DEVELOPMENT OF A NEW CQCM/TQCM SENSOR TO ACHIEVE GOOD USABILITY AND TEMPERATURE MEASUREMENT

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ABSTRACT

The Quartz Crystal Microbalance (QCM) sensor is a key device used in evaluating contamination characteristics such as deposition, desorption, and temperature-dependent properties. We propose a new cryogenic QCM (CQCM) sensor with good usability and temperature measurement characteristics using the twin-electrode technique. The sensor was named "Twin-CQCM" after its unique technique aspect. Its operating temperature ranges from –190 to +125°C. The temperature-dependent drift of the frequency was less than ±10 ppm over the whole temperature range; note that the frequency shift value is experimental data. To confirm the accuracy of temperature measurement, we installed an additional platinum temperature sensor at the center of the crystal. The sensor output temperature value was compared to that of the additional sensor, with the difference between both temperature sensors being +0.4 to +2.6°C in the temperature range from –130 to +100°C.
The user can easily replace the CQCM sensor's crystal. This good usability enables not only simplified sensor replacement but also new applications, such as atomic oxygen (AO) measurement and the fixation of contaminants by ultraviolet (UV) irradiation.
In addition, a new thermoelectric QCM (TQCM) is under development using the twin-electrode technique. The TQCM sensor was designed to ensure good tolerance to mechanical environments, similar to the long-life requirement for onboard satellite components.

1. INTRODUCTION

The QCM sensor is widely utilized to measure the deposition/desorption amounts of contaminants in a vacuum chamber [1]. As contamination phenomena depend on the temperature, the sensor used for in-situ contamination measurement must have a wide operating temperature range. The operation of commercial QCM sensors is generally guaranteed at around room temperature. Therefore, a sensor's operating temperature range should be extended for space use. And because the quartz crystal oscillation frequency depends on the temperature caused by the quartz characteristics, a compensation technique for the frequency drift is needed to extract only the mass effect of contamination phenomena. Some QCM sensors were developed for space use [2-3] by using two sensor crystals to compensate the temperature effect. One sensor crystal is used to detect the deposited contaminant mass (F_d), and the other is used to detect the frequency drift caused by the temperature effect as a reference frequency value (F_r). The sensor output frequency (F) is calculated by the two frequency values ($|F_d - F_r|$).

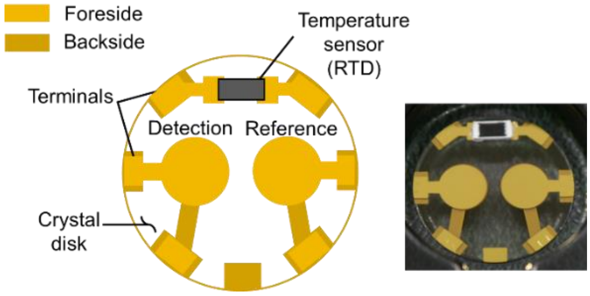

Fig. 1. The twin-electrode sensor crystal including the electrodes for detection and reference

However, the use of two separate quartz crystals tends to complicate sensor crystal replacement due to the sensor unit structure. Once contaminant substances adhere to the sensor crystal, the sensor output frequency (F) gradually increases in accordance with the remaining contaminants that cannot be refreshed by baking. Therefore, a refreshment technique is required to maintain the sensor conditions for contamination tests.
The twin-electrode technique is thus proposed to simplify the sensor structure and enable easy sensor replacement. The proposed structure improves not only replacement but also the equivalence of each electrode's temperature condition. And by using the free space close to the twin electrodes on the crystal, a temperature sensor can be installed to improve the accuracy of temperature measurement. This paper describes the proposed twin-electrode technique and discusses the evaluation results.

2. TWIN-ELECTRODE SENSOR CRYSTAL

Figure 1 shows the twin-electrode sensor crystal including the two oscillation electrodes for detection (F_d) and reference (F_r). The reference electrode is placed next to the detection electrode to prevent the interaction of oscillation energy. The oscillation electrodes and the crystal are 4 mm and 14 mm in diameter, respectively. The crystal is approximately 162 μm in thickness, that is,

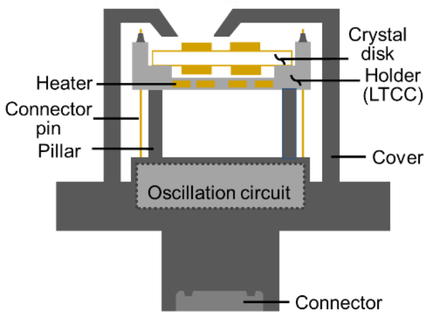

Fig. 2. Cross-sectional drawing of the Twin-CQCM sensor

Fig. 3. Photos of the Twin-CQCM sensor unit (Left: unit with the cover; Right: unit without the cover)

