KAUNAS UNIVERSITY OF TECHNOLOGY

MECHANICAL ENGINEERING AND DESIGN FACULTY

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Development of Piezoelectric Composite material for Microresonators

Master's Degree Final Project

Supervisor

Associate Professor. Dr. Janusas Giedrius

KAUNAS 2016

KAUNAS UNIVERSITY OF TECHNOLOGY

MECHANICAL ENGINEERING AND DESIGN FACULTY

I APPROVE

Head of Department

Prof. Dr.

Development of Piezoelectric Composite material for Microresonators

Final Project for Master degree

Mechanical Engineering (Code 621H30001)

Supervisor

Associate Professor Dr Janusas Giedrius

Project made by

Farusil Najeeb Mullaveettil

Reviewer

Professor Dr.Arvydas Palevicius

KAUNAS 2016

KAUNAS UNIVERSITY OF TECHNOLOGY

FACULTY OF MECHANICAL ENGINEERING AND DESIGN

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MASTER STUDIES FINAL PROJECT TASK ASSIGNMENT
Study programme
MECHANICAL ENGINERING - 621H30001

Approved by the Dean’s Order No.V25-11-7 of May 3rd , 2016 y

Assigned to the student **FARUSIL NAJEEB MULLAVEETTIL**

(Name, Surname

1. Title of the
Project

Development of Piezoelectric Composite material for Microresonators

2. Aim of the project

To develop the possibility for piezo electric composite material for microresonators and to create a finite element model for the proposed microresonator and investigate its dynamical and electrical characteristics.

3. Tasks of the project

- To investigate the piezo electric composite material developed for application in microresonators
- To propose a microresonator for quality control in injection molding.
- To create a finite element model for the proposed micro resonator.
- To investigate its dynamical and electrical characteristics of designed microresonator.

4. Specific Requirements

Created microresonator should fit into 15mm diameter hole.

5. This task assignment is an integral part of the final project

6. Project submission deadline: 2016 May 20th.

Task Assignment received FARUSIL NAJEEB MULLAVEETTIL ______________________

(*Name, Surname of the Student*) (*Signature, date*)

Supervisor Associate Professor Dr. Janusas Giedrius ______________________

(*Position, Name, Surname*) (*Signature, date*)

KAUNAS UNIVERSITY OF TECHNOLOGY

FACULTY OF MECHANICAL ENGINEERING AND DESIGN

(Faculty)

FARUSIL NAJEEB MULLAVEETTIL

(Student's name, surname)

Mechanical Engineering (621H30001)

(Title of study programme, code)

Development of Piezoelectric Composite material for Microresonators

DECLARATION OF ACADEMIC INTEGRITY

20 MAY 2016

Kaunas

I confirm that the final project of mine, **FARUSIL NAJEEB MULLAVEETTIL**, on the subject “**Development of Piezoelectric Composite material for Microresonators**” is written completely by myself; all the provided data and research results are correct and have been obtained honestly. None of the parts of this thesis have been plagiarized from any printed, Internet-based or otherwise recorded sources; all direct and indirect quotations from external resources are indicated in the list of references.

No monetary fund’s (unless required by law) have been paid to anyone for any contribution to this thesis.

I fully and completely understand that any discovery of any facts of dishonesty inevitably results in me incurring a penalty under procedure effective at Kaunas University of Technology.

(name and surname filled in by hand) *(signature)*

Mullaveettil, Farusil Najeeb, Development of Piezoelectric Composite material for Microresonators, Masters Final project/ Supervisor Assoc. Prof. Dr. Janusas Giedrius; Kaunas University of Technology, Mechanical Engineering and Design faculty, Mechanical department.

Keywords: PZT; Microresonators; Eigen Frequency

Kaunas, 2016,

SUMMARY

The presence of microresonators are more evident these days ranging from biometric sensors to gyroscopes. The application of sensors into the molding cavities is a new challenge that is yet to be studied more. On the other hand microresonators designed using piezo materials are showing some promising results. In this study the author aim to develop a microresonator with piezoelectric materials for quality control in injection molding. This aim is achieved by investigating PZT composite material by experimental testing. A finite element of the proposed microresonator is created and simulated using the comsol multiphysics software. From the experiment results, the young's modulus is calculated and used in further simulation work to find the excitation frequencies of the new PZT material. In this research them microresonators are integrated with piezo materials. Piezo materials can generate electrical signals which can be used to measure natural micro level changes, where devices like vibrometers or some other measurement methods are destructive and which are really hard to be used. Moreover these materials could assure high functionality and accuracy for micromechanical systems.

Mullaveettil, Farusil Najeeb, Pjezoelektrinės kompozicinės medžiagos, naudojamos mikrorezonatoriuose, kūrimas, magistro baigiamojo projekto / Supervisor doc. Dr Janušas Giedrius ; Kauno technologijos universitetas , Mechanikos inžinerijos ir dizaino fakultetas , Mechanikos katedra.

Raktiniai zodziai: PZT; mikrorezonatoriu; Eigen dažnis

Kaunas, 2016

SANTRAUKA

Mikrorezonatoriai yra plačiai naudojami, pradedant biometriniais jutikliais, giroskopais ir t.t. Tačiau jų taikymas liejimo formose yra naujas iššūkis. Tačiau sukurtus mikrorezonatorius galima efektyviai panaudoti technologinių procesų liejimo formose stebėsenai. Šio darbo tikslas – panaudojant naujas *pjezoelektrines medžiagas, sukurti mikrorezonatorių skirtą liejimo proceso stebėsenai. Siekiant įgyvendinti iškeltą tikslą buvo atlikti naujai sukurtos pjezoelektrinės medžiagos mechaninių ir elektrinių savybių tyrimai. Lyginant teorinius eksperimentinius rezultatus, nustatytas naujai sukur*tos *PZT medžiagos jungo modulis. Mechaniškai deformuojant šią medžiagą gaunamas tiesioginis pjezoelektrinis efektas, t.y. generuojama įtampa. Naudojant šią medžiagą, sukurtas mikrorezonatorius, leidžiantis identifikuoti procesus vykstančius liejimo formoje. Be to ši medžiaga gali užtikrinti aukštą mikromechaninių sistemų funkcionalumą ir tikslumą..*

Contents

1. Introduction

One of the fundamental components used in a MEMS application is the microresonators. The presence of microresonators are more evident these days ranging from biometric sensors to gyroscopes. The application of sensors into the molding cavities is a new challenge that is yet to be studied more. On the other hand microresonators designed using piezo materials are showing some promising results. In this study the authors aim to develop a sensor for quality control in injection molding.

Injection molding was created and patented in the year 1872 and this machine was relatively simple compared to present machines today. It works as a plunger by injecting a material through a heated cylinder into the mould. The materials used in injection molding are mostly Polymers or resins including all thermoplastics and some elastomers and thermosets. The rate of materials used for injection molding increases at a rate of 750 per year, so it will be very convenient for product designers to choose the material with the best possible properties from a very wide range of selection. Cost, strength of material and function required for the final part are the main criteria for selection of the materials. By understanding in the importance of molding process using the injection molding it is important to study and investigate the problems or drawbacks occurs during the molding process. Therefore the authors in this research work focuses more on investigating a new sensor which can assure quality assurance to the formed molds.

In this research the sensors are integrated with piezo materials. Piezo materials can generate electrical signals which can be used to measure natural micro level changes, where devices like vibrometers or some other measurement methods are destructive and which are really hard to be used. Moreover these materials could assure high functionality and accuracy for micromechanical systems. The three main piezo materials used, due to their high piezoelectric coefficients, good flexibility and with strong electromechanical coupling are Lead oxide ferroelectrics, known by lead zirconium titanate (Pb(Zr,ti)O3) or PZT, poly vinylidene fluoride(PVDF) films and zinc oxide(ZnO) films.

1.1 Background Information of the research

This report deals with the field of micro electro mechanical systems (MEMS) which encompasses the process based technologies used to fabricate micro integrated devices and systems that is a combination of different functionalities from different physical domains into one device.

1.2 Aim

The objective of this work is twofold. First objective is to develop the possibility for piezo electric composite material for microresonators The second objective is to create a Finite element model for the proposed microresonator and investigate its dynamical and electrical characteristics.

1.3 Research Problem

According to John Bozzelli, founder from injection molding Solutions (2010) “Processors face a multitude of challenges whenever they approach an injection molding machine.” Whenever it comes to injection molding one of the problems that went on for years is the difficulty of obtaining multicavity tools to produce identical parts. Due to this quality problems, in many cases it shows that single cavity production are actually less expensive. By the theory of mold making a perfect geometrical balance of the runners would be a possible solution for obtaining identical multi cavities. Even though the mold makers ensures a pretty good geometrical balance the problem still persist. Therefore a proper sensing system can make sure the non uniformity or geometrical unbalances in the mold cavities [8].

1.4 Research Questions

The author have performed the research based on these research questions.

- What are the properties that govern the features of the new composite Piezo material?
- What are the different fabrication techniques that can be used to fabricate these microresonators?
- What are the possible improvements and application of these microresonators?

1.5 Tasks

- To investigate the piezo electric composite material developed for application in microresonators
- To propose a microresonator for quality control in injection molding.
- To create a finite element model for the proposed micro resonator.
- To investigate its dynamical and electrical characteristics of designed microresonator.

1.6 Research Methods

The authors in this research work followed both qualitative and quantitative research methods. The stages in qualitative approach includes a study into the works done in the similar field and analyzing the works done in the field of MEMS. A deep study into the field of micro mechanics was done by focusing on topics like micro resonators with implant structure and also performing literature review into different Microresonator fabrication techniques ranging from Substrate making , planar technologies to 3D technologies.

The quantitative Research method stages include designing a Sensor (Comb Structure) and simulating it in Comsole Multiphysics Software and comparing its results with Experimental results. The sensors are designed based on the application of the work.

2. Literature Review

2.1 Piezoelectric effect

Piezoelectric effect was first seen in 1880 and was initially worked by Pierre and Jacques Curie. Integrating the knowledge of pyroelectricity to crystal structures behavior, the brothers showed the first piezoelectric effect with quartz crystals, tourmaline, topaz, cane sugar and Rochelle salt. In the first demonstration it was found that quartz and Rochelle salt exhibited the most piezoelectricity.

Piezoelectric effect is defined as the ability of certain unique materials to exhibit electric charge in response to applied mechanical stress. This effect is reversible, meaning that the materials exhibiting the direct piezoelectric effect, which is the generation of electricity when stress is applied, also can exhibit the reverse piezoelectric effect that is the generation of stress when an electric field is applied. When the piezoelectric material is exposed to mechanical stress, a shifting of the positive and negative charge centers takes place, which will then result in external electric field. When reversed, the electric field stretches or compresses the material [9].

This effect is utilized in application that deals with production and detection of sound, generation of high voltages, electronic frequency generation, micro balances and ultra fine focusing of optical assemblies. These days, lots of sensors are being developed using this effect [9].

Piezoelectric materials are many, both natural and manmade. Berlinite (structurally identical to quartz), cane sugar, quartz, Rochelle salt, topaz, tourmaline, and bone (due to the apatite crystals dry bone exhibits some piezoelectric properties, like in biological force sensor) are categorized as natural piezo materials. Barium titanate and lead zirconium titanate are some of the man made piezo materials that are used widely these days. The authors here used Lead zirconium titanate for the research [9].

Lead Zirconium titanate Pb(ZrxTi12x)O3 also known as PZT is a ferroelectric material which shows a marked piezoelectric effect. In the past year there has been lots of fabrication techniques used to make powdered PZT like coprecipitation, sol-gel process or by hydrothermal conditions. Generally PZT's are prepared by conventional mixed oxide method using PbO, TiO2, and ZrO2.

For the past few years researchers concentrated on topics relating to effective energy harvesting in modern technologies, MEMS or MOEMS systems. Energy harvesting can be done with various methods, for instance, using of electromagnetic devices, but mostly changes were noticed while using piezoelectric materials in small scale objects. Based on the piezoelectric, pyroelectric, and electrostrictive properties of ferroelectric thin film, Dennis L. Polla and Joon Han Kim manufactured a micromechanical device or microactuator using ceramic materials such as PZT. A typical micro device will have a device substrate and a deflectable component. For observing the deflection, the deflectable component is mounted on the device substrate and is integrated with sensor/actuator. These sensor/actuator consist of two electrodes and a piezoelectric film in between them, the thin film is made of PZT. The sensor/actuator which is disposed on a substrate, is formed by a material which is resistive to attack by hydrofluoric acid vapor. This invention relates to a method for the fabrication of micromechanical devices or microactuators [14].

The presence of piezoelectric can be seen in making micro scale motors. Author Takefumi Kanda mentioned the fabrication of a micro ultrasonic motor by introducing a micro-machined bulk piezoelectric transducer. In this paper, a diameter of 0.8 mm and a height of 2.2 mm cylindrical shaped bulk piezoelectric transducer was developed to be used a stator transducer for traveling wave type ultrasonic motor [2].

2.2 Injection molding

Injection molding is considered as one of the major processes for producing plastics. It is comparatively fast and large numbers of identical parts from high precision engineering components to other consumer goods can be manufactured. Almost all thermoplastics can be processed by injection moulding. The most commonly used materials are acrylonitrile-Butadiene-Styrene, Nylon, polycarbonate, polypropylene and polystyrene [10].

