AN INTRODUCTORY STUDY ON FABRICATION OF PIEZOELECTRIC CERAMIC

(KAJIAN PENGENALAN TERHADAP PEMBUATAN SERAMIK PIEZOELEKTRIK)

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DECLARATION

This work has not previously been accepted in substance for any degree and is not being concurrently submitted in candidature for any degree

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Regards,
Zuraihana Bachok
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SYMBOLS & UNITS

Symbol	Description
ε_0	permittivity of free space (8.85 x 10^{-12} farad / m)
ΔT	temperature difference (^{0}C)
ε^T	permittivity of ceramic material (farad / m) (at constant stress)
ABO_3	Type of structure
d	diameter of ceramic disc or rod (m)
d_{33}	piezoelectric charge constant (C / N)
$E_{(pyro)}$	electric field in (volts/meter)
F	force
g_{33}	piezoelectric voltage constant (V.m / N)
h	height (thickness) of ceramic element (m)
in.	inch
k	electromechanical coupling factor
K_3	dielectric constant
kNf	kilo Newton force
l	initial length of ceramic element (m)
m	meter
mm	millimeter
N	frequency constant (Hz.m)
pkpk	peak to peak
Q_m	mechanical quality factor
r	radius
rms	root mean square
s	elastic compliance (m^2 / N)
S	surface area of ceramic element (m^2)
sdev	standard deviation
t	thickness
T	stress

$\tan \delta$	dielectric dissipation factor
T_c	curie temperature / Curie point (^{0}C)
V	volt
Y	young's modulus (N / m^{2})
α	pyroelectric coefficient (Coulomb/^{0}C m^{2})

ABBREVIATION

Abbreviation	**Description**
AMREC	Advanced Material Research
Ba	Barium
$BaCO_3$	Barium Carbonate
$BaTiO_3$	Barium Titanate
C	Carbon
$CaTiO_3$	Calcium Titanate
CO_2	Carbon Dioxide
DC	Direct current
HIP	Hot isostatic press
$K(Ta_xNb_{l-x})O_3$	Potassium Tantalate Niobate
$KNbO_3$	Potassium Niobate
$K_xNa_{l-x}NbO_3$	Potassium Sodium Niobate
MgO	Magnesium Oxide
O	Oxygen
Pb	Lead
PbO	Lead Oxide
$PbTiO_3$	Lead Titanate
PLZT	Lead Lanthanum Zirconate Titanate
PMN	Lead Magnesium Niobate
PZT/ $PbZrO_3$	Lead Zirconate Titanate
Ti	Titanium
TiO_2	Titanium Oxide
ZrO_2	Zirconia

ABSTRACT

This thesis gives a quite detailed overview of piezoelectric ceramics. It will be beneficial to general reader to get basic ideas about this smart material once the reader going through this thesis. The scope of this work is limited to the development of ceramic piezoelectric, related to the scope of work which the objectives of the study are to focus on its manufacture of the green material, the poling process and the study of its physical and electrical characteristics. Processing of Barium Titanate ($BaTiO_3$) ceramic by mixed-oxide method for high technology applications involves the sequential steps; (a) powder preparation (b) powder calcining (c) de-agglomeration (d) forming (e) sintering (f) electroding (g) and poling. Each step is critical to the realization of desirable structure-property relationships.

To this end of the thesis, I have presented a brief introduction of the history of piezoelectric ceramics and a discussion on processing of the Barium Titanate ceramic ($BaTiO_3$) and testing of the ceramic that define the behavior of a piezoelectric material. The ceramic was manufactured using Barium Carbonate ($BaCO_3$) powder and Titanium Oxide (TiO_2). The testing was done by using oscilloscope (waverunner) to observe the characteristic of piezoelectric ceramics.

This work is organized as follows: in chapter 2 the previous work of processing a piezoelectric ceramic. Chapter 3 details the apparatus and experimental procedures used to fabricate and testing of piezoelectric ceramic. The result and discussion of the work are presented in chapter 4.

