Preliminary Study on the Electromechanical Characterization of Commercial Lead Zirconate Titanate Piezoelectric Ceramic Materials for Flying Micro Robot Actuators

by

Halimatulssaadiah binti Wan Jusoh

Dissertation submitted in partial fulfilment of
the requirements for the
Bachelor of Engineering (Hons)
(Mechanical Engineering)

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CERTIFICATION OF APPROVAL

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Approved by,

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(AP Dr. Bambang Ari Wahjoedi)

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

December 2009

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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HALIMATULSSAADIAH BINTI WAN JUSOH

ABSTRACT

The objectives of this project are to compare the characteristics of several commercial ceramic disks of Lead Zirconate Titanate as well as to measure the piezoelectric coefficient, study the microstructure and the correlation between the microstructure and the piezoelectric coefficient so that we can come up with the best ceramic disk for the Flying Micro Robot Actuators application.

Currently, there are a lot of commercial Lead Zirconate Titanate piezoelectric ceramics offered in the market that are broadly used as mechanically resonant oscillators in electric circuitry, as electromechanical transducers such as underwater sound detectors and also as actuators in micromechanical flying insects.

In addition to the simple mechanical structure, other beneficial general properties of piezoelectric actuators that are suitable for a Flying Micro Robot Actuators are; a short response time, an ability to create high forces, a high efficiency and a high mechanical durability. On the disadvantage side, piezoelectric actuators have small strains: only 0.1-0.2% and a high supply voltage are needed – typically between 60 and 1000 Volts.

Keywords for this project would include piezoelectric ceramic, resonance, pressure sensor, impedance, Lead Zirconate Titanate, piezoelectric coefficient, flying micro robot actuators and microstructure.

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CHAPTER 1

INTRODUCTION

1.0 INTRODUCTION

1.1 Background Of Study

Piezoelectric ceramics are known for what are called the piezoelectric and reverse piezoelectric effects. The piezoelectric effect causes a crystal to produce an electrical potential when it is subjected to mechanical vibration. In contrast, the reverse piezoelectric effect causes the crystal to produce vibration when it is placed in an electric field. Of piezoelectric materials, Rochelle salt and quartz have long been known as single-crystal piezoelectric substances. However, these substances have had a relatively limited application range chiefly because of the poor crystal stability of Rochelle salt and the limited degree of freedom in the characteristics of quartz. Recently, a Lead Titanate-Lead Zirconate system ($PbTiO_3$-$PbZrO_3$) appeared which has electromechanical transformation efficiency and stability including temperature characteristics far superior to existing substances. It has dramatically broadened the application range of piezoelectric ceramics [1].

Apart from that, the piezoelectric actuators are widely used in smart structure applications due to their high bandwidth, high output force, compact size, and high power density properties [2]. For such reasons they are very appealing for mobile micro robotic applications such as the Micromechanical Flying Insect (MFI) where, because of strict size or weight constraints and smart structures capable of both actuating and sensing are preferred [3].

1.2 Problem Statement

Currently, there are various of commercial Lead Zirconate Titanate piezoelectric ceramics available in the market that are widely used as mechanically resonant oscillators in electric circuitry, as electromechanical transducers such as in underwater sound detectors and also as actuators in Micromechanical Flying Insects.

As an example, in addition to the simple mechanical structure, other beneficial general properties of piezoelectric actuators that are suitable for a Micromechanical Flying Insects actuators are; a short response time, an ability to create high forces, a high efficiency and a high mechanical durability. On the disadvantage side, piezoelectric actuators have small strains: only 0.1-0.2% and a high supply voltage are needed – typically between 60 and 1000 Volts.

1.2.1 Problem Identification

The applications of Lead Zirconate Titanate piezoelectric ceramics are so wide but the characteristics of these piezoelectric actuators are not properly identified and catalogued by the industry. The correlation of piezoelectric coefficient with the properties and microstructure are discussed in this project.

1.2.2 Significance of Project

The significance of this project is to integrate the differing methods, and ultimately provide industries with a more reliable and robust means of characterizing piezoelectric materials for the application of Micromechanical Flying Insect Actuators. As this is critical to quality, it is hoped it will lead to improvements in the competitiveness.

1.3 Objective & Scope Of Study

The objectives of this project are to compare the characteristics of several commercial ceramic disks of Lead Zirconate Titanate as well as to measure the piezoelectric coefficient and study its correlation with microstructure so that we can come up with the best solution for the Flying Micro Robot Actuators application.

The whole project would start with the knowledge gathering. The scope of study includes studying the characteristics of piezoelectric ceramics along with the properties of typical piezoelectric materials. Taking the sample from several manufacturers would be the first step in this project. Then, experiments will be carried out to correlate the theoretical knowledge with practices. The experiments include the analytical technique involved in measuring the piezoelectric coefficient. Then, a methodology will be developed according to the step-by-step procedures from identifying, measuring, and analyzing the coefficient of the piezoelectric. Other scopes would include find out its correlation with microstructure by using Scanning Electron Microscopy (SEM). It is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition and other properties such as electrical conductivity. Further research would be continuously practiced to ensure satisfactory results are achieved.

1.3.1 Feasibility of Project

The project is feasible as it utilizes experiments and analysis of the experiments that is within the scope and time frame of two semesters. Besides, this project is low in cost for the experiments and brings huge benefits for the future.

CHAPTER 2

LITERATURE REVIEW

2.0 LITERATURE REVIEW

2.1 Definition And History Of Piezoelectric Ceramics

Piezoelectricity is a linear effect that is related to the microscopic structure of the solid. Some ceramic materials become electrically polarized when they are strained; this linear and reversible phenomenon is referred to as the direct piezoelectric effect. The direct piezoelectric effect is always accompanied by the converse piezoelectric effect where a solid becomes strained when placed in an electric field. The microscopic origin of the piezoelectric effect is the displacement of ionic charges within a crystal structure. In the absence of external strain, the charge distribution within the crystal is symmetric and the net electric dipole moment is zero. However, when an external stress is applied, the charges are displaced and the charge distribution is no longer symmetric. A net polarization develops and results in an internal electric field [5, 6].

Piezoelectricity is a property possessed by a group of materials, discovered in 1880 by Pierre and Jacques Curie during their study of the effects of pressure on the generation of electrical charge by crystals such as Quartz, Tourmaline, and Rochelle salt. In 1881, the term "piezoelectricity" was first suggested by W. Hankel, and the converse effect was deduced by Lipmann from thermodynamics principles. In the next three decades, collaborations within the European scientific community established the field of piezoelectricity; and by 1910, Voigt's "Lerbuch der Kristallphysic" was published and became a standard reference work detailing the complex electromechanical relationships in piezoelectric crystals. However, the complexity of the science of piezoelectricity made it difficult for it to mature to an application until a few years later [6].

Langevin et al. developed a piezoelectric ultrasonic transducer during World War I. Their success opened up opportunities for piezoelectric materials in underwater applications as well as a host of other applications such as ultrasonic transducers, microphones, accelerometers, etc. In 1935, Busch and Scherrer discovered piezoelectricity in potassium dihydrogen phosphate (KDP). The KDP family was the first major family of piezoelectric and ferroelectrics to be discovered.

During World War II, research in piezoelectric materials expanded to the U.S., the Soviet Union and Japan. Up until then, limited performance by these materials inhibited commercialization but that changed when a major breakthrough came with the discovery of barium titanate and lead zirconate titanate (PZT) in the 1940s and 1950s respectively. These families of materials exhibited very high dielectric and piezoelectric properties. Furthermore, they offered the possibility of tailoring their behavior to specific responses and applications by the use of dopants. To date, PZT is one of the most widely used piezoelectric materials [6].

2.2 Properties Of Piezoelectric Ceramics

Piezoelectric ceramics are a type of multi-crystal dielectric with a high dielectric constant and are formed by two processes: first is high temperature firing and second is polarization. After firing, they have the characteristic crystal structure shown in Fig. 1 (a) but do not yet exhibit the piezoelectric property because the electrical dipoles within the crystals are oriented at random and the overall moment of the dipoles is canceled out. To make ceramic piezoelectric, the dipoles must be polarized. A DC electric field of several kV/mm is applied to the piece of ceramic to align the internal electrical dipoles in a single orientation (see Fig. 1 (b)). Due to the strong dielectric property of the ceramic, the dipole moment remains unchanged after the electric field is removed, and the ceramic thus exhibits a strong piezoelectric property (see Fig. 1 (c)). When an AC signal is applied to a piezoelectric ceramics in a frequency matching the specific elastic frequency of the ceramics (which depends on the shape of the material), the ceramic exhibits resonance. Since the ceramic has a very high electromechanical transforming efficiency at the point of resonance, many applications use this resonance point. Also piezoelectric ceramics when molded in certain shapes have more than one point of resonance depending on vibration mode. In such a case, the vibration mode most suited for the application is selected [2].

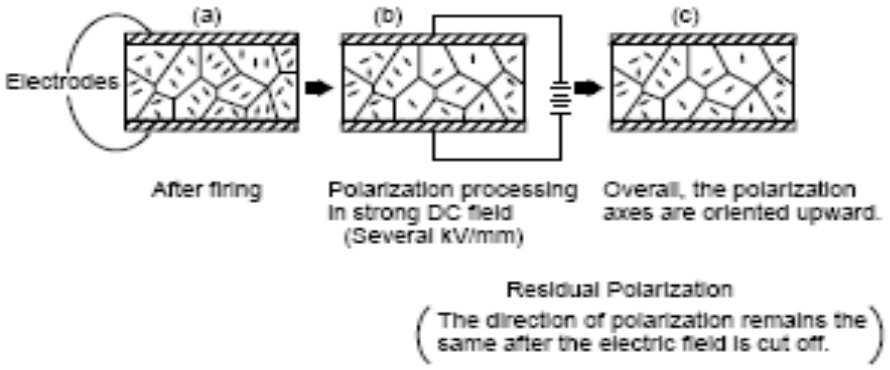


Figure 1 Polarization processing of Piezoelectric Ceramics [2]

2.3 Characteristics Of Piezoelectric Ceramics

When a piezoelectric material is subjected to stress T, it produces polarization P which is a linear function of T: P=*d*T (*d*: piezoelectric strain constant). This effect is called the normal piezoelectric effect. In contrast, when a piezoelectric substance has an electric field E applied across its electrodes, it produces distortion S which is a linear function of the electric field: S =*d*E. This effect is called the reverse piezoelectric effect [2]. For an elastic material, the relationship of distortion S to the stress T is given by S =*s*ET (*s*E: compliance); for a dielectric substance, the relationship of electrical displacement D with electric field strength E is given by D=εE. For a piezoelectric ceramic, these relationships are given by the following equations; both being associated with piezoelectric strain constants:

$$\left.\begin{aligned} S_i &= s_{ij}^{E} T_j + d_{mi} E_m \\ D_n &= d_{nj} T_j + \varepsilon_{nm}^{T} E_m \end{aligned}\right\} \rightarrow (1)$$

$$(m, n = 1,2,3; i, j = 1,2 \rightarrow (6)$$

These equations are called the basic piezoelectric equations, where the electric field E and electrical displacement D is represented in vector magnitudes; whereas stress T and distortions S are given in symmetrical tensile magnitudes. When the symmetry of the crystals is taken into account, Eq. (1) is simplified because some constants in the equations are nullified and some other constants become equal to a third set of constants.