2.2.1 Injection Molding Process

During the injection molding process, the preheated material to its melting temperature is injected right before the molds press against each other. The material is still heated with the heater to maintain its temperature during the period of clamping. After clamping, the heater is shutdown and the mold and material is cooled down. In injection molding, the plastic is fed from a hopper into a heated barrel by gravity. As they are slowly pushed further by a plunger (screw-type), the plastic will be forced to a heated chamber (barrel), where it is melted. As the plunger advances, the molten plastic is forced through a nozzle that is placed against the mold sprue bushing, therefore allowing it to enter the mold cavity by a runner and a gate system. The mold will remain to a set temperature, so that the plastic can be solidified almost as soon as the mold is filled [10].

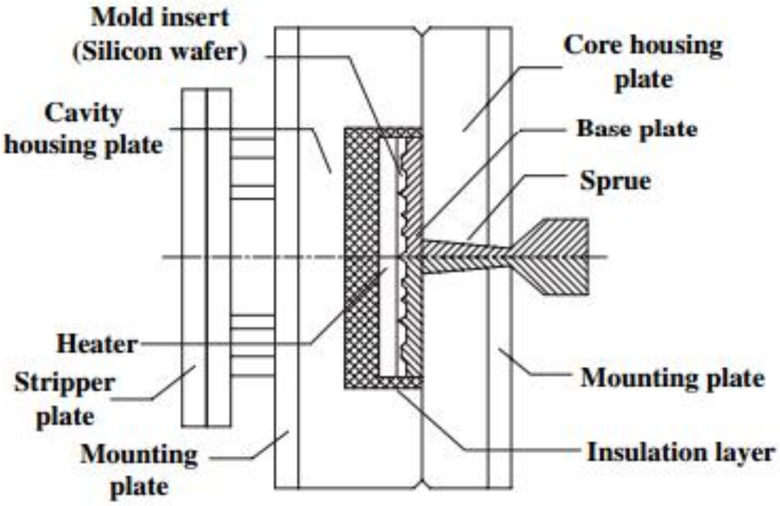


Figure 1 Injection Molding

As much as it is fast and can produce most of the plastics, injection molding also possess many drawbacks like Short shot, flash, sink, splay, warpage, burn marks, contamination, voids, bubbles , gate blush, jetting, weld lines and so on. Most of these problems arises due to multiple reasons, for instance the molten plastic material may not be reaching the mold cavity sections or it may be flowing into unwanted sections of the mold cavity. Shrinkage of plastics, differential cooling rate can also be another reason for these defects [10].

However lots of works have been carried out to minimize these defects. To optimize the injection molding process, a combination of artificial neural network and genetic algorithm method was proposed [3]. In this method, the authors developed a BP neural network model which can be used to map the complex non-linear relationship of the process conditions and quality indexes for the injection molded parts. Also on the other hand an Artificial Intelligence System in plastic injection molding operation was developed for obtaining the magnitudes in process parameters. It possesses a user interactive system and could be seen in use in shop floors. For the system, two techniques are performed, Rule-Based and Case-Based reasoning. Case-Based reasoning is to get the setting of processing parameters, while the Rule-Based system, suggests rectified solution to handle possible corresponding variations in molding. From this paper optimization time and human expert dependency is reduced [4].

Some works were also done to improve the quality of the injected plastic parts. For predicting the quality of the parts a neural network model is formed based on key process variables and material grade variations. The approach uses a back propagation 4-2-3 net (BPN) data which were taken from simulation works done through a computer aided engineering software. The outcome of this work was to reduce the time required for planning and optimizing operating parameters [5].

2.3 Fabrication Techniques

Fabrication of Micro sensors or resonators is very complex and extremely exciting endeavor due to the customized nature of process technologies and processing diversity. Fabrication of MEMS uses mostly the same techniques used in Integrated circuits domain such as oxidation, diffusion, ion implantation, LPVCD, sputtering and so on. By combining these capabilities with highly specialized micromachining process, the authors are mentioning some of the fabrication techniques used in this chapter [11].

MEMS fabrication techniques is primarily categorized based on the substrates used and the technology platforms. For products aiming at high volume markets such as consumer electronics, manufacturers try to refine production methods to deliver highly reliable products. For robust products like flow sensors, micromirrors, Pressure sensors and accelerometers micromachining based on KOH etching is preferred. Although this technology platform has the advantage that it does not need large equipment investments, the technology is difficult to integrate with CMOS, it is time consuming, and the fragile wafers are difficult to handle. Moreover, only relatively large structures can be created. The shifting onto Polysilicon based micromachining was a major paradigm. For materials with short processing time and smaller structures this method is preferred. For obtaining excellent mechanical properties, single crystalline silicon based machining is preferred [11].

There are many techniques for fabricating MEMS. Technologies which are based on photolithography, sputtering, evaporating, chemical vapour deposition are termed under planar technologies.

Photolithography deals with photographic technique for transferring copies of a pattern (master) onto the surface of a substrate material (silicon wafer is usually preferred) [11].

Etching is mostly done with silicon. Silicon etching is an important step in bulk and surface micromachining. Etching is further divided into wet and dry etching. Wet etching refers to the removal of material by immersion of a material (typically a silicon wafer) in a liquid bath of a chemical etchant. These are either isotropic or anisotropic. Dry etching depends on vapour phase or plasma-based etching methods , using suitable reactive gases or vapours, usually at high temperatures [11].

2.3.1 Screen Printing

To achieve thick films at temperatures compatible with silicon processing, alternative technique of composite film technology has been developed. The technique involves forming a slurry of PZT powder and a solution of PZT. This slurry is then deposited onto the substrate. It is then dried at 710'C. Density and functional properties of the film can be improved with intermediate Sol Infiltration and pyrolysis. PZT films can be printed by screen printing using a suitable slurry [13].

For the past years many works have been done in making PZT films. A MEMS based PZT/PZT thick film bimorph vibration energy harvester with an integrated silicon proof mass is presented by R Xu and A lei et al .Basically a piezoelectric energy harvesting devices uses a cantilever beam of a non piezoelectric material as support between the piezoelectric materials; it provides mechanical support but also may reduces the power output. In this work by R Xu and A lei they replaced the supporting material with another layer of the piezoelectric material. With the absence of an inactive mechanical support all stresses induced by vibrations will be harvested by the active piezoelectric elements [14].

Studying the properties of PZT is of great importance in order to adapt PZT for various applications Many works have been done in order to study its properties. Some of the works include making a thick film of composites of lead–zirconate–titanate (PZT) particulate and polyvinylidene-trifluoroethylene (PVDF-TrFE) copolymer ,are produced by screen-printing on indium–tin-oxide (ITO)-coated glass substrates. This microstructure was studied using scanning electron microscopy and X-ray diffraction [15].

2.3.2 Physical Vapour Deposition

Physical Vapour Deposition (PVD) is a deposition method used to deposit thin films at a time onto various surfaces usually onto semiconductors. This type of deposition uses coating source which is physical in nature (i.e. solid or liquid) rather than chemical as in chemical vapour deposition. Evaporation and sputtering are commonly used PVD process, for instance for the deposition of aluminum or gold conductors [11]

2.3.3 Chemical Vapour Deposition

For producing high-purity and high-performance solid materials Chemical Vapour Deposition (CVD) is used. The substrate is exposed to one or more volatile precursors in a typical CVD process, which react and/or decompose on the substrate surface to produce the desired deposit. Frequently, volatile by-products are removed by gas flow through the reaction chamber. Plasma Enhanced Chemical Vapour Deposition (PECVD) is a single wafer process, while Low Pressure Vapour Deposition (LPCVD) deposit very uniform layers on a batch of wafers simultaneously. Typical LPCVD process temperatures are above 600o C, limiting its use in general to the very beginning of the processing. In MEMS it is often used to deposit polysilicon (as mechanical layer in surface micromachining) and low stress silicon nitride (as a membrane layer or to be used as a masking material in bulk micromachining) [11].

There has been lots of works being done in making PZT composites, for instance Jiunnjye Tsaur et al prepared a PZT film deposited by hybrid process using solution gel method and laser ablation[6]. Skinner, et al. prepared PZT/polymer composites using the coral replamine process. The composites were found to have, for certain applications, greatly improved electromechanical and physical properties over those of conventional piezoelectric materials [7]. On another paper it is said to have developed a wet procedure to prepare stoichiometric and homogeneous PZT powder. The Starting reagents are tetra-*n*-butyl zirconate, titanate, and lead acetate. The synthesis is based upon the coprecipitation of metal hydroxides Pb(OH)2, ZrO(OH)2, TiO(OH)2 and lead oxalate PbC2O4 in the pH range 9–10 [12].

2.4 Sensors in Molding process

The adaption of sensors in molding process is common phenomenon we see these days. Sensors ranging from pressure to temperature are implemented in molding process. But why we need to do this? Why we need to sense the cavity? It is simply because that what happens in mold stays in part. Cavity pressure and temperature sensors are very common in injection molding. These parameters are important to study in order to identify sinks, short, flash, dimensional variation, weight, chemical resistance, warp or in molded stress. Implementation of sensors in injection molding would help realize these parameters and can in turn increase the quality of the molded parts[8].

Many mold making industries make use of pressure sensors to assist them in gaining valuable information as the mold is developed. As the part is filled, the pressure sensor measures the amount of plastic between the machine nozzle to the cavity area. Generally sensors are placed in the nozzle, hot and cold runner systems and at the cavity area of a mold. However, mostly the focus is on the cavity area [8].

Location of sensors are really important in order to get the most efficient results. In order to get the good amount of information for molders these sensors should be located at precise locations. When it comes to placing the sensor there are many specific exceptions, sensors for process monitoring are located near the one-third of the cavity end to fill. Sensors used for controlling the process by transferring the press are placed to the first one-third of the cavity. For very small parts, sensors are located in the runner system also at the end-of-cavity, sensors are key for sensing short-shots and automated part containment. Digital sensors makes it easy to put sensors in every cavity and can have one network wire from the mold to the press. This allows a maximum insurance against short-shots by simply keeping the sensors at the end of cavity without any further process control interface to the machine [8].

3. Experimental details

Piezoelectric material is another structural part of a microresonator. Piezoceramics act as an actuator in the structure by employing its ability to generate electrical signal from deformations, which are incident to the material. Additionally, it can be used to identify natural frequency changes, where vibrometers and other indirect measurement methods are destructive and hardly can be used in-situ analysis. To fully control microresonator's parameters, piezoelectric layer is essential. The control of parameters assure higher accuracy and functionality of micromechanical systems.

In this paper, a finite element model of new type piezoelectric material is proposed and experimental and theoretical results of its behavior are compared. The experimental works are divided into practical and software phases. The practical works were done at The experiments were held in the Institute of Mechatronics of Kaunas University of Technology

3.1 Analysis of Dynamical Characteristics

3.1.1 PZT

The piezo composite material is initially formed and is then subjected to different studies. Piezoelectric substances in electromechanical systems show theoretically no deflection, when affected by compression. When an extended amplitude interval is applied, it would give ruggedness and a high natural frequency.

Using PZT powder and 20% solution of polyvinyl butyral in benzyl alcohol a new PZT type piezoelectric material was created. A lamellar periodical microstructure is formed on the piezoelectric thin top layer of 4 μm, by the thermal embossing technique. Periodical microstructure was dip-coated by silver (Ag) nanoparticles formed in deionized water from a solution of 0.05 $AgNO_3$ [17].

The thickness of piezoelectric layer is 54 μm

3.1.2 Copper Film

Copper thin film was used as the bottom layer of multilayer structure. UNS C10100 copper alloy was used for this purpose. The thickness of copper thin film is 40 μm. The Vickers hardness value for the copper ranges from 75.0 to 90.0 with tensile strength ranging from 221 to 445 Mpa. The elongation at break is 55%, modulus of elasticity is 115Gpa. The copper possesses a poisons ratio of 0.31 with 20% machinability and 44.0Gpa shear modulus

3.1.3 Aluminum Layer

The top layer of multilayer structure was constructed of aluminum foil covered with epoxy. Epoxy was used to glue the aluminum layer and PZT layer together. The density of aluminum is $2700kg/m^3$. The modulus of elasticity is 66Gpa with poisons ratio of 0.33. The relative permeability is 1 for the aluminum. The modulus of elasticity of aluminum is lowered by adding epoxies to it [17].

3.1.4 Multi Layer Structure

A cantilever-type multilayer element was created. It is a combination of three different layers: aluminum layer, piezoelectric material and copper thin film (Figure 5.). The dimensions of proposed structure is 18x15x0.139 mm. Layers are with thickness 45 µm, 54 µm, and 40 µm respectively [17].