To understand the behavior of a piezoelectric polycrystalline ceramics, we should know the principle of natural crystal like Quartz, Rochelle salt, and Tourmaline. Quartz crystal is naturally piezoelectric material and polycrystalline ceramic is an artificially polarized that manufactured material.

ABSTRAK

Tesis ini memberikan gambaran yang agak terperinci tentang seramik piezoelektrik. Sekiranya pembaca meneliti tesis ini, ia mungkin berguna kepada mereka untuk mendapatkan idea tentang seramik piezoelektrik ini. Bidang tugas yang dilakukan hanya terhad kepada penghasilan seramik piezoelektrik, yang berkaitan dengan pembuatan seramik, proses pengkutuban "poling" dan kajian tentang sifat fizikal dan elektrikal. Kaedah campuran oksida digunakan dalam proses penghasilan Barium Titanate seramik untuk menghasilkan barangan yang menggunakan teknologi tinggi. Proses penghasilan Barium Titanate seramik ini adalah seperti berikut: (a) penyediaan serbuk (b) pembakaran serbuk (c) penghancuran (d) pembentukan (e) pembakaran pada suhu yang tinggi (f) pengelektrodan (g) dan pengkutuban. Setiap langkah adalah kritikal bagi menghasilkan barangan yang mempunyai sifat-sifat yang dikehendaki.

Di dalam tesis ini, saya menerangkan secara ringkas sejarah seramik piezoelektrik, membincangkan proses penghasilan Barium Titanate dan menjalankan ujikaji terhadap seramik piezoelektrik untuk mengenalpasti sifat keadaannya. Serbuk Barium Karbonat dan Titanium Oksida digunakan untuk menghasilkan seramik ini. Ujikaji dijalankan dengan menggunakan osiloskop jenis "waverunner" untuk memerhatikan sifat piezoelectrik ke atas seramik ini.

Tesis ini disusun seperti berikut: di dalam bab 2, ia menerangkan kajian ilmiah tentang proses-proses penghasilan seramik piezoelektrik yang telah dijalankan. Bab 3 pula menerangkan secara terperinci tentang peralatan dan kaedah eksperimen yang digunakan untuk membentuk dan menganalisa seramik piezoelektrik ini. Seterusnya huraian tentang keputusan dan perbincangan yang diperolehi daripada eksperimen yang dijalankan diterangkan di dalam bab 4.

Untuk memahami sifat keadaan seramik piezoelektik ini, kita perlu mengetahui terlebih dahulu tentang prinsip hablur seperti Kuarza, "Rochelle salt" dan "Tourmaline". Hablur Kuarza menunjukan sifat piezoelektrik secara semulajadi manakala seramik piezoelektrik pula perlu melalui proses pengkutuban "poling" terlebih dahulu untuk menghasilkan sifat piezoelektrik. Seramik piezoelektrik terbentuk daripada hasil buatan manusia dan tidak menunjukan sifat piezoelektrik secara semulajadi seperti hablur kuarza.

CHAPTER 1: INTRODUCTION

1.1 Background

Electrical energy can be produced from sources of energy such as friction, chemical reaction, light, magnetic and heat. Another source that can produce electrical energy is a material known as piezoelectric, which is unique compared to any other source of electrical energy sources. This material will produce electrical energy upon receiving a mechanical impact on its poling ends. This material, in reverse, will produce a physical deformation upon receiving electrical charge also at its poling ends.

In this work, an introductory study on the fabrication of piezoelectric ceramic was exercised by using Barium Carbonate powder and Titanium Oxide powder. This was chosen in conjunction with the work being carried out in School of Material and Mineral Resources Engineering, Universiti Sains Malaysia, conducted by Miss Teoh Wah Tzu (PhD student) under the supervision of Prof Madya Dr. Zainal Arifin Ahmad.

