Prototype 9:

Thermocouple probe (FEMTO-ST)

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Based on our experience to develop Scanning Probe Microscopy systems and specific sensors such as microthermocouples, Scanning Thermal Microscopy (SThM) instrument has become a major imaging system which overcomes many drawbacks encountered in both standard far field thermal imager and local batch fabricated thermal probes. A simple wire thermocouple that is robust, versatile and can operate in a large temperature range is used as passive (temperature) [1] and active (thermal conductivity) sensor [2].

Probe Specifications

Microthermocouples are made of Wollaston wires of Platinum and Platinum-10% Rhodium, which are welded together by a sparking technique after having removed their silver cladding. The resulting junction corresponds to a standard S type thermoelectric sensor. Available diameters are 5, 2.5 and 1.3 micrometres. Due to their stiffness, the use of a cantilever is prohibited for contact detection. Only a Quartz Tuning Fork (QTF) allows detecting and controlling the tip to surface contact strength through its resonance frequency response [3]. As shown in Figure 1, two among the eight electrodes of the QTF are used for connecting the thermocouple. The embedded mass is then limited for insuring a sufficient quality factor (better than 5000 in air).

Figure 1 depicts the general aspect of the probe, with a 1.3 µm thermocouple junction. The tip junction is systematically reshaped by means of a Focused Ion Beam (FIB) for insuring a minimal contact area and the best possible spatial resolution.

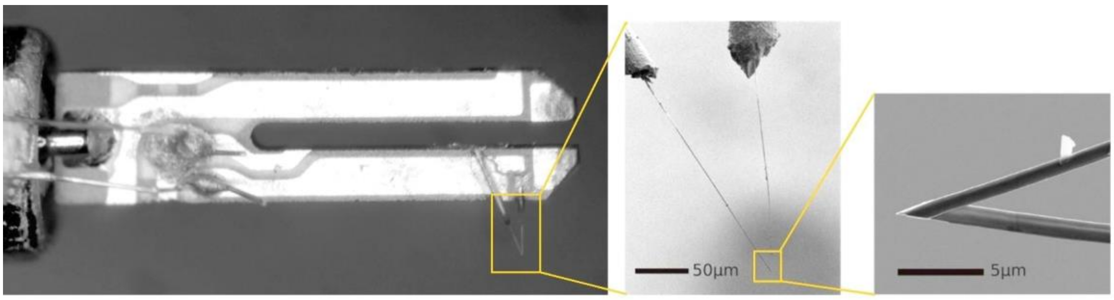


Figure 1: Optical image (left) of FEMTO-ST QTF thermocouple probe; (middle and right) SEM views of the thermoelectric junction made of 1.3µm wires after FIB reshaping.

QTF size: length 5.8 mm and width 1.5 mm. Resonance frequency near 32 kHz.

In Figure 2, the contact response of the QTF and the resulting force between the 1.3 µm probe-tip and a silicon surface are presented. During scanning, the contact force is maintained constant for each measurement point.

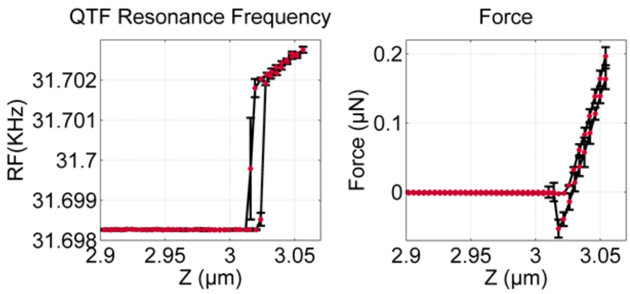


Figure 2: Approach and contact response of (left) QTF resonance frequency; (right) tip-to-surface contact force.

Applications

- Mapping of topography and surface temperature (dc or ac regimes of sample heating) in passive mode, from ambient to 800°C [1].
- Mapping of topography and contact conductance (thermal conductivity if bulk material) in active mode (using 2ω and 3ω methods when ac heating of the probe).
- Scanning area up to a few millimetres square.
- Adjustable pressure environment: from primary vacuum (1 Pa) to atmosphere.

As examples, Figure 3 depicts the topography and surface temperature images of a heated platinum thin film of 600 nm width on a substrate (oxide on silicon wafer). This reveals a lateral resolution far below one micrometre, and hotspot occurrences. Since the imaging process is performed point by point, the topography image is obtained from the vertical position of the probe when the contact occurs. As shown in Figure 2, a resonance frequency shift of the QTF is used as a set value corresponding to a fixed force.