Table. 1 Twin-CQCM specifications

Item	Value
Oscillation Frequency	10.278 [MHz]
Crystal	AT-cut 14 mmϕ Twin electrodes
Mass sensitivity	2.39×10^8 (Fund.) [Hz/(g/cm^2)] 7.17×10^8 (3rd.) [Hz/(g/cm^2)]
Heater power	less than 9 [W] (typ. 3.5 [W])
Temperature sensor	RTD platinum 1000

the oscillation frequency in fundamental mode is approximately 10 MHz. A resistance temperature detector (RTD) is installed on the same crystal disk using the free area to measure the crystal temperature itself for improved accuracy. Given the sensitive reaction to temperature conditions by contamination phenomena, accurate temperature measurement is meaningful. There are six terminals along the edge of the disk for small clips to clamp the crystal disk, and the Low Temperature Co-fired Ceramics (LTCC) holder (Figs. 2 and 3) without any mechanical strain. The heater for temperature control is embedded in the LTCC holder. The connector pins transmit the oscillation frequency signals, heater current, and temperature sensor signal. The Twin-CQCM sensor provides two kinds of signals: one for fundamental mode frequency (approx. 10 MHz), and the other for 3rd overtone mode frequency (approx. 30 MHz). In 3rd overtone mode, the sensor sensitivity is three times

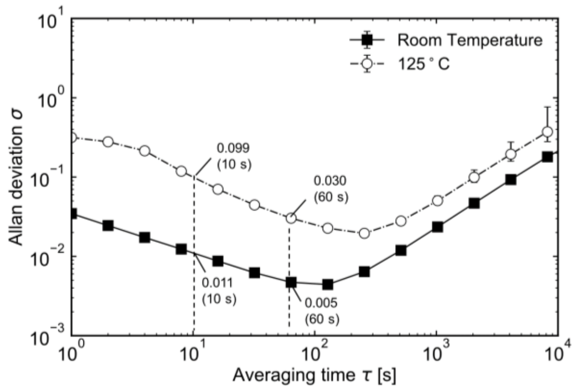

Fig. 4. Allan deviation of the Twin-CQCM sensor at room temperature and +125 °C under a vacuum

higher than that in fundamental mode. Table 1 lists the sensor specifications.

2.1 Sensor stability and resolution

Figure 4 shows the stability of the Twin-CQCM sensor in fundamental mode as the values of Allan deviation [4] expressed by Eq. (1), with a one-second sampling period. When the averaging time is 10 seconds, the frequency deviation values were 0.011 Hz and 0.099 Hz at room temperature and +125°C, respectively. In the case of 60 seconds, the values were 0.005 Hz and 0.030 Hz; therefore, the output frequency is sufficiently stable for contamination measurement even at the temperature of +125°C because 0.1 Hz (ΔF) represents approximately 0.42 ng/cm^2.

$$\sigma^2(\tau) = \left\{ \sum_{i=0}^{M-1} \left[\frac{x(i\tau + \tau) - x(i\tau)}{\tau} \right]^2 - \frac{1}{2} \left[\sum_{i=0}^{M-1} \frac{x(i\tau + \tau) - x(i\tau)}{\tau} \right]^2 \right\} \quad (1)$$

Where,

M	:	Number of sampling points	[-]
τ	:	Averaging time	[s]
x	:	Data	[-]
$\sigma^2(\tau)$	:	Allan variance	[-]
$\sigma(\tau)$	:	Allan deviation, $\sigma(\tau) = \sqrt{\sigma^2(\tau)}$	[-]

2.2 Frequency drift

The operating temperature of the Twin-CQCM sensor ranges from –190 to +125°C. Figures 5 and 6 show the frequency drift caused by the temperature-dependent effect of quartz crystal in both modes. Thanks to the twin-electrode technique, the drift of the output frequency was less than ±10 ppm in the range of –175 to +125°C without the crystal matching procedure. The value is sufficiently small so as not to affect estimation of the contaminant deposition/desorption mass.

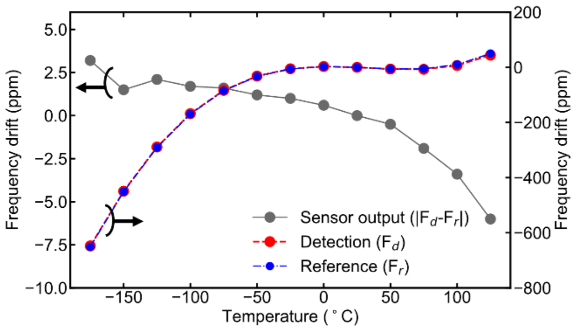

Fig. 5. Twin-CQCM frequency drift in fundamental mode caused by temperature-dependent effect