This work is the first work of its kind conducted in School of Mechanical Engineering. The reason to do this work is because there is an urgent need for Mechanical Engineer's field of study to get involved in the piezoelectric study, since this material is playing an important role in the “submicron scale” of Mechanical Engineering, such as the application of piezoelectric components in micro actuators used in many automatic intelligent micro positioning devices.

Piezoelectric

Crystal like quartz can produce electrical energy when pressures are applied on the surface like the one that shown in Figure 1.1. This effect called piezoelectric effect. The piezoelectric effect is often encountered in daily life. For example, in small butane cigarette or gas grill lighters, a lever applies pressure to a piezoelectric crystal creating an electric field strong enough to produce a spark to ignite the gas. Furthermore, alarm clocks often use a piezoelectric element. When DC voltage is applied, the piezoelectric material moves at the frequency of the applied voltage and the resulting sound is loud enough to wake even the strongest sleeper.

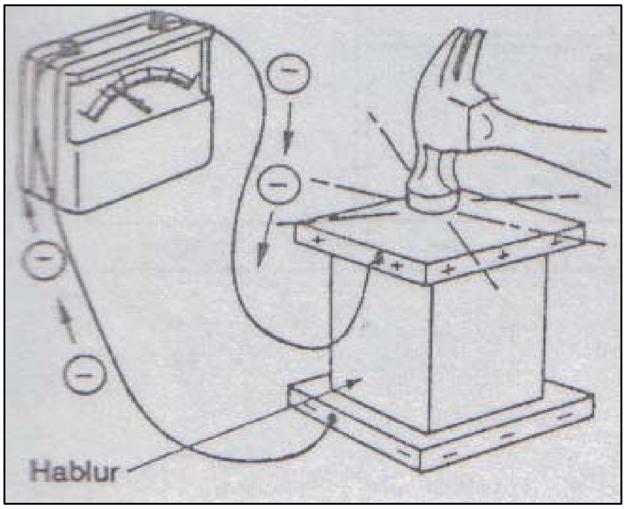

Figure 1.1 Electrical energy produced caused of pressure effect.

Piezoelectricity is a property of certain classes of crystalline materials including natural crystal of Quartz, Rochelle salt and Tourmaline plus manufactured ceramics such as Barium Titanate and Lead Zirconate Titanates (PZT). As we know, natural crystals are quiet expensive and difficult to find. Therefore, piezoelectric ceramics are widely used today because they are more versatile in terms of their physical, chemical and piezoelectric characteristics which are able to be tailored to specific applications. Furthermore, piezoelectric ceramics can be manufactured in almost any given shape or size and they are chemically inert, and immune to moisture and other atmospheric conditions. Piezoelectric ceramics are more sensitive and offer a less expensive alternative to Quartz and other piezoelectric single crystals which are presently widely used for pressure, force, acceleration and vibration sensors.

A phenomenon of piezoelectricity or “pressure electricity” occurs in a certain class of natural crystalline materials such as Quartz, Rochelle salt and Tourmaline. They are typically referred to as ferroelectric materials. Piezoelectric ceramics work in two ways which convert electrical energy into mechanical (vibration) and mechanical into electrical. When pressure or a force is applied to a piezoelectric ceramic (like hitting it or talking to it), it will generate a voltage. This effect is used in microphones, sonar and medical ultrasound equipment. The other ways is when voltage is applied to the piezoelectric ceramic, it will expands and contracts and changing the shape. It either begins to vibrate or exerts pressure on whatever is touching it.

