

SMASIS2010-' * , ,

FEASIBILITY OF USING PIEZOELECTRIC PROBES TO MEASURE VISCOSITY IN NEWTONIAN FLUIDS

Rachel Waxman Ugur Erturun Karla Mossi
 Department of Mechanical Engineering, Virginia Commonwealth University,
 Richmond, VA, USA

ABSTRACT

Viscosity plays an important role in modeling fluid flow in different systems. In Newtonian fluids, viscosity is a constant, measureable property. Currently, viscosity is measured using viscometers that use an array of different techniques depending on the application. In this study, pre-stressed lead zirconate titanate (PZT) composites were used as probes to monitor changes in viscosity. The probes are used as an actuator-sensor pair: a voltage of $1V_{rms}$ will be applied to one probe, the actuator; the second probe, the sensor, receives a vibration wave and turns it into an output voltage. Measurements of gain and phase at different input signal frequencies are analyzed. The fluid-medium where the probes are tested consists of different glycerin-deionized water solutions. Results indicate that the frequency of peak phase shift can be correlated to fluid viscosity. This correlation is exponential with viscosity, with an R^2 of 0.99. Results included viscosity values in the range of 0.8cP to 612cP. Possible applications for this type of sensor are numerous, and are both non-time-dependent (simple viscosity measurements of fluids), and time-dependent.

INTRODUCTION

Viscosity is the measure of a fluid's resistance to shear stress, tensile stress, or deformation. Dynamic viscosity is defined by the ratio of shearing stress (force over area) to the velocity gradient in a fluid [1], or:

$$\mu = \frac{F / A}{dv_x / dz} \quad (1)$$

The instrument used to measure viscosity is called a viscometer. There are five different categories of conventional viscometers commercially available. These categories include: vibration based, rotational, capillary, falling-ball, and cup-type

[2]. Figure 1 shows examples of each type, in the order listed above.



Figure 1. Common viscometers: (a) vibration based [15], (b) rotational [16], (c) capillary [17], (d) falling-ball [18], (e) cup-type [19].

The five different categories of viscometers all function and take their measurements in different ways. The vibration based viscometer immerses a transducer in the fluid. While the amplitude of the transducer is controller, the current driving the transducer is recorded and correlated to viscosity.

The rotational viscometer immerses cylindrical rotors in the fluid. The rotors are spun at a constant speed. The torque required to maintain that constant speed is related back to viscosity.

Capillary viscometers function by allowing a fluid to flow downward through a capillary, and measuring a pressure difference between the tube's ends. This difference is found by measuring the time it takes the fluid to flow, and using the viscometers inherent viscosity constant to correlate to viscosity. These viscometers are fairly cheap, but are designed for use over a specific kinematic viscosity range (e.g., 0.8 to 4 cSt).

The falling-ball viscometer is somewhat similar to a capillary viscometer. Instead of the fluid flowing downward,

an object (a column or sphere) of known dimensions and density is allowed to fall through the fluid. The time it takes for the object to fall a certain distance is related back to viscosity. The falling-ball and capillary viscometers cannot constantly measure viscosity, while the vibration based and rotational viscometers can.

The last type, the cup-type viscometer is often used in measuring the viscosity of paint or ink. For the cup-type viscometer, a cup has an open tube extending from its bottom surface. The user fills the cup with a fluid until the cup is completely full, while plugging the hole at the bottom of the tube. The time it takes for the cup to drain completely is then measured and correlated to viscosity. This type of viscometer is also not appropriate for non-constant measurements [2].

In spite of the options available, there is still a need for miniaturized, faster, and more advanced viscosity measurement techniques for various applications. A recent alternative technique for viscosity-sensing devices has been developed mostly based on the mechanism of resonance [10].

