DESIGN FABRICATION AND SYNTHESIS OF RECTANGULAR BEAM OF PZT AT VARIOUS Zr AND Ti RATIO

A thesis submitted in partial fulfillment of the requirements for the degree of Master of Technology in Instrumentation and Electronics Engineering

Submitted By

Jhelum Ray Chaudhuri

University Reg. No. : 129476 of 2014-2015

Class Roll No. : 001411103001

Examination Roll No. : M4IEE1601

Under the guidance of

Prof. Subhankar Bandyopadhyay

Department of

Instrumentation & Electronics Engineering

FACULTY OF ENGINEERING AND TECHNOLOGY
JADAVPUR UNIVERSITY
SALT LAKE CAMPUS
KOLKATA-700098

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FACULTY OF ENGINEERING AND TECHNOLOGY

JADAVPUR UNIVERSITY

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I hereby recommend that the work done by ***Ms. Jhelum Ray Chaudhuri (Registration No. 129476 of 2014-2015)*** *under my guidance and the thesis entitled* ***"DESIGN, FABRICATION AND SYNTHESIS OF RECTANGULAR BEAM OF PZT AT VARIOUS Zr AND Ti RATIO"****, be accepted in partial fulfilment of the requirement for the degree of* ***Master of Technology*** *in* ***Instrumentation and Electronics Engineering*** *from the Department of Instrumentation and Electronics Engineering of Jadavpur University.*

(Prof. Subhankar Bandyopadhyay)
Thesis Supervisor
Department of Instrumentation
& Electronics Engineering
Jadavpur University

Contersigned:

(Prof. Rajanikanta Mudi)
Head of the Department
Department of Instrumentation
& Electronics Engineering
Jadavpur University
Salt Lake Campus
Kolkata-700098

(Prof. Shivaji Bandyopadhyay)
Dean
Faculty of Engineering
and Technology
Jadavpur University
Kolkata-700032

FACULTY OF ENGINEERING AND TECHNOLOGY

JADAVPUR UNIVERSITY

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I hereby declare that this thesis contains literature survey and original research work by me, as a part of my Master of Technology in Instrumentation & Electronics engineering studies.

All information in this document have been obtained and presented in accordance with academic rules and ethical conduct.

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Name : JHELUM RAY CHAUDHURI

Examination Roll No. : M4IEE1601

Thesis Title : DESIGN, FABRICATION AND SYNTHESIS OF RECTANGULAR BEAM OF PZT AT VARIOUS Zr AND Ti RATIO

Signature with Date :

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Date:

Jhelum Ray Chaudhuri

PREFACE

This thesis is organized in a series of five chapters as follows:

Chapter 1 is an **Introduction** to piezoelectric materials, their classification, mechanism, storage and applications.

Chapter 2 named **Literature Survey** contains the citations of various works that have been done in the PZT (Lead Zirconate Titanate) field. They are arranged in chronological order for the ease of understanding. Rapid advancement in this field has been highlighted in this chapter.

Chapter 3 named **The Research Objective** deals with different chemical methods involving for preparation of PZT and its electrical characterization. This chapter also includes the plan of this research work.

Chapter 4 named **Experimental Procedure** mentions the steps of PZT sample preparation, sintered rectangular beam preparation. It contains the details of furnace time-temperature profile. Characterisation of the samples by XRD and SEM analysis has also been discussed in this chapter. Lastly, it contains the percentage change in length and width due to variation of composition.

Chapter 5 named **Results and Discussion** contains discussion and comparative study of the results obtained in XRD and SEM. It also deals in the percentage change in length and width of the PZT samples with different Zr/Ti ratio before and after sintering. Future scope of this thesis work has also been discussed in this chapter.

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CHAPTER 1
INTRODUCTION

1.1 INTRODUCTION

Certain crystals like natural Quartz (SiO_2) or quartz like other natural and artificially synthesized materials acquire a charge when compressed, twisted or distorted are said to be piezo-electric.

The piezo-electric effect can be made to respond to mechanical deformations of the material in many different modes, such as Thickness Expansion, Transverse Expansion, Thickness Shear and Face Shear. The mode of motion affected depends on the shape and orientation of the body relative to the crystal axes and the location of the electrodes. Metal electrodes are plated onto selected faces of the piezo-electric material so that lead wires can be attached for bringing in or leading out the electric charge. Since the piezo-electric materials are insulators, the electrodes also become the plates of a capacitor. A piezo-electric element used for converting mechanical motion to electric signals thus may be thought of as a Charge Generator and a Capacitor. Mechanical deformation generates a charge, this charge then results in a definite voltage appearing between the electrodes according to the usual law of capacitors, $V = \frac{Q}{C}$. The piezo-electric effect is direction sensitive in that tension produces a definite voltage polarity while compression produces the opposite.

Piezo-electric materials could be used to harvest energy in buildings. If the entire flooring of a building could be made from these materials, then there would be sufficient amount of energy produced to meet a part of the building’s energy requirement. This energy is ‘clean’ i.e., there is no pollution in its production and comes under the purview of CDM (Clean Development Mechanism).

1.2 CLASSIFICATION OF PIEZO-ELECTRIC MATERIALS

There are a wide variety of materials that exhibit piezoelectricity. Many naturally occurring substances show these properties which can be broadly classified as crystals, bone and biological materials. Synthetic materials like ceramics, crystals, III-V and II-VI semiconductors, lead-free ceramics and polymers also show piezoelectric effects.

Quartz, Berlinite ($AlPO_4$), a rare phosphate mineral that is structurally identical to quartz, Sucrose (table sugar), Rochelle salt, Topaz, Tourmaline-group minerals and Lead titanate ($PbTiO_3$) (naturally occurring as mineral macedonite) are some of the naturally occurring crystals that exhibit piezoelectricity.

Dry bone exhibits some piezoelectric properties. These are not due to the apatite crystals, which are centrosymmetric and thus non-piezoelectric, but due to collagen which exhibits the polar uniaxial orientation of molecular dipoles in its structure and can be considered as bioelectret, a sort of dielectric material exhibiting quasi-permanent space charge and dipolar charge. When a number of collagen molecules are stressed in the same way displacing significant numbers of the charge carriers from the inside to the surface, potentials are thought to occur.

DNA, Tendon, Enamel, Silk, Dentin, Wood and Viral proteins are biological materials which show piezoelectric effect.

Ceramics with randomly oriented grains must be ferroelectric to exhibit piezoelectricity. The macroscopic piezoelectricity is possible in textured polycrystalline non–ferroelectric piezoelectric materials, such as AlN and ZnO. The family of ceramics with perovskite, tungsten-bronze and related structures exhibits piezoelectricity

Synthetic crystals comprise of Langasite ($La_3Ga_5SiO_{14}$), a quartz analogic crystal, Lithium niobate ($LiNbO_3$), Gallium orthophosphate ($GaPO_4$), a quartz analogic crystal and Lithium tantalate ($LiTaO_3$).

Barium titanate ($BaTiO_3$) was the first synthetic piezoelectric ceramic discovered. Lead zirconate titanate ($Pb[Zr_xTi_{1-x}]O_3$ $0 \leq x \leq 1$), more commonly known as PZT, is the most common piezoelectric ceramic in use today. Other ceramics include Potassium niobate ($KNbO_3$), Sodium tungstate (Na_2WO_3), $Pb_2KNb_5O_{15}$, $Ba_2NaNb_5O_5$ and Zinc oxide (ZnO) (Wurtzite structure).

Zinc-oxide (ZnO) is an interesting material that is pushing the piezoelectric field to a nanometric scale. It is used to grow one dimensional hair-like nanowires, with diameters in the sub-one hundred nanometre scale and lengths ranging from several hundreds of nanometres to a few centimetres. Zinc exhibits both semiconductor and piezoelectric properties, it is relatively bio safe and biocompatible, so it can be involved in biomedical applications with little toxicity **[5]**.

There is growing concern regarding the toxicity in lead-containing devices driven by the result of restriction of hazardous substances directive regulations. To address this concern, there has been a resurgence in the compositional development of lead-free piezoelectric materials.

Bismuth ferrite ($BiFeO_3$), Sodium potassium niobate ($(K,Na)NbO_3$), Sodium niobate $NaNbO_3$, Bismuth titanate $Bi_4Ti_3O_{12}$ and Sodium bismuth titanate $Na_{0.5}Bi_{0.5}TiO_3$ are lead-free piezo ceramics.

A piezoelectric potential can be created in any bulk or nanostructured semiconductor crystal having non central symmetry, such as the Group III-V and II-VI materials, due to polarization of ions under applied stress and strain. This property is common to both the zincblende and wurtzite crystal structures.

Polyvinylidene fluoride (PVDF) exhibits piezoelectricity several times greater than quartz. Unlike ceramics, where the crystal structure of the material creates the piezoelectric effect, in polymers the intertwined long-chain molecules attract and repel each other when an electric field is applied.

Table 1.1 shows the classification of the different types of piezoelectric materials found.

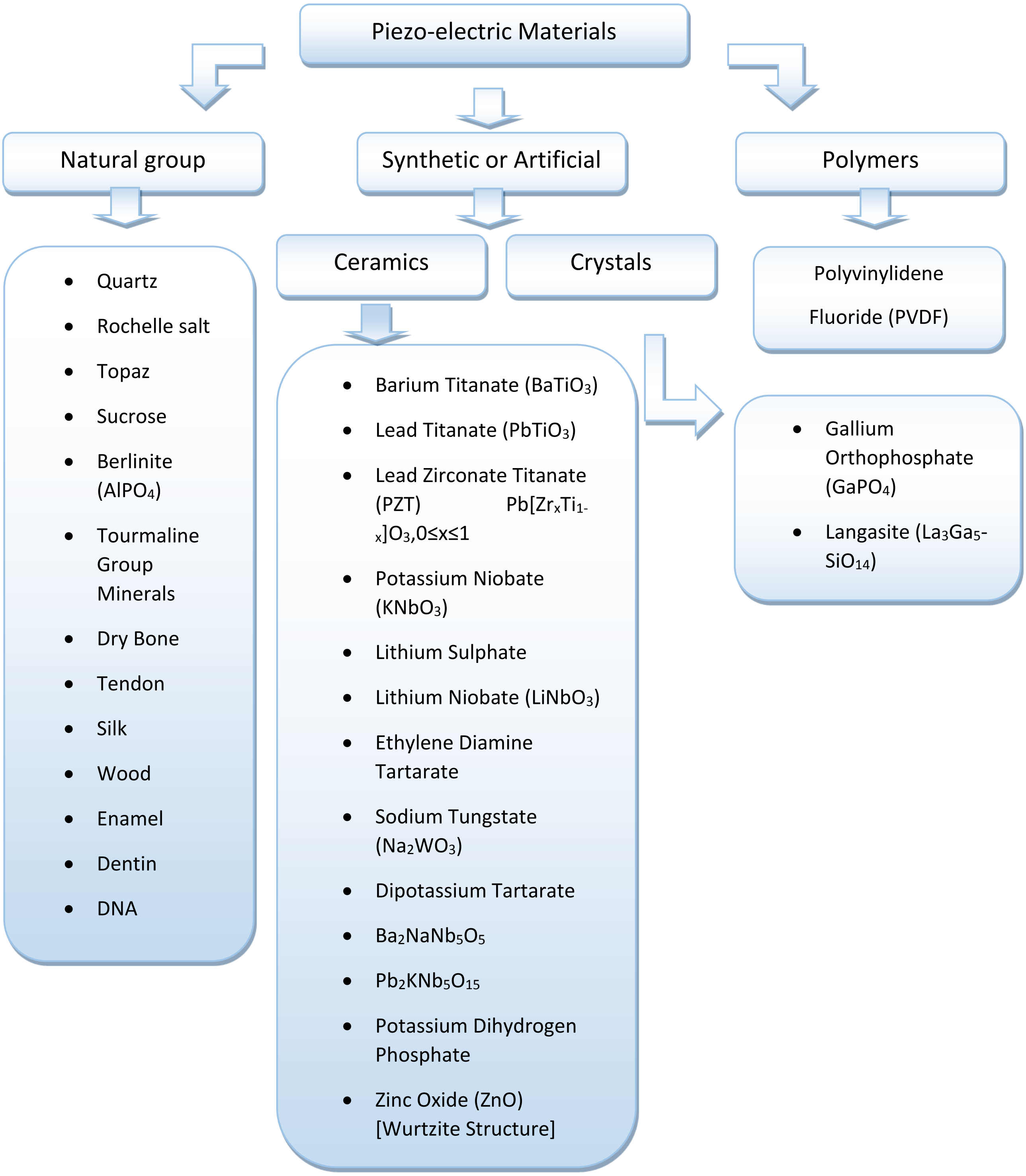

Table 1.1: Classification of piezo-electric materials

1.3 MECHANISM

A piezoelectric substance is one that produces an electric charge when a mechanical stress is applied (the substance is compressed or stretched) on it. Conversely, a mechanical deformation of the substance is produced when an electric field is applied across it. It is based on the fundamental structure of a crystal lattice. Certain crystalline structures have a charge balance with negative and positive polarization, which neutralize along the imaginary polar axis. When this charge balance is perturbed with external stress onto the crystal mesh, the energy is transferred by electric charge carriers creating a current in the crystal. Conversely, with the piezoelectric effect an external charge input will create an unbalance in the neutral charge state causing mechanical stress.

The connection between piezoelectricity and crystal symmetry are closely established. This effect is formed in crystals that have no centre of symmetry. This relationship can be explained with monocrystal and polycrystalline structures.

In a monocrystal (Figure 1.1) the polar axes of all of the charge carriers exhibit one-way directional characteristics. These crystals demonstrate symmetry, where the polar axes throughout the crystal would lie unidirectional even if it was split into pieces.

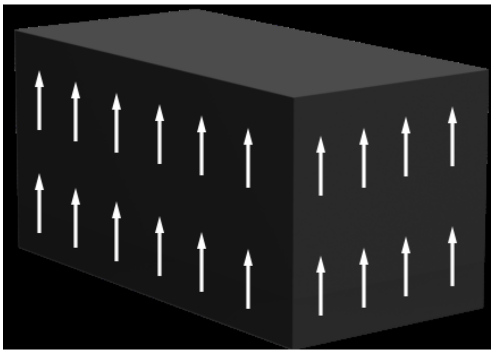

Figure 1.1: Monocrystal

Instead, a polycrystal (Figure 1.2) is characterized by different regions within the material with different polar axes. It is asymmetrical because there is no point at which the crystal could be cut that would leave the two remaining pieces with the same resultant polar axes.

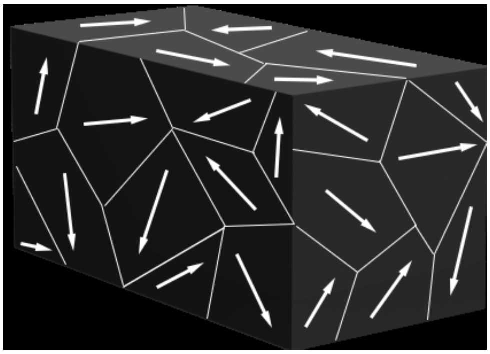

Figure 1.2: Polycrystal

In order to attain the piezoelectric effect, the polycrystal is heated to the Curie point along with strong electric field. The heat allows the molecules to move more freely and the electric field forces the dipoles to rearrange in accordance with the external field (Figure 1.3).