Table 1.1 Comparisons of Piezoelectric Materials (*PCB Piezotronics)*

Quartz Crystal	**Polycrystalline Ceramic**
naturally piezoelectric material	artificially polarized, man-made material
high voltage sensitivity	high charge sensitivity
stiffness comparable to steel	unlimited availability of sizes and shapes
exhibits excellent long term stability	materials available which operate at 1000 F (540^0C)
non-pyroelectric	output due to thermal transients (pyroelectric)
low temperature coefficient	characteristics vary with temperature

Piezoelectricity is an anisotropic characteristic and green materials (ceramic shape) are isotropic and are not piezoelectric before poling. Therefore, in this work, there is an explaining of poling process after producing the ceramics because once they are polarized, the ceramics become anisotropic that have piezoelectric characteristic. The piezoelectric effect depends on the type of piezoelectric material and the mechanical and electrical axes of operation can be precisely orientated within the shape of the ceramic. These axes are set during "poling"; the process that induces piezoelectric properties in the ceramic. The orientation of the dc poling field determines the orientation of the mechanical and electrical axes. After the poling process is complete, a voltage lowers than the poling voltage changes the dimensions of the ceramic for as long as the voltage is applied. The poling process permanently changes the dimensions of a ceramic element. After the poling process is complete, compressive and tensile forces applied to the ceramic elements that will generate a voltage.

Piezoelectric materials therefore can be classified as smart materials that have the ability to senses a change in its environment and through the use of a feedback system, adapts to correct or eliminate such a change.

Knollenberg, 2000 in his study found that *pyroelectricity and ferroelectricity* are phenomena related to piezoelectricity that arise from the polar nature of these materials, but are only observed in specific sub-sets of the family of piezoelectric materials. *Ferroelectricity* is a subgroup of piezoelectricity, where a spontaneous polarization exists that can be reoriented by application of an AC electric field and crystals that develop an electric charge due to change in the magnitude of a unique polar dipole upon uniform heating are labeled *pyroelectric.*

1.2 Purpose and scope of work

The purpose of this work is to develop further the piezoelectric material using $BaTiO_3$ composition on the interest that this material has a better dielectric properties compared to the existing widely used PZT material.

The scope of work

The work carried out in this work will be focused on a fabrication of green material (Barium Titanate ceramic), an overview of its associated poling process and an observation on its physical and electrical characteristics.

1.3 Definition and History

1.3.1 Definitions

1.3.1.1 Piezoelectricity

The literal translation of the term "piezoelectricity" comes for the Greek word piezin, which means "to press". In a more specific sense, the term refers to the ability to create electricity by pressure, and conversely, the development of mechanical strain by electricity. Piezoelectricity is a linear effect that is related to the microscopic structure of the solid. In a subsequent study *(Jordan & Ounaies, 2001*), it was shown some ceramic materials become electrically polarized when they are strained; this linear and reversible phenomenon is referred to as the *direct piezoelectric effect*. The direct piezoelectric effect is always accompanied by the *converse piezoelectric effect* where a solid becomes strained when placed in an electric field. The microscopic origin of the piezoelectric effect is the displacement of ionic charges within a crystal structure. In the absence of external strain, the charge distribution within the crystal is symmetric and the net electric dipole moment is zero. However, when an external stress is applied, the charges are displaced and the charge distribution is no longer symmetric. A net polarization develops and results in an internal electric field. A material can only be piezoelectric if the unit cell has no center of inversion.

1.3.1.2 Ceramics

Ceramics are solid, inorganic, nonmetallic materials made by firing (sintering) compacted powders, where the particles of the powder coalesced or fused together. The aggregate (collection of particles of powder clustered in a dense mass) is accomplished by firing at an elevated temperature. At that temperature the sintering process occurs. This process will shrink the aggregate body, caused by the grains bond. Thus a solid is produced with an increased in density. Ceramics are typically hard, high temperature resistant, corrosion resistant and inexpensive.

In order to understand crystal structure, it is necessary to know something about atomic structure, but explaining atomic structure in detail is not within the scope of this work. There are ionic bonds, covalent bonds, van der Waals bonds and metallic bonds. Oxides ceramics that used in the fabrication of ceramic mainly have ionic bonding.

