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2017 J. Phys.: Conf. Ser. 785 012005

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Recent improvements on micro-thermocouple based SThM

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Abstract. The scanning thermal microscope (SThM) has become a versatile tool for local surface temperature mapping or measuring thermal properties of solid materials. In this article, we present recent improvements in a SThM system, based on a micro-wire thermocouple probe associated with a quartz tuning fork for contact strength detection. Some results obtained on an electrothermal micro-hotplate device, operated in active and passive modes, allow demonstrating its performance as a coupled force detection and thermal measurement system.

1. Introduction

Probe improvements and the full comprehension of heat transfer at micro/nano-scales are still in progress and remain a major objective for different groups and laboratories for which quantitative thermal microscopy systems remain challenging. SThM measurements can be divided in two groups. The first concerns the measurement of the temperature fields of a surface and the second corresponds to the characterization of the thermal properties of solid materials. The latter requires an active thermal probe, from which the heat dissipation to the sample provides relevant data, to deduce some local thermophysical quantities [1,2]. We have shown that a 2-wire micro-thermocouple is able to operate in these two modes of measurement [3,4]. To complete the SThM probe, a combination of a thermocouple sensor and a quartz tuning fork (QTF) force sensor has been developed [4,5]. In the most common available systems, a resistive thermal sensor is integrated on a micro-fabricated cantilever whose deflection is optically detected to derive the contact force between the probe tip and the surface. Unlike that, the dynamic force detection principle of a QTF consists to derive an interaction force from its resonance frequency shift while the contact occurs. In this case, the embedded thermocouple tip acts as a spring, which interacts with the sample surface. If the QTF quality factor is high enough (above 5000 typically) any resonance frequency change can be interpreted as a force and used as a reliable tip-to-sample distance control loop. We first present the design of a thermal/force sensor. The experimental setup is then described and examples of topographical and thermal results in active and passive modes are depicted.

2. Thermocouple probe design and microscopy setup

Here we focus on recent advancements in the development of a SThM system based on a probe associating a micro-thermocouple with a Quartz Tuning Fork (QTF) force sensor [4,5]. “Wollaston” wires (Pt and Pt-10%Rh) are used to make the micro-thermocouple which is directly connected to QTF



electrodes using conductive adhesive. Figure 1 shows the tuning fork with its thermocouple probe. The thermocouple junction is obtained by welding two wires using a sparking technique. Among the eight available electrodes of a QTF, two electrodes are disconnected and reserved for the thermocouple connexions, and a third is used as a RTD sensor to provide the reference thermocouple temperature.

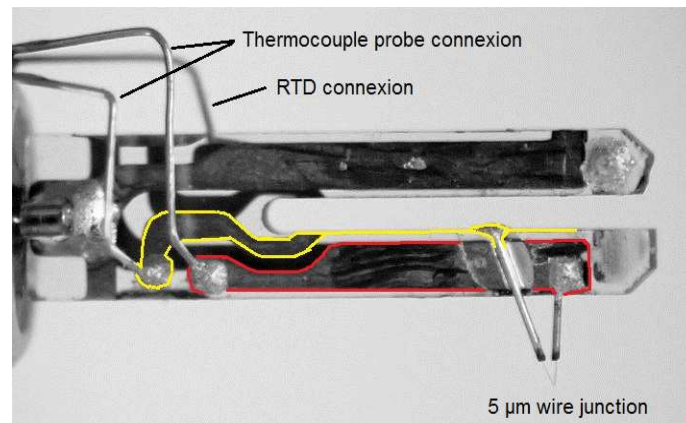


Figure 1. Quartz resonator (QTF) with its thermocouple probe and electrical connection principle.

The shape of the thermocouple junction after ion etching (FIB) is depicted in Figure 2. It clearly shows that a very sharp tip can be obtained in order to ensure a quasi-punctual contact on the sample surface and subsequently the best possible lateral resolution. Three wire diameters are available and used for probe design: 5 μm , 2.5 μm and 1.3 μm .

