Scanning thermal microscopy: A review

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Fundamental research and continued miniaturization of materials, components and systems have raised the need for the development of thermal-investigation methods enabling ultra-local measurements of surface temperature and thermophysical properties in many areas of science and applicative fields. Scanning thermal microscopy (SThM) is a promising technique for nanometer-scale thermal measurements, imaging, and study of thermal transport phenomena. This review focuses on fundamentals and applications of SThM methods. It inventories the main scanning probe microscopy-based techniques developed for thermal imaging with nanoscale spatial resolution. It describes the approaches currently used to calibrate the SThM probes in thermometry and for thermal conductivity measurement. In many cases, the link between the nominal measured signal and the investigated parameter is not straightforward due to the complexity of the micro/nanoscale interaction between the probe and the sample. Special attention is given to this interaction that conditions the tip–sample interface temperature. Examples of applications of SThM are presented, which include the characterization of operating devices, the measurements of the effective thermal conductivity of nanomaterials and local phase transition temperatures. Finally, future challenges and opportunities for SThM are discussed.

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1 Introduction In the last 20 years nanotechnologies have led to growing needs for fundamental knowledge in thermal and energy sciences at scales always smaller, from the micrometre to the nanometre. In particular, the development of novel materials is dependent on significant advances in the understanding of the energy transport at these scales. As an example, modern electronic and optoelectronic devices have features of several nanometres in size but their accurate thermal characterization at nanoscale stays difficult to achieve. Various issues regarding the impact of nanometre-scale heat transfer on engineered systems justify the importance of developing new experimental methods with this purpose. The scientific and commercial activities of numerous industrial sectors such as semiconductors, aeronautics, aerospace, information technologies are deeply concerned. Precise thermal measurements at sub-30 nm scales are, for example incontestably needed in order to:

- characterize and optimize the properties of nanostructured materials such as nanoscale multi-layered interphases and superlattices, nanoporous media, nanoobjects and nanomaterials such as graphene, carbon nanotubes (CNT) or nanowires integrated in components,
- fill the lacks of understanding of failure mechanisms (reliability and lifetime) in micro- or nanoelectronic devices and components involving silicon-on-insulator nanotransistors, light-emitting diodes, etc. – their design has often been based on theoretical analyses without proper experimental verification,
- improve the accuracy and validity of prediction tools for the ultra-integrated technologies that will appear in the years to come.

Besides energy transport, any phenomenon involving exchanges of energy and entropy with the surroundings such as changes in atomic structures or magnetic domains requires



Review Article



heating or cooling to a certain degree. This includes phase transformations and chemical as well as biochemical reactions. Nanoscale thermal probing should enable to study the energy balances of these phenomena at very small scales.

From a more fundamental point of view, many notions related to heat transfer should be analysed at nanometre scale. Heat transfer is usually associated with a temperature difference. As temperature is only defined under local thermodynamic equilibrium, it can be reasonable to estimate that the mean free path of energy carriers, often in the 10–



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500 nm range, is a limiting lengthscale for many concepts. Since the important quantity is the rate at which energy flows and situations without temperature difference or local thermodynamic equilibrium are possible, these concepts should be revisited. Nonequilibrium conditions have then to be taken into account. So far, mechanisms of energy transfer at very small scales are not completely understood. Measurements of energy transfer at these very small lengthscales will provide first insights into this poorly explored regime.

Contrarily to far-field optical techniques, scanning thermal microscopy (SThM) is not limited in lateral resolution by optical diffraction at few hundreds of nanometres: it can perform thermal imaging and measurements far beyond the micron-scale. SThM techniques are based on scanning probe microscopy (SPM) methods. As a consequence, their spatial resolutions depend on the characteristic lengths associated to the heat transfer between the small thermal probe and the sample to be characterized. SThM probes can be tailored with tips of curvature radii in the range of few tens of nanometres. Because of its high spatial resolution, SThM is now an integral part of the experimental landscape in submicron heat transfer studies. Since the 1990s, it has been developed actively and applied to diverse areas such as microelectronics, optoelectronics, polymers and CNTs.

This article is divided into four main sections. Section 2 provides a review of the main and promising SThM techniques. Section 3 presents the approaches currently used to calibrate usual SThM probes. The calibration step is particularly important because the link between the nominal measured signal and the investigated parameter (temperature, thermal conductivity, etc.) is more complex than for many other SPM techniques due to the entanglement of the various micro/nanoscale heat transfer channels between the probe and the sample. In Section 4, special attention is paid to the physics of the tip-sample interaction, which involves these heat transfer channels. Section 5 introduces some examples of selected applications of SThM. This includes the characterization of operating devices, the measurement of the thermal conductivity of nanomaterials and the determination of phase change temperatures. The final section concludes the article mentioning perspectives and areas where progress could be made in the future, in order to develop novel opportunities for SThM.

2 Instrumentation and SThM methods

2.1 General principle The first SPM instrument exploiting thermal phenomena for nanoscale measurements was invented in 1986 by Williams and Wickramasinghe [1], soon after the invention of scanning tunnelling microscopy. The goal was to extend the possibilities of imaging topography to insulators and was termed scanning thermal profiler (STP). Although the STP was not intended for thermal imaging, it stimulated efforts to develop SPM-based



Figure 1 Set-up of an AFM-based SThM system. Here, the output signal is the voltage V_{out} delivered by a "thermal control unit" and a balanced Wheastone bridge can be used to maintain the probe mean temperature at constant value.

techniques in the thermal area. Since then, various types of scanning thermal microscopes have emerged. These instruments have been mainly based on atomic force microscopy (AFM), because AFM enables using a wider variety of samples and are very versatile systems. Measurements can be performed as a function of the tip–sample force and distance, and, as discussed in the following, various types of sensors can be placed at the tip of an AFM probe. Figure 1 describes the set-up of an AFM-based SThM system.

