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Quartz Crystal Microbalance for viscosity measurement with temperature self-compensation

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ABSTRACT

This paper presents the design, fabrication, and experimental demonstration of a portable viscosity measurement system using an AT-cut quartz crystal resonator with a temperature compensation technique. The proposed sensor is designed to measure viscosity from room temperature to 65 °C with complete temperature compensation. A novel fixture to hold the crystal for the viscosity sensing is designed using CAD (Computer Aided Design) software and a 3D model prototype was fabricated using a 3D printer. Subsequently, the fabricated fixture was integrated with a crystal resonator and tested with various mixtures of glycerol and water solutions each with different viscosities. A beat frequency based temperature measurement and compensation technique was also proposed and has been implemented in the crystal resonator based force measurement system in the literature. The measurement system is portable, simple, and low cost making it convenient for laboratory-based viscosity measurements. The proposed fixture of the measurement system can be altered for real-time monitoring to quantify the viscosity of the continuous flowing aqueous liquids.

1. Introduction

Viscosity is an important rheological property describing the physical behavior of a liquid. Once determined, it can be used to form conclusions about the condition of the examined liquid. Viscosity monitoring is widely used for the monitoring of a lubricant's quality and age; other applications are: characterizing polymers, pumping of waste slurries, and characterization of DNA solutions. Viscosity monitoring is applied in several industries such as food research, biochemistry, blood analysis, and paper technology. In laboratory-based viscosity measurements, there is a need for portable handheld viscometers that have a lower maintenance cost and require smaller amounts of sample liquid compared to conventional viscometers. During the last two decades, different viscometers were proposed; the viscometers can be distinguished by the mechanical structure [1,2], manufacturing methods, materials used, actuation, and readout principles [3,4]. One type of viscosity sensor is based on piezoelectric thin film resonators (FBAR) [5–8], however, the temperture stability of this type of sensor is not ideal. Another type of viscosity sensor is based on Quartz crystal resonator (QCR) (usually called QCM (Quartz Crystal Microbalance) when its mass sensitivity is applied), which normally operate in the thickness shear mode [9,10]; QCR sensors are widely used because of the high sensitivity, high accuracy, good stability, portability, low manufacturing cost, and reliability in measuring the deposited sample in either gaseous or liquid media [11–13]. In thickness shear mode (TSM), the QCR undergoes shear displacement upon excitation and resonates at specific frequencies. The QCR sensors have been widely studied for several decades by different investigators to obtain theoretical models that explain the behavior of the sensor. QCRs have also been used widely in many applications such as current, mass, density, humidity, chemical, and biological sensors [14–17].

The advantage of these QCR based sensors is that they operate at high frequencies in the range of MHz, which is typically a limit for other conventional viscometers. A novel continuous flow cell system was designed and fabricated to monitor viscosity continuously using an 11 MHz quartz crystal resonator [18]. QCR in liquid media is also limited to low viscosity measurements because the oscillations are significantly damped in high viscosity liquids. A research has been performed by utilizing an inexpensive sensor with a quartz crystal using an automatic gain control oscillator to measure the sample mass and viscosity simultaneously [19]. A new approach using QCRs to distinguish between mass loading and liquid damping was proposed and tested experimentally on a 10 MHz AT-cut quartz resonator with glycerol [20]. Another approach using a novel quartz crystal sensor for the measurement of the density-viscosity product for Newtonian liquids with two circular Plano-convex AT-cut quartz crystals [21] was demonstrated. A droplet quartz crystal microbalance to measure the viscosity of industrial oils is reported. In this approach, a small volume of test fluid is employed to investigate the frequency shift of the quartz resonator [22]. Similarly, a viscosity sensor using quartz resonator for online condition monitoring of lubricant

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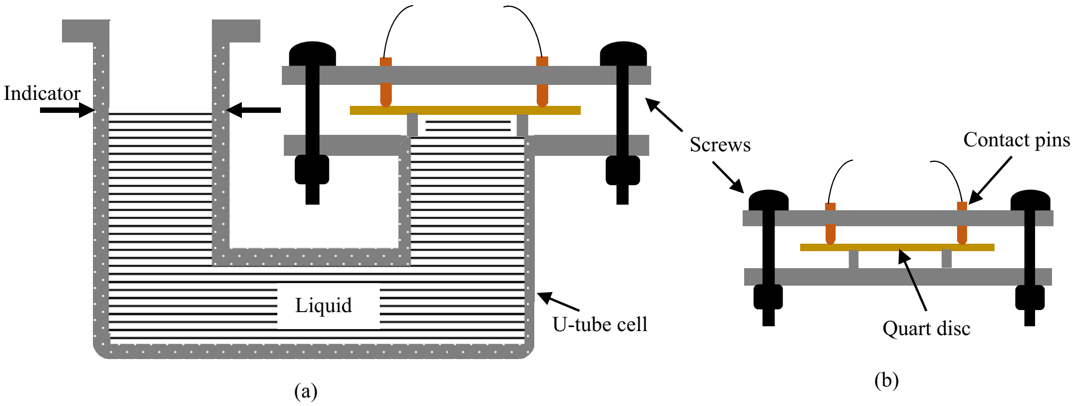


Fig. 1. Cross-sectional view of the (a) measurement system (b) fixture for the crystal.

oils has been proposed and examined [23]. The drawback of this sensor is that measurement cannot be performed at high temperatures since the crystal resonator is dependent on temperature. A new theoretical model and a method to measure density and viscosity separately with a single quartz crystal resonator was explored and experimentally verified [24]. Another similar work has been suggested for the field measurement of liquid viscosities using mass-sensitivity based method. A complete analytical mode describing the influence of the liquid properties on the oscillation frequency has also been demonstrated [25]. This method is not a standard method and has a severe effect as the temperature of the liquid rises. A detailed study to investigate viscous liquids at elevated temperatures was also examined and experimentally verified [26]. Although quartz works exceptionally well in most applications, it is not used at high temperatures since quartz loses piezoelectric properties above its Curie temperature. Temperature compensation of a quartz crystal resonator sensor has been applied to determine the charge level of lead-acid batteries, which takes into account the thermal variations of density and viscosity along with the thermal behavior of quartz resonator [27].

In this paper, a new laboratory-based measurement system to measure the viscosity of various liquids using commercially available quartz crystal resonator operating in the thickness shear mode was designed, fabricated, and tested. The sensor was also tested for viscosity at high temperatures, and a beat frequency method for temperature measurement and compensation was proposed and demonstrated through a series of experiments on the sensor which was not reported in the literature.

