



Localised heating effects on AT-cut Quartz Crystal Microbalances: Experimental characterisation through admittance measurements

Chiara Martina^{a, *}, Diego Scaccabarozzi^{a, *}, Bortolino Saggin^b, Emiliano Zampetti^c, Ernesto Palomba^d

^a Politecnico di Milano, Mechanical Engineering Department, Milano, Italy

^b CISAS - Università degli Studi di Padova, Industrial Engineering Department, Padova, Italy

^c Institute of Atmospheric Pollution Research – CNR, Rome, Italy

^d Institute for Space Astrophysics and Planetology – INAF-IAPS, Rome, Italy

ARTICLE INFO

Keywords:

AT-cut quartz crystals

Signal loss

Localised heating performance assessment

Thermal gradient effects

Admittance analyses

Infrared images analyses

ABSTRACT

The research focuses on the experimental characterisation of the effect of localised heating on the sensing area for 5 MHz AT-cut Quartz Crystal Microbalances (QCMs). Through contactless temperature and admittance measurements, the work explores the performance of the QCMs under challenging conditions, up to about 300 °C, and analyses the trends and magnitudes of the admittance modulus and phase changes with respect to temperature. Experimental results show that all tested QCMs experienced irreversible oscillation loss well below the Curie temperature, providing weak oscillation amplitudes beyond 250 °C and a permanent failure before 305 °C, despite maintaining their mechanical integrity; furthermore, it is found that the oscillation loss is independent of any ageing effects. Infrared imaging confirms that surface temperatures never exceed 300 °C, indicating that oscillation loss is driven by thermal gradients rather than intrinsic material limitations. Localised heating also induces sharper resonance frequency shifts at high temperatures and steep thermal gradients, resulting in shifts of the oscillation frequency about 50% higher than what is measured under uniform heating. These findings highlight the impact of non-uniform temperature fields on AT-cut QCMs operation, leading to signal loss that should be overcome by proper accommodation strategies aiming to minimise the thermal gradient during operative conditions.

1. Introduction

1.1. QCMs applications

Quartz crystals are widely spread electronic components that play a significant role in many commercial, industrial and military high-tech systems, valued for their high precision, high quality factor and stability in timing, synchronisation and frequency control [1–3]. These characteristics led the components to be integrated as resonators in high-resolution and precision instrumentation, such as Quartz Crystal Microbalances (QCMs) [4–8]. Quartz Crystal Microbalances are highly sensitive mass measurement devices capable of detecting changes at the nanogram scale, and when integrated with heaters and temperature sensors, they are commonly employed for precise thermogravimetric analyses up to 150 °C, both in ambient and space environments [9–11].

The working principle of the instrument is based on the piezoelectric

properties of the quartz, which vibrates in thickness-shear mode under a controlled electrical field applied by the two-surface electrodes. The mass variation can be related to the measured frequency shifts leveraging Sauerbrey's equation, and with the respective temperature and activation energy for adsorption/desorption of chemical species by the Langmuir equation [12,13]. Quartz Crystal Microbalances are versatile instruments, and their adaptability extends across various domains and across a wide range of disciplines [14,15]. Some examples of their utility include biosensing applications and biomolecular research, where QCMs are used for virus and disease detection and diagnosis, and in general, they are extensively used to investigate interactions at the molecular level, such as protein binding or antigen-antibody recognition, offering real-time and label-free detection [16–20].

Moreover, QCMs contribute significantly to quality control in the food industry, where they are employed to ensure product consistency and safety by analysing moisture content, detecting contaminants and

* Corresponding author.

E-mail address: diego.scaccabarozzi@polimi.it (D. Scaccabarozzi).

<https://doi.org/10.1016/j.sna.2026.117801>

Received 15 December 2025; Received in revised form 1 March 2026; Accepted 30 March 2026

Available online 1 April 2026

0924-4247/© 2026 The Author(s). Published by Elsevier B.V. This is an open access article under the CC BY license (<http://creativecommons.org/licenses/by/4.0/>).

bacteria, such as *Escherichia coli*, and monitoring shelf-life stability [21–24]. Indeed, the quartz microbalances are fundamental devices in gas sensing and humidity control, where they play a critical role in detecting and quantifying gases and volatile compounds through adsorption and desorption processes; characteristics that make the gravimeter invaluable for monitoring hazardous gas emissions, detecting environmental pollution, ensuring industrial safety, and supporting the sustainability control of processes and environmental preservation [25–30].

The importance of contaminant monitoring extends to space applications, where outgassing phenomena can compromise instrument reliability and satellite environments. Stringent space mission and agencies' requirements demand precise measurement systems for characterising chemical contaminants, including volatiles and refractory materials, in both space and ambient conditions. When mass measurement of traditional QCMs is combined with heating and temperature measurement, the devices can be employed as thermogravimeters and provide interesting outcomes in terms of chemical composition of the sample, performing thermo-gravimetric analyses. Thus, microbalances are often used for material characterisation, both for laboratory usage and in-space vacuum conditions [31–33]. Quartz Crystal Microbalances (QCMs) typically operate across a wide range of temperatures, from cryogenic conditions to higher temperatures; most research on thermo-gravimetric systems has focused on relatively low maximum temperatures around 150 °C, with only a limited number of studies investigating QCMs operating at temperatures up to 250 °C [34–36]. These higher temperatures are generally achieved using uniform heating methods without localised temperature control [37–39]. In fact, in current commercial QCMs and most technological implementations, quartz is externally heated, resulting in a nominal uniform temperature field across the device.

A few innovative solutions in the literature propose directly delivering heat to the quartz surface and simultaneously measuring the localised temperature using embedded resistance temperature detectors (RTDs) [40–44]. These integrated systems offer enhanced temperature control over the sensing area, optimising the configuration for acquiring detailed chemical and physical properties of analysed samples. However, localised heating technologies for QCMs operating at high temperatures, up to 400 °C, remain largely unexplored.

Although some research has addressed the high-temperature challenges of various piezoelectric materials, such as the work in [45], which showed the application of GaPO₄ sensors for deposition monitoring, the specific piezoelectric behaviour and response of QCMs under localised high-temperature heating have not yet been characterised or monitored. This gap emphasises the importance of evaluating the behaviour of existing technologies under such extreme conditions and highlights the need to characterise not only the piezoelectric properties of quartz at relatively high temperature conditions, but also its performance under significant thermal gradients, both aspects poorly covered in the literature. Indeed, the application of quartz resonators at elevated temperatures offers several advantages, since higher operating temperatures enhance the cleaning capability of the quartz surface and expand the range of contaminants that can be analysed without the need to change the instrument's material, thus opening different interesting applications either for industrial or space-related research. Indeed, thermo-gravimetric analysis up to 300 °C is essential for studying the initial thermal decomposition of polymeric and carbon-based materials. Above 100 °C, materials undergo desorption of adsorbed compounds, releasing species such as H₂O, CO, SO₂, NO, and NO₂, while resin degradation generates volatile organic compounds (aldehydes, ketones, and carboxylic acids). In graphite oxide-based materials, heating up to 300 °C induces decomposition of oxygenated groups, releasing CO₂ and CO, with SO₂ detection confirming the breakdown of sulphur-containing components [46–48]. For planetary and asteroid missions instead, thermogravimetric sensors capable of operation at high temperatures are essential for quantifying initial volatiles, identifying terrestrial

contamination, and characterising the early decomposition of organic species [49,50]. Moreover, Space agencies, including NASA, ESA, and JAXA, are actively investing in high-temperature QCM technologies to monitor material outgassing, refine testing procedures, and develop validated dynamic outgassing models for long-term missions [51].

1.2. Study objectives

The work aims to assess the output frequency signal of quartz resonators, address critical design challenges at elevated temperatures, and focus on localised power delivery solutions. High thermal loads and gradients can significantly introduce thermo-elastic stress in the quartz component [52–56], and they can impact the behaviour and performance of the instrument, as evidenced by preliminary experimental investigations up to 100 °C [57,58], and numerical analyses up to 250 °C [59].

Although high-temperature QCM applications have been discussed in the literature, most studies rely on external heating elements and sensors rather than integrated components within the quartz itself [60–63]. These advancements in integrated, high-temperature QCM technologies hold the potential to redefine their capabilities for both scientific and practical applications. There remains a knowledge gap regarding the upper temperature limits for current QCM configurations under localised heating conditions. Thus, the work aims to experimentally determine the feasible temperature ranges for QCMs, specifically focusing on the high-temperature-frequency relationship, frequency shift dependence on thermal gradient and the reversible and irreversible behaviours of the electric impedance when subjected to a non-uniform temperature field.

In Section 2, the studied crystals, the methodology, and the test procedures followed are presented. Section 3 shows and discusses experimental results, focusing on the frequency-temperature trend, the electromechanical properties of the system, and the characterisation of the temperature field.

2. Material and methods

2.1. Methodology

The study investigates a 14 mm diameter, 330 µm thick AT-cut quartz crystal disk, with a nominal resonance frequency of approximately 5 MHz. The circular Platinum (Pt) electrodes, which have a diameter of 6 mm and were deposited via Physical Vapor Deposition (PVD) using a Titanium (Ti) as an adhesion layer, are structured with their leads positioned at a 120° angle.

Moreover, Ti-Pt thin-film electrical resistances are deposited on both surfaces, characterised by a nominal thickness up to 400 nm (resulting in electrical resistances of approximately 50 Ω [44]) and functioning either as a heater or as a temperature sensor (RTD). The layout of the microbalance with the deposited electrodes and heater/temperature sensor, designed to provide the best thermal uniformity within the electrodes' areas [43], is shown in Fig. 1.

The numerical evaluation of the thermal gradient and thermoelastic stresses on the quartz disc is conducted using finite element analysis (FEA) based on previously validated finite element models (FEM), which confirms the mechanical integrity of the 5 MHz quartz crystals under elevated temperature conditions [59]. Therefore, an experimental test campaign is performed to monitor the quartz admittance with different localised heating conditions. The tests not only demonstrate the structural resistance of the material but also examine the piezoelectric behaviour under different operating temperature conditions. Three AT-cut 5 MHz quartz microbalances, named hereafter as “QCM₁”, “QCM₂” and “QCM₃”, are selected from the same batch of crystals and subjected to admittance studies at different stable operating temperatures. In particular, the modulus and the phases are analysed in terms of magnitude and resonance shifts. The deposited Pt-Ti thin-films (heater

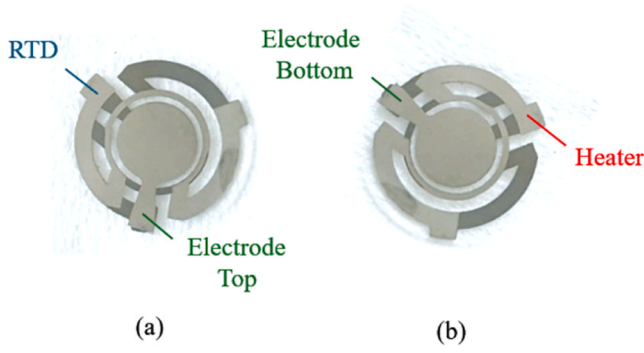


Fig. 1. AT-cut QCM layout: the RTD is deposited on the top surface (a), while the heater is deposited on the bottom surface (b). Both the deposited patterns are identical in shape but rotated by 120° .

and RTD) are previously stabilised and calibrated in a thermal bath (model BK40M-125 Giussani, temperature stability: $\pm 0.02^\circ\text{C}$), enabling accurate localised measurements of temperature by means of the embedded RTDs [64]. It should be noted that, before calibration, all tested crystals are annealed following the thermal treatment described in [64], allowing for the accommodation of any residual stresses in the crystals due to manufacturing and/or the deposition of embedded resistors.

The heating cycles are then repeated on a different micro-balance—named later “QCM₄”—taken from the same production batch of the previous QCMs. This selection was done to further investigate the QCM behaviour with particular attention to highlight any thermal hysteresis effects [65–68].

For each tested temperature condition, the admittance is measured and compared to assess the repeatability and reproducibility of the obtained results. Furthermore, admittance is measured under uniform temperature conditions using a reference QCM (named “QCM_{REF}”),

taken from the same batch as the other items tested in this study. The QCM_{REF} is subjected to uniform heating in controlled temperature conditions (in ambient pressure), by using the crystal holding system in a oven (Binder FD 115, with 1.7°C temperature uniformity). This was deemed necessary to ensure proper temperature uniformity during the testing. The testing allowed for differentiating the effects of temperature from those of localised thermal stress caused by targeted power delivery.

In addition, infrared imaging is used to characterise the temperature field on the quartz surface, quantitatively mapping the thermal gradient on the sensing elements and ensuring that the temperature does not exceed the Curie point (about 573°C [69]), which would compromise the piezoelectric properties of the quartz. Infrared testing under operative conditions is performed on three additional crystals, named “QCM₅”, “QCM₆” and “QCM₇”, selected from the same batch of QCMs. Experimental setups, test protocols and data processing are presented in the following paragraphs.

