A Room Temperature Compensated Lateral Field Excited Lithium Tantalate Sensor Platform

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***Abstract*—The quartz crystal monitor (QCM) is a common sensor platform based on the room temperature compensated pure shear mode (PSM) of thickness field excited (TFE) AT-cut quartz. However, with electrodes on both crystal faces, TFE only allows sensing of mechanical property changes. Lateral field excitation (LFE), where both electrodes are on a single face, enables the detection of both mechanical and electrical changes, potentially leading to higher sensitivity. As lithium tantalate (LT) has an LFE PSM and piezoelectric coupling several times greater than that of quartz, LFE LT was chosen as a possible replacement for TFE quartz. A theoretical search of all LT cuts identified those exhibiting a room temperature compensated PSM. A set of orientations ranging from (YXwl) 0°/-85° to 0°/-90° was chosen for experimental verification. The temperature response of each sample was shown to be parabolic, with a roughly linear relationship between crystal cut angle and temperature inflection point/turnaround temperature. Specifically, the (YXwl) 0°/-87° cut with a turnaround temperature at 26.4°C demonstrates that a room temperature PSM in LT can be excited via LFE. Future work focusing on the development of an LT sensing platform could profoundly impact sensor systems in agriculture, homeland security, global warming, and medical applications.**

Keywords*—lateral field excitation, lithium tantalate, temperature compensation, bulk acoustic wave sensor

I. INTRODUCTION

The critical components in a sensor system are the sensor platform which documents the target analyte capture as an electrical signal determining sensitivity, and the target analyte selective film or layer deposited on the sensor platform which determines selectivity. Depending upon the specific sensor application a considerable amount of work has been devoted toward the target selective film or layer and far less on the sensor platform. In the case of the latter, one of the most common sensor platforms has been the "off the shelf" quartz crystal microbalance (QCM) which employs a thickness field excited (TFE) room temperature compensated pure shear mode (PSM) in AT-cut quartz. Lateral field excitation (LFE), where both electrodes are on a single quartz face, enables the detection of both mechanical and electrical property changes leading to higher sensitivity as shown in several recent publications [1], [2]. The use of quartz as the piezoelectric crystal also further inhibits sensitivity due to the very low piezoelectric coupling of quartz. The low coupling of quartz was recognized as a drawback in the late 1960's in bulk and surface acoustic wave signal processing devices leading to work on other piezoelectric materials and in particular lithium niobate (LN) and lithium tantalate (LT) [3]. LN had much higher piezoelectric coupling but poor temperature stability. LT was also briefly studied since it had better temperature stability than LN but not the temperature stability associated with quartz. As a result combinations such as LN on quartz were examined [4]. Another problem which affected both LN and LT was the fact that both of these crystals were pyroelectric. The deleterious effects associated with pyroelectricity could however be minimized in the growth process leading to two types of LN and LT, namely as grown or congruent LN and LT and black LN or LT. Since LT was more strongly pyroelectric, as grown LN was chosen as the crystal of choice for surface and bulk acoustic wave signal processing devices. In order to carefully select specific orientations for acoustic wave devices the material constants and their temperature dependence were measured for both as grown LN and black LT [5]. Using these material constants it was determined theoretically that LN did not have bulk acoustic wave orientations that were temperature compensated and LT had several temperature compensated bulk wave orientations some of which were pure shear modes (PSM) [6]. Subsequently, temperature compensated bulk wave orientations in LT were confirmed experimentally which led to an exhaustive search for black LT crystals which were successfully located.

The primary focus of this paper is the development of a sensor platform whose major component consists of a room temperature compensated LFE excited PSM in black LT. It is envisioned that this sensor platform will have a wide range of applications in diverse areas such as medicine, agriculture, global warming, and homeland security to name just a few.

II. Methods

A. Verification of Previous Results

In previous work [6], a theoretical search of all crystallographic orientations of LT identified those exhibiting an LFE PSM with a 1st order temperature coefficient of frequency (TCF) that is equal to zero at room temperature. The PSM's 1st order TCF is defined as the derivative of its series resonance frequency with respect to temperature. To experimentally study these temperature compensated cuts, LT samples between (YXwl) 0°/-84.2° and (YXwl) 0°/-90° were acquired and tested.