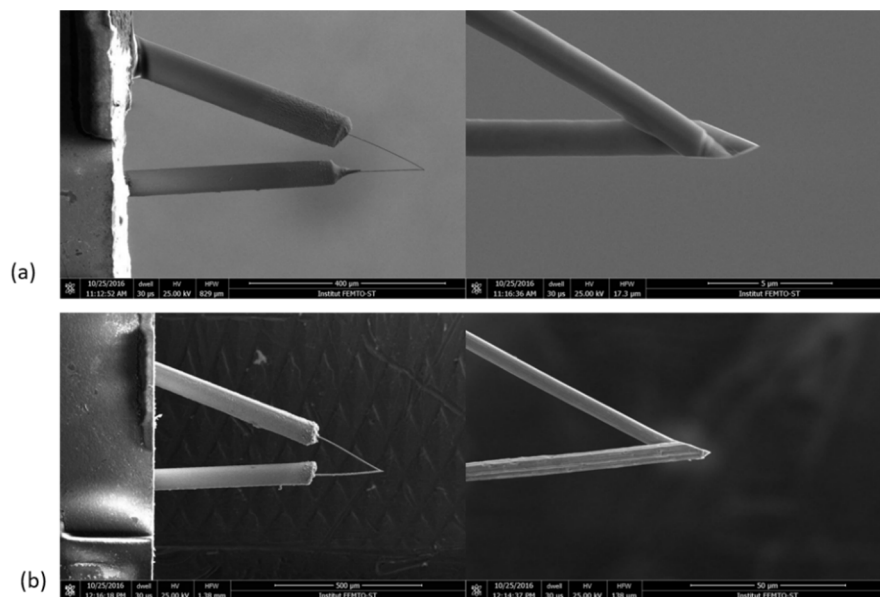


Figure 2. Thermocouple junctions: a) 1.3 μm probe and b) 5 μm probe.

The QTF is excited thermally by a laser whose magnitude is modulated at its main resonance frequency as shown in Figure 3. The photo-thermal excitation allows us to obtain a very high quality factor (10000 in air), eliminates the anti-resonance signal and parasitic capacitance effects.

The new system integrates horizontal X-Y piezo-actuators and stepper motors on which samples are placed in order to provide high resolution and large path length respectively. The probe holder is fixed along the Z axis which is associated with an independent linear motor stage and a high resolution piezo-actuator as shown in Figure 3.

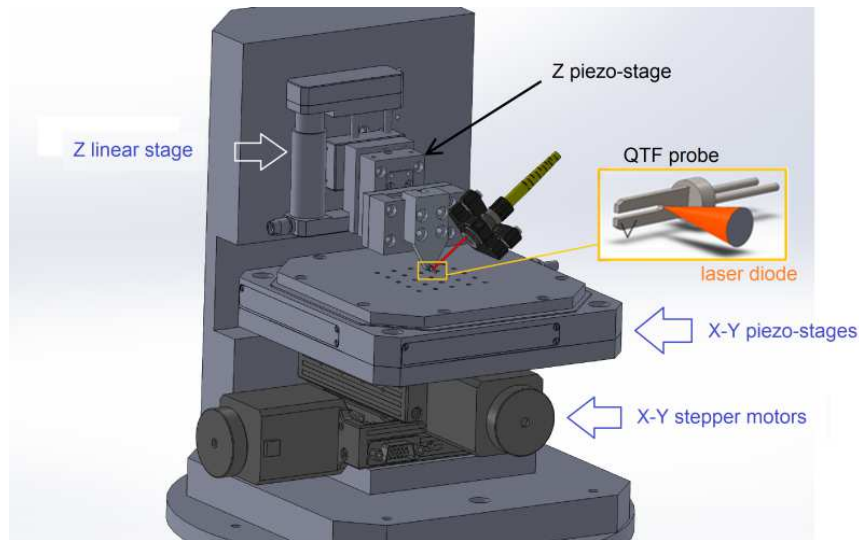


Figure 3. 3D view of the second generation FEMTO-ST SThM system.

The interaction force between the probe and the surface modifies the QTF's resonance frequency. This can be detected by measuring its phase-shift using a lock-in amplifier (open-loop) or frequency shift (close-loop) using a phase locked loop.

Figure 4 depicts the different signal responses of the thermocouple in contact with a silicon surface. When the tip-surface contact occurs, the QTF resonance frequency is shifted (figure 4a). Because the thermocouple probe is considered as a spring [5], the contact force between probe and surface can be deduced from the QTF frequency shift value and the tip's displacement as described in the Figure 4c.

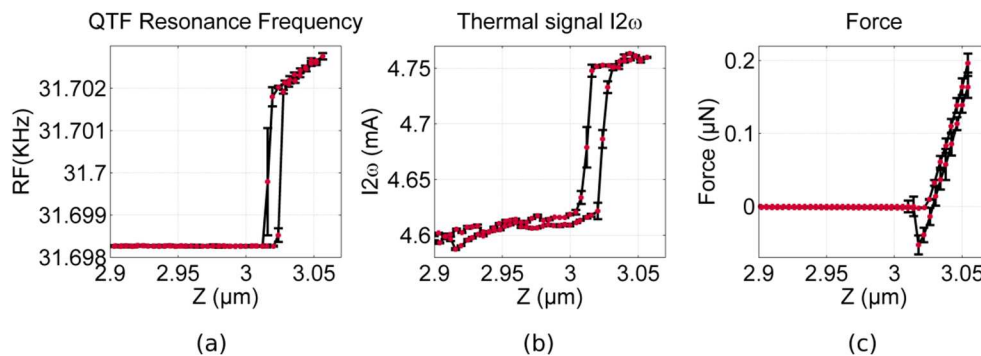


Figure 4: Effects of probe tip contact on a silicon surface.