Cantilever deflections are probed by reflecting a laser beam on a reflective part of the probe, such as the cantilever itself or a mirror appropriately glued on its back, towards a photodiode. Other deflection measurement systems can involve piezoresistive cantilevers [2]. The deflection generates an electrical signal that is detected. In the imaging mode, the deflection signal is used in a feedback control loop to maintain a constant tip-sample contact force while the tip scans laterally. Piezoelectric scanners are used to move the sample vertically and to scan the sample surface laterally. The combination of the X-Y scan position data, the force feedback signal and the thermal signal measured by the sensor located either at the tip or on the cantilever gives the raw data for both the topography image and the "thermal" image of the surface. The thermal image contrast reflects the change in the amount of heat locally exchanged between the tip and the sample. The force feedback control system operates simultaneously but, in contrast to the STP, independently of the process of the thermal measurement. Usually real-time thermal signal analysis is performed with the help of a thermal control unit.

Since 1993, various thermal methods based on the use of different thermosensitive sensors or phenomena have been developed. They can be classified according to the temperature-dependent mechanism that is used: thermovoltage [3–11], change in electrical resistance [2, 12–17], fluorescence [18–20] or thermal expansion [21].

2.2 Thermovoltage-based methods Thermovoltage-based methods exploit the thermoelectric voltage generated at the junction between two electrodes to carry out thermometry. Measurements can be performed either in the non-contact or contact modes in a STM or an AFM system. Thermovoltage-based methods include the tunnelling thermometry [22–25] and the point-contact thermocouple method [26] in which the thermoelectric junction is established between the tip and the sample surface. For both methods, thermal imaging with a nanometric spatial resolution was reported. However, the probe and the sample should have an electrically conducting surface or a surface covered with a metallic film. This limits their applications.

Thermovoltage-based methods also involve probes with a built-in thermal sensor such as a thermocouple [8, 27–29] and a Schottky diode [11, 30]. The first is by far the most popular. Advancements in microfabrication and characterization technologies have enabled to significantly improve the design, operation and use of thermocouple probes [27]. The miniaturization of the cantilever, the tip and the junction at the tip end could lead to a decrease of the probe thermal time constants and to an improvement of the spatial resolution [8, 27–29]. Figure 2 shows an example of nanojunction that it is possible to fabricate at the end of an AFM tip [31].

Until 2002, nanoscale thermal imaging was essentially qualitative with thermocouple-based SThM probes. Different factors limited quantitative characterization. Shi and Majumdar [32] showed that the temperature rise locally measured by a probe depends on the size of the heated area on the sample surface because heat transfer occurs through the surrounding gas. The heat transfer through the surrounding gas may be considered as a perturbation for the measured thermal signal, because it degrades the spatial resolution in comparison to the one of the tip end. Such phenomenon applies to all the methods involving a built-in thermal sensor. In addition, tip and sample are not necessarily at the same temperature, as a large temperature drop can occur at the tip-sample junction due to a thermal contact resistance between the tip and the sample [32]. As discussed later in this article, the value of this thermal contact



Figure 2 Scanning electron microscopy images of a Au–Cr thermocouple SThM probe. Reprinted with permission from Ref. [31]. Copyright 2008, AIP Publishing LLC.

resistance depends on various physical properties and on the surface of the sample. It is often unknown and difficult to determine. Moreover, the sample temperature just below the tip can be modified due to the heat flux flowing through the tip–sample thermal contact. Even if this perturbation is often neglected in SThM, it would be better to specify it for each technique.

To perform real nanoscale quantitative temperature measurement, Nakabeppu and Suzuki [33] proposed to place the set-up under vacuum conditions (below 0.1 Pa) with an active thermal feedback scheme allowing maintaining the tip temperature equal to the sample surface temperature. The tip was a single-wire thermocouple AFM probe and the probe mount was instrumented with an additional thermocouple and a heater. These measurements suggested that maintaining zero heat flux between the tip and the sample may be an alternative for quantitative temperature measurement and profiling. The temperature of the sample surface can then be measured and temperature profiles can be obtained despite unknown tip-sample contact thermal resistance or changes during a scan. However, only point measurements were performed because of the large thermal time constant $(\sim 0.5 \text{ s})$ of the used probe.

The null-point SThM (NP SThM) method was recently proposed by Chung et al. [34–36]: it requires two scans [31]. The method, based on the aforementioned thermodynamic principle, makes it possible to perform quantitative thermal profiling at nanoscale for experiments under ambient condition. Point-by-point temperature measurements of an electrically heated multiwall CNT and continuous temperature profiles of a 5 μ m-wide aluminium line heater deposited on Pyrex glass were demonstrated.

More recently, Kim et al. [37] described an ultra-high vacuum (UHV)-based SThM technique that is capable of quantitatively mapping temperature fields with $\sim 15 \text{ mK}$ temperature resolution and $\sim 10 \text{ nm}$ spatial resolution.

In 2006, the use of thermocouple probes was extended to the investigation of material thermal conductivity with the proposition of a 2ω method [38, 39]. In this 2ω method, the thermocouple probe is heated by Joule effect with an ac current, consequently operating in an ac active mode, and the amplitude of the 2ω signal from the thermocouple junction is monitored (see more details on the various operating modes of resistive metallic probes in next section). The first measurements were performed in contact mode [38, 39]. Thermal conductivity contrast imaging with a nanoscale spatial resolution was reported. Since then, passive or active mode is applied with the thermocouple-based SThM probes depending on the applications.