Two pairs of Z-cut plano-plano LT samples were obtained from two different manufacturers. The first pair was purchased from Yamaju Ceramics Co. LTD, the same source used in previous research [6]. The second pair was purchased from Hangzhou Freqcontrol Electronic Technology LTD. These samples were 2" in diameter and 0.5mm thick. As shown in Fig. 1 below, in each pair, one sample was treated for suppression of pyroelectricity, a process called "blackening," and the other was left untreated. Treated and untreated LT will be referred to as black LT and white LT respectively.

These samples were studied to verify the previous results for (YXwl) 0°/-90° LT, confirm the necessity that LT be black to suppress pyroelectric effects, and compare crystal quality between the two manufacturers. In both white LT samples irregular drifts in the resonant frequency were observed when temperature was varied. This irregular behavior was also observed in previous work [6]. Possibly, the pyroelectric properties of these crystals cause an accumulation of charge resulting in spontaneous polarization. In any case, the instability of the white LT samples likely precludes any practical benefits to be exploited from their temperature behavior. On the other hand, the black LT samples from both manufacturers behaved as expected, based on what was demonstrated in previous studies. Both samples exhibited a zero TCF at about 50.5°C.

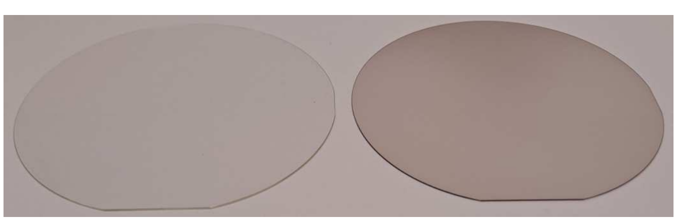

Fig. 1. White (left) and black (right) LT samples.

B. Identifying the Room Temperature Compensated Cut

To narrow in on the room temperature compensated cut, additional black LT samples of the orientations (YXwl) 0°/-85°, 0°/-86°, 0°/-86.5°, 0°/-87°, and 0°/-90° were purchased from Hangzhou Freqcontrol Electronic Technology LTD. These plano-plano 0.55" diameter circular samples were 0.5mm thick. Room temperature baseline measurements were taken to assess the frequency responses of each of the samples. An Agilent E5071C ENA Vector Network Analyzer (VNA) and an LFE test fixture, constructed on a PCB, were used to obtain scattering parameter (S-parameter) data for each sample. The LFE test fixture electrodes were 0.9mm in diameter with a 0.5mm gap. The fundamental resonances, as well as up to the 13th harmonics, of each of the samples were assessed by analyzing their frequency response in terms of admittance magnitude. The fundamental frequency for each sample was found to be between 3.5MHz and 3.6MHz. As the samples were approximately 0.5mm thick, this agrees with the numerical expectation of a fundamental resonant frequency at about 3.6MHz for an LT crystal plate of this thickness. The fundamental resonances were first located using a wide span of 1.6MHz on the VNA, then were examined qualitatively at a narrower span of 48 kHz. It can be seen in Fig. 2 below that the fundamental resonances were plagued by significant coupling to spurious modes. This is likely due to the poor energy trapping associated with the plano-plano geometry of the samples. This spuriousness caused shifts in the fundamental frequency to be difficult to differentiate and track across wide temperature ranges. However, as also demonstrated in Fig. 2, the higher order harmonics tended to be much cleaner and less spurious. For this reason, higher order harmonics up to the 13th were tracked within a narrower span of 16kHz during temperature tests on the samples.

The temperature responses of each of the LT samples were tested using the same VNA and PCB test fixture described above. In this case, the samples and test fixture were placed inside a proportional integral derivative (PID) controlled Tenney environmental test chamber (ETC), with a resistance

