SYNTHESIS AND CHARACTERIZATION OF LEAD ZIRCONATE TITANATE ($Pb[Zr_{0.52}Ti_{0.48}]O_3$) PROPERTIES VIA HIGH ENERGY PLANETARY BALL MILLING

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DOCTOR OF PHILOSOPHY

UNIVERSITI MALAYSIA PAHANG

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for the award of the degree of
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LIST OF SYMBOLS

%	Percentage
°	Degree
θ	Theta
°C	Degree Celsius
Å	Armstrong
M_D	Dry Weight
M_S	Suspended Weight
M_W	Saturated Weight
MPa	Mega Pascal
MHz	Mega Hertz
GHz	Giga Hertz
kHz	Kilo Hertz
ml	Milliliter
g	Gram
rpm	Rotation per Minutes
pC/N	Coulomb per Newton
nm	Nanometer
µm	Micrometer
mm	Millimeter
cm	Centimeter
kV	Kilo Volts
K	Kelvin
m	Meter
wt %	Weight percent

Ω	Ohm
s	Second
min	Minutes
ε	Permittivity
tan δ	Tangent lost or dissipation factor
C	Capacitance
V	Volt
T_c	Curie temperature
d_{33}, d_{31}, d_{15}	Piezoelectric Charge Constants
s^D_{33}, s^D_{11}, s^E_{11}, s^E_{33}, s^E_{55}, s^D_{55}	Elastic Constants
g_{33}, g_{31}	Piezoelectric Voltage Constants
Q_m	Mechanical Quality Factor
k_{33}, k_{31}	Piezoelectric Coupling Factors
k_p	Planar Coupling Factor
k_t	Thickness Coupling Factor
f_a	Anti-Resonance Frequency
f_r	Resonance Frequency
Z_r	Impedance at Resonance Frequency
P_s	Spontaneous Polarization
P_r	Remanent Polarization
F_R	Rhombohedral Phase
F_RL	Low Temperature Rhombohedral Phase
F_RH	High Temperature Rhombohedral Phase
F_T	Ferroelectric Tetragonal

P	Polarization
E	Electric Field

LIST OF ABBREVATIONS

$Pb(Zr_{0.52}Ti_{0.48})O_3$ (PZT)	Lead Zirconate Titanate
PbO	Plumbum Oxide
ZrO_2	Zirconia Oxide
TiO_2	Titanium Oxide
ABO_3	Perovskite Family
$MgTiO_3$	Magnesium Titanate
$PbTiO_3$	Plumbum Titanate
$CaTiO_3$	Calcium Titanate
$BaTiO_3$	Barium Titanate
Pt	Platinum
Ag	Silver
RuO_x	Ruthenium Oxides
MPB	Morphotropic Phase Boundary
SEM	Scanning Electron Microscopy
XRD	X-ray Diffraction
EDX	Energy-dispersive X-ray Spectroscopy
EDS	Energy Dispersive Spectrometry
DTA	Differential Thermal Analysis
TGA	Thermal Gravimetry Analysis

ABSTRACT

$Pb(Zr_{0.52}Ti_{0.48})O_3$ piezoelectric was successfully prepared by solid state reaction method via high energy planetary ball milling. This method which implements a single firing step where the calcinations process has been skipped thus successfully controls the higher evaporation amount of PbO during sintering process. Optimization is important on sintering parameters when using a single firing process. With the optimum processing parameters and sintering conditions (temperature and duration), the excellent PZT formation and optimum electrical properties of PZT ceramics was produce. The properties of piezoelectric materials were modified by different milling duration (20, 40 and 60 hours) and sintering parameters (temperature: 1000, 1050, 1100, 1150 and 1200 °C; soaking time: 1, 2 and 3 hours). Based on the study, sample milled for 40 hours show suitable milling duration with SEM analysis promotes fine particles and densification, thus achieved an excellent dielectric and piezoelectric properties. Preliminary works have been done in order to find suitable sintering temperature and durations to obtain the best PZT formation with good piezoelectric properties. This study continued to the next phase with 40 hours and sintered at different sintering temperatures (1050, 1100, 1150 and 1200 °C) and durations (1, 2 and 3 hours). According to the results, XRD analysis indicate that samples sintered at different sintering temperature and duration show single perovskite phase and no crystal structure changes were detected. However, sample sintered at 1200 °C for longer sintering duration (3 hours), new phase which mainly of excess PbO oxides were identified. SEM analysis was carried out and shows that, fine and dense grain sizes were achieved after sintering at 1050, 1100 °C for 1 hour. Increasing the sintering temperature and duration, larger and irregular grain size were observed. SEM observation indicated that sample sintered at 1200 °C for 2 hours durations shows better grain morphology whereby homogenous and dense microstructure were obtained. Increasing of the sintering temperature and durations, produces an increase of the grain size, increase in dielectric and piezoelectric properties thus decrease the value of dielectric loss for PZT ceramic. However, higher sintering temperature (1200 °C) and longer sintering durations (3 hours), the relative density slightly decreases to about 92 % of the theoretical density and causes the electrical properties to decrease. This phenomenon happens due to the evaporation of PbO whereby the existence of PbO phase was detected in the XRD patterns. Based on the results, sample sintered at 1200 °C for 2 hours show optimum sintering parameter and exhibits excellent room temperature electrical properties, which are listed as follows: ε_r = 2400, tan δ = 0.009, d_{33} = 256 pC/N, k_t = 0.48, k_p = 0.42 and Q_m = 1716.