There are several types of piezoelectric ceramic materials available which each type is tailored towards the requirements of particular applications. This is achieved by changing the chemical composition of the ceramic to enhance specific properties. The ceramic materials are arranged in three groups: Hard Materials, Soft Materials and Customized Materials.

- **High Power "Hard" Materials**

  High power or "hard" ceramics can withstand high levels of electrical excitation and mechanical stress. These materials are suited for high voltage or high power generators and transducers.

- **High Sensitivity "Soft" Materials**

  High sensitivity or "soft" ceramics feature high sensitivity and permittivity, but under high drive conditions are susceptible to self-heating beyond their operating temperature range. These materials are used in various sensors, low-power motor-type transducers, receivers, and low power generators.

- **Customized Materials**

  Several ceramic materials have been formulated to meet the particular needs of specific applications.

Table 1.2 Characteristics of Soft Ceramics and Hard Ceramics Compared *(APC International Ltd.)*

CHARACTERISTIC	**SOFT CERAMIC**	**HARD CERAMIC**
Piezoelectric Constants	larger	smaller
Permittivity	higher	lower
Dielectric Constants	larger	smaller
Dielectric Losses	higher	lower
Electromechanical Coupling Factors	larger	smaller
Electrical Resistance	very high	lower
Mechanical Quality Factors	low	high
Coercive Field	low	higher
Linearity	poor	better
Polarization / Depolarization	easier	more difficult

1.3.1.3 Ferroelectricity

There are many piezoelectric materials which are not ferroelectric but all ferroelectric materials are piezoelectric. A ferroelectric material possesses a spontaneous polarization that can be reversed in direction by application of a realizable electric field over some temperature range. It creates part of the piezoelectric effect. Most ferroelectric materials have a Curie temperature, T_c below.

Ferroelectric materials exhibit self polarization, that is adjacent dipoles align themselves by common interaction and these parallel dipoles arrange themselves into domains. All have high dielectric constants and high refractive indices. The polarization of these materials is not proportional to the applied electric field as for paraelectric materials (i.e. polarization is proportional to applied electric field) but instead exhibits hysteresis. There is a coercive field required to reduce the polarization to zero and a remanent polarization which remains when the field is reduced to zero. These materials are called ferroelectric by analogy with ferromagnetic materials which also have domains and exhibit hysteresis.

1.3.1.4 Pyroelectricity

Piezoelectric materials are also pyroelectric. They produce electric charge as they undergo a temperature change. When their temperature is increased, a voltage develops having the same orientation as the polarization voltage. When their temperature is decreased, a voltage develops having an orientation opposite to the polarization voltage, creating a depolarizing field with the potential to degrade the state of polarization of the part. The maximum electric field which arises due to a temperature shift is:

$$E(\text{pyro}) = \frac{\alpha\ (\Delta T)}{K_3\ \varepsilon_0}$$

where $E_{(pyro)}$ is the induced electric field in volts/meter, α is the pyroelectric coefficient in Coulomb/° C meter ^{2}, ΔT is the temperature difference in °C, K_3 is the dielectric constant, and ε_0 is the dielectric permittivity of free space. For PZT piezoceramic, α is typically ~ 400×10^{-6} coulomb/°C meter^2.

Crystals that are piezoelectric (no centre of symmetry) but not pyroelectric (no dipole) do not show a charge on heating or cooling. Summarizing, all pyroelectric crystals are piezoelectric, but not all piezoelectric crystals are pyroelectric.