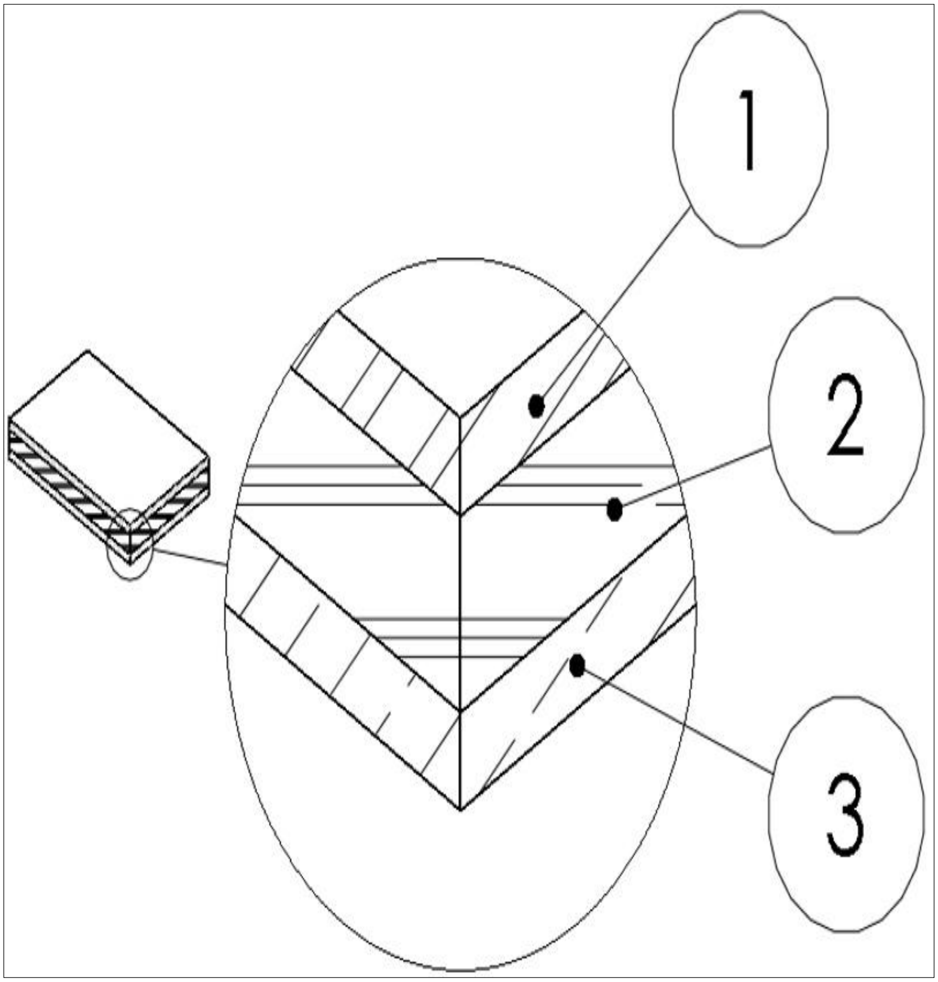


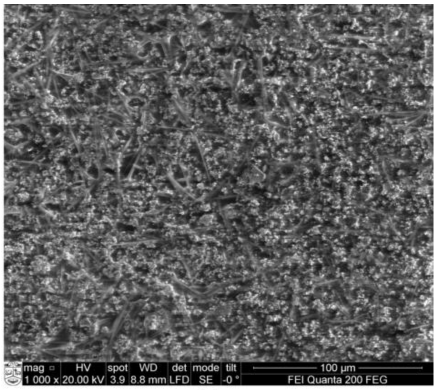
Figure 5. Geometrical representation of multilayer structure: aluminum foil – 1, piezoelectric material – 2, copper foil – 3

3.1.5 Morphology analysis of piezoelectric element surface

To study the surface morphology of the piezoelectric structure, chemical, electrical, dynamical results, three different concentrations of PZT (40%, 60% and 80%) were formed.

By using scanning electron microscopy the surface morphology of piezoelectric element were studied. Different grain sizes are studied here. The surface of the first element is with PZT 40 %. The surface is smooth with small 5-15 µm diameter pileups were observed on top. When the concentration of PZT was increased to 60%, individual grains were formed. The concentration was again raised to 80% and the piezoelectric structure became granular with smaller grain size below 4 µm(Fig 2.) [18].

The origin of the irregularity, nucleation and growth in the solution can be due to PZT particle loaded in a Polyvinyl Butyral, thus forming the smaller grains group. PZT island structures (Fig. 2) were formed where the granular grains surrounds larger grains. It can be observed that a high density is achieved in PZT 40% and 60% except few pinholes. However, these micro cracks with the length of micron distribute uniformly in the surface (Fig 2) [18].



(c)

Figure 2 . SEM view of piezoelectric elements with PZT 80%.

SEM surface views in scale of 5-10 micrometer are presented in Fig 3. The biggest difference in microstructure of the layer was observed in the element with 80% concentration of PZT (Fig 3). It posses three dimensional microstructure with empty cavities of about 6-8 µm diameter. [18]

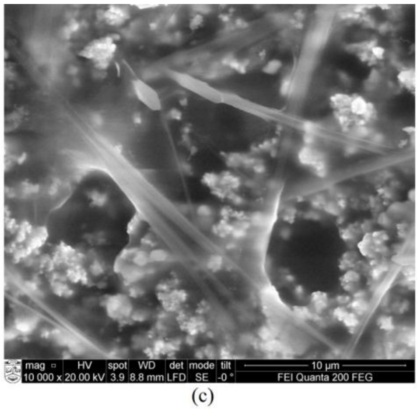


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Figure 3. SEM views of sensing elements with 80%.

Using PRISM system piezoelectric elements’ response to electrical excitation was investigated. When excited periodically (acceleration 0.007g, frequency 50 Hz) the cantilever type piezoelectric elements, generated up to 50 μV potential (for open circuit) when PZT 40% , and up to 40 μV potential when PZT concentration is 60 %. At 80% concentration of PZT (Fig 4), it generated electrical potential up to 80 μV. The results were pre-processed by low-pass filter of 500Hz. [18].

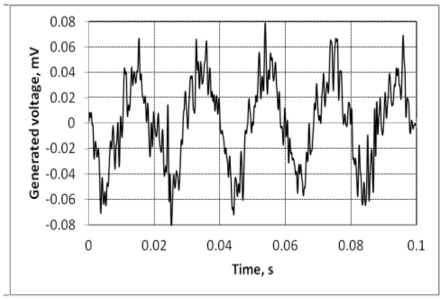

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Figure 4. Electrical potential generated by piezoelectric elements with PZT 80%.

The piezoelectric element with 80% concentration of PZT shows significant results in power generation as a thin layer at low frequencies. Other elements had no signs of ability to convert electrical potential into mechanical energy. This was analyzed using electronic speckle pattern interferometer PRISM. The piezo electric element with 80% PZT was exited by a sinusoidal function with amplitude of 5mV at frequency of 13 Hz. It vibrates as a cantilever at the first resonant frequency.

Advantage of such element is the ability to apply this novel piezoelectric composite material on any uniform or non uniform vibrating surface [18].

3.1.6 Experiment Setup

The experiments were held at the Mechatronics institute and at the department of Mechanical Engineering and Design of Kaunas University of Technology. The analysis of the piezoelectric material is performed with COMSOL Multiphysics 3.5a simulation software, by COMSOL, Inc. in Burlington, USA. COMSOL. This software is a general-purpose software platform, which is governed by advanced numerical methods and simulating physics oriented problems.

For the study of dynamic characteristics the multilayer structure a mathematical pendulum is used(Figure 3).The specimen is clamped vertically and an impulse is given by the pendulum onto the specimen. A single impulse is given in a measurement instance. By the aid of laser triangular displacement sensor LK-G3000 from the company Keyence from Illinois in United States of America, the vibration response measurements are performed. The specifications of laser triangular displacement measuring system component sensor head LK-G82 are with reference distance 80mm-diffuse reflection and 75.2mm with specular reflection. The measuring range is ±15mm (diffuse reflection) and ±14mm (specular reflection), the light source is red semiconductor laser with 655nm. The spot diameter is approximately with 70µm. Repeatability and operating temperature range is 0.2µm and 0 to 50'C. The operating ambient humidity ranges from 35 to 85% and the vibration is from 10 to 55Hz with double amplitude of 1.5mm and the weight is approximately 380g. [17]

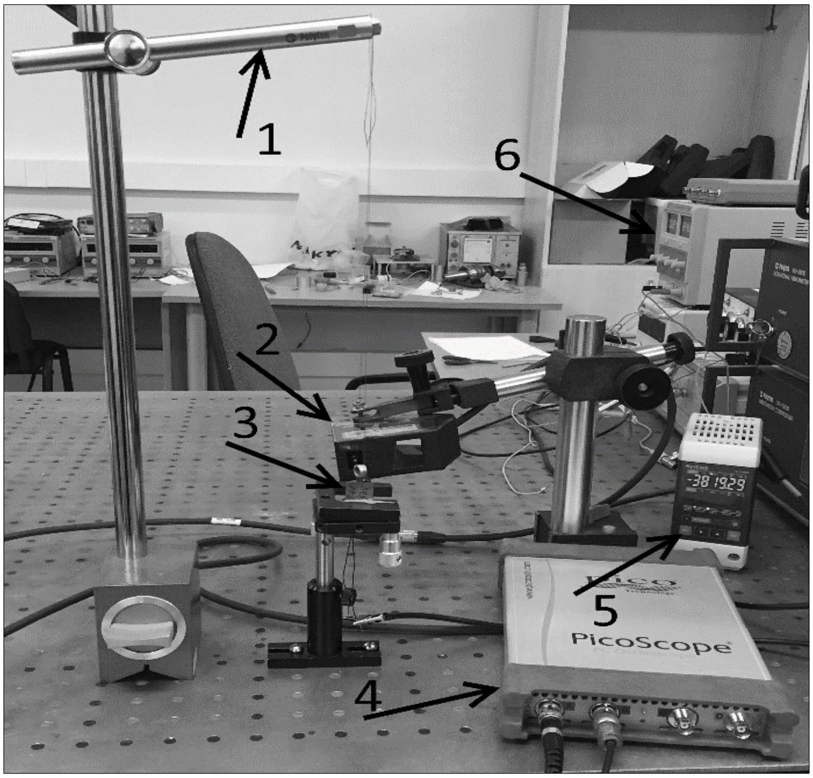

Figure 6. Experimental setup for measuring dynamic characteristics consists of: (1) mathematical pendulum, (2) LK-G82, (3) Specimen material (4) Oscilloscope (Pico scope), (5) LK-G3001PV (6) power

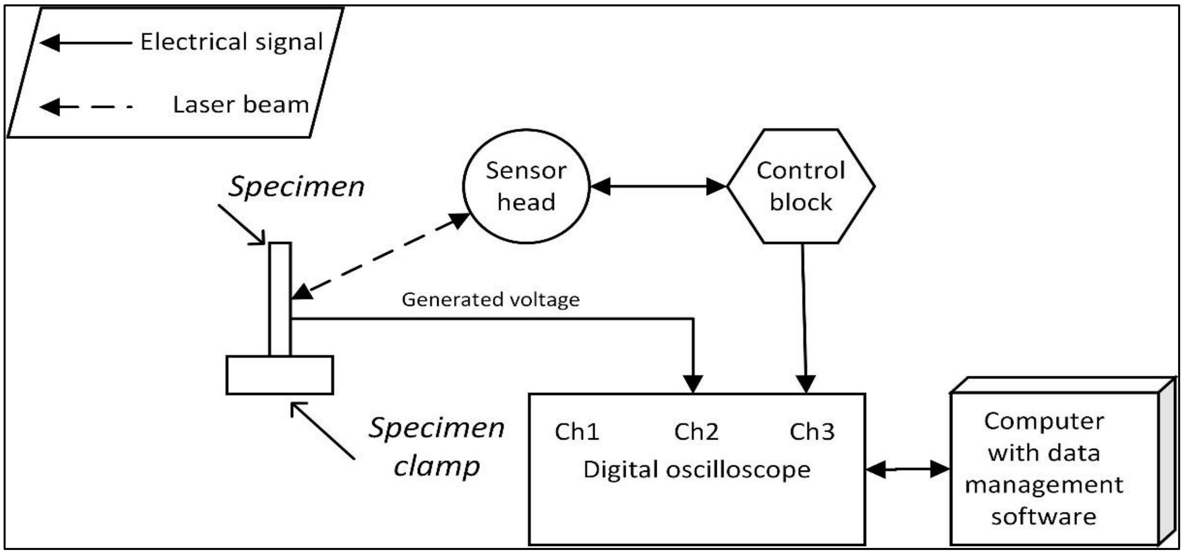

Figure 7. Experiment setup scheme for measuring harmonic excitations of piezoelectric elements

The system also uses a control block LK-G3001PV. The control block is of PNP type with minimum display unit of 0.01µm. The refresh rate is 10times per second. The analog output voltage and analog output current is ±10V for 2 outputs with output impedence of 100Ώ and 4 to 20mA for 2 outputs with maximum load resistance of 350Ώ. The operating temperature range is 0 to 50'C and operating ambient humidity is 35 to 85% RH. A 4-channel USB oscilloscope PICO 3424, is used to collect the experimental data and forward it to the computer. The obtained signal from control block and generated voltage is analyzed using Pico-Scope 6 software. The setup scheme for measuring dynamic characteristics of proposed multilayer structure is illustrated in Figure 7. [17]

3.1.7 Natural Frequency Analysis

The natural frequency calculation of the proposed multilayer element is done using the experimental vibrational response curve registered with laser triangular displacement measurement system. The natural frequency was used for comparison of theoretical and experimental models. For calculating the natural frequency the following formulae was used.

$$\omega_d = \omega_n\sqrt{1-\xi^2} \tag{1}$$

Where, ω_d – damped natural frequency, ω_n – natural frequency, ξ – damping coefficient.

$$\xi = \frac{1}{\sqrt{1+\left(\frac{2\pi}{\delta}\right)^2}} \tag{2}$$

Where, ξ – damping ratio, δ – logarithmic decrement.

$$\delta = \frac{1}{n} ln \frac{x(t)}{x(t+nT)} \tag{3}$$

Where, δ – logarithmic decrement, n – number of subsequent periods, $x(t)$ – the amplitude at time t, $x(t+nT)$ – the amplitude at time $t+nT$, T – period of oscillations.