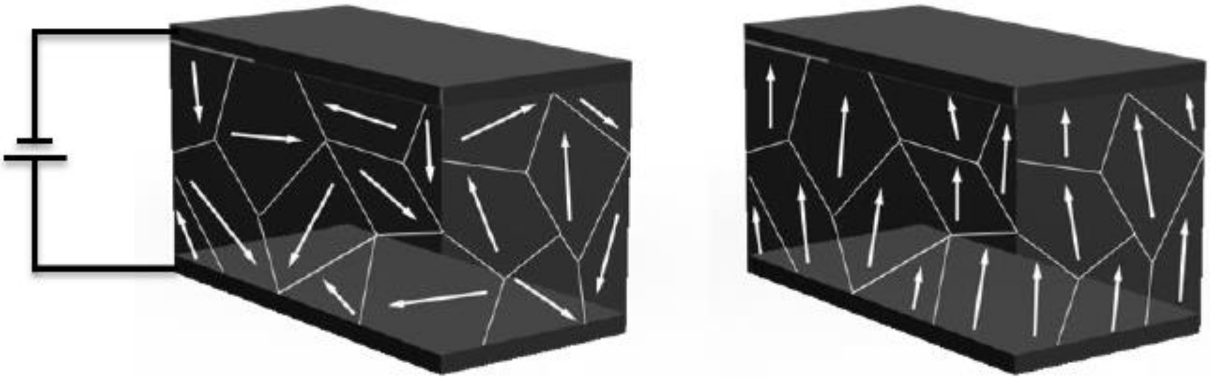

Figure 1.3: (a) Polarizations (b) Surviving Polarity

As a result, the material possesses piezoelectric effect: a voltage of the same polarity as of the poling voltage appears between electrodes when the material is compressed; and opposite polarity appears when stretched. Material deformation takes place when a voltage difference is applied, and if an AC signal is applied the material will vibrate at the same frequency as the signal. This is illustrated in figure 1.4.

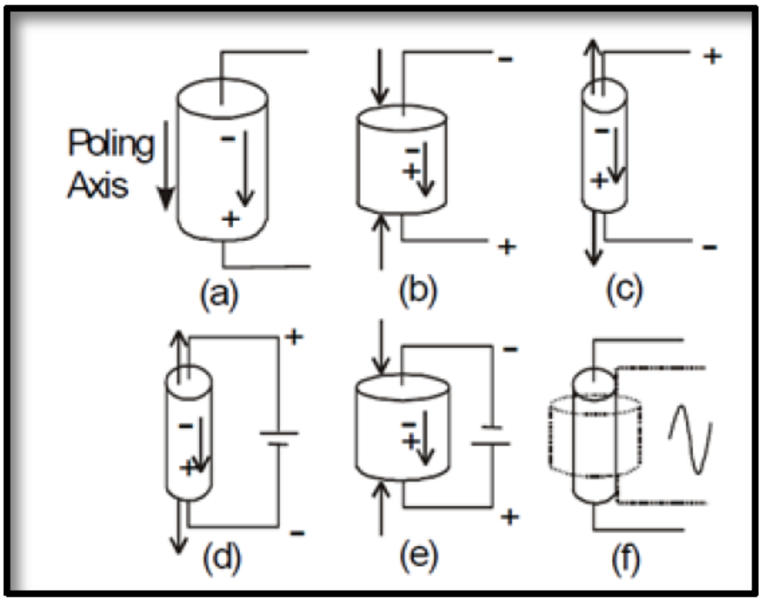

Figure 1.4: Illustration of Piezoelectric Effect

Figure 1.4 (a) shows a piezoelectric material without any stress or charge. If the material is compressed, then a voltage of the same polarity as the poling voltage will appear between the electrodes (b). If stretched, a voltage of opposite polarity will appear (c). A voltage with the opposite polarity as the poling voltage will cause the material to expand, (d), and a voltage with the same polarity will cause the material to compress (e). If an AC signal is applied then the material will vibrate at the same frequency as the signal (f) **[1-5]**.

1.4 EQUIVALENT CIRCUIT OF A PIEZO HARVESTER

An input vibration applied on to a piezoelectric material as shown in Figure 1.5 causes mechanical strain to develop in the device which is converted to electrical charge **[6]**. Lead-zirconate-titanate (PZT) is a commonly used piezoelectric material for power generation. The equivalent circuit of the piezoelectric harvester can be represented as a mechanical spring mass system coupled to an electrical domain as shown in Figure 3. Here, LM represents the mechanical mass, CM the mechanical stiffness and RM takes into account the mechanical losses. The mechanical domain is coupled to the electrical domain through a transformer that converts strain to current. On the electrical side, Cp represents the plate capacitance of the piezoelectric material. At or close to resonance, the whole circuit can be transformed to the electrical domain, where the piezoelectric element when excited by sinusoidal vibrations can be modelled as a sinusoidal current source in parallel with a capacitance Cp and resistance Rp.

One of the challenges in a power generator of this type is the design and construction of an efficient power conversion circuit to harvest the energy from the PZT membrane. Another unique characteristic of this power source is that it outputs relatively low output voltages for the low levels of input vibration typically encountered in ambient conditions. This low output voltage makes it challenging to develop rectifier circuits that are efficient since many diode rectifiers require nonzero turn-on voltages to operate.

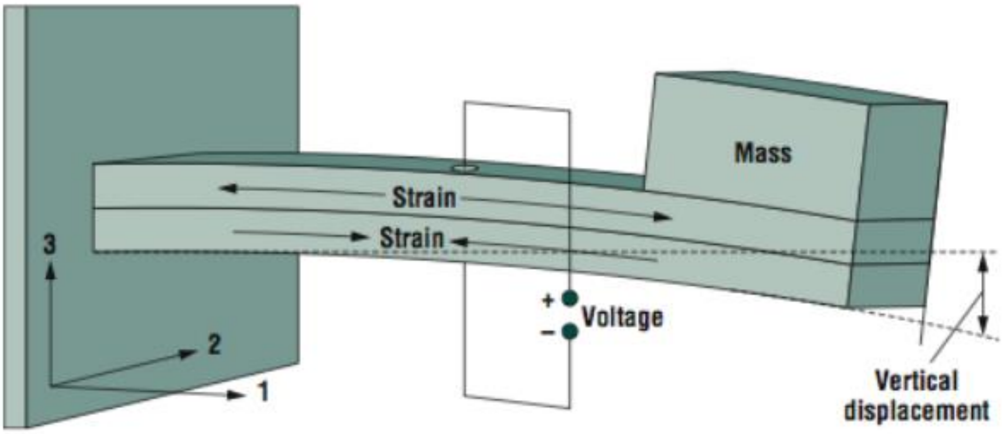

Figure 1.5: Working of a piezo harvester **[17]**

1.5 STORING AND INTERFACING

1.5.1 STORAGE DEVICES

The energy generated from piezoelectric devices is extremely low, and can be stored in temporary power storage devices such as capacitors or rechargeable batteries. Supercapacitors are being investigated as low power storage devices, as they offer higher energy densities than regular capacitors. Their performances are comparable to that of Lithium-Ion batteries and have better characteristics, such as cycle life (the number of times it can be charged and discharged). There has been less implementation of supercapacitors as energy storage devices. But there can be more to utilization of supercapacitors as storage devices with piezoelectric generation. **[7]**

1.5.2 STANDARD AC-DC INTERFACE

The standard AC-DC interface is one of the simplest ways to generate electric voltage from a piezoelectric element. The electrical energy supplied by a piezoelectric element is an AC voltage, whereas the battery connected to the element requires a steady DC voltage. To ensure that the electrical circuit has proper compatibility, an AC-DC interface is installed between the piezoelectric element and the battery. The AC-DC interface consists of a bridge rectifier, and a controller. The function of a bridge rectifier is to achieve full-wave rectification. The controller that is installed is generally a DC-DC converter, whose function is to optimize the power being delivered to the battery as well as regulate the voltage, such that the voltage being delivered is suited to the requirements of the battery (Figure 1.6) **[7][8]**

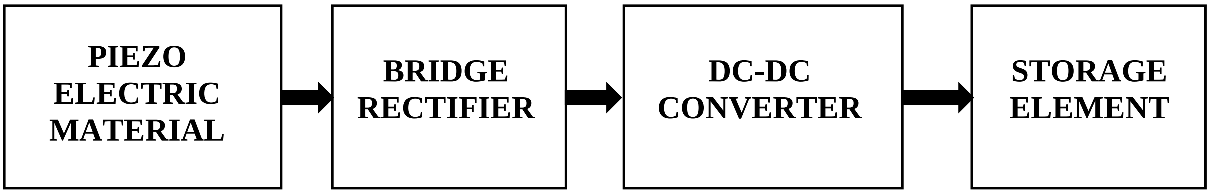

Figure 1.6: Standard Electrical Interface

1.5.3 DC-DC CONVERTERS

The energy produced by a piezoelectric material is low compared to other forms of energy harvesting techniques. The efficiency obtained by the DC-DC converter when used in low power applications is high. Efficiencies can reach the range of 80%-99%. Another unique characteristic of the DC-DC converters are dynamic controlling of power and voltage regulation. **[7][8]**

1.6 PZT (LEAD ZIRCONATE TITANATE)

PZT, or lead zirconate titanate ($Pb[Zr_xTi_{1-x}]O_3$), is one of the world's most widely used piezoelectric ceramic materials. When fired, PZT has a perovskite crystal structure, each unit of which consists of a small tetravalent metal ion in a lattice of large divalent metal ions. In the case of PZT, the small tetravalent metal ion is usually titanium or zirconium. The large divalent metal ion is usually lead. Under conditions that confer a tetragonal or rhombohedral symmetry on the PZT crystals, each crystal has a dipole moment **[9]**.

PZT, lead zirconate titanate, is the most commonly used piezo ceramic today. In general, piezo ceramics are the preferred choice because they are physically strong, chemically inert and relatively inexpensive to manufacture. Plus, they can be easily tailored to meet the requirements of a specific purpose. PZT ceramic is revered because it has an even greater sensitivity and higher operating temperature than other piezo ceramics.

A variety of methods have been developed to synthesise mixed-oxide ceramic powders **[10]**. These methods have become available for both laboratory and industrial production. The different techniques of PZT synthesis are Solid-state Reaction, Coprecipitation, Sol-gel, Spray/Freeze Drying Spray, Pyrolysis, Emulsion Synthesis and Hydrothermal Synthesis. Most of them have been used to make PZT powders. A general comparison of the synthesis routes for oxide ceramic powders is listed in Table 1.1.

Synthesis Method	**State of Development**	**Compositional Control**	**Morphological Control**	**Purity (%)**	**Costs**
Solid-state reaction	Commercial	Poor	Poor	<99.5	Low-Moderate
Coprecipitation	Commercial	Good	Moderate	>99.5	Moderate
Sol-gel	R&D	Excellent	Moderate	>99.9	Moderate-High
Spray/Freeze Drying	Demonstration	Excellent	Moderate	>99.9	Moderate-High
Spray Pyrolysis	R&D	Excellent	Excellent	>99.9	High
Emulsion Pyrolysis	Demonstration	Excellent	Excellent	>99.9	Moderate
Hydrothermal Synthesis	Demonstration	Excellent	Good	>99.9	Moderate

Table 1.2: Oxide powder synthesis route comparison

1.7 APPLICATIONS

Piezoelectric materials have a wide range of applications. They can be used as high voltage and power sources, sensors, actuators, frequency standard, piezoelectric motors, reduction of vibrations and noise, infertility treatment, surgery and other potential applications.

1.7.1 HIGH VOLTAGE AND POWER SOURCES

Direct piezoelectricity of some substances, like quartz, can generate potential differences of thousands of volts. The electric cigarette lighter is the best-known application: pressing the button causes a spring-loaded hammer to hit a piezoelectric crystal, producing a sufficiently high voltage electric current that flows across a small spark gap, thus heating and igniting the gas. The portable sparkers used to ignite gas stoves work the same way, and many types of gas burners now have built-in piezo-based ignition systems.

A similar idea is being researched by DARPA in the United States in a project called Energy Harvesting, which includes an attempt to power battlefield equipment by piezoelectric generators embedded in soldiers' boots. However, these energy harvesting sources by association have an impact on the body. DARPA's effort to harness 1–2 watts from continuous shoe impact while walking were abandoned due to the impracticality and the discomfort from the additional energy expended by a person wearing the shoes.

The pressure exerted by a person while walking can converted into electrical energy to power portable devices. This is done by embedding a moonie harvester **[4] [11]** into a shoe. The "Moonie,' is a metal ceramic composite transducer that has been developed by sandwiching a poled lead zirconatetitanate (PZT) ceramic between two specially designed metal end caps. The operating principle of the moonie harvester is also shown in Figure 1.6. The structure serves as an amplifier for the input force which, in this case, is the weight of the person wearing the shoe. The force on the heel presses the curved plates which in turn expand the piezoelectric disk sandwiched in between the steel plates. The stress is evenly distributed on the disk as opposed to beam structures where the majority of the stress is located at the fixed end of the beam.

The energy output of one step was recorded as 81 μJ which translates to 162 μW for two shoes when walking 2 steps per second. The power density at 1 step / s frequency was measured as

56 μW/cm^3. The size of the piezo element was 17.5 mm in diameter and the thickness was 500 μm. The material used was PZT-5H.

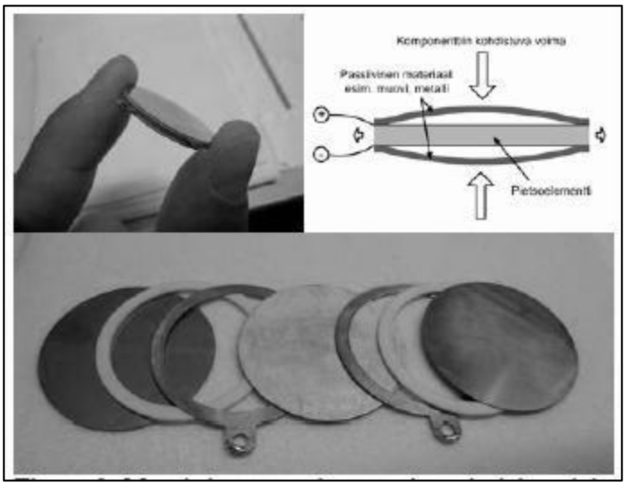

Figure 1.6: Moonie harvester

Other energy harvesting ideas include harvesting the energy from human movements in train stations or other public places and converting a dance floor to generate electricity. Vibrations from industrial machinery can also be harvested by piezoelectric materials to charge batteries for backup supplies or to power low-power microprocessors and wireless radios.

Airports and railways are vital transportation hubs that will greatly benefit from new energy technologies. Lower costs along with cleaner day-to-day operations from green forms of energy will allow airports to operate more efficiently and effectively. One such idea that will fit well in such setting is the capturing of kinetic energy from passenger foot traffic. This novel idea is not only clean but it is also renewable.

Using the floor space in airport terminals allows for a large source of otherwise wasted energy to be captured and utilized as an alternative form of energy for the lighting systems within airports **[12]**. Placing piezoelectric devices that are used to capture energy from foot traffic underneath airport terminals can effectively capture electrical energy and send it back to the power grid through inverters, which are needed in order to convert the DC power, from the piezoelectric, into AC power used by terminal lighting systems.

The East Japan Railway Company (JR East) conducted a demonstration experiment from January 19 to March 7, 2008, at Yaesu North Gate, Tokyo Station, on a new power-generating floor. Installed at the ticket gate area, it generates electricity from the dynamic pressure created by passengers walking through the ticket gates.