1.3.1.5 Perovskite structure

Perovskite is a family name of a group of materials and the mineral name of calcium titanate ($CaTiO_3$) having a structure of the type ABO_3 *(Safari, et.al. 1998)*. Many piezoelectric ceramics such as Barium Titanate ($BaTiO_3$), Lead Titanate ($PbTiO_3$), Lead Zirconate Titanate (PZT), Lead Lanthanum Zirconate Titanate (PLZT), Lead Magnesium Niobate (PMN), Potassium Niobate ($KNbO_3$), Potassium Sodium Niobate ($K_xNa_{1-x}NbO_3$), and Potassium Tantalate Niobate ($K(Ta_xNb_{1-x})O_3$) have a perovskite type structure. These materials are usually ceramics with a perovskite structure (see Figure 1.2). The perovskite structure exists in two crystallographic forms. Below the Curie temperature they have a tetragonal structure and above the Curie temperature they transform into a cubic structure. In the tetragonal state, each unit cell has an electric dipole, i.e. there is a small charge differential between each end of the unit cell. Basically, Curie point of Barium Titanate ceramic is approximately 120^0C-130^0C.

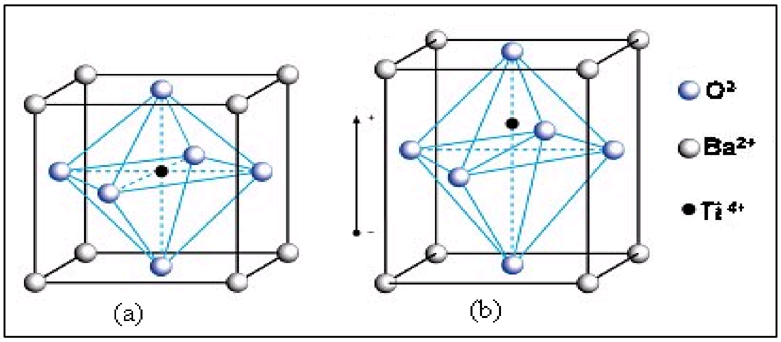

Figure 1.2 $BaTiO_3$ unit cell: a) Perovskite-type Barium Titanate ($BaTiO_3$) unit cell in the symmetric cubic state above the Curie temperature. b) Tetragonally or rhombohedral distorted unit cell below the Curie temperature

1.3.2 History of piezoelectric ceramics

The discovery of the phenomena of piezoelectricity is credited to Jacque Currie and his brother Pierre (who along with his wife, Marie, and Henri Becquerel shared the 1903 Nobel Prize in Physics). In 1880, the two brothers demonstrated that when a stress in the form of a weight was applied to quartz and other crystals, positive and negative charges on certain portions of their surfaced developed (*Trainer et.al. 2003).* These charges were proportional to the pressure exerted and disappeared when the pressure was withdrawn. Later they also confirmed that the change in crystal's dimension occurred when a voltage was applied.

It was during World War I that French scientist, Paul Langevin, used piezoelectric. In subsequent work on ceramics in the late 1940's and 1950's led to their widespread application. The first commercial device to be made from piezoelectric Barium Titanate was a phonograph pickup and was produced in 1947 *(Alpha Ceramics Inc. et.al).*

A number of other ceramics were found to have piezoelectric properties and in 1952, piezoelectric Lead Niobate was discovered in the United States and in 1955, Lead Zirconate Titanate (PZT) compositions were found, thus leading to the mass production of PZT products in the late 1950's.

There are several piezoelectric ceramic compositions in common use today: Barium Titanate, Lead Zirconate Titanate (and modified iterations such as PLZT), Lead Metaniobate and Lead Magnesium Niobate, PMN, (including electrostrictive formulations). The Lead Zirconate Titanate, PZT, compositions are the most widely usage in applications involving light shutters, micro-positioning devices, speakers and medical array transducers. Barium Titanate is considered an outmoded piezoelectric material and is the least used material. The Lead Metaniobate compositions have excellent electrical characteristics for use in transducers for nondestructive testing and high temperature applications as well as high resolutions medical applications.

The major applications for piezoelectric ceramics are ultrasonic transducers, audio transducers, speakers, medical ultrasound, measurement devices, filters and resonators, and high voltage devices.