3.1.8 Observations

The response of multilayer element when simulated is shown in the following figure

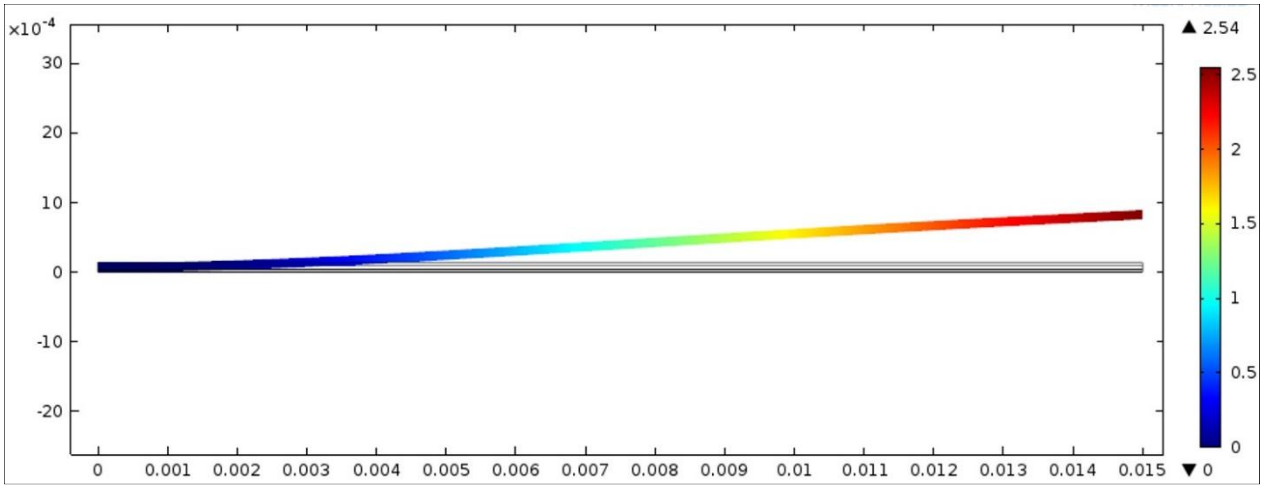

Figure 8. First form of vibrations of piezoelectric element model in COMSOL Multiphysics environment

Analyzing the graph Fig. 6, the calculated damped natural frequency was 186 Hz.

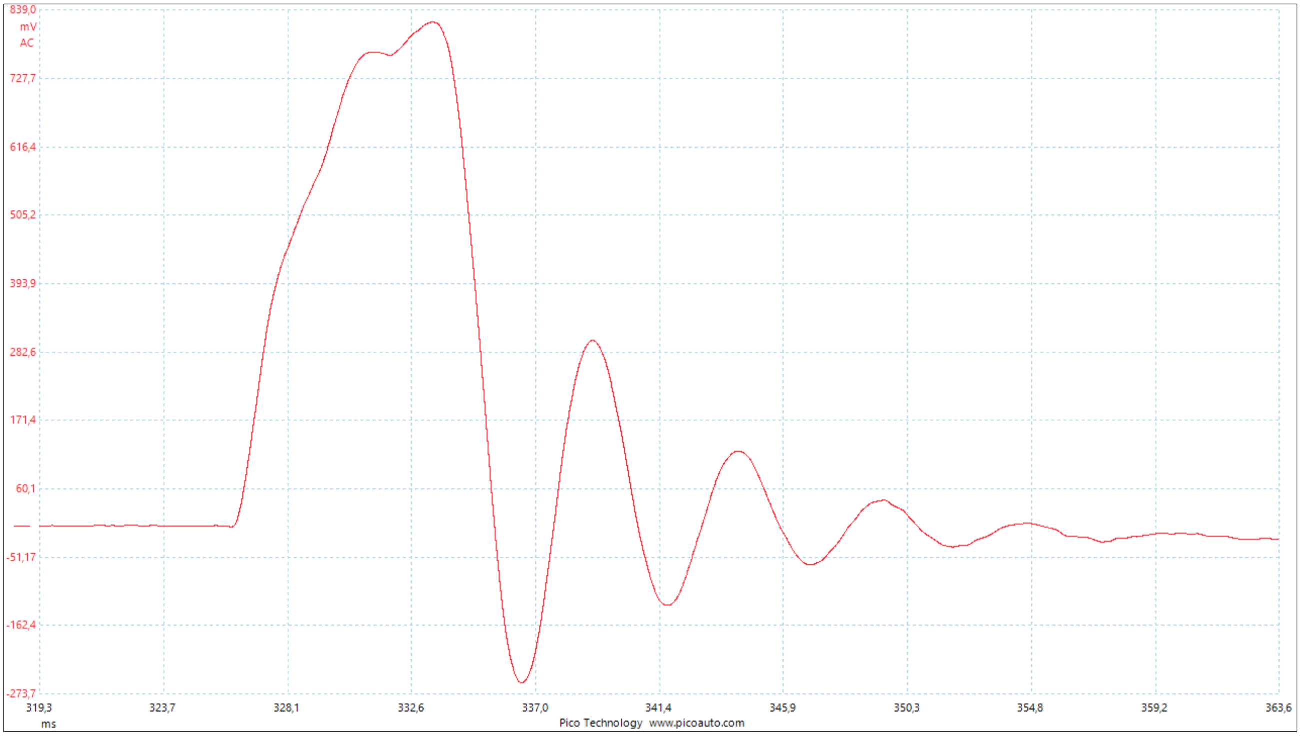

Figure 9. Vibrational response of multilayer element

Amplitudes in the measuring interval which were used in calculations, were 150 µm and 6 µm after one period. Using formulae 2, 3, and 4 the natural frequency of 187.056 Hz was calculated. The first vibration form in COMSOL Multiphysics model is achieved with 187.728 Hz. This result is only 0.4% higher than the experimental result. Stating that the frequencies (natural) of both models, theoretical and experimental were equal. Therfore it could be observed that determination of modulus of elasticity of created piezoelectric material as 1 MPa is shown to be correct.

Because the natural frequencies of both models were equal, the Young's modulus of piezoelectric material could be determined. It was taken from COMSOL Multiphysics model and the values of modulus of elasticity was 1 MPa.

3.2 Comsole multiphysics software analysis

Theoretical analysis of proposed piezoelectric element was done with COMSOL Multiphysics simulation software, created by COMSOL, Inc. in Burlington, USA. This software is a general-purpose software platform, which is governed by advanced numerical methods and simulating physics oriented problems. A model of piezoelectric element was created in the software's environment.

3.2.1 Polarization

In ferroceramics polarization is a factor to be studied. Ferroceramics are poly crystalline in nature and it does not possess piezo electric properties. This is explained by the orientation of the polar axes of the individual crystallites in it. Besides, the crystallites are divided into domains with various directions of the vectors of spontaneous polarization. Under the influence of an external constant electric field, a reorientation of the spontaneous polarization along the field direction occurs. As a result of this, the ferroceramics acquires piezoelectric properties. The efficiency of the polarization depends on the values of the external electric field, the duration of the impact and on the polarization temperature. For the sensor being developed here the polarization is noted to be in the Y direction. The authors came to this conclusion by observing that when polarized in Y direction the results were maximum compared to polarization in X and Y direction.

3.2.2 Eigen frequency analysis

By implementing the result from the practical experiments, the eigen frequencies of the sensor is observed. The steps of the analysis is given below:

Geometry

A block of 9.74 mm in length and height of 40 micrometers is drawn in 2D plane and revolved to 3D plane to form a circular plate. On the 2D plane the piezo comb structures are drawn with 2mm and 2.25 mm width with a thickness of 54 micrometers and revolved to obtain the following structures.

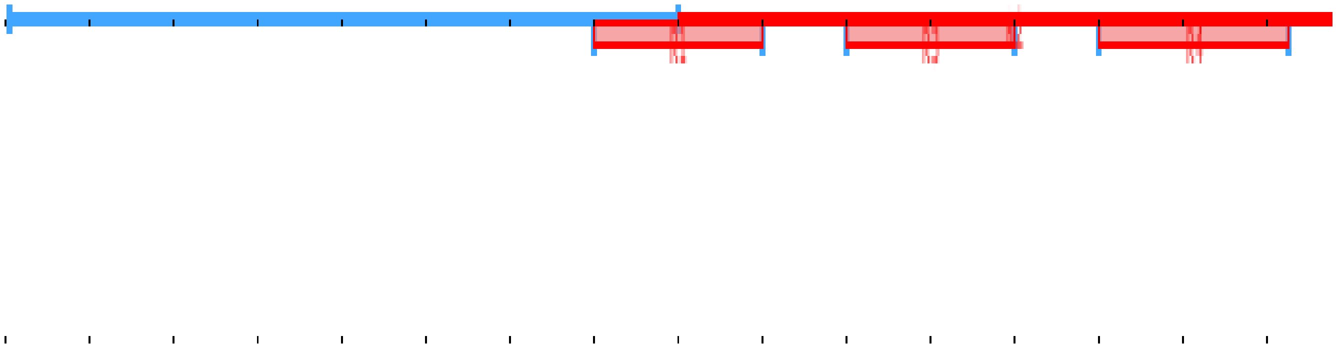

Figure 10. Plate in 2D. Comsol Drawing

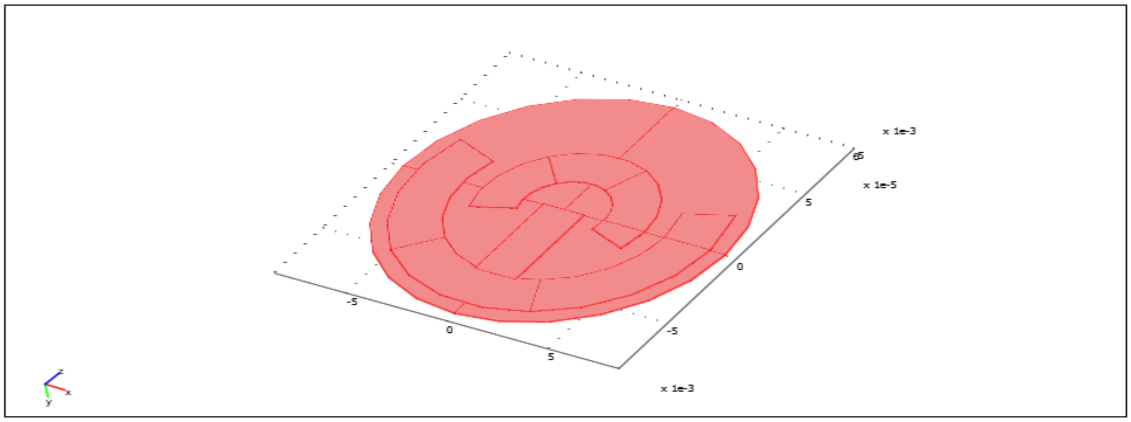

Figure 11. Plate drawing in 3D. Comsol Drawing

Mesh Parameters

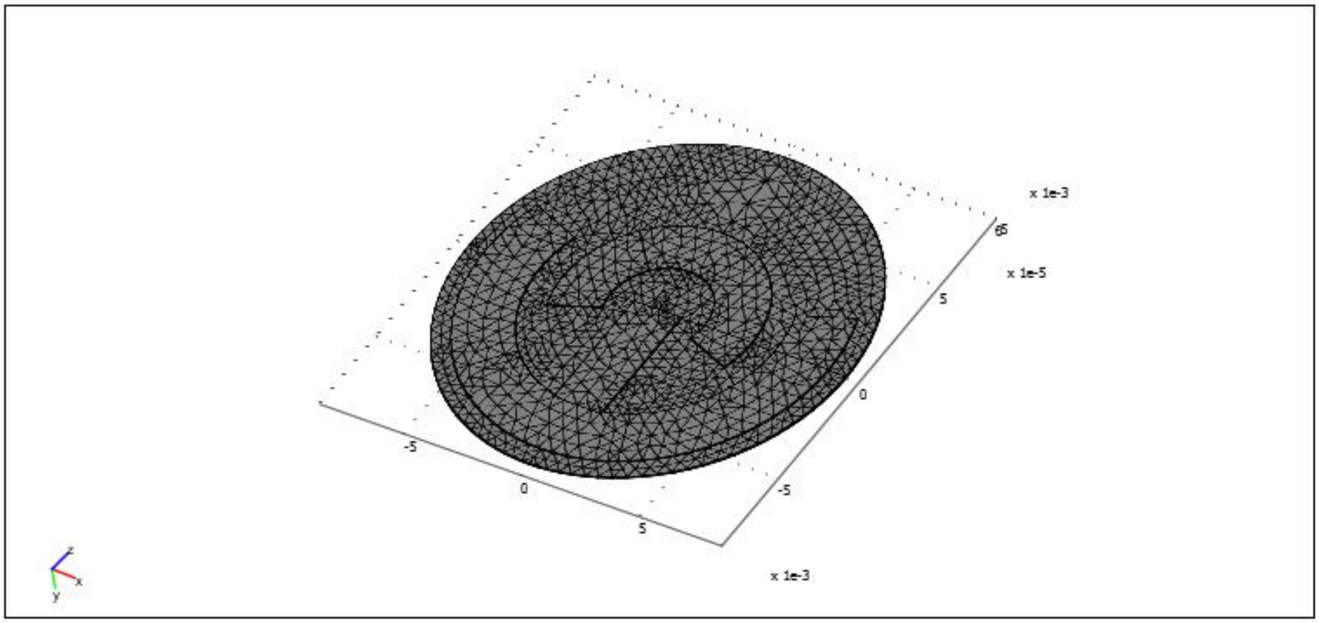

Figure 12 Mesh

Table 1 Mesh Parameters

Mesh parameters	Value
Number of degrees of freedom	50172
Number of mesh points	2696
Number of Elements	9062
Tetrahedral	9062
Prism	0
Hexahedral	0
Number of boundary elements	5538
Triangular	5538
Quadrilateral	0
Number of edge elements	772
Number of vertex elements	71
Minimum element quality	0.036
Element volume ratio	0.007