2.2. Experimental setups description

An overview of the experimental setup for the admittance measurements is presented in Fig. 2. The setup, as illustrated in the black box scheme in Fig. 2(a), features two main interfaces: one for signal acquisition connected to a multiplexer unit (model Keysight 34970 A)—responsible for measuring voltage and current acting on both the deposited heater and RTD, labelled V_{HT} , I_{HT} , V_{RTD} , and I_{RTD} , respectively—and another for power supply (model HP Agilent Keysight E3647A), which provides respectively controlled voltage to the heater and controlled current (I_{RTD}) to the RTD, stabilized at $1.00 \pm 0.05\text{ mA}$. In fact, the setup includes a current generator for evaluating the RTD deposition resistance, and a multimeter in series with the current generator to monitor the generated current (Fig. 2(b)). Then, an impedance analyser (model Keysight E4990A) connected to the electrodes is integrated into the setup to measure and collect the crystal's admittance data. Admittance moduli and phases are acquired via input

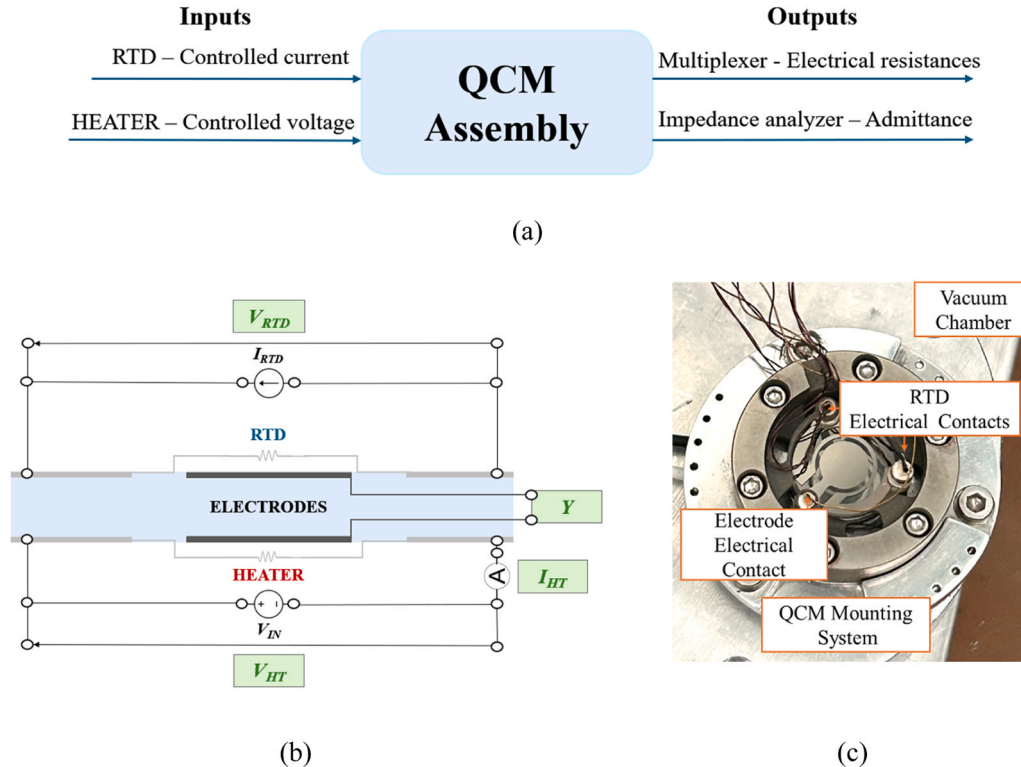


Fig. 2. Experimental setup in vacuum chamber: (a) black-box scheme of the setup; (b) schematic of the electrical circuits – acquired signals; (c) close-up view of the QCM assembly, featuring a three-pin holding system.

sweep signals, whose range covers the resonance shifts of interest, from 4.96 MHz to 5.04 MHz, with 1602 acquired points.

The QCMs are mounted on a compliant support system, developed to assure mechanical resistance of the crystal in challenging thermal and mechanical environments, and validated in previous research activity [40]. The compliant support system featuring three clamping points positioned at 120°, as shown in Fig. 2(c), ensures electrical contact with all deposited elements on the crystal and crystal mechanical support. The QCMs are tested in a vacuum chamber operating at 10^{-3} mbar, to exclude the effect of the environmental pressure variation on the QCMs' output.

The experimental activity oversees a heating test for each QCM following voltage-step changes (V_{IN}) provided to the heaters, allowing for a stable temperature condition on which to perform the admittance measurements. At least ten different temperature conditions are explored for every QCM.

As previously mentioned, a subsequent test is performed using the same setup on QCM₄, taken from the same batch, to evaluate potential performance degradation due to thermal hysteresis. This was achieved by subjecting the QCM₄ to four consecutive thermal cycles ranging between 25°C and about 300°C.

Eventually, a dedicated test session is conducted in air to characterise the temperature field acting on the sensing area and evaluate the thermal gradient across the quartz surface. This is achieved using a thermal imaging infrared camera (model FLIR T640-45), featuring a 640×480 -pixel resolution and a temperature measurement range of -40 °C to 650 °C, allowing contactless measurements with a temperature uncertainty of about 2% of the measured value. The main instrumentation consists of the infrared camera positioned at 40 mm above the QCM assembly, the multiplexer unit (model Keysight 34970 A) to acquire the electrical resistance of the RTD and a power supply (model HP Agilent Keysight E3647A) to apply the thermal load on the crystal. To eliminate unwanted background effects on the measurements, a shield was positioned between the microbalance assembly and the camera to minimise convective and radiative unmeasured disturbances. Three Pt1000 Class A sensors are positioned along the system to monitor the background temperature. The schematic of the measurement system is depicted in Fig. 3.

The testing procedure involves capturing thermal maps at a minimum of four different nominal temperature conditions for each QCM.

2.3. Admittance measurements: theoretical background and data analyses

The impedance properties of an unloaded piezoelectric material oscillator can be modelled by the lumped-parameter Butterworth–Van Dyke equivalent circuit showed in Fig. 4, in accordance with IEEE Standards [64]; the model consists of a series branch —comprising the motional inductance (L), motional capacitance (C), and motional resistance (R) —that simulates the energy conversion and damping associated with the mechanical vibrations of the crystal, modelling the

mechanical oscillation and inherent losses of the crystal are described. In parallel with this series branch is a static capacitance (C_0), which accounts for the capacitance between the crystal's electrodes.

The overall impedance (Z) of the resonator is determined by the parallel combination of the series RLC branch (Z_s) and the impedance of the quartz C_0 (Z_{C_0}) expressed as:

$$Z_s = R + j \left(\omega L - \frac{1}{\omega C} \right) \quad (1)$$

$$Z_{C_0} = \frac{1}{j\omega C_0} \quad (2)$$

At the series resonance frequency (f_s), defined in Eq. 3, the inductive and capacitive reactances cancel each other, producing a minimum impedance that is predominantly resistive. In contrast, the parallel frequency f_p is defined as the frequency at which the impedance reaches its maximum value, corresponding to maximum resistance (Eq. 4).

$$f_s = \frac{1}{2\pi\sqrt{LC}} \quad (3)$$

$$f_p = \frac{1}{2\pi} \sqrt{\frac{1}{LC} - \frac{R^2}{L^2}} \quad (4)$$

Furthermore, for a low-loss material, the resonance frequency (f_r) is characterised by the lowest impedance Z, enabling maximum energy transfer with minimal loss. Conversely, the antiresonance f_a occurs at a higher frequency, where the interaction between the RLC branch and the parallel static capacitance results in a peak of the impedance. Thus, the admittance (Y) - the inverse of the impedance - reaches a minimum, leading to reduced energy transfer.

This leads to considering the series frequency as the resonance frequency, and the parallel as the anti-resonance frequency, based on the low-loss assumption where the resistance is much smaller than the reactive impedance (ωL), resulting in a circuit with a high-quality factor (Q) [70].

The analyses of the admittance of the circuit under different temperature-loading conditions performed in the work are therefore based on the measurement of the series frequency as the maximum of the admittance:

$$f_s \approx f_r, \quad f_r @ \max|Y| \quad (4)$$

2.4. Deposited RTDs calibration and thermal images processing

The electrical resistance of the deposited RTDs on the tested crystals allows for the direct measurement of the localised temperature on the crystals, as shown in Eq. 5.

$$T_{RTD} = \frac{R_{RTD} - R_0}{m} \quad (5)$$

The measurement sensitivity (m) and offset (R_0) are determined during the thin films' calibration phase [40,59] through linear

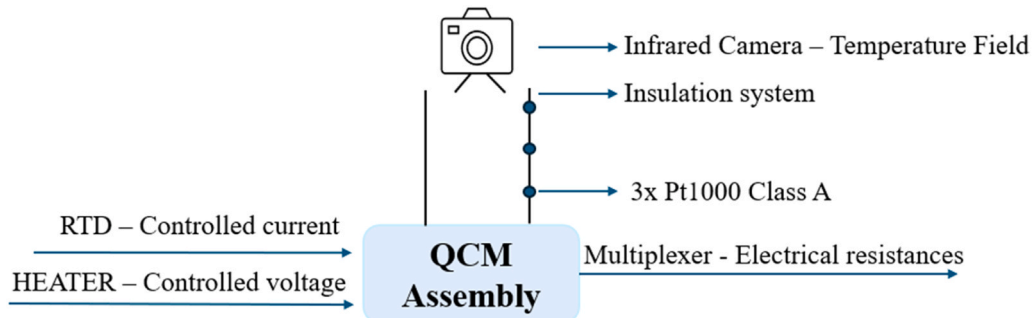


Fig. 3. Schematic of the experimental setup for temperature field evaluation.

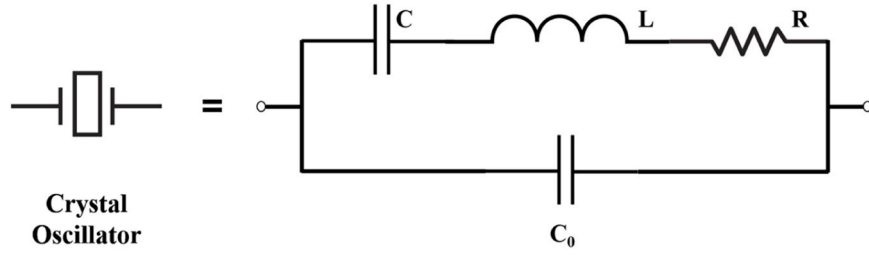


Fig. 4. Butterworth–Van Dyke equivalent circuit.

regression analysis (worst $R^2 \geq 0.997$, considering all the tested QCMs). Table 1 summarises the measured parameters.

Furthermore, to evaluate the thermal gradient on the crystal surface and characterise the temperature fields acting on the QCM under different operating conditions, the acquired thermal maps (e.g., shown in Fig. 5) with unit emissivity are analysed, accounting for the actual emissivity of the quartz (0.55 ± 0.015), which was experimentally determined in [59].

The temperature of the quartz is measured by means of Eq. 6, in which T_{th} is the local temperature read by the thermal imaging camera with unit emissivity, ε_q is the emissivity of the quartz, T_{bk} is the background temperature, i.e. the measured temperature of the testing environment by averaging the three thermistors on the shield (Pt1000 sensors, positioned as illustrated in Fig. 3). Temperatures in Eq. 6 are in K units.

$$\bar{T} = \sqrt[4]{\frac{T_{th}^4 - (1 - \varepsilon_q)T_{bk}^4}{\varepsilon_q}} \quad (6)$$

Then, all the images were analysed by rotating the system (Fig. 6(a)), considering the symmetric plane passing through the electrode (named y-direction) and the normal direction to that plane (named x-direction). Also, the thermal gradient and temperature statistics (maximum and minimum) are calculated for each column of a rectangular matrix (7×15 pixels), selected as a Region of Interest (ROI) for the thermal gradient assessment. The ROI is depicted in Fig. 6(b), specifically selecting the quartz region with the highest temperature near the heater area.

For the thermal gradient computation, a linear regression is applied to each row (horizontal) and column (vertical) within the region of interest. The absolute value of each regression slope corresponds to the gradient component along that direction, and the highest slope value identifies the most critical gradient condition. Moreover, to provide a physical reference, the thermal gradient initially expressed in temperature variations along pixels is converted using a scaling factor of the single pixel area, equal to $0.088 \times 0.088 \text{ mm}^2$, based on the focusing characteristics of the camera. Hence, this allows the thermal gradient magnitude to be expressed in $^\circ\text{C}$ per millimetre ($^\circ\text{C}/\text{mm}$).