The power-generating floor is embedded with piezoelectric elements, which are 35 millimeters in diameter, and disc-shaped components used for loudspeakers. It uses 600 of these elements per square meter. While the loudspeaker creates sound by converting electric signals to vibrations, the floor adopts the reverse mechanism that produces electricity by harnessing the vibrational power generated from passengers' steps. It is being developed by JR East with the aim of making stations more environmentally friendly and energy efficient (Japan for Sustainability, 2008).

The piezo devices, due to their small thin shape, could be placed underneath floor tiles or carpet with few complications. In order to harness the power a capacitor could be used to store the electricity like in the train stations or inverters. The power could then be routed directly to specific electrical devices such as lights or billboards or it could be sent to the main power grid in order to supplement the main power supply. There are many installation options and applications of these devices; the specific type of installation will depend upon the intended use of the piezo devices within the terminals. Experimentation with different areas and by observing locations of high foot traffic in such terminals are important in determining the optimal locations for capturing kinetic energy from walking.

A piezoelectric transformer is a type of AC voltage multiplier. Unlike a conventional transformer, which uses magnetic coupling between input and output, the piezoelectric transformer uses acoustic coupling. An input voltage is applied across a short length of a bar of piezoceramic material such as PZT, creating an alternating stress in the bar by the inverse piezoelectric effect and causing the whole bar to vibrate. The vibration frequency is chosen to be the resonant frequency of the block, typically in the 100 kilohertz to 1 megahertz range. A higher output voltage is then generated across another section of the bar by the piezoelectric effect. Step-up ratios of more than 1000:1 have been demonstrated. An extra feature of this transformer is that, by operating it above its resonant frequency, it can be made to appear as an inductive load, which is useful in circuits that require a controlled soft start. These devices can be used in DC-AC inverters to drive cold cathode fluorescent lamps. Piezo transformers are some of the most compact high voltage sources.

1.7.2 SENSORS

The principle of operation of a piezoelectric sensor is that a physical dimension, transformed into a force, acts on two opposing faces of the sensing element. Depending on the design of a sensor, different "modes" to load the piezoelectric element can be used: longitudinal, transversal and shear. Detection of pressure variations in the form of sound is the most common sensor application, e.g. piezoelectric microphones (sound waves bend the piezoelectric material, creating a changing voltage) and piezoelectric pickups for acoustic-electric guitars. A piezo sensor attached to the body of an instrument is known as a contact microphone.

Piezoelectric sensors especially are used with high frequency sound in ultrasonic transducers for medical imaging and also industrial nondestructive testing (NDT).

For many sensing techniques, the sensor can act as both a sensor and an actuator – often the term transducer is preferred when the device acts in this dual capacity, but most piezo devices have this property of reversibility whether it is used or not. Ultrasonic transducers, for example, can inject ultrasound waves into the body, receive the returned wave, and convert it to an electrical signal (a voltage). Most medical ultrasound transducers are piezoelectric.

In addition to those mentioned above, various sensor applications include:

- Piezoelectric elements are also used in the detection and generation of sonar waves.
- Piezoelectric materials are used in single-axis and dual-axes tilt sensing.
- Power monitoring in high power applications (e.g. medical treatment, sonochemistry and industrial processing).
- Piezoelectric microbalances are used as very sensitive chemical and biological sensors.
- Piezoelectric materials are sometimes used in strain gauges.
- A piezoelectric transducer was used in the penetrometer instrument on the Huygens Probe
- Piezoelectric transducers are used in electronic drum pads to detect the impact of the drummer's sticks, and to detect muscle movements in medical acceleromyography.
- Automotive engine management systems use piezoelectric transducers to detect Engine knock (Knock Sensor, KS), also known as detonation, at certain hertz frequencies. A piezoelectric transducer is also used in fuel injection systems to measure manifold absolute pressure (MAP sensor) to determine engine load, and ultimately the fuel injectors milliseconds of on time.

- Ultrasonic piezo sensors are used in the detection of acoustic emissions in acoustic emission testing.

1.7.3 ACTUATORS

As very high electric fields correspond to only tiny changes in the width of the crystal, this width can be changed with better-than-μm precision, making piezo crystals the most important tool for positioning objects with extreme accuracy — thus their use in actuators. Multilayer ceramics, using layers thinner than 100 μm, allow reaching high electric fields with voltage lower than 150 V. These ceramics are used within two kinds of actuators: direct piezo actuators and Amplified piezoelectric actuators. While direct actuator's stroke is generally lower than 100 μm, amplified piezo actuators can reach millimetre strokes.

- Loudspeakers: Voltage is converted to mechanical movement of a metallic diaphragm.
- Piezoelectric motors: Piezoelectric elements apply a directional force to an axle, causing it to rotate. Due to the extremely small distances involved, the piezo motor is viewed as a high-precision replacement for the stepper motor.
- Piezoelectric elements can be used in laser mirror alignment, where their ability to move a large mass (the mirror mount) over microscopic distances is exploited to electronically align some laser mirrors. By precisely controlling the distance between mirrors, the laser electronics can accurately maintain optical conditions inside the laser cavity to optimize the beam output.
- A related application is the acousto-optic modulator, a device that scatters light off soundwaves in a crystal, generated by piezoelectric elements. This is useful for fine-tuning a laser's frequency.
- Atomic force microscopes and scanning tunneling microscopes employ converse piezoelectricity to keep the sensing needle close to the specimen.
- Inkjet printers: On many inkjet printers, piezoelectric crystals are used to drive the ejection of ink from the inkjet print head towards the paper.
- Diesel engines: High-performance common rail diesel engines use piezoelectric fuel injectors, first developed by Robert Bosch GmbH, instead of the more common solenoid valve devices.
- Active vibration control using amplified actuators.
- X-ray shutters.

- XY stages for micro scanning used in infrared cameras.
- Moving the patient precisely inside active CT and MRI scanners where the strong radiation or magnetism precludes electric motors.
- Crystal earpieces are sometimes used in old or low power radios.
- High-intensity focused ultrasound for localized heating or creating a localized Cavitation can be achieved, for example, in patient's body or in an industrial chemical process

1.7.4 FREQUENCY STANDARD

The piezo electrical properties of quartz are useful as a standard of frequency.

- Quartz clocks employ a crystal oscillator made from a quartz crystal that uses a combination of both direct and converse piezoelectricity to generate a regularly timed series of electrical pulses that is used to mark time. The quartz crystal (like any elastic material) has a precisely defined natural frequency (caused by its shape and size) at which it prefers to oscillate, and this is used to stabilize the frequency of a periodic voltage applied to the crystal.
- The same principle is critical in all radio transmitters and receivers, and in computers where it creates a clock pulse. Both of these usually use a frequency multiplier to reach gigahertz ranges.

1.7.5 PIEZOELECTRIC MOTORS

Types of piezoelectric motor include:

- The traveling-wave motor used for auto-focus in reflex cameras
- Inchworm motors for linear motion
- Rectangular four-quadrant motors with high power density (2.5 W/cm^3) and speed ranging from 10 nm/s to 800 mm/s.
- Stepping piezo motor, using stick-slip effect.

Aside from the stepping stick-slip motor, all these motors work on the same principle. Driven by dual orthogonal vibration modes with a phase difference of 90°, the contact point between two surfaces vibrates in an elliptical path, producing a frictional force between the surfaces. Usually, one surface is fixed, causing the other to move. In most piezoelectric motors, the piezoelectric crystal is excited by a sine wave signal at the resonant frequency of the motor. Using the resonance effect, a much lower voltage can be used to produce a high vibration amplitude.

A stick-slip motor works using the inertia of a mass and the friction of a clamp. Such motors can be very small. Some are used for camera sensor displacement, thus allowing an anti-shake function.

1.7.6 REDUCTION OF VIBRATIONS AND NOISE

Different teams of researchers have been investigating ways to reduce vibrations in materials by attaching piezo elements to the material. When the material is bent by a vibration in one direction, the vibration-reduction system responds to the bend and sends electric power to the piezo element to bend in the other direction. Future applications of this technology are expected in cars and houses to reduce noise. Further applications to flexible structures, such as shells and plates, have also been studied for nearly three decades.

In a demonstration at the Material Vision Fair in Frankfurt in November 2005, a team from TU Darmstadt in Germany showed several panels that were hit with a rubber mallet, and the panel with the piezo element immediately stopped swinging.

Piezoelectric ceramic fibre technology is being used as an electronic damping system on some HEAD tennis rackets.

1.7.7 INFERTILITY TREATMENT

In people with previous total fertilization failure, piezoelectric activation of oocytes together with intracytoplasmic sperm injection (ICSI) seems to improve fertilization outcomes.

1.7.8 SURGERY

A recent application of piezoelectric ultrasound sources is piezoelectric surgery, also known as piezosurgery. Piezosurgery is a minimally invasive technique that aims to cut a target tissue with little damage to neighbouring tissues. For example, Hoigne et al. **[13]** reported its use in hand surgery for the cutting of bone, using frequencies in the range 25–29 kHz, causing microvibrations of 60–210 μm. It has the ability to cut mineralized tissue without cutting neurovascular tissue and other soft tissue, thereby maintaining a blood-free operating area, better visibility and greater precision.

1.7.9 OTHER POTENTIAL APPLICATIONS

When a piezo ceramic transducer is stressed mechanically by a force, its electrodes receive a charge that tends to counteract the imposed strain. This charge may be collected, stored and delivered to power electrical circuits or processors.

In 2015, Cambridge University researchers working in conjunction with researchers from the National Physical Laboratory and Cambridge-based dielectric antenna company Antenova Ltd, using thin films of piezoelectric materials found that at a certain frequency, these materials become not only efficient resonators, but efficient radiators as well, meaning that they can potentially be used as antennas. The researchers found that by subjecting the piezoelectric thin films to an asymmetric excitation, the symmetry of the system is similarly broken, resulting in a corresponding symmetry breaking of the electric field, and the generation of electromagnetic radiation.

In recent years, several attempts at the macro-scale application of the piezoelectric technology have emerged to harvest kinetic energy from walking pedestrians. The piezoelectric floors have been trialed since the beginning of 2007 in two Japanese train stations, Tokyo and Shibuya stations. The electricity generated from the foot traffic is used to provide all the electricity needed to run the automatic ticket gates and electronic display systems. In London, a famous nightclub exploited the piezoelectric technology in its dance floor. Parts of the lighting and sound systems in the club can be powered by the energy harvesting tiles. However, the piezoelectric tile deployed on the ground usually harvests energy from low frequency strikes provided by the foot traffic. This working condition may eventually lead to low power generation efficiency.

In this case, locating high traffic areas is critical for optimization of the energy harvesting efficiency, as well as the orientation of the tile pavement significantly affects the total amount of the harvested energy. A Density Flow evaluation is recommended to qualitatively evaluate the piezoelectric power harvesting potential of the considered area based on the number of pedestrian crossings per unit time. In X. Li's study, the potential application of a commercial piezoelectric energy harvester in a central hub building at Macquarie University in Sydney, Australia is examined and discussed **[15]**. Optimization of the piezoelectric tile deployment is presented according to the frequency of pedestrian mobility and a model is developed where 3.1% of the total floor area with the highest pedestrian mobility is paved with piezoelectric tiles. The modelling results indicate that the total annual energy harvesting potential for the proposed optimized tile pavement model is estimated at 1.1 MW h/year, which would be sufficient to meet close to 0.5% of the annual energy needs of the building. In Israel, there is a company which has installed piezoelectric materials under a busy highway. The energy generated is adequate and powers street lights, billboards and signs.

Tyre Company Goodyear has plans to develop an electricity generating tyre which has piezoelectric material lined inside it. As the tyre moves, it deforms and thus electricity is generated **[16]**.

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CHAPTER 2

REVIEW OF PAST WORK

2.1 INTRODUCTION

The word ‘piezo’ is derived from the Greek word for pressure. The piezoelectric effect was discovered by Jacques and Pierre Curie in 1880. They found that pressure applied to a quartz crystal creates an electric charge in the crystal, a phenomenon they referred to as the (direct) piezoelectric effect. Later they also verified that an electric field applied to the crystal leads to a deformation of the material: the inverse piezoelectric effect. In the subsequent century, research has been performed into the development of materials with improved piezoelectric properties, enabling commercial utilization of the piezoelectric phenomenon. To date, the number of applications of piezoelectric materials is still increasing.

Piezo-electric materials have become very popular for last fifty years due to its wide frequency range, high stability, high output impedance, ruggedness, very large dynamic range. The main advantages of this type of material are low weight, compact, less immune to electromagnetic noise. PZT has been studied in the past thirty years and research work is still continuing. It can be used as sensors as well as actuators. **[1]**

In the scientific circles of the day, this effect was considered quite a "discovery," and was quickly dubbed as "piezoelectricity" in order to distinguish it from other areas of scientific phenomenological experience such as "contact electricity" (friction generated static electricity) and "pyroelectricity" (electricity generated from crystals by heating).

The Curie brothers asserted, however, that there was a one-to-one correspondence between the electrical effects of temperature change and mechanical stress in a given crystal, and that they had used this correspondence not only to pick the crystals for the experiment, but also to determine the cuts of those crystals. To them, their demonstration was a confirmation of predictions which followed naturally from their understanding of the microscopic crystallographic origins of pyroelectricity (i.e., from certain crystal asymmetries).

The Curie brothers did not, however, predict that crystals exhibiting the direct piezoelectric effect (electricity from applied stress) would also exhibit the converse piezoelectric effect (stress in response to applied electric field). This property was mathematically deduced from fundamental thermodynamic principles by Lippmann in 1881. The Curies immediately confirmed the existence of the "converse effect," and continued on to obtain quantitative proof of the complete reversibility of electro-elasto-mechanical deformations in piezoelectric crystals.

For the next few decades, piezoelectricity remained something of a laboratory curiosity. At this point in time, after only two years of interactive work within the European scientific community, the core of piezoelectric applications science was established: the identification of piezoelectric crystals on the basis of asymmetric crystal structure, the reversible exchange of electrical and mechanical energy, and the usefulness of thermodynamics in quantifying complex relationships among mechanical, thermal and electrical variables.

More work was done to explore and define the crystal structures that exhibited piezoelectricity. This culminated in 1910 with the publication of Woldemar Voigt's Lehrbuch der Kristallphysik (Textbook on Crystal Physics), which described the 20 natural crystal classes capable of piezoelectricity, and rigorously defined the piezoelectric constants using tensor analysis.

During the 25 years that it took to reach Voigt's benchmark, however, the world was not holding its breath for piezoelectricity. A science of such subtlety as to require tensorial analysis just to define relevant measurable quantities paled by comparison to electro-magnetism, which at the time was maturing from a science to a technology, producing highly visible and amazing machines. Piezoelectricity was obscure even among crystallographers; the mathematics required to understand it was complicated; and no publicly visible applications had been found for any of the piezoelectric crystals.

The first practical application for piezoelectric devices was sonar, first developed during World War I. In France in 1917, Paul Langevin and his coworkers developed an ultrasonic submarine detector. The detector consisted of a transducer, made of thin quartz crystals carefully glued between two steel plates, and a hydrophone to detect the returned echo. By emitting a high-frequency pulse from the transducer, and measuring the amount of time it takes to hear an echo from the sound waves bouncing off an object, one can calculate the distance to that object.

