A Lateral-Field-Excited $LiTaO_3$ High-Frequency Bulk Acoustic Wave Sensor

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***Abstract*—The most popular bulk acoustic wave (BAW) sensor is the quartz crystal microbalance (QCM), which has electrodes on both the top and bottom surfaces of an AT-cut quartz wafer. In the QCM, the exciting electric field is primarily perpendicular to the crystal surface, resulting in a thickness field excitation (TFE) of a resonant temperature compensated transverse shear mode (TSM). The TSM, however, can also be excited by lateral field excitation (LFE) in which electrodes are placed on one side of the wafer leaving a bare sensing surface exposed directly to a liquid or a chemi/bio selective layer allowing the detection of both mechanical and electrical property changes caused by a target analyte. The use of LFE sensors has motivated an investigation to identify other piezoelectric crystal orientations that can support temperature-compensated TSMs and operate efficiently at high frequencies resulting in increased sensitivity. In this work, theoretical search and experimental measurements are performed to identify the existence of high-frequency temperature-compensated TSMs in $LiTaO_3$. Prototype LFE $LiTaO_3$ sensors were fabricated and found to operate at frequencies in excess of 1 GHz and sensitively detect viscosity, conductivity, and dielectric constant changes in liquids.**

I. Introduction

Bulk acoustic wave (BAW) crystal plate resonators may be classified according to their style of excitation. The "thickness-field-excited" (TFE) resonator [Fig. 1(a)] has electrodes on both plate faces. The electric field that is induced by the electrodes in the TFE case is impressed across the thickness of the plate, collinear with the acoustic wave propagation direction. The lateral-field-excited (LFE) resonators, as shown in Fig. 1(b), have both electrodes on a single plate face, leaving the opposing plate face bare with the exciting electric field impressed mostly across the gap between the electrodes. With thickness field excitation, the choice of a particular crystal and plate orientation completely determines the electro-acoustical properties of the resonator, because it determines both the propagation direction and the excitation direction. Lateral field excitation allows the excitation direction to be chosen independently, which in turn allows a degree of control over the electromechanical coupling to the acoustic modes at a particular plate orientation [1]. This control may be used to optimize LFE resonators for different applications.

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The respective electrode configurations of TFE and LFE resonators also lead to differences in performance. Neglecting fringing effects, the electric field produced by the TFE resonator electrodes can not penetrate into the measurand or sensing layer due to the conducting electrode on the sensing surface, and thus, electrical property changes such as relative permittivity and conductivity can not be measured [2], [3]. However, in many sensor applications such as biological or chemical analyte detection, it may be desirable to maximize the electrical sensitivity to the environment. Efforts have been made to increase the fringing interactions in TFE resonators [4], [5], but the sensing surface in these devices still has an electrode. Recent reports have noted progress in the development of LFE sensors [6]–[8]. This work has been performed using the well-known AT-cut of α-quartz. The choice of the AT-cut of quartz has allowed direct comparison to TFE devices of the same material, particularly the quartz crystal microbalance (QCM) [9].

It may be possible, however, to enhance the performance of LFE sensors by using another piezoelectric platform. The purpose of the present work is to theoretically identify and experimentally investigate orientations in $LiTaO_3$ (LTO), a material known for its high electromechanical coupling [10]–[12]. In particular, factors that are desirable for an LFE-sensing platform are identified. A theoretical search was then undertaken to identify orientations suitable for an LTO–LFE-sensing platform. Using the theoretical results as a guide, the (YXwl) $-16.5°$ cut was selected for experimental testing. The frequency response, temperature characteristics, and liquid sensing properties of this cut were measured and compared with LFE and QCM sensors fabricated on AT-cut quartz.

II. LFE Sensor Platform Requirements

Four factors are important in the identification of other possible LFE sensing platforms. It is desirable for an LFE sensor platform to have a piezoelectric active transverse shear mode (TSM) at that orientation and, preferably, a pure TSM at that orientation; that is, an allowable piezoelectrically active acoustic mode for which the mechanical displacements normal to the plate face are zero. Although some effort has been made to include and exploit longitudinal displacements in sensor applications [13], such displacements generally degrade performance. Second, given the existence of a pure TSM, it is desirable for one to be able to selectively excite this mode with zero coupling

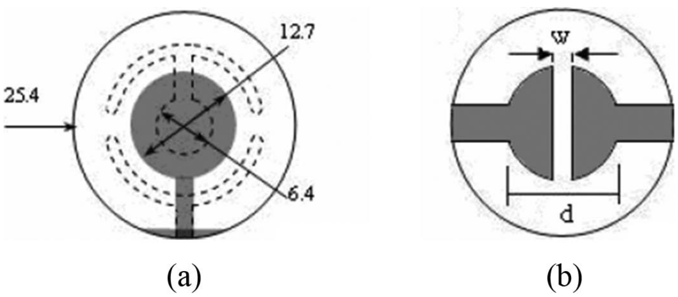


Fig. 1. (a) Top view (sensing surface) of a standard QCM sensor. Shaded (sensing surface) and dotted (bottom surface) regions are gold and all dimensions are in millimeters. (b) Bottom view (reference surface) of an LFE sensor. Shaded regions are gold. Typical values for the electrode gap width, w, are 1 mm and 13 mm for the electrode diameter, d.

to other modes. Lateral excitation has the advantage of permitting a degree of control over the mode coupling [1]. The challenge is then to identify those orientations that maximize the electromechanical coupling to a pure TSM mode decoupled from interfering modes. Third, the identified TSM should be temperature compensated so that the sensor's frequency response does not fluctuate with temperature changes. Fourth, it is desirable for the TSM to possess larger piezoelectric coupling than that observed for AT-cut quartz, the substrate that has been used in previous LFE work [6]–[8].