2. Sensor design and principle

The measurement system consists of a hollow U tube cell, a fixture, and an AT-cut quartz disc resonator as the main element for viscosity sensing as shown in Fig. 1(a). The U-shaped hollow cell is used to supply the sample liquid under test, and the fixture consists of a top frame with two metallic brass pins that provide electrical contacts for the electrodes on the crystal and a bottom base as shown in Fig. 1(b). The U-tube cell is designed so that one side is larger than the other side restricting the sample liquid to overflow. The crystal is mounted on the shorter side of the U-tube cell and using two screws the fixture is adjusted to ensure a tight seal that prevents the liquid from contacting the top electrode of the crystal. Thus one side of the crystal is exposed to the measuring liquids and the other side is used for the electrical contacts. A quartz disc with diameter “d”, thickness “t”, and wrap around electrodes is chosen to meet to the sensor design requirement. Fig. 2(a) shows the bottom electrode on the rear side of the quartz disc, and Fig. 2(b) shows a wrap-around extended electrode from the bottom electrode to the front side, and Fig. 2(c) shows the top electrode of the quartz disc. This configuration allows both electrical contacts to be made on the front side of the quartz disc allowing the rear side to be exposed to measuring liquids.

The quartz disc will vibrate at its natural thickness shear mode frequency when not exposed to the testing liquid. When a liquid of viscosity ‘v’ is filled in the U tube cell to the indicator level, the liquid comes in contact with the rear side of the crystal as shown in Fig. 1(a). The contact with the liquid surface results in damping that alters the natural thickness shear mode frequency of the quartz disc. Beat frequency technique is also used for temperature measurement and as a procedure for temperature compensation [28,29]. The beat frequency is the difference between three times the first and third mode frequency; this difference is used to measure the temperature. The beat frequency is independent of viscosity load and a linear function of temperature, thus the beat frequency is used to measure the temperature and either the first and third mode frequency is used to measure the viscosity.

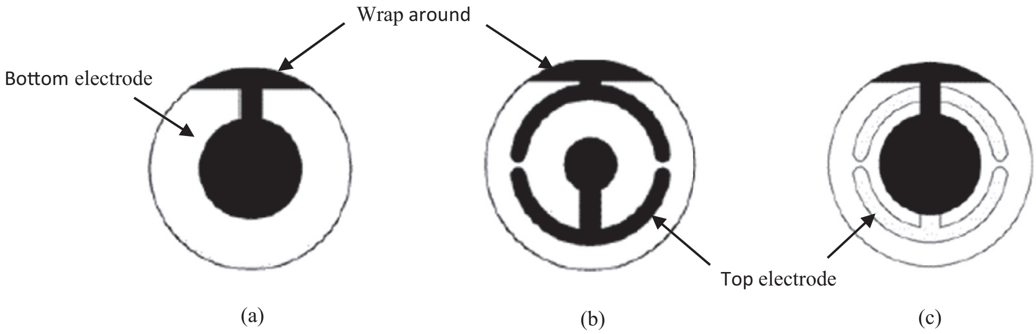


Fig. 2. Quartz crystal showing (a) rear view (b) front view (c) both electrodes.

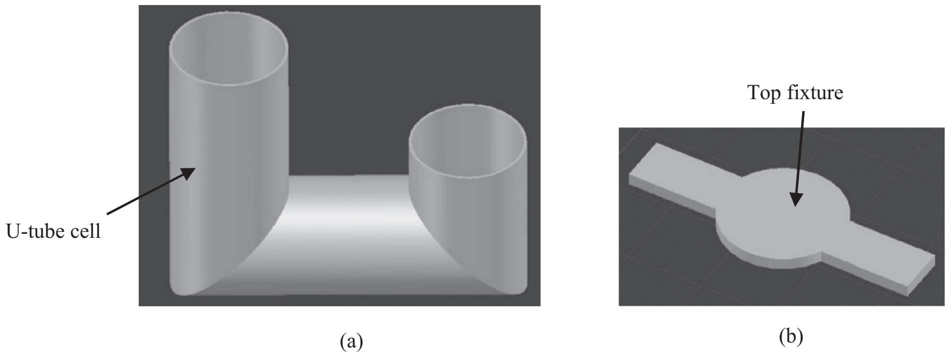


Fig. 3. CAD model (a) U-tube cell (b) fixture.

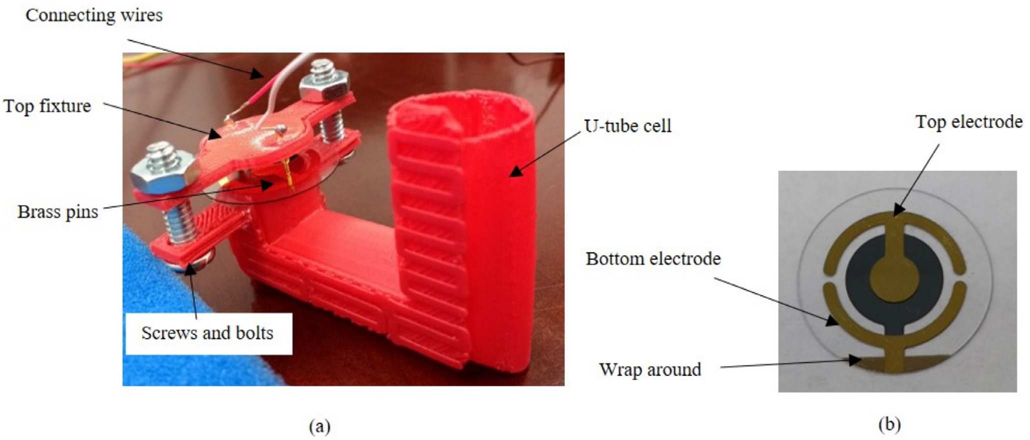


Fig. 4. (a) 3D printed model of the measurement system with complete assembly (b) AT-cut Quartz disc.

Table 1
Dimensions of the hollow U-Tube cell.

Parameter	Value
Length	5 cm
Height of longer side	4 cm
Height of shorter side	3 cm
Hollow hole diameter	1 cm
Diameter of the top fixture	2 cm

3. Fabrication and testing

The measurement system design is shown in Fig. 1(a) was designed in AutoCAD. The diameter of the U-tube cell openings on both sides is fixed based on the quartz disc dimensions. The lengths of the U-tube cell are adjusted so that 30 ml of liquid is sufficient to fill the U-tube cell completely and its dimensions are shown in Table 1. The CAD model of the U-tube cell and the fixture are shown in Fig. 3. A 3D model of the design was fabricated using a 3D printer with ABS plastic and assembled with the quartz crystal disc as shown in Fig. 4(a). A standard AT-cut 5 MHz quartz disc with a 25.4 mm diameter and 0.33 mm thick wrap around gold-coated electrodes was purchased from INFICON, which are generally used in liquid applications as shown in Fig. 4(b). Two brass spring pins are glued to the top fixture to ensure that they were in contact with both the electrodes on the quartz disc after assembly.