The Quartz Crystal Microbalance (QCM) and the electrochemical quartz crystal microbalance (EQCM) are well known methods of viscosity measurement based on the mechanism of resonance. The QCM sensor consists of an AT-cut quartz crystal with two electrodes on opposite sides. Applied electrical field leads to the vibration of the crystal (through the piezoelectric effect). A transverse acoustic wave is generated by this vibration for an AT-cut quartz crystal in thickness-shear mode. This wave propagates across the thickness of the crystal [3]. According to Kanazawa, the QCM frequency changes with the change of viscosity and density of the liquids which are in contact with the resonator as shown in Equation 2.

$$\Delta f = f_0^{3/2} \sqrt{\frac{\eta_L \rho_L}{\pi \mu_Q \rho_Q}} \quad (2)$$

In equation (2), Δf is measured frequency shift, f_0 is resonance frequency, η_L and ρ_L are viscosity and density of the liquid in contact with the QCM, respectively, and μ_Q and ρ_Q are the shear modulus and density of the quartz crystal, respectively [4].

Some of the QCM applications include viscosity and mass measurements of glycerol-water mixtures [5]; viscosity measurements of aqueous solutions of sucrose, urea, PEG-400, glucose, and ethylene glycol at $25^\circ\text{C} \pm 0.5^\circ\text{C}$ [6]; viscosity measurements of a selection of industrial oils, including commercial automotive lubricants, heavy fuel oils, calibration oils, and used automotive lubricating oils [7]; and determination of concentrations of Endotoxin, a type of fever-inducing pyrogen [8]. Thickness-shear mode (TSM) resonators

are alternative devices to measure viscosity and density of liquids. Like QCMs, they are based on AT-cut quartz crystal, but their structures are different. The quartz of a TSM resonator is a thin disk with circular electrodes patterned on both sides. Smooth- and textured-surface TSM resonators have been studied to measure density and viscosity of liquids [9].

Piezoelectric Unimorph cantilevers have also been examined as liquid viscosity and density sensors. The cantilever mainly consists of a PZT driving electrode and PZT sensing electrode, which is permitting direct measurement of the resonance frequency. The shift in the resonance frequency is closely related to the change in the viscosity of a glycerol-water test liquid [10]. Multimode dynamic response of composite cantilever beams in various viscous media such as air, water, ultrapure ethanol, and silicon oil has also been investigated for determining viscosity [11]. Agoston et al. evaluated a vibrating cantilever sensor for viscosity measurements of complex organic liquids and compared it with conventional viscometer and micro acoustic sensor [12]. Another sensor application of PZT cantilevers is real-time in-water cell detection [13]. Two cantilevers with different sensitivities were used to detect yeast cells.

Previous successes of viscosity measurements with piezoelectrics (namely with QCM and PZT) had noticeable disadvantages. QCM methods utilize quartz crystal, which is far more expensive than PZT and materials being used in other methods. The studies conducted with PZT cantilevers presented an accurate model, but the model only worked in the range of 1cP to 600cP [10]. The study presented here investigates the use of piezoelectric probes to detect viscosity by way of phase shifts between input and output signals. Two pre-stressed PZT composites were used as probes. One probe was used as actuator, which caused vibrations in the fluid. The second probe was used as a sensor to detect these vibrations and turns them into an output voltage. Success was achieved in the range of 0.8cP to 612cP. The experimental setup is explained below.

EXPERIMENTAL SETUP

Two piezoelectric probes were utilized as an actuator-sensor pair. These probes were pre-stressed lead zirconate titanate type 4195HD, with a brass backing material and an aluminum foil top layer. These probes were manufactured by Face International Co. of Norfolk, Virginia. The dimensions of the probes are overall length of 12.395mm, width of 1.981mm, and a total thickness of 0.4064mm. The brass backing extends farther than the PZT and aluminum, and was therefore used for mounting. This is shown in Figure 2 as a tab holder. Figure 2 also shows a diagram and a description of each layer. The thicknesses of the layers were 0.0254mm aluminum, 0.0127mm adhesive, 0.2032mm PZT, another adhesive layer, and a metal brass layer of 0.1524mm.