III. Search Results

A theoretical search was performed to identify possible orientations in LTO, a piezoelectric material that has been reported to have temperature-compensated bulk waves [14], that may be used as LFE-sensing platforms. Acoustic bulk wave velocities and their displacement data were computed for LTO using the extended Christoffel-Bechmann method [15]. The material constants and temperature coefficients used for LTO were taken from [14]. The loci of orientations with pure TSMs and the loci of orientations with zero temperature coefficient of frequency (TCF) at 25°C (room temperature) were calculated. For those orientations that lie at the intersections of these loci, namely, the temperature-compensated pure TSMs, the piezoelectric coupling for each mode was calculated. The coordinate systems detailed in the IEEE Standards on Piezoelectricity [16] as seen in Fig. 2 and Fig. 3 were used for the calculations.

In LTO 2 cuts, (YXwl) $-16.5°$ and (YXwl) $-84.3°$, offering significant improvements in coupling (39% and 47%, respectively) compared with AT-cut quartz (6.3%) were identified as possible LFE sensor platforms. As shown in Fig. 4, although the (YXwl) $-84.3°$ exhibits higher piezoelectric coupling than the (YXwl) $-16.5°$ cut, a slight variation in the direction of the electric field would also excite the slow quasi-shear mode, which has relatively high piezoelectric coupling, leading to interference. The (YXwl) $-16.5°$ cut has piezoelectric coupling (38.8%) more than

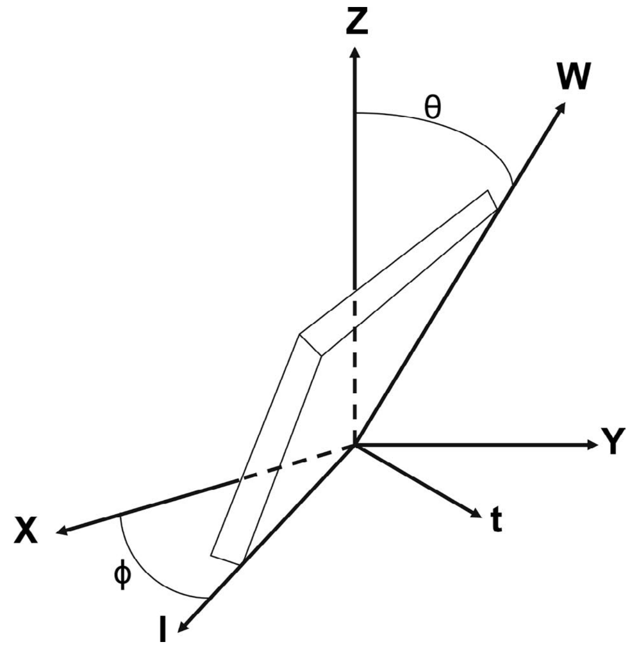


Fig. 2. IEEE Standard on Piezoelectricity [16] coordinate system used to calculate acoustic mode data.

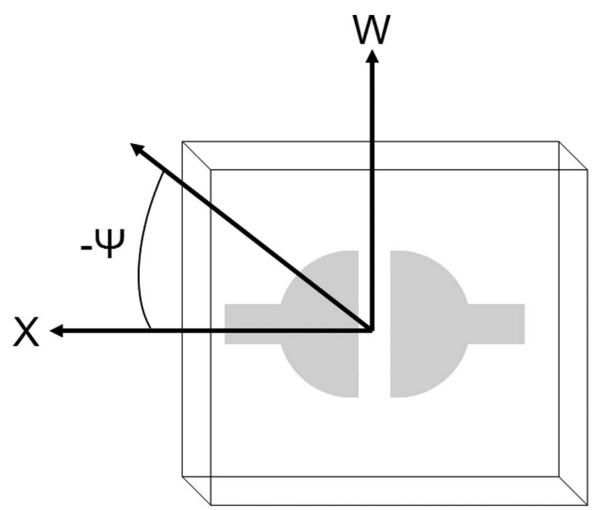


Fig. 3. LFE sensor notation system for electric field direction relative to the crystallographic axes.

6 times that of AT-cut quartz (6.3%). Also, the (YXwl) $-16.5°$ cut of LTO has been previously investigated for use in RF-filtering applications [17] and has theoretically been shown to be temperature compensated at 25°C. This cut of LTO was therefore selected for further testing as a possible LFE sensor platform.