NP SThM has also recently been shown promising for quantitative thermal conductivity profiling [36].

2.3 Resistive probes Various kinds of SThM probes, based on resistance thermometry, in particular metallic probes [2, 12–17, 40–42] and doped silicon probes [43–45], have been implemented.

2.3.1 Operating modes of resistive metallic probes All resistive metallic probes can also be used in passive and active modes.

The passive mode is used for thermometry. In this mode, a very small electrical current is passed through the probe. This results in minimal Joule self-heating and enables the measurement of the electrical resistance. During a scan, heat flows from the hot sample to the probe and changes the electrical resistance R_p of the probe. Indeed at first order

$$R_{\rm p}(T) = R_{\rm p0}(1 + \alpha(T - T_0)), \tag{1}$$

where $R_{\rm p}(T)$ is the electrical resistance of the probe thermosensitive element at a reference temperature T, $R_{\rm p0}$ is the electrical resistance of the element at temperature T_0 and α is the temperature coefficient of its electrical resistivity.

The active mode is used to measure thermophysical properties of materials such as thermal conductivity. In this case, a larger electrical current is passed through the probe, resulting in a significant Joule heating. Part of the Joule power flows into the sample, depending on its thermal conductivity. The probe temperature is monitored by measuring the probe voltage. This temperature is related to the thermal conductivity of the sample.

The active mode can also be used to locally heat the sample in order to induce and study thermo-dependent phenomena such as in scanning thermal expansion microscopy or with the dynamic localized thermomechanical analysis method, both developed by Hammiche et al. [46].

Under both passive and active modes, dc, ac or both measurements can be performed. Exciting the probe with an ac electrical current can be useful for an improved signal-to-noise ratio, since lock-in detection is possible. It is worth mentioning that a 3ω method can be used [47], which consists in measuring the third-harmonic voltage $V_{3\omega}$ of the resistor. The $V_{3\omega}$ amplitude is directly proportional to an increase of temperature due to Joule heating: $V_{3\omega} = \alpha T_{2\omega} R_{p0} I_{\omega}/2$, where I_{ω} is the amplitude of the exciting current and $T_{2\omega}$ is the amplitude of second-harmonic of the probe mean temperature. Different configurations of electrical bridge can be used to measure the electrical resistance of the probe and deduce its temperature [40–41, 48–50].

2.3.2 Wollaston wire probe The Wollaston wire probe was the first SThM metallic resistive probe proposed by Pylkki et al. [12, 51] in 1994. The cantilever is made of Wollaston wire consisting of a silver shell of 75 μ m in diameter and a core of an alloy of platinum and rhodium (Pt₉₀/Rd₁₀) of 5 μ m in diameter [52]. At the extremity of the cantilever, the wire is bent in a V-shape and electrochemically etched to uncover the Pt₉₀/Rd₁₀ part over a length of approximately 200 μ m as shown in Fig. 3. The liberated core constitutes the thermal sensitive element. A mirror is stacked on the cantilever arms, so that the cantilever deflection can be



Figure 3 Scanning electron microscopy image of a Wollaston wire probe [71].

controlled by the optical way. The cantilever of the Wollaston wire probe has a spring constant of $\sim 5 \text{ N m}^{-1}$. Its thermosensitive element has a temperature coefficient $\alpha = 0.00166 \text{ K}^{-1}$ and its time response has been estimated to 200 µs in air [47, 53, 54]. Because of these characteristics and its high endurance, the Wollaston probe has been attractive. This probe has been used widely for microsystem diagnostics [48, 55–57], local thermophysical characterization of various materials [58–68] and investigation of the thermal interaction between the probe and samples [69–71] (see also Section 4). However, its large active area limits the thermal investigations at nanoscales.

2.3.3 Smaller metallic probes To improve the method using a miniaturized resistive metallic element, several probe designs based on the deposit of a resistive element on the AFM cantilever have been proposed [2, 13–17, 40–42]. Figure 4 shows an example of design of such probe. The thermosensitive element of the probe consists of a thin Pd film positioned at the very end of a flat tip located at the extremity of a thin cantilever. The cantilever was initially made of silicon oxide but it has been recently

extended to silicon nitride Si₃N₄ as reported by Weaver and co-workers [16]. The cantilever has a low spring constant (0.35 N m⁻¹). The tip is tall (around 10 μ m) to maximize the cantilever–sample separation, minimizing the heating of the cantilever by the hot sample in case of thermometry measurement [16]. This tip has a curvature radius of around 50 nm [72] and its electrical resistance has a temperature coefficient $\alpha \sim 0.0012 \text{ K}^{-1}$ [73]. Its time response has been estimated to a few tens of μ s [73, 74].

Designs incorporating a multiwall CNT or similar high thermal conductivity graphene sheet material with longitudinal dimensions on micrometre length scale have been recently proposed [72] and are expected to improve the thermal and spatial resolutions. Quantitative temperature sensing at the nanoscale point contact has already been developed using a platinum hot film sensor with a CNT as the thermal probe [75]. The quantitative local temperature at the CNT probe contact point was determined by bringing the probe in and out of contact and controlling the amount of heat released by the Pt hot film.