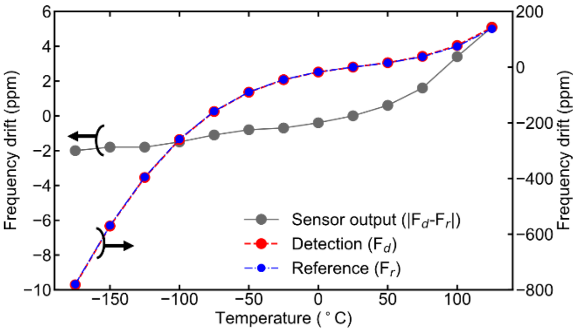

Fig. 6. Twin-CQCM frequency drift in 3rd overtone mode caused by temperature-dependent effect

2.3 Temperature measurement accuracy

As shown in Fig. 1, a temperature sensor is installed in the free area of the crystal to measure the crystal itself. Because the temperature of interest is at the center of the crystal, the difference between the sensor temperature (*Ts*) and the center temperature (*Tc*) was estimated. Figure 7 shows the result of the difference. To measure the center temperature, an additional sensor (*Tc*) was installed. The difference between the two temperature sensors (*Ts* - *Tc*) was +0.4 to +2.6°C in the range of –130 to +100°C under cooling by liquid nitrogen. The slope was only 0.009, which confirmed the accuracy of temperature measurement.

2.4 Usability

As the sensor crystal is installed on the LTCC holder using six small clips, the user can easily replace the crystal disk in a few minutes without complicated instructions. The simple procedure is as follows:

1. Open the sensor cover.
2. Remove the clips and the sensor crystal disk.
3. Put a new sensor on the LTCC holder.
4. Close the cover.

The Twin-CQCM sensor provides the frequency values of both detection and reference so that user can comprehend more detailed phenomena. A simultaneous increase or decrease in both detection and reference frequency might represent a change in a surrounding environmental factor, such as the vacuum condition or sensor temperature. Otherwise, the data acquired would imply physical interaction on the sensor electrodes.

The output frequency signals at around 10 and 30 MHz square waves are converted to Low Voltage Differential signalling (LVDS) signals in the oscillation circuit to optimize the signal qualities for long transmittance between the sensor unit and the controller. This guarantees a communication distance of at least 20 m.

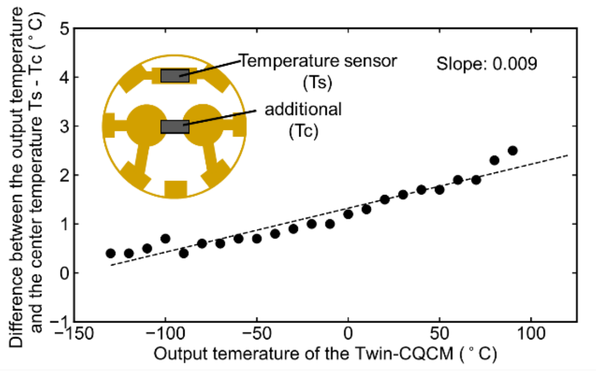

Fig. 7. Twin-CQCM temperature measurement accuracy

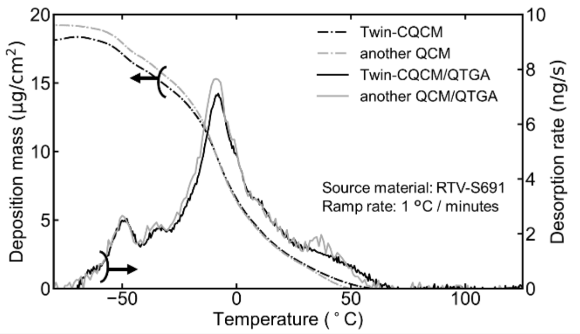

Fig. 8. Comparison between the QCM sensors by QTGA analysis (Source material: RTV-S691)

3. CONTAMINANT MEASUREMENT

As shown in Fig. 8, contaminant deposition and a QCM Thermogravimetric Analysis (QTGA) [1] measurement were conducted using the Twin-CQCM sensor and another QCM sensor for a comparison of both sensor outputs. The temperature of the sensors was set to –190°C at the deposition phase, and then the QTGA spectrum was acquired at a ramp rate of 1°C per minute. The deposition masses acquired by the Twin-CQCM sensor and the other QCM sensor were 19.0 and 19.9 μg/cm^2, respectively. The 4.5% difference between the two kinds of QCMs includes a slightly different view factor from the contaminant source. When looking at the QTGA spectrum, the temperature and rate of the desorption peaks are almost the same. From these results, compatibility with another QCM sensor was confirmed.