Figure 4b corresponds to the thermal response of the junction due to the Seebeck effect at the double frequency of the AC heating current inside the probe wires [3]. It corresponds to the current value required to ensure a constant tip temperature (active 2ω mode). In these graphs, the error bars have been obtained by recording 30 consecutive values for every 2 nm step.

3. Active and passive mode measurements

To demonstrate the performance of our system, an electrothermal low power consumption device (micro-hotplate) which is composed of a thin insulating membrane of silicon nitride (500 nm thick) and a 150 nm suspended platinum heater, is used. Figure 5 presents the sample side view with the probe setup (a) and a top view of the platinum heater on the silicon nitride membrane (b). In passive mode, the platinum heater is supplied with a DC current. A scan is performed on the membrane surface (1mm x 1 mm) to extract a temperature map of the whole device surface. Absolute temperature is obtained using the S type thermocouple conversion law using the reference given by the embedded RTD sensor.

In active mode (2ω , 3ω method), the heater is not supplied whereas the thermocouple is heated with an AC current at ω frequency. When the contact occurs, the probe's temperature is affected by the thermal conductance with the sample surface, notably related to its thermal conductivity and topography (roughness). If the current is constant, one can extract both the variation of 2ω and 3ω components. The conversion of the thermoelectric voltage is based on the knowledge of the Seebeck coefficient at the mean junction temperature (DC value).

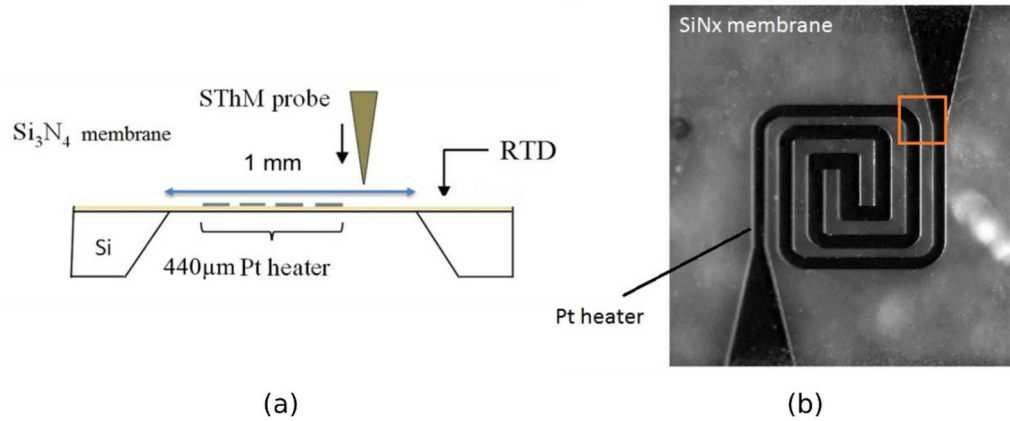


Figure 5. Side-view (a) and top-view (b) of the micro-hotplate.

Passive mode (for the probe)

During the contact, the probe temperature is obtained directly by measuring the Seebeck's voltage. The calibration methodology was already presented using standard hotplates or an optimized device with an integrated temperature sensor [6]. The real surface temperature is calculated thanks to the calibration processes. The temperature map of the device and the topography obtained when the heater operates have shown the sensitivity of a QTF in terms of force detection and the high stability of the thermocouple for temperature measurement. The system is able to reach 0.1°C of temperature resolution and works in a very wide range of temperature (from ambient to 800°C).