2.3.4 Doped Si resistor probes (DS probes) The silicon nanoprobes were first developed by IBM for high data-storage systems and high-speed nanoscale lithography applications [76]. The cantilever is a U-shaped cantilever consisting of two micrometric legs with high-doping level and a low-doped resistive element platform. The tip possesses a nanometric curvature radius (it can reach 10 nm) and is of micrometric height. It has a conical shape and is mounted on top of the resistive element. Later, Nelson and King [43] developed similar silicon probes but with pyramidal tips as shown in Fig. 5.

An electrical current flowing through the resistive element causes resistive heating and a temperature rise of the tip. Note that the first-order expansion of Eq. (1) is not sufficient to describe the variation of this electrical resistance as a function of temperature.

Operating in active mode, this kind of probe has mainly been used for nanothermal analysis, thermomechanical actuation, nanolithography and data storage [77]. A review of its applications is given in Ref. [77] and some examples of



Figure 4 SEM image of a palladium probe.



Figure 5 SEM image of a doped silicon probe.

481



its application in SThM are described in Section 4 of this article.

2.4 Other probes and techniques In addition to the thermoresistive and the thermocouple phenomena, few other thermally dependent physical effects can be exploited for thermal investigations at the micro and nanoscale.

2.4.1 Thermoacoustic effect Thermal expansion can be utilized to measure the increase of temperature. Majumdar et al. invented a scanning-Joule expansion microscope (SJEM) in the 1990s by measuring the dilation of a material in which a Joule heating resistor was embedded [78, 79]. Due to Joule heating, the material could be heated and a standard AFM was placed on top of the surface to measure precisely the surface position. Knowing that thermal expansion coefficient is usually in a range close to 10^{-5} m K^{-1} , a minimal size of few tens of microns is required for the sample. Similar technique has also been applied by Cretin [80] and Gurrum et al. [81]. We note however that the geometry of the material or device should be known prior to the experiment if one wants to apply this technique.

The thermal expansion can also be generated optically. This is the principle of AFM-infrared (AFM-IR) spectroscopy developed by Dazzi et al. [82]. Here, an infrared pulse is generated either by a pulsed source facility [82] or by a less-resolved but more practical table-top infrared light source [83]. The goal is to detect if the sample absorbs radiation in the infrared spectrum at the chosen exciting frequency (or frequency band). It is interesting to note that here the technique takes advantage of the dynamics of the phenomenon: the short pulse leads to the thermal expansion which will shake the AFM tip in contact with the excited sample. As a consequence, the cantilever will start to oscillate. If the heating frequency is close to the cantilever resonance f_{res} , an amplification of the signal can happen. The cantilever should indeed be considered as a frequency filter with some resonances. This technique can be used to perform a spectroscopic analysis of the sample. This technique is not strictly speaking a SThM, but involves heating to perform spectroscopy. Interesting applications are found in biology, e.g. for localizing viruses in cells [84].

2.4.2 Bimetallic probes When two materials in contact with different thermal expansion coefficients are involved, thermal expansion can lead to a bending effect. This especially happens in the case of a bimetallic cantilever. Because of the lengths of cantilevers in the micron to millimetre range and the very sensitive detection of the cantilever position in AFMs, very tiny changes in the average temperature of cantilevers can be observed. Majumdar et al. showed that changes down to few 10^{-5} K could be observed [85]. The idea was then to link the temperature of the cantilever to the one of the sample. This can be done by considering a physical model describing

the configuration of the experiment. Refs. [86-88] took advantage of this technique.

This technique has been applied for the measurement of thermal radiation between objects separated by nano to micrometre-scale distances [89, 90]. The exchanged heat flux is increased in comparison to large separation distances due to the opening of near-field radiative heat transfer channels. Here tip and sample are not in mechanical contact, and no feedback is needed in order to maintain the cantilever in its initial position: only its displacement is monitored.

2.4.3 Fluorescent-particle based tips Fluorescence is a phenomenon which depends strongly on temperature, because the emitted intensity I_{em} is proportional to the population of excited states. This population is proportional to the Bose–Einstein statistics when the fluorescent material is in thermal equilibrium at temperature T:

$$I_{\rm em} \approx \frac{1}{\mathrm{e}^{(\hbar\omega/k_{\rm B}T)} - 1} \approx \mathrm{e}^{-(\hbar\omega/k_{\rm B}T)}, \qquad (2)$$

with $k_{\rm B}$ the Boltzmann constant and $\hbar\omega$ the photon energy. $\hbar = h/2\pi$, where *h* is the Planck constant.

By monitoring the evolution of the intensity of an emitting material at a given frequency, one can deduce the temperature variation of the sample. Interestingly, the ratio of the intensities of two bands depends only on their frequencies and on temperature. Aigouy has proposed a new SThM tool based on fluorescence by gluing a small fluorescent particle (Er-based) at the very end of an AFM tip [18, 91]. The temperature of the tip is then assimilated to the temperature of the fluorescent particle.

2.4.4 Near-field thermal radiation Kittel et al. have developed a SThM which operates within an STM [29, 92–94]. Here a thermocouple is set at the end of the tip and the instrument is located in a UHV chamber to avoid any contamination issue. Interestingly, this set-up can operate in non-contact mode and enables to measure the near-field thermal radiative heat transfer between the tip and the sample (see Section 4.5). Such an instrument could show for example that the heat radiated by a monolayer dielectric island can be detected [95].

2.4.5 Towards new tips? Beyond the currently used ones, few techniques appear promising. Among them, one can notice another scanning probe technique called 'thermal radiation STM' (TR-STM), which is based on the detection of a heated sample. Here, it is the thermal radiation emitted by the sample that is scattered by the probe, which is modulated in height by means of the tapping mode [96, 97]. The scattered radiation is collected with the help of a detector locked to the tapping frequency. A Fourier-transform infrared (FTIR) analysis of this radiation can be performed to observe the spectroscopic features of the sample. It is also possible to heat the tip directly instead of the sample [98].