IV. Experiment

A. X-ray Diffraction Measurements

LTO wafers were purchased from Sawyer Technical Materials, LLC (Eastlake, OH). X-ray diffraction (XRD)

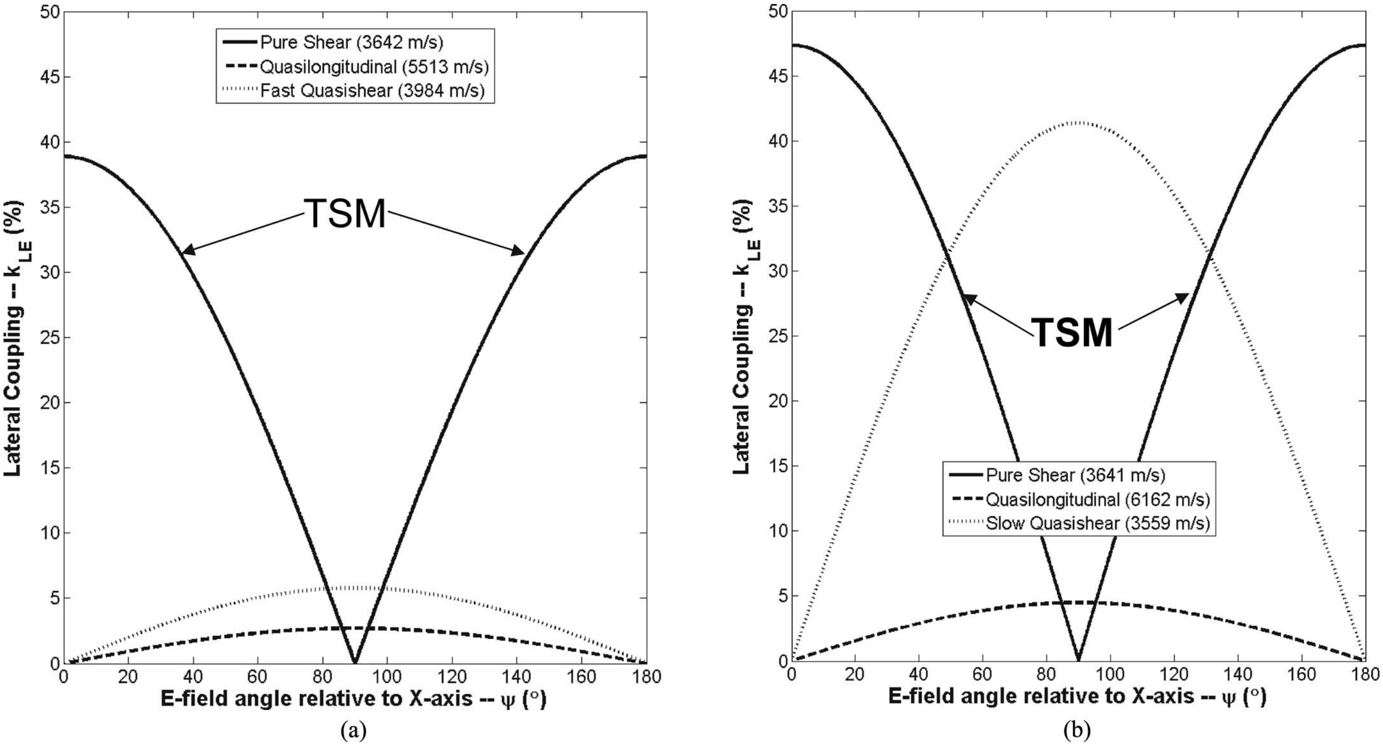


Fig. 4. Lateral field coupling coefficients k_{LE} for a) (YXwl) $-16.5°$ and b) (YXwl) $-83.4°$ rotate-Y cuts of lithium tantalate versus electric field angle ψ with regard to the x-axis.

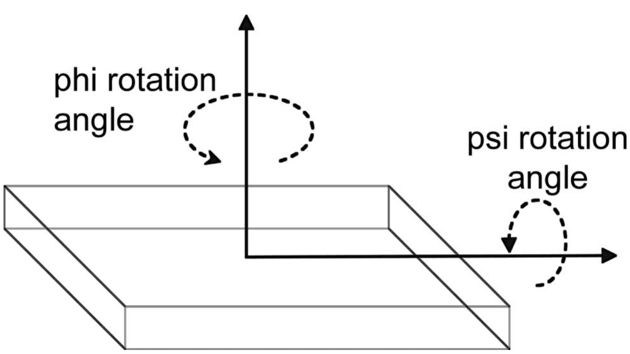


Fig. 5. Coordinate system with respect to the crystal face used in XRD measurements and calculations.

tests were first performed on the wafer to verify that the wafer was the desired (YXwl) $-16.5°$ cut of LTO. Fig. 5 shows the coordinate system with respect to the crystal face used in the XRD tests. Verification of the orientation was performed through the use of pole figures generated by a Panalytical (Westborough, MA) X'Pert PRO MRD Diffractometer using Cu k-α radiation configured with an X-ray lens and parallel plate collimator/proportional detector, a Matlab (MathWorks, Natick, MA) modeling script, and a miscut procedure performed by Sawyer Technical Materials, LLC.

B. Frequency Response of the LTO Sensor Platform

An LFE sensor platform was fabricated by photolithographically depositing thin-film gold electrodes with a chromium adhesion layer on one side (Fig. 6) of an LTO wafer. A network analyzer (HP 8571A, Hewlett Packard, Palo Alto, CA) was used to monitor the resonant frequency of the LTO LFE sensor platform (henceforth referred to as the LTO LFE). The device's fundamental frequency was 5.2 MHz. Due to the high piezoelectric coupling of LTO, it is possible to operate the device at very high frequencies by exciting high-order harmonics in the device. Resonances as high as 1.4 GHZ (269th harmonic) were detected. Although LFE and QCM sensors fabricated on AT-cut quartz may be operated at elevated harmonics [18], efficiently exciting above the 7th harmonic is difficult due to the relatively low piezoelectric coupling of α-quartz [19].

