

Characterization of a Pt-core PZT Fiber/Al Matrix Composite

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ABSTRACT

The objective of this study is to design and characterize a piezoelectric composite and evaluate its suitability for viscosity-measuring applications, i.e., monitoring the coagulation rate of blood. The composite is manufactured of a platinum-core lead zirconate titanate (PZT) fiber inserted into an aluminum matrix. This study characterizes the described composite by testing its impedance, capacitance, voltage sensitivity response to vibrational inputs, and deformation due to electrical input. As actuators, different voltage inputs are fed into the probes and displacement is measured with results on the range of nanometers. As sensors, the devices are used to monitor cantilever beam vibrations. The probe's response is in the mV range and follows the same pattern as an accelerometer. Additional tests in air, water, and deionized water are carried out to evaluate the sensor's suitability for measuring viscosity using two probes: one as an actuator and the other as a sensor. Results of the gain and phase between the two probes indicate that the phase shift may be used as an indicator of viscosity changes. The first significant phase shift was measured as 2.45, 2.77, and 4.065×10^7 Hz, for water, air, and oil, respectively, which is directly proportional to the kinematic viscosity of each fluid.

Keywords: Piezoelectric fiber, viscosity, sensor, actuator

1. INTRODUCTION

Monitoring the blood coagulation process is critical during anesthesia and surgical procedures. Conventional coagulation monitoring methods such as point-of-care (POC) analyzers have several drawbacks, such as requiring specialized laboratory personnel and large samples of blood (Ganter and Hofer, 2008). Limitations of the conventional methods create a need for the development of better methods. Several existing methods for measuring blood coagulation using vibrating sensors have been designed and tested but never fully implemented. A selection of these methods are described in the following paragraphs.

Mechanically damped quartz crystals have been investigated for the detection of IgG (a type of antibody in blood) from a solution (Thompson et al., 1986). Protein A, a bacterial protein that binds IgG, was immobilized on the crystal surface. Reaction of protein A and human IgG changes the resonance frequency since there is a correlation between resonance frequency shifts and the change of the mass. This correlation monitors the change in human IgG concentration (Muramatsu et al., 1987, 1991).

Piezoelectric Quartz Crystal (PQC) sensors which are traditionally called *Quartz Crystal Microbalance* (QCM) have been used for the determination of blood coagulation time (Cheng et al., 1998). A thin AT-cut quartz wafer is sandwiched between two metal excitation electrodes to prepare a PQC sensor. Coagulation time is determined by the graph of oscillating frequency response to the plasma coagulation reaction (Chang et al., 2000). QCM-D is a variation of QCM that allows simultaneous frequency (f) and dissipation factor (D) measurements (Hook et al., 1998). The QCM-D technique was refined and used for real time measurement of blood coagulation density. It was found that the blood plasma clot density can be assessed with the use of the factor D (Andersson et al., 2005). *Surface Plasmon Resonance* (SPR) is another method which has been used for blood coagulation analysis (Hansson et al., 1999). The response signals of this method are similar in shape to QCM-D but they have different time scales and QCM-D has a longer response time (Viking et al., 2000b).

Magnetoelastic sensors have also been used to monitor blood coagulation (Puckett et al., 2003). Ribbon-like magnetoelastic sensors oscillate at a fundamental resonance frequency when they are in a magnetic field. The changing viscosity of blood as it coagulates shifts the characteristic resonance frequency of the sensor. A remotely located pick-up coil detects the magnetic flux which is emitted by magnetoelastic sensors (strips). Therefore there is no need for a direct connection between the sensor and the detector. Thanks to their simple design these sensors can be miniaturized and used disposably.

The objective of this study is to design and characterize a piezoelectric composite probe and evaluate its suitability for viscosity-measuring applications. The composite is manufactured of a platinum-core lead zirconate titanate (PZT) fiber (Sato, et al., 2002) inserted into an aluminum matrix, which serves as the ground terminal. This type of sensor utilizes the piezoelectric effect to measure the viscosity of a fluid by using one or more fibers as actuator and sensors. The shift of resonance peaks between the sensing and receiving fibers may be related to the difference in viscosity. This study proposes a design for a sensor/actuator that may be used for this application. This report outlines the construction of the probes, characterization of the probes' piezoelectric sensing and actuating properties, and assessment of the probes' suitability to be used as viscosity sensors. The ultimate goal is to use this probe to create a system for measuring the coagulation of blood that is competitive with presently available systems.