With piezoelectric ceramics, when the polarization axis is placed along the *z* (3) axis and two arbitrary orthogonal axes (which are also orthogonal to the *z* axis and assumed to be the *x* (1) and *y* (2) axis), the crystal structure of the ceramic can be represented in the same way as that of 6mm crystals, in which case the only independent non-zero coefficients are the following ten constants:

$$s_{11}^{E}\left(\frac{1}{Y_{11}^{E}}\right), s_{12}^{E}\left(\frac{1}{Y_{12}^{E}}\right), s_{13}^{E}\left(\frac{1}{Y_{13}^{E}}\right), s_{33}^{E}\left(\frac{1}{Y_{33}^{E}}\right), s_{44}^{E}\left(\frac{1}{Y_{44}^{E}}\right),$$
$$d_{31}, d_{33}, d_{15}, \varepsilon_{11}^{T}, \varepsilon_{33}^{T}$$

The proportionality constant of charge to stress (direct effect) is equal to that of strain to field (inverse effect). Either is called the piezoelectric coefficient, d_{ij}.

$$d_{ij} = \frac{charge/area}{force/area} = \frac{coul/m^2}{Newtons/m^2} = \frac{coul}{Newtons} = \frac{coul \times m}{Newtons \times m} = \frac{coul \times m}{coul \times volt} = \frac{m}{volt}$$

or

$$d_{ij} = \frac{strain}{field} = \frac{meters/meter}{volts/meter} = \frac{m}{volt}$$

The subscript *I* denotes the direction of the electric field (applied or generated) and is given values of 1, 2, 3 for the *x*, *y*, and *z* axes with 3, by convention being the axis of polarization. The subscript *j* stands for the type and direction of mechanical stress or strain and has values of 1, 2, 3 for compression along *x*, *y*, and *z* axes and 4, 5, 6 for shear stresses with the plane of shear perpendicular to the 1, 2 ,3 axes respectively. If a compressive stress is applied perpendicular to the faces of a disc-shaped specimen, a charge density is generated on the electrodes on the faces of the discs, so that

$$q = d_{33}P$$

which is equivalent to

$$Q = d_{33}F$$

where Q is the total charge released when a force F is applied. The charge generated therefore is a function of the force applied and the characteristic of the material and not of the size of the sample. Since disc-shaped elements are used for many piezoelectric applications, d_{33} is an important material constant. High *d*-coefficients are desirable in materials utilized as actuators, such as in motional and vibrational applications [11].

2.4 Application Of Piezoelectric Ceramics

Piezoelectric materials are used as mechanically resonant oscillators in electronic circuitry, as electromechanical transducers such as underwater sound detectors, as accelerometers, phonograph pick-ups, load cells, strain gauges, igniters for automobiles and ammunition, etc. Below are the lists of other application of piezoelectric ceramics [9, 11].

- Mechanical power sources (electrical-to-mechanical transducers):
  Piezoelectric actuators, piezoelectric fans, ultrasonic cleaners, and etc.
- Sensors (mechanical-to-electrical transducers):
  Ultrasonic sensors, knocking sensors, shock sensors, acceleration sensors, etc.
- Electronic circuit components (transducers):
  Ceramic filters, ceramic resonators, surface acoustic wave filters, etc.

2.4.1 Micromechanical Flying Insects (MFI)

The Micromechanical Flying Insect (MFI) project aims to create an autonomous flying robotic insect the size of a housefly. To do this, biologists have identified mechanisms in real insects which are necessary for flight, thus solving the mystery of how insects produce adequate lift for stable flight. The results show that to produce enough lift, while also having a structure which is capable of creating thrust vectors necessary for control of the insect, there are a number of key kinematics and dynamic requirements [5, 6].

The MFI wings must be capable of independently going through a wing stroke of 120±, while being able to rotate 90± at a resonant frequency of 150*Hz*. To do this the body of the MFI consists of two wings, each driven by separate thorax structures.

The thorax structures consist of two actuators, two mechanically amplifying four-bar structures, and a differential. Since the work done on the air is proportional to the velocity of the wing squared, the most important requirements are a high resonant frequency and a large stroke angle [5, 6].

To keep the weight of the thorax low, the MFI uses unimorph piezoelectric actuators. Such actuators produce large forces and small displacements, thus it is necessary to use a mechanical amplifier to transform these small displacements into large wing strokes. The actuators currently use stainless steel as the elastic layer, thus again causing high inertias and construction problems. By utilizing the high stiffness to weight ratios of composites this inertia can be decreased while allowing for the tailoring of the anisotropy of the layer. This anisotropy makes it possible to create an actuator with kinematic properties that allow simplifications to be made within the kinematics of the four-bar structure. The figure below shows an example of MFI. [5, 6]

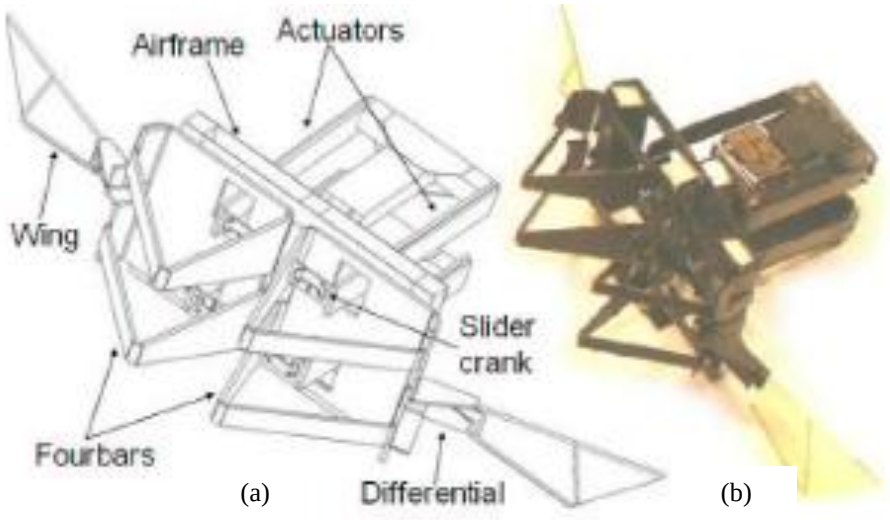


Figure 2 (a) Detail drawing of 26 joints, 4 actuator, 4 DOF, 2 wings MFI,

(b) Prototype of MFI

CHAPTER 3

METHODOLOGY

3.0 METHODOLOGY

Before proceeding with the experiment itself, steps were drawn out diagrammatically to clear out on the flow. Firstly, measure the change in the impedance of each load when a fixed frequency, the anti-resonance frequency, is applied to the Lead Zirconate Titanate. Impedance changes can be measured according to changes of current via Ohm's law, and thus pressure is measured by voltage in the Lead Zirconate Titanate piezoelectric ceramic. The load is increased from 0 kg to 3 kg, and the result is measured in voltage. It is expected that the increasing in load will result in increasing of voltage reading. Secondly, collect the test results and characterize response of the load tests. Thirdly, calculate the piezoelectric coefficient at different loads and plot the graph of piezoelectric coefficient, d_{33} versus load followed by analyzing and synthesizing results as well as drawing conclusions. Besides, the correlation between piezoelectric ceramics and the microstructure will be studied too by doing the Scanning Electron Microscopy (SEM). High *d*-coefficients are desirable in materials utilized as actuators, such as in motional and vibrational applications so I look forward for the sample that has the highest *d*-coefficients.

Below are the rough methodologies of this project

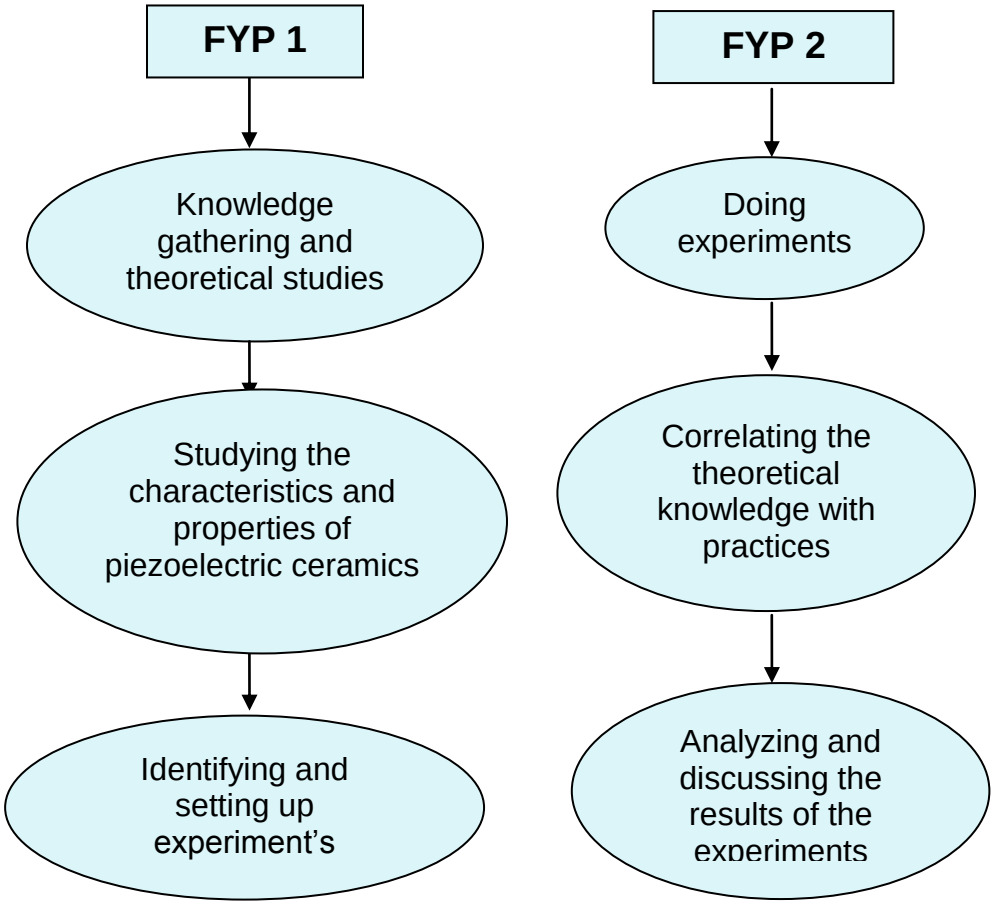


Figure 3 Methodology

*Please refer Appendix for the Gantt chart

3.1 Project Activities

Below are the steps and explanation on the experiment expansion.