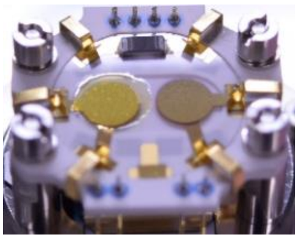

Fig. 9. Photo of the quartz crystal with a polyimide thin film on the detection (left) electrode

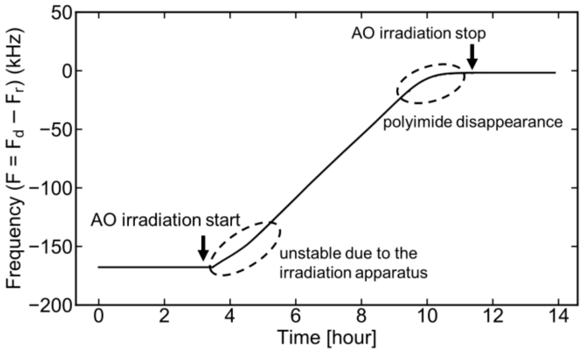

Fig. 10. AO flux measurement by the Twin-CQCM sensor with a polyimide thin film

4. ATOMIC OXYGEN MEASUREMENT

Thanks to the replaceable sensor crystal, the sensor is easily applicable to Atomic Oxygen (AO) flux measurement. To demonstrate such application, an AO irradiation test was conducted. Polyimide thin film was selected as a target material under AO irradiation. The polyimide film is eroded in accordance with the flux, and the Twin-CQCM sensor measures the erosion rate. Figure 9 shows the sensor crystal with a polyimide film coated using a spin coater on the sensor electrode for detection. When the thickness of the film was 5.4 μm, the frequency of the sensor was approximately 168.7 kHz. The sensor was installed in an AO irradiation chamber, and another polyimide film sheet was put next to the Twin-CQCM sensor for monitoring the AO total fluence. Figure 10 shows the frequency during the irradiation. The sensor successfully determines the AO flux, and the slope of approximately 7 Hz/s is consistent with the sensitivity of 9.44 (atoms/cm^2)/Hz calculated by Eq. (2). Note that the start of AO irradiation in Fig. 10 was unstable due to the irradiation apparatus.

5. TWIN-TQCM SENSOR

A new Twin-Thermoelectric QCM (TQCM) sensor now under development does not utilize liquid nitrogen for cooling. Instead of the heater in the LTCC holder (Fig. 2), a Peltier unit is inserted between the holder and the oscillation circuit. Figure 11 shows a conceptual drawing of the Twin-CQCM sensor. Because the LTCC holder is supported by the Peltier unit, it is important to assure the tolerance for vibration and shock environments. Using the consumer model that we designed, vibration and

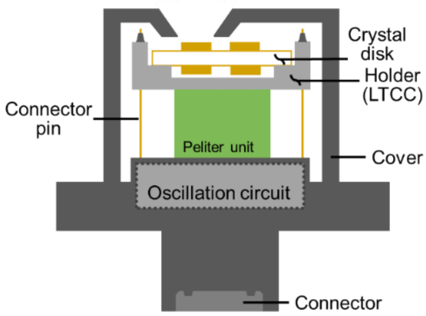

Fig. 11. Conceptual drawing of the Twin-TQCM sensor

Table 2. Vibration and shock test conditions

Item	Value	Result
Random vibration	In-plane axis: 20 - 70 Hz: +6 dB/oct. 70 - 700 Hz: 0.2 G^2/Hz 700 - 2000 Hz: -8 dB/oct. (overall: 14.1 grms) Out-of-plane axis: 20 - 70 Hz: +6 dB/oct. 70 - 270 Hz: 0.5 G^2/Hz 270 - 400 Hz: -8 dB/oct. 400 - 1000 Hz: 0.23 G^2/Hz 1000 – 2000 Hz: -8 dB/oct. (overall: 19.7 grms) Duration: at least 120 seconds	Pass
Shock	1000 G (duration: 1ms) all axes	Pass

$$\alpha = \frac{\Delta m}{A \rho Re \Delta F} \tag{2}$$

Where,

α	:	Sensor sensitivity	[/cm^2/Hz]
Δm	:	Mass loss of the monitor film	[g]
A	:	AO irradiation area	[cm^2]
ρ	:	Polyimide density	[g/cm^3]
Re	:	AO reaction efficiency of polyimide (3.00×10^{-24}) [5]	[cm^3]
ΔF	:	Frequency change	[Hz]

shock tests were conducted. The consumer model passed all the mechanical vibration and shock tests listed in Table 2. As sufficient tolerance is expected from the results, the sensor could be possibly be applied to a flight model for space use.

6. CONCLUSION

The new Twin-CQCM sensor has many advantages such as an easily replaceable sensor and accurate temperature measurement. The sensor is particularly applicable as an AO sensor and for contamination measurement under UV irradiation not needing sensor refreshment. Accurate temperature measurement might also improve measurement at high temperature, given the larger temperature difference between the sensor base and the sensor crystal.

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