The use of piezoelectricity in sonar, and the success of that project, created intense development interest in piezoelectric devices. Over the next few decades, new piezoelectric materials and new applications for those materials were explored and developed.

Piezoelectric devices found homes in many fields. Ceramic phonograph cartridges simplified player design, were cheap and accurate, and made record players cheaper to maintain and easier to build. The development of the ultrasonic transducer allowed for easy measurement of viscosity and elasticity in fluids and solids, resulting in huge advances in materials research. Ultrasonic time-domain reflectometers (which send an ultrasonic pulse through a material and measure reflections from discontinuities) could find flaws inside cast metal and stone objects, improving structural safety.

During World War II, independent research groups in the United States, Russia, and Japan discovered a new class of synthetic materials, called ferroelectrics, which exhibited piezoelectric constants many times higher than natural materials. This led to intense research to develop barium titanate and later lead zirconate titanate materials with specific properties for particular applications **[2]**.

One significant example of the use of piezoelectric crystals was developed by Bell Telephone Laboratories. Following World War I, Frederick R. Lack, working in radio telephony in the engineering department, developed the “AT cut” crystal, a crystal that operated through a wide range of temperatures. Lack's crystal didn't need the heavy accessories previous crystal used, facilitating its use on aircraft. This development allowed Allied air forces to engage in coordinated mass attacks through the use of aviation radio.

Development of piezoelectric devices and materials in the United States was kept within the companies doing the development, mostly due to the wartime beginnings of the field, and in the interests of securing profitable patents. New materials were the first to be developed — quartz crystals were the first commercially exploited piezoelectric material, but scientists searched for higher-performance materials. Despite the advances in materials and the maturation of manufacturing processes, the United States market did not grow as quickly as

Japan's did. Without many new applications, the growth of the United States' piezoelectric industry suffered.

In contrast, Japanese manufacturers shared their information, quickly overcoming technical and manufacturing challenges and creating new markets. In Japan, a temperature stable crystal cut was developed by Issac Koga. Japanese efforts in materials research created piezoceramic materials competitive to the U.S. materials but free of expensive patent restrictions. Major Japanese piezoelectric developments included new designs of piezoceramic filters for radios and televisions, piezo buzzers and audio transducers that can connect directly to electronic circuits, and the piezoelectric igniter, which generates sparks for small engine ignition systems (and gas-grill lighters) by compressing a ceramic disc. Ultrasonic transducers that transmit sound waves through air had existed for quite some time but first saw major commercial use in early television remote controls. These transducers now are mounted on several car models as an echolocation device, helping the driver determine the distance from the rear of the car to any objects that may be in its path.

2.2 LITERATURE SURVEY

San-Yuan Chen et al. used two solution-based methods, metallo-organic decomposition and sol–gel processes to study the effects of precursor solution type on the microstructure evolution and texture development of oriented PZT films **[3]**. Microstructure development and perovskite content are strongly dependent on the heating rate. Fast heating rate forms a dense fine-grained microstructure with (111) orientation. Intermediate-temperature pyrolysis followed by a fast heating rate forms clustered or island structures of submicrometer grains with (100) orientation. Intermediate temperature pyrolysis followed by a very slow heating rate forms larger spherical rosettes with random orientations. Pt5–7Pb is a (111) textured transient intermetallic phase that nucleates PZT (111) texture. PbO is a (001) textured layer compound that nucleates PZT (100) texture. The texture selection of PZT films is independent of precursor systems but sensitive to the film thickness especially when sol–gel precursors and oxidizing atmosphere are used. Correlation and comparison of oriented sol–gel and MOD PZT films with electrical properties are also made.

A wide variety of preparation techniques have been employed to produce Pb $(Zr_{1-x}Ti_x)$ O_3 (PZT) thin films. Among those methods, the solution-based methods to fabricate ferroelectric thin films offer numerous advantages, including low processing temperature, excellent compositional control, uniform homogeneity, ease of fabrication over large areas, and low cost. Films prepared by such methods have displayed ferroelectric properties comparable to those of bulk ceramics.

The texture selection of PZT films can be controlled through similar heating schedules independent of precursor systems. The texture selection is sensitive to the film thickness especially when sol–gel precursors and oxidizing atmosphere are used. This is related to the lower carbon content of sol–gel. Microstructure development and perovskite content are strongly dependent on the heat treatment, especially on the heating rate. Fast heating rate forms a dense fine-grained microstructure with (111) orientation. Intermediate-temperature pyrolysis followed by a fast heating rate forms clustered or island structures of submicrometer grains with (100) orientation. Intermediate-temperature pyrolysis followed by a very slow heating rate forms larger spherical rosettes with random orientations. The amount of perovskite decreases in the above order and is especially low in sol–gel films undergoing slower heating. This is because of the higher crystallization temperature in the sol–gel films, which in turn suffer more PbO loss.

R. Seveno et al. prepared $PbZr_{0.45}Ti_{0.55}O_3$ ferroelectric films by sol-gel method, using alkoxide precursor compounds and multi-layer technique **[4]**. The gel films were deposited by spin-coating onto stainless steel substrates. In order to obtain crystallization in the perovskite phase, the samples were annealed at 600-700^{0}C for 1 min. The dependence of the electric properties on the heat-treatment temperature was studied, and the coercive electric field as a function of the material thickness was determined. By SEM photography, the microstructure of the films could be shown to be homogeneous. Sol-gel processing is based on the polymerization of alkoxide compounds producing a gel, which then is crystallized by heat-treatment.

By varying the spin coating parameters, the maximum obtainable film thickness for a single coating step was determined to be approximately 330 nm, and was limited by cracking of the film during the annealing process. In order to obtain thicker films, therefore multi-layer processing has been used, where the samples were annealed after each individual deposition step. A good adhesion of the gel on the substrate could be obtained only when cooling down the substrates to room temperature prior to the consecutive deposition step. By this method, homogeneous PZT films with reproducible ferroelectric properties can be processed.

$PbZr_{0.45}Ti_{0.55}O_3$ films of up to 3.2 cm thickness were successfully deposited by multi-layer sol-gel method with alkoxid precursor compounds onto stainless steel substrates. The individual coatings had a uniform thickness and the overall film is homogenous. Rather good values for the remanent polarization are in contrast to a coercive electric field, which appears to be higher than cited in literature for deposition on Si-wafers. This disadvantage, probably stemming from a non-adapted interface between the PZT film and the substrate, is assumed to be healed by the introduction of an additional oxide layer which should improve the conditions for the ferroelectric crystallization process and thus decrease the coercive electric field and while increasing remanent polarization. The modification of the sol-gel process might be envisaged for depositing thicker mono-layers of the PZT in order to obtain thicker overall films; the utilization of stainless steel substrates, as an alternative to the traditional Si-wafer, seems to be promising.

The electrical measurements of PZT thin films have been studied with hysteresis, capacitance-voltage (C-V) and current density – electric field (S-E) measurements by H. Keuppers et al. **[5]**. A thin piezoelectric PZT layer was deposited between two platinum electrodes on a silicon cantilever as the active material. The fabrication process consisted of deposition and RIE structuring of SiO_2 and LPCVD-Si_3N_4, respectively. Then a low stress poly-silicon layer and a thin phosphor silicate glass (PSG) layer was deposited. A PZT thin film with a Zr/Ti ratio of 45/55 was deposited by a modified butoxyethanole-based CSD route to yield a high piezoelectric coefficient. On the PZT layer a 100nm Pt top electrode was sputtered. Then a PECVD-Si_3N_4 film was deposited and structured using a RIE process. The same mask was used for structuring the poly-silicon layer in a TMAH-based wet etching process. Then the contact pads and the metalization layer was deposited and structured using a lift-off process.

To have an overview of the PZT film characteristics hysteresis, capacitance-voltage (C-V) and current density – electric field (S-E) measurements on Pt/PZT/Pt capacitors were carried out. The hysteresis measurements were carried out in virtual ground modus at a frequency of 500Hz and yield a remnant polarisation P_r of 22µC/cm^2. The coercive field E_c was determined to be 47kV/cm. C-V measurements of these films yield a permittivity value of 480(at 500 Hz). Last the S-E measurements were carried out. In this paper PZT-based actuator were presented which can run at low voltages of 5-10V and hence can be integrated in microelectronic circuits.

Yi-Chu Hsu et al. developed a sol–gel process to fabricate PZT thin films with thickness of 2 m in three coatings **[6]**. The crack-free area can be as large as 5mm × 5mm. Recent development of next-generation medical devices, such as endoscopes and hearing aids, call for PZT (lead zirconate titanate oxide) thin-film sensors and actuators with thickness in the range of 1–30µm to enhance actuation strength and sensor sensitivity. Currently, sol–gel derived PZT films often have thickness less than 0.2µm per coating. Moreover, thermal stresses in the films limit the crack-free area to less than $1mm^2$. This paper has four specific goals. The first goal is to demonstrate an improved sol–gel process using rapid thermal annealing and a diluted sealant coating. The resulting thickness can reach 2µm in three coatings with a crack-free area as large as 5mm×5mm. The second goal is to characterize piezoelectric properties of the fabricated PZT films experimentally. The resulting piezoelectric constant d_{33} is 120 pC/N and the dielectric constant ranges from 200 to 400. The third goal is to demonstrate the use of the PZT thin film as a calibrated sensor. The specimen is a silicon cantilever (30mm × 7.5mm× 0.4mm) with a PZT thin film (4mm × 4mm× 1µm). Moreover, a tiny shaker excites the cantilever at the fixed end, and a charge amplifier detects the charge accumulated in the PZT film. In the meantime, a laser vibrometer measures the deflection of the cantilever at three points along the PZT film, from which the strain is calculated using Euler–Bernoulli beam theory. Comparison of the strain and the charge amplifier voltage determines the calibration constant of the PZT thin-film sensor. The last goal is to demonstrate the use of the PZT thin film as a powerful actuator through active vibration control. In experiments, a tiny bulk PZT patch is first glued to the silicon cantilever. A function generator drives the bulk PZT simulating a source of disturbance exciting the silicon cantilever. In the meantime, a laser Doppler vibrometer (LDV) measures velocity of the cantilever tip. With a phase shifter as the controller, the LDV measurement is fed back to the PZT thin-film actuator to actively control the cantilever vibration. To evaluate the effectiveness of the active vibration control, a spectrum analyzer measures the frequency response functions (FRF) from the bulk PZT voltage to the LDV response. Experimental results show that the simple active vibration control scheme can reduce resonance amplitude of the first bending mode by 66%.

A fast firing technique for densification of PZT (up to 97% of theoretical density) at a low temperature (950˚C) has been found out by A. Seal et al. **[7]**. A small amount of excess PbO (3–5 wt. %) and a fast firing schedule are required to achieve the desired sintering. The final composition of the sintered ceramics can be kept close to the morphotropic phase boundary by modifying the firing time and post-sintering annealing treatment at 800˚C. The g_{33} values of fast fired samples are comparable to those of conventionally sintered samples, though the d_{33}

values are somewhat lower than those reported for conventionally sintered (at 1250 ◦C or above) samples.

Lead zirconate titanate (PZT) piezoelectric ceramics of composition $Pb(Zr_{0.52}Ti_{0.48})O_3$, which is close to the morphotropic phase boundary, find a host of applications in recent time. Conventionally, PZT ceramics require a sintering temperature of 1250 ◦C or more. Such high temperature sintering is undesirable because of higher energy consumption and above all, volatilization of lead with consequent change in composition and deterioration of piezoelectric properties. To get around the problems, the common practice is to use controlled atmosphere sintering. Probably, a better alternative is to use a sintering aid so as to get the desired densification exploiting the liquid phase sintering at a lower temperature. However, the selection of sintering aid is crucial because they should not degrade the piezoelectric properties of PZT. So far, a lot of sintering aids have been tried by different workers with varying success. Some examples of sintering aids are P_2O_5, Li_2CO_3, Na_2CO_3, B_2O_3, Bi_2O_3, V_2O_5, Cu_2O–PbO, etc. However, the incorporation of such extraneous components in PZT degrades the d_{33} and g_{33} values in most of the cases. As an alternative approach, there are a few reports of low temperature liquid phase sintering of PZT using excess PbO as a sintering aid.

In the present work, we report a technique of obtaining highly dense PZT ceramics of acceptable properties at a temperature of 950˚C by employing a fast firing schedule and using excess PbO as a sintering aid. The process is simple, economic and does not require any controlled atmosphere. The final composition of the sintered ceramics can be kept close to the morphotropic phase boundary by a post-sintering annealing treatment at a lower temperature.

In A. Chaipanich's study Lead zirconate titanate (PZT) and cement composites of 0–3 connectivity were produced using PZT of 30–90% by volume **[8]**. Lead zirconate titanate (PZT) ceramic particles of composition $Pb(Zr_{0.52}Ti_{0.48})O_3$ were produced using PZT powder sintered at 1250˚C for 3 h. PZT powder was produced from the two-stage mixed oxide method by calcining lead titanate and lead zirconate at 800˚C. Particle size distribution of PZT and Portland cement (ordinary type) were obtained using a laser particle size analyser (CILAS 1064L). Phase characterisations of the composites were carried out by room temperature X-ray diffraction (XRD; Philips PW 1729 diffractometer) using Ni-filtered CuK radiation. The capacitance and the dissipation factor (dielectric loss or tan δ) of the composites were measured using an impedance meter (Hewlett Packard 4194A) at room temperature and at the frequency of 1 kHz.

The dielectric constant of the composites were found to increase with increasing PZT content and the continuous effect of PZT was seen at up to 90% PZT by volume where the ε_r value was found to be at 291. In addition, the dielectric loss (tan δ) was found to decrease with PZT content and the benefit of increasing PZT was seen with the tan δ value was lowest at 0.63 for 90% PZT composite. Piezoelectric coefficient (d_{33}) on the other hand was also found to increase with PZT content as expected. However, the effect was most significant at 30% where immediate effect was seen and then at 90% PZT volume content where the d_{33} value reached 43 pC/N.

M. Es-Souni et al. processed 3 μm thick lead zirconate titanate films via a hybrid sol-gel-powder method on Inconel substrates for pressure sensor applications **[9]**. Microstructure, ferroelectric, dielectric and piezoelectric properties are reported. It is shown that crack- free films with good high remnant polarization, and high effective piezoelectric coefficient, d_{33}, could be processed. Sensor membranes were obtained from these film heterostructures, and their resonance behaviour was studied. Characterization of the membranes under different pressure conditions shows that the resonance frequency of the membrane decreases with increasing pressure. The FEM modelling using mechanic-acoustic coupling of PZT coated membranes agrees largely with this finding.

Multifunctional materials based on the solid solution $PbTiO_3$-$PbZrO_3$ (PZT) exhibit high piezoelectric properties which make them promising candidates for many sensor and actuator applications. These properties may even be amplified via using composite structures such as PZT/metal, e.g. unimorph, flex-tensional actuators and Moonie actuators, which have been shown to lead to high deflection amplitudes. Modern devices require small, miniaturized structures which can easily be produced via thin film technology, and in fact the last decades have seen a plethora of work dealing with thin PZT films for micro-sensor and actuator applications. Sol-gel processing certainly constitutes a versatile and cheap method for the processing of thin films, and still has the advantage of a high degree of microstructure and stoichiometry control. Films of up to 1 μm thickness can be fabricated in a cost-effective way. However, thicker films which are essential for micro-actuator applications may require an unacceptable number of coating sequences which makes the method no more interesting. An alternative route is to make use of a sol filled with a fine dispersion of ceramic particles. In recent work we showed that it is possible to process thick, crack-free and dense PZT films using this processing route. In the present paper a hybrid powder-sol-gel processing route is employed in order to fabricate thick PZT films on metallic foils. New results on the dielectric and piezoelectric properties of such films are presented, and perspectives for their use as resonant membranes for pressure sensor applications are shown. Metallic substrates are of great interest for many applications including embedded capacitors and sensors for automotive applications. Although a recent interest has been devoted to ferroelectric thin films on metallic substrates, no work has been found on the application of PZT thin film/metal heterostructures for piezoelectric applications.