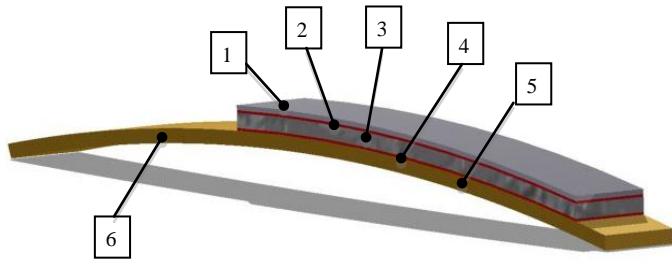


Figure 2. Pre-stressed piezoelectric probe composite. The layering sequence consists of: (1) aluminum,(2) adhesive, (3) PZT, (4) adhesive, (5) brass, (6) tab holder.

The experimental setup is based on the cantilever beam setup used most often with piezoelectric materials. The probes were mounted on holders with aluminum layers facing each other as shown in Figure 3a. The devices were monitored by an HP4194A impedance analyzer in Gain-Phase mode set to sweep a number of frequency ranges. The probes were submerged in solutions of glycerin and DI water. The solutions were each of known concentrations, ranging from pure water to pure glycerin. Five different solutions were tested, including 0, 25, 50, 75, and 100% concentrations. Each solution was placed in the setup shown in Figure 3a and the probes were connected to the impedance analyzer. The impedance analyzer was set such that the actuator probe's input is a $1V_{rms}$ sinusoidal input with frequencies in the range of 20 to 40MHz. The sensor probe's output is read by the impedance analyzer and a ratio of output/input signal, or gain, is recorded at each frequency. Along with gain, the phase shift between the actuator and sensor signal is also recorded. Other parameters used in the gain-phase setup included single input mode and $1M\Omega$ input impedance for input and output ports.

The distance between the probes was 4mm and approximately 4mm of the probes were submerged in the liquid concentrations. The container was 37mm tall with a bottom diameter of 24mm. All samples were 10ml of liquid concentration. Details of this setup are shown in Figure 3b.

In the case of gain, Figure 4a, the frequency of maximum gain increases as the concentrations of liquid contain more glycerin, and therefore become more viscous. The magnitude of the gain seems to mostly follow these pattern except for the case of the 25% glycerin solution. Note that the magnitude of the gain is high due to this specifically high impedance setup used on the HP4194A.

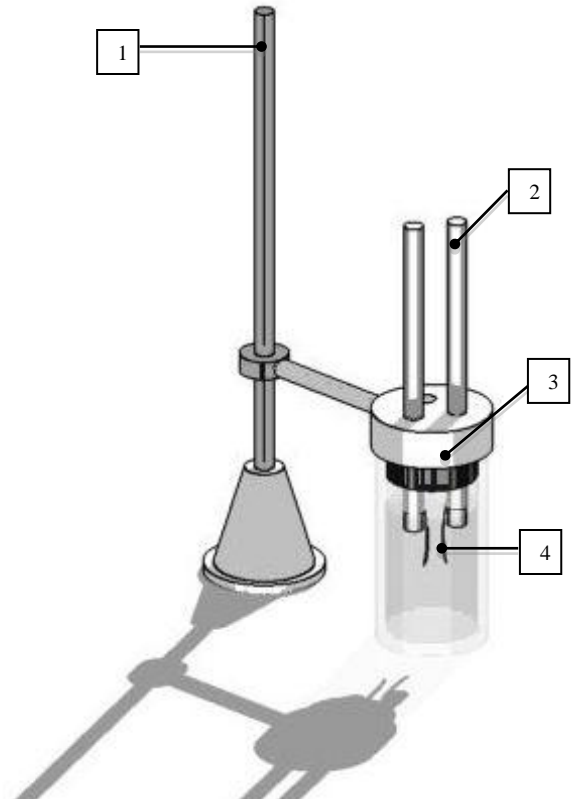


Figure 3(a). Measurement setup using probes and glycerin concentrations: (1) stand, (2) probe holder, (3) probe, (4) sample container.