The solutions to be tested are prepared in the laboratory by mixing water and glycerol solutions in an appropriate ratio at room temperature to obtain viscosities ranging from 5 to 30 mPa.sec. Due to the hygroscopic nature of glycerol, the solutions were prepared quickly and tested immediately. The prepared solutions with different viscosity are shown in Fig. 5.

Under the initial no-load conditions (air) the sensor is assembled as shown in Fig. 4(a) and the first and the third mode frequency of the quartz disc are tracked using a network analyzer (KEYSIGHT) manually by measuring the amplitude of the mode frequencies. The frequency spectrum obtained from network analyzer is shown in Fig. 6. The first

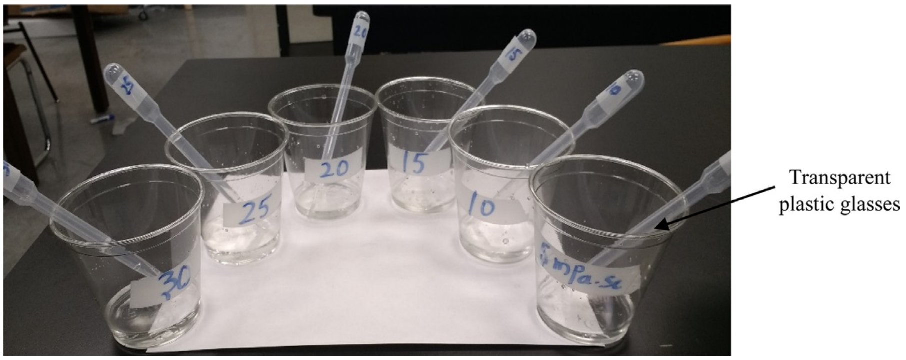
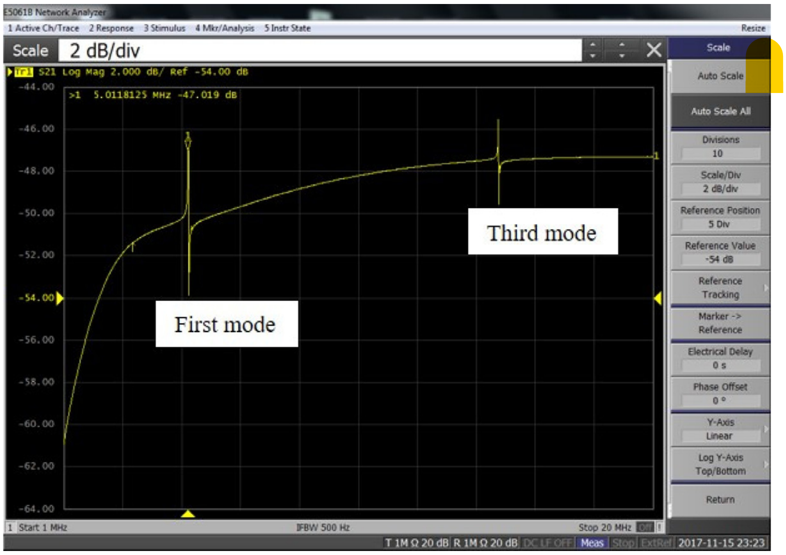


Fig. 5. Test solutions showing different viscosity.



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Tr1 S21 Log Mag 2.000 dB/ Ref -54.00 dB
>1  5.0118125 MHz -47.019 dB
1  Start 1 MHz        IFBW 500 Hz        Stop 20 MHz
T 1M Ω 20 dB  R 1M Ω 20 dB  DC LF OFF  Meas  Stop  ExtRef  2017-11-15 23:23
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Scale 2 dB/div