It has been shown that crack- free PZT thick films can be processed via a hybrid powder sol-gel method on metallic substrates. The ferroelectric and dielectric properties lie in the range of those obtained on platinized substrates, though the dielectric loss was found to be higher on metallic substrates. The piezoelectric properties are similar to those reported for flex-tensional actuators. The high remnant strains obtained resemble those of shape memory ceramics. Finally the potential of such heterostructures as resonant membranes for pressure sensor applications is clearly demonstrated. It is shown that the resonance frequency drops substantially with increasing pressure, and follows a quadratic behaviour. The modelling of the membranes using structural-acoustic coupling largely agrees with the experimental results, though a linear drop of resonance frequency was found.

Chemical-solution-deposited PZT films were prepared on copper substrates by Mark D. Losego et al. using solution chemistries where the relative strengths of chemical chelating agents were systematically varied **[10]**. The ferroelectric material lead zirconate titanate (PZT) has traditionally been considered incompatible with base metal technology because PbO volatility makes conventional thermodynamic equilibrium processing impractical. However, by strategically designing solution chemistry and processing conditions to avoid interfacial reaction, chemical-solution-deposited PZT films can be prepared on copper surfaces without oxidizing the base metal or cracking the oxide film. A limited set of thermal and atmospheric processing conditions to kinetically maintain an unoxidized copper substrate are available and not necessarily optimal for processing sol–gel films. Solutions processed within these confined conditions must form gels with sufficiently reduced organic content and properly consolidated gel networks such that phase-pure and crack-free ceramic films can be crystallized. The current work explores three solution chemistries that use different chelating ligands: alkanolamines, acetylactone, and acetic acid. It is found that the alkanolamine solution frustrates perovskite formation and is prone to cracking under processing conditions compatible with the copper substrate. The introduction of water vapor into the processing atmosphere is moderately successful in resolving these issues. Using a more volatile chelating ligand (acetylacetone or acetic acid) shifts the thermal process window nearer a copper-compatible regime. Because of its weaker chelation strength, acetic acid solutions are most compatible with the processing constraints required for copper substrate compatibility.

The experiments demonstrate that by properly optimizing the chelating agent strength, a favourable combination of solution stability and gel structure development could be achieved. This favourable combination enables PZT film synthesis with thermal budgets that are compatible with sensitive substrates like copper foils.

The application of piezoelectric transducers as an energy source has been depicted by Camila Gianini Gonsalez et al. Nowadays, a major concern is the need to develop new energy sources **[11]**. In this context, a sector that has attracted much interest is one in which devices that are able convert other types of energy into electrical energy. This technique is known as Energy Harvesting and consists of energy capture and storage from ambient sources. This paper works with piezoelectric and electromagnetic transducers, which are able to convert mechanical vibrations into electrical energy. However, when an electrical circuit is coupled to the transducer the mechanical system is strongly influenced by it. This paper presents a model that considers the coupling influence between these systems. The structure modelled as a free sliding beam with one electrical load. A program was developed to analyze the behaviour of this system, as well as the optimal conditions for power harvested.

Energy Harvesting, Power Harvesting or Energy Scavenging is about the act of converting ambient energy in electrical energy (electrical power). In every cases this energy was been wasted or lost before. Normally, the electrical energy converted is stored in a kind of battery to be used later but that don’t prevent the energy be used in the same time that is converted too. In this form, the Energy Harvesting may be a solution for source energy in many cases, mainly in remote, inaccessible or hostile environments applications where the connection with the electrical energy network is difficult. Frequently, these devices are small, wireless autonomous, like those used in wearable electronics and wireless sensor networks. The external source can be solar, wind, thermal, salinity gradients and kinetic. In this paper the source is kinetic; specifically, vibration sources that can be anything that have periodic motion. For example the small vibrations of a machine, the motion of walking, even the motion of blood circulation. However, for this conversion to be possible the transducer should transform mechanical energy in electrical energy. The transducers mostly used for this are electromagnetic, electrostatic and piezoelectric. In this work, the harvesting of energy is through piezoelectric and magnetic transducers. Piezoelectric transducers are constituted by piezoelectric materials. This material has the ability to directly convert applied strain into electrical charge. It happens because when a load is applied in the material it causes the molecular structure deformation that in turn causes a separation of the positive and negative gravity centres, resulting in the macroscopic polarization of the material. This paper has described an electromechanical model for an energy harvesting device for piezoelectric and electromagnetic transducers.

Sutrisno W. Ibrahim et al. investigated the necessary conditions to enhance the extracted AC electrical power from the exciting vibration energy using piezoelectric material **[12]**. The effect of tip mass and its mounting position on maximum power extraction are investigated theoretically and experimentally. The optimal load impedance is also investigated to maximize the output power. With the advances being made in wireless technology and low power electronics, wireless sensors are being developed and can be placed almost anywhere. Wireless sensor networks are progressively used in many applications such as: structure health monitoring, automation, robotics swarm, and military applications. However, these wireless sensors require their own power supply which in most cases is the conventional electrochemical battery. Once these finite power supplies are discharged, the sensor battery has to be replaced. The task of replacing the battery is tedious and can be very expensive when the sensor is placed in a remote location. These issues can be potentially alleviated through the use of power harvesting devices.

Piezoelectric energy harvesting is a promising avenue of research to develop self-powered microelectronic devices. Wireless remote monitoring of mechanical structures, low power wireless sensors, and biomedical sensors are strongly candidate for piezoelectric energy harvesting applications. The piezoelectric energy harvester has a limited power and optimization to extract maximum power in the whole stages is necessary to enhance the device performance. The maximum (mechanical/electrical) power transfer depends on piezoelectric material properties and other matching conditions. In this paper, the experimental work validated the theoretical results to enhance the system performance in increasing the output power. Resonance frequency of the harvesting cantilever can be identified experimentally by tracking the maximum extracted electrical power. Increasing tip mass will decrease the resonance frequency. Output power increases as the value of tip mass increases that means; the Q-factor is also increased. After certain limit; increasing tip mass will decrease Q-factor due to damping effect. This phenomenon not yet addressed in this paper. The change of tip mass position has a great effect on the effective mass of the harvesting cantilever, and hence its resonance frequency and the output power are also changed. Piezoelectric harvester has effective internal impedance as capacitive type which depends on the operating frequency. Maximum power transfer to the resistive load occurs at matching condition between harvester's internal impedance and the resistive load. For a fixed level of excitation, the output power is inversely proportional to the natural frequency of a harvesting structure and hence it is desirable to operate at the first harmonic (fundamental frequency) within the available vibration spectrum.

Renato Caliò et al. presents the basics of piezoelectricity and discusses materials choice **[13]**. Though piezoelectric energy harvesting has been thoroughly investigated since the late 1990s, it still remains an emerging technology and critical area of interest. Energy harvesting application fields so far mainly focused on low power devices due to their limited transduction efficiencies. To date, researchers are following distinct ways in developing piezoelectric energy harvesting technology. New materials, configuration approaches and operating modes are under study, and some of these valuable solutions were proposed in order to achieve large bandwidth harvesters that are able to scavenge energy from diverse environments. Resonant cantilever beams need optimization, but several interesting solutions and approaches that were published can push forward the research. Harvesters are still too complex to be fabricated, but exhibit great potential.

In this paper these configurations have been briefly studied using a comparison table with respect to, reporting various factors. From this analysis it is concluded that is more efficient than other modes, has higher output voltages, simplifying the power conversion process, whereas is the simplest solution found in terms of fabrication process and performances (in most cases). Considering nanoscale harvesters, they represent a promising but still emerging technique that requires to be consolidated. Non-resonant solutions, as well as frequency tuning methods, are powerful instruments to push forward the growth of vibration harvesting techniques. However, though several non-resonant solutions were demonstrated, new roads can be explored. As an example, in electromagnetic vibration harvesting a well-known technique to achieve bistability involves mechanical bumpers. Furthermore, all these piezoelectric harvesting research branches could be merged. Likely, a bistable harvester involving a high efficiency material, equipped with a proper conditioning circuitry, would achieve significant results. The limit in terms of harvested energy density has still to be overcome. This has been the main technological challenge so far. A well-integrated roadmap was designed in the framework of the Guardian Angels Coordination Action within the Future and Emerging

Technologies Flagship initiative funded by the European Commission. In this framework, research efforts are focusing on the transducer and also on the integration with the downstream conditioning circuitry, power management circuits and application devices.

Vincent Chalvet et al. proposed the characterization of a new generation of piezoelectric cantilevers called thick-films piezoelectric actuators **[14]** Based on the bonding and thinning process of a bulk PZT layer on to a silicon layer, these cantilevers can provide better static and dynamic performances compared to traditional piezo cantilevers, additionally to the small dimensions.

PZT (lead, zirconate, titanate) material is widely used in the microworld for the design of highly performant actuators and sensors. Thanks to their high displacement resolution and high bandwidth, piezoelectric actuators are prized for several applications such as in microrobotic domain (cantilevers for micro tweezers, AFM piezo-scanner, etc.) or biomedical applications. Despite their high resolution, piezoelectric actuators present several well-known nonlinearities, such as hysteresis or creep. Many studies are currently trying to overcome these nonlinearities by using compensation techniques, or through feedback control strategies. One of the mainly used piezo actuators is the unimorph cantilever, it is made of one piezoelectric active layer bonded on to a passive elastic layer, resulting in an out of plane displacement when an electric field is applied between the two sides of the piezoelectric layer.

The fabrication process of the piezoelectric actuator is based on the gold bonding (at room temperature) of a PZT bulk layer (200 μm thickness) onto a SOI (Silicon On Insulator) wafer. Mechanical thinning and polishing of the PZT layer is then per- formed. A thin gold film is then deposited on the top surface of the PZT layer, which serves as a top electrode for the cantilever actuation. The gold layer used for the bonding is also used as the bottom electrode. The characterization setup is composed of: the piezoelectric actuator, an optical sensor (LC2420 from Keyence Company) capable of measuring the deflection of the cantilever (displacement) with a resolution of tens of nanometer and bandwidth in excess of 5 kHz, and a computer and a dSPACE board used to generate the input voltage and to acquire the measurement. The sampling time is tuned to be 50 μs.

This paper dealt with the static and dynamic characterization of a novel piezoelectric cantilever actuator. Along the paper, it is shown that the actuator exhibit much larger gain (in excess of 3.7 μm/V) and higher dynamics (first resonant frequency 1100 Hz) than in classical piezoelectric PZT cantilever actuators. During the characterization, it is also shown that the classical properties, i.e. hysteresis and creep nonlinearities and badly damped oscillations, of piezoelectric actuators are found in this novel generation of actuators.

Sarabjeet Kaur et al. discussed the effect of various shapes and materials which open the gateway towards the choice of maximum power generation for the micro and nano world **[15]**. Comsol Multiphysics was used to simulate the four designed shapes named as Pi, E, Rectangular and T in the size range of less than 1mm but greater than 1 micron. Designed shapes worked under the impact of ambient vibrations using few piezoelectric materials for the maximum power generation so that traditional power sources can be replaced with such piezoelectric energy harvester. A layer of piezoelectric material (PZT-5H, AlN, $BaTiO_3$) of thickness 0.5 μm is added to the cantilever and the base material is silicon of thickness 1.5 μm. Simulations were performed using the piezoelectric device module of Comsol Multiphysics. All three materials were studied for the all four cantilever geometries. The generated power was observed maximum as 382.5 μW in case of the barium titanate material with rectangular shape geometry but the displacement is 0.132 μm which is very less whereas E shape cantilever shows the maximum displacement of 0.6078 μm in case of PZT-5H, Hence rectangular shape with barium titanate material is concluded to be good for maximum power generation but the displacement factor cannot be neglected, hence the cantilever with E shape geometry is considered as the best with a generated power of 49.005 μW and a displacement of 0.6078 μm.

The whole processing of a Micro scale device is well defined from the aspects of its used materials. The supposed designed and simulated cantilevers were composed of two layers, one is piezoelectric and the rest one is semiconducting in nature. The substrate of thickness 1.5 μm has been selected and 0.5 μm is the thickness of the piezoelectric material. The piezoelectric energy harvesting system sector is booming as shown from yole market report of MEMS. The decision to choose the piezoelectric material was totally oriented from their highly valuable properties. They are having the high piezoelectric coefficient and less Young's modulus which predicts greater displacement as per Stoney's formulation. They give the best piezoelectric effect which on getting any outer potential generate the reverse mechanical change either the expansion or the contraction or vice versa depending upon the kind of the application. The piezoelectric generator principle is based on direct piezoelectric effect. The conversion chain consist of the vibration source or mechanical energy source (e.g. cantilever beam).The movements of the cantilever beam produces ambience vibrations and they are converted to electricity by using piezoelectric element. The piezoelectric materials are highly advantageous to use for generating power, as they harvest energy and produce significant amount of power at the output. It is considered to be the innovative approach of power generation and power can be stored for future also.

The vibrational energy harvesting is used to generate power using piezoelectric materials. The basic process of power generation involves energy source, energy conversion circuit and the capture circuit. The conversion chain starts with mechanical vibrations source from environment. The vibrations from the surroundings cause movement and deflection in the cantilever. The deflection in the cantilever exerted by the force provide by vibration in micro Newton. As per theoretical criteria, we observed an increase in deflection with decrease in spring constant and leading to a corresponding increase in generated piezoelectric voltage which intensifies the output power. The resistance is kept constant, hence the power generation is directly proportional to v^2/R_{load}. Among the various traditional geometries, E shape gives maximum displacement (0.6078 μm) and Rectangular shape generates highest voltage (0.19125 V).

A.Sh. Kherbeet et al. investigated the factors affecting the performance of a vibration based micropower generator for a power plant wireless monitoring application **[16]**. A bimorph bender, one of micropower generator methods, was used in this work. The ANSYS program was used to study the distribution of stress–strain in each model design, and MATLAB was used to simulate and investigate the extracted power. Triangular, rectangular and trapezoidal cantilevers were chosen to analyse the effect of the bender shape on the power produced. The tests were conducted using the same input excitation conditions ($10\ m/s^2$), frequency range of (50–150 Hz). The effect of the configuration arrangement (horizontal and vertical) of the PZT bimorph cantilever on the power generation (poling series) was also investigated. The simulation result showed that the maximum stress–strain value was produced in the triangular bender shape with equal distribution on all the surface areas. The measurements' results showed that the triangular cantilever produced maximum power compared to other bender shapes for both horizontal and vertical directions and single test. The horizontal and vertical arrangements of the cantilevers showed that the vertical arrangement could produce more power than the horizontal arrangement. The experimental results showed concurrence with theoretical models.