C. Temperature Stability Measurements

Because the (YXwl) $-16.5°$ cut of LTO is theoretically predicted to be temperature compensated at 25°C [14] the temperature behavior of this cut was experimentally examined. An LTO LFE sensor was placed in a temperature-controlled oven and operated at temperatures ranging from 20°C to 90°C. The changes in the resonant frequency of the LTO LFE sensor was monitored over the temperature range at the fundamental frequency (5.2 MHz), 15th harmonic (78 MHz), and 19th harmonic (98.8 MHz).

D. LTO Sensor Platform as a Liquid Phase Sensor

To demonstrate the applicability of the LTO LFE sensor, the mechanical and electrical property changes of liquids were measured and compared with standard AT-cut quartz QCM and LFE sensors. All tests were performed at a constant temperature of 25°C.

The LTO LFE sensor was operated at a different harmonic for each test to demonstrate its ability to operate over a wide range of frequencies. The wide frequency range of operation for the LTO LFE sensor opens the possibility of tuning the LTO LFE's operating frequency for a given application. It has recently been shown that, for certain acoustic wave biological-sensing applications, proper selection of the sensor operating frequency is critical due to the fact that the depth at which the acoustic wave penetrates the sensing layer depends on the operating frequency of the sensor [20].

1) Liquid Viscosity Measurements: To demonstrate the applicability of the LTO LFE sensor, it was first used to detect changes in the viscosity of liquids. The LFE LTO sensor's performance was compared with the performance of standard QCM and LFE sensors, both fabricated on AT-cut quartz with a fundamental frequency of 5 MHz. Eight solutions of varying viscosities were made by mixing Karo (Memphis, TN) brand corn syrup with deionized water at varying ratios from 0 to 80% corn syrup. The viscosity of each solution was first measured using a Cannon-Fenske Routine Viscometer (Cannon Instrument Company, State College, PA). Each solution was applied to a standard QCM, an AT-cut quartz LFE sensor, and a LTO LFE sensor. The change in frequency (from the operating frequency when only deionized water was present) was measured for each device. The QCM and AT-cut quartz LFE sensor responses were measured using a Maxtek PLO-10i phase lock oscillator and an EZ FC-705U 100 MHz Universal Counter while the network analyzer was used to monitor the LTO LFE at the fundamental frequency and 63rd harmonic of the TSM. The QCM and AT-cut quartz LFE sensor were fabricated from identical 1-in. diameter AT-cut quartz wafers obtained from Maxtek, Inc. (Beaverton, OR).

2) Liquid Conductivity Measurements: The LTO LFE sensor was also tested to determine its response to electrical property changes in liquids. Its response to the conductivity of NaCl water solutions in the range of 0 to 0.07 wt% was compared with the response of a QCM and quartz LFE. The LTO LFE was operated at the 87th harmonic for the conductivity tests, and its resonant frequency was monitored by the network analyzer. The quartz LFE and QCM were operated at their fundamental frequency, and their resonant frequency was measured using the Maxtek PLO setup described in Section IV-D-1.

3) Liquid Relative Permittivity Measurement: Permittivity measurements were performed by monitoring the responses of the LTO LFE, AT-cut LFE, and AT-cut QCM to changes in 2- propanol concentrations from 0 to 60 wt% in water. The 2 propanol was chosen because its liquid permittivity changes significantly when it is added to water [21]. The LTO LFE was operated at the 49th harmonic for the permittivity tests, and its resonant frequency was monitored by the network analyzer. The quartz LFE and QCM were operated at their fundamental frequency, and their resonant frequency was measured using the Maxtek PLO setup described in Section IV-D-1.

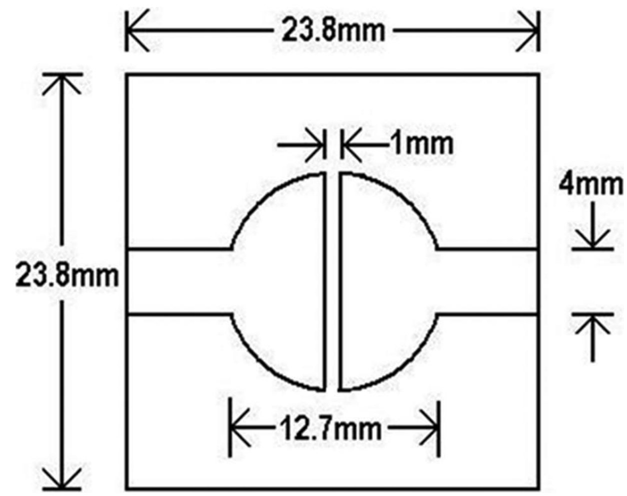


Fig. 6. LTO LFE test crystal with dimensions.