Furthermore, the linear regression analysis was performed on both the vertical (column-wise) and horizontal (row-wise) directions of the ROI, aiming to investigate the trend of the temperature moving away from the central heated area.

Table 1

Calibration of the deposited resistors: measurement sensitivity (m) and offset (R_0).

QCM	$m [\Omega \cdot ^\circ\text{C}^{-1}]$	$R_0 [\Omega]$
QCM ₁	0.11384 ± 0.00003	64.912 ± 0.002
QCM ₂	0.09047 ± 0.00008	40.501 ± 0.006
QCM ₃	0.11581 ± 0.00004	51.194 ± 0.003
QCM ₄	0.10724 ± 0.00004	44.973 ± 0.003
QCM ₅	0.12112 ± 0.00005	64.015 ± 0.006
QCM ₆	0.13059 ± 0.00006	62.333 ± 0.005
QCM ₇	0.09321 ± 0.00007	38.952 ± 0.006

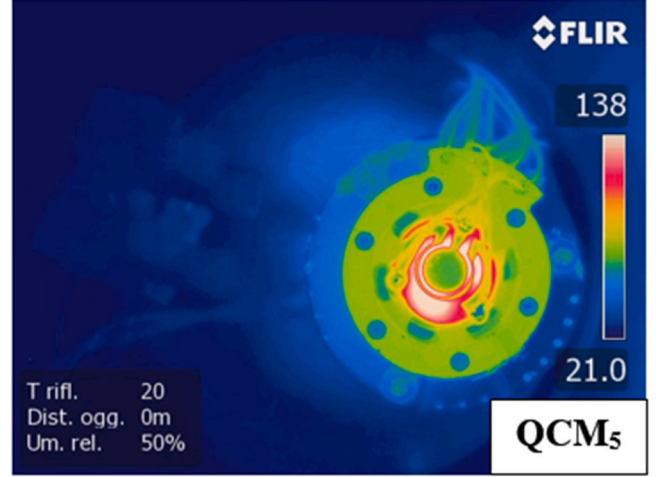


Fig. 5. Thermal image acquired on QCM₅ (unitary emissivity), at $T_{\text{RTD}} = 190.0 \pm 0.1 \text{ }^\circ\text{C}$. Temperatures are in $^\circ\text{C}$ units.

The angular coefficients m_j and m_i of the linear regressions in Eq. 7 and Eq. 8, represent temperature gradient along a specific column j -indexed, and row i -indexed, respectively. In the equations, T_i denotes the temperature at the i^{th} row and T_j the temperature at j^{th} column of the matrix, while x represents the pixel index along the investigated direction.

$$T_j = m_j \cdot x + q_j \quad (7)$$

$$T_i = m_i \cdot x + q_i \quad (8)$$

3. Experimental campaign results

3.1. Admittance measurements

The thermo-electromechanical behaviour of the tested items is shown in the following, where the measured modulus and the phase of the admittances are given as a function of the operating temperature over the sensing area. Specifically, Fig. 7 is related to the first crystal (QCM₁), Fig. 8 to the second tested item (QCM₂), and Fig. 9 refers to QCM₃. It is interesting to notice that for particular ranges of temperature, such as $180 \pm 10 \text{ }^\circ\text{C}$, $245 \pm 5 \text{ }^\circ\text{C}$ and after $270 \text{ }^\circ\text{C}$, the admittance modulus shows two peaks and phase changes. This behaviour is still under investigation and might be related to the coupling of the main share mode with spurious flexural modes.

Moreover, a significant decrease in the peak of the admittance modulus is observed before the complete loss of oscillation, which occurs between $252.9 \text{ }^\circ\text{C}$ and $302.8 \text{ }^\circ\text{C}$ for the tested items. This loss proved to be an irreversible phenomenon, as the crystals no longer vibrated even after being cooled back to ambient conditions. An example of such a loss at $25 \text{ }^\circ\text{C}$ is shown in Fig. 10, where the admittance modulus of QCM₁ no longer exhibits any resonance peaks. The irreversible effect

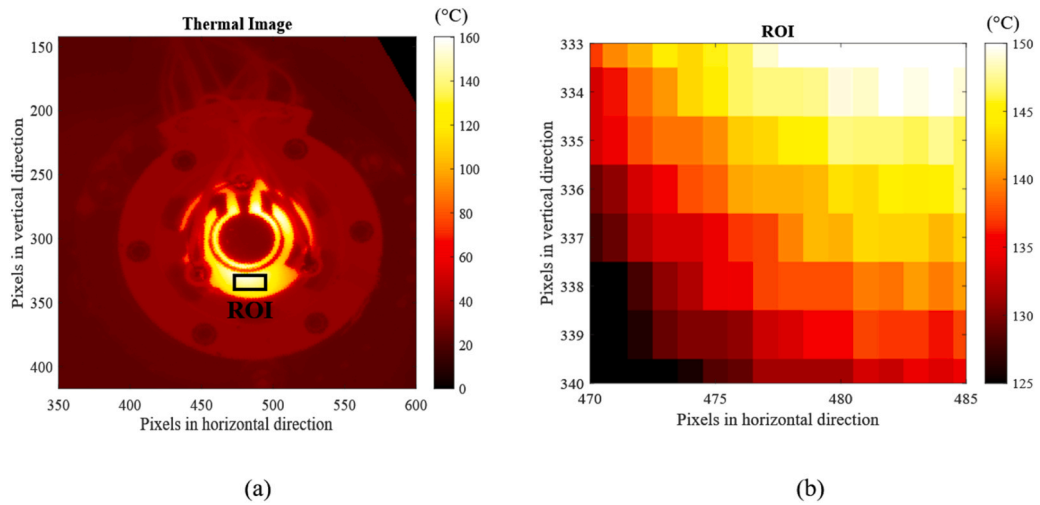


Fig. 6. (a) Rotated image of the temperature field acting on the sensing area, $T_{RTD} = 190.0\text{ }^{\circ}\text{C}$ – QCM₅; (b) ROI for thermal gradient assessment and temperature statistical analysis.

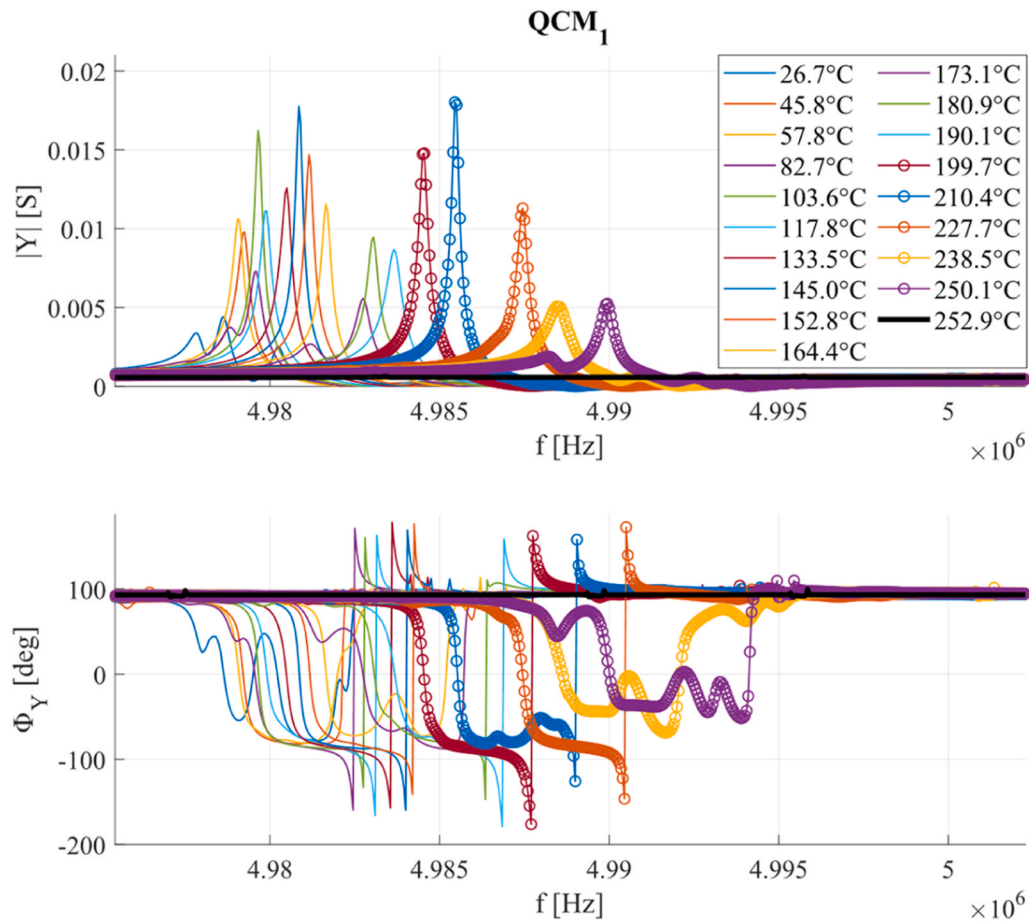


Fig. 7. Acquired admittance magnitude and phase for different temperature conditions (QCM₁).

was verified for all the tested QCMs, confirming that none of them regained vibration performance when heated above the temperature of the loss. Moreover, comparing the obtained results with the measured admittance of the QCM_{REF} in uniform heating conditions, as shown in Fig. 11, it was found that no signal losses were detected either during the heating up to 300 °C or back to ambient temperature. This strengthens the finding that the loss is therefore correlated with the application of

localised thermal load from the heater, rather than the temperature condition itself.

The magnitude of the admittance peaks is analysed as well, searching for any correlation of the crystal quality factor with the temperature. Fig. 12 shows that no correlation exists considering all the available measurements under different conditions. Moreover, the performed analysis highlights a difference between the two found typologies of

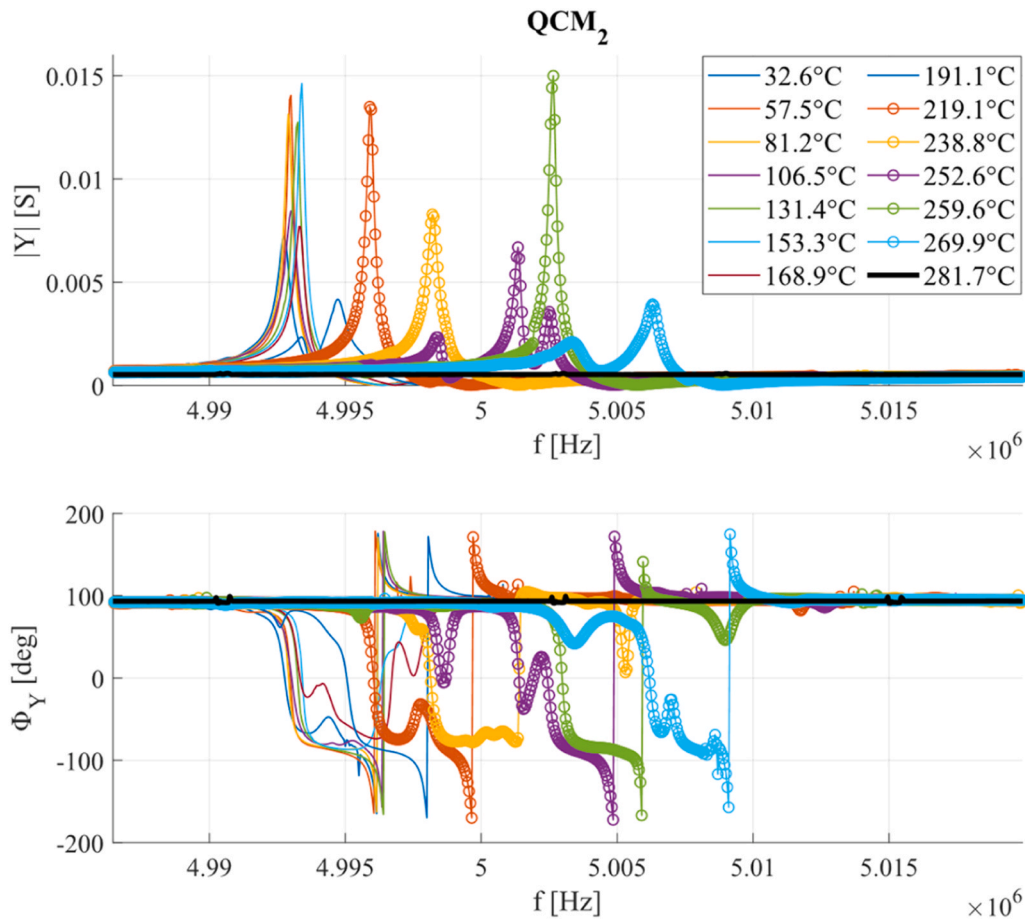


Fig. 8. Acquired admittance magnitude and phase for different temperature conditions (QCM₂).

admittance shapes, i.e. the single peak (SP) in blue colour and the double peak (DP) one in purple, with the relative uncertainty bands indicating the measured variability. The mean values for each group are marked in Fig. 12 by horizontal lines. It can be noticed that, for the temperature conditions where a double-peak admittance is observed, the mean value is systematically lower than for the others. This suggests a present but weaker output signal, as the maximum admittance is directly related to the oscillation efficiency. Measured statistics related to the admittance values are summarised in Table 2, both for the detection of single peaks and double peaks: the maximum admittance values and their respective standard deviations are reported, noting that the double-peak occurred sporadically with respect to the behaviour of the single peak. However, the single peak magnitude is at least 40% higher than the peak when double peaks are detected.