Divisions 10

Scale/Div 2 dB/div

Reference Position 5 Div

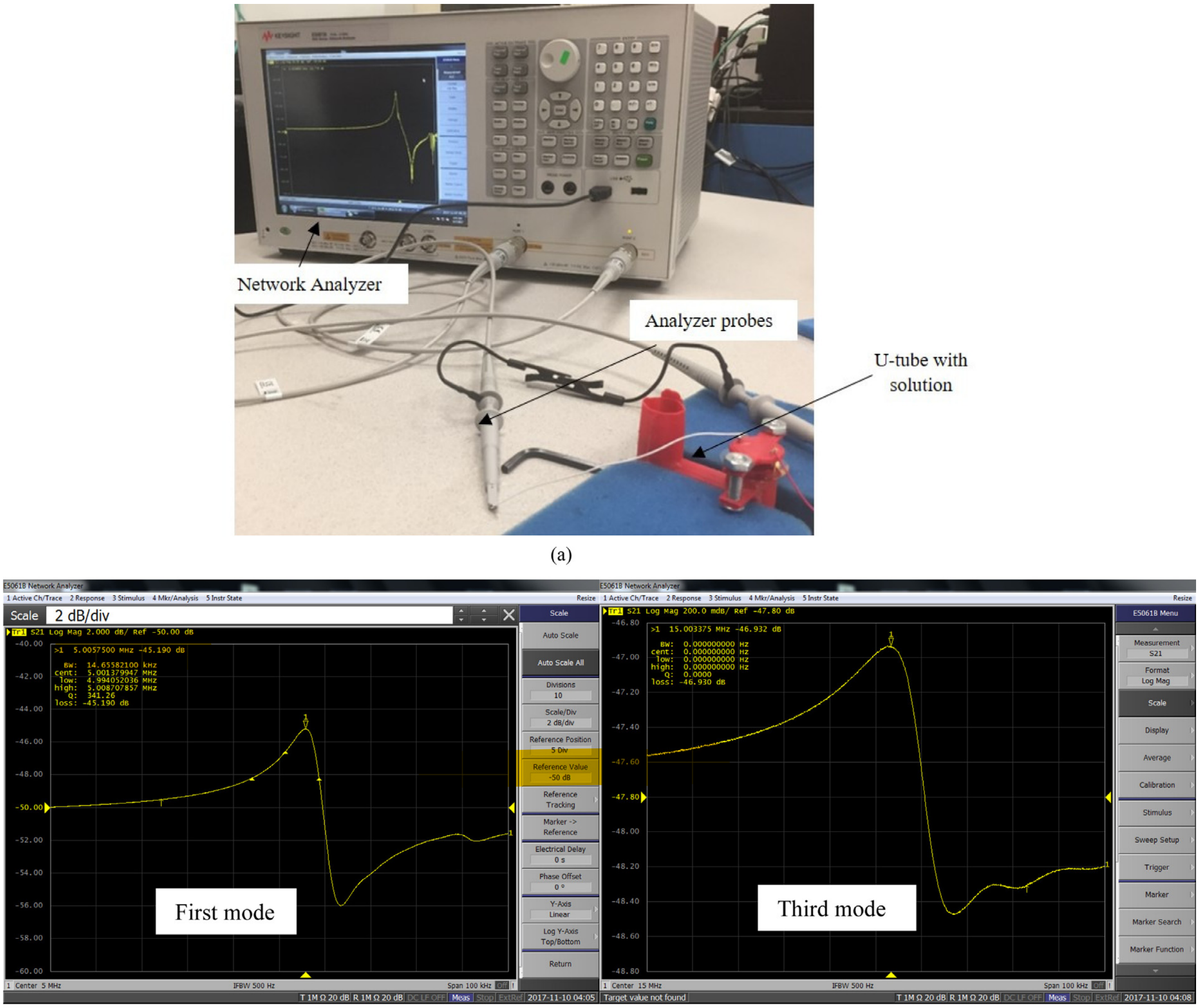
Reference Value -54 dB

Electrical Delay 0 s

Phase Offset 0 °

Y-Axis Linear

Fig. 6. Frequency spectrum showing two modes under no load.



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Tr1 S21 Log Mag 2.000 dB/ Ref -50.00 dB
>1  5.0057500 MHz -45.190 dB

  BW:  14.65582100 kHz
cent:  5.001379947 MHz
 low:  4.994052036 MHz
high:  5.008707857 MHz
   Q:  341.26
loss: -45.190 dB
1  Center 5 MHz        IFBW 500 Hz        Span 100 kHz
T 1M Ω 20 dB  R 1M Ω 20 dB  DC LF OFF  Meas  Stop  ExtRef  2017-11-10 04:05
```

Scale 2 dB/div

Divisions 10

Scale/Div 2 dB/div

Reference Position 5 Div

Reference Value -50 dB

Electrical Delay 0 s

Phase Offset 0 °

Y-Axis Linear

```
Tr1 S21 Log Mag 200.0 mdB/ Ref -47.80 dB
>1  15.003375 MHz -46.932 dB

  BW:  0.000000000 Hz
cent:  0.000000000 Hz
 low:  0.000000000 Hz
high:  0.000000000 Hz
   Q:  0.0000
loss: -46.930 dB
1  Center 15 MHz        IFBW 500 Hz        Span 100 kHz
Target value not found
T 1M Ω 20 dB  R 1M Ω 20 dB  DC LF OFF  Meas  Stop  ExtRef  2017-11-10 04:08
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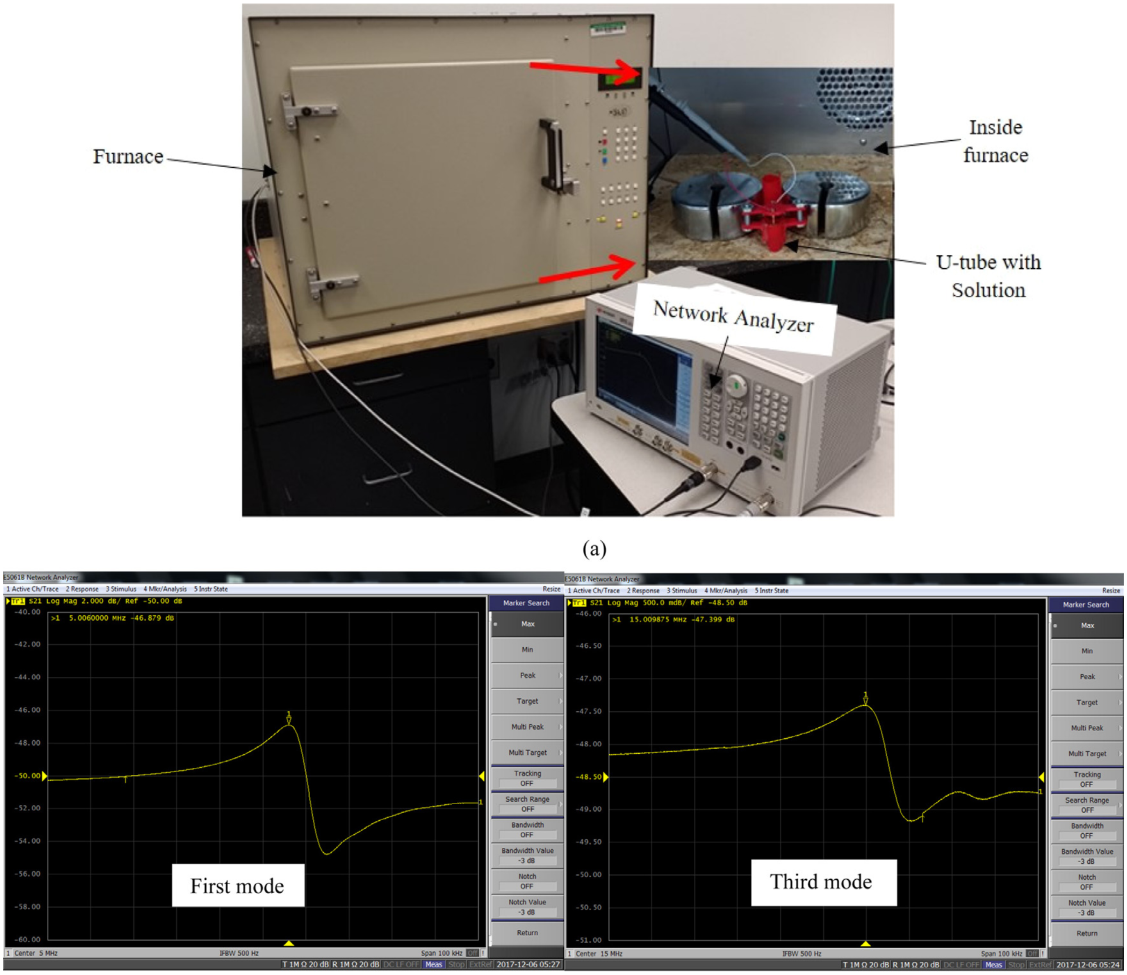
Measurement S21

Format Log Mag

(a)

(b)

Fig. 7. (a) Experimental set up at room temperature (b) Frequency spectrum showing two modes with load at room temperature.