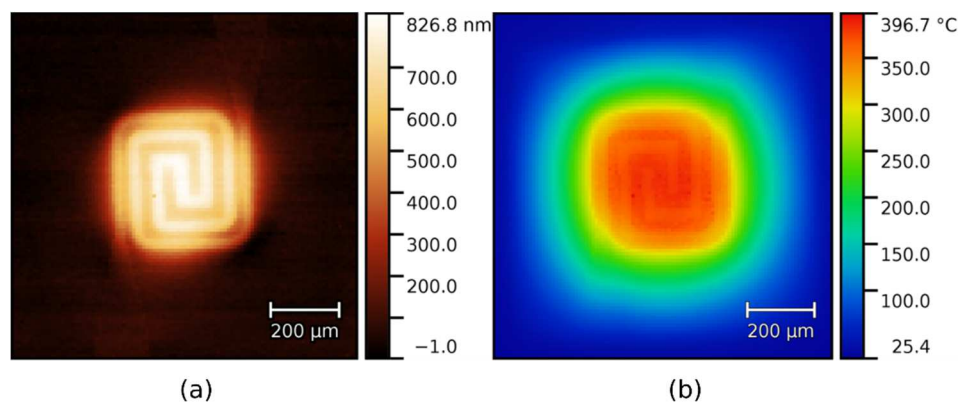


Figure 6. Topography (a) and surface temperature map (b) of active micro-hotplate.

Active mode

To illustrate the possibility to obtain the thermal contrast of a sample surface with our system, a smaller scan area ($100 \times 100 \mu\text{m}^2$) represented by the yellow rectangle in Figure 5b has been performed. In this experiment, the sample is passive. The probe tip temperature is extracted at the double frequency of the exciting current allowing an increase in its temperature by Joule effect. Then, it is possible to measure two thermal components: the 2ω thermoelectric voltage that corresponds to the junction temperature, called 2ω method and the 3ω voltage derived from the resistive effect and the mean probe temperature

(3ω method). Since the chosen frequency of this current is low, the measured $2f$ temperature is identical to the DC temperature variation. Figure 7b and c show the thermal contrast obtained from 2ω and 3ω methods respectively. The tip temperature generally decreases when it approaches the platinum step. At the boundary of two materials, the tip temperature is irregularly high or low due to the topographical artifacts. A stronger cooling is measured in the middle of platinum step, far away from silicon nitride membrane due to maximum heat diffusion into the sample.

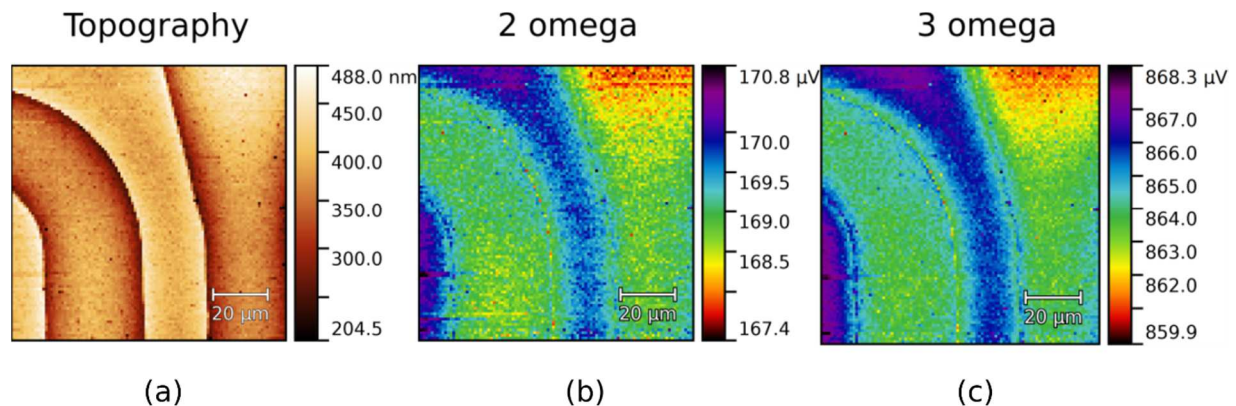


Figure 7. Topography and thermal maps in active mode of a portion of platinum heater on silicon nitride membrane with a $5\mu\text{m}$ thermocouple probe. The sample is passive.

4. Conclusions

The combination of a microwire thermocouple with a QTF has been demonstrated polyvalent. The probe can operate correctly in active mode as well in passive mode. The probe voltage is a highly stable in both modes and the available temperature range is higher than any other SThM system. Our system can also perform scans over area of millimeters square for big samples or smaller areas with resolution down to sub-nanometer scale. The system can operate at different pressures from ambient to 10^{-5} bar. Further measurements and calibration procedures under air and under vacuum conditions are still to be performed in order to obtain quantitative results. The possibility to vary the wire diameter of the thermocouple probe will be also helpful for a better understanding of the thermal spatial resolution significance.

Acknowledgments

The authors would like to acknowledge the developer group of Gwyddion for SPM data visualization software. They would also like to thank the European Union Seventh Framework Program FP7-NMP-2013-LARGE-7 under GA N°604668 Project “QUANTIHEAT” for funding this work.

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