3.1.1 Measuring the piezoelectric coefficient

1. Turn on the electrometer and let it warm up for 5 minutes.
2. Place one of the Lead Zirconate Titanate ceramic disks in the sample holder.
3. Connect to the electrometer using a capacitor of appropriate capacitance to obtain the readings in the range of the electrometer.
4. The charge generated by the sample due to the application and removal of three known loads, is obtained from $Q=CV$. Be sure that V is within the rating of capacitor.
5. Measure the charge due to the unknown weight.
6. Repeat the measurement on the different Lead Zirconate Titanate specimen by repeating steps 2, 3, 4 and 5.

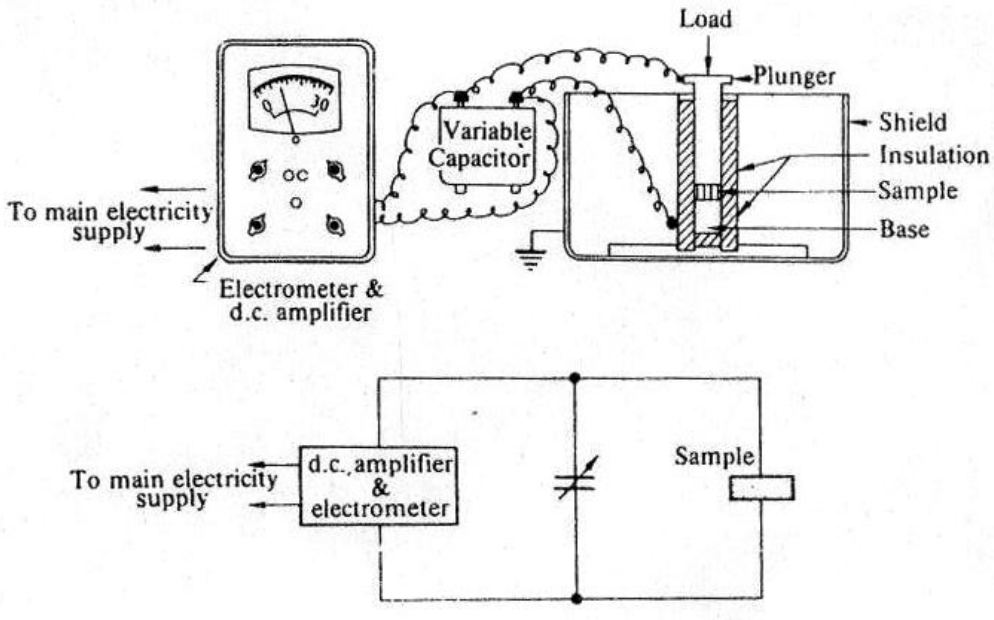


Figure 4 Experimental set up for measuring piezoelectric constant, d_{33} (top) and the electrical connections (bottom). The insulation is shaped from Teflon or Nylon rod and the base and plunger are made of brass

3.1.2 Scanning Electron Microscope (SEM)

The scanning electron microscope (SEM) is a type of electron microscope that images the sample surface by scanning it with a high-energy beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition and other properties such as electrical conductivity. [12]

The types of signals produced by an SEM include secondary electrons, back scattered electrons (BSE), characteristic x-rays, light (cathodoluminescence), specimen current and transmitted electrons. These types of signal all require specialized detectors for their detection that are not usually all present on a single machine. The signals result from interactions of the electron beam with atoms at or near the surface of the sample.

All samples must be of an appropriate size to fit in the specimen chamber and are generally mounted rigidly on a specimen holder called a specimen stub. Several models of SEM can examine any part of a 6-inch (15 cm) semiconductor wafer, and some can tilt an object of that size to 45 degrees.

Metals, geological specimens, and integrated circuits all may also be chemically polished for viewing in the SEM. Special high resolution coating techniques are required for high magnification imaging of inorganic thin films.

Specimens that are studied in the SEM can be divided into two categories, namely conductors and non-conductor. However there are several factors to consider during all specimens preparation:

1. The size and weight necessitate some reduction of the specimen to fit holder and to ease the specimen manipulation for observation.
2. The specimen should be able to withstand the high vacuum of the SEM as it might be deformed.

3. It should be clean and dry, i.e. free of dust, moisture, oils and grease as their presence can lead to charging, contamination and longer pump down times.
4. Porous samples will also take a long time to pump down.
5. Coat non-conductors to prevent charging.
6. There should be good electrical connection between the surface of the specimen and specimen holder.

The procedures in Scanning Electron Microscopy (SEM) are;

1. The specimens were coated with gold using the sputter coater for 15 minutes.
2. The specimens then were examined with LEO 1430VP Scanning Electron Microscope with appropriate magnification and field of view.
3. The microscope has been linked to a computer which could display and store the microstructure images. In the computer, the AcQuis Software has been launched and the photomicrographs have been captured to save in JPEG file format.
4. All the photomicrographs of the specimens will be hand-sketch with the most appropriate magnification (usually>100x).
5. The field of view will be indicated and drawings will be annotated carefully with the corresponding micro-constituents identity.
6. All the relevant microscopic observation in results is recorded.

3.1.3 Energy Dispersive X-Ray Spectroscopy

Energy dispersive X-ray spectroscopy (EDS) is an analytical technique used for the elemental analysis or chemical characterization of a sample. It is one of the variants of XRF. As a type of spectroscopy, it relies on the investigation of a sample through interactions between electromagnetic radiation and matter, analyzing x-rays emitted by the matter in response to being hit with charged particles. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing x-rays that are characteristic of an element's atomic structure to be identified uniquely from each other. [13]

To stimulate the emission of characteristic X-rays from a specimen, a high energy beam of charged particles such as electrons or protons, or a beam of X-rays, is focused into the sample being studied. At rest, an atom within the sample contains ground state electrons in discrete energy levels or electron shells bound to the nucleus. The incident beam may excite an electron in an inner shell, ejecting it from the shell while creating an electron hole where the electron was. An electron from an outer, higher-energy shell then fills the hole, and the difference in energy between the higher-energy shell and the lower energy shell may be released in the form of an X-ray. The number and energy of the X-rays emitted from a specimen can be measured by an energy dispersive spectrometer. As the energy of the X-rays are characteristic of the difference in energy between the two shells, and of the atomic structure of the element from which they were emitted, this allows the elemental composition of the specimen to be measured. [13]

There are four primary components of the EDS setup: the beam source; the X-ray detector; the pulse processor; and the analyzer. EDS systems are most commonly found on scanning electron microscopes since scanning electron microscopes are equipped with a cathode and magnetic lenses to create and focus a beam of electrons. A detector is used to convert X-ray energy into voltage signals; this information is sent to a pulse processor, which measures the signals and passes them onto an analyzer for data display and analysis.

The excess energy of the electron that migrates to an inner shell to fill the newly-created hole can do more than emit an X-ray. Often, instead of X-ray emission, the excess energy is transferred to a third electron from a further outer shell, prompting its ejection. This ejected species is called an Auger electron, and the method for its analysis is known as Auger Electron Spectroscopy. [12]

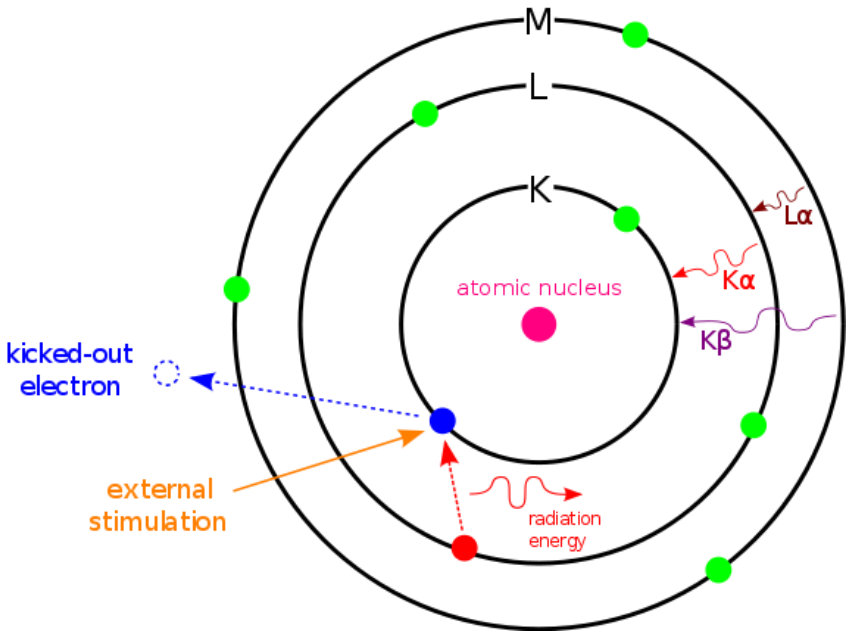


Figure 5 The principle of EDS

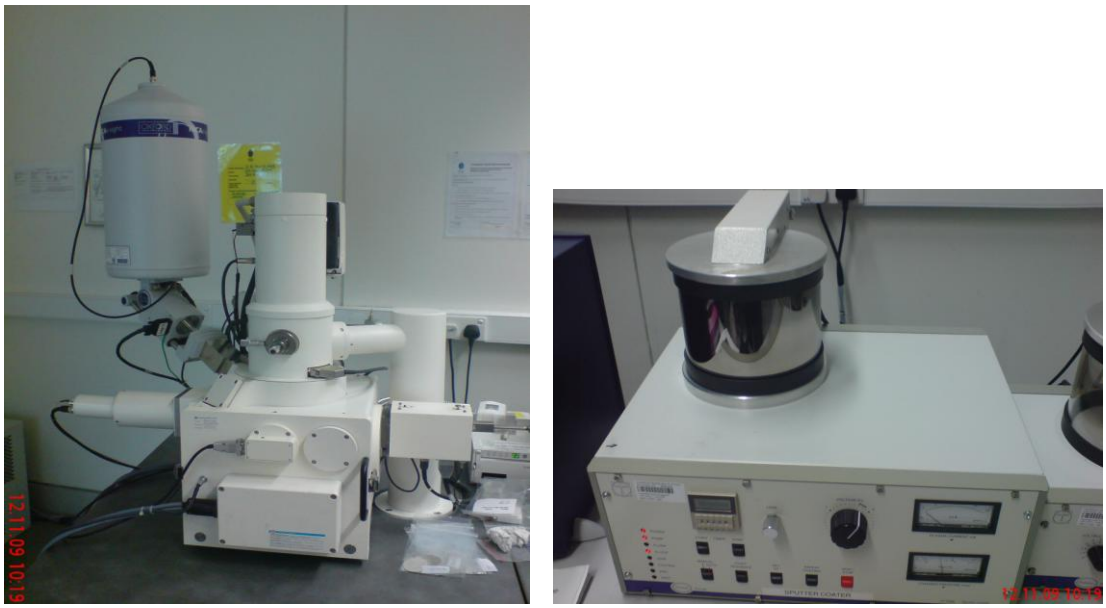
The procedures in Energy Dispersive X-Ray Spectroscopy (EDS) are;

1. The specimens were coated with gold using the sputter coater for 15 minutes.
2. The specimens then were examined with LEO 1430VP Scanning Electron Microscope with appropriate magnification and field of view.
3. The microscope has been linked to a computer which could display the chemical characterization of the sample. In the computer, the Oxford Inca Software has been launched and the characteristic of the element's atomic structure have been analyzed to save in Microsoft Words file format.
4. The field of view will be indicated carefully with the corresponding micro-constituents identity.
5. All the relevant chemical characterization in results is recorded.