2. MANUFACTURING

2.1 Materials

The probes are constructed using 0.2mm and 0.4mm thick, 30mm wide, and 30mm long pure aluminum plates (A1050P-O), a platinum-core lead zirconate titanate (PZT) fiber of 0.22mm outside diameter with a platinum core of 0.05mm diameter produced by Nagamine Manufacturing Co., Ltd., and a 0.01mm thick, 30mm wide and 30mm long copper foil of 99.9% purity.

2.2 Fabrication

Piezoelectric ceramic fibers are very fragile and usually reactive with aluminum. In order to overcome these problems, the interphase forming/bonding (IF/B) method (Asanuma, 2000) shown in Figures 1a through 1c is applied. In Figure 1a, to form the U-groove, a steel wire 0.35mm in diameter is pressed on the copper foil and the 0.4mm thick aluminum plate together with a pressure of 98MPa for a period of 0.18ks. At 0.35mm deep, the groove is larger than the 0.22mm diameter of the piezoelectric ceramic fiber filament. Next, the fiber filament is placed in the groove and covered with the 0.2mm thick aluminum plate. In the second step, Figure 1b, the layered materials are hot-pressed in a low vacuum (100Pa) under the pressure of 2.2MPa. When the sample is heated to around the eutectic temperature of Al-Cu alloy, the copper insert reacts with the aluminum matrix very quickly, and the molten alloy partially flows in the void between the U-groove and the fiber filament. After further heating to 873K the molten Al-Cu alloy with a composition close to that of the eutectic alloy, it can be squeezed out smoothly and the void can be closed by additional hot pressing for 2.4ks as shown in Figure 1c.

The composite is then cut to the specified dimensions of the sensor and soldered to a connector for ease of testing. A voltage of 300V is applied between the aluminum matrix and the platinum-core for 1.8ks to polarize the piezoelectric ceramic.

3. EXPERIMENTAL SETUP

3.1 Samples

Two probes are used in this study and designated probes A and C. The probes, shown in Figure 2, have slightly different dimensions as shown in Table 1. The probes consist of a sensing area and a connector area. The sensing section has a rectangular cross-sectional area with a circular fiber embedded in it. The diameter of the Pt-core of PZT fiber is 0.22mm. Details of a typical cross section are shown in Figure 3.

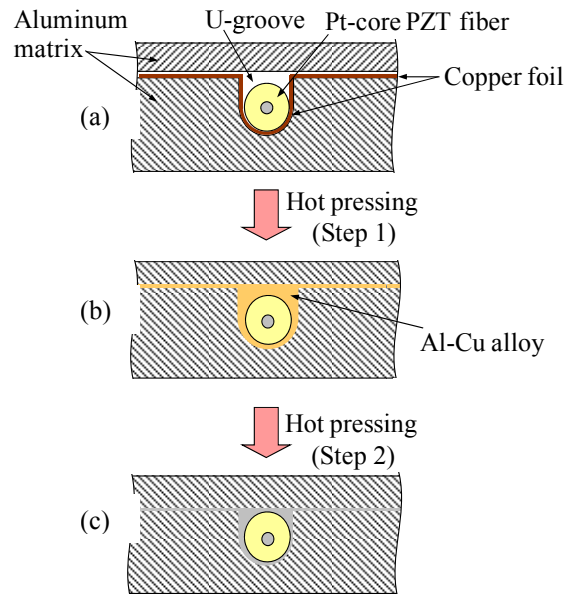


Figure 1 Interphase forming/bonding method: (a) Preparation; (b) Low Vacuum Hot-pressing just after eutectic reaction; (c) High Temperature Hot-pressing