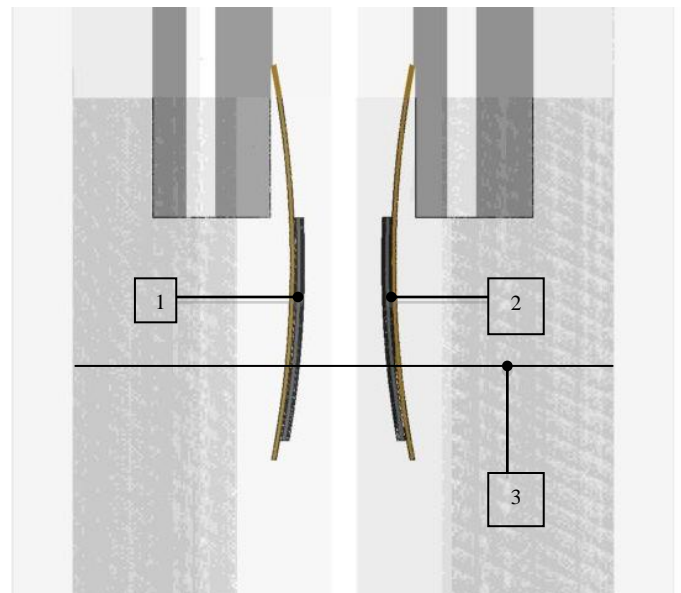


Figure 3(b). Detailed schematic of the probe Setup: (1) actuator probe, (2) sensor probe, (3) fluid level.

RESULTS AND DISCUSSIONS

The first set of results for each concentration was narrowed down since the most notable changes occurred in the range of 25MHz to 35MHz. The gain and phase plots are displayed in Figures 4a and 4b respectively. The legend on both figures indicate percent glycerin for the different concentrations.

The same measurements using an oscilloscope would be much lower due to the low impedance of the input devices. For the phase angle, as shown in Figure 4b, the changes in phase angle are more significant and are clearly shifting with concentration levels. Though the whole frequency sweep is not shown here, this phase shift observed is the highest in the range displayed on the graph. In order to more clearly identify these frequencies for the phase peaks, the first derivative of phase was taken with respect to frequency, and is displayed in Figure 4c. A clear shift with concentration is observed then. This shift however is not obviously proportional.

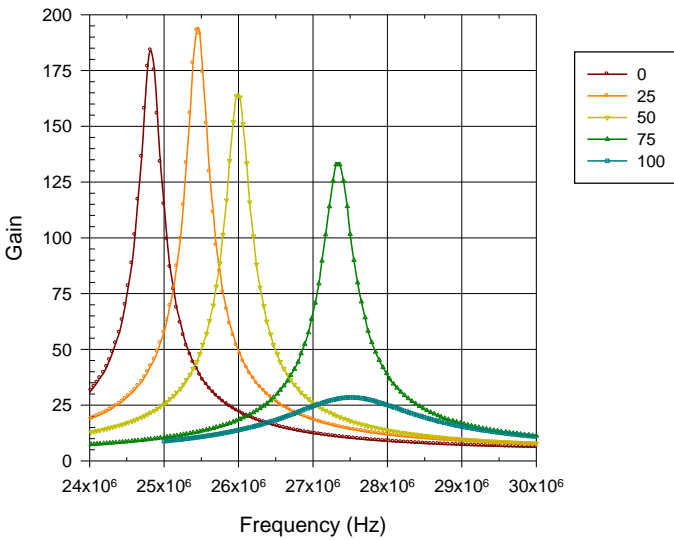


Figure 4(a). Gain versus frequency for different concentrations of glycerin and DI water.