(a)

(b)

Fig. 8. (a) Experimental set up for high-temperature viscosity measurement (b) Frequency spectrum showing two modes with load at high temperature.

and third mode frequency was found to be 5 MHz and 15 MHz respectively. The sensors were tested at room temperature and with the load conditions applied. This test was run multiple times with the various different sample solutions. The photograph of the experimental setup and the frequency spectrum of both first and third mode frequency is shown in Fig. 7.

The viscosity of the liquid decreases as temperature increases; to measure the viscosity at high-temperature separate solutions were prepared to have viscosities of 80, 100, and 120 mPa.sec by mixing appropriate solutions of water and glycerol. The viscosity variation of the prepared sample solutions with temperature is recorded in Table 2. The measurement assembly with the prepared solutions was placed inside a temperature furnace (SUN SYSTEMS EC 16HA) that is set to the required temperature of 65 °C. The photograph of the experimental set up is shown in Fig. 8(a). A network analyzer (KEYSIGHT E5061B) was once again used to track both the first and third mode frequencies of

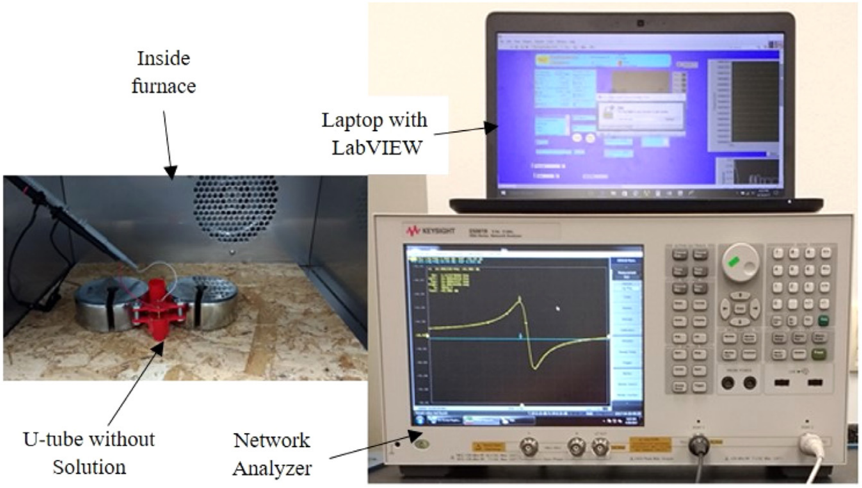
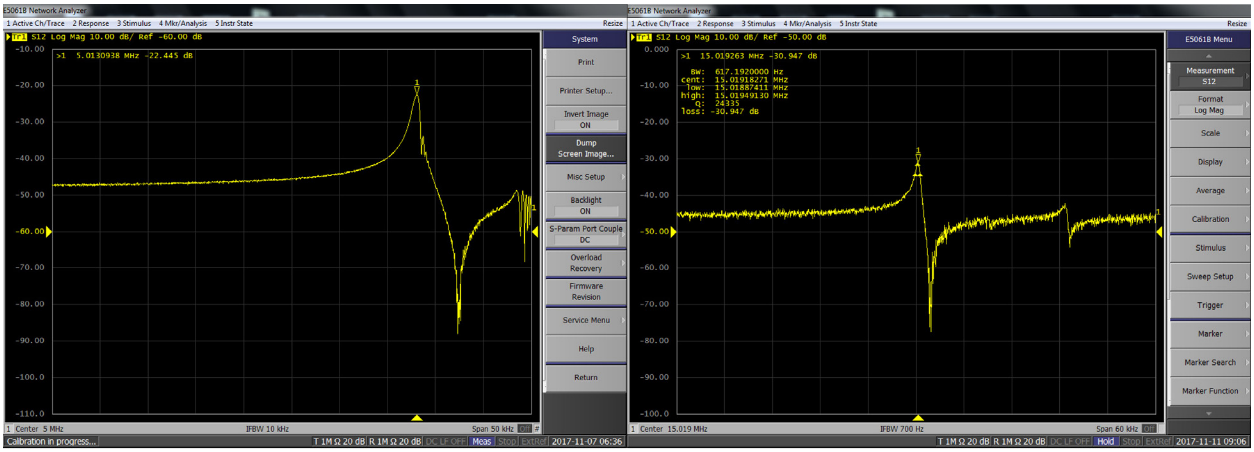


Fig. 9. Experimental set up for temperature-frequency characteristics.



(a) (b)

Fig. 10. Frequency tracking showing (a) first mode and (b) third mode frequency.

Table 2
Temperature vs viscosity data of the sample solutions.

Temperature (°C)	Viscosity (mPa.sec)		
25	80	100	120
30	59.4	73.3	89.4
35	44.8	54.8	66.2
40	34.5	41.8	50
45	27	32.4	38.5
50	21.5	25.6	30
55	17.3	20.5	24
60	14.2	16.7	19.4
65	11.8	13.7	15.8

the quartz disc manually by observing the resonance peak as shown in Fig. 8(b).