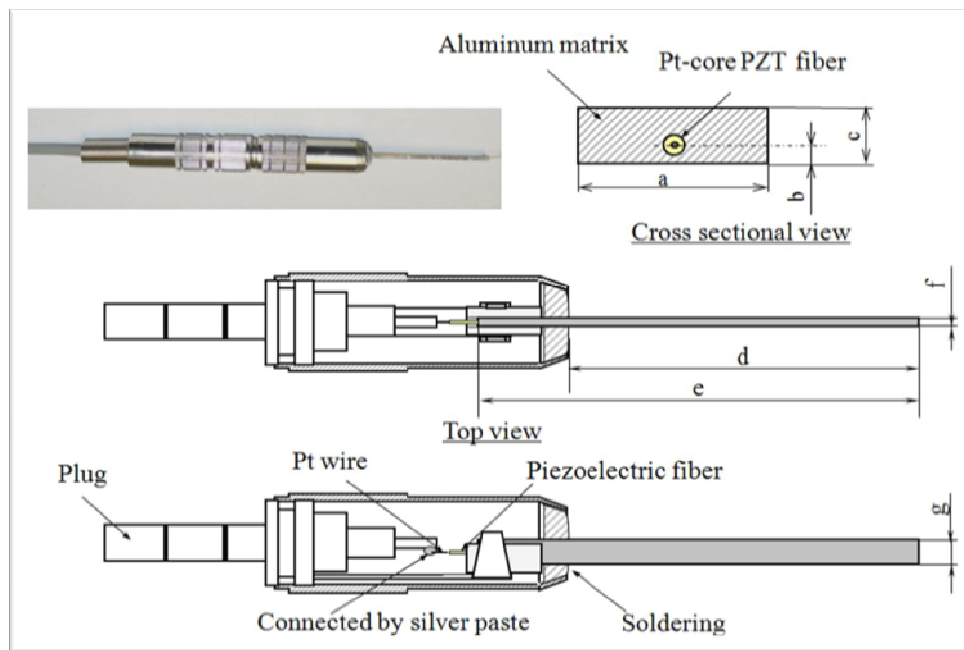


Figure 2 Sectional views of the Pt-core PZT fiber/Al matrix composite

Table 1 Probe Dimensions

Dimensions (mm)	A	b	c	d	e	F	g
Sensor A	1.620	0.140	0.625	24.00	30.00	0.625	1.620
Sensor C	2.00	0.140	0.650	24.00	30.00	0.650	2.00

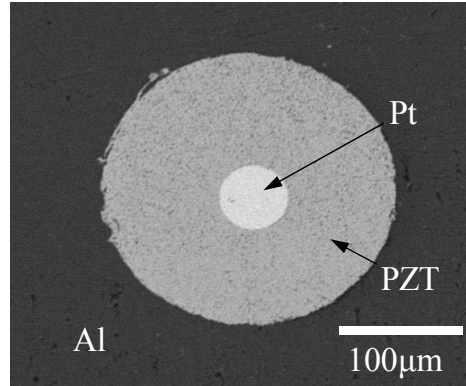


Figure 3 SEM image of a cross section of the Pt-core PZT fiber

3.2 Quality Assurance Tests

The probes are subjected to several tests. Capacitance and impedance measurements are accomplished using a HP4194A impedance analyzer and using Labview[®] for data acquisition. Because of the expected high electrical resonance, a faraday cage is utilized, into which the probes were placed for all tests.

3.3 Sensor Output Setup

The experimental setup in this case consisted of placing a PT-core fiber/Al matrix composite probe at the end of a cantilever beam. The beam serves as the vibration source. The voltage output of the probe is monitored through a Tektronix TDS 2024 oscilloscope and collected through a GPIB connection. The vibration source, the cantilever beam, is monitored using a Bruel & Kjaer Deltatron accelerometer Type 4508B.

3.4 Displacement Monitoring Setup

The Pt-core PZT fiber/Al matrix composite probe is placed on a probe holder and powered using a signal generator and an amplifier. The probes are powered using an HP3567A signal generator and a TREK model PZD700 voltage amplifier. In this manner, two sinusoidal input voltages, 400 and 800V_{pp} can be used for input for the probes. Displacement of the sensor is monitored using an optic fiber, an Angstrom Resolver Series Dual Channel Model 201 placed on a three-dimensional stage for precise positioning. The fiber optic probe output is monitored through an oscilloscope and a computer data acquisition system as shown in Figure 4.

3.5 Actuator-Sensor Detection Setup

In this case two probes are connected to the Gain-Phase Analyzer: one as an actuator and the other one as a receiver. The two probes are placed inside a covered container as shown in Figure 5. Probe A is the actuator with a sinusoidal input waveform of 0.9V_{rms} input. Probe C is the sensor probe. The frequency is swept at different frequency ranges and data is collected through a GPIB card.