V. Results and Discussion

A. XRD Results

Using [16], the desired orientation is described as (YXwl) −16.5°, which places the unit plate normal at [0x, ± cos(16.5°)y, ± sin(16.5°) z] in an orthonormal coordinate system. Numerical rotations appropriate for a trigonal 3m type lattice [22] were performed to generate the plate normals corresponding to the equivalent crystallographic planes. Because the International Centre for Diffraction Data (ICDD) database for this crystal is defined in terms of Miller-Bravais indices on an abc $\alpha\beta\gamma$ [5.147, 5.147, 13.766] [90°, 90°, 120°] system a transformation of indices is desirable [23]. Transformation to Miller-Bravais indices for this orientation results in the 3 crystallographically equivalent planes hk.l ⟨0 −1.0931 1⟩, ⟨−1.0931 1.0931 0⟩, and ⟨1.0931 0 1⟩. The ⟨2 0 2⟩ plane was selected for measurement via XRD because it is in the same family as the ⟨1 0 1⟩ and near the theoretical ⟨1.0931 0 1⟩ wafer orientation. Figure 7(a) shows a ⟨2 0 2⟩ pole figure obtained with a diffractometer set at 2-theta = 45.569°. A Matlab modeling script was created that would simulate a theoretical XRD pole figure for a (YXwl) −16.5°; see Fig. 7(b). The XRD ⟨2 0 2⟩ pole figure and results of the modeling script for (YXwl) −16.5° are very similar. It can be concluded that a member of the family of ⟨2 0 2⟩ planes is very close to the cut surface of the sample, (YXwl) −16.5° family (which all possess identical characteristics due to crystallographic equivalence [24]) and that the received wafer was in fact cut very close to our desired orientation.

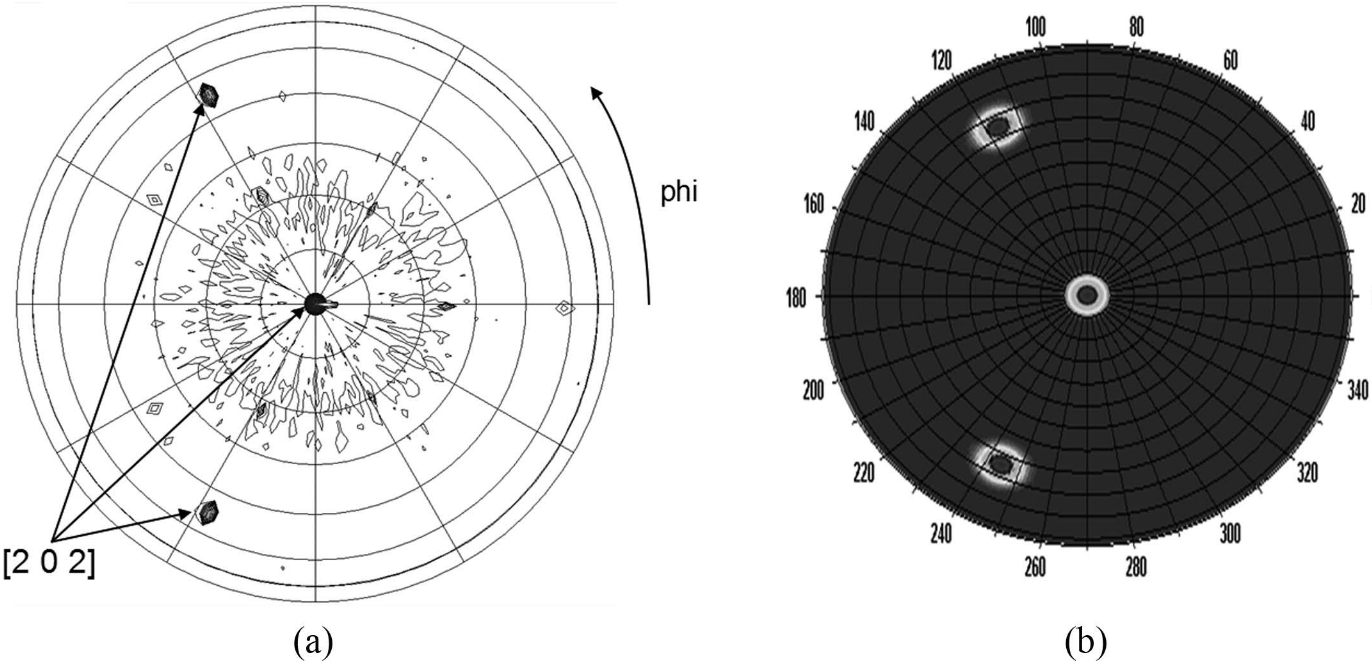


Fig. 7. a) X'Pert Texture generated experimental pole figure of the ⟨202⟩ plane for (YXwl) −16.5° LTO (concentric circles represent values of psi ranging from 0° to 90° in 15° increments) and b) theoretical Matlab simulation for (YXwl) −16.5° LTO (concentric circles represent values of psi ranging from 0° to 90° in 10° increments).

For a more precise measurement and confirmation of these conclusions, the wafer was sent to Sawyer Technical Materials, LLC, for a miscut analysis. Sawyer determined that the orientation was either (YXwl) −16.467° or (YXwl) −19.433°. The (YXwl) −19.433° possibility was excluded through an XRD measurement of the ⟨0 0 6⟩ pole figure. In the case of a (YXwl) −16.5° orientation, the ⟨0 0 6⟩ pole would be located at psi = 73.5°. In the case of the (YXwl) −19.433° orientation, the ⟨0 0 6⟩ pole would be located at psi = 70.467°. The fact that the ⟨0 0 6⟩ pole was observed to be at psi = 73.5° in the XRD measurements further verified that the wafer was the correct orientation.