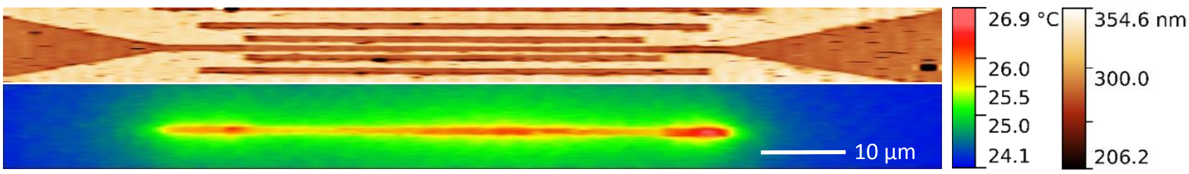


Figure 3. Topography (up) and surface temperature map (down) of a heated resistive film of 600 nm width.

Larger scan areas and higher temperature levels are available as shown in Figure 4 that depicts a 1 mm² hotplate silicon nitride membrane surface in which a platinum thin film heater is embedded.

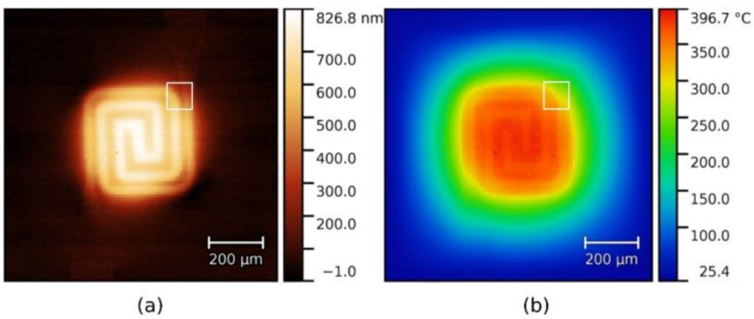


Figure 4. Topography (a) and surface temperature map (b) of an active micro-hotplate heated at 400°C.

Active mode of operation allows extracting thermal conductivity contrast on any kind of surface. In this mode, the thermocouple is heated by an ac current and the resulting thermoelectric voltage is measured at the double frequency. Resistive response of the thermocouple is also available at the triple frequency. Figure 5 shows the topography and the thermal conductance contrast of a portion of the Figure 4 hotplate (white square) while it is not heated (heater not supplied).

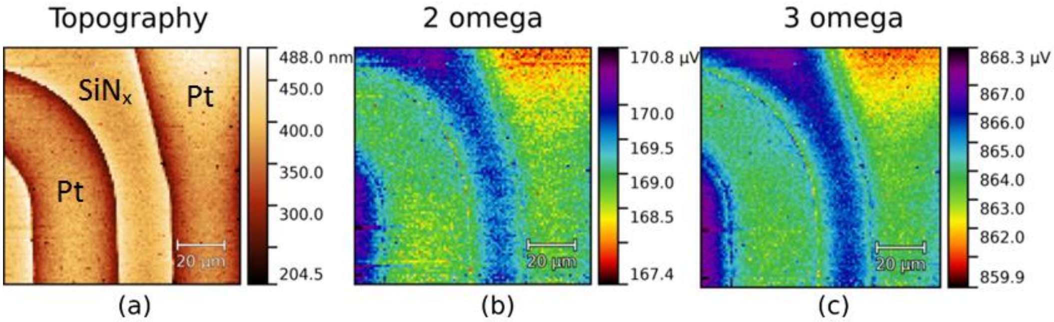
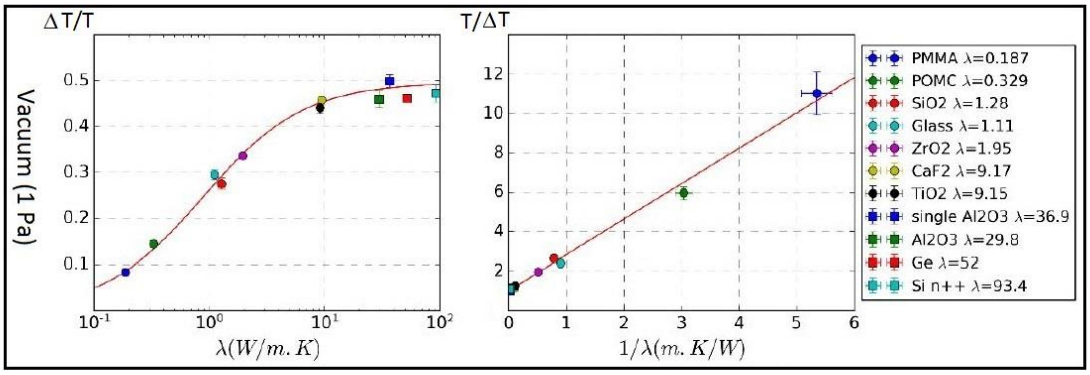