This points out to the new possibilities of using probes for various purposes in the same time. It is well known that SPM can be used to investigate, at the nanoscale, electrical potential, electrical and electromechanical properties [99], magnetic properties [100], chemical characteristics [101], mechanical properties [102–105], etc. It may be possible to combine SThM with at least one of these other applications, resulting in a multifunctional probe. This has already induced many fruitful developments [106–109] and will open totally new era for SThM.

3 SThM measurement approaches

3.1 Energy balance As in all thermal measurement method involving a sensor in contact or in proximity with the sample surface to be characterized, the quantity of heat exchanged between the sample and the tip Q_{s-t} depends on the energy balance of the system that consists of the sensor in contact with the sample and interacting with its surrounding environment. Q_{s-t} is consequently a function of the effective thermal properties of sample and probe, and temperatures of probe, sample and their surrounding environment.

For thermometry in steady-state regime and passive mode, a heat quantity is exchanged between the hot sample and the probe that is initially at room temperature. In a very simplistic way, this may be described by the thermal resistance network represented in Fig. 6. The corresponding expression of Q_{s-t} can then be written as

$$Q_{s-t} = \frac{T_s - T_{t,c}}{R_{th,s} + R_{th,c}}$$

= $\frac{(T_p - T_a)(R_{th,pe} + R_{th,cant.})}{R_{th,pe}R_{th,cant.}} + \frac{T_{t,c} - T_a}{R_{th,env}},$ (3)

where T_s is the sample temperature to be determined and T_p is the probe temperature that is measured. The contact of the



Figure 6 Thermal resistance network model for a probe used in passive mode. Here T_{sp} is the sample surface temperature perturbed by the heat transfer between the sample and the cantilever far from the tip–sample thermal exchange area.

hot sample with the probe initially at ambient temperature $T_{\rm a}$ leads to a decreasing of the temperature within the sample under the probe-sample contact of temperature $T_{s,c}$ and an increasing of the temperature at the tip apex $T_{t,c}$. $R_{th,s}$, $R_{th,c}$ and $R_{\text{th,t}}$ are the thermal resistances associated respectively to the heat transfer within the sample at the level of the constriction near the contact (sample thermal spreading resistance), to the heat transfer from the sample to the tip and to the heat transfer between the tip apex and the thermosensitive element of temperature $T_{\rm p}$. The heat losses to the environment are included in three thermal resistances: $R_{\rm th,env}$ that describes the heat losses to the surrounding environment between the probe apex and the sensitive element, $R_{\rm th,pe}$ that represents the probe heat losses after the sensitive element by convection and radiation to the environment and $R_{\rm th,cant.}$ that corresponds to the heat losses by conduction in the probe support or cantilever. Let $R_{th,p}$ be the equivalent for the last two thermal resistances. We note that a parasitic heat transfer from the sample directly to the cantilever, here represented by $R_{\rm th,gap}$, can take place, but this will be neglected in the following.

In the simple case of a sensitive element at the tip apex, which is the case for almost all the thermovoltage-based SThM probes ($T_{t,c} = T_p$ and $R_{th,env} = R_{th,t} = 0$), and no heat transfer between the cantilever and the sample surface, Q_{s-t} may be written as

$$Q_{s-t} = \frac{T_s - T_p}{R_{th,s} + R_{th,c}} = \frac{(T_p - T_a)(R_{th,pe} + R_{th,cant.})}{R_{th,pe}R_{th,cant.}}$$
$$= \frac{T_p - T_a}{R_{th,p}}$$
(4)

and the value of the correction to be applied to the nominal measurement of the instrument T_p is

$$\delta T_{\rm p} = T_{\rm s} - T_{\rm p} = \frac{(T_{\rm p} - T_{\rm a})(R_{\rm th,s} + R_{\rm c})}{R_{\rm th,p}}.$$
 (5)

This last expression shows that δT_p is dependent on the heat transfer within the sample (through $R_{th,s}$) and from the sample to the whole probe and its surrounding (through $R_{th,p}$). In addition, it depends on the heat transfer between the tip apex and the thermosensitive element if this last element is not located at the tip apex. δT_p also depends on the resistance of the tip–sample thermal contact R_c .

If δT_p is unknown, only the probe temperature T_p can be measured: SThM temperature measurement requires the determination of the various thermal resistances of the network shown in Fig. 6.

In practice, the error δT_p is very variable from a sensor to another one and from an experimental configuration to another one. The power rate transferred from the sample to the probe Q_{s-t} depends on many parameters characterizing:

the surrounding gas (pressure, temperature, degree of relative humidity),



- the tip–sample mechanical contact: mechanical properties of tip and surface, tip–sample force, surface roughness and topography,
- the thermophysical properties of probe and sample.

As shown in Section 3.2, experimental calibration methodologies have been proposed for the determination of $\delta T_{\rm p}$. The estimation of all the involved parameters through modelling is not trivial and is still one of the main limitations of SThM involving nanoprobes. Indeed, heat transfers at micro and nanoscales within the tip and sample, exchanged between the probe and the sample through surrounding gas and radiation or through nanoscale contacts must be considered in the estimation of $\delta T_{\rm p}$. The tip–sample heat transfer is described in details in Section 4.