Comparison of the measured averages and related variability in case of single peak admittance trends shows good compatibility: in particular, the mean values for QCM₁ (0.0127 S), QCM₂ (0.0115 S) and QCM₃ (0.0129 S) are all very close to each other, with differences between 0.0002 S and 0.0014 S. Comparing these values with their standard deviations, which are between 0.0033 S and 0.0036 S, it can be stated that the observed differences are within the experimental measured variability.

Moreover, the relative variability, i.e. the ratio of the standard deviation to the mean value (1 σ %), does not exceed 30% considering the complete dataset. The substantial overlap of the uncertainty bands indicates that all three measures are statistically consistent, confirming the overall agreement among the results and the absence of significant incompatibilities between the three conditions, as illustrated in Fig. 13.

The effect of applying a high thermal gradient in high operative temperature led to the upper operative condition limit up to around 250

°C, beyond which irreversible loss of the signal may occur.

Further analyses conducted on QCM₄, aiming to investigate a possible degradation of performance due to thermal hysteresis, show a lack of correlation between the signal loss condition and the number of thermal cycles performed. The performed thermal cycles are illustrated in Fig. 14, where the temperatures measured by the deposited RTD are shown; for each stable condition, the temperatures are evaluated as the mean value of 10 points, providing a relative uncertainty on the average temperature lower than 2% (1 σ band).

The measured admittance on QCM₄ remains largely unchanged, as shown in Fig. 15, where the acquired moduli, recorded before and after three thermal cycles, serve as the baseline for the repeatability measurement before the last cycle, when the permanent signal loss was targeted and achieved. Analysing the repeatability of the modulus shape and magnitudes shown in Figs. 14(a), 14(b) and 14(c), tests performed at ambient, 165 °C and 235 °C, respectively, shapes remain consistent in terms of resonance and antiresonance positions, as well as the number of peaks, confirming the stability of the response. In particular, the modulus varies by less than 30% between the first and last tested conditions, while the frequency shift within the shown interval is below 0.1%.

The admittance moduli and phases related to the last operative cycle for QCM₄ are shown in Fig. 16: the measured trends confirm findings obtained with the previous tests, i.e. 1) no correlation between temperature and admittance peaks, 2) presence of trends with single peak and double peaks within the measured temperature range and 3) irreversible loss of signal loss at temperature lower than Curie limit (for the QCM₄ this occurred at 290.4 ± 0.1 °C). Moreover, it has to be noted that despite what happened in the previous study [59], focusing on thermoelastic modelling and preliminary testing of 10 MHz AT-cut crystals

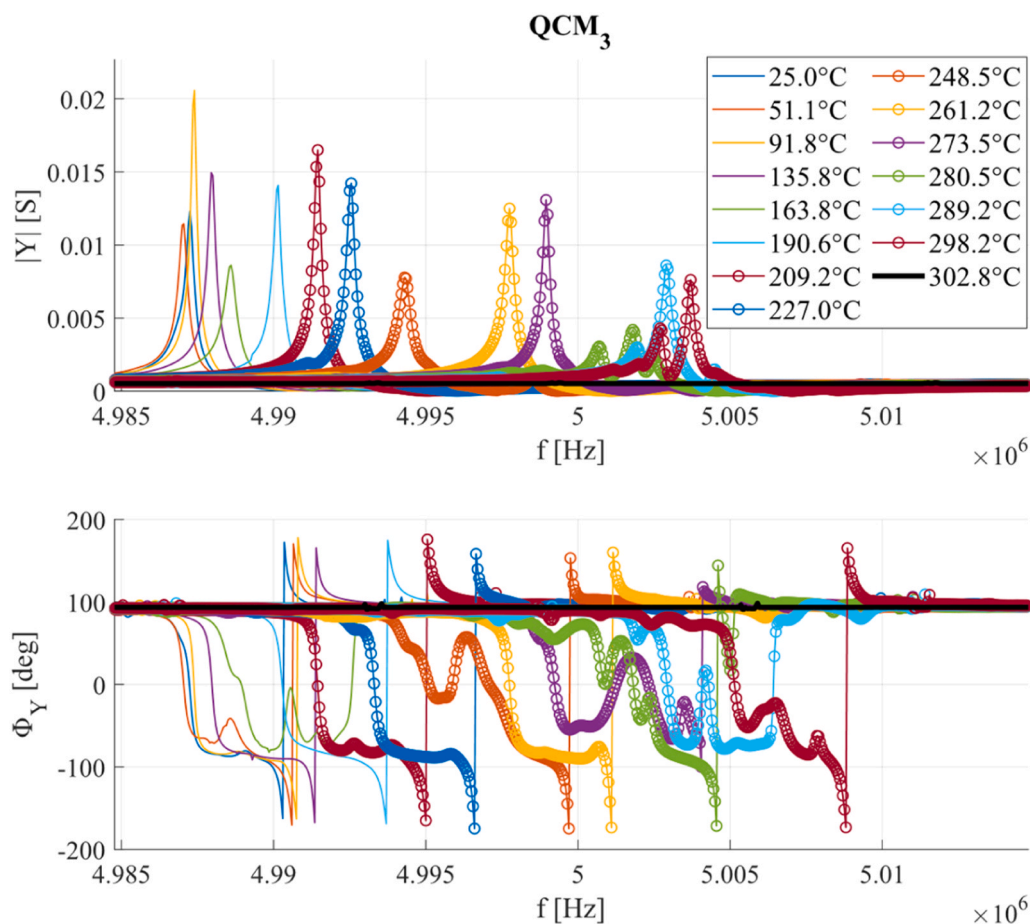


Fig. 9. Acquired admittance magnitude and phase for different temperature conditions (QCM₃).

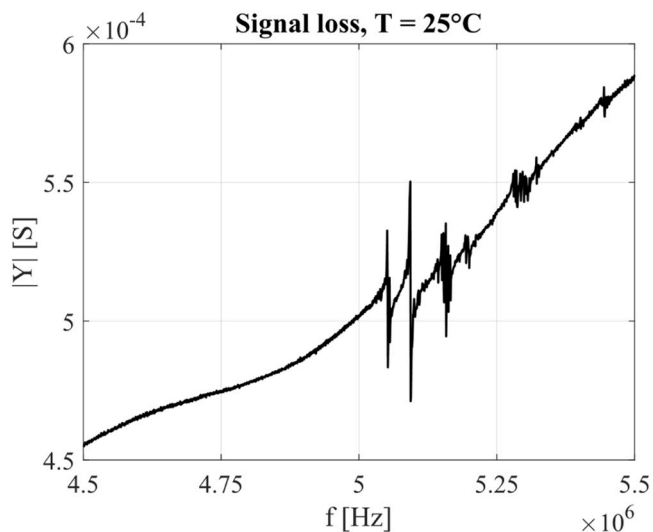


Fig. 10. Example of permanent oscillation loss at ambient temperature – QCM₁ admittance modulus after heating up to 252.9 °C.

with localised heating, mechanical failure was observed, demonstrating that the thermoelastic stress value reaches critical levels for the mechanical resistance of the crystal. This was not observed in the present work, given the higher mechanical resistance thanks to a double thickness of the crystal.

Summarising, the obtained results strengthen the finding that until

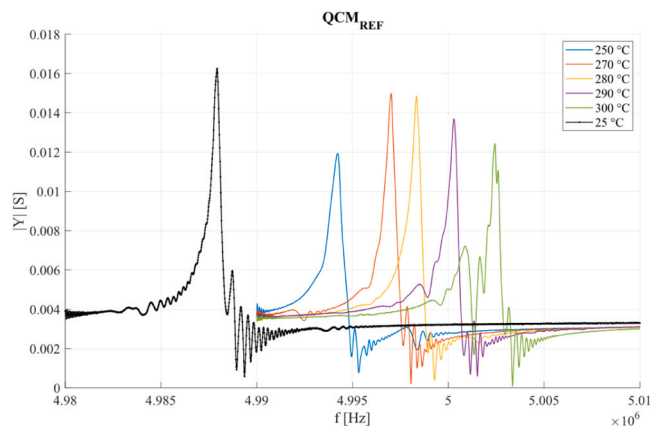


Fig. 11. Acquired admittance magnitude for QCM_{REF} – uniform heating conditions (from 25 °C to 300 °C).

the critical combination between stress and temperature given by the localised heating is not achieved, the AT-cut 5 MHz quartz crystals can be safely operated, showing repeatable behaviour, and allowing for thermo-gravimetric analyses in high temperature conditions.

3.2. QCM's temperature-frequency behaviour

The relation between output frequency and temperature is crucial for the instrument's working principle, as it defines the metrological parameters required for thermogravimetric analysis and the correction

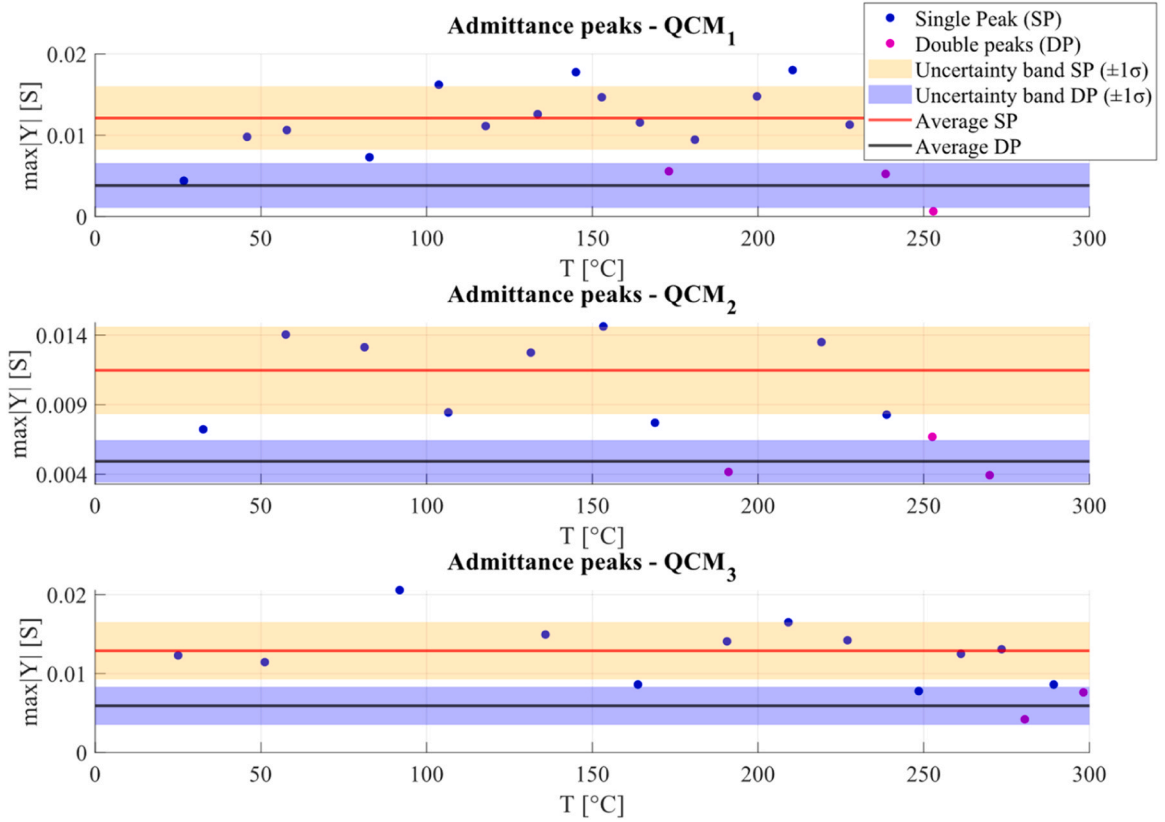


Fig. 12. Measured maximum of the admittance magnitude under varying operative temperature.

Table 2

Mean values and standard deviation of the measured admittance magnitude peaks.