Figure 5. Topography and thermal maps in active mode of a portion of platinum thin film (200 nm thick) on silicon nitride membrane (500 nm thick) with a 5 µm thermocouple probe heated in ac regime.

Thermal images contrast is given by the cooling effect of the active probe part while contact occurs on a surface. The more heat transfers between the probe and the sample are efficient (and the thermal conductivity of the material is high), the more the probe temperature is decreased. However, the extraction of an effective thermal conductivity is not straightforward for thin-film materials and complex structures such for Figure 5. As a result, only apparent values can be deduced from bulk materials calibration as shown in Figure 6. Air or primary vacuum measurements have provided calibration curves (red lines) that relate the relative temperature drop of the thermocouple junction at 2 omega frequency to the sample thermal conductivity.



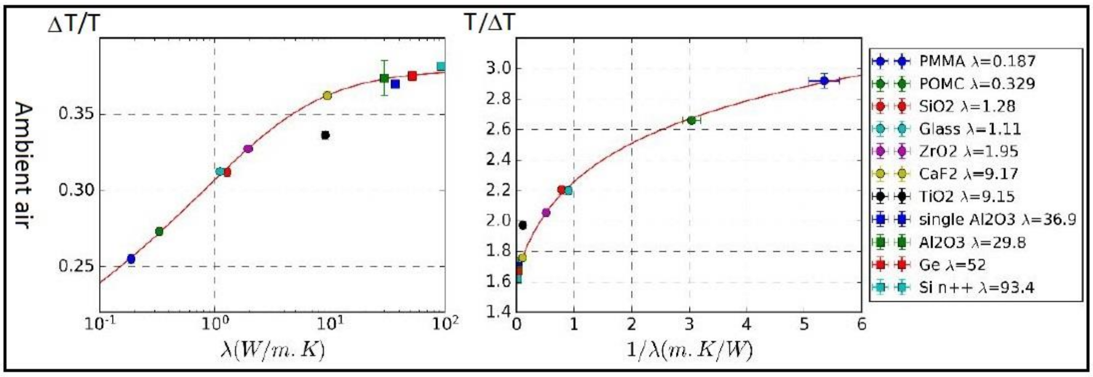


Figure 6. Thermal conductivity calibration curves of 1.3 µm thermocouple probe in vacuum and ambient air obtained on a series of known bulk samples.

Perspectives and recommendations

Due to the complexity of tip-to-sample heat transfers at this scale, complementary studies using different probe size, pressure conditions and specific samples remain necessary to reach the objectives of quantification. Among further perspectives, the next evolution of our system will consist in a "real-time" close-loop control of the tip-to-surface distance (z) in order to reduce the scan duration (from 3 seconds per point to less than 1 second) and improve the contact force control.

References

1. Bontempi A., Thiery L., Teyssieux, D., Briand D., Vairac P., Quantitative thermal microscopy using thermoelectric probe in passive mode, *Rev. Sci. Instrum*. **2013**, 84, 103703; DOI: 10.1063/1.4824069.
2. Bontempi A., Nguyen T.P., Salut R., Thiery L., Teyssieux D., Vairac P., Scanning thermal microscopy based on a quartz tuning fork and a microthermocouple in active mode (2ω method), *Rev. Sci. Instrum.* **2016**, 87, 063702; DOI: 10.1063/1.4952958.
3. Bontempi A., Teyssieux D.,Friedt J. M.,Thiery L.,Hermelin D., Vairac P., Photo-thermal quartz tuning fork excitation for dynamic mode atomic force microscope, *Appl. Phys. Lett.* **2014**, 105, 154104; DOI: 10.1063/1.4896784.

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