In general, sources of vibration are plentiful and easily accessed for example via air ducts and buildings' structures. Piezoelectric conversion is one method producing a significant increase in use for power harvesting by utilizing the vibrations surrounding a system which have many ranges. Low vibrations generated in different environments attract more attention, such as vibrations generated in factories or other buildings. Growing interest in the improvement of wireless sensor and actuator networks (WSN) has resulted in its use across a range of applications. One WSN application is monitoring the status and technical conditions of machines, using a distance sensor. In this field researchers have studied the numerical and experimental enhancement of the micropower generation utilizing a piezoelectric cantilever. The configuration of the fixed free cantilever with a proof mass at the free end was chosen in this study since the one-edge fixed configuration can generate larger strain and thereby higher power compared with the two-edge fixed bridge and an all-edge fixed diaphragm at the same input force. Secondly it can easily achieve low resonance frequency corresponding to most vibration sources in the environment. However, the power of the piezoelectric vibrator is proportional to its strain. In the other words, the more strain at each location the greater the generated voltage output.

The design of triangular, rectangular and trapezoidal piezoelectric cantilevers was proposed, simulated, then evaluated experimentally and numerically to improve the extraction of energy from the environmental vibration. The results illustrated that the triangular cantilever could be used to generate the highest output power value when compared with the other shapes. The triangular cantilever was found to be a better choice for a wideband generator. The trapezoidal and rectangular cantilever shapes showed less than the total power generated by the triangular cantilevers. Under the same volumetric conditions and vibration excitation for all the shapes, the experimental results showed that two triangular cantilevers at designated natural frequencies of 85 Hz and 95 Hz combined in vertical direction had the ability to generate 11979.527 lW for a volume of 1 cm3 which is high enough to generate a wireless sensor. This value represents the highest value of produced power compared to the rectangular and trapezoidal shapes. The results revealed the increases of the designated natural frequency led to the decrease in the harvested power. The increment of cantilever numbers to more than two cantilevers led to an increase of harvested power in the horizontal combination while the extracted power decreased with a vertical increment of cantilevers.

Y. Kebbati et al. presented the optimization of the energy harvesting in the case of piezoelectric converter **[17]**. The conversion of mechanical energy from environmental vibrations into electrical energy is a key point for powering sensor nodes, towards the development of autonomous sensor systems.

Within power electronic electromagnetic transformers have been the dominating component for converting and transforming of electrical power. The trend of power converters goes in the direction of higher efficiency and smaller volume. Research has shown that piezoelectric converters (PC) can compete with traditional electromagnetic transformers on both efficiency and power density. PCs are therefore an interesting field of research. A PC cantilever model includes the inverse and direct piezoelectric effect which harvesting the energy from the motion.

A number of crystals present a piezoelectric behaviour; we can quote the quartz, the tourmaline, the salt of Seignette, the sugar. This behaviour appears in crystals presenting an asymmetric structure and ionic connections. Unlike the crytals which the structure is fixed, the piezoelectric ceramic has a crystallography structure that can vary. An important family of piezoelectric ceramic, most used in industry, is the PZT (Lead-Zirconate-Titanate) which possess excellent piezoelectric properties. The PZT is generally formed by cristals of Lead Zirconate $Pb^{2+}Zr^{4+}O_3^{2-}$ and by Lead Titanate $Pb^{2+}Ti^{4+}O_3^{2-}$, in near equal proportions. The Titanate of Barium $Ba^{2+}Ti^{4+}O3^{2-}$ is another piezoelectric ceramic which possesses the same crystallography structure as the PZT.

The experimental validation is realized using test bench with laser, lenses, reflector, photo receiving cell and PZT system. The PZT system is composed: ceramic PZT with 280 multilayers of 60 μm thickness each, an electronic circuit supplying polarization in the ceramic PZT and an actuator which applies mechanical stress. The photo receiving cell measures the movements of the PZT, whereas energy harvesting is converted in volt. From FEM simulations, it was seen that the sinusoidal polarization gives poorer results compared to DC polarization. Thus, the experimental tests will be only made for DC polarization. As predicted with simulation, the polarization allows enhancement in energy harvesting. To reach the same energy, we need to increase the mechanical stress applied to the PZT through the actuator about 18%. However, this increase of the stress also increases risk of destruction of the micro layers in ceramic.

An energy harvester which is a self-generating energy source able to run small scale electronic devices was developed by Mahidur R. Sarker et al. **[18]**. It largely reduces the dependency on external power source. This paper represents the utilization of micro cantilever beam to develop a vibration based electromechanical systems (MEMS) piezoelectric energy harvester system. The proposed micro cantilever part has been developed by finite element method (FEM) using

COMSOL. The motivation behind this research is to calculate the resonant frequency of the harvesting and electrical voltage behaviour considering cantilever beam length and thickness. The materials (i.e., silicon, PZT-5H) have been studied to improve voltage efficiency. The proposed micro cantilever beam has been able to generate 0.4V in COMSOL. Finally, the result of proposed method is compared with other existing methods to validate the outcomes.

There are two types of piezoelectric materials, piezoceramics like Lead Zirconate Titanate (PZT) and piezopolymers like Polyvinylidene Fluoride (PVDF). This work has been designed a single layer material (i.e. PZT-5H, silicon) based piezoelectric cantilever beam to harvest the energy. One of the most important parameter to design a piezoelectric cantilever beam vibration based energy harvesting device is resonant frequency has been considered. The range of frequency is common for environmental vibrations between 60 Hz and 200 Hz. The mesh application, which defines the relation between the 3-D structure and reference structure, solves mash smoothing equations inside the COMSOL to define the coordinate transformations of the beam.

This paper represented the modelling, simulation of piezoelectric energy harvester based on micro cantilever beam. The micro cantilever was developed using COMSOL multiphysic software. The developed micro cantilever beam with the silicon and PZT-5H material in COMSOL was produced 0.4V. Thereafter, the results obtained in this study were compared with others well known methods. Finally, it is concluded that the proposed method performs better than other methods.

Mohammad H. Malakooti et al. fabricated nanostructured piezoelectric beam using vertically aligned lead zirconate titanate (PZT) nanowire arrays and its capability of continuous power generation is demonstrated through direct vibration tests. The lead zirconate titanate nanowires are grown on a PZT thin film coated titanium foil using a hydrothermal reaction **[19]**. The PZT thin film serves as a nucleation site while the titanium foil is used as the bottom electrode. Electromechanical frequency response function (FRF) analysis is performed to evaluate the power harvesting efficiency of the fabricated device. Furthermore, the feasibility of the continuous power generation using the nanostructured beam is demonstrated through measuring output voltage from PZT nanowires when beam is subjected to a sinusoidal base excitation. The effect of tip mass on the voltage generation of the PZT nanowire arrays is evaluated experimentally. The final results show the great potential of synthesized piezoelectric nanowire arrays in a wide range of applications, specifically power generation at nanoscale.

In recent years, the idea of having of self-powered small electronics which eliminates the use of batteries has led to the development of various nanoscale piezoelectric materials along with many efforts in utilizing them in compact electronics. Piezoelectric materials are well-known active materials that convert the ambient mechanical energy into electrical energy in their "generator" mode of operation and they deform once they are subjected to an electric field which this behavior is often termed as "motor" mode. Since piezoelectric materials preserve their coupled behavior even in a smaller scale (i.e. nano and micro scales), there have been efforts to synthesize them in these scales to shrink the required space for integrating them into a system. Piezoelectric nanomaterials have been synthesized in different geometries: nanowires, nanoparticles, nanorods, nanotubes, nanotubes, and etc. The shape of these nanomaterials is generally controllable during the synthesis process and varies based on their potential applications.

In order to investigate the energy harvesting potential of the vertically aligned PZT nanowires in conversion of the mechanical energy to electrical energy, electromechanical frequency response function (FRF) characterization of the output voltage response of the device with respect to base acceleration was performed. A cantilever beam configuration was chosen for the simplicity and compatibility of the common energy harvesting devices to conduct the vibration characteristics of the beam. The beam was clamped on an electromagnetic LDS shaker in order to stimulate the vibrational motion of a host structure. Then the open voltage generated from the PZT nanowires and the PZT thin film was measured using a high impedance voltage follower (LTC6240CS8) while the base acceleration of the beam was monitored using a PCB shear accelerator (PCB 352C22).

Fumiya Kurokawa et al. fabricated multilayer ceramics (MLCs) composed of multilayered $Pb(Zr,Ti)O_3$ (PZT) piezoelectric thin films with internal electrodes and evaluated their dielectric and piezoelectric properties **[20]**. Multilayer thin films were deposited on SOI substrates by multi-target RF magnetron sputtering. In this study, SOI is used as a substrate to fabricate micro-cantilevers from the MLCs. The thickness of Si device layer, buried oxide (BOX) layer, and substrate layer were 35, 1, and 325 μm, respectively. Deposition of the SRO/Pt/Ti layer on the SOI substrate as a bottom electrode was done first. SRO thin films acted as not only electrode layers but also seed layers to promote the crystal growth of perovskite phase for PZT thin films. Then, an internal SRO electrode was deposited after moving the shadow mask in the same direction to maintain insulation with the bottom electrode. The depositions of internal SRO electrode layers and PZT ferroelectric layers were alternately conducted by reciprocating the movable shadow mask. The effective area of multilayer structure is an overlapping part of the separated internal electrodes, and both ends of the electrodes act as external electrodes. The Ti and Pt layers were deposited on the heated substrate around 500 °C, and during the growth of SRO and PZT layers the substrate was heated to 775 °C. Ti adhesion layer and Pt electrodes were sputtered in an argon gas atmosphere, while PZT and SRO layers were deposited in a mixed gas atmosphere of argon and oxygen. The gas pressure was around 0.5 Pa during each sputtering deposition. The thicknesses of Ti, Pt, SRO, and PZT layers were typically around 3, 80, 100, and 550 nm, respectively.

The stack of PZT ferroelectric layers and internal SRO electrodes were deposited by single RF magnetron sputtering and the micro-cantilevers composed of multi-layered PZT thin films were fabricated for the first time. The stack of PZT ferroelectric layers and SRO electrodes were fabricated on SOI substrates by alternatively deposition through movable shadow mask. We measured the dielectric properties with one, three, and five PZT layers. The capacitances increased with the number of PZT layers, while the relative dielectric constants are almost constant. The all MLCs exhibited almost saturated and symmetric P–E hysteresis loops. We evaluated the piezoelectric properties of MLCs using FEM simulation. The calculated effective transverse piezoelectric coefficient ($d_{31,eff}$) also increased with the number of PZT layers and reached to −2964 pC/N at 25 PZT layers, which is much larger than those of conventional single-layer piezoelectric thin films. These results indicate that multilayered PZT thin films with separated internal electrodes are effective to enhance dielectric and piezoelectric properties.

2.3 CONCLUSION

Earlier bulk materials were synthesized like pellets whereas in the recent years the focus has shifted to smaller things. PZT thin films are being produced instead of thick films. As technology is advancing researchers are trying to fabricate materials which are smaller in magnitude. In the VLSI fields microstructures and nanostructures are being studied. Work on PZT thin films on metallic substrates (such as copper and stainless steel) is being done extensively throughout the world.

Energy harvesting from piezoelectric materials is also being done. The shapes and sizes of PZT for optimal energy harvesting is being studied. Multi-layered piezoelectric films are also being developed which will aid in energy production.

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CHAPTER 3

THE RESEARCH OBJECTIVE

3.1 INTRODUCTION

After an exhaustive review work on the various field of research work that has been done till date on this subject no work has been observed regarding a consistent and comparative study on the microstructures and the dielectric properties of PZT materials synthesized from the nitrate precursor materials solid state sintering method.

Therefore, in this work a comparative study of the role of different ratios of Zr/Ti on the piezoelectric behaviour as an energy harvester and also a vibration sensor in relation to micro structural modification have been done.

3.2 PLAN OF THE WORK

The following steps were involved for the entire proposed work:

1) Conventional powder mixing techniques are used for preparing $PbZrTiO_3$ (PZT) powders in various Zr/Ti ratios.

2) Calcinations of the oxide mixture from the corresponding nitrates and rutile powders of Pb-Ti and Pb-Zr at 600°C were carried out.

3) The calcined oxide mixture Pb-Ti and Pb-Zr again were mixed stoichiometrically for preparing oxide mixture of Pb-Zr-Ti at 52:48; 51:49; 50:50; 49:51 and 48:52 ratios in powder mixing technique.

4) Finally calcination of the oxide mixture of Pb-Zr-Ti at 600°C was carried out.

5) Rectangular beam with those calcined oxide mixture of Pb-Zr-Ti were prepared in a die press machine.

6) The compacted wafers were sintered at 1000°C for 2 hours.

7) Microstructures and surface morphology of the sintered samples were studied with the help of XRD analysis and SEM pictures respectively.

CHAPTER 4

EXPERIMENTAL PROCEDURE

4.1 INTRODUCTION

Various synthesis routes that have been reviewed from the literature and described in the previous chapters possess both merits and demerits. In the proposed work the Conventional and low cost process has been followed for preparing doped PZT bulk materials by powder mix technique. In the process as described in chapter 3, Lead Nitrate, Zirconium and Titanium dioxide were mixed in powder form separately to ensure adequate mixing and homogeneity of the ingredients. The ingredients were mixed in different ratios to develop precursors of PZT of different molar ratios of $PbTiO_3$ and $PbZrO_3$.The detailed experimental method has been described in the next section. Starting materials used were inorganic salts of Pb, Zr and Ti. In stage wise powder mix process and calcinations there will be no wastage of material was observed and almost theoretical yield was obtained.

4.2 EXPERIMENTAL PROCEDURE

The starting materials were annular grade Lead Nitrate, Zirconium and Titanium dioxide (Rutile) in the powder form. The whole synthesis process was carrying in three steps as described in the following sections for the preparation of the specimens.

4.2.1 FIRST STEP

The powders of Lead Nitrate and Zirconium oxide were mixed maintaining proper Stoichiometry for the preparation of PZT in a grinder mixer. The mixing process was continued for 2 hours in steps i.e. it was alternately run for 5 mins and stopped for another 5 mins throughout the whole period of time. The white powder material was then calcined at 600˚C in a muffle furnace for a period of 2 hours. The calcined material became oxide mixture of Pb and Zr.

4.2.2 SECOND STEP

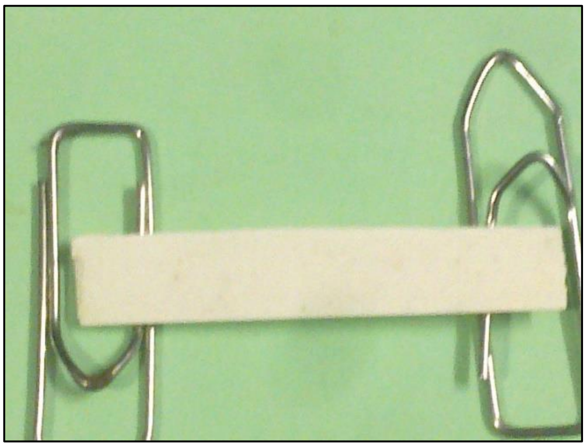
A calcined oxide mixture of lead and titanium was prepared in a similar way as described in section 4.2.1, using proper molar weight ratio of Lead Nitrate and Titanium Oxide. After calcinations of the mixture the material became oxide mixture of Pb and Ti.

