

Cobalt oxide is an inorganic compound. The colour is greyish or black powder. CoO has a 74.9326 g/mol of molar mass and 6.44 g/cm^3 of density. This compound is insoluble in water, very stable in air and has a melting point at 1933 ˚C. The band gap of cobalt oxide is around 2.4 eV. Cobalt is widely used in alloys. It is also used in electroplating to give an attractive surface to the objects that resist oxidation.

1.5.2 Soda Lime Silica glass

Soda lime silica (SLS) glass is one type of glass that is often used use for glass containers such as (bottle and jars), windowpanes and other commodity things. Soda lime silica has a major composition of silicon dioxide (SiO_2), sodium oxide (Na_2O), and calcium oxide (CaO). The SLS glass melting point is about 1000 ˚C. The silica rich waste can potentially be as material for electronic device such as varistor.

1.6 Problem Statement

The most notable characteristics of ZnO varistor are their nonlinear I-V behaviour. However, pure ZnO has a linear I-V characteristic. The non-ohmic ZnO varistor can be fabricated by doped with metal oxide like Bi_2O_3, Sb_2O_3, MnO_2, TiO_2 and Co_3O_4 (Matsuoka, 1971). Additives play a major role in getting non ohmic characteristics. Previous research has proven glass as additives can exhibit the non-linear characteristics of ZnO varistor. The additives can form at ZnO grains and grain boundaries that build a Double Schottky Barrier (DSB) to charge carriers transport into the ZnO grains via diffusion. Majority of the reported studies shows the preparation of the glass mixtures in the laboratory and the process took a longer time and also costly for the preparation of the samples. This project focuses on the application of recyclable glass in the varistor, where it differs from other reported studies that involve the preparation of glass mixtures in the laboratory. To save the time and money, SLS glass is chosen as additives. The SLS glass consists of silicon dioxide, sodium oxide (soda) and calcium oxide (lime). SiO_2 is the highest composition about 74% in the SLS glass which can control ZnO grain growth and also improve the nonlinearity of ZnO varistor (Zaid et al., 2016). Majority recycles SLS glass is focused to optoelectronic application such as military services, LED traffic lights, photodiodes, automatic access control systems and medical equipment. There is lack of study such SLS glass on electronic device and focused on varistor application. Previous study for ZnO varistor shown wet milling method is use as mixture preparation. The mixing operation is much easier in liquid phase such as distilled water and organic polymerization agent (Frigura-Iliasa et al., 2019). There is lack of study that chooses dry milling method for mixture preparation. Dry milling is a good technique for producing fine and homogeneous sample powders (Sarrigani & Amiri, 2019). Other than that, sample preparation generally consumes less time compared to wet milling methods. The effect of different milling method will be investigated and undergoes characterization on morphological, density and electrical properties. ZnO based varistor ceramics commonly prepared by solid state method in which sintering process to remove impurity as well as form highly dense ceramic product. Sintering process that are affected by several of indicators, including sintering temperature, sintering time, heating rate, cooling methods and sintering methods (Zhang et al., 2013). However, there is little concern on the sintering time. In this sense, the aim of this work to study the influence of sintering time upon microstructural and electrical characteristics of 2.0 mol% SLS glass doped ZnO-CoO varistor ceramic sintered at 1100˚C through the similar preparation method.

1.7 Hypothesis

The use of SLS glass as dopant in varied composition will affect the electrical properties of ZnO varistor. As composition of the SLS glass increased, the nonlinearity coefficient of I-V characteristic will increase and resulting in improvement of the electrical stability. However, the concentration of SLS glass will be expected less than 3 mol% to achieve excellent electrical properties. This is due to the other chemical composition in SLS glass such Na_2O and CaO might be effect electrical properties of ZnO varistor. Besides, the different milling method which is wet milling and dry milling will affect the nonlinearity of the ZnO varistor.

1.8 Objectives

In the present research work, ZnO based varistor ceramic were fabricated by doped with metal oxide CoO and SLS glass, different milling methods which is wet milling method and dry milling method. All samples were characterized to study the morphology, density, and electrical properties.

The objectives of this research are;

1. To synthesis ZnO-CoO_2-SLS_x based varistor ceramics where *x* is 0.5 to 2.0 mol% by using conventional solid state method sintered at 1100 °C for 120 minutes.
2. To study the effect of varied SLS glass concentration on microstructure and electrical properties of ZnO-CoO based varistor ceramics by wet and dry milling method sintered at 1100 °C for 120 minutes.
3. To study the effect of different sintering time (60 to 180 minutes) on microstructure and electrical properties of ZnO-CoO based varistor ceramics by dry milling method sintered at 1100 °C at constant of 2.0 mol% SLS glass.

1.9 Scope of Study

The study focuses on the effect of the varied concentration of SLS glass doped ZnO varistor ceramics on the nonlinear electrical properties such as nonlinear coefficient α, leakage current, breakdown voltage and barrier height calculated from the current-voltage curve. For microstructure properties, the study is concerned the effect of varied concentration of SLS glass on diffraction angle, *d*-spacing, lattice parameter, Full Widths Half Maximum (FWHM), density, and grain size. The study is limited to the varistor ceramics prepared using conventional solid-state method and for the low voltage varistor application

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LIST OF PUBLICATIONS

Ismail, N. Q. A., SA, N. K., & Zaid, M. H. M. (2020). Enhancement of non-ohmic properties of CoO doped ZnO varistor ceramics using soda lime silica (SLS) glass. *Sains Malaysiana*, *49*(4), 871-876.

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