3.2 Tools/Equipment Required

The tools and equipment which are required in this project are;

- Piezoelectric ceramic disks of Lead Zirconate Titanate from different manufacturers (1 to 3 cm in diameter and 1 to 2 mm in thickness)
    - China Ideal Mechanical & Electrical Equipment Group Company Ltd. (FMQ 2724 and FMQ 2012)
    - Xinhua Huaxing Machinery & Electronics Co. Ltd (SFM 27)
- Sample holder
- Set of known capacitors or variable standard decade capacitor (0.01 to 1 µF)
- Some known and one unknown weights (500 to 3000 g)
- Electrometer
- LEO 1430VP scanning electron microscope (refer Figure 5a)
- Sputter Coater (refer Figure 5b)
- Computer



(a) (b)

Figure 6 (a) LEO 1430VP Scanning Electron Microscope and (b) Sputter Coater

CHAPTER 4

RESULTS AND DISCUSSION

4.0 RESULTS AND DISCUSSION

4.1 Experiment Observation

4.1.1 Experiment 1: Measuring the piezoelectric coefficient.

The data is tabulated as follows:

Sample 1: FMQ 2724 from China Ideal Mechanical & Electrical Equipment Group Company Ltd

Table 1: The result for sample 1

Weight, kg	Force, F (Newton)	Capacitance, C (Farad)	Voltage, V (Volts)	Charge, Q (=CV) (coulombs)	d_{33} (=Q/F) (Coul/Newton)
0.5	5	1×10^{-8}	0.068	68×10^{-11}	136×10^{-12}
1.0	10	1×10^{-8}	0.135	135×10^{-11}	135×10^{-12}
1.5	15	1×10^{-8}	0.203	203×10^{-11}	135×10^{-12}
2.0	20	1×10^{-8}	0.270	270×10^{-11}	135×10^{-12}
2.5	25	1×10^{-8}	0.338	338×10^{-11}	135×10^{-12}
3.0	30	1×10^{-8}	0.405	405×10^{-11}	135×10^{-12}

Sample 2: FMQ 2012 from China Ideal Mechanical & Electrical Equipment Group Company Ltd

Table 2: The result for sample 2

Weight, kg	Force, F (Newton)	Capacitance, C (Farad)	Voltage, V (Volts)	Charge, Q (=CV) (coulombs)	d_{33} (=Q/F) (Coul/Newton)
0.5	5	1×10^{-8}	0.136	136×10^{-11}	272×10^{-12}
1.0	10	1×10^{-8}	0.271	271×10^{-11}	271×10^{-12}
1.5	15	1×10^{-8}	0.407	407×10^{-11}	271×10^{-12}
2.0	20	1×10^{-8}	0.542	542×10^{-11}	271×10^{-12}
2.5	25	1×10^{-8}	0.678	678×10^{-11}	271×10^{-12}
3.0	30	1×10^{-8}	0.813	813×10^{-11}	271×10^{-12}

Sample 3: SFM 27 from Xinhua Huaxing Machinery & Electronics Co. Ltd

Table 3: The result for sample 3

Weight, kg	Force, F (Newton)	Capacitance, C (Farad)	Voltage, V (Volts)	Charge, Q (=CV) (coulombs)	d_{33} (=Q/F) (Coul/Newton)
0.5	5	1×10^{-8}	0.205	205×10^{-11}	410×10^{-12}
1.0	10	1×10^{-8}	0.410	410×10^{-11}	410×10^{-12}
1.5	15	1×10^{-8}	0.615	615×10^{-11}	410×10^{-12}
2.0	20	1×10^{-8}	0.820	820×10^{-11}	410×10^{-12}
2.5	25	1×10^{-8}	1.025	1025×10^{-11}	410×10^{-12}
3.0	30	1×10^{-8}	1.230	1230×10^{-11}	410×10^{-12}

Graph

The graph below shows the relationship between the load applied on the samples and the voltage readings on the electrometer. The voltage is directly proportional to the load applied with the piezoelectric coefficient, d_{33} and capacitance, C being the constant of proportionality and it is proved from the equation earlier:

$$Q = CV = d_{33}F$$

$$V = \frac{d_{33}}{C}F$$

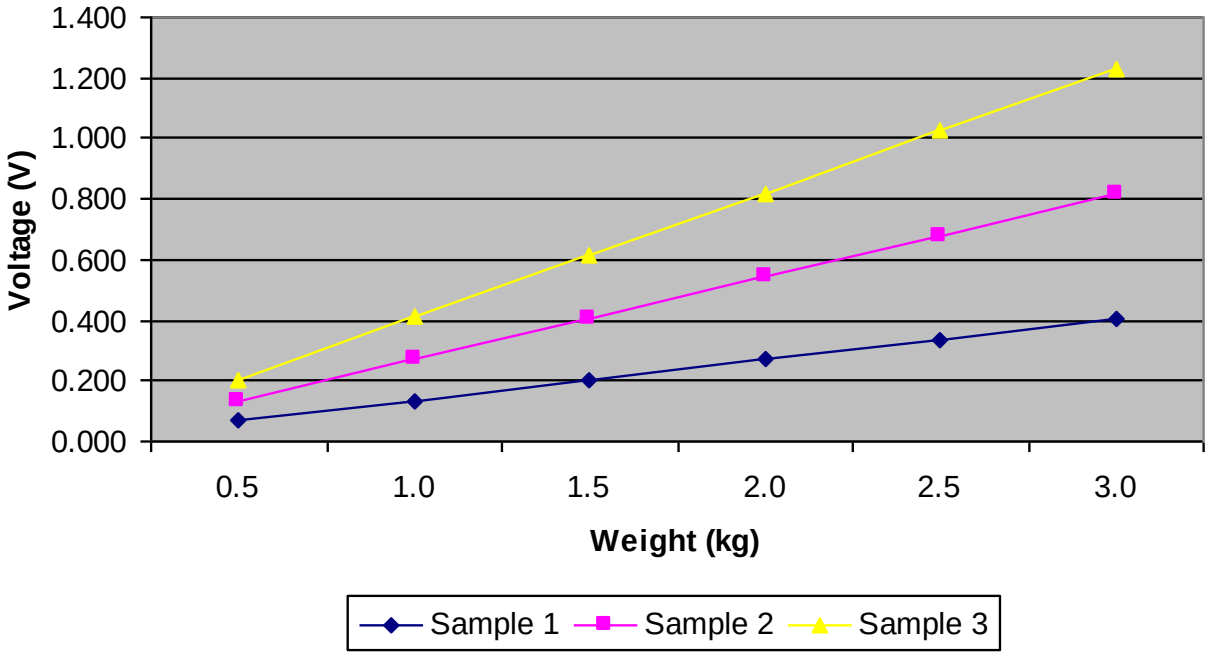


Figure 7 Relationship between the load applied and the voltage output

The graph below shows the relationship between the load applied on the samples and the piezoelectric coefficient measured from the voltage reading. The piezoelectric coefficient, d_{33} remain the same with the increasing of load applied.

$$Q = CV = d_{33}F$$

$$d_{33} = \frac{Q}{F}$$

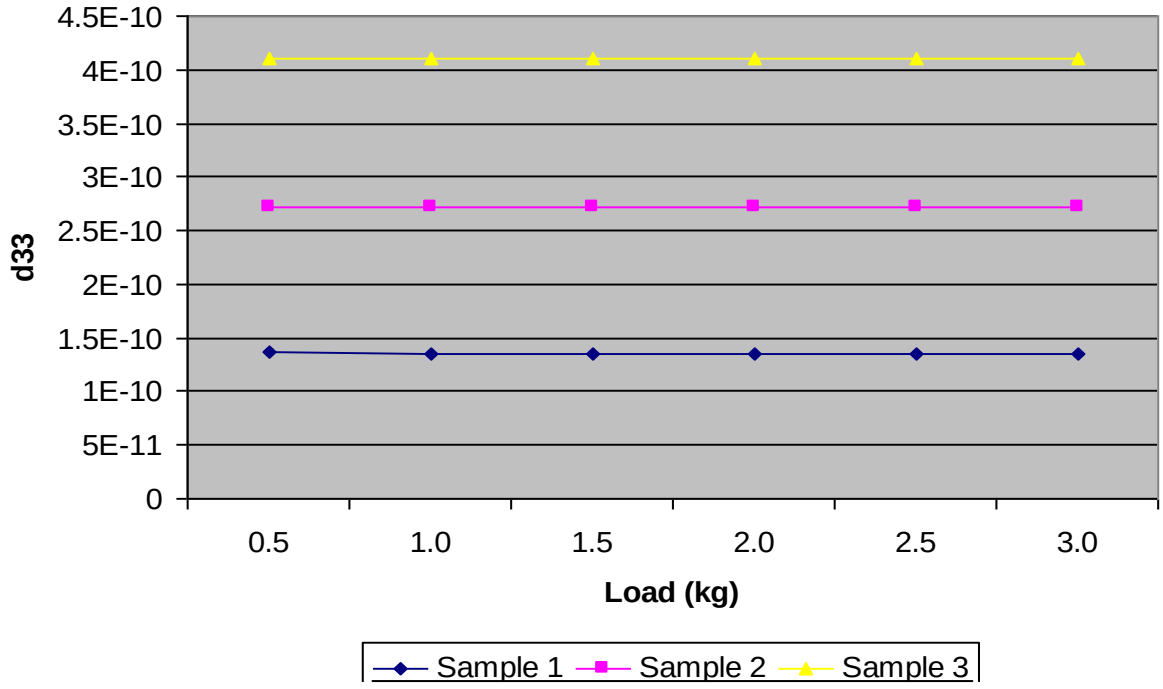


Figure 8 Piezoelectric coefficient, d_{33} vs load graph

4.1.2 Scanning Electron Microscope

Sample 1: FMQ 2724 from China Ideal Mechanical & Electrical Equipment Group Company Ltd.