Tested Item	Parameter	Single Peak	Double Peaks	Ratio SP/DP
QCM ₁	Mean value Max Y	0.0127 S	0.0054 S	42%
	1σ	0.0033 S	0.0002 S	
	Mean value Max Y	0.0115 S	0.0049 S	42%
QCM ₂	Mean value Max Y	0.0031 S	0.0015 S	
	1σ	0.00129 S	0.0059 S	46%
	Mean value Max Y	0.0129 S	0.0059 S	
QCM ₃	Mean value Max Y	0.0036 S	0.0024 S	
	1σ			
	Mean value Max Y			

bias to assess the measured frequency shift due to desorption or condensation. For each tested stable temperature condition, the resonance frequency of the tested QCMs (i.e. QCM₁, QCM₂ and QCM₃) is measured by the admittance modulus maximum, and its correlation with the measured temperature on the tested item is analysed. The experimental frequency variations are then compared to the theoretical third-order polynomial curve describing the response of an AT-cut quartz crystal under a uniform temperature field, as given by Eq. 9; the corresponding coefficients are listed in Table 3 below and derived from the literature [71,72]. Further, the table collects the coefficients for the localised heating scenario, computed by means of third-order polynomial best fitting of the acquired experimental data.

$$\frac{\Delta f}{f_0} = a_0 \cdot \Delta T + b_0 \cdot \Delta T^2 + c_0 \cdot \Delta T^3 \quad (9)$$

In Fig. 17 comparison between the theoretical trend of the frequency shifts and the measured ones is presented: the frequency shifts exhibit a clear temperature dependence, properly following a cubic trend

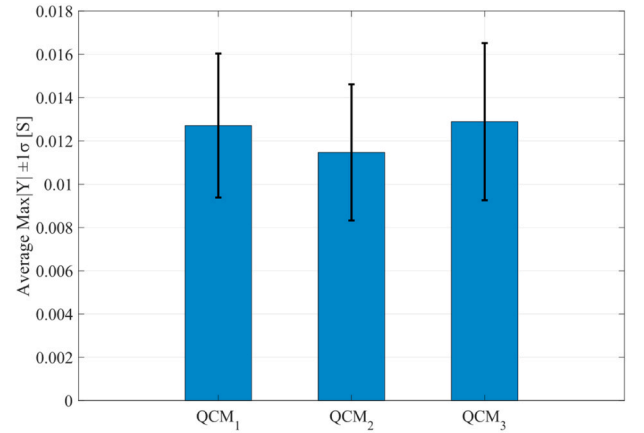


Fig. 13. Measurement compatibility of the admittance maximum magnitudes - single peak conditions.

($R^2=0.9644$). However, when the thermal gradient increases, the frequency variation becomes more pronounced. The thermal gradient across the quartz crystal, along with the resulting thermoelastic stress, alters its standard temperature response. This effect is reflected in the temperature coefficients, leading to deviations from the theoretical model.

Consequently, the average percentage difference between the uniform temperature condition and experimental results is computed as illustrated in Eq. 11, where Δf_{fit} denotes the fitted value through regression and df_{theo} the corresponding theoretical value.

$$\text{Percentage error} = 100 \times \frac{|\Delta f_{\text{fit}} - df_{\text{theo}}|}{\max(|df_{\text{theo}}|)} \quad (11)$$

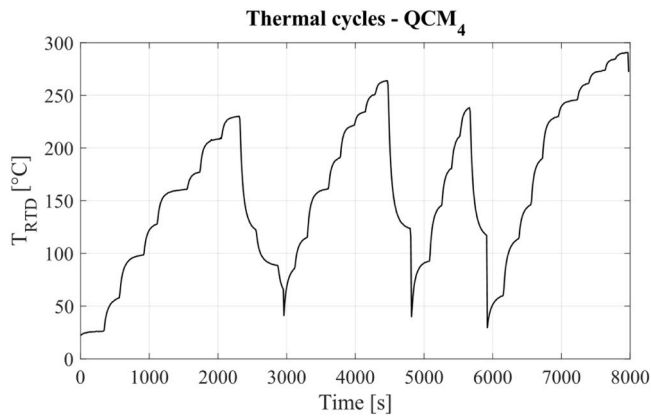


Fig. 14. Measured temperature during thermal cycles (QCM₄).

The error increases, reaching up to 60% at high temperatures (in the range of 280 °C), as shown in Fig. 18. This stresses the need for proper quantification and correction of the thermal gradient instrumental effect when operating with AT-cut crystal microbalances.

3.3. Thermal gradient assessment

The thermal behaviour of the tested microbalances is assessed by analysing temperature variations on the selected QCMs in air. In particular, Fig. 19(a) compares the minimum ($T_{\min}$) and maximum

($T_{\max}$) temperatures observed in the ROI (already corrected for the quartz emissivity) with the measured temperature by the deposited RTD (T_{RTD}), while Fig. 19(b) illustrates the temperature difference ($T_{\max} - T_{\min}$) vs the measured temperature T_{RTD} . The thermal maps of the ROI for two conditions are compared in Fig. 18(c) and Fig. 18 (d), highlighting that the temperature gradient increases with the operative temperature. As shown by the thermal maps and measured temperature differences, the thermal gradient is expected to increase with the average measured temperature T_{RTD} .

Moreover, analysis of the maximum temperature reached before permanent signal loss confirms that, under the tested conditions, none of the microbalances approached critical temperatures, remaining well below the Curie point.

Consequently, the observed admittance signal loss cannot be attributed to any known phase transition of the quartz for the temperature alone, but a likely explanation would be the combined effect of the average temperature of the crystal and the thermal stress due to the thermal gradient for the localised heating. This effect, never documented in the literature, is directly related to the way in which the crystal is heated and can be partially accommodated by designing a new heater shape with a lower thermal gradient on the crystal.

Table 3 reports the angular coefficients for each column for the thermal gradient assessment (for QCM₅). The negative values of the angular coefficients confirm that the temperature always decreases moving away from the central heated area. Moreover, the coefficient of determination (R^2) was evaluated for every regression, and for the thermal gradients along the vertical direction (m_j) high R^2 values are found, confirming a stronger correlation with the mean temperature.

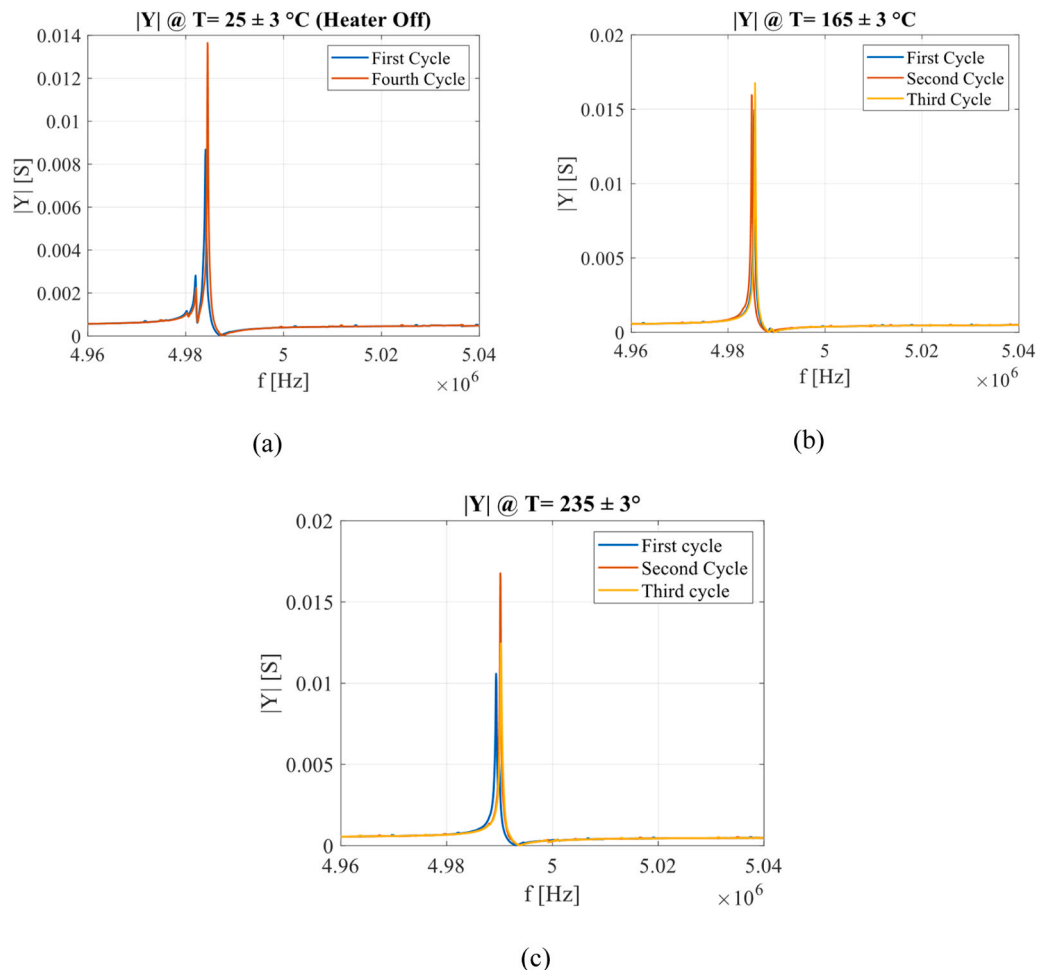


Fig. 15. Repeatability of the admittance modulus - testing on QCM₄. (a) Ambient temperature (heater switched off); (b) 165 °C conditions; (c) 235 °C conditions.

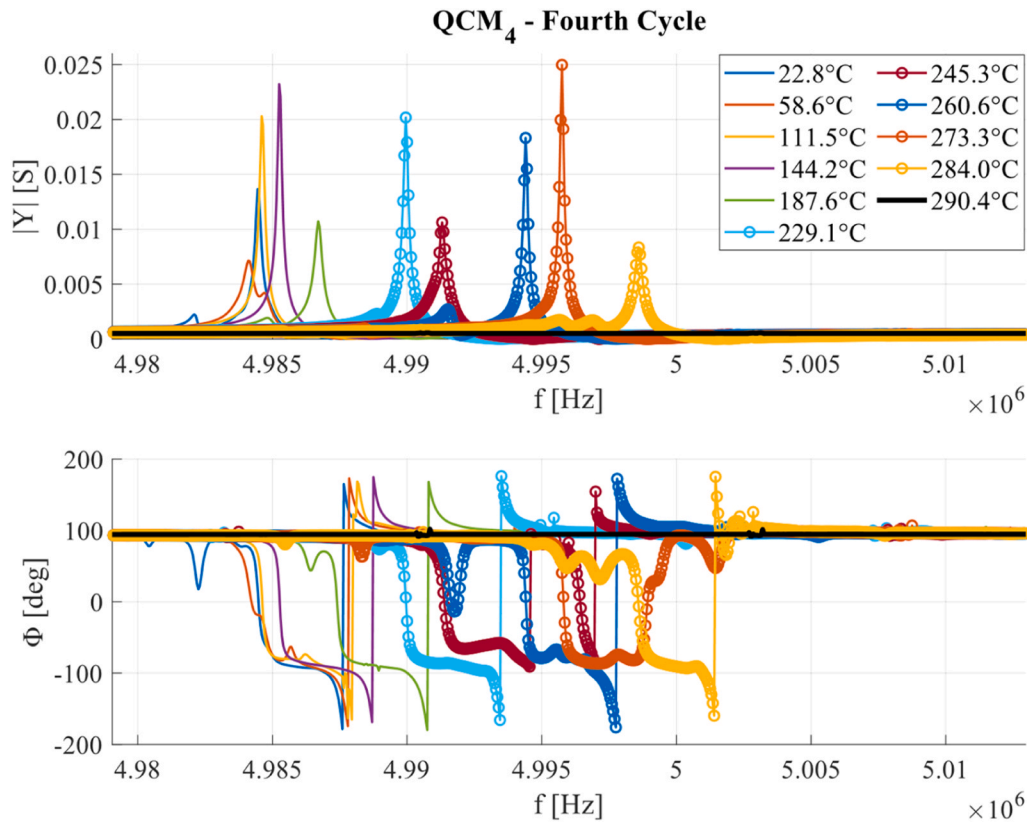


Fig. 16. Acquired admittance for different temperature conditions (QCM₄), fourth cycle with signal loss.

Table 3

Temperature coefficients of frequency for AT-cut quartz. Best fitting with the cubic trend. R^2 equals 0.9644.

	a_0 (10^{-6} K $^{-1}$)	b_0 (10^{-8} K $^{-2}$)	c_0 (10^{-12} K $^{-3}$)
Uniform heating - as in [65, 66]	0	-0.45	108.6
Localized heating (results in this study)	1.63	-3.78	292.2

Table 4

Thermal gradient assessment along vertical direction, $T_{RTD} = 190.0$ °C, QCM₅.

x	m [°C/pixel]	R^2
1	-2.666	0.991
2	-2.290	0.989
3	-2.382	0.979
4	-2.675	0.991
5	-2.272	0.989
6	-2.650	0.997
7	-2.367	0.986
9	-2.467	0.998
10	-2.002	0.977
11	-2.330	0.998
12	-2.265	0.998
13	-1.851	0.984
14	-2.149	0.998
15	-1.783	0.968

Considering the thermal gradient along the rows (m_i), the R^2 calculation showed a lower value with respect to the vertical direction and a lower thermal gradient, about one order of magnitude.