For thermophysical measurement (active mode), the heating of the sample by the probe operating in active mode (in dc or ac regimes) is required. The temperature sensor is heated through Joule effect and plays the role of heat source for the sample. Under this condition, the rate of the heat transferred by the probe to the sample $Q_{t,s}$ may be written as a function of the thermal power *P* used for the heating of the probe, the measured probe temperature T_p and the thermal conductivity of the sample k_s . Depending on the probe, various analytical and numerical models have been proposed [6, 36, 47, 54, 67, 70, 72, 73, 110–114] to link the nominal signal effectively measured (voltage) and the parameters to be determined (k_s).

For resistive probes, the thermal sensor cannot be assumed to be located at the probe apex. The probe temperature at the probe apex $T_{t,c}$ must be known to rigorously establish the expression of the power exchanged between the probe and the sample. We note also that some studies have been proposed for thermocouple probes [31, 115]. A majority of models have considered the geometrical and dimensional parameters and the physical properties of materials to describe the probe. They also include effective parameters such as:

- (i) an effective coefficient *h* of heat losses by the whole probe surface to its environment, which is key to the expression of the thermal resistance $R_{\text{th,pe}}$ in the Eqs. (3) and (4),
- (ii) the effective thermal resistance $R_{\text{th,c}}$ generally used for describing the probe–sample thermal interaction at the level of the probe–sample contact. Thermal interaction is then assumed to take place across an area generally described as a disc of effective radius *b* at the sample surface.

For bulk and homogeneous thick samples, the sample thermal conductivity k_s is included in the expression of the sample thermal spreading resistance $R_{th,s}$ that can be written as

$$R_{\rm th,s} = \frac{1}{4k_{\rm s}b},\tag{6}$$

if one assumes that the heated area on the sample surface is circular of effective radius b and isothermal [116].

As explained in the next section, the comparison of the effective tip–sample system thermal modeling with measurements enables the determination of the unknown modeling parameters. Once calibrated for a given experimental configuration (probe and surrounding environment), the modelling is used to characterize unknown specimens from measurements performed in the same configuration such as in Refs. [65, 73, 107].

3.2 Calibration The calibration consists in performing measurements with a reference sample and comparing the determined value to the expected one. If they are not equal, one can correct the setting of the unit. In SThM, the calibration consists mainly in specifying the link between the thermovoltage (thermocouple junction) or the electrical resistance (resistive probe) measured with either the sample temperature or the sample thermal conductivity. Purely experimental methods with known samples and methods involving modelling of the measurement have been used.

3.2.1 Experimental calibration for thermometry The calibration methods implemented for the determination of the error δT_p have mainly used laboratory self-heating samples. They have been based on comparisons of SThM measurements with either measurements obtained by optical thermometry methods or results of simulation of the sample surface temperature (or both). However optical thermometry methods have spatial resolution limited to few hundreds of nanometres and simulations at micro and nanometric scales are often dependent on simplifications or critical parameters. As a result, these comparisons are not always perfectly applicable to SThM temperature measurements with spatial resolution of few tens of nanometres, so precautions are necessary and limit the temperature measurement at nanoscales. One regularly evoked solution would be to exploit the null-point SThM (NP SThM method [34-36], see Section 2.2) based on specific measurements, which ensure that Q_{s-t} nullifies:

$$Q_{\rm s-t} = \frac{T_{\rm s} - T_{\rm p}}{R_{\rm th,s} + R_{\rm th,c}} = 0.$$
(7)

Self-heating samples that have been used or fabricated for SThM calibration include instruments that are specifically designed for absolute temperature measurements on the scale of 1 μ m. They are based on the measurement of the Johnson–Nyquist noise in a small metallic resistor [117], or instrumented membrane [118]. Other samples have been based on hot sources implemented in subsurface volume with a metallic line heated through Joule effect [20, 31, 32, 36, 37, 48, 119, 120]. The samples are generally heated in ac regime to demonstrate thermal mapping with low signal-tonoise ratio. They have been also used to characterize the dynamic response of sensors, which is also an important parameter to be considered. Let us notice that accurate temperature measurements after calibration with such samples will only be possible on samples that hold surface properties close to the ones of the calibration samples.

3.2.2 Experimental calibration for thermal **conductivity analysis** For thermal conductivity analysis, an experimental calibration can be performed with a set of experiments involving flat bulk samples of well-known thermal conductivities in a range that covers the expected value of the thermal conductivity k_s to be measured. Practically, the tip is usually heated with an increase of temperature ΔT larger than 80 K to ensure a good signal-tonoise ratio and avoid issues related to the presence of a water meniscus [121, 122] (see Section 4.3). A stable dc current heats up the tip through Joule effect, and the electrical resistance of the tip is constantly monitored. The probe is measured with a balanced Wheatstone bridge that involves a feedback loop enabling to set a constant value of its electrical resistance (see Fig. 1). The average probe temperature is then kept constant during the measurement, and it is the electrical current that can vary. The data associated to each known sample are reported on a $\Delta P/P_{in} = f(k_s)$ plot, where P_{in} is the Joule power required to heat the tip at the set temperature when the tip is in contact with the sample of thermal conductivity $k_{\rm s}$ and $\Delta P = P_{\rm in} - P_{\rm out}$ is the difference with the Joule power required to set the tip temperature when the probe is far from the contact [123]. The method can also be performed with measurements in the ac regime.