ABSTRAK

Piezoelektrik $Pb(Zr_{0.52}Ti_{0.48})O_3$ telah berjaya dihasilkan menggunakan kaedah tindak balas pepejal melalui proses pengisar bebola planetari. Kaedah ini menggunakan satu proses pembakaran yang mana pengkalsinan dapat dielakkan dan sekali gus Berjaya mengawal pemeruapan jumlah PbO semasa proses pensinteran. Pengoptimunan adalah penting untuk parameter pensinteran apabila menggunakan satu proses pembakaran. Dengan optimum parameter pemprosesan dan keadaan pensinteran yang bagus (suhu dan tempoh), pembentukan PZT yang baik dan sifat elektik yang optimum dapat dihasilkan. Sifat – sifat bahan seramik piezoelektrik diubah suai dengan tempoh pengisaran (20, 40 dan 60 jam) dan parameter pensinteran yang berbeza (suhu pensinteran: 1000, 1050, 1100, 1150 dan 1200 °C; tempoh pensinteran: 1, 2 dan 3 jam). Hasil daripada kajian, sampel yang dikisar selama 40 jam menunjukkan tempoh pengisaran paling sesuai dengan analisis mikrostruktur menunjukkan saiz partikel yang seragam dengan ketumpatan yang tinggi dan memperolehi nilai lesapan dielektrik dan sifat piezoelektik yang bagus. Penyelidikan awal telah dijalankan untuk mengenalpasti suhu dan tempoh pensinteran yang sesuai bagi memperoleh pembentukan PZT dan sifat piezoelektrik yang bagus. Kajian ini diteruskan kepada fasa lain dengan menggunakan tempoh pengisaran 40 jam dan sampel disinter pada suhu (1050, 1100, 1150 dan 1200 °C) dan masa (1, 2 dan 3 jam) pensinteran yang berbeza. Hasil daripada keputusan ujikaji, analisis belauan sinar-X menunjukkan sampel yang disinter dengan suhu dan tempoh pensinteran yang berbeza menghasilkan satu fasa tunggal (PZT) dan tiada perubahan struktur kristal. Walaubagaimanapun, sampel yang disinter pada suhu 1200 °C untuk tempoh pensinteran yang lama (3 jam), fasa baru iaitu lebihan oksida PbO telah dikenal pasti. Analisis mikrostruktur menunjukkan, butiran saiz yang kecil dan sekata telah diperolehi selepas pensinteran pada suhu 1050 dan 1100 °C selama 1 jam. Apabila suhu dan masa pensinteran ditingkatkan, saiz butiran yang membesar dan tidak sekata terhasil. Pemerhatian pada mikrostruktur menunjukkan sampel yang disinter pada suhu 1200 °C selama 2 jam memaparkan morfologi butiran yang lebih baik yang mana butiran lebih sekata dan seragam yang jelas kelihatan. Kenaikan suhu dan tempoh pensinteran menghasilkan saiz butir yang besar, peningkatan pada sifat dielektrik, menurunkan nilai lesapan dan meningkatkan nilai sifat piezoelektrik bagi seramik PZT. Namun, lebih tinggi suhu pensinteran menjangkau 1200 °C untuk tempoh pensinteran yang panjang (3 jam), nilai ketumpatan menurun sehingga 92 % daripada ketumpatan teori dan sifat elektrik seramik PZT menurun. Hal ini berlaku disebabkan pemeruapan PbO yang mana fasa PbO telah dikenal pasti melalui analisis belauan sinar X-ray. Dalam kajian ini, sampel yang disinter pada suhu 1200 °C selama 2 jam menunjukkan parameter pensinteran yang paling sesuai dan mencapai sifat elektrik yang paling bagus seperti yang disenaraikan: ε_r = 2400, tan δ = 0.009, d_{33} = 256 pC/N, k_t = 0.48, k_p = 0.42 and Q_m = 1716.

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