At high temperatures, the thermal gradient becomes increasingly pronounced, in agreement with the large increase observed in the experimental frequency-temperature curve: as illustrated in Fig. 20,

Table 5

Thermal gradient assessment along horizontal direction, $T_{RTD} = 190.0$ °C, QCM₅.

x	m_i [°C/pixel]	R^2
1	-0.336	0.412
2	-0.354	0.446
3	-0.253	0.368
4	-0.213	0.230
5	-0.221	0.300
6	-0.067	0.028
7	-0.104	0.063

showing all the measured maxima of the thermal gradient derived from the infrared mapping, grouping data from testing QCM₅, QCM₆ and QCM₇: measured thermal gradients are consistent among the tested items, proving the robustness of the implemented procedure, and showing values up to 70 °C/mm.

Modelling of the thermal gradient with a quadratic function is proposed as shown in Fig. 19, resulting in an R^2 set to 0.96 and the following expression: here, the quadratic regression was performed on the thermal gradient data, with the additional constraint that the gradient is zero at ambient temperature ($T_{RTD} = 25$ °C). The remaining parameters were determined via least-squares fitting, yielding a model that accurately describes the temperature dependence of the gradient ($R^2 \approx 0.958$), within the operative temperature range of interest.

$$G_{\max} = 7.93 \cdot 10^{-4} \cdot T_{RTD}^2 + 4.17 \cdot 10^{-2} \cdot T_{RTD} - 1.54 \quad \left(\frac{^\circ\text{C}}{\text{mm}} \right) \quad (11)$$

4. Conclusions

The study evaluated the performance of 5 MHz AT-cut quartz crystal microbalances under localised heating. By analysing the resonance frequency, admittance modulus, and their variations with temperature, the

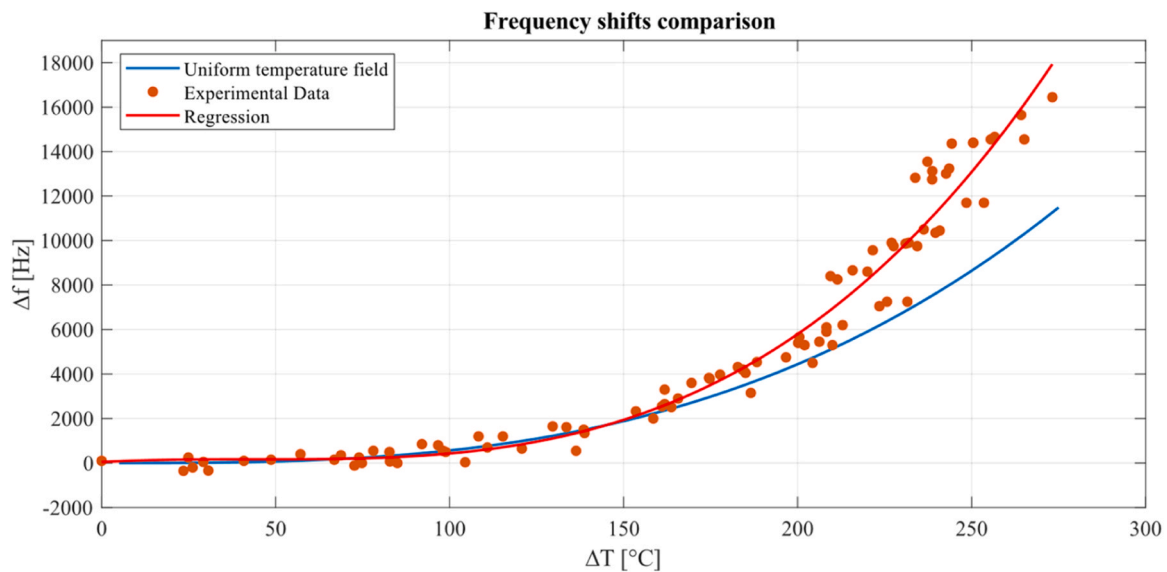


Fig. 17. Frequency shifts under different temperature fields.

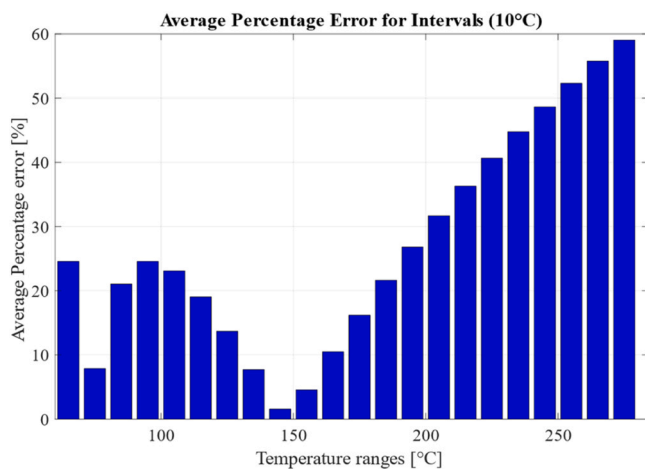


Fig. 18. Average percentage error (10 °C temperature ranges) between theoretical and experimental models.

study identified performance and upper operational temperature limits. The obtained results revealed that all tested microbalances ceased oscillation irreversibly well below the Curie temperature, with weak signal admittance magnitudes measured after 250 °C and a permanent signal loss occurring even before 305 °C, despite maintaining their mechanical integrity. Infrared imaging confirmed that, under the tested operating conditions, the crystal surface temperature never exceeded the Curie point. This supports the conclusion that oscillation loss is not directly determined by the intrinsic material limit but rather arises from the combined effects of the applied thermal load, the resulting thermal gradient, and associated thermal stresses, which can lead to permanent failure. This was confirmed by measurements on a reference quartz crystal microbalance from the same production batch, heated under uniform conditions up to 300 °C, which did not show any signal losses.

Furthermore, while the magnitude of the admittance modulus maxima did not exhibit a clear correlation with temperature, a consistent decrease was observed in cases where multiple peaks appeared. Additional analyses demonstrated that the loss of oscillation was not dependent on the number of thermal cycles, indicating that thermal hysteresis did not play a relevant role in performance degradation.

As a side result, the activity allowed evaluating the experimental

frequency-temperature curve for the 5 MHz quartz crystal microbalances, highlighting that sharper frequency variations in high-temperature and high thermal gradient conditions are expected, comparing the experimental results with the standard reference. In general, about a 50% shift of the resonance was found with respect to a uniform heating scenario, confirming also preliminary results presented in literature on similar microbalances. Moreover, double admittance peaks were detected as well, suggesting a mode splitting phenomenon, to be addressed in future research activity, due to the possible impact on the accuracy of microbalances operated in double crystal configuration.

The obtained findings highlight the critical impact of non-uniform temperature fields on AT-cut quartz crystal microbalances' operation, which induce mechanical constraints and energy dissipation mechanisms leading to premature and irreversible vibration suppression. This emphasises the need for further research, e.g. exploring new heater designs to mitigate the observed instrumental effects but extending QCM operability at high-temperature ranges, assessing possible ageing phenomena (either at macro or micro levels) by dedicated mechanical testing, and additional cross-validation activities, either through contactless localised heating or by recording synchronised thermoelastic stress maps with the related admittance measurements. Eventually, similar studies on different crystal cuts (e.g. IT cut), theoretically less sensitive to temperature changes in uniform conditions, but not studied yet with localised heating, might be performed, unlocking the possibility of chemical and thermo-gravimetric analyses in high-temperature operative conditions.

CRedit authorship contribution statement

Emiliano Zampetti: Writing – review & editing, Resources, Methodology, Investigation. **Ernesto Palomba:** Writing – review & editing, Methodology, Investigation. **Chiara Martina:** Writing – review & editing, Writing – original draft, Validation, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Diego Scaccabarozzi:** Writing – review & editing, Validation, Supervision, Methodology, Investigation, Formal analysis, Conceptualization. **Bortolino Saggin:** Writing – review & editing, Methodology, Investigation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence

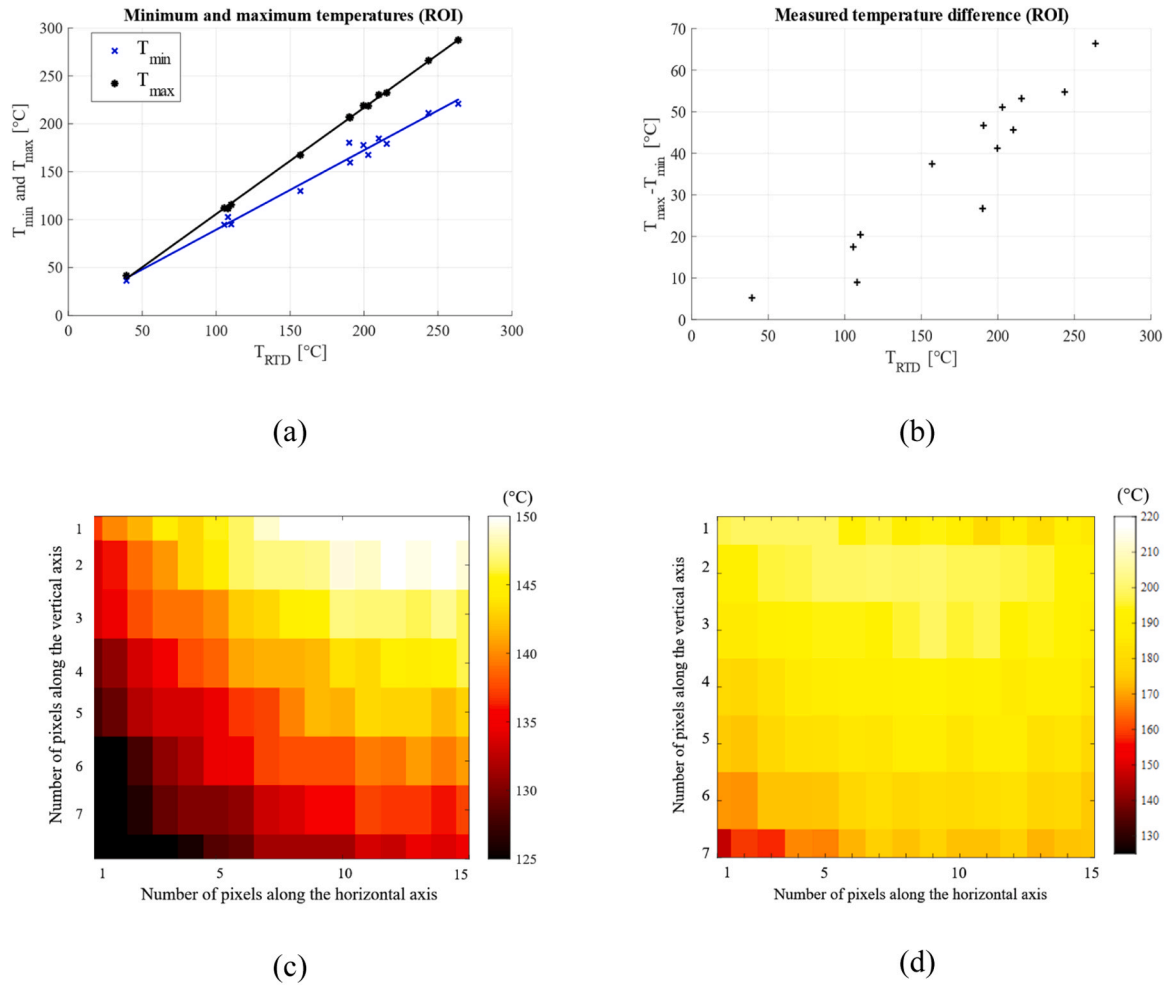


Fig. 19. Measured minimum, maximum temperatures (a) and temperature ranges (c) in different operative conditions, corrected by quartz emissivity ($\epsilon=0.55$); (c) ROI thermal map for $T_{RTD}=190^{\circ}C$ ($\epsilon=1$) and (d) ROI thermal map for $T_{RTD}=245^{\circ}C$ ($\epsilon=1$).