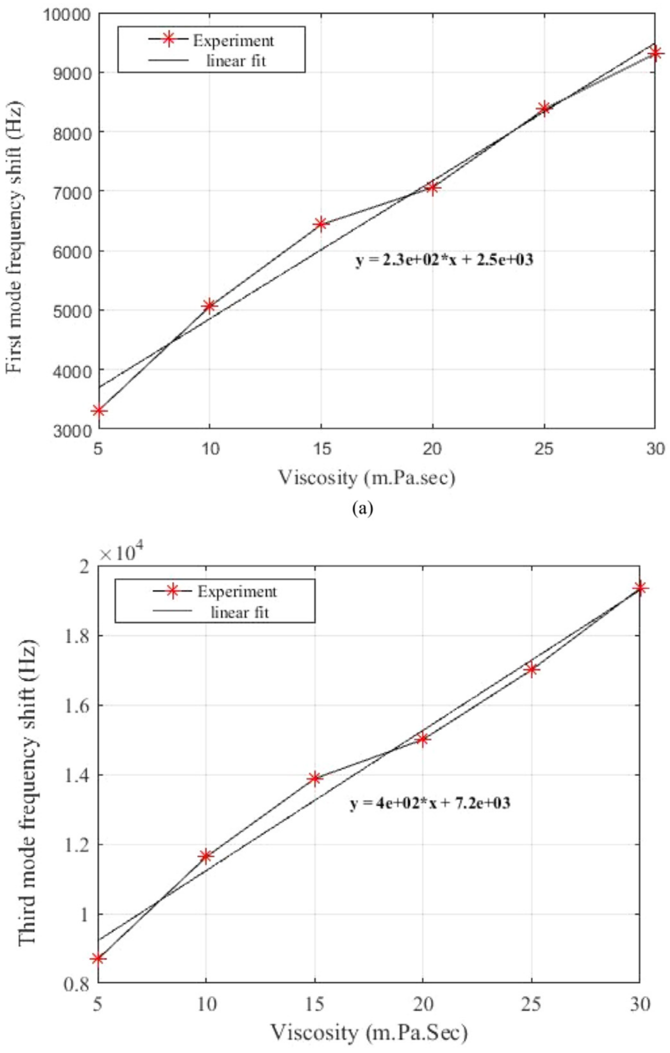
For obtaining the temperature-frequency characteristics, the measurement set up was assembled without any solution and enclosed in a furnace system to study the frequency variation of the quartz disc in relation to temperature. Both the network analyzer and temperature furnace system were interfaced to a computer with LabVIEW software through a GPIB connector and the experimental set up is shown in Fig. 9. The network analyzer was programmed in the software to filter the crystal's first and third mode frequencies individually from the frequency spectrum by specifying the central frequencies and the span bandwidth. Initially, under no liquid load, the frequency-temperature analysis was carried out from an operating temperature of 25 °C to 65 °C for both the first and third mode frequency of the quartz crystal disc. The frequency tracking of both the first and third mode frequency in network analyzer is shown in Fig. 10. Theoretically fractional changes in the resonant frequency of a thickness-shear mode based resonator when subject to static temperature change and in-plane forces can be represented by the following equation [30]:

$$\frac{\Delta \mathrm{f}}{\mathrm{f}_0} = \mathrm{A}(\Delta \mathrm{T}) + \frac{B(\Delta T)^2}{2!} + \frac{C(\Delta T)^3}{3!} + \ldots D(\Delta P)$$

where higher-order terms can be ignored, and where “D” is the stress coefficient of frequency, and ΔP is the resonator stress. The coefficients “A”, “B” and “C” are, respectively, the first, second and third-order temperature coefficients of frequency and ΔT is the temperature difference.

4. Results and discussion

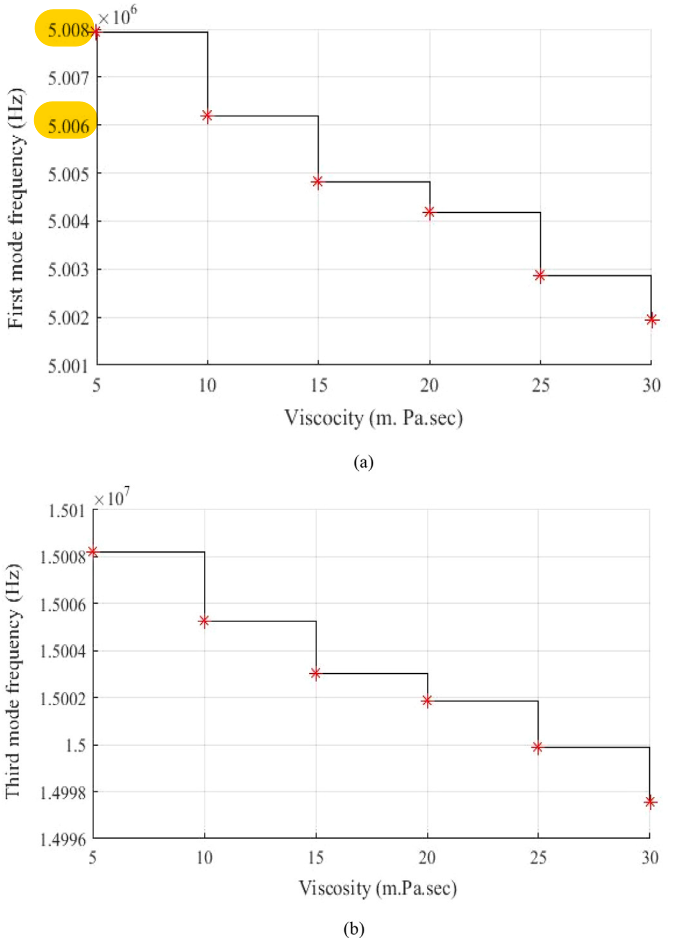
The fabricated and assembled sensor is tested with sample solutions of different viscosities using the experimental setup in Fig. 7(a) and the



(a)

(b)

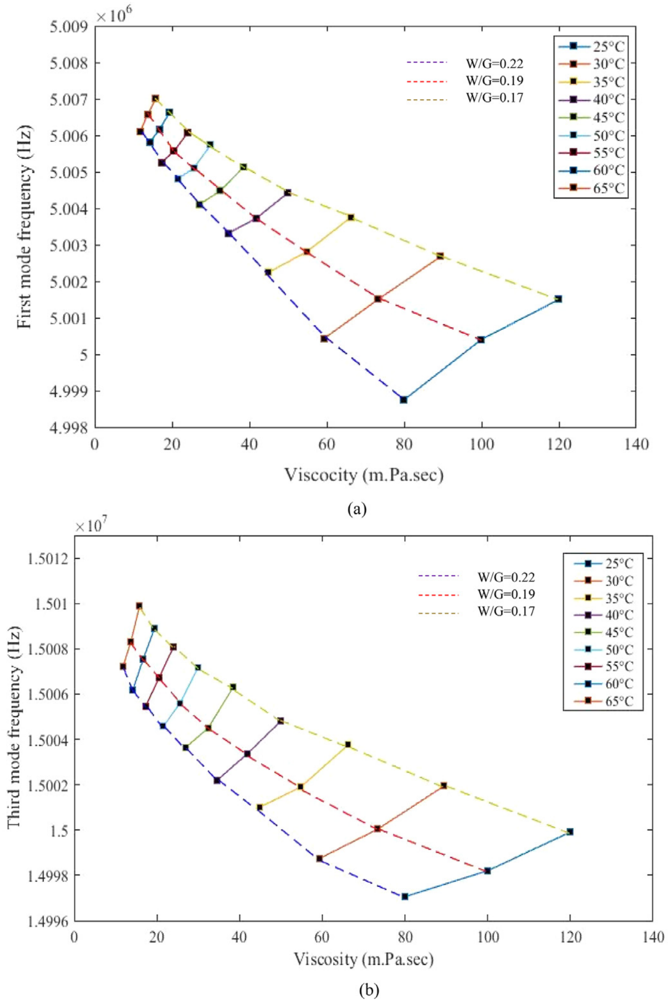
Fig. 11. Frequency shift with viscosity (a) first mode and (b) third mode.