CHAPTER 2: PREVIOUS WORK

In just over 100 years piezoelectricity has moved from a laboratory curiosity to big business. During this period, several technologies have been developed to utilize the piezoelectric effect such as transducer, actuator, sensor and generator. In turn, each of these technologies has become an essential component in many kinds of electronic products.

A variety of methods are employed for preparation of powder to produce a highly pure powders such as hydrothermal method, flux growth method, mixed oxide route (conventional method), co-precipitation method and melting or Czochralski growth method. However, this work is based upon ferroelectric ceramics that are prepared from a conventional ceramic powder processing route utilizing the individual constituent oxides.

Basically, the traditional ceramic powder processing is almost the same which starting with powder preparation, then powder calcinations, forming and sintering. The only difference is the set up of the operating temperature, time and pressure. Moreover, the other facilities used will also be different, depending on the suitability of the materials and processes carried out. To produce piezoelectric ceramic that have piezoelectric effect, the ceramic materials that obtained from variety methods as mentioned above should be put through a process called poling which aligns the domains and hence the dipoles to achieve saturation polarization. In order to create a dipole, electrodes are applied to the desired surfaces of the shaped product. After the poling process finished, a piezoelectric ceramics were produced with piezoelectric effect that can be use in application of piezoelectric igniter. In this work, the preparation powder method used is the mixed oxide method.

2.1 Preparation of piezoelectric ceramic by using mixed-oxide method

In a subsequent study *(Ballato et.al, 1996),* mixed-oxide methods for producing piezoelectric ceramics were used. Mixed-oxide route is a conventional ceramic powder processing that prepared via mixed oxides and this is the most common powder preparation.

According to *Ballato et.al. (1996),* the conventional mixed-oxide process consists of:

1. Weighing the oxides to the appropriate accuracy
2. Blending in a liquid medium
3. Drying to completeness

Powder preparation

4. Calcining the powder at approximately 900°C for 1 hour
5. Milling in a liquid medium
6. Drying with binder addition
7. Cold pressing a specific shape
8. High-temperature firing (sintering) at approximately 1275°C for several hours
9. Shaping
10. Electroding
11. Poling

During sintering, covered crucible approaches are typically used in conjunction with PbO-based atmosphere powders to compensate for lead oxide loss. *Safari et.al. (1998)* proposed a flowchart for the processing of piezoelectric ceramics (see Figure 2.1). It can be seen in *Safari's* flowchart, that in powder preparation stage, *Safari* does not explain the process as detailed as *Ballato* did in his 1 to 3 steps of eleven steps.

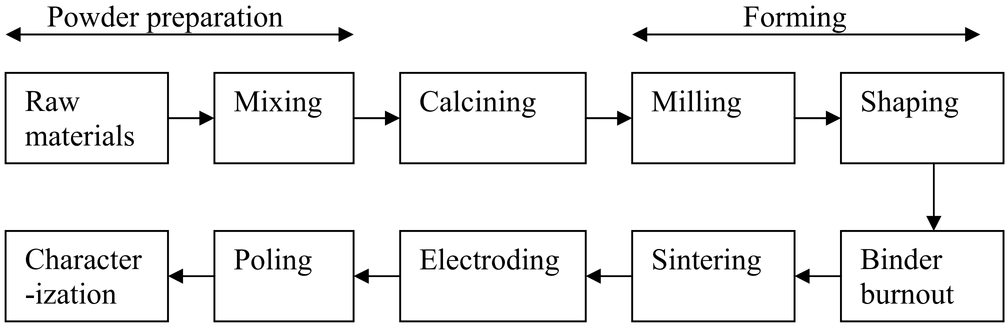

Figure 2.1 Flowchart for the processing of piezoelectric ceramics *(Safari et.al. 1998)*

Referring to Figure 2.1, the fabrication of most bulk piezoelectric ceramics starts with powder preparation. The powder is then pressed to the required shaped to produce mechanically strong and dense ceramics. However, the most important process that will influence the product characteristics and properties are the preparation of the powder and the calcining of the product. From the piezoelectric characteristics and properties point of view, the shaping stage, which consist of milling and shaping of the powder into the shape of the components that are going to be produced as well as the burn out of the binder if the binder is used, is not as critical because this will affect the physical shape of the product only. However, the next stage that is the sintering, the electroding, the poling and the characterization are the most important steps in the final process of the ceramic piezoelectric fabrication.