B. Temperature Stability Measurements

The (YXwl) −16.5° cut of LTO is theoretically temperature compensated at 25°C. However, as seen in Fig. 8(b), experimental measurements show this cut is actually temperature compensated at approximately 70°C, not at 25°C. It is interesting to note that the temperature behavior of the LTO LFE sensor changes very little when it is operated at higher harmonics. We believe that the discrepancy between our predicted and measured temperature behavior for this cut is due slight inaccuracies in the temperature coefficients used for the theoretical calculations because small changes in these values can have significant impacts on the predicted results. Given that the (YXwl) −16.5° cut of LTO is experimentally temperature compensated at 70°C, it is possible that an orientation close to the (YXwl) −16.5° cut is in fact temperature compensated at 25°C. These temperature stability results clearly point to a need for updated and more accurate material constants and temperature coefficients for LTO.

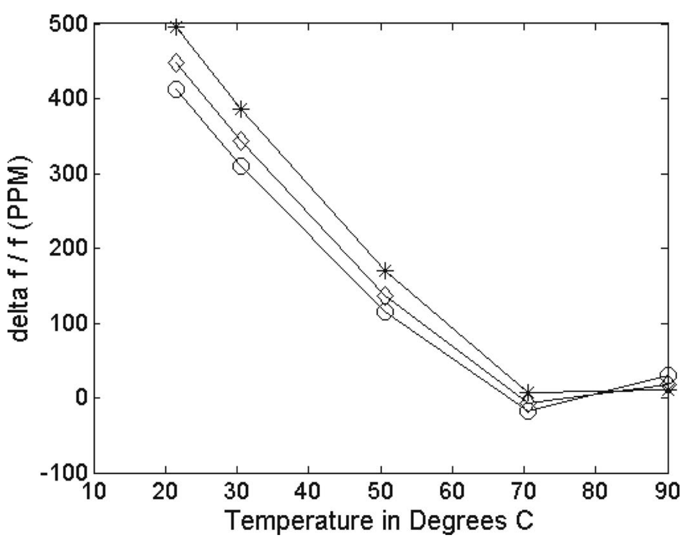
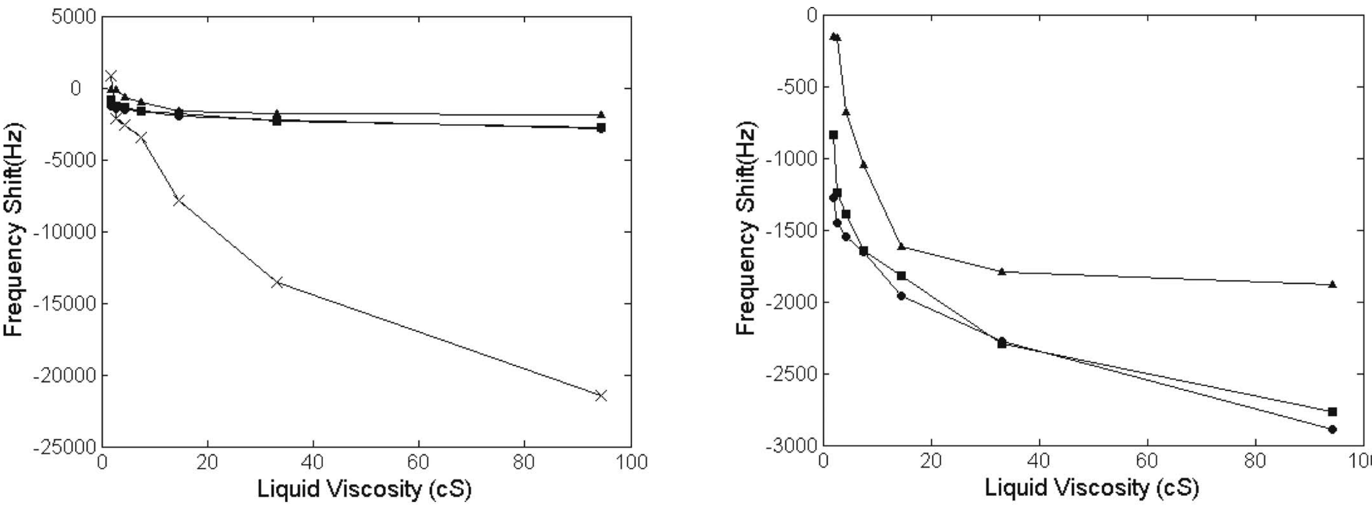


Fig. 8. Experimental temperature behavior for (YXwl) −16.5° LTO at its fundamental frequency (5.2 MHz, connected '○'), 15th harmonic (78 MHz, connected '*'), and 19th harmonic (98.8 MHz, connected '◇').

C. LTO Sensor Platform as a Liquid Phase Sensor

1) Liquid Viscosity Measurements: Quartz LFE, quartz QCM, and LTO LFE sensors were tested by applying solutions of corn syrup water solutions with viscosities ranging from 1 to 94 cS. As can be seen in Fig. 9(a), the LTO LFE sensor had significantly larger responses to viscosity change than either the standard QCM or quartz LFE when it was operated at the 63rd harmonic. Specifically, the LTO LFE sensor showed an approximately 12 times larger frequency shift when compared to either the stan-



(a) (b)

Fig. 9. Sensor response to change in corn syrup viscosities for the a) AT-cut QCM (fundamental frequency, connected '▲'), AT-cut quartz LFE (fundamental frequency, connected '■'), (YXwl) −16.5° LTO LFE (fundamental frequency, connected '●'), and (YXwl) −16.5° LTO LFE sensor (63rd Harmonic, connected 'x'), and b) AT-cut QCM (fundamental frequency, connected '▲'), AT-cut quartz LFE (fundamental frequency, connected '■'), (YXwl) −16.5° LTO LFE (fundamental frequency, connected '●') only.

dard QCM or quartz LFE sensors. It is interesting to note that, as seen in Fig. 9(b), the LTO LFE and the quartz LFE exhibited almost identical frequency shifts when they were both operated at their fundamental frequencies.