(a)

(b)

Fig. 12. Frequency variation with viscosity (a) first mode and (b) a third mode.



(a)

(b)

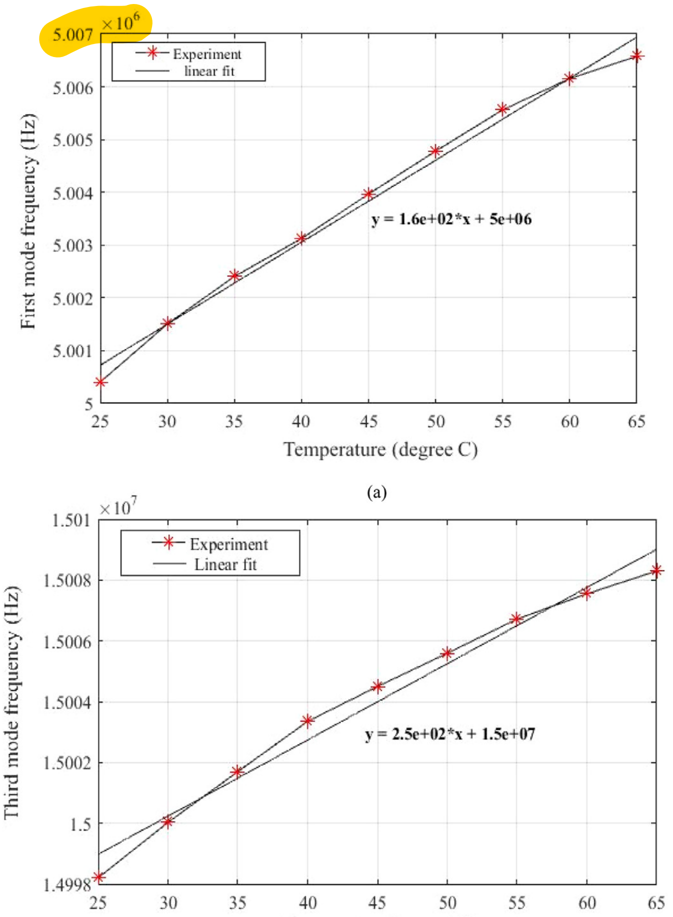
Fig. 13. Frequency variation with the viscosity at different temperatures (a) first mode and (b) third mode.

frequency shift of both the first and third mode at room temperature is plotted as shown in Fig. 11. The frequency shift is calculated by taking the difference between without load (air) and with a load for each solution separately. The below graph shows a linear frequency shift in both modes and increases as the viscosity increases. It shows that as the viscosity increases, the damping induced by the solution to the quartz disc resonator increases and results in an increase in the frequency shift. Fig. 12 shows the decreasing trend in both the frequency modes with viscosity. The steps in Fig. 12 indicate that the viscosity measurement is not continuous and the data is obtained by testing all the sample solutions one after the other. As the viscosity increases the frequency decreases gradually in both modes and it is also observed that the frequency shift is 6 KHz for the first mode and 10.5 KHz for the third mode with a viscosity variation from 5 to 30 mPa.sec respectively.

For high-temperature viscosity measurement and compensation using the beat frequency technique the assembled sensor is tested with sample solutions, each having viscosities 80, 100, and 120 mPa.sec respectively by taking different water to glycerol (W/G = 0.22, 0.19, and 0.17) ratios with the experimental setup described in Fig. 8. The frequency variation in both the first and third mode with a viscosity at different temperatures (from room temperature to 65 °C) were obtained and plotted as shown in Fig. 13. As the temperature increases, the viscosity of the solutions gradually decreases, thereby reducing the damping on the quartz disc resonator resulting in an increase of the frequency in both modes. To show the variation of frequency with temperature, separate graphs are plotted to show the change in both modes as the temperature increases from room temperature to 65 °C using the solution with viscosity 100 mPa.sec (W/G = 0.19) as shown in Fig. 14(a) and (b).

Fig. 14 gives the variation in the first mode and third mode frequency of the crystal resonator with an increase in temperature under viscosity load. For these two graphs, the static characteristic linearity was calculated in terms of Non-linearity as a percentage of span within the temperature range of 25–65 °C from the experimental data. The Non-linearity percentage for the first mode frequency (55%) was found to less as compared to the third mode frequency (73%) obtained from the linear curve fitting. So we can conclude that, the first mode frequency variation with temperature is slightly more linear than the third mode frequency. As the temperature increases the quartz disc will also contribute some frequency variation in addition to the viscosity effect. The temperature measurement and compensation can be done simultaneously by using the beat frequency technique. Thus the beat frequency is calculated offline from the experimental data and plotted for all the three solutions with viscosities 80,100 and 120 mPa.sec as shown in Fig. 15. The graph shows a linear variation with temperature up to 65 °C for all the solutions and can be used to measure temperature. Once the temperature is estimated, temperature compensation can be performed using the temperature frequency characteristic of the sensor under no load conditions.

The temperature-frequency characteristics of the sensor (quartz disc resonator) without load is obtained from the experimental setup in Fig. 9 is plotted as shown in Fig. 16. The graph shows a variation of



(a)

(b)

Fig. 14. Frequency variation with temperature under viscosity load (a) first mode and (b) third mode.