Boundary and Sub domain Settings

Table 2 Boundary Parameters

Boundary	1	2	3
Material	Copper	Lead Zirconium Titanate	Leaad Zirconium titanate
Constraint Condition	Fixed	Free	Free
Young's modulus (E)	110e9[Pa]	2.0e6	2.0e6
Density (rho)	8700[kg/m^3]	7500[kg/m^3]	7500[kg/m^3]
Thermal expansion coeff. (alpha)	17e-6[1/K]	1.2e-5	1.2e-5
Poisson's ratio (nu)	0.35	0.33	0.33

Post processing and results

Table 3 Eigen Frequency parameters

Parameter	**Value**
Desired number of eigen values	12
Search for eigenvalues around	0
Eigenvalue tolerance	0.0
Maximum number of eigenvalue iterations	300
Dimension of Krylov space	0

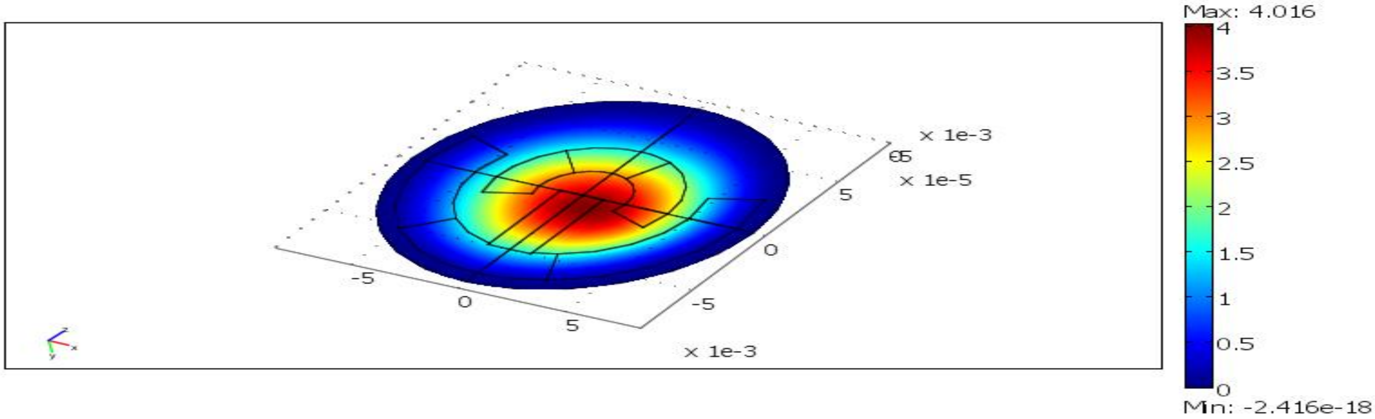

Figure 13. Frequency 1105.4928Hz

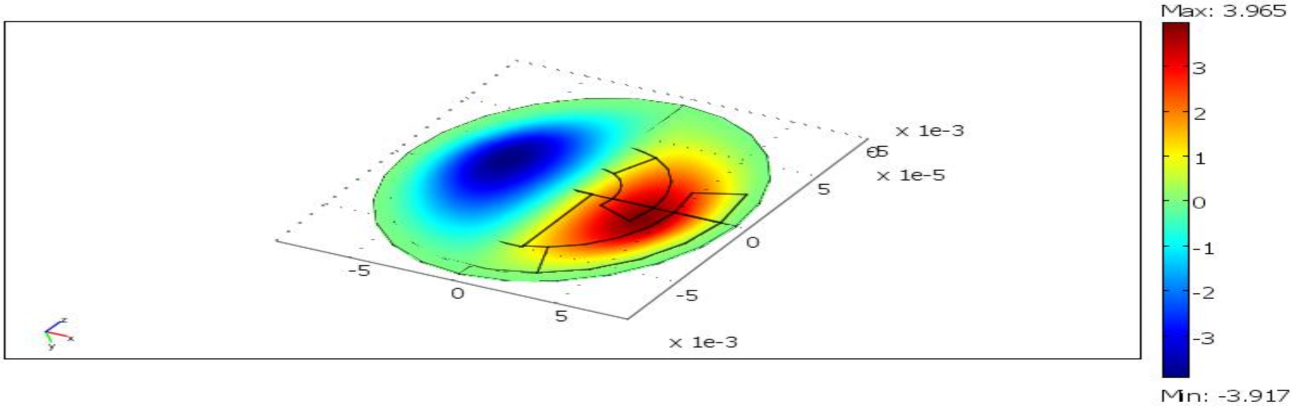

Figure 14. Frequency 2750.0449Hz

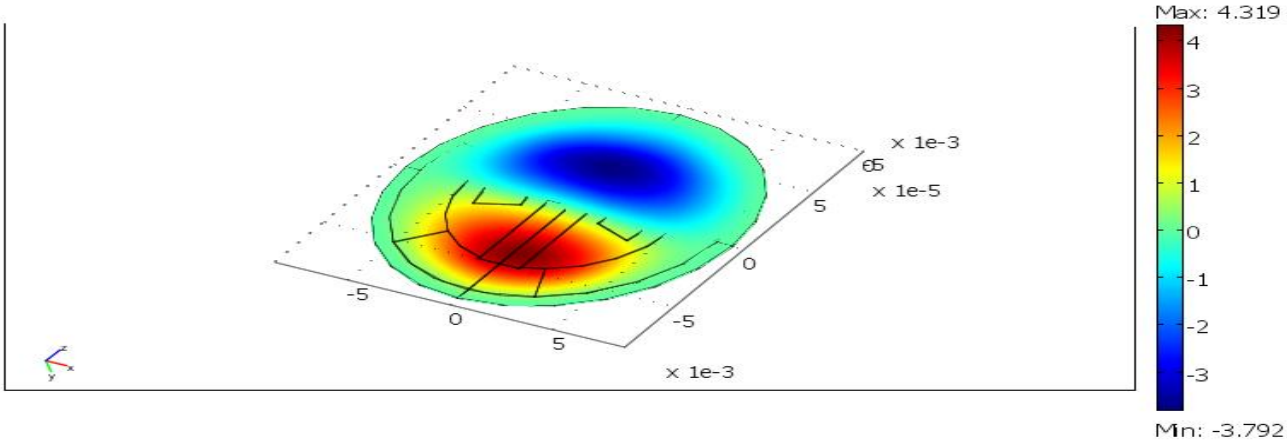

Figure 15. Frequency 2756.4896Hz

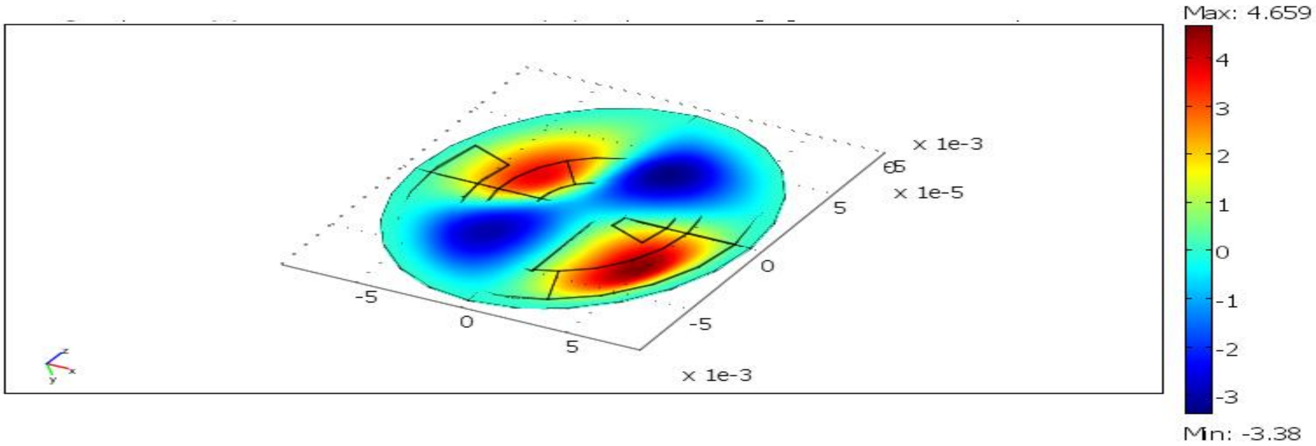

Figure 16. Frequency 4849.7608Hz

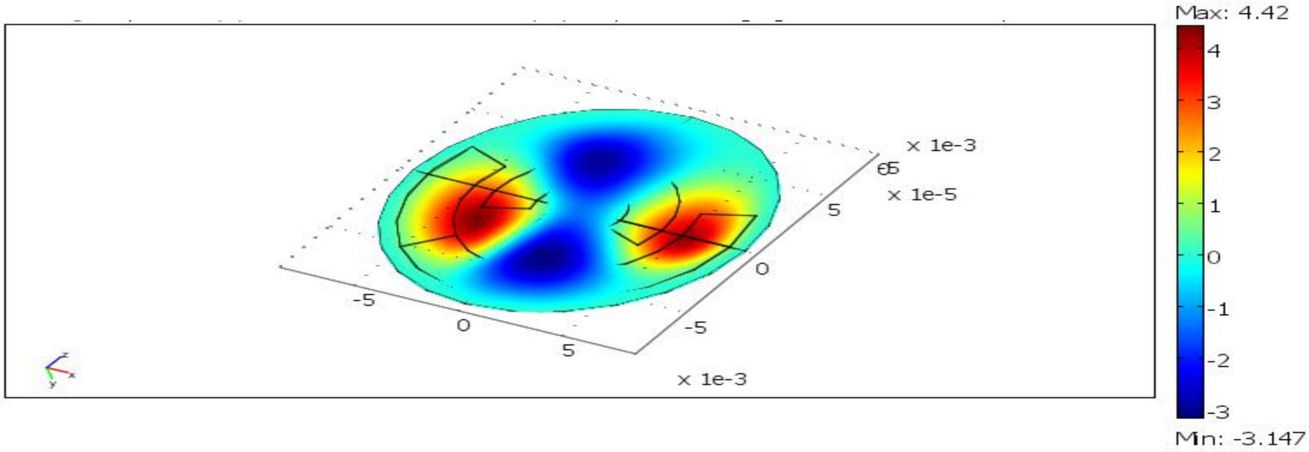

Figure 17. Frequency 5156.8353

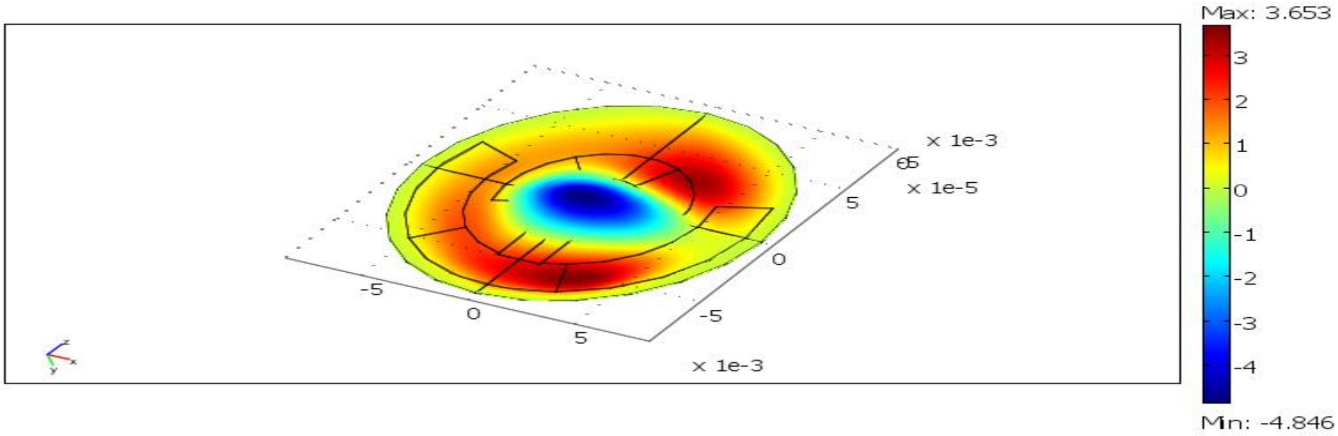

Figure 18. Frequency 6331.2160

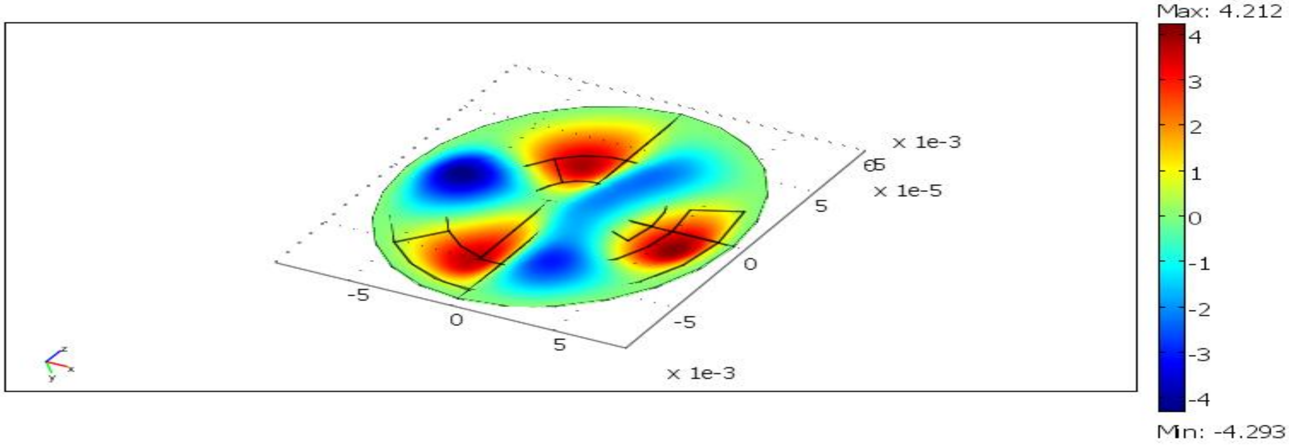

Figure 19. Frequency 7422.4828Hz

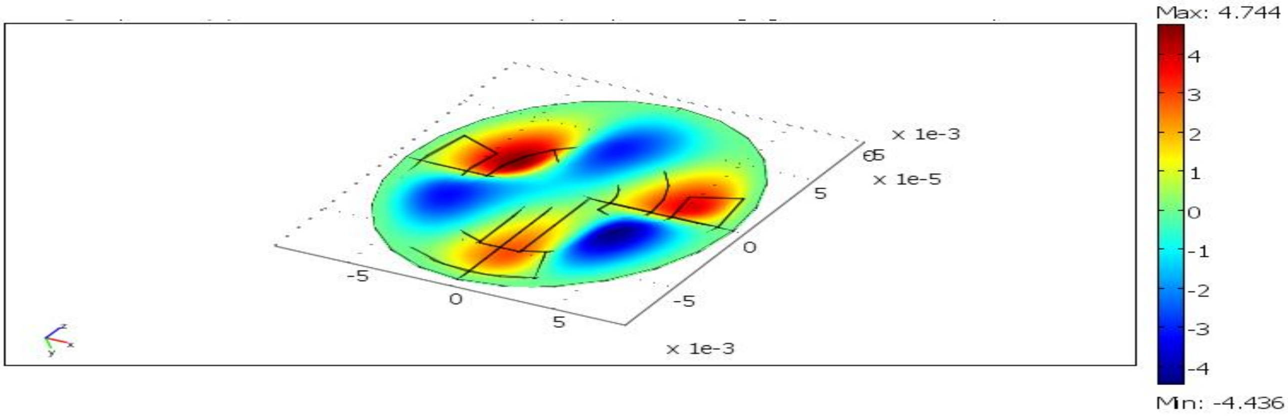

Figure 20. Frequency 7947.1283Hz

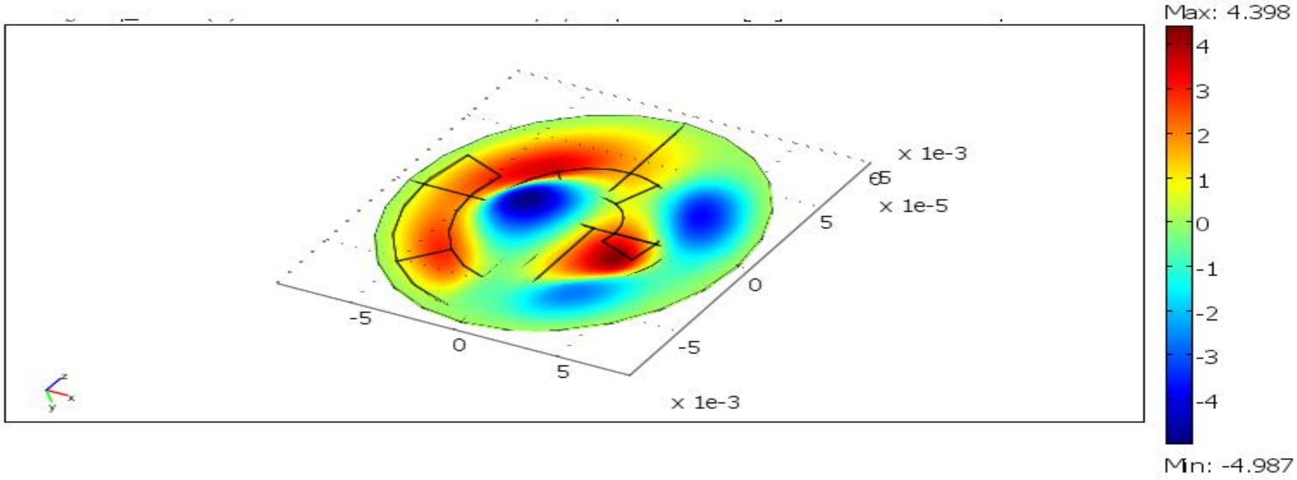

Figure 21. Frequency 9395.8320Hz

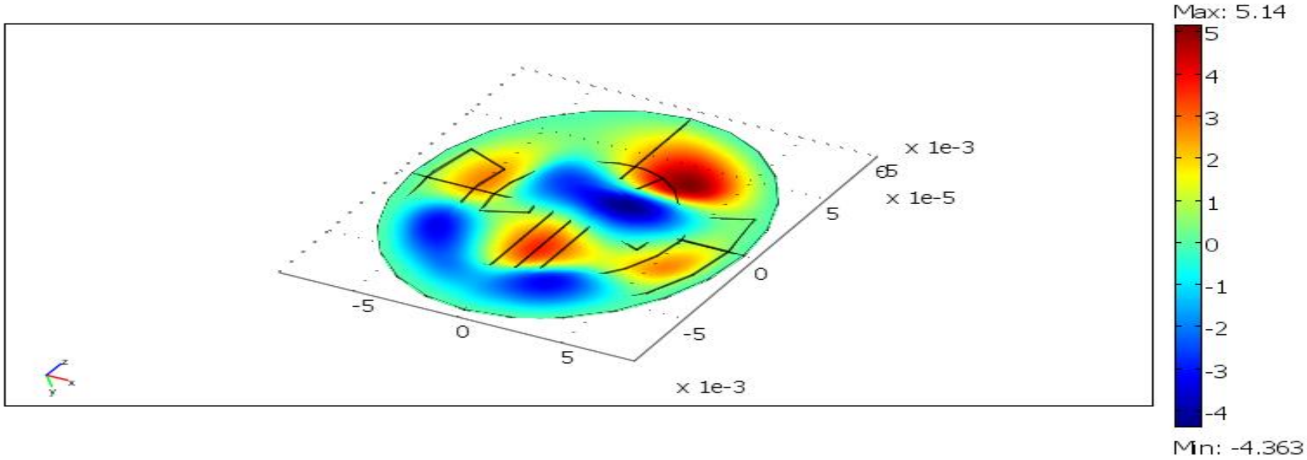

Figure 22. Frequency 10879.4343Hz

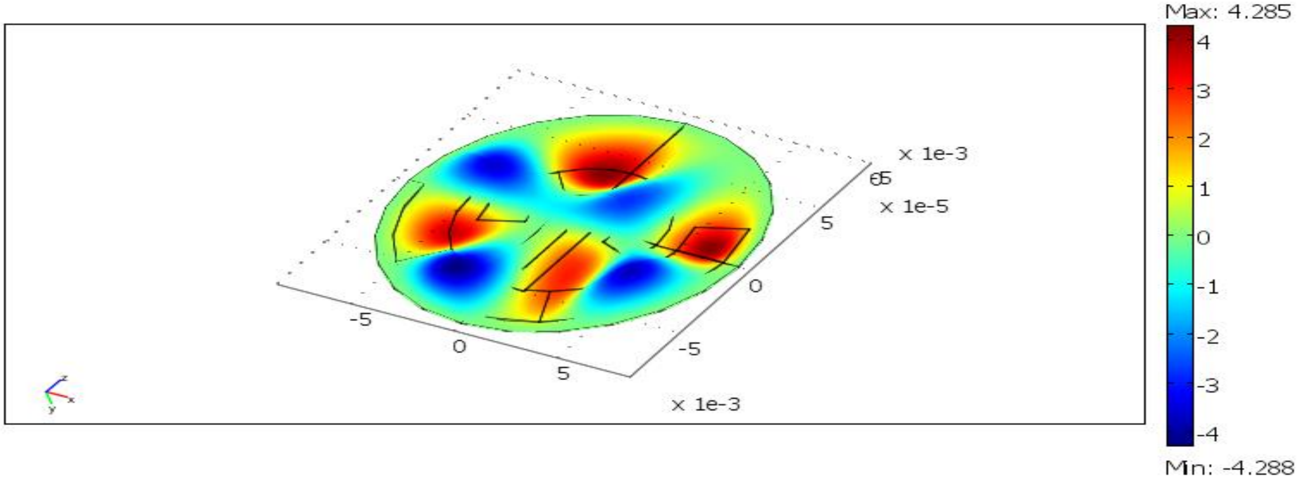

Figure 23. Frequency 11421.8126Hz

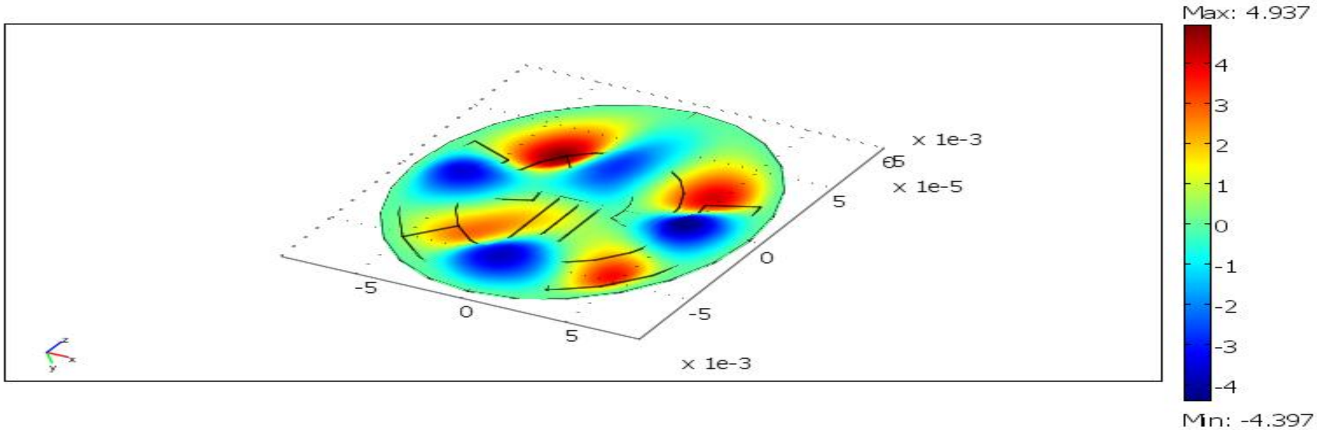

Figure 24. Frequency 11731.7650Hz