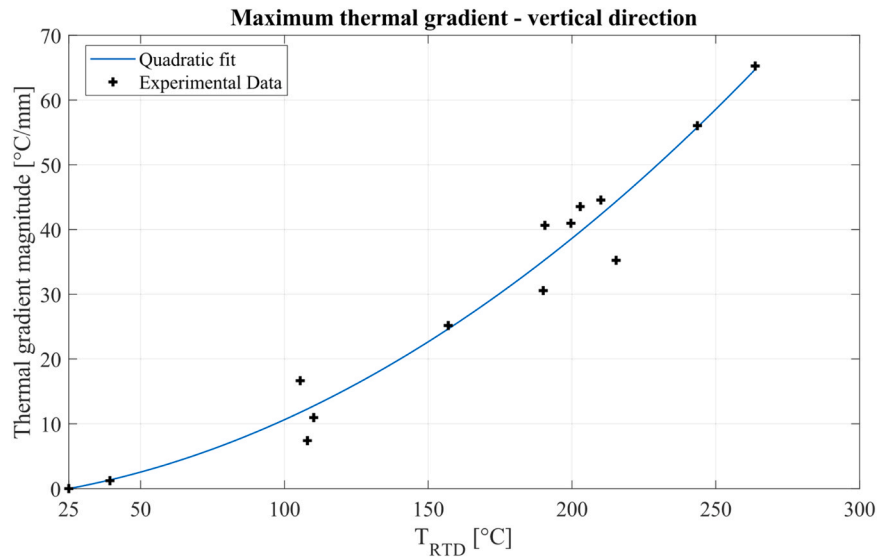


Fig. 20. Maximum absolute magnitude of the thermal gradient in the vertical direction under different operating conditions. All the measured infrared maps for QCM₅, QCM₆ and QCM₇ are grouped.

the work reported in this paper.

Data Availability

Data will be made available on request.

References

- [1] A. Shapira, et al., An ultra-stable oscillator for the 3GM experiment of the JUICE mission (York, UK), *Eur. Freq. Time Forum (EFTF)* 2016 (2016) 1–5, <https://doi.org/10.1109/EFTF.2016.7477766>.
- [2] J.R. Norton, J.M. Cloeren, Precision quartz oscillators and their use aboard satellites, *Johns. Hopkins APL Tech. Dig.* 15 (1) (1994) 30–37.
- [3] C.S. Lam, A review of the recent development of MEMS and crystal oscillators and their impacts on the frequency control products industry (Beijing, China), 2008 IEEE Ultrason. Symp. (2008) 694–704, <https://doi.org/10.1109/ULTSYM.2008.0167>.
- [4] F. Pascal-Delannoy, B. Sorli, A. Boyer, Quartz crystal microbalance (QCM) used as humidity sensor, *Sens. Actuators A Phys.* 84 (3) (2000) 285–291, [https://doi.org/10.1016/S0924-4247\(00\)00391-5](https://doi.org/10.1016/S0924-4247(00)00391-5).
- [5] C. Lu, A.W. Czanderna, *Applications of Piezoelectric Quartz Crystal Microbalances*, Elsevier, Amsterdam, 1984.
- [6] M.A. Mohammadzadeh Kashan, V. Kalavally, N. Ramakrishnan, Sensing film-coated QCM coupled resonator sensors: approach, fabrication, and demonstration, *Sens. Actuators A Phys.* 274 (2018) 64–72, <https://doi.org/10.1016/j.sna.2018.03.006>.
- [7] L. Rodríguez-Pardo, A.M. Cao-Paz, J. Fariña, Design and characterization of an active bridge oscillator as a QCM sensor for the measurement of liquid properties and mass films in damping media, *Sens. Actuators A Phys.* 276 (2018) 144–154, <https://doi.org/10.1016/j.sna.2018.04.012>.
- [8] X. Huang, Q. Chen, W. Pan, Y. Yao, Advances in the mass sensitivity distribution of quartz crystal microbalances: a review, *Sensors* 22 (14) (2022) 5112, <https://doi.org/10.3390/s22145112>.
- [9] N. Debabhuti, S. Neogi, S. Mukherjee, A. Dhar, P. Sharma, R.L. Vekariya, M. Poddar Sarkar, B. Tudu, N. Bhattacharyya, R. Bandyopadhyay, M. Muddassir, Development of QCM sensor to detect α -terpinyl acetate in cardamom, *Sens. Actuators A Phys.* 319 (2021) 112521, <https://doi.org/10.1016/j.sna.2020.112521>.
- [10] E. Zampetti, M.A. Mancuso, A. Capocercera, P. Papa, A. Macagnano, Improving aerosol characterization using an optical particle counter coupled with a quartz crystal microbalance with an integrated micro-heater, *Sensors* 24 (2024) 2500, <https://doi.org/10.3390/s24082500>.
- [11] F. Dirri, E. Palomba, A. Longobardo, E. Zampetti, B. Saggin, D. Scaccabarozzi, A review of quartz crystal microbalances for space applications, *Sens. Actuators A Phys.* 287 (2019) 48–75, <https://doi.org/10.1016/j.sna.2018.12.035>.
- [12] G.Z. Sauerbrey, Use of quartz vibration for weighing thin films on a microbalance, *Physik* 155 (1959) 206–212.
- [13] I. Langmuir, The vapour pressure of metallic tungsten, *Phys. Rev.* 2 (1913) 329–342, <https://doi.org/10.1103/PhysRev.2.329>.
- [14] L. Wang, J. Song, C. Yu, The utilization and advancement of quartz crystal microbalance (QCM): A mini review, *Microchem. J.* 199 (2024) 109967, <https://doi.org/10.1016/j.microc.2024.109967>.
- [15] S. Na Songkhla, T. Nakamoto, Overview of quartz crystal microbalance behavior analysis and measurement, *Chemosensors* 9 (2021) 350, <https://doi.org/10.3390/chemosensors9120350>.
- [16] A. Afzal, A. Mujahid, R. Schirhagl, S.Z. Bajwa, U. Latif, S. Feroz, Gravimetric viral diagnostics: QCM-based biosensors for early detection of viruses, *Chemosensors* 5 (2017) 7, <https://doi.org/10.3390/chemosensors5010007>.
- [17] Y. Montagut, J. Garcia, Y. Jiménez, C. March, Á. Montoya, A. Arnau, QCM technology in biosensors, *Biosens. Emerg. Mater. Appl.* (2011) 153–178.
- [18] A. Juste-Dolz, W. Teixeira, Y. Pallás-Tamarit, et al., Real-world evaluation of a QCM-based biosensor for exhaled air, *Anal. Bioanal. Chem.* 416 (2024) 7369–7383, <https://doi.org/10.1007/s00216-024-05407-5>.
- [19] F. Bonyadi, M. Kavruk, S. Ucak, B. Cetin, G. Bayramoglu, A.D. Dursun, V.C. Ozalp, Real-time biosensing bacteria and viruses with quartz crystal microbalance: recent advances, opportunities, and challenges, *Crit. Rev. Anal. Chem.* 54 (2023) 2888–2899, <https://doi.org/10.1080/10408347.2023.2211164>.
- [20] M. Rodríguez-Torres, V. Altuzar, C. Mendoza-Barrera, G. Beltrán-Pérez, J. Castillo-Mixcoatl, S. Muñoz-Aguirre, Acetone detection and classification as biomarker of diabetes mellitus using a quartz crystal microbalance gas sensor array, *Sensors* 23 (2023) 9823, <https://doi.org/10.3390/s23249823>.
- [21] G.B. Eshun, H.A. Crapo, I. Yazgan, L. Cronmiller, O.A. Sadik, Sugar–lectin interactions for direct and selective detection of *Escherichia coli* bacteria using QCM biosensor, *Biosensors* 13 (2023) 337, <https://doi.org/10.3390/bios13030337>.
- [22] H.A. Huellemeyer, N.M. Eren, T.D. Payne, Z.D. Schultz, D.R. Heldman, Monitoring and characterization of milk fouling on stainless steel using a high-pressure high-temperature quartz crystal microbalance with dissipation, *Langmuir* 38 (2022) 9466–9480, <https://doi.org/10.1021/acs.langmuir.2c00419>.
- [23] M. Forinová, et al., A reusable QCM biosensor with stable antifouling nano-coating for on-site reagent-free rapid detection of *E. coli* O157:H7 in food products, *Food Control* 165 (2024) 110695.
- [24] F. Deng, W. Chen, J. Wang, Z. Wei, Fabrication of a sensor array based on quartz crystal microbalance and the application in egg shelf life evaluation, *Sens. Actuators B Chem.* 265 (2018) 394–402.
- [25] L. Wang, Metal-organic frameworks for QCM-based gas sensors: a review, *Sens. Actuators A Phys.* 307 (2020) 111984, <https://doi.org/10.1016/j.sna.2020.111984>.
- [26] Y. Ishiguro, T. Nishitani, C. Li, K. Hirakuri, A graphitic carbon nitride-coated quartz crystal microbalance gas sensor for H₂ detection, *J. Mater. Chem. C* 11 (2023) 10178–10184, <https://doi.org/10.1039/D3TC00870C>.
- [27] E. Zampetti, M.A. Mancuso, F. Dirri, et al., Effects of oscillation amplitude variations on QCM response to microspheres of different sizes, *Sensors* 23 (2023) 5682, <https://doi.org/10.3390/s23125682>.
- [28] Z. Golshadi, M. Dinari, A. Knebel, J. Lützenkirchen, B.H. Monjezi, Metal-organic and covalent organic framework-based QCM sensors for environmental pollutant detection and beyond, *Coord. Chem. Rev.* 521 (2024) 216163, <https://doi.org/10.1016/j.ccr.2024.216163>.
- [29] A. Bearzotti, A. Macagnano, P. Papa, I. Venditti, E. Zampetti, A study of a QCM sensor based on pentacene for the detection of BTX vapors in air, *Sens. Actuators B Chem.* 240 (2017) 1160–1164, <https://doi.org/10.1016/j.snb.2016.09.097>.
- [30] E. Zampetti, A. Macagnano, P. Papa, A. Bearzotti, F. Petracchini, L. Paciucci, N. Pirrone, Exploitation of an integrated microheater on QCM sensor in particulate matter measurements, *Sens. Actuators A Phys.* 264 (2017) 205–211, <https://doi.org/10.1016/j.sna.2017.08.004>.
- [31] D. Lansade, J.F. Roussel, D. Faye, et al., Contamination level prediction: progress in species separation by TGA/MS, *IOP Conf. Ser. Mater. Sci. Eng.* 1287 (2023) 012022, <https://doi.org/10.1088/1757-899X/1287/1/012022>.
- [32] A. Suliga, O. Ergincan, R. Rampini, Modeling of spacecraft outgassed contamination levels by thermogravimetric analysis, *J. Spacecr. Rockets* 58 (2021) 1010–1016, <https://doi.org/10.2514/6.2021-135020>.
- [33] W.M. Grundy, S.C. Tegler, J.K. Steckloff, et al., Laboratory measurement of volatile ice vapor pressures with a quartz crystal microbalance, *Icarus* 410 (2024) 115767, <https://doi.org/10.1016/j.icarus.2023.115767>.
- [34] D. Scaccabarozzi, et al., Design of VISTA, a quartz crystal thermogravimetric analyzer for Hera mission, in: *IEEE 9th International Workshop on Metrology for Aerospace (MetroAeroSpace)*, Pisa, Italy, 2022, 2022, pp. 28–32, <https://doi.org/10.1109/MetroAeroSpace54187.2022.9856401>.
- [35] D. Scaccabarozzi, F. Dirri, E. Palomba, et al., Feasibility design of DIANA, a dust analyzer developed for the Tianwen-2 mission, in: *11th International Workshop on Metrology for Aerospace (MetroAeroSpace)*, Lublin, Poland, 2024, 2024, pp. 422–426, <https://doi.org/10.1109/MetroAeroSpace61015.2024.10591512>.
- [36] C. Martina, et al., DIANA, a cometary dust in-situ analyzer for Tianwen-2 mission: Thermomechanical design, *IAC-24-A3. 4B* 10 (2024), <https://doi.org/10.52202/078357-0084>.
- [37] H.A. Huellemeyer, T.D. Payne, Z.D. Schultz, D.R. Heldman, Assessing the scalability of the high-pressure high-temperature quartz crystal microbalance with dissipation (HPHT QCM-D) for milk fouling studies, *ACS Food Sci. Technol.* 3 (2023) 1216–1228.
- [38] S.A. Zhgoon, A.S. Shvetsov, S.A. Sakharov, O. Elmazria, High-temperature SAW resonator sensors: electrode design specifics, *IEEE Trans. Ultrason. Ferroelectr. Freq. Control* 65 (2018) 657–664, <https://doi.org/10.1109/TUFFC.2018.2797093>.
- [39] V.M. Mecea, J.O. Carlsson, P. Heszler, M. Bärtn, Development and testing of a high-temperature quartz crystal microbalance, *Vacuum* 46 (1995) 691–694, [https://doi.org/10.1016/0042-207X\(94\)00147-2](https://doi.org/10.1016/0042-207X(94)00147-2).
- [40] D. Scaccabarozzi, F. Dirri, A. Longobardo, et al., Characterization of thermally controlled quartz crystal microbalances, in: *IEEE Metrology for Aerospace (MetroAeroSpace)*, Florence, Italy, 2016, 2016, pp. 614–618, <https://doi.org/10.1109/MetroAeroSpace.2016.7573287>.
- [41] A. Zinzi, E. Palomba, F. Dirri, D. Scaccabarozzi, Exploring the feasibility of volatile desorption studies by means of a quartz crystal microbalance with an integrated micro-heater, *Sens. Actuators A Phys.* 172 (2011) 504–510.
- [42] J. Sakai, T. Morishita, T. Yamazaki, T. Sato, Microheater-integrated quartz crystal microbalance array for thermal desorption spectroscopy, *IEEJ Trans. Sens. Micro* 135 (2015) 112–113.
- [43] D. Scaccabarozzi, F. Dirri, A. Longobardo, et al., Thermo-mechanical design and testing of a microbalance for space applications, *Adv. Space Res.* 54 (2014) 2386–2397.
- [44] C. Martina, R. Sala, G. Moschioni, B. Saggin, E. Zampetti, E. Palomba, D. Scaccabarozzi, Temperature Measurement Accuracy of Thin-Film Heaters for Quartz Crystal Microbalances. 2025 IEEE 12th International Workshop on Metrology for AeroSpace (MetroAeroSpace), IEEE, 2025, June, pp. 800–805.
- [45] J.W. Elam, M.J. Pellin, GaPO₄ sensors for gravimetric monitoring during atomic layer deposition at high temperatures, *Anal. Chem.* 77 (11) (2005) 3531–3535, <https://doi.org/10.1021/ac050348e>.
- [46] Célia Lourenço, et al., Instrumentation for quantitative analysis of volatile compounds emission at elevated temperatures. Part 2: analysis of carbon fibre reinforced epoxy composite, *Sci. Rep.* 10 (1) (2020) 8702.
- [47] Y.G. Kryazhev, I.V. Anikeeva, M.V. Trenikhin, T.I. Gulyaeva, V.P. Melnikov, V. A. Likholtobov, O.B. Belskaya, Porous carbon-carbon composite materials obtained by alkaline dehydrochlorination of polyvinyl chloride (Art. no), *Materials* 15 (21) (2022) 7636, <https://doi.org/10.3390/ma15217636>.
- [48] H. Li, T. Li, T. Zhang, J. Zhu, W. Deng, D. He, Construction and adsorption performance study of GO-CNT/activated carbon composites for high efficient adsorption of pollutants in wastewater (Art. no), *Polymers* 14 (22) (2022) 4951, <https://doi.org/10.3390/polym14224951>.
- [49] S.P. Verevkin, R. v Ralys, D.H. Zaitsau, V.N. Emel'yanenko, C. Schick, Express thermo-gravimetric method for the vaporization enthalpies appraisal for very low volatile molecular and ionic compounds, *Thermochim. Acta* 538 (2012) 55–62, <https://doi.org/10.1016/j.tca.2012.03.018>.