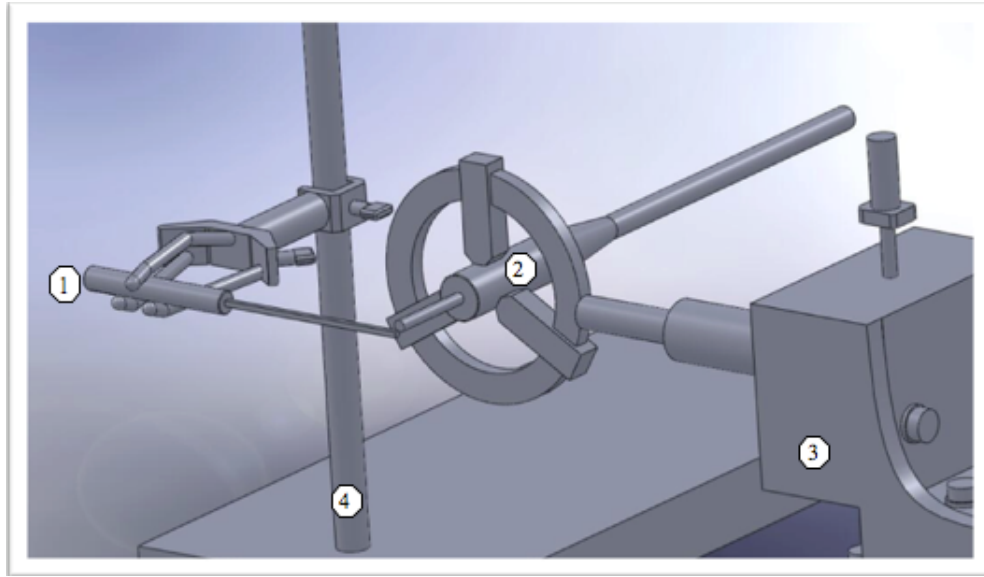


Figure 4 Displacement Monitoring Setup: (1) Pt-core PZT fiber/Al matrix composite probe; (2) Fiber optic probe; (3) Three-dimensional stage; (4) Pt-core PZT fiber/Al matrix composite probe holder.

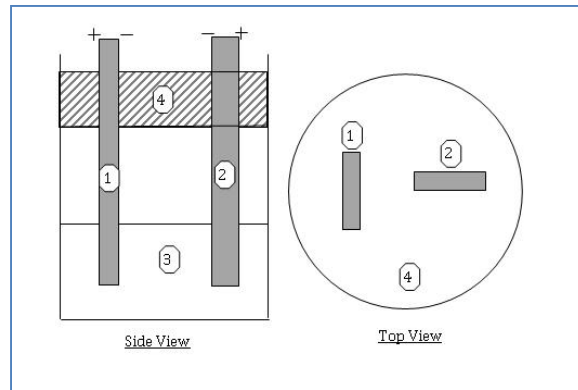


Figure 5 Actuator - Sensor Setup: (1) Actuator Probe [Probe A]; (2) Sensor Probe [Probe C]; (3) Fluid; (4) Top Cover

4. RESULTS AND DISCUSSION

The capacitances of the tested probes in the frequency range 100Hz to 1MHz are as shown in Figure 6 for probes A and C. A resonant peak is observed around 8.6MHz and anti-resonance peak at 11.6MHz for probe A with an average capacitance of approximately 0.6nF. For probe C the resonant and anti-resonant peaks are observed at 30.2 and 30.5MHz respectively with an average capacitance of 0.1nF.