From the above eigen frequency analysis, it can be noted that the sensor operates as a simple membrane. This is because the plate thickness is small (40µm). In this study the entire plate is excited. But for more in depth analysis of the system, it is necessary to analyze just the PZT. It is expected to have a frequency shift when sensor is excited by active PZT layer.

3.2.3 Frequency Response Analysis

From the previous results we have obtained the eigen frequencies for the entire plate. This results are quite broader and it is mandatory to analyse by exciting just the sensing section (Piezo). Therefore in this section the author have excited just the sensing section and analyze the results. For this study, the results of the previous analysis that is the eigen frequencies are utilized. By utilizing these results it is possible to study the excitation frequency and the amplitude corresponding to these frequencies. The input voltage is 10V.

Application Mode Properties

Table 4 Application Mode

Property	**Value**
Default element type	Lagrange - Quadratic
Analysis type	Frequency response
Specify eigenvalues using	Eigenfrequency
Electrical formulation	Symmetric, Electrostatic
Large deformation	Off
Structural frame	Frame (xyz)
Electrical frame	Defined by structural frame
Deform frame	Frame (defined_by_structural_frame)
Weak constraints	Off
Constraint type	Ideal

Boundary and Sub domain Settings

Table 5 Boundary Conditions

Boundary	1	2	3
Material	Copper	Lead Zirconium Titanate	Leaad Zirconium titanate
Constraint Condition	Fixed	Free	Free
Electric type	Zero charge/Symmetry	Electric Potential	Ground
Electric Potential	0	10	0

Post processing and results

The excitation frequency is studied from the frequency 1000Hz to 15000Hz at an interval of 10Hz. The excitation frequencies are noted along with its corresponding amplitudes. The selected frequencies are plotted below.