3.2.3 Calibration through comparison between measurement and modelling As for numerous experimental methods of characterization, the fitting of simulated measurements with experimental data in well-known conditions can be used for the identification of the modelling parameters in SThM. These parameters may include probe parameters. The thermal resistance $R_{\text{th,t}}$, $R_{\text{th,cant}}$ and $R_{\text{th,env}}$, $R_{\rm th,pe}$ indeed depend on the shape and sizes of the probe. For probes that have been partially made by hand, such as the Wollaston one, these parameters may vary from one tip to another. Even nanofabricated probes, which benefit from the reproducibility associated to cleanroom CMOS standard technology, may have little variations in the parameters. The user certainly does not want to inspect systematically all the tips with scanning electron microscopy (SEM). In addition, SEM check does not necessarily provide all the relevant information. For this reason, Lefèvre et al. [47] have proposed to use a sweep in frequency $V_{3\omega}(f)$ $(f = \omega/2\pi \text{ with } \omega$ the angular frequency) in order to determine the cut-off frequency f_c , which is linked to the size of the heating element in their Wollaston resistive tip (length of the resistive filament) [47]. The amplitude of the signal close to the static operation $V_{3\omega}(f \rightarrow 0)$ then provides the radius of the filament, so that a full determination of the resistor is performed with such sweep. The method enables a useful direct experimental determination of some modelling parameters for the Wollaston probe. Puyoo et al. [73,

124] did similar analysis for the Pd probe and showed that its smallest dimension increases the value of the cut-off frequency, therefore higher-frequency operation and faster scans are possible. The drawback of such characterization lies in the determination of f_c , with some uncertainty due to experimental measurement. These works used probes operating in active mode but such calibration can be performed for thermometry in a similar fashion [54].

Modelling parameters, such as the effective coefficient hof heat losses by the whole probe surface to its environment and the effective parameters $R_{th,c}$ and b generally used for describing the probe-sample thermal interaction, can also be determined through the fitting of simulated measurement with experimental data in well-known conditions (various surrounding conditions [125], various frequencies of heating of the probe [47, 73, 113], various samples...). The method has been used not only for thermal conductivity measurement of various materials [65, 73, 107] but also to study the probe-sample heat transfer. In particular, the comparison of a tip-sample heat transfer model with measurements under ambient air on a set of samples of various thermal conductivity has suggested that both $R_{\rm th,c}$ and b depend on the thermal conductivity of sample [126]. Values of $R_{\rm th,c}$ and b for the Wollaston probe were then respectively found varying from 2×10^6 KW⁻¹ to 1×10^5 KW⁻¹, and from 550 to 150 nm when the sample thermal conductivity increases [126]. Assy et al. have recently shown that this dependence on the sample thermal conductivity is not negligible from similar measurements [127]. Furthermore, other works [70, 128] suggested a non-negligible contribution of the thermal resistance of the solid-solid contact between the probe and the sample. This contribution depends on the sample thermal conductivity but also on the sample mechanical properties and roughness [128, 129]. To date, no analytical expression of $R_{th,c}$ depending on the sample physical properties is available. Numerical simulation appears appropriate for describing the probe-sample interaction through air. As a consequence, the purely experimental methods previously described are preferable for simple measurements.

We note that an almost similar calibration methodology has recently been demonstrated comparing measurements performed with a Wollaston probe far from contact and without contact but at a small distance (<100 nm) from the sample surface, confirming that heat transfer is mainly exchanged through air with the Wollaston probe [66, 125, 130] (see Section 4.4).

It has also been demonstrated that this calibration methodology is applicable for the resistive palladium probe (see Fig. 4) operated in air [131, 132] and under vacuum conditions [132].

Current Si probes with nanotips may be sensitive to a reduced range of thermal conductivities [133] compared to the one of larger and metallic probes. This is specifically due to nanoscale size effects. At room temperature the phonon averaged mean free path Λ_{ph} in pure crystalline silicon is approximately 300 nm [134] and exceeds the actual sizes of



the tip apex of the Si probes. As a result a macroscopic description of heat transfer does not accurately model the heat flow and the heat conduction through the tip is modelled as quasi-ballistic transport. A Mathiessen rule can be applied to calculate a mean free path modified from the bulk value by accounting boundary scattering. Analytical expressions have already been proposed for simple geometries [135]. As a consequence an additional thermal resistance must be considered to describe heat transfer at the probe apex [45, 111, 136]. As discussed in the following, the expression of the sample thermal spreading resistance $R_{th,s}$ as well as the microscale description of the probe–sample heat transfer may also be strongly affected and fails due to nanoscale size effects.

Whatever the probe, a better understanding of the thermal interaction between a SThM tip and a sample is however crucial for a good understanding of measurement and a good interpretation of the contrast of thermal images. The following part focuses on the current description of the different heat transfers contributing to the probe–sample thermal interaction.

4 Probe-sample heat transfer

4.1 Heat transfer channels Various mechanisms of heat transfer between the probe and the sample coexist. Figure 7 shows the heat flow paths from a hot probe to the sample for experiments performed under ambient air. The heat transfer channels between the probe and the sample include radiative heat transfer, thermal transfer through the surrounding gas, heat conduction through the liquid meniscus formed at the tip–sample junction and heat conduction through the mechanical contacts between both objects.

Very schematically, the effective thermal resistance $R_{th,c}$ describing the probe–sample thermal interaction at the level of the probe–sample interface may be written as proposed by Majumdar [27, 32]:

$$R_{\rm th,c} = \frac{1}{G_{\rm th,c}} = \frac{1}{G_{\rm rad} + G_{\rm gas} + G_{\rm w} + G_{\rm mc}}$$
(8)

with $G_{\rm th,c}$ the effective thermal conductance describing the probe–sample thermal interaction, and $G_{\rm rad}$, $G_{\rm gas}$, $G_{\rm w}$ and $G_{\rm mc}$, the thermal conductances describing the probe–sample heat transfer through radiation, gas conduction, liquid meniscus conduction and conduction through the mechanical contacts, respectively.