- Under 100X magnifications

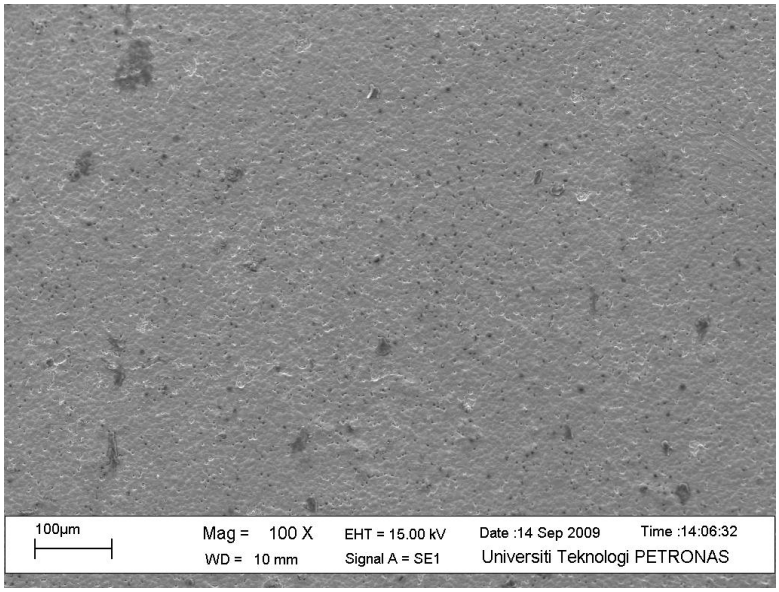


Figure 9 Under 100X magnification

- Under 1000X magnifications

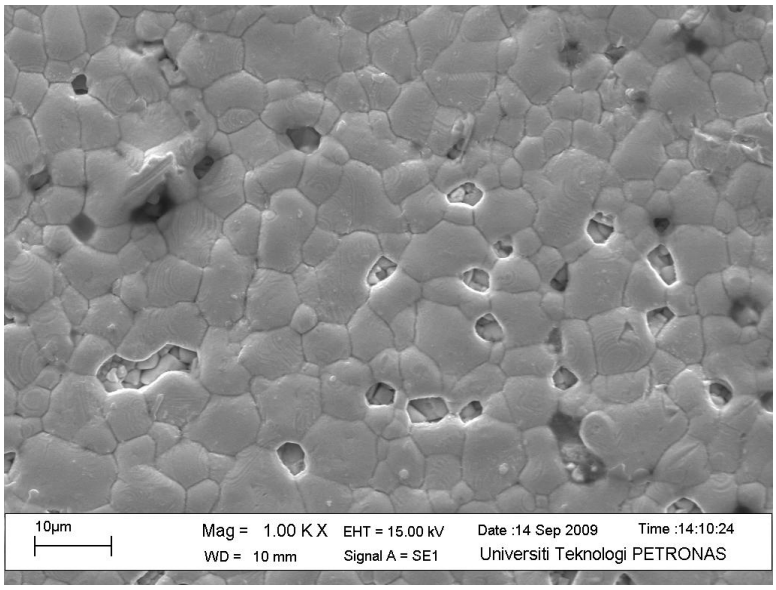


Figure 10 Under 1000X magnification

- Under 5000X magnifications

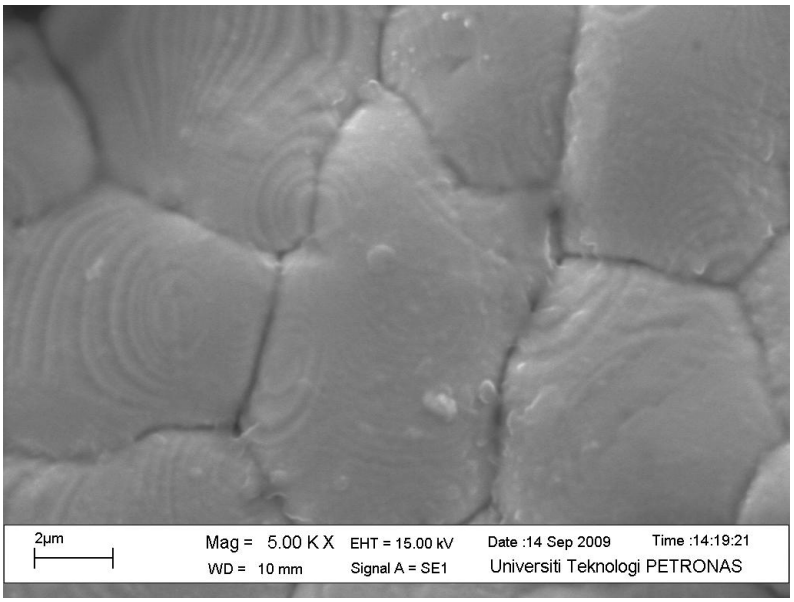


Figure 11 Under 5000X magnification

Sample 2: FMQ 2012 from China Ideal Mechanical & Electrical Equipment Group Company Ltd.