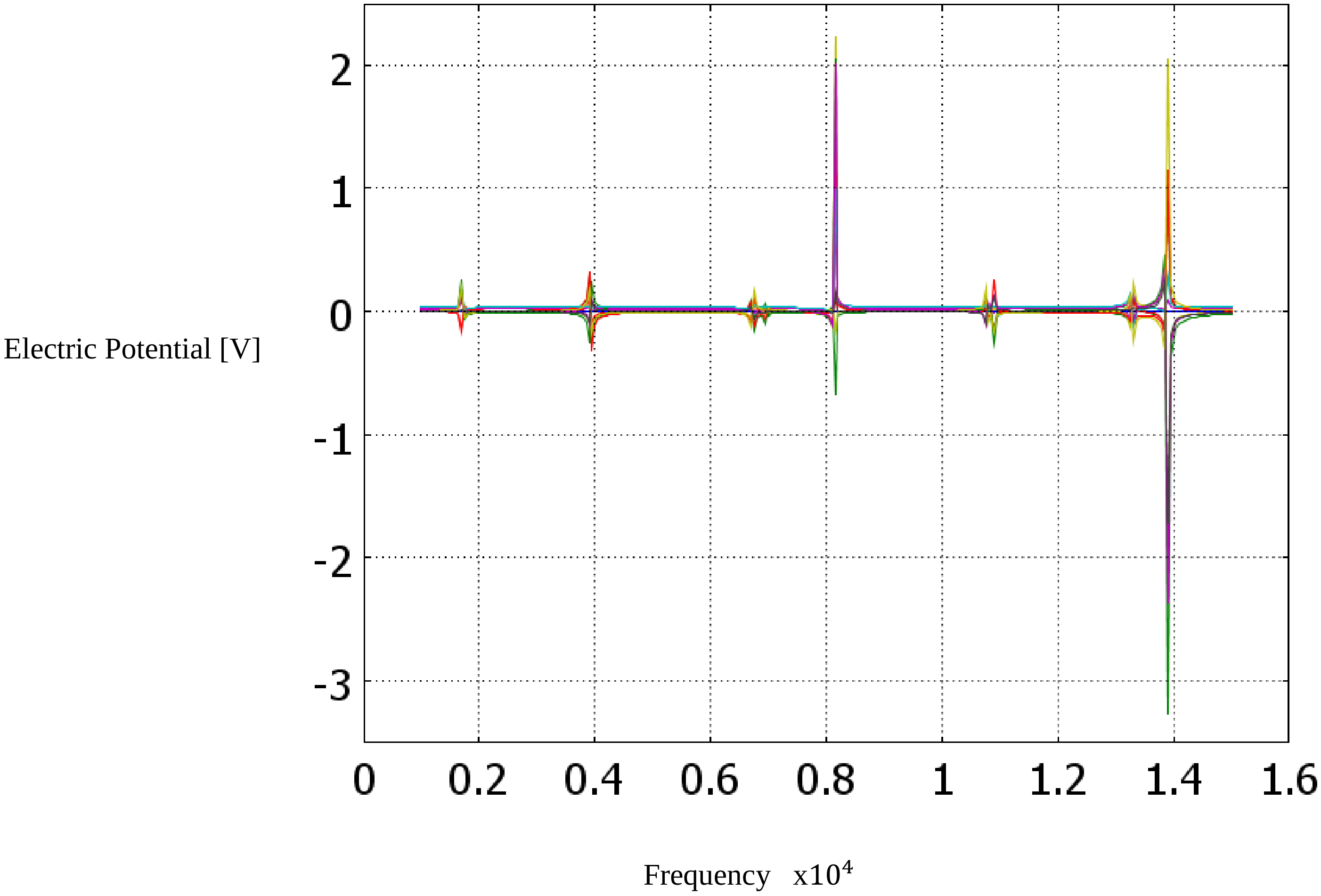

Figure 22. Graph Frequency vs Electric potential

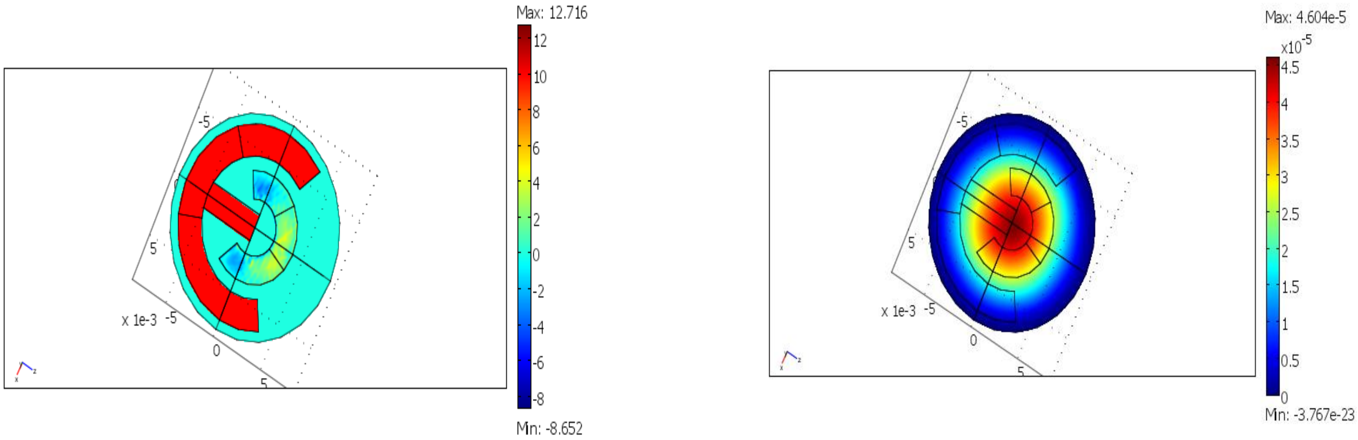

Figure 23. Frequency 1710Hz

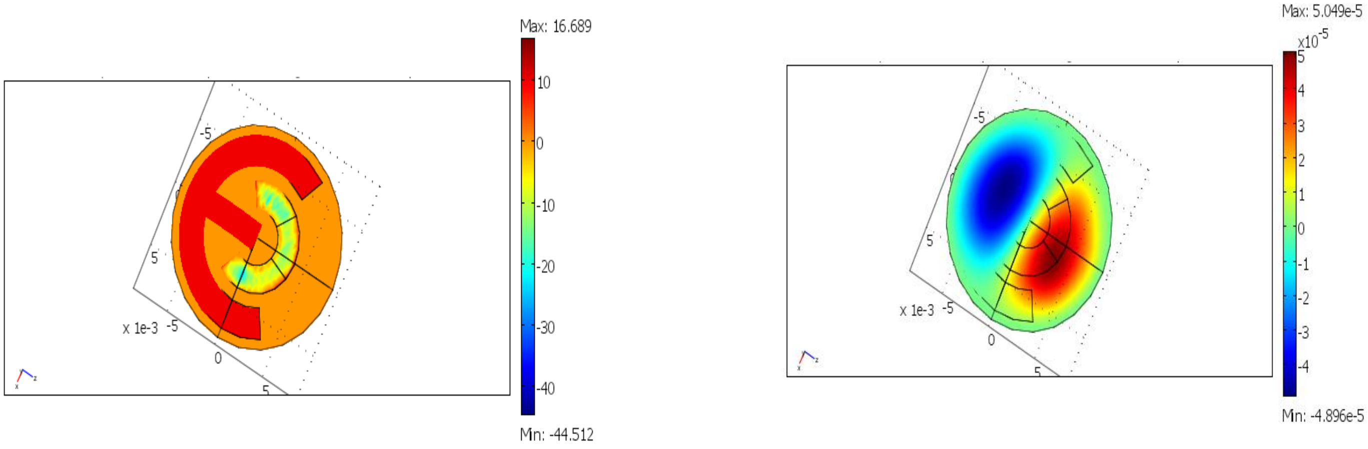

Figure 24. Frequency 3924Hz

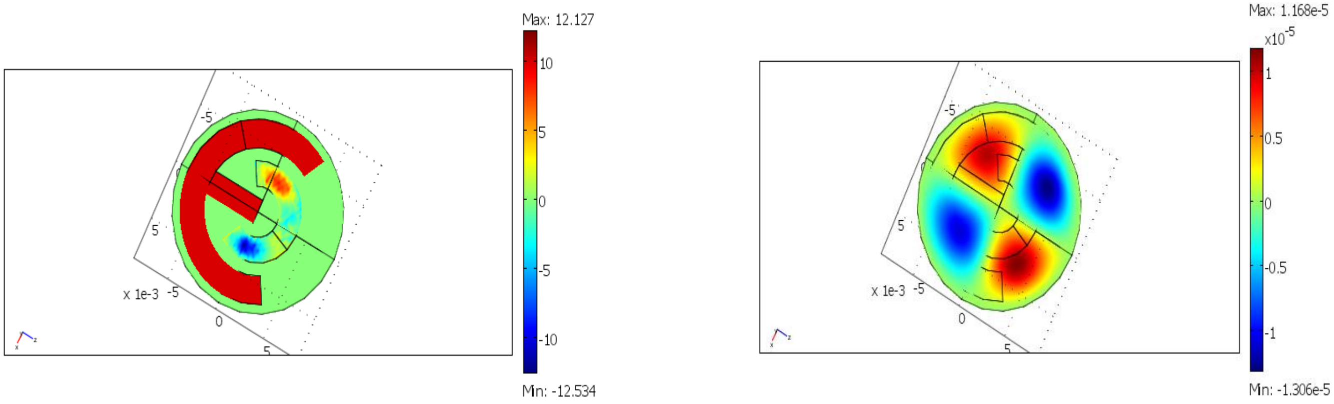

Figure 25. Frequency 6730Hz

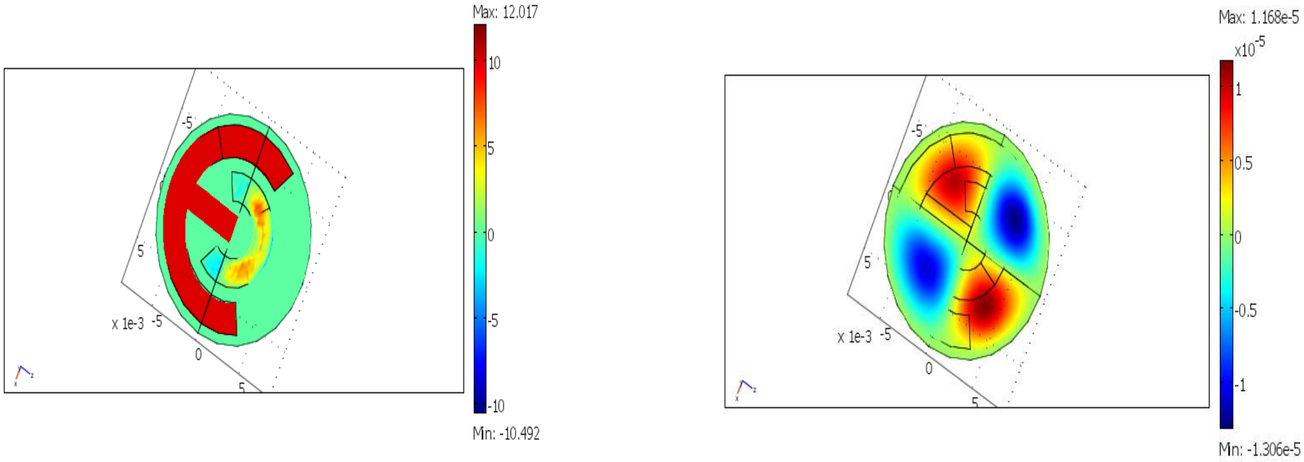

Figure 26. Frequency 6964Hz.