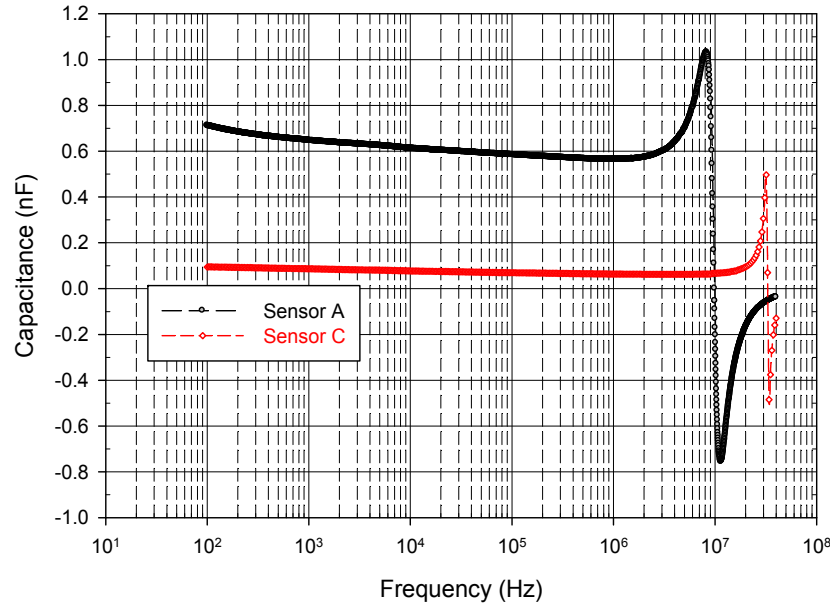


Figure 6 Capacitance vs. Frequency

Impedance measurements are also performed for both probes and results are shown in Figure 7. Note that both probes follow similar trends until a peak occurs at 30MHz and 10MHz for probes A and C respectively. At 1kHz the impedance for probes A and C are $2\text{M}\Omega$ and $0.25\text{M}\Omega$ respectively.

Results of capacitance and impedance are indicators that the probes are in good condition and can be used as actuators and sensors since both curves are typical for piezoelectric materials with impedances of the 10^7ohms order of magnitude and resonant and anti-resonant peaks.

Then the displacement of the probes can be quantified. Using the displacement setup described, signals were collected at different frequencies. A typical signal at 250Hz is shown in Figure 8 for two input sinusoidal voltages 100V_{pp} and 200V_{pp} [Probe A]. The displacement signals have noise on them, so smoothed data lines are shown for clarity. The displacements at 100V_{pp} and 200V_{pp} are 698.5 and 1397nm (filtered data).

Since the probes can also be sensors using the setup described in section 3.3 their response is recorded with certain limitations: the probe is not directly put in contact with the beam to avoid damage to the probe. A typical result is shown in Figure 9 for the probe and accelerometer outputs. Therefore the test verifies that the probe produces a measurable output (mV range).

Once the probes are characterized as sensors and actuators, they can be tested as health monitoring devices. By testing them in different fluid mediums, the feasibility of using them to detect changes in viscosity is assessed. The test is performed using air as a baseline in a controlled environment. Cooking oil and deionized water were the other fluids chosen due to their properties: they have different viscosities and are both electrical insulators. Distance between the probes, level of liquid, and probe direction at this stage of the study are not optimized and are arbitrarily chosen. Even though these factors are not controlled, the results shown in Figure 10 show significant differences in the gain measured in the MHz frequency range. More significant are the changes in phase angle as shown in Figure 11. To more clearly identify significant changes in phase angle, the first derivative of the phase angle vs. frequency is taken for all data and is shown in Figure 12. Note that the first highest peak for each frequency sweep is $2.446 \times 10^7\text{Hz}$ for water, $2.777 \times 10^7\text{Hz}$ for air, and $4.065 \times 10^7\text{Hz}$ for oil. These values can then be correlated to the kinematic viscosity of each fluid as shown in Table 2 where the viscosity is linearly proportional to the frequency shift. These results are encouraging and are the target of ongoing studies.