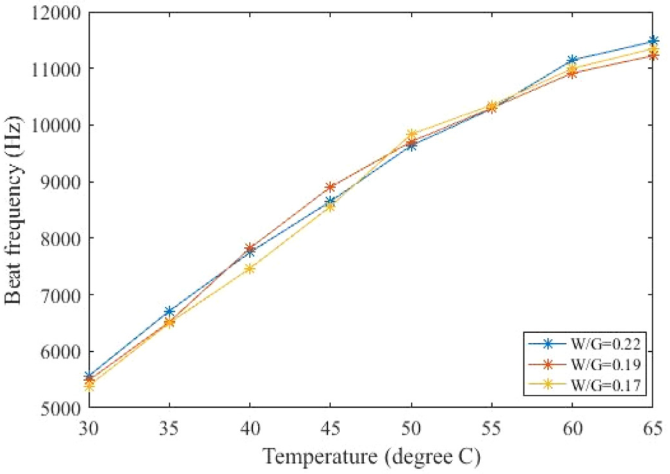
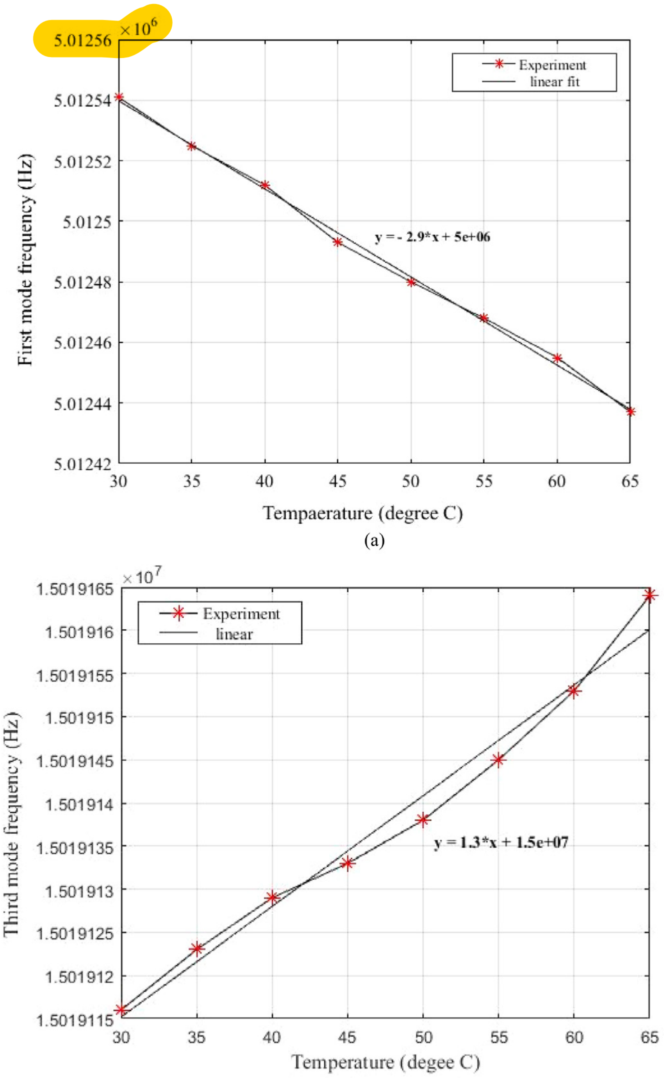


Fig. 15. Beat frequency with temperature for all the three solutions.



(a)

(b)

Fig. 16. Temperature frequency characteristics of (a) first mode and (b) third mode.

−3 Hz/°C in first mode frequency and 1.5 Hz/°C in third mode frequency with an increase in temperature. The frequency variation obtained is purely due to temperature effect due to the internal heating of the quartz disc resonator. It is also observed that the temperature-frequency characteristics are almost linear for the first mode, but not for the third mode. Hence for temperature compensation, this frequency variation with temperature must be subtracted from the results obtained in Fig. 14(a) and the resultant graph of the first mode frequency with the solution having viscosity 100 mPa.sec is shown in Fig. 17. A similar temperature compensation technique using the beat frequency method was experimentally demonstrated by the authors in their previous work [28]. The beat frequency alone is used to measure the temperature and either the first mode or third mode frequency is used to estimate the viscosity. The temperature measurement is limited to 65 °C due to the material used in the fabrication of the measurement set up and this temperature range can be increased further by fabricating the U-tube cell with glass and by using Langasite as a disc resonator instead of quartz. In the case of Langasite resonator we can use the dual mode SBTC cut for simultaneous temperature measurement and compensation instead of beat frequency method [31,32].

The variation in the first mode frequency with viscosity for the three solutions (W/G = 0.22, 0.19, 0.17) with temperature compensation is shown in Fig. 17. The compensated and uncompensated lines are overlapping on one another for each sample solution since the frequency shift of the quartz resonator with temperature alone is very small as compared to the frequency shift due to viscosity variation with temperature. This variation will be visible if we conduct the experiment at very high temperatures.

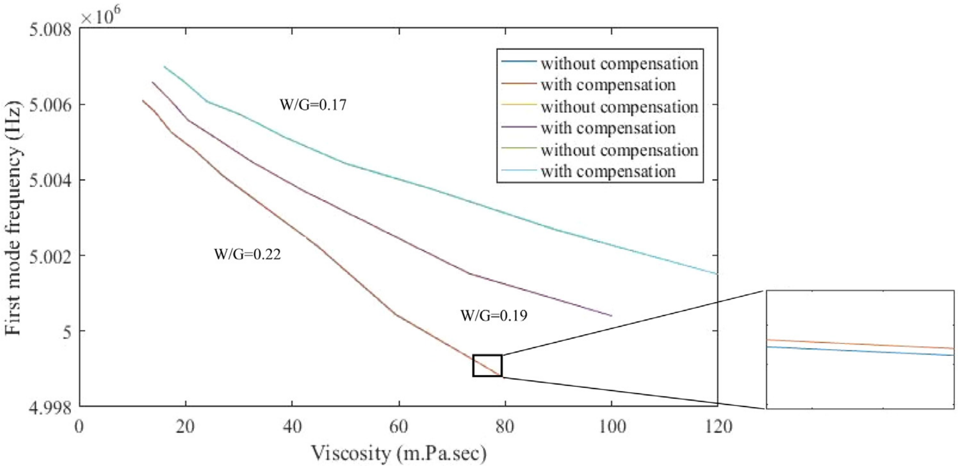


Fig. 17. Temperature compensation under viscosity load for the first mode frequency.

5. Conclusion

A portable viscosity measurement system using an AT-cut quartz crystal resonator was proposed to measure viscosity from room temperature to 65 °C with complete temperature compensation. Different solutions of water and glycerol were mixed to obtain solutions of different viscosity and tested experimentally. The experimental results show a good variation in the first mode frequency of the resonator for solutions with low viscosity. Beat frequency technique is implemented offline to show the temperature measurement and compensation for solutions having viscosity 80, 100, and 120 mPa.sec. The entire measurement system design is portable, simple, and low cost making it applicable for laboratory-based viscosity measurements. The proposed measurement system can be fabricated with glass for high-temperature viscosity measurements. The temperature range can be still further increased by replacing quartz with High temperature langasite resonators.

Conflict of intrest

None.

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