To better illustrate the pattern of where these phase shifts occur and how that relates to the solution concentration, frequency of peak phase shift is plotted versus percent glycerin in Figure 5. As viscosity and glycerin concentration are not linearly related (viscosity increases exponentially with glycerin percentage [14]), viscosity is plotted against frequency of phase shift to better illustrate the correlation, as shown in Figure 6. In addition to using the corresponding published values of dynamic viscosity for glycerin solutions [14], a 25% glycerin concentration was tested using a CANNON-FENSKE viscometer size 50 rated from 0.8 to 4 cP. The published value and the measured values were extremely close, differing by only 6%. In particular, Table 1 shows the numerical values of dynamic viscosity for the different concentrations. In addition,

the frequency at which the peak phase shift occurred is also shown.

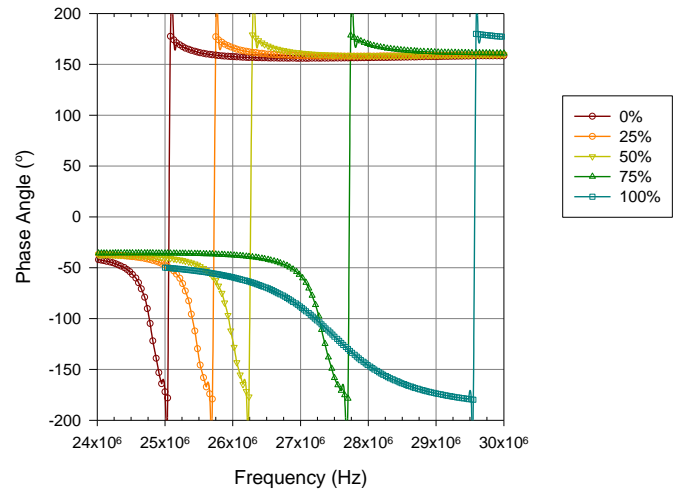


Figure 4(b). Phase angle versus frequency for different concentrations of glycerin and DI water.

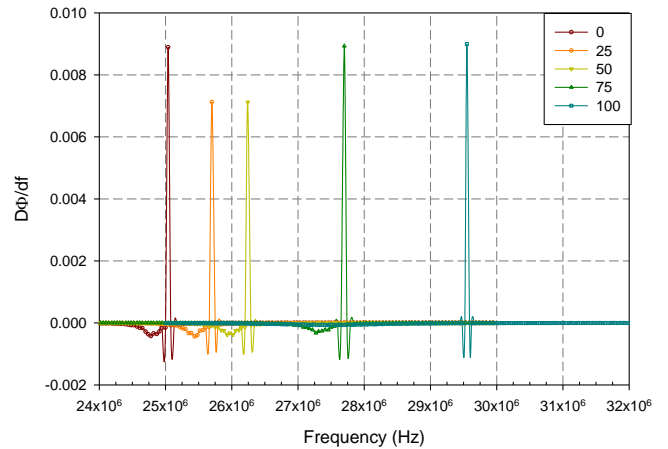


Figure 4(c). First derivative of phase with respect to frequency.

A regression was applied to the data and is also displayed in Figure 6. The nonlinear regression is of the form shown in Equation 3. The results of the regression show an R^2 of 0.99 and a P-value less than 0.0001 making the regression significant with a 95% confidence level as shown in Table 3.

$$\mu = \mu_0 \cdot e^{(b \cdot f_\phi)} \quad (3)$$

In the above equation, μ is the dynamic viscosity in cP at room temperature and μ_0 and b are constants with P-values less

than 0.005 and an overall regression coefficient of 99%. f_ϕ is the frequency of peak phase shift, in MHz. The details of the regression are shown in Table 2.