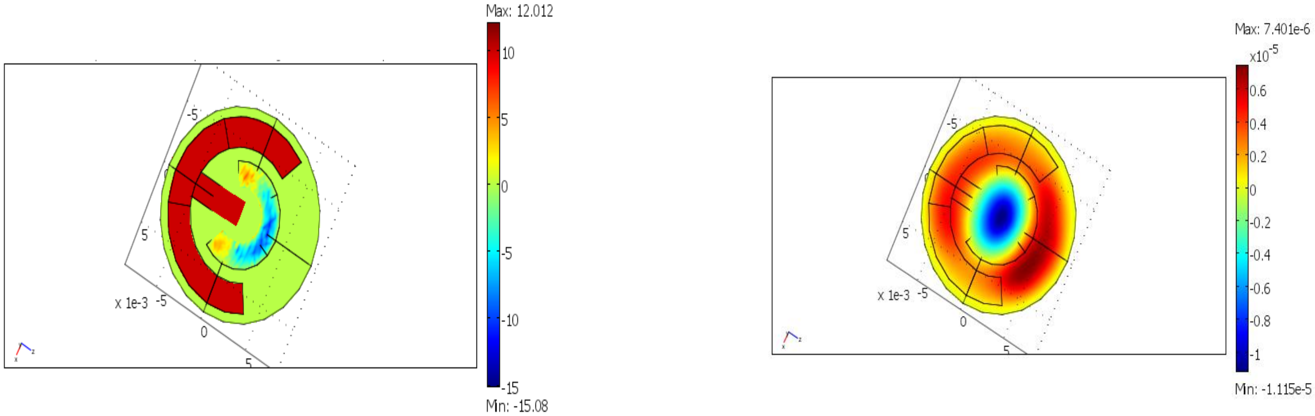

Figure 27. Frequency 8150Hz

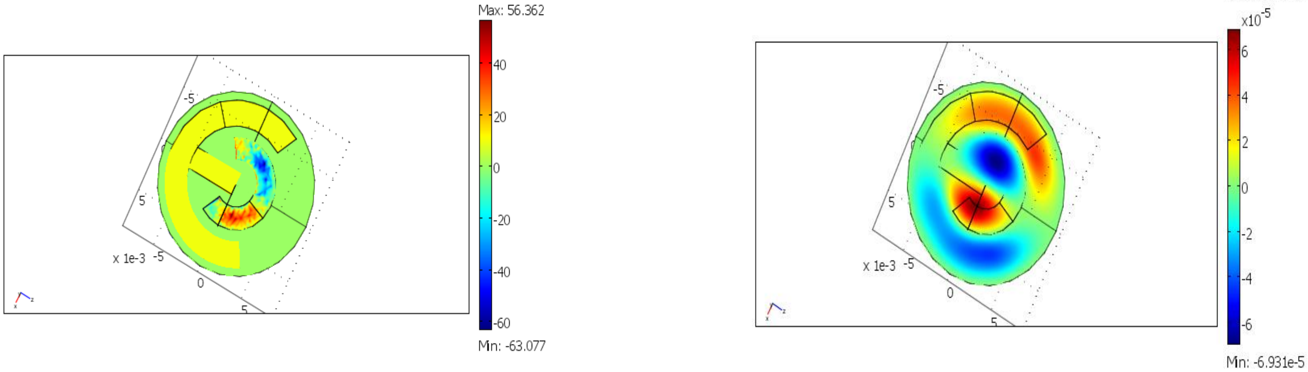

Figure 28. Frequency 10770Hz

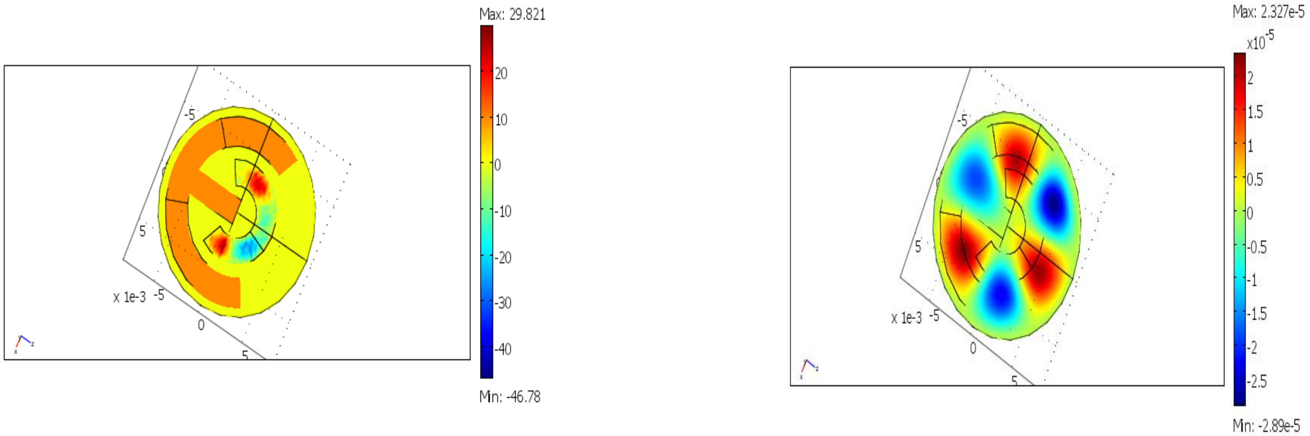

Figure 29. Frequency 10896Hz

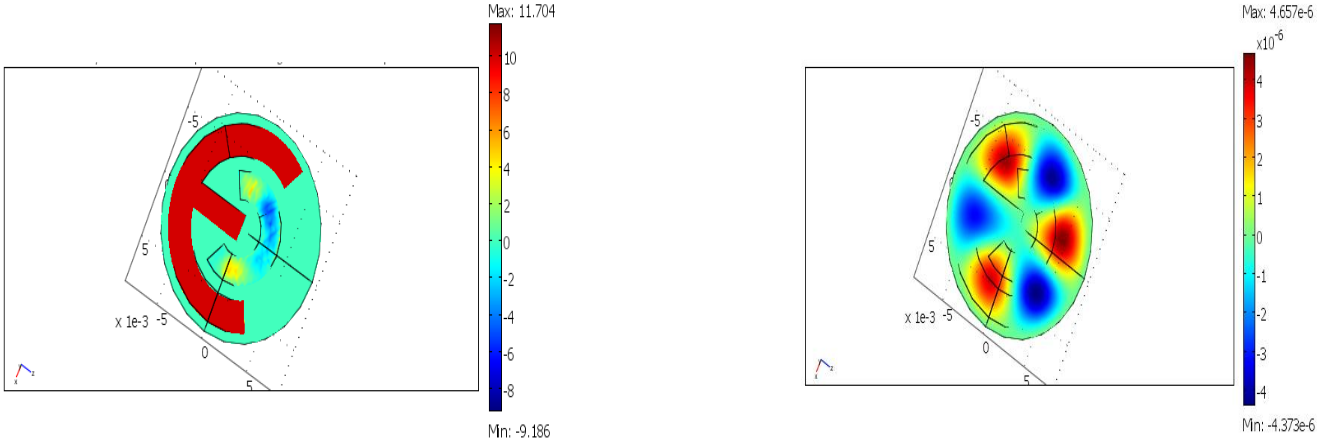

Figure. 30 Frequency 13280Hz

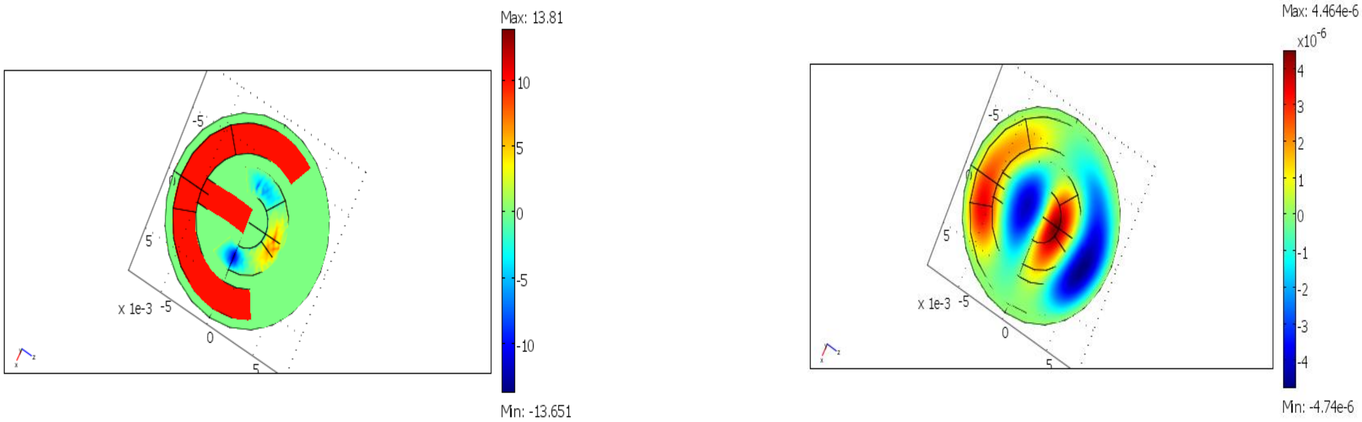

Figure 31. Frequency 13900Hz

By careful observation of the above graph, it can be noted that at a frequency of 8150Hz it is recorded to have a maximum voltage of 2.2V for the corresponding 10V input. At the frequency 8150Hz the sensing electrode shows same sign charge. Therefore it can be concluded that at this Frequency it gives maximum voltage when it not divided into smaller segments. At the frequency 13900Hz also shows a larger value of voltage but in negative sign. If the sensor is subjected to further segmentation then this frequency would suit the best as the total generated voltage is higher at this frequency.

In this report the author is not subjecting the sensors to segmentation therefore at 8150Hz , the sensor is furthere analyzed with variable input voltages and accounting the variation in output voltages.

3.2.4 Input Voltage Variation

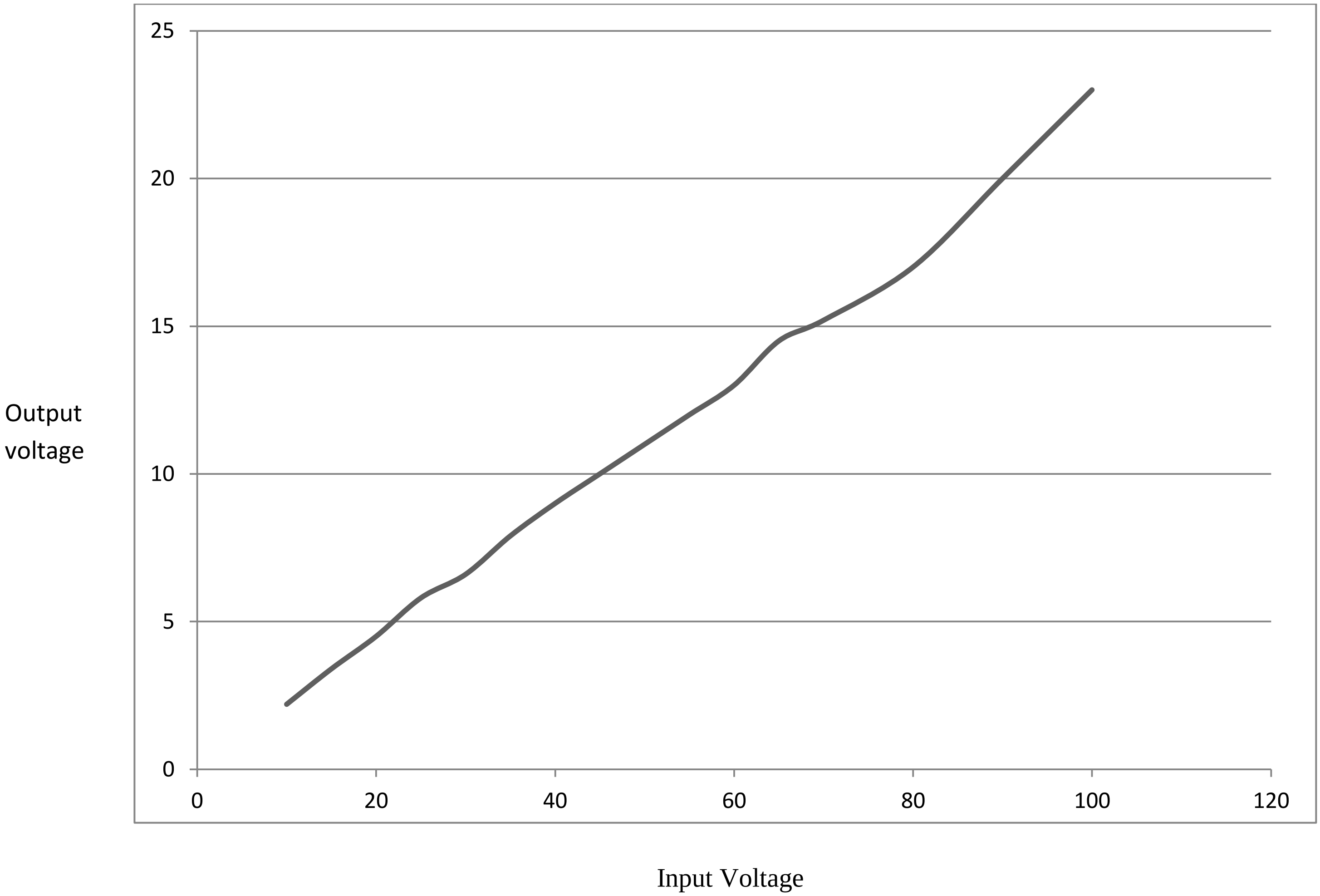

Figure 32 Input Voltage vs Output Voltage

The above graph shows the variation between input voltages and output voltages. Like explained in the earlier sections, maximum voltage was recorded at 8150Hz and the input voltages were varied at this frequency. So at this frequency as the input voltage increases , the output voltage also tends to increase resulting in a linearly dependent graph.

Conclusions

Experimental and theoretical approaches of a multilayer element with new type piezoceramic material were carried out. Using the experimental results, the damped natural frequency of proposed multilayer structure was 186 Hz and 187.056 Hz (natural frequency). The finite element model have shown the first form of vibrations at 187.728 Hz frequency. Comparison of these two models have shown only 0.4% difference. By comparing experimental and numerical models modulus of elasticity of novel piezoceramic material of 1 MPa was calculated.

From the Comsol multiphysics analysis it can be concluded that the sensor shows maximum voltage at a frequency 8150Hz for non segmented case and 13900Hz for segmented case. The output voltage increases as the input voltage increases for the sensing body.

In the beginning of this report, the author proposed few questions that revolves around this research.

- What are the Frequencies that govern the features of the new composite Piezo material?

  In the measuring interval amplitude is 150 µm and 6 µm after a period of one interval. A natural frequency of 187.056 Hz was calculated. The first mode of vibrations in COMSOL Multiphysics model is achieved with 187.728 Hz. This result is only 0.4% higher than the experimental result. The properties of the new composite material is described in this report. From the experimental results, the new composite material possess a young's modulus of 1MPa. The highest voltage is generated when it is excited at 8150Hz. The output voltage increases in accordance with the input voltage.

- What are the different fabrication techniques that can be used to fabricate these microresonators?

  Different methods of fabrication of PZT has been described in this report. The author uses Thermal embossing method for PZT. The PZT powder is mixed with 20% solution of polyvinyl butyral in benzyl alcohol. A lamellar periodical microstructure was formed on the surface of piezoelectric thin top layer with 4 µm. Periodical microstructure was dip-coated by silver (Ag) nanoparticles formed in deionized water from a solution of 0.05 $AgNO_{3.}$

- What are the possible improvements and application using these microresonators?

By developing a new PZT composite sensor it is expected to improve the quality of the molds. It would help to analyze the defects in the mold and is expected to give a clear understanding of the mold cavity.In the field of automobile industry , efficiency plays a key role in injection molding and in space industry reliability plays a key role. The results, conclusions of this work could help improve the sensors in injection molding. Issues related to curing time, viscosity of the melt , uniformity in the cavity is expected to have some remedies using the results of this work.

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