- Under 100X magnifications

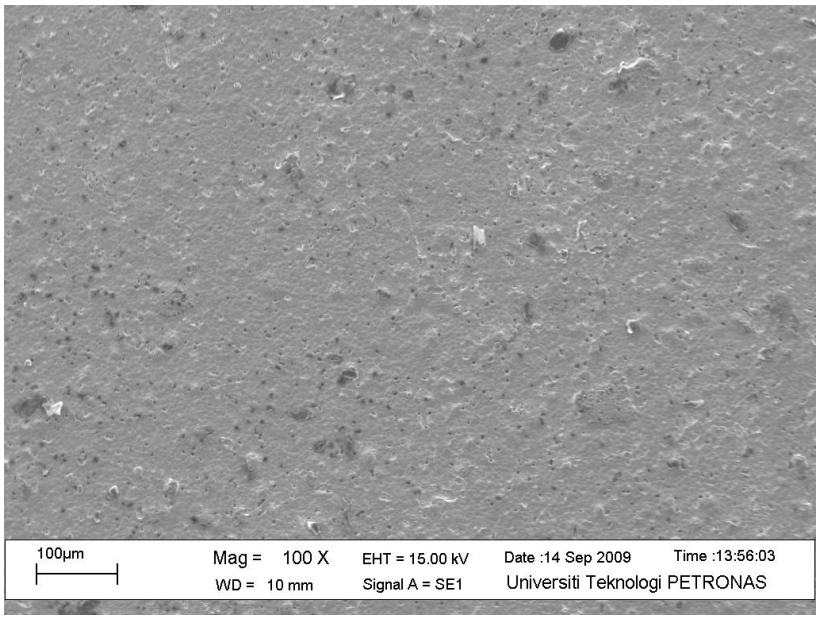


Figure 12 Under 100X magnification

- Under 1000X magnifications

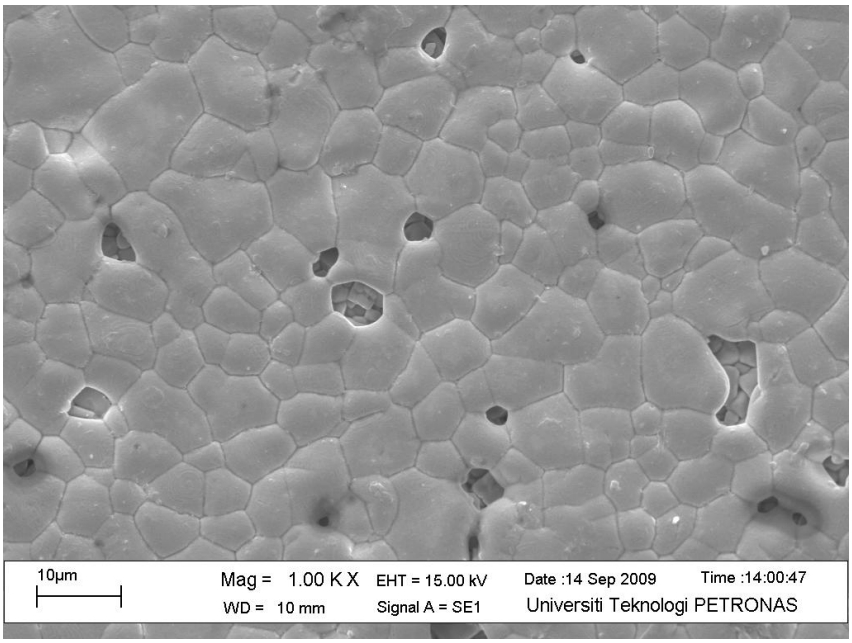


Figure 13 Under 1000X magnification

- Under 5000X magnification

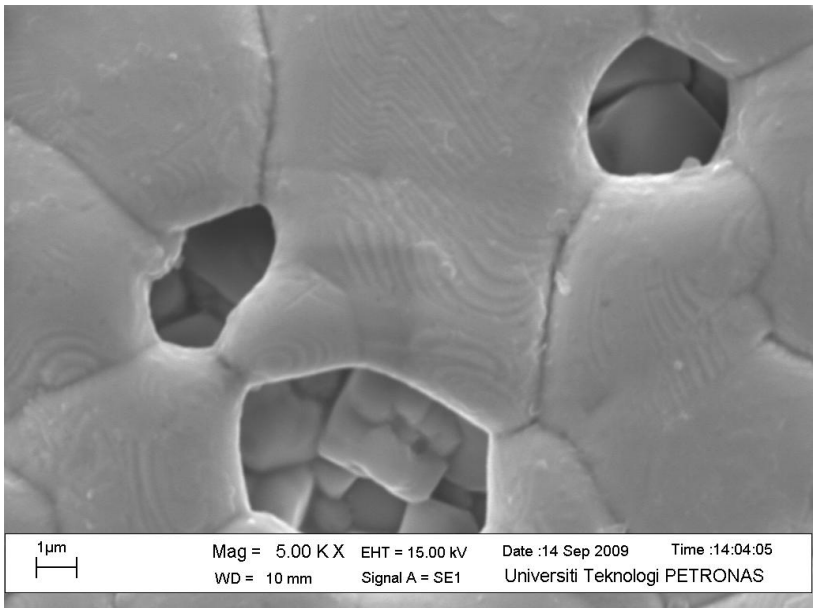


Figure 14 Under 5000X magnification

Sample 3: SFM 27 from Xinhua Huaxing Machinery & Electronics Co. Ltd

- Under 100X magnifications

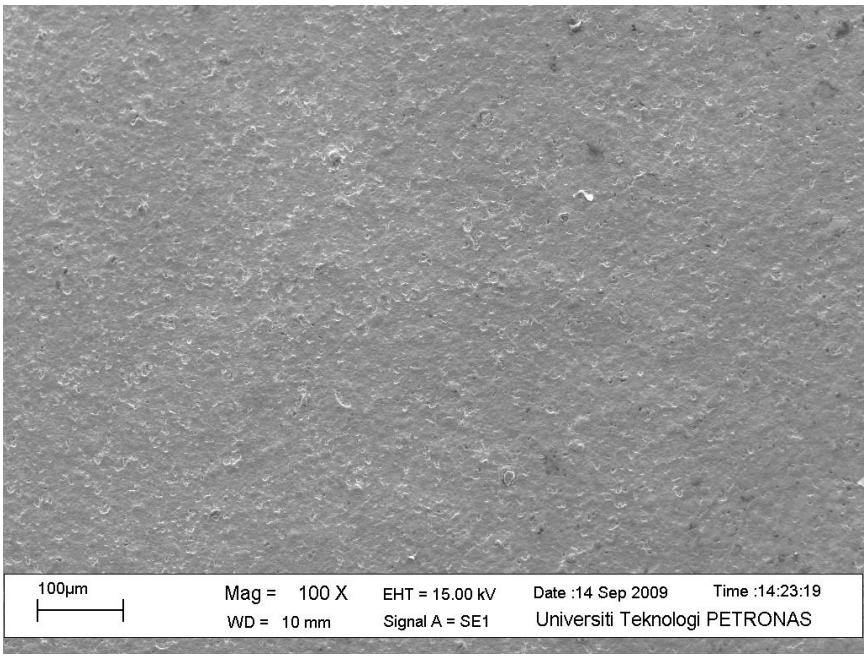


Figure 15 Under 100X magnification

- Under 1000X magnifications

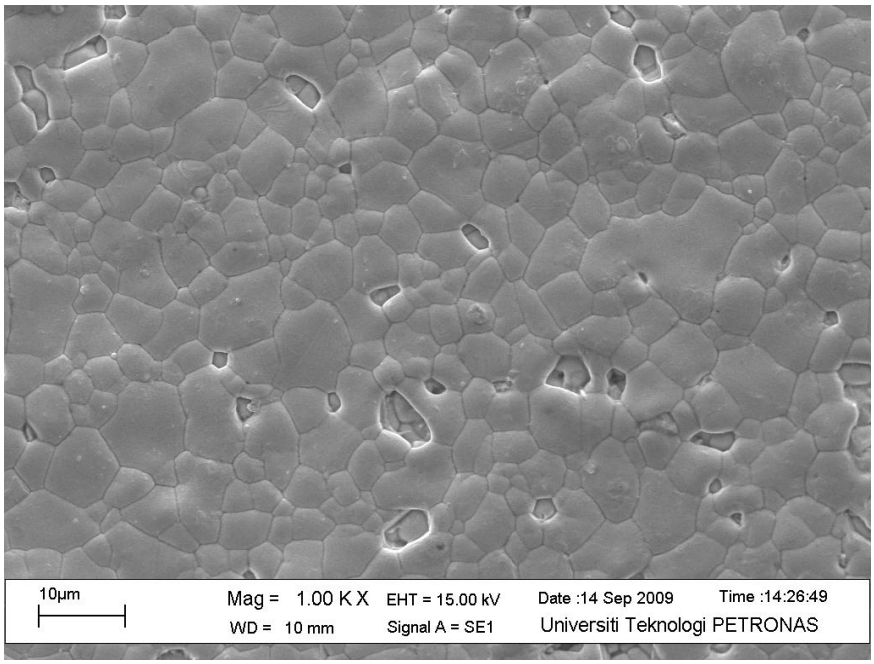


Figure 16 Under 1000X magnification

- Under 5000X magnifications

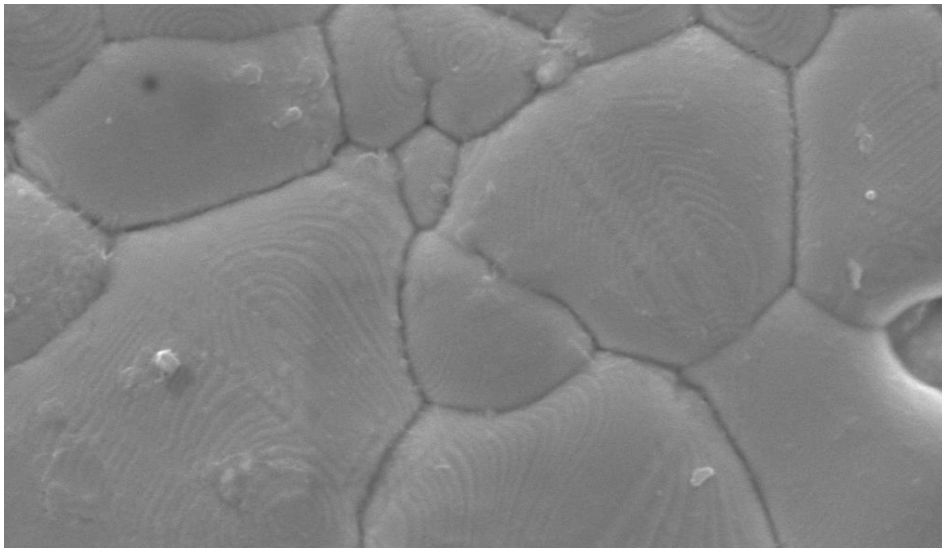


Figure 17 Under 5000X magnification

4.1.3 Energy Dispersive X-ray Spectroscopy

Sample 1: FMQ 2724 from China Ideal Mechanical & Electrical Equipment Group Company Ltd.

Table 4: The concentration of elements in Sample 1

Element	Weight %	Atomic %
C K	5.01	26.39
O K	5.46	21.56
Ag L	88.00	51.58
Pb M	1.53	0.47
Totals	100.00	

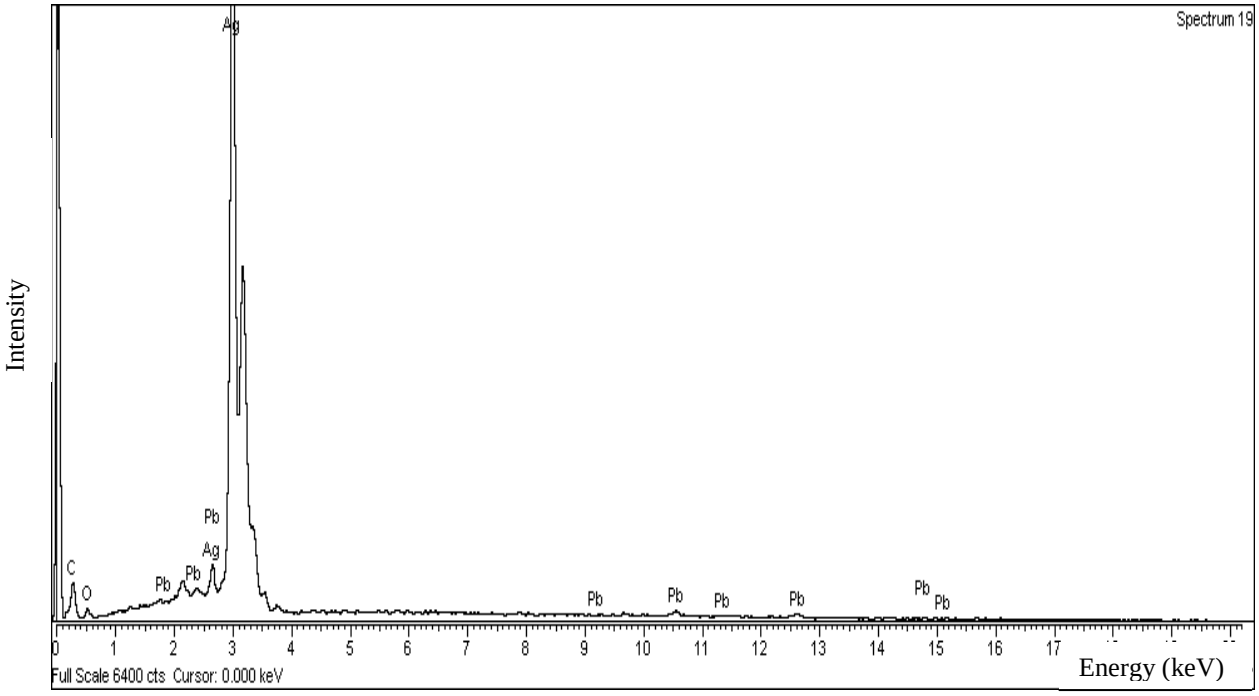


Figure 18 EDX result for sample 1

Sample 2: FMQ 2012 from China Ideal Mechanical & Electrical Equipment Group Company Ltd.