4.2.3 THIRD STEP

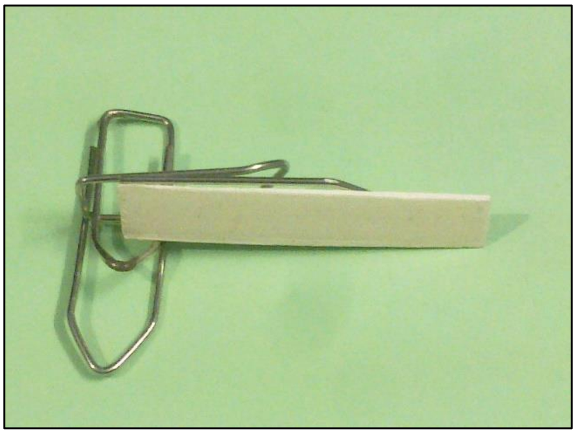
At this stage, the oxide mixture of Pb-Zr and oxide mixture of Pb-Ti were again mixed at weight ratios 52/48, 51/49, 50/50, 49/51 and 48/52 in a powder mix technique. The resulting white power material was then calcined at 600˚C in a muffle furnace for two hours for removing residual nitrate and water molecules. The obtained products were the oxide mixture of Pb-Zr-Ti at previously stated weight ratios respectively. The samples were used to make rectangular beams and sintered (the detailed sintering process is described in the next section) to form a solid solution of Lead Zirconium Titanate (PZT) of the said ratios.

4.2.4 PREPARATION OF SINTERED RECTANGULAR BEAMS

This calcined powder was ground with very small amount of 4% PVA solution (aqueous) in an agate mortar. The ground mix was then casted into rectangular beams having a dimensions of 25 mm length, 5 mm width and 0.5 mm thickness using a in a die press machine at 100 kg/cm^2 pressure and then fired in an electrically heated temperature controlled Box Furnace at final temperature 1000˚C. Figure 4.1 shows the actual pictures of the rectangular beams compared to a paper clip.



(a) Top view



(b) Side view

Figure 4.1: Pictures of the rectangular beam

4.2.5 DETAIL OF FURNACE TIME-TEMPERATURE PROFILE

The palletized green compacts of all the samples were marked properly and placed into a Box furnace. Temperature of the furnace was controlled by a set-point programmable PID controller. The set-point of the controller was time programmed and the furnace temperature changed with time in a controlled manner according to the pre-programmed rule base stored into the controller memory. A setting of the time-temperature profile used to sinter the green has been briefly described as follows.

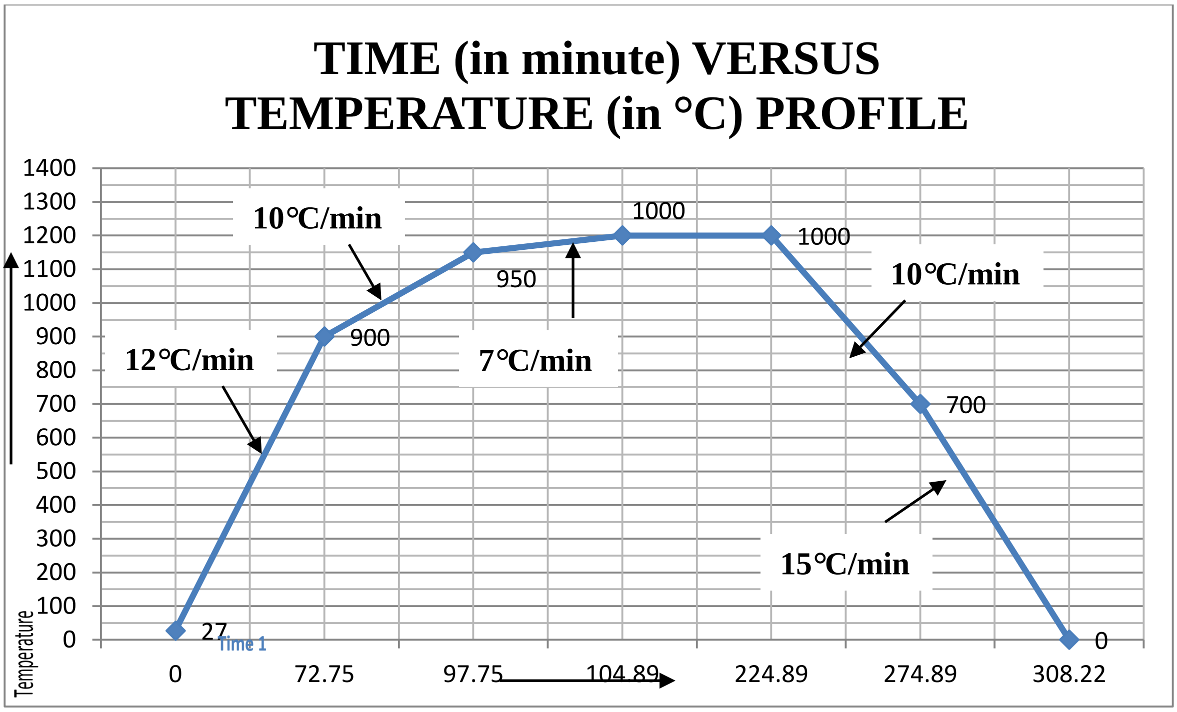


Figure 4.2: Time - Temperature Profile

The rise in temperature from ambient i.e. 30˚C up to 900˚C was maintained at a rate of 12˚C per minute, then the ramp rate was set at 10˚C per minute up to 950˚C and for the last segment the ramp setting was 7˚C per minute up to the temperature 1000˚C The dwell period was set for 120 min. After finishing the dwell period the furnace was cooled down at the rate of 10˚C per min up to 700˚C and then at the rate of 15˚C per min until the furnace temperature became equal to the ambient temperature i.e. 30˚C (approx.). Figure 4.2 demonstrates time-temperature profile of the furnace temperature control. The microstructures and the surface morphology of these sintered compact specimens were characterized using XRD and SEM.

4.2.6 CHARACTERIZATION OF THE SAMPLES

4.2.6.1 XRD ANALYSIS

XRD characterization of the sintered specimen was carried out with a Rigaku X-ray diffractometer with Cu target (Miniflex, Japan). Figure 4.3 demonstrates a pictorial view of the Instrument.

Figure 4.3: Rigaku X-ray diffractometer with Cu target (Miniflex, Japan)

Both the sintered samples were thoroughly ground to 50 mesh size. Around 2 mg of the finely grinded sample was taken in a standard XRD sample holder and subjected to XRD analysis under normal air pressure at room temperature. The collar level was fixed at zero. CuKα light was used and 15mA current was passed from a source of 30 kV. Miniflex goniometer and Kb filter were used to obtain the diffraction data. The diffraction data were analyzed using standard commercial software and the standard diffraction data provided by the Joint Committee on Powder Diffraction Standards (JCPDS). The XRD data are presented graphically in the Figure 4.4 to Figure 4.8.

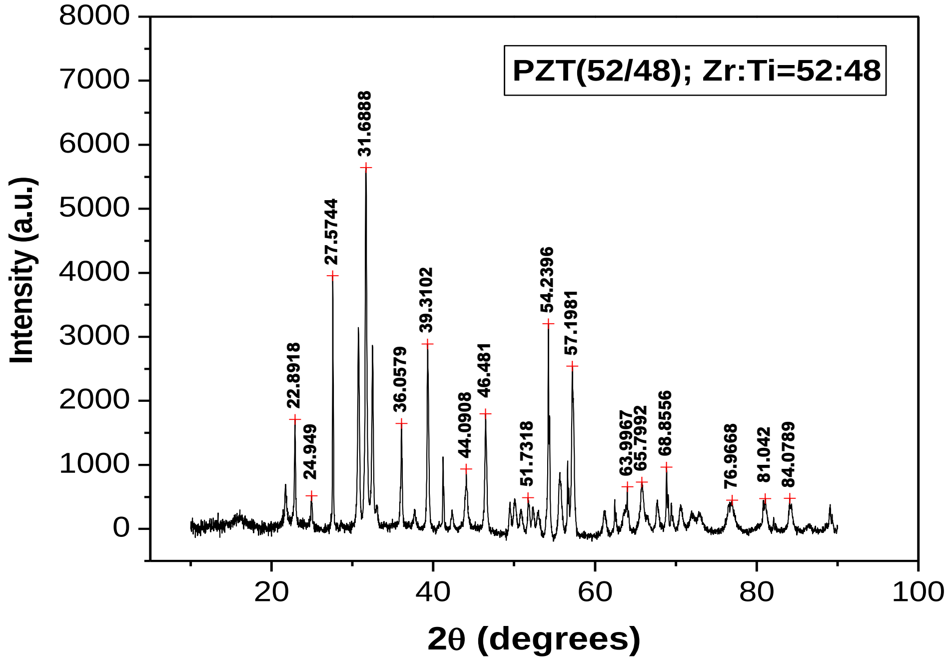


Figure 4.4: XRD Diagram of PZT (Zr/Ti = 52/48)

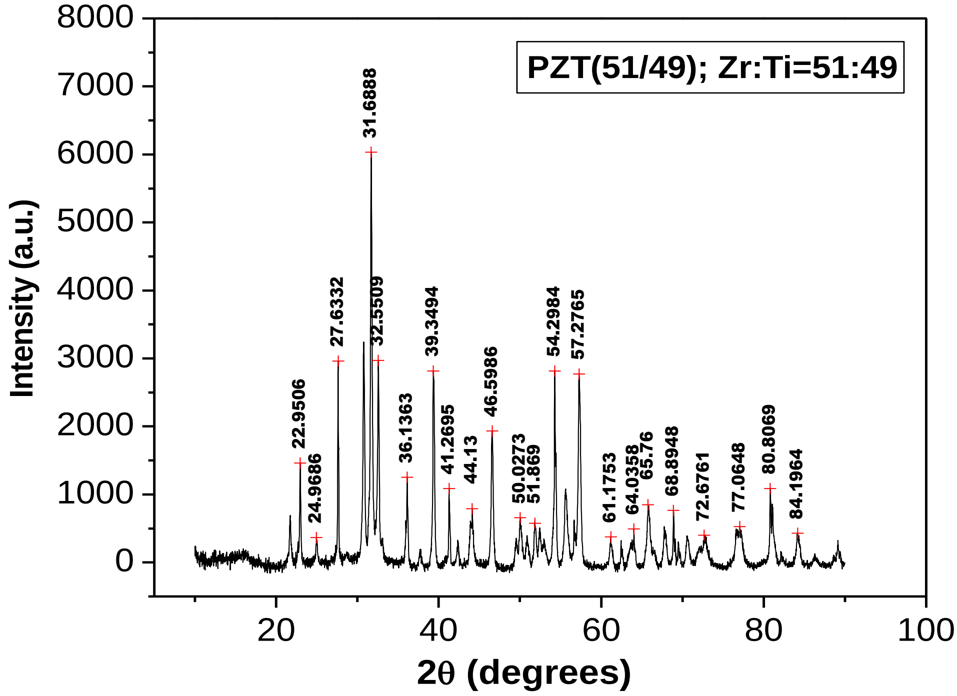


Figure 4.5: XRD Diagram of PZT (Zr/Ti = 51/49)

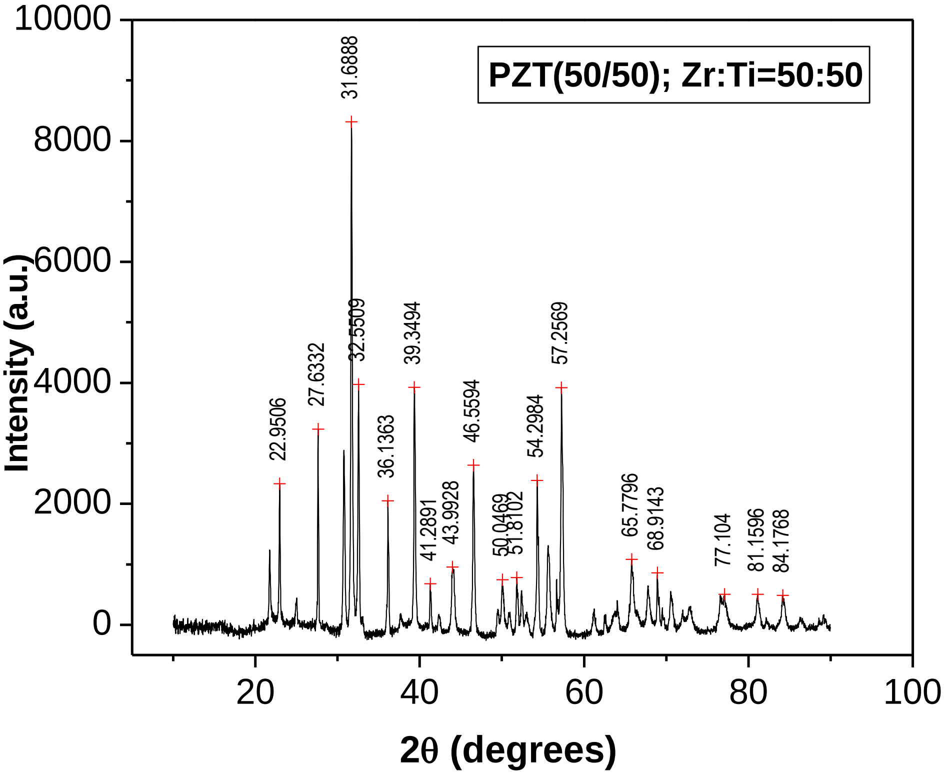


Figure 4.6: XRD Diagram of PZT (Zr/Ti = 50/50)

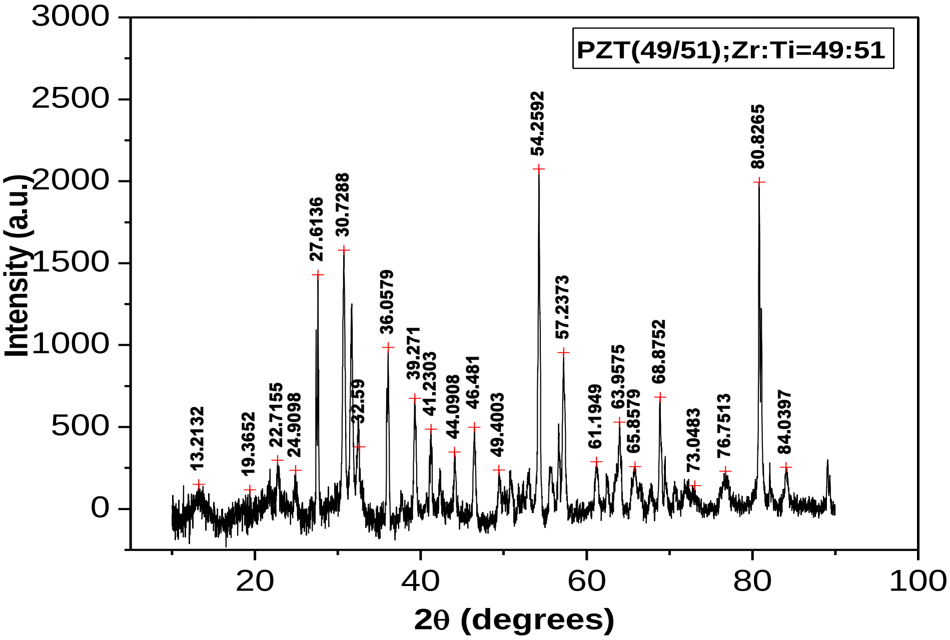


Figure 4.7: XRD Diagram of PZT (Zr/Ti = 49/51)