2) Liquid Conductivity Measurements: As discussed in Section I, LFE sensors are also capable of detecting electrical changes because there is no metal layer on the sensing surface as opposed to the QCM where a metal electrode is placed on both surfaces of the plate [6]–[8].

To test the LTO LFE sensor's ability to measure changes in the conductivity of a liquid, the change in resonant frequency of an LTO LFE sensor was monitored using an HP 8751A network analyzer while the sensor was subjected to liquids with various concentrations of NaCl. These measurements were performed at the 87th harmonic of the LTO LFE sensor. The response of a standard QCM and quartz LFE to the same liquids was measured using the PLO setup previously discussed. The resonant frequency changes of the sensors with respect to their resonant frequencies in deionized water as a function of NaCl concentration is shown in Fig. 10(a).

Because the resonant frequency change of the LFE sensors is due to both mechanical and electrical property changes in the liquid, the NaCl concentrations chosen for this experiment (0 to 0.07% wt) have very small variations in mechanical property changes such as density and viscosity. The frequency shift for a 0.5% wt NaCl solution predicted by mechanical perturbation theory was found to be only 5 Hz [4]. It can therefore be assumed that the mechanical properties of the liquid had negligible effects on the frequency response of the sensors.

As can be seen in Fig. 10(a) and Fig. 10(b), the LTO LFE sensor was able to measure the changes in the liquid conductivity while the QCM could not. As can be seen in Fig. 10(b), the quartz LFE exhibited a frequency shift of 434 Hz for the 0.07 wt% solution (11 500 μS/cm). The frequency fluctuations of the QCM for all of the liquids were within the noise of the sensor. The frequency change of the LTO LFE sensor was 30.75 kHz for the 0.07 wt% (11 500 μS/cm) solution leading to the conclusion that the LTO LFE sensor is extremely sensitive to small electrical property changes.

3) Liquid Relative Permittivity Measurement: The LTO LFE's sensor response to changes in relative permittivity (ε_r) was also explored. Fig. 11(a) shows the resonant frequency changes of the LTO LFE, quartz LFE, and QCM sensors with respect to their resonant frequencies in deionized water to changes in 2-propanol concentrations in water between 0 wt% ($\varepsilon_r = 80$) and 60 wt% ($\varepsilon_r = 44$). As can be seen in Figs. 11(a) and (b), the frequency of the QCM decreased by 338 Hz at 60 wt% 2-propanol concentration while the frequency of the LTO and quartz LFE sensors increased. Unlike the LFE sensors, which respond to both mechanical and electrical liquid property changes, the resonant frequency of the QCM is only influenced by the mechanical properties of the liquid, in this case, the product of density and viscosity, which reaches a maximum at approximately 50 wt% ($\varepsilon_r = 50$) [21]. The LFE sensors also respond to changes in density and viscosity. However, in the case of the permittivity changes in this experiment, the sensor's frequency response is dominated by the changes in electrical properties. It has previously been reported that relative permittivity decreases approximately linearly with 2-propanol concentrations in water from 0 wt% to 70 wt% [25]. In the case of the LFE sensors, the resonant frequency increases as the relative permittivity decreases. The frequency shift of the LTO LFE sensor was approximately 29 times larger when it was operated