Basically, the processes are almost the same but only certain process can be choosing either needed or not. The binder is optional that we can choose either want to add or not. Attention should also be given to the mixing process during the powder preparation stage, where the powders can be mixed by mechanically either using dry or wet ball milling or attrition milling for a short time. These methods have advantages and disadvantages. *Jordan & Ounaies, 2001* was mentioned that wet ball-milling is faster than dry-milling. However, the disadvantage is the added step of liquid removal like done by *Ballato et.al. 1996* which need to dry the mixture before calcining the powder. This choice is depends on the individual to decide the methods of processing either want to use dry or wet ball milling.

Figure 2.2 shows a flowchart of the mixed oxide route for making PZT ceramics by *Jordan & Ounaies, 2001.* They claimed that the most common method for making PZT ceramics is through wet-ball milling. Ethanol and stabilized Zirconia media are added for the wet milling process. A vibratory mill may be used rather than a conventional ball mill that reduces the risk of contamination by the balls and the jar. Zirconia media are used to further reduce the contamination risks.

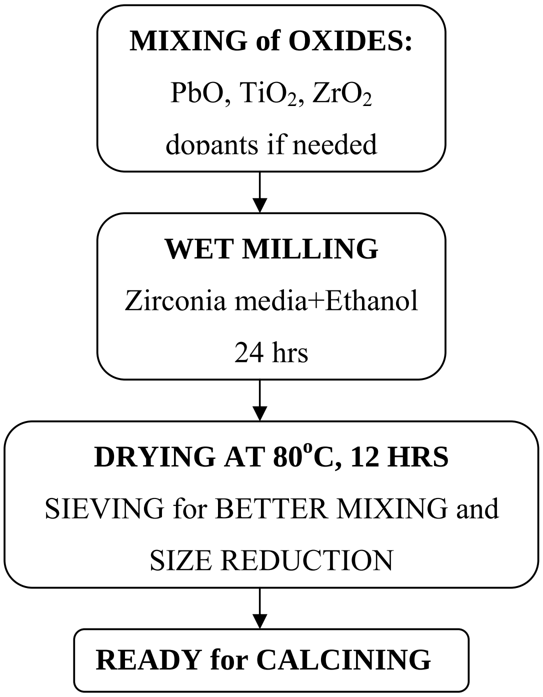

Figure 2.2 Mixed-oxide route of preparing PZT

The calcination step is a very crucial step in the processing of PZT ceramics and during this step the solid phase reaction takes place between the constituents giving the ferroelectric phase or perovskite phase. The calcination of PZT by *Safari et.al. (1998)* should be started by mixing raw materials PbO, TiO_2 and ZrO_2 in the molar ratio of 2:1:1. The mixture is then pressed into lumps and then calcined in ambient air at 800° C to obtain the perovskite phase. The objectives of this step are to remove any organics, water or other volatiles left after mixing and to form the desired phase composition before the ceramic is processed to useful devices. Beside that, it can also reduce volume shrinkage and allow for better homogeneity during and after sintering.

After calcining, a binder is added to the powder, and then the mixture is shaped usually by dry-pressing in a die for simple shapes. Extrusion and casting can also use for more complicated bodies. Next, the shapes are placed in an oven for binder burn-out and densification. This step can also be considered as the sintering step, but in much lower temperature. The major problem in the sintering of the PZT ceramic is the volatility of PbO which is at about 800°C. To minimize this problem, the PZT samples are sintered in the presence of a lead source, such as $PbZrO_3$, and placed in closed crucibles. The