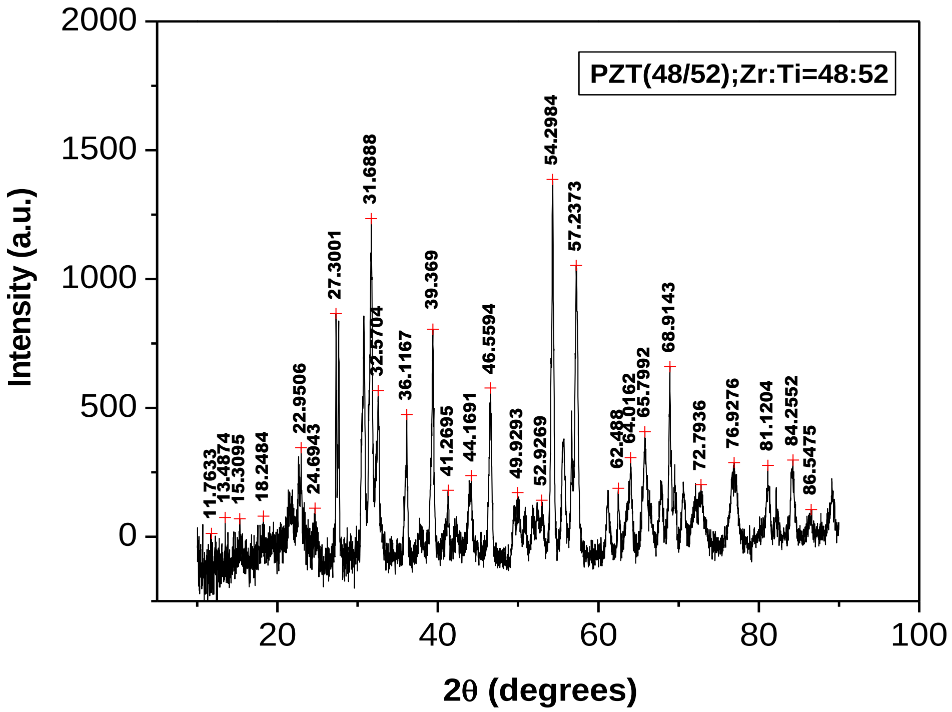


Figure 4.8: XRD Diagram of PZT (Zr/Ti = 48/52)

4.2.6.2 SEM ANALYSIS

A study of the photomicrograph (SEM) analyses of the sample was carried out with FEI Quanta microscope (US) for determining the particle size and morphology of the prepared samples. The Fig.4.9 demonstrates a pictorial view of the Instrument. The chamber pressure was kept in between 70 to 120 Pascal. The chamber was filled with water vapour. The sample was placed on the carbon tape and the whole system was placed on a 1 cm stab. The SEM photographs of the samples are as shown in the Figures 4.10 to Figures 4.12.

Figure 4.9: FEI Quanta Microscope (US)

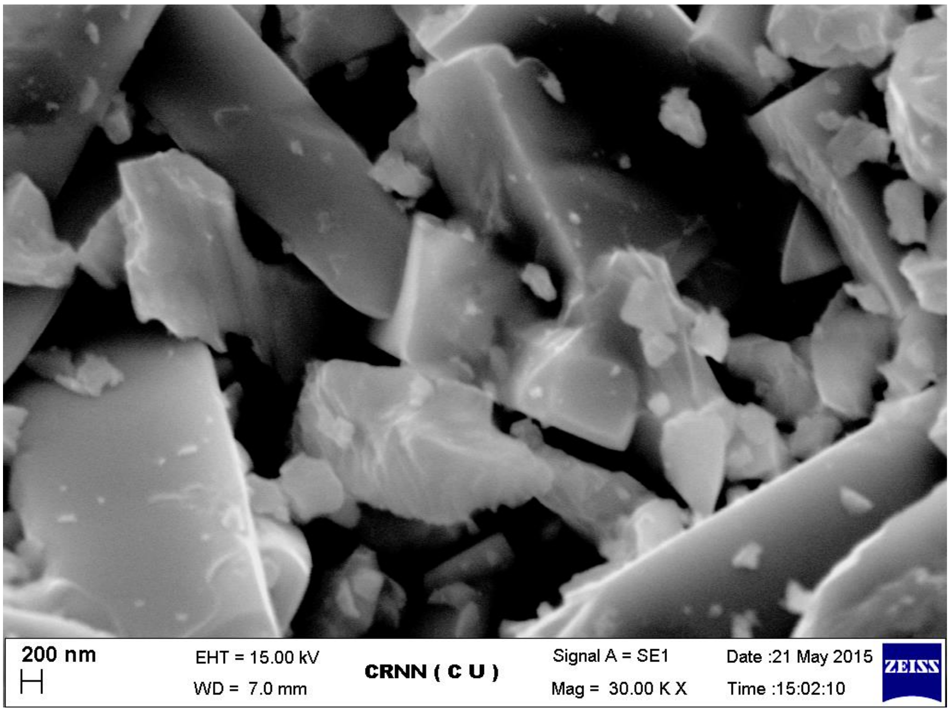


Figure 4.10: SEM Image of PZT (Zr/Ti = 51/49)

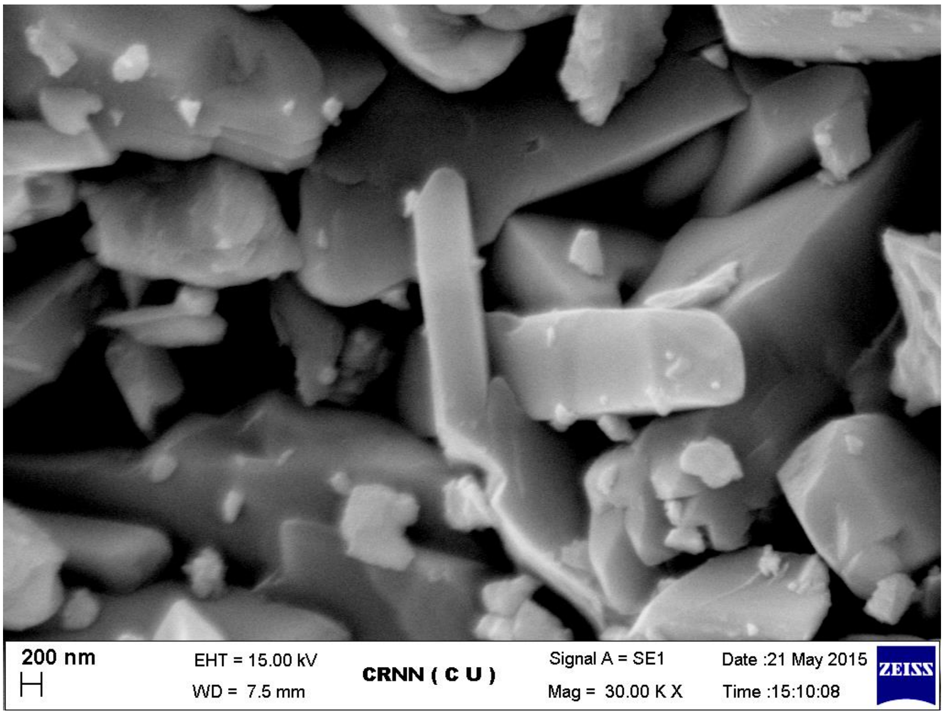


Figure 4.11: SEM Image of PZT (Zr/Ti = 50/50)

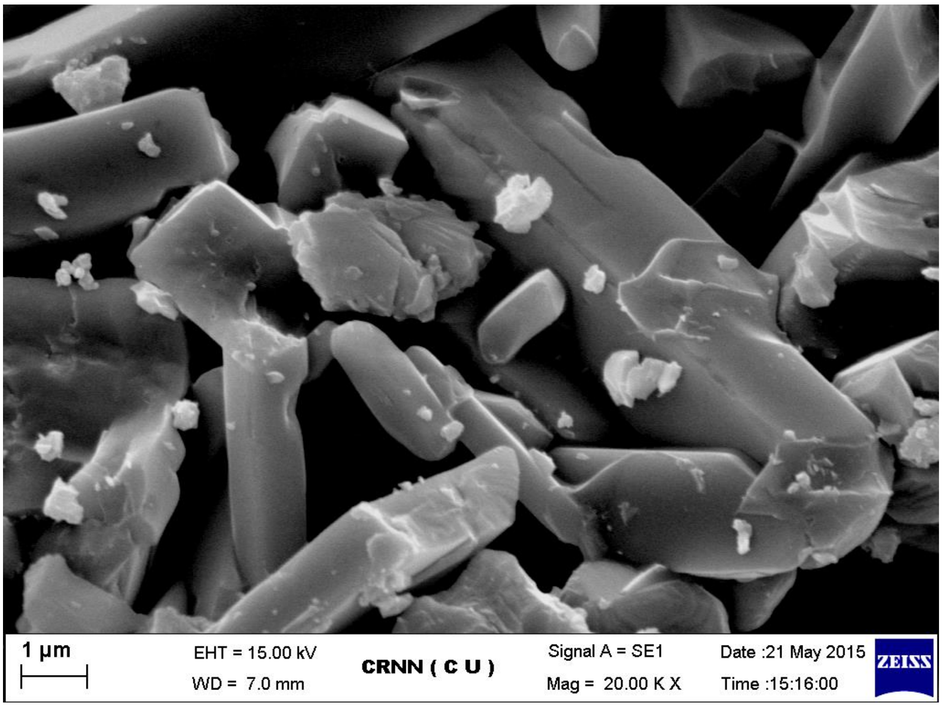


Figure 4.12: SEM Image of PZT (Zr/Ti = 49/51)

4.2.7 EFFECT OF CHANGE IN LENGTH AND WIDTH DUE TO VARIATION OF COMPOSITION

Length and width of the PZT samples having different Zr/Ti ratios such as 52/48, 51/49, 50/50, 49/51, 48/52 were measured before sintering and after sintering. Sintering process was carried out at a temperature of 1000°C for 2 hours. The percentage shrink in length and width were calculated from the values of the dimensions before sintering and after sintering. Effect of change in length due to variation of composition is shown in Table 4.1. Effect of change in width due to variation of composition is shown in Table 4.2.

From Table 4.1, it can be concluded that the percentage shrink in length at first decreases significantly with increasing Ti content from 48% to 50% and then increases with increasing Ti content from 50% to 52% (little change is found when Ti content increases from 51% to 52%).

From Table 4.2, it can be concluded that the percentage shrink in width decreases slightly with increasing Ti content from 48% to 52% but there is an exception when the Ti content is equal to 51%.

SERIAL NO.	COMPOSITION	LENGTH BEFORE SINTERING (in mm)	LENGTH AFTER SINTERING (in mm)	CHANGE IN LENGTH (in mm)	% CHANGE IN LENGTH
1	Zr/Ti = 52/48	25.00	21.98	3.02	12.09
2	Zr/Ti = 51/49	25.00	22.20	2.80	11.18
3	Zr/Ti = 50/50	25.00	22.36	2.64	10.55
4	Zr/Ti = 49/51	25.00	21.87	3.13	12.52
5	Zr/Ti = 48/52	25.00	21.85	3.15	12.60

Table 4.1: Effect of change in length due to variation of composition

SERIAL NO.	COMPOSITION	WIDTH BEFORE SINTERING (in mm)	WIDTH AFTER SINTERING (in mm)	CHANGE IN WIDTH (in mm)	% CHANGE IN WIDTH
1	Zr/Ti = 52/48	5.00	4.21	0.69	13.82
2	Zr/Ti = 51/49	5.00	4.33	0.67	13.38
3	Zr/Ti = 50/50	5.00	4.34	0.66	13.24
4	Zr/Ti = 49/51	5.00	4.28	0.72	14.38
5	Zr/Ti = 48/52	5.00	4.35	0.65	13.02

Table 4.2: Effect of change in width due to variation of composition

CHAPTER 5

RESULTS AND DISCUSSION

5.1 XRD RESULTS

XRD characterization of the samples was carried out with a Rigaku X-ray diffractometer with Cu target (Miniflex, Japan). The XRD patterns of PZT having Zr/Ti equals to 52/48, 51/49, 50/50, 49/51 and 48/52 are shown in the Figure 4.4, 4.5, 4.6, 4.7 and 4.8 respectively.

The intensity of the PZT peak having Zr/Ti equal to 52/48 is found to be maximum value of 5500 (approx.) at 2θ of 31.6888°. The maximum value of the intensity of the PZT peak having Zr/Ti equal to 51/49 is 6000 (approx) at 2θ of 31.6888°. In case of the PZT having Zr/Ti equal to 50/50, the intensity of the PZT peak is observed to be maximum value of 8000 (approx.) at 2θ of 31.6888°. Two PZT peaks are found to have almost same intensity of 2000 (approx.) at 2θ of 54.2592° and 80.8265° in case of PZT with Zr/Ti equal to 49/51. In case of PZT having Zr/Ti equal to 48/52, two PZT peaks have almost same intensity of 1250 (approx.) at 2θ of 31.6888° and 54.2984°. So, from these observations, it can be concluded that the intensity of the PZT peak value first increases with increasing the Ti content from 48% to 50% and then decreases with increasing the Ti content from 50% to 52%.

5.2 SEM RESULTS

The SEM micrographs, grain size and morphology were carried out with FEI Quanta microscope (US). The SEM images of PZT having Zr/Ti equals to 51/49, 50/50, 49/51 are shown in the Figure 4.10, 4.11 and 4.12 respectively.

In all the samples of PZT, the fracture mode is completely inter-granular. The average grain sizes of about 6 µm, 5 µm and 8 µm is observed in case of PZT having Zr/Ti ratio equal to 51/49, 50/50, 49/51 respectively. So, it can be concluded that the average grain size at first decreases with increasing the Ti content from 49% to 50% and then increases with increasing the Ti content from 50% to 51%.

5.3 RESULTS OF PERCENTAGE CHANGE IN LENGTH AND WIDTH

Length and width of the PZT samples having different Zr/Ti ratios such as **52/48, 51/49, 50/50, 49/51, 48/52** were measured before sintering and after sintering. Effect of change in length and width due to variation of composition is shown in Table 4.1 and Table 4.2 respectively.

From the results obtained, it can be concluded that the percentage shrink in length at first decreases significantly with increasing Ti content from 48% to 50% and then increases with increasing Ti content from 50% to 52% (little change is found when Ti content increases from 51% to 52%). The percentage change in length is found to be maximum for PZT having Zr/Ti equal to 48/52. It can also be concluded that the percentage shrinkage in width decreases slightly with increasing Ti content from 48% to 52% but there is an exception when the Ti content is equal to 51%. The percentage change in width is found to be maximum for PZT having Zr/Ti equal to 49/51.

5.4 FUTURE SCOPE OF THIS THESIS

This thesis work has got a lot of future scope such as making of an energy harvester or vibration sensor from the rectangular beam fabricated. The beam can be used as a cantilever to produce energy as discussed in section 1.4. The electrical characterization of the beam will help in understanding the amount of charge it can create by applying a known amount of dynamic pressure. After the characterization the beams of different Zr/Ti ratio can be compared to see which one produces most charge and that can be further used to study the energy harvesting property of PZT.

In today’s fast advancing world, we need to find more and more ways to produce green electricity to reduce carbon emission. PZT is the most sensitive piezoelectric material for energy harvesting. This thesis work has completed the fabrication of the PZT rectangular beam. The beam is now ready for future work on it.