Table 5: The concentration of elements in Sample 2

Element	Weight %	Atomic %
C K	7.70	33.44
O K	7.85	25.58
Cl K	0.19	0.28
Ag L	84.04	40.64
Pb M	1.53	0.47
Totals	100.00	

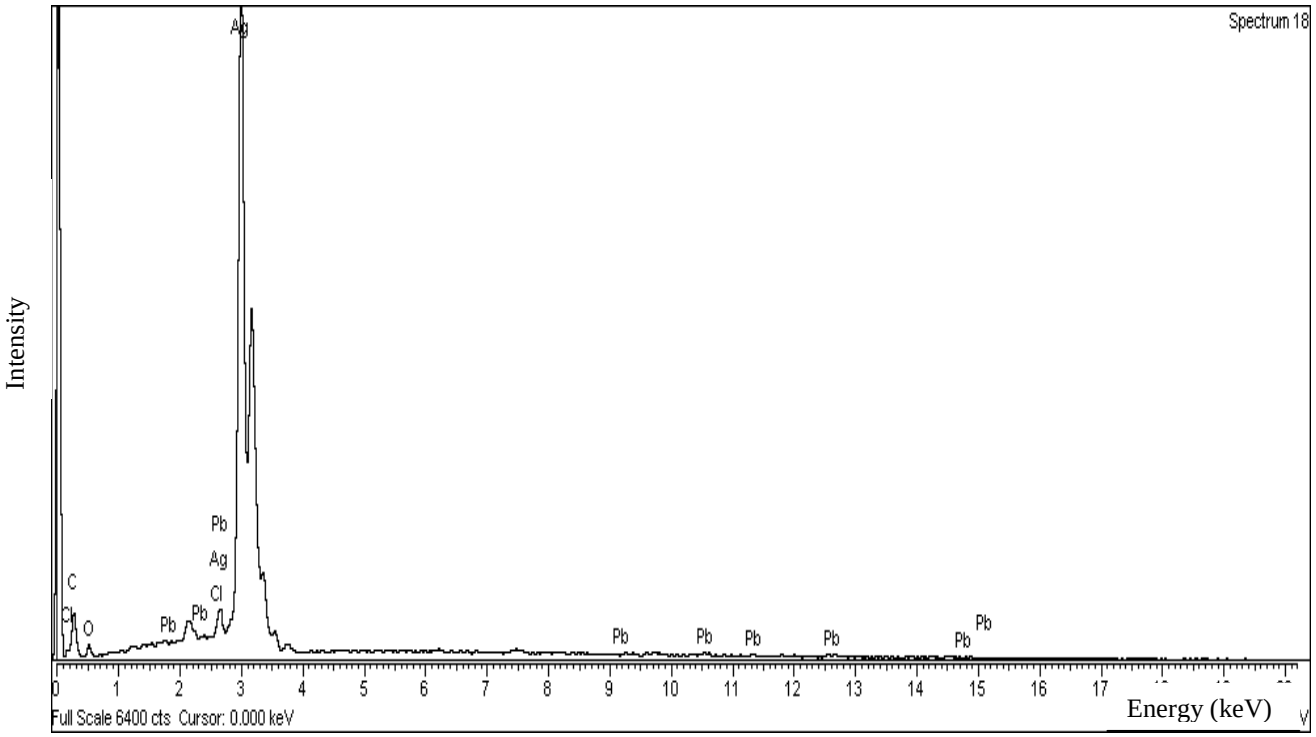


Figure 19 EDX result for sample 2

Sample 3: SFM 27 from Xinhua Huaxing Machinery & Electronics Co. Ltd

Table 6: The concentration of elements in Sample 3

Element	Weight %	Atomic %
C K	4.44	24.38
O K	4.71	19.42
Mg K	0.55	1.49
Ag L	88.57	54.16
Pb M	1.73	0.55
Totals	100.00	

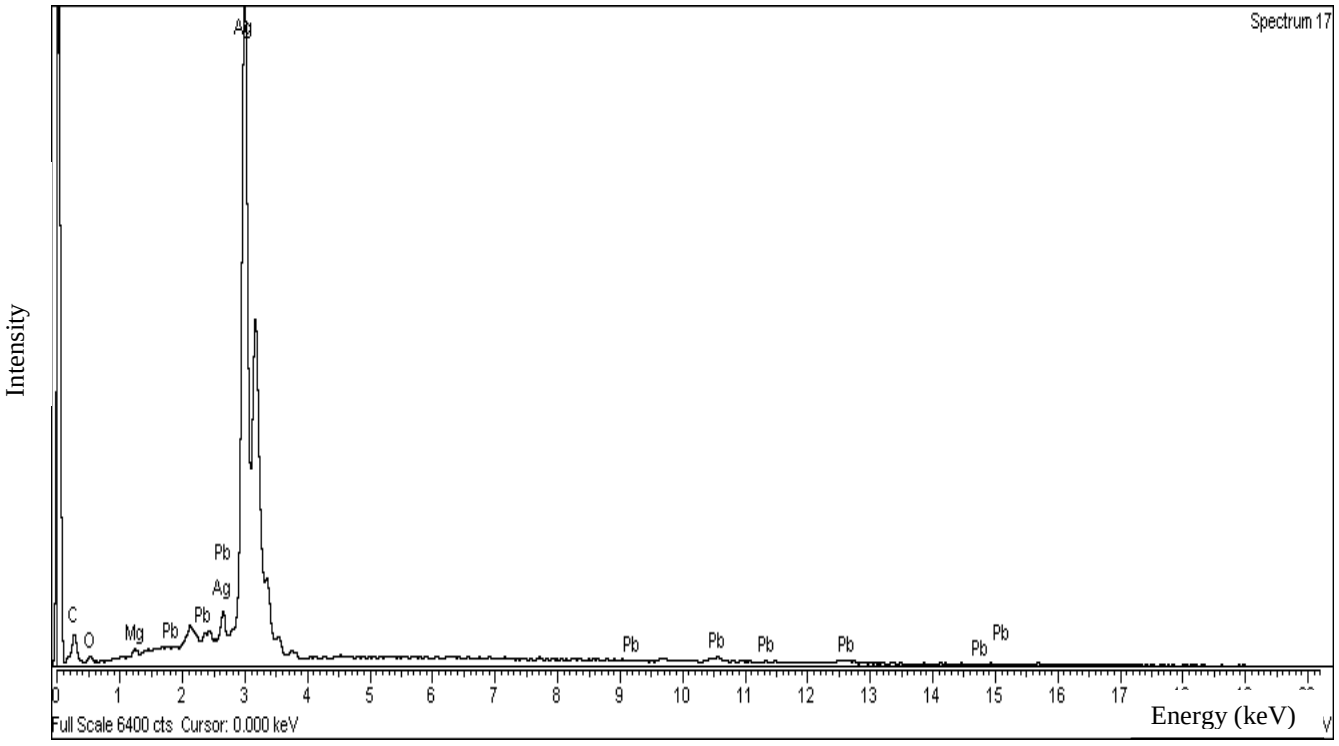


Figure 20 EDX result for sample 3

4.2 Discussion

The d_{33} values of the discs at different loads are calculated using the formula as per below:

$$Q = d_{33}F$$

where Q is the total charge released when a force F is applied. The value for charge, Q, is obtained from Q = *CV*. The value for the capacitance in the circuit is the appropriate value to obtain the readings in the range of electrometer (0.01 to 1 µF). It is observed that the suitable value of capacitance for all samples in experiment 1 is 0.01 µF. 1 µF capacitor cannot be used since it is not suitable for sample 1 because the voltage reading is so small and it will affect the accuracy of the result.

The voltage reading increase with the increasing of the load applied. It was shown in the graph in Figure 7. The voltage is directly proportional to the load applied with the piezoelectric coefficient, d_{33} and capacitance, C being the constant of proportionality and it is proved from the equation earlier:

$$Q = CV = d_{33}F$$
$$V = \frac{d_{33}}{C}F$$

The graph in Figure 8 shows the relationship between the load applied on the samples and the piezoelectric coefficient measured from the voltage reading. The piezoelectric coefficient, d_{33} remain the same with the increasing of load applied. It is because each piezoelectric has a specific piezoelectric coefficients.

$$Q = CV = d_{33}F$$
$$d_{33} = \frac{Q}{F}$$

The piezoelectric coefficients, d_{33} for all samples measured from experiment 1 are as per below:

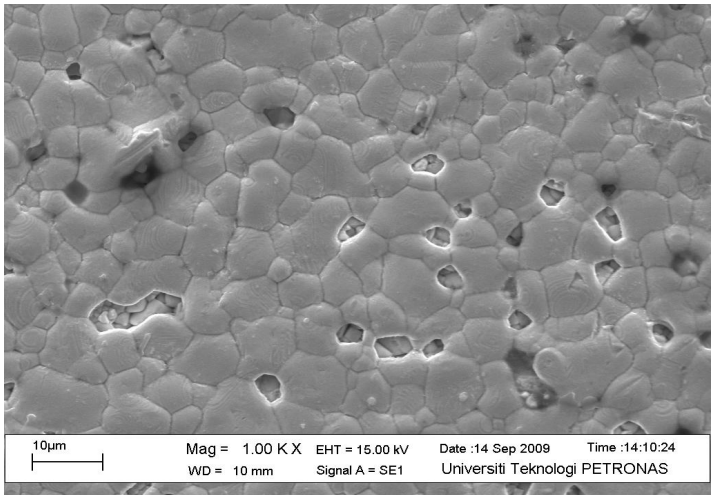
- Sample 1: 135 x 10^{-12} Coulomb/Newton
- Sample 2: 271 x 10^{-12} Coulomb/Newton
- Sample 3: 410 x 10^{-12} Coulomb/Newton

Sample 3 has the highest piezoelectric coefficients amongst all of the samples. Although Sample 1 and 2 are from the same manufacturer, the difference in the piezoelectric coefficients value maybe due to the manufacturing process and the composition of the Lead Zirconate Titanate [15].

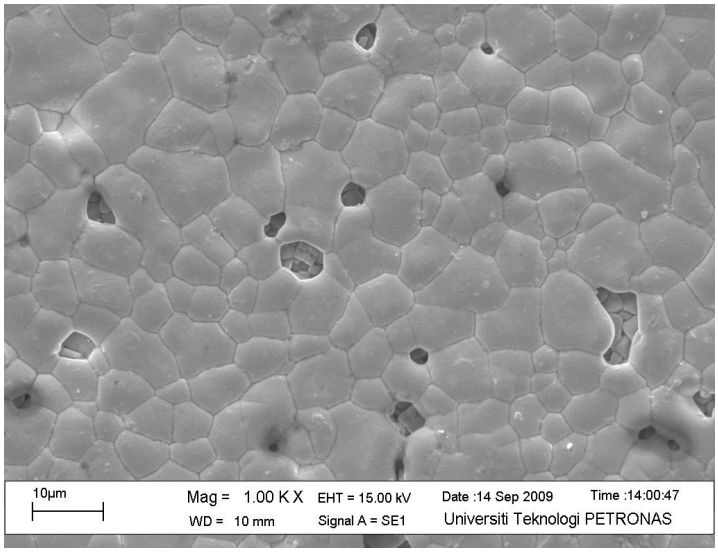
From the results of the Scanning Electron Microscope Experiment, we can see the difference between the microstructure of the samples. There was a slightly difference in the microstructure between Sample 1 and Sample 2 since both of the samples are from the same manufacturer. For Sample 3, the microstructure is different than Sample 1 and 2 since they are from different manufacturers.