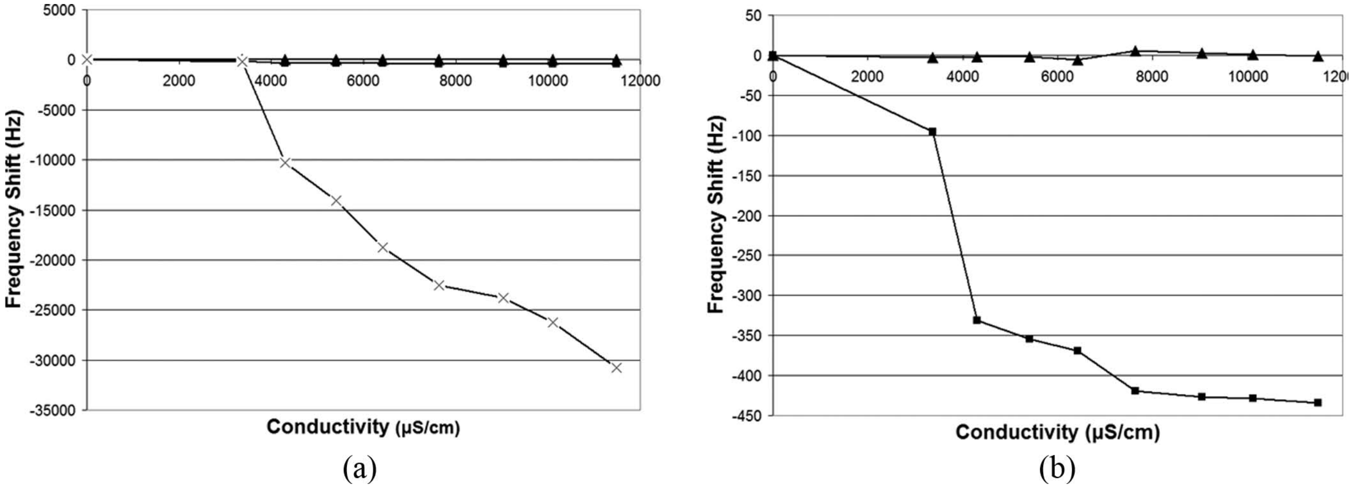


Fig. 10. Sensor response to changes in NaCl concentration for a) AT-cut QCM (fundamental frequency, connected '▲'), AT-cut quartz LFE (fundamental frequency, connected '■'), and (YXwl) −16.5° LTO LFE (87th Harmonic, connected 'x') sensors and b) AT-cut QCM (fundamental frequency, connected '▲'), AT-cut quartz LFE (fundamental frequency, connected '■') only.

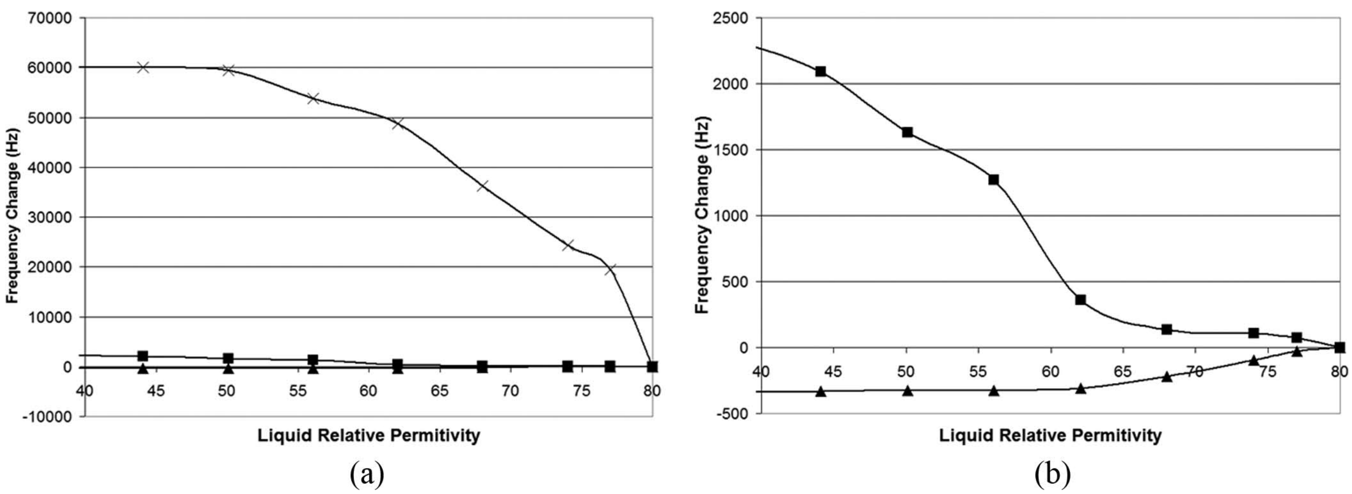


Fig. 11. Sensor response to changes in 2-propanol concentration for a) AT-cut QCM (fundamental frequency, connected '▲'), AT-cut quartz LFE (fundamental frequency, connected '■'), and (YXwl) −16.5° LTO LFE (49th Harmonic, connected 'x') sensors and b) AT-cut QCM (fundamental frequency, connected '▲'), AT-cut quartz LFE (fundamental frequency, connected '■') only.

at its 49th harmonic compared with the quartz LFE sensor operated at its fundamental frequency.

VI. Conclusions

A search was made for orientations in lithium tantalate that, when excited with a lateral electric field, would offer improved sensor performance over standard AT-quartz LFE and QCM sensors. Two candidate orientations of lithium tantalate were theoretically identified that showed significant improvements in electromechanical coupling and predicted to have temperature compensated pure TSMs at 25°C, namely, the (YXwl) −16.5° and (YXwl) −84.3° cuts. The (YXwl) −16.5° cut was chosen for further testing based on the separation of interfering modes and the body of literature available on it from its use in RF-filtering applications. XRD measurements were performed on the wafer that verified that it was the correct orientation. An improperly aligned wafer was then excluded as a possible explanation for the differences observed in the theoretical and measured temperature characteristics. An LFE sensor with a fundamental frequency of 5.2 MHz was fabricated on this cut and found to be capable of operating at frequencies as high as 1.4 GHz (269th harmonic). The temperature behavior for this cut of LTO was experimentally measured and found to be temperature compensated at 70°C instead of 25°C as theoretically predicted. We believe that inaccuracies in the material constants and temperature coefficients used to perform the theoretical calculations are the cause of this discrepancy.

This result points to the need for more experimental work to determine exactly the orientation in LTO that is temperature compensated at 25°C. When tested as a sensor, the LTO LFE was found to be capable of detecting both mechanical and electrical property changes in liquids, unlike the standard QCM. This sensor was used to measure viscosity, conductivity, and relative permittivity changes in liquids and found to have significantly larger frequency changes than AT-cut QCM and LFE sensors when it was operated at high frequencies. It is expected that the LTO LFE sensor will yield significant increases in sensor sensitivity for both chemical and biological sensing applications.

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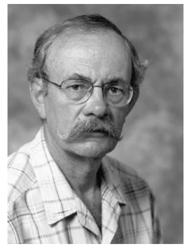

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