- [50] M.M. Grady, I.P. Wright, Elemental and isotopic abundances of carbon and nitrogen in meteorites, *Space Sci. Rev.* 106 (1/4) (2003) 231–248, <https://doi.org/10.1023/A:1024645906350>.
- [51] M. Orlandi et al., DOK: THE NEW ESA DYNAMIC OUTGASSING HIGH TEMPERATURE FACILITY.
- [52] J. Wang, J.D. Yu, Y.K. Yong, et al., A finite element analysis of frequency–temperature relations of AT-cut quartz crystal resonators with higher-order Mindlin plate theory, *Acta Mech.* 199 (2008) 117–130, <https://doi.org/10.1007/s00707-007-0538-5>.
- [53] J. Wang, J. Du, L. Shen, Z. Yang, The thermal effect of metal electrodes on thickness-shear vibrations of crystal plates, *IEEE Trans. Ultrason. Ferroelectr. Freq. Control* 54 (11) (2007) 2331–2336, <https://doi.org/10.1109/TUFFC.2007.537>.
- [54] R.D. Mindlin, Frequencies of piezoelectrically forced vibrations of electrode, doubly rotated, quartz plates, *Int. J. Solids Struct.* 20 (1984) 141–157.
- [55] P.C.Y. Lee, Y.K. Yong, Frequency–temperature behavior of thickness vibrations of doubly rotated quartz plates affected by plate dimensions and orientations, *J. Appl. Phys.* 60 (7) (1986) 2327–2342.
- [56] Timoshenko, S.P., Goodier, J.W., 1970. *Theory of Elasticity*. 3rd ed., International Student Edition. McGraw-Hill, New York, p. 122.
- [57] M. Magni, D. Scaccabarozzi, E. Palomba, E. Zampetti, B. Saggini, Characterization of thermal gradient effects on a quartz crystal microbalance, *Sensors* 22 (2022) 7256, <https://doi.org/10.3390/s22197256>.
- [58] M. Magni, D. Scaccabarozzi, B. Saggini, Compensation of thermal gradient effects on a quartz crystal microbalance, *Sensors* 23 (2023) 24, <https://doi.org/10.3390/s23010024>.
- [59] C. Martina, B. Saggini, E. Palomba, E. Zampetti, M.A. Mancuso, D. Scaccabarozzi, Finite element modelling of thermoelastic behavior for high-temperature quartz crystal microbalance. 2023 IEEE 10th International Workshop on Metrology for Aerospace (MetroAeroSpace), Milan, Italy, 2023, pp. 109–113, <https://doi.org/10.1109/MetroAeroSpace57412.2023.10190026>.
- [60] R. Stadlmayr, P.S. Szabo, H. Biber, H.R. Koslowski, E. Kadletz, C. Cupak, R. A. Wilhelm, M. Schmid, C. Linsmeier, F. Aumayr, A high-temperature dual-mode quartz crystal microbalance technique for erosion and thermal desorption spectroscopy measurements, *Rev. Sci. Instrum.* 91 (12) (2020) 125104, <https://doi.org/10.1063/5.0012028>.
- [61] W. Ding, et al., Investigation of high-temperature and high-pressure gas adsorption in zeolite H-ZSM-5 via the langatate crystal microbalance: CO₂, H₂O, methanol, and dimethyl ether, *J. Phys. Chem. C* 119 (41) (2015) 23478–23485, <https://doi.org/10.1021/acs.jpcc.5b05760>.
- [62] M. Nicoli, et al., High-temperature oxidation evaluation using crystal microbalance, *Corros. Eng. Sci. Technol.* 55 (5) (2020) 365–371, <https://doi.org/10.1080/1478422X.2020.1732122>.
- [63] B. Acharya, M.A. Sidheswaran, R. Yung, J. Krim, Quartz crystal microbalance apparatus for study of viscous liquids at high temperatures, *Rev. Sci. Instrum.* (2017), <https://doi.org/10.1063/1.4973822>.
- [64] E.V. Vaz Junior, et al., Influence of annealing in deposited Ti–Pt thin films sensing elements for quartz crystals microbalances. 2023 IEEE 10th International Workshop on Metrology for Aerospace (MetroAeroSpace), Milan, Italy, 2023, pp. 699–703, <https://doi.org/10.1109/MetroAeroSpace57412.2023.10189942>.
- [65] R.L. Filler, Thermal hysteresis in quartz crystal resonators and oscillators. 44th Annual Symposium on Frequency Control, IEEE, 1990, pp. 52–57.
- [66] A. Hashmi, A.N. Kalashnikov, Sensor data fusion for responsive high-resolution ultrasonic temperature measurement using piezoelectric transducers, *Ultrasonics* 99 (2019) 105969, <https://doi.org/10.1016/j.ultras.2019.105969>.
- [67] J.A. Kusters, J.R. Vig, Hysteresis in quartz resonators: a review, *IEEE Trans. Ultrason. Ferroelectr. Freq. Control* 38 (3) (1991) 281–290, <https://doi.org/10.1109/58.79613>.
- [68] X. Deng, S. Wang, X. Huang, H. Liu, B. Cui, Modified modeling method of quartz crystal resonator frequency–temperature characteristic considering thermal hysteresis, *IEEE Trans. Ultrason. Ferroelectr. Freq. Control* 68 (3) (2021) 890–898, <https://doi.org/10.1109/TUFFC.2020.3014887>.
- [69] O. Knacke, O. Kubaschewski, H. Hesselmann, *Thermochemical properties of inorganic substances*, Springer, 1991.
- [70] IEEE, IEEE Standard on Piezoelectricity, ANSI/IEEE Std, 1988, pp. 176–1987, <https://doi.org/10.1109/IEEESTD.1988.79638>.
- [71] R. Bechmann, A.D. Ballato, T.J. Lukaszek, Higher-order temperature coefficients of the elastic stiffnesses and compliances of alpha-quartz, *Proc. IRE* 50 (8) (1962) 1812–1822, <https://doi.org/10.1109/JRPROC.1962.288222>.
- [72] A. Ballato, Effect of second rotation on frequency–temperature characteristics of AT-cut crystals, *NASA STI/Recon Tech. Rep.* 77 (1976) 16274.

Chiara Martina earned her B.Sc. in Aerospace Engineering (2020) and M.Sc. in Aeronautical Engineering (2022) from Politecnico di Milano. She is a Ph.D. Candidate in Mechanical Engineering, developing Advanced Measurement Systems for space applications, with a focus on developing high-temperature microbalance and characterisation of instrumental effects from mechanical and thermal disturbances. She was a Visiting Researcher at ESA–ESTEC with a project devoted to sensor fusion for long-term outgassing monitoring, and she was Co-Investigator on different space-related projects (DIANA microbalance for the Tianwen-2 mission, MiLi miniaturised Lidar for Martian atmospheric analyses, and PANCAM project).

Diego Scaccabarozzi is an Associate Professor in Mechanical and Thermal Measurements at the Polytechnic of Milan. His research focuses on the design, miniaturisation, and thermo-mechanical characterisation of space instrumentation, with extensive experience in infrared spectrometers and high-precision sensors for planetary exploration. He contributed and held leadership roles for thermo-mechanical and optomechanical development to major international missions and projects, Mars Express (PFS), ExoMars (MIMA and MicroMED), ESA Hera mission (VISTA), Tianwen-2 (DIANA) and MiLi – EU Horizon programme. Beyond instrument development and optimization, his research addresses metrological characterisation, development of measurement techniques, uncertainty reduction in mechanical and thermal measurements, and vibration analysis for both industrial, space, aerospace and sport applications.

Bortolino Saggini is a Full Professor in Mechanical and Thermal Measurements at the University of Padua. Since his PhD studies, he has specialised in the thermo-mechanical design and structural modelling of space instrumentation for extreme environments. He was responsible for thermo-mechanical development and testing in numerous international missions. He held leadership roles and Co-Investigator status for payloads on Cassini (HASI), Rosetta (VIRTIS, OSIRIS), MarsExpress/VenusExpress (PFS), and JUICE (MAJIS). His expertise spans thermal modelling, cryogenic testing, vibration damping and the development of miniaturised spectrometers and instruments for planetary observation, such as MIMA and MicroMED for ExoMars, with a focus on innovative data processing methods. Beyond space applications, he contributed to high-precision metrology and uncertainty reduction in thermal and mechanical measurements.

Emiliano Zampetti is a Senior Researcher at the National Research Council of Italy (CNR-IIA). His research focuses on the development of advanced sensors for both space exploration and environmental monitoring. He lead development of quartz crystal microbalance (QCM) technology and in-situ chemical analysis for many industrial and space applications. He contributed and held leadership roles for the crystal technology and electronics development in the DIANA instrument (on the Tianwen-2 mission) and successfully led the electronics and crystals development of the VISTA thermogravimetric analyser for the ESA Hera mission (launched in 2024). Beyond deep-space payloads, he spearheaded significant environmental projects, including the GrIN-BOX system for indoor air quality and the NOGAS project for greenhouse gas monitoring, while managing ESA-funded initiatives like CAMLAB for space component outgassing characterisation.

Ernesto Palomba is a Director of Research at the Institute for Space Astrophysics and Planetology (IAPS-INAF) in Rome. His research focuses on the design, development, and calibration of advanced instrumentation for Solar System exploration. Since earning his PhD in Astrophysics and Matter Physics from the Université de Provence in 2001, he has established himself as an expert in hyperspectral Vis-IR data analysis and the laboratory study of extraterrestrial materials. He serves as the Principal Investigator for several high-profile projects, including the VISTA instrument (ESA Hera mission) and the MASTER spectrometer (JAXA's OKEANOS mission). His extensive portfolio of international collaborations includes Co-Investigator roles on landmark missions such as Rosetta, Hayabusa 2, Dawn, BepiColombo, and Mars Express. Beyond his technical contributions to spacecraft instrumentation, he coordinated national and international research initiatives, leading multidisciplinary teams in space weathering and micro-thermogravimetry.