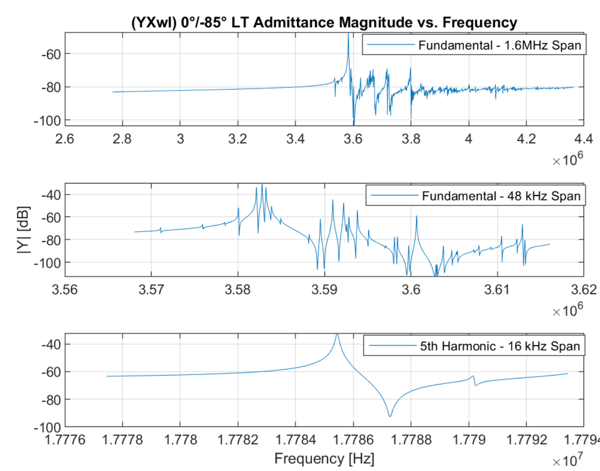

Fig. 2. Example baseline responses from the (YXwl) 0°/-85° sample's fundamental with a wide span (top), fundamental with a narrow span (middle) and 5th harmonic with a narrow span (bottom).

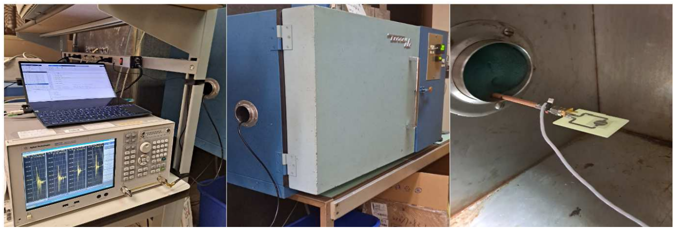

Fig. 3. (left) PC running a MATLAB script to control the VNA and (middle) PID controlled Tenney ETC. (right) PCB test fixture and LT sample inside the ETC.

temperature detector (RTD) measuring local temperature. The VNA outside the chamber obtained port parameter data for each sample through standard 50Ω coaxial cables connected to the test fixture. A PC running MATLAB® swept the temperature in the ETC, recorded the fixture temperature, and acquired data from the VNA. This test setup can be seen in Fig. 3 above.

III. Results

Following the collection of resonant frequency data, plots of the data, along with 2nd order fits were generated for analysis and comparison with previous research. The data was portrayed in terms of the deviation in resonant frequency, as a function of temperature, from the resonant frequency at room temperature. This can be expressed as Δf/f(25°C), in parts per million (PPM). In Fig. 4 below it can be seen that each sample demonstrates a parabolic temperature response.

The fundamental resonance for each plano-plano sample was generally too spurious to track across a range of temperatures. As such, higher order harmonics were primarily used for analysis. The curves in Fig. 4 below were generated from S-parameter data for each sample's 9th harmonic. The temperature responses of each of the samples align with previous theoretical predictions and the (YXwl) 0°/-90° sample matches the previous experimental results for the same cut with a zero TCF in the vicinity of 50°C. The triangles in Fig. 4 mark the inflection point of the second order fitted parabolic curves,

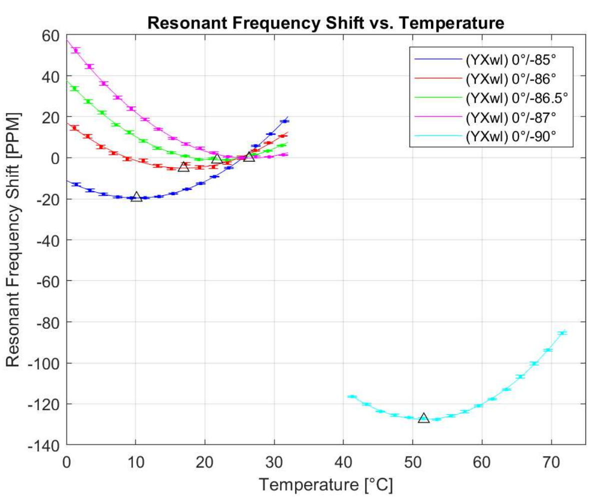

Fig. 4. Temperature responses of black LT samples.