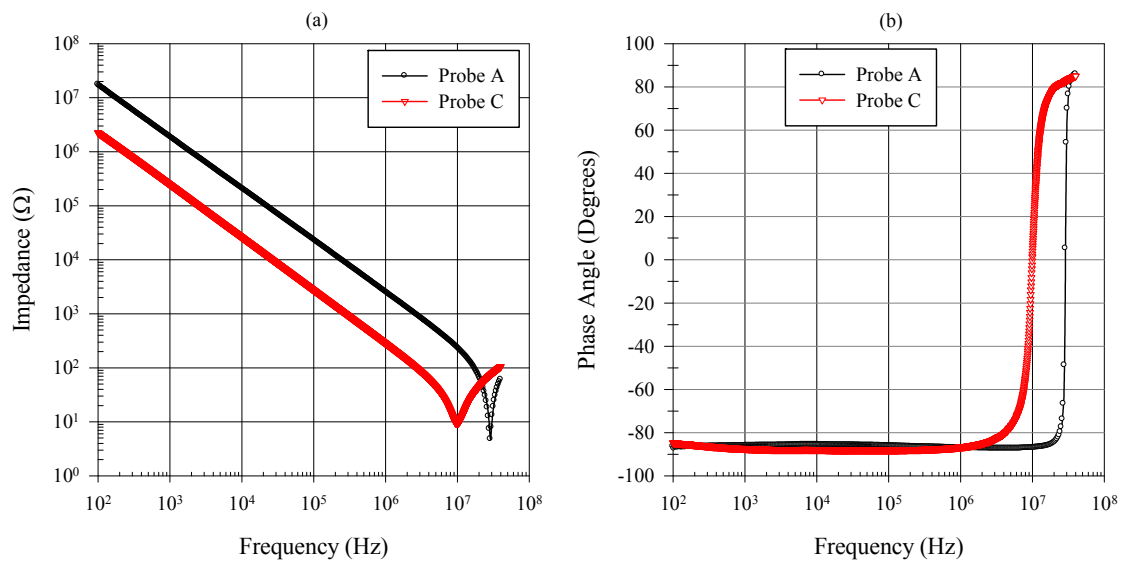


Figure 7 (a) Impedance and (b) Phase Angle for Probes A and C

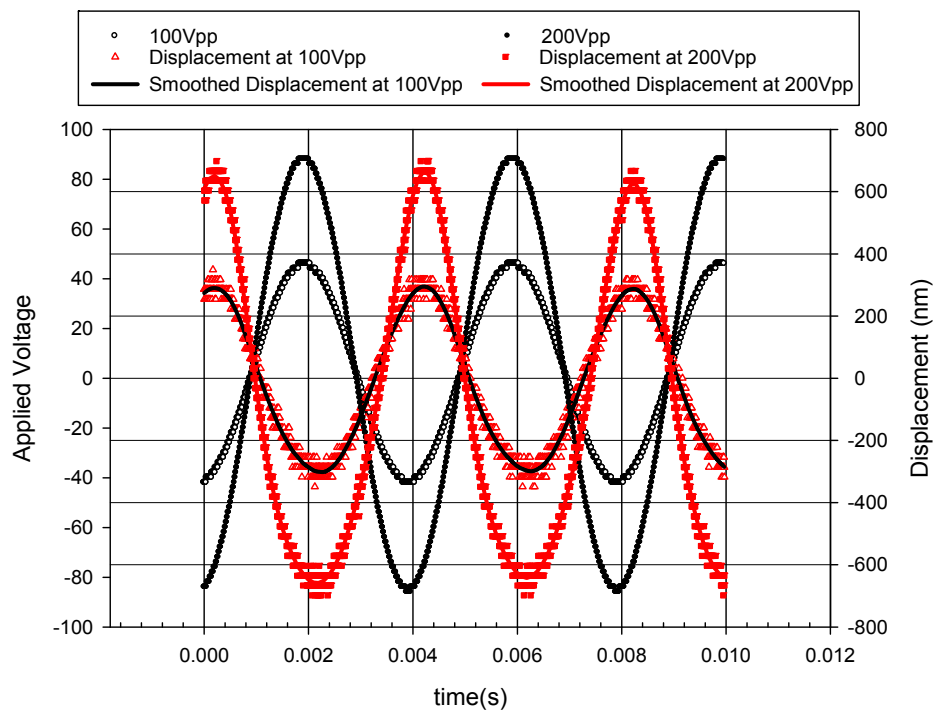


Figure 8 Applied Voltage vs. Displacement at 250Hz for Probe A

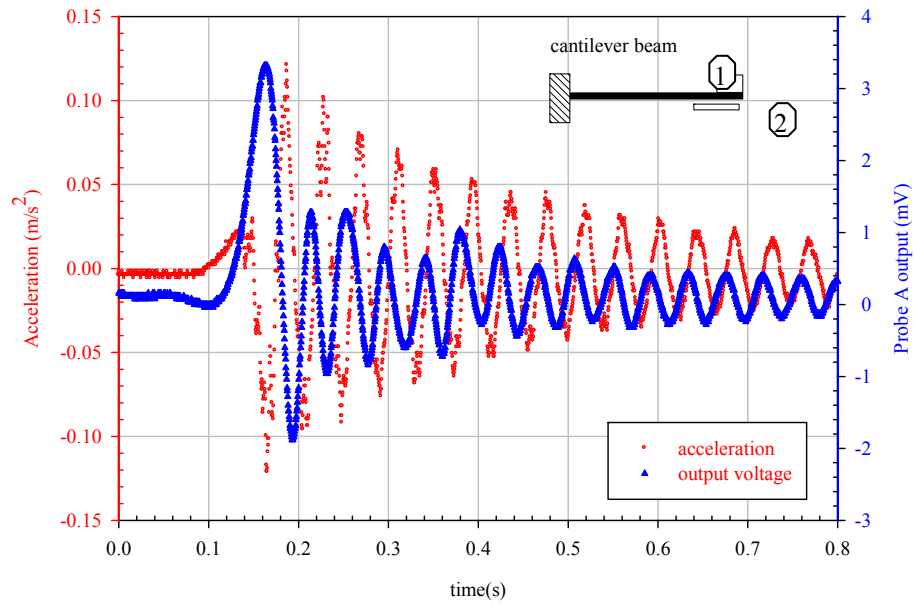


Figure 9 Cantilever beam vibration experiment (1) accelerometer, and (2) Probe A

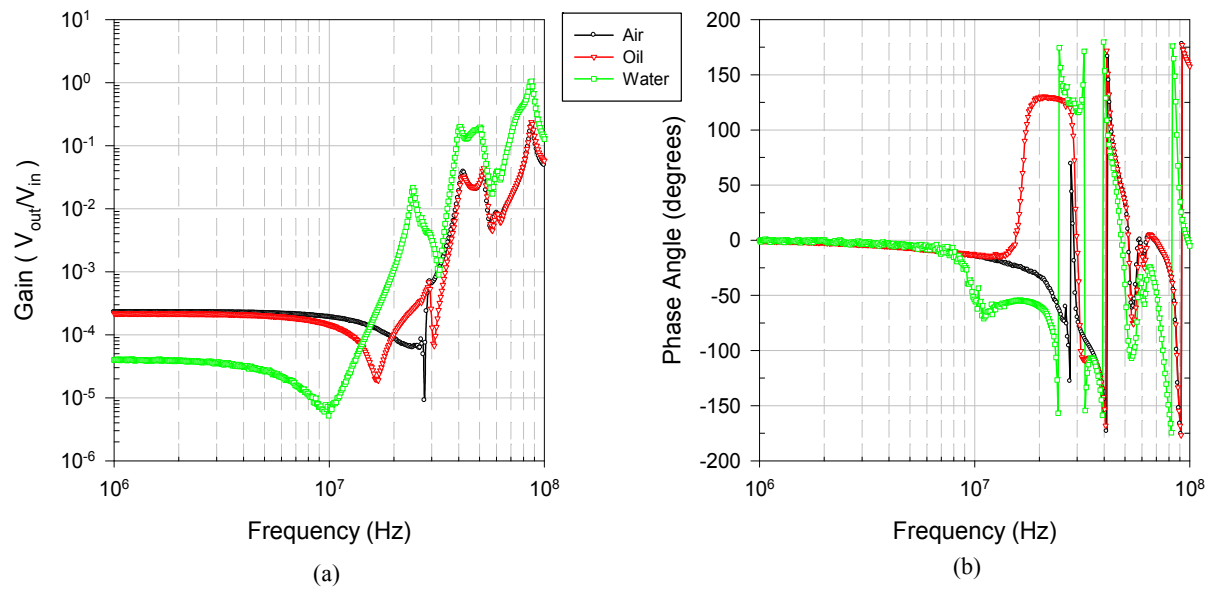


Figure 10 Actuator-Sensor outputs with frequency (a) Gain (b) Phase Angle

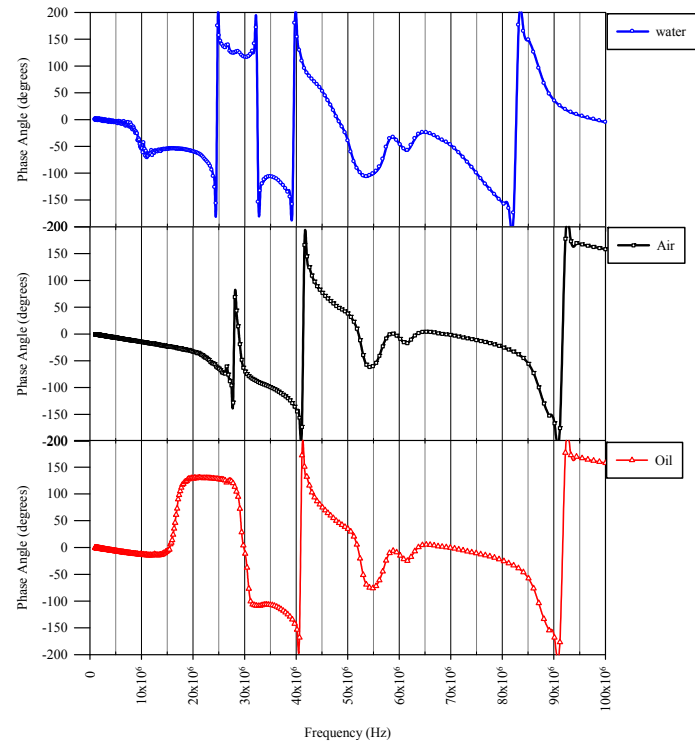


Figure 11 Phase angle vs. frequency linear sweeps for water, air and oil

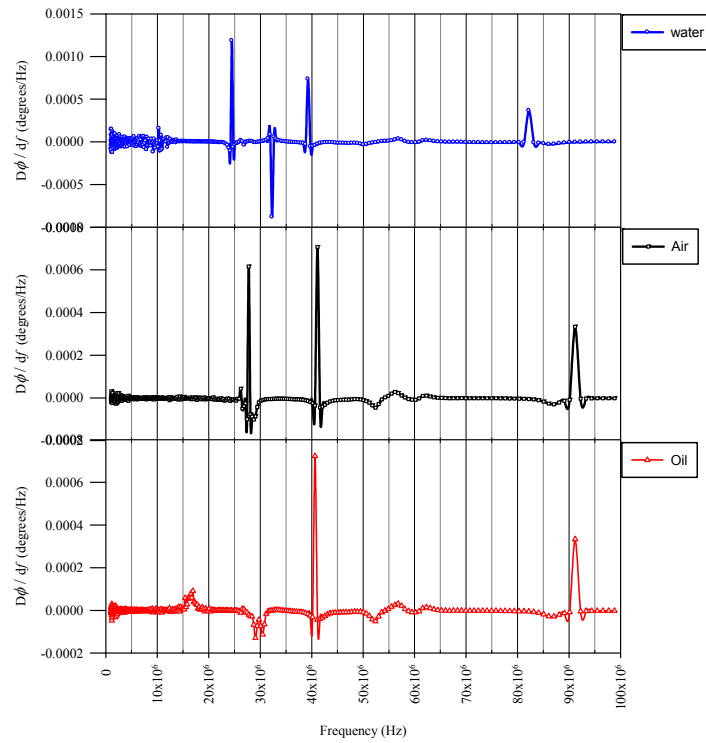


Figure 12 First derivative of the phase angle with frequency vs. frequency for water, air, and oil

Table 2 Kinematic Viscosity and peak frequency for water, air, and oil

Liquid	Kinematic Viscosity (cST)	Peak frequency shift of ϕ $\times 10^7 \text{Hz}$
Water ^a	01.0038	2.446
Air ^b	15.2500	2.777
Oil ^c	63.0000	4.065

^aengineeringtoolbox.com

^bTracy et al., 1980

^cLang et al., 1992

5. CONCLUSIONS

A process to manufacture Pt-core PZT fiber/Al matrix composite probes is described. The probes' capacitance and impedance are documented and checked for quality control purposes. Then, these probes are characterized as actuators and sensors. As actuators, different voltage level inputs are fed into the probes and displacement is measured with results on the range of nanometers. As a sensor, the devices are used to monitor the vibrations produced by a cantilever beam. The probe's response is compared to an accelerometer and measured in the mV range. Finally, the probes are tested as an actuator-sensor pair in air, oil and deionized water in a controlled environment. The results show significant differences in gain and phase angle for the different mediums. To better quantify differences, the first derivative of phase change is taken with respect to frequency to enhance the differences. The results showed that the frequency where phase angle changes is directly proportional to the kinematic viscosity of the three fluids tested.

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