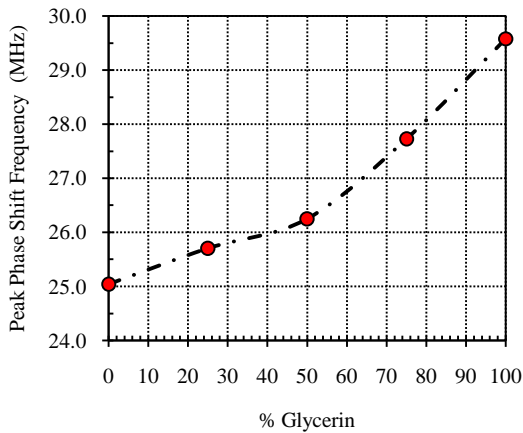


Figure 5. Frequency of maximum phase shift versus percent glycerin in solution.

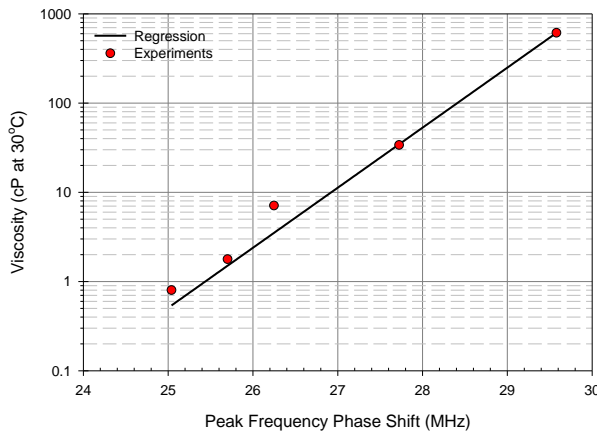


Figure 6 Viscosity versus peak phase shift.

Table 1. Dynamic viscosity and frequency for different glycerin concentrations.

Glycerin (%)	Frequency (MHz)	Viscosity (cP)
0	25.040	0.800
25	25.701	1.772*
50	26.248	7.100
75	27.725	33.900
100	29.579	612.00

*Validated through direct measurement

Table 2. Nonlinear regression for Viscosity and Phase Shift.

Nonlinear Regression					
Equation: Exponential Growth, Single, 2 Parameter					
$\mu = \mu_0 * \exp(b * f_\phi)$					
R	Rsqr	AdjRsqr	Std. Err. of Estimate		
1	1	0.9999	2.1223		
		Coeff.	Std. Err.	t	P
μ_0		7.65E-18	7.34E-18	1.042	0.374
b		1.5494	0.0325	47.745	<0.0001
Analysis of variance:					
		DF	SS	MS	
Regression		2	375733.9	187866.9	
Residual		3	13.5	4.5	
Total		5	375747.4	75149.5	
Corrected for the mean of the observations:					
		DF	SS	MS	F
Regression		1	289776.9	289776.9	64335.8
Residual		3	13.5	4.5	
Total		4	289790.4	72447.6	

CONCLUSIONS

Experiments were carried out using piezoelectric pre-stressed probes as an actuator-sensor pair to monitor changes in kinematic viscosity. A range of concentration values of glycerin and DI water were used to measure kinematic viscosity since their values are well known and published. Results indicate that in the MHz range, the frequency of peak phase shift angle changes can be directly correlated to changes in kinematic viscosity. The exponential correlation is a 99% fit with experimental data and the results are statistically significant values with 95% confidence levels. Future work includes testing of other solutions with different ranges of viscosity and developing correlations for changes of viscosity over

ACKNOWLEDGMENTS

Materials for preliminary testing were provided by Dr. Hiroshi Asanuma of Chiba University in Japan. Also, advice and consultation was provided Dr. Kevin Ward and Dr. Nathan White of the Medical College of Virginia, at Virginia Commonwealth University in Richmond, VA.