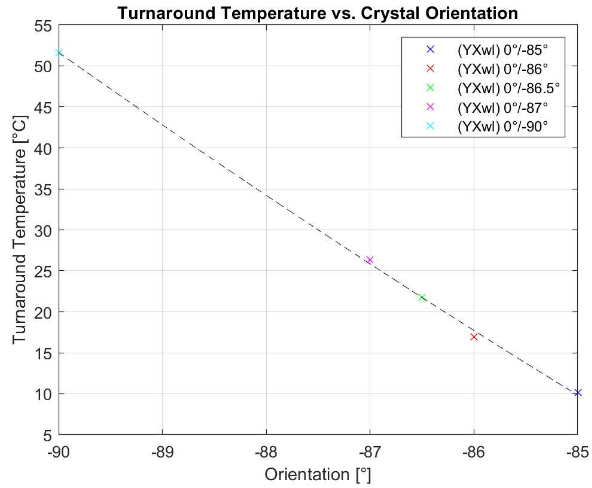

Fig. 5. Temperature at which zero-valued TCF occurs versus crystal orientation.

which also coincides with the temperature at which the derivative of the resonant frequency with respect to temperature is equal to zero. It can be seen in Fig. 4 that the zero TCF for the (YXwl) 0°/-87° sample is located at about 26.4°C, making this the room temperature compensated LT cut that has been sought. Furthermore, Fig. 5 above shows that a roughly linear relationship between crystal cut angle and temperature inflection point/turnaround temperature occurs, which implies that the temperature at which the zero-valued TCF occurs could be adjusted with relative ease by modifying the LT crystal orientation.

It should be noted that the data points in Fig. 5 above correspond with the average temperature values of 55 measurements taken at each temperature setpoint.

IV. Conclusions

A lateral field excited room temperature temperature-compensated lithium tantalate cut was identified theoretically and demonstrated experimentally. Based on theoretical analysis and simulation, a set of orientations ranging from (YXwl) 0°/-85° to 0°/-90° was chosen for experimental verification. These plano-plano samples demonstrated spurious responses near the fundamental resonance. Therefore, higher-order harmonics, up to the 13th, were used for TCF measurements. The temperature response of each sample was parabolic, with a roughly linear relationship between crystal cut angle and turnaround temperature. In particular, the (YXwl) 0°/-87° cut was shown to exhibit a turnaround temperature at room temperature (26.4°C). The next phase of this research will focus on investigating the surface curvature to optimize the LFE response and to suppress the spurious response. Future work focusing on the development of an LT sensing platform could profoundly impact sensor systems in agriculture, homeland security, global warming, and medical applications.

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REFERENCES

[1] L. A. French, D. F. McCann, M. Wark, S. Winters and J. F. Vetelino, "A Lateral Field Excited Acoustic Wave Sensor," *TRANSDUCERS 2007 - 2007 International Solid-State Sensors, Actuators and Microsystems Conference*, Lyon, France, 2007, pp. 1287-1290, doi: 10.1109/SENSOR.2007.4300373.

[2] Jequil S. R. Hartz, Nuri W. Emanetoglu, Caitlin Howell,and John F. Vetelino, "Lateral field excited quartz crystal microbalances for biosensing applications", Biointerphases 15, 030801 (2020) https://doi.org/10.1116/6.

[3] J. J. Hannon, P. Lloyd and R. T. Smith, "Lithium Tantalate and Lithium Niobate Piezoelectric Resonators in the Medium Frequency Range with Low Ratios of Capacitance and Low Temperature Coefficients of Frequency," in *IEEE Transactions on Sonics and Ultrasonics*, vol. 17, no. 4, pp. 239-245, Oct. 1970, doi: 10.1109/T-SU.1970.29572.

[4] T. Baron *et al*., "RF oscillators stabilized by temperature compensated HBARs based on LiNbO3/Quartz combination," *2011 Joint Conference of the IEEE International Frequency Control and the European Frequency and Time Forum (FCS) Proceedings*, San Francisco, CA, USA, 2011, pp. 1-4, doi: 10.1109/FCS.2011.5977814.

[5] R. T. Smith, F. S. Welsh; Temperature Dependence of the Elastic, Piezoelectric, and Dielectric Constants of Lithium Tantalate and Lithium Niobate. *Journal of Applied Physics* 1 May 1971; 42 (6): 2219–2230. https://doi.org/10.1063/1.1660528.

[6] J. M. McGann, D. F. McCann and J. F. Vetelino, "Lateral field excited LiTaO3 acoustic wave sensing platform," *2010 IEEE International Ultrasonics Symposium*, San Diego, CA, USA, 2010, pp. 938-941, doi: 10.1109/ULTSYM.2010.5935758.