The microstructure for Sample 1 and 2 have higher porosity than Sample 3. The d_{33} values of porous PZT ceramics are considerably lower than that of the conventional PZT ceramics since because of the existence of the non-piezoelectric air phase where it increases the interconnectivities between the pores and pores size.

a)



(b)



(c)

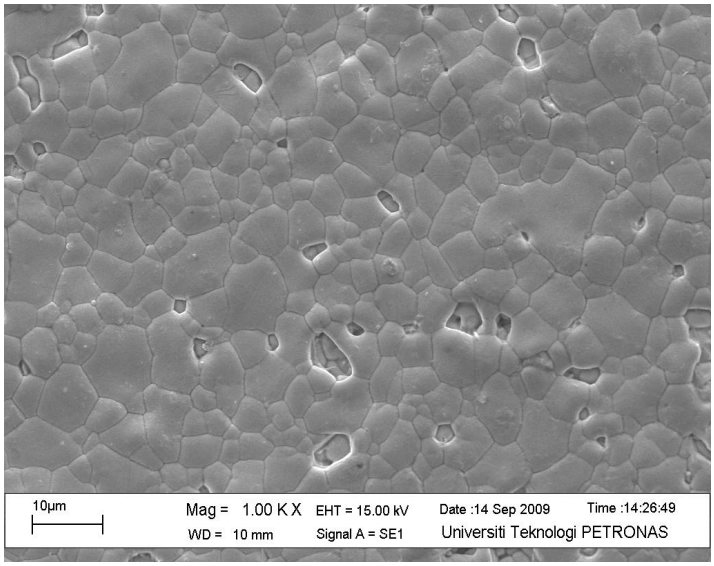


Figure 21 Difference between Sample 1, 2 and 3. (a) Sample 1, (b) Sample 2, (c) Sample 3

In the Energy Dispersive X-ray Spectroscopy (EDS), the only composition that can be considered is Lead, Pb. Since Sample 1 and Sample 2 are from the same manufacturer, the composition of the lead are the same in samples, 1.53 weight% and 0.47 atomic%. Sample 3 has higher lead composition which is 1.73 weight% and 0.55 atomic%.

The result obtained is not as expected because there is none of Zirconate, ZrO elements and Titanate, TiO elements shown up in the graph. The Zirconate, ZrO elements and Titanate, TiO elements should be in the graph because the K_{α} and K_{β} have higher energy to detect those elements. It is because during the experiment, the beam of electron is only focused on a small part on the surface. In order to get a better composition, the beam of electron should be focused on the entire surface.

The relations between piezoelectric coefficient,d_{33} and microstructure is the piezoelectric coefficient,d_{33} is higher if the sample has lower porosity and smaller pores and pores size. Sample 3 has high piezoelectric coefficient and at the same time has lower porosity. While for Sample 1 and 2, both of them have lower piezoelectric coefficient since they have higher porosity.

CHAPTER 5

CONCLUSION

5.0 CONCLUSION

For the conclusion, the methodology which is used in this project has succeed in supporting the objectives in the project which are to compare the characteristics of several commercial ceramic disks of lead Zirconate Titanate as well as to measure the piezoelectric coefficient and study its correlation with microstructure and come up with the best solution.

From the analysis, Sample 3 has high piezoelectric coefficient and at the same time has lower porosity. While for Sample 1 and 2, both of them have lower piezoelectric coefficient since they have higher porosity. Here, we can conclude that the relations between piezoelectric coefficient,d_{33} and microstructure is the piezoelectric coefficient,d_{33} is higher if the sample has lower porosity and smaller pores and pores size. For the composition, the sample with higher concentration of Lead has higher piezoelectric coefficient.

For the future projects, Sample 3 is the best solution as the actuator for Micromechanical Flying Insect since it has high piezoelectric coefficient that is preferable for an actuator for MFI.

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APPENDICES

Appendix 1: Gantt Chart for FYP I

No.	Detail/ Week	1	2	3	4	5	6	7	8	9	10	11	12	13	1
1	Selection of Project Topic														
	- Propose Topic														
	- Confirmation of Topic Selection														
2	Preliminary Research Work														
	- Data collection														
	- Identifying equipment and tools required														
	- Literature review on piezoelectric ceramics														
3	Submission of Preliminary Report														
4	Project Work														
	- Data reduction and presentation														
5	Submission of Progress Report														
	-Literature Review														
	-Project Description														
6	Project work continue														
	- Analyze data														
7	Submission of Interim Report Final Draft														
8	Oral Presentation														
9	Submission of Interim Report														

Milestone

Process

Appendix 2: Gantt Chart for FYP II

No.	Detail/ Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Project Work Continue														
	-Practical/Laboratory Work														
2	Submission of Progress Report 1														
3	Project Work Continue														
	-Practical/Laboratory Work														
4	Submission of Progress Report 2														
5	Project work continue														
	-Practical/Laboratory Work														
6	Submission of Dissertation Final Draft														
7	Oral Presentation														
8	Submission of Project Dissertation														

Suggested milestone

Process

Appendix 3: Calculation

The d_{33} values of the discs at different loads are calculated using the formula as per below:

$$Q = d_{33}F$$

where Q is the total charge released when a force F is applied. The value for charge, Q, is obtained from Q = *CV*. The value for the capacitance in the circuit is the appropriate value to obtain the readings in the range of electrometer (0.01 to 1 µF). After a couple of try and error, I found that the capacitance value of 0.01µF was suitable for the entire samples.

a) Sample 1

i. Load = 0.5 kg

Q = CV

= 0.01µF x 0.068 V

= 68 x 10^{-11} Coulombs

Q = d_{33}F

d_{33} = 68 x 10^{-11} Coulombs ÷ 5 N

= 136 x 10^{-12} Coul/N

ii. Load =1.0 kg

Q = CV

= 0.01µF x 0.135 V

= 135 x 10^{-11} Coulombs

Q = d_{33}F

$d_{33} = 135 \times 10^{-11}$ Coulombs ÷ 10 N

$= 135 \times 10^{-12}$ Coul/N

iii. Load = 1.5 kg

Q = CV

$= 0.01\mu F \times 0.203$ V

$= 203 \times 10^{-11}$ Coulombs

$Q = d_{33}F$

$d_{33} = 203 \times 10^{-11}$ Coulombs ÷ 15 N

$= 135 \times 10^{-12}$ Coul/N

iv. Load = 2.0 kg

Q = CV

$= 0.01\mu F \times 0.270$ V

$= 270 \times 10^{-11}$ Coulombs

$Q = d_{33}F$

$d_{33} = 270 \times 10^{-11}$ Coulombs ÷ 20 N

$= 135 \times 10^{-12}$ Coul/N

v. Load = 2.5 kg

Q = CV

$= 0.01\mu F \times 0.338$ V

$= 338 \times 10^{-11}$ Coulombs

$Q = d_{33}F$

d_{33} = 338 x 10^{-11} Coulombs ÷ 25 N

= 135 x 10^{-12} Coul/N

vi. Load = 3.0 kg

Q = CV

= 0.01µF x 0.405 V

= 405 x 10^{-11} Coulombs

Q = d_{33}F

d_{33} = 405 x 10^{-11} Coulombs ÷ 30 N

= 135 x 10^{-12} Coul/N

b) Sample 2

i. Load = 0.5 kg

Q = CV

= 0.01µF x 0.136 V

= 136 x 10^{-11} Coulombs

Q = d_{33}F

d_{33} = 136 x 10^{-11} Coulombs ÷ 5 N

= 272 x 10^{-12} Coul/N

ii. Load =1.0 kg

Q = CV

= 0.01µF x 0.271 V

= 271 x 10^{-11} Coulombs

$Q = d_{33}F$

$d_{33} = 271 \times 10^{-11}$ Coulombs ÷ 10 N

$= 271 \times 10^{-12}$ Coul/N

iii. Load = 1.5 kg

$Q = CV$

$= 0.01\mu F \times 0.407$ V

$= 407 \times 10^{-11}$ Coulombs

$Q = d_{33}F$

$d_{33} = 407 \times 10^{-11}$ Coulombs ÷ 15 N

$= 271 \times 10^{-12}$ Coul/N

iv. Load = 2.0 kg

$Q = CV$

$= 0.01\mu F \times 0.542$ V

$= 542 \times 10^{-11}$ Coulombs

$Q = d_{33}F$

$d_{33} = 542 \times 10^{-11}$ Coulombs ÷ 20 N

$= 271 \times 10^{-12}$ Coul/N

v. Load = 2.5 kg

$Q = CV$

$= 0.01\mu F \times 0.678$ V

$= 678 \times 10^{-11}$ Coulombs

$Q = d_{33}F$

$d_{33} = 678 \times 10^{-11}$ Coulombs ÷ 25 N

$= 271 \times 10^{-12}$ Coul/N

vi. Load = 3.0 kg

$Q = CV$

$= 0.01\mu F \times 0.813$ V

$= 813 \times 10^{-11}$ Coulombs

$Q = d_{33}F$

$d_{33} = 813 \times 10^{-11}$ Coulombs ÷ 30 N

$= 271 \times 10^{-12}$ Coul/N

c) Sample 3

i. Load = 0.5 kg

$Q = CV$

$= 0.01\mu F \times 0.205$ V

$= 205 \times 10^{-11}$ Coulombs

$Q = d_{33}F$

$d_{33} = 205 \times 10^{-11}$ Coulombs ÷ 5 N

$= 410 \times 10^{-12}$ Coul/N

ii. Load =1.0 kg

$Q = CV$

$= 0.01\mu F \times 0.410$ V

$= 410 \times 10^{-11}$ Coulombs

$Q = d_{33}F$

$d_{33} = 410 \times 10^{-11}$ Coulombs $\div$ 10 N

$= 410 \times 10^{-12}$ Coul/N

iii. Load = 1.5 kg

$Q = CV$

$= 0.01\mu F \times 0.615$ V

$= 615 \times 10^{-11}$ Coulombs

$Q = d_{33}F$

$d_{33} = 615 \times 10^{-11}$ Coulombs $\div$ 15 N

$= 410 \times 10^{-12}$ Coul/N

iv. Load = 2.0 kg

$Q = CV$

$= 0.01\mu F \times 0.820$ V

$= 820 \times 10^{-11}$ Coulombs

$Q = d_{33}F$

$d_{33} = 820 \times 10^{-11}$ Coulombs $\div$ 25 N

$= 410 \times 10^{-12}$ Coul/N

v. Load = 2.5 kg

$Q = CV$

$= 0.01\mu F \times 1.025$ V

$= 1025 \times 10^{-11}$ Coulombs

$Q = d_{33}F$

$d_{33} = 1025 \times 10^{-11}$ Coulombs $\div$ 30 N

$= 410 \times 10^{-12}$ Coul/N

vi. Load = 3.0 kg

$Q = CV$

$= 0.01\mu F \times 1.230$ V

$= 1230 \times 10^{-11}$ Coulombs

$Q = d_{33}F$

$d_{33} = 1230 \times 10^{-11}$ Coulombs $\div$ 30 N

$= 410 \times 10^{-12}$ Coul/N