REFERENCES

- [1] Elert, Glen. *The Physics Hypertextbook*. "Viscosity" © 1998-2010.
<http://physics.info/viscosity>
- [2] A&D Weighing. "Measuring Viscosity." © 2004-2010.
<http://www.andweighing.com/and.nsf/html/Measuring+Viscosity>
- [3] Lin, Z., Yip, C.M., Joseph, I.S., and Ward, M.D., 1993, "Operation of an Ultrasensitive 30-MHz Quartz Crystal Microbalance in Liquids" *Anal. Chem.*, 65, pp. 1546-1551.
- [4] Kanazawa, K.K. and Gordon, J.G., 1985, "Frequency of a Quartz Microbalance in Contact with Liquid" *Anal. Chem.*, 57, pp. 1770-1771.
- [5] Hayward, G.L. and Chu, G.Z., 1994, "Simultaneous measurement of mass and viscosity using piezoelectric quartz crystals in liquid media" *Analytical Chimica Acta*, 288, pp. 179-185.
- [6] Saluja, A. and Kalonia, D.S., 2004, "Measurement of Fluid Viscosity at Microliter Volumes Using Quartz Impedance Analysis" *AAPS PharmSciTech*, 5(3), Article 47.
- [7] Ash, D.C., Joyce, M.J., Barnes, C., Booth, J.C. and Jefferies, A.C., 2003, "Viscosity measurement of industrial oils using the droplet quartz crystal microbalance" *Meas. Sci. Technol*, 14, pp. 1955-1962.
- [8] Muramatsu, H., Tamiya, E., Suzuki, M. and Kabure, I., 1988, "Viscosity Monitoring with a Piezoelectric Quartz Crystal and its Application to Determination of Endotoxin by Gelation of Limulus Amebocyte Lysate" *Analytical Chimica Acta*, 215, pp. 91-98.
- [9] Martin, S.J., Frye, G.C. and Wessendorf, K.O., 1994, "Sensing liquid properties with thickness-shear mode resonators" *Sensors and Actuators, A* 44, pp. 209-218.
- [10] Shih, W.Y., Li, X., Gu, H., Shih, W.H. and Aksay, I., 2001, "Simultaneous liquid viscosity and density determination with piezoelectric unimorph cantilevers" *Journal of Applied Physics*, 89(2), pp. 1497-1506.
- [11] Bergaud, C. and Nicu, L., 2000, "Viscosity measurements based on experimental investigations of composite cantilever beam eigen frequencies in viscous media" *Rev. Sci. Instrum.*, 71(6), pp. 2487-2492.
- [12] Agoston, A., Keplinger, F., and Jakoby, B., 2005, "Evaluation of a vibrating micromachined cantilever sensor for measuring the viscosity of complex organic liquids" *Sensors and Actuators, A* 123-124, pp. 82-86.
- [13] Yi, J.W., Shih, W.Y., Mutharasan, R. and Shih, W.H., 2003, "In situ cell detection using piezoelectric lead zirconate titanate-stainless steel cantilevers", *Journal of Applied Physics*, 93(1), pp. 619-626.
- [14] The Dow Chemical Company. "Viscosity of Aqueous Glycerin Solutions" © 1995-2010.
<http://www.dow.com/glycerine/resources/table18.htm>
- [15] Ferret. "SV-A Series Vibroviscometer." © 2009.
<http://www.ferret.com.au/c/A-D-Australasia-228302/SV-A-Series-Vibro-Viscometer-n849377>
- [16] Ningbo Hinotek Tehcnology Co., Ltd. "NDJ Series Rotational Viscometer." NDJ-4 Rotational Viscometer. © 2007 – 2009.
<http://www.hinotek.com/laboratory-Equipment/Viscometer/Rotational-Viscometer.html>
- [17] Society of Tribologists and Lubrication Engineers. "Basics of Rheology: Capillary Viscometers." © 2008.
<http://www.stle.org/resources/lubelearn/rheology/capillary>
- [18] Gardco (Paul N. Gardner Company, Inc.). "Brookfield Falling Ball Viscometer."
http://www.gardco.com/pages/viscosity/vi/bf_fallingball.cfm
- [19] Ningbo Hinotek Tehcnology Co., Ltd. "NDJ Series Rotational Viscometer." NDJ-5 Cup Type Viscometer. © 2007 – 2009.
<http://www.hinotek.com/laboratory-Equipment/Viscometer/Rotational-Viscometer.html>