4.2 Heat conduction at the tip-sample contact The radius of the mechanical contact $b_{c,p}$ is generally estimated from mechanical contact theories [137–140]. These theories apply to the contact between a sphere and a flat surface and their use leads to consider an ideal contact between surfaces of perfect quality. Whatever the considered SThM tip, $b_{c,p}$ is estimated to be lower than a few tens of nanometres. For such nanoscale contacts, phonon mismatch and mechanical contact geometry have to be



Figure 7 Schematic representations at different length scales (not at scale) of the heat flow paths from the very end of a heated tip at temperature T_{tip} to a sample at lower temperature T_{sample} . (a) Microscopic probe–sample contact; *b* is the radius of the effective thermal contact. (b) Microscopic multi-asperity contact. The dotted line represents the ideal profile of the tip apex. b_c and $b_{c,p}$ are, respectively, the mechanical contact radii associated to the ideal profile and the real profile of the tip apex. (c) Thermal resistance network model of the thermal resistance of the probe–sample thermal interaction $R_{th,c}$: R_{rad} , R_{gas} , R_w , and R_{mc} are, respectively, the thermal resistances describing the probe–sample heat transfer through radiation, gas conduction, liquid meniscus conduction, and conduction through solids close to the mechanical contact. $T_{tip,c}$ is the mean temperature of the tip apex.

accounted for the estimation of the contribution of the solidsolid interface to the measurement.

An interfacial contact thermal resistance (thermal boundary resistance) should be considered at the tip–sample interface due to the difference in phonon dispersion between the two materials in contact [141–145]. This boundary

resistance can be estimated as

$$R_{\rm th,contact B} = \frac{R_{\rm th,B}}{\pi b_{\rm c,p}^2},\tag{9}$$

with $R_{\text{th,B}}$ a thermal boundary resistance that has the same units as the bulk thermal contact resistance. The values of $R_{\text{th,B}}$ experimentally determined for solid–solid contacts near room temperature typically lie in the range 5×10^{-7} to $5 \times 10^{-9} \text{ m}^2 \text{ KW}^{-1}$ [43, 144, 146].

Since the tip–sample interface is never perfect, (i) water, contamination or oxide layers can cover surfaces and (ii) the contact area is non-continuous due to surface roughness or weak coupling bonds between the atoms of solids. Advanced contact models have been developed to account for the weak coupling. The transmission probability is then related to the mechanical coupling spring between the two solids [147, 148]. Moreover, due to the roughness of the tip and sample surfaces, the apparent contact surface is not continuous. This surface can be divided into smaller contact area (of size b_c in Fig. 7b). Appropriate modelling of this effect depends on the ratio of the averaged mean free path of heat carriers Λ_c to the size b_c . If $b_c \gg \Lambda_c$, diffusive transport applies. Classical solutions are applicable:

$$G_{\rm th,contact} = Kk_{\rm s}b_{\rm c},\tag{10}$$

where K is a geometrical factor describing the heat spreading within the sample (see Eq. (6)).

On the other hand, if $b_c < \Lambda_c$, ballistic solutions must be considered [149]. When $b_c \ll \Lambda_c$, the notion of finite contact spots may be extrapolated to the atomic scale [150, 151]. Gotsmann and Lantz [151] have recently suggested that a quantization of thermal conductance could occur when $b_{\rm c}$ becomes lower than $\lambda_{\rm coh}$, the phonon coherence length, while the distance between individual contact spots may exceed λ_{coh} . This may take place, e.g. for single-atom contacts. Related experimental works were performed under UHV with doped silicon probes with end tip specifically prepared for the purpose [151]. Under such environmental conditions, probe-sample thermal interaction involves nearfield heat transfer and conduction through mechanical contact. From almost similar experiments under UHV, Pettees and Shi [136] studied the phonon transport through a nanoscale point contact between a Si tip and a Si sample. Several models were used to evaluate the contact area critical for interpreting $R_{\rm th,c}$. These works characterized the thermal boundary resistance $R_{\text{th,B}}$ for the considered Si-Si nanocontact by using a nanoconstriction model. These authors also examined the expression of the quantum thermal conductance and suggested the inaccuracy of such a model to explain measurement results obtained above room temperature [136].

4.3 Water meniscus When the SThM probe contacts the sample, a water meniscus appears due to the capillary condensation of humidity. The heat path through water



Figure 8 Schematic of the meniscus between probe and sample.

meniscus was scarcely studied in the literature and little information exists about the meniscus thermal conductance. The reason behind is that most of the properties of the water at small scales are still unknown and especially the thermal properties. Luo et al. [6] suggested that the meniscus conductance G_w is dominant among the heat transfer mechanisms. Based on the Kelvin equation, G_w was given as [6]

$$G_{\rm w} \approx 2\pi k_{\rm w} R_{\rm a} \left[\ln \left(1 + \frac{R_{\rm a}}{a} (1 - \cos \beta) \right) + 1 - \cos \beta \right].$$
(11)

Here, k_w , *a* and R_a are, respectively, the meniscus thermal conductivity, the probe–sample separation and the apex radius, $\sin \beta = w/2R_a$ where $w = 2r_2$ is the meniscus width as shown in Fig. 8. Their probes were made of thermocouple junctions (Au–Ni or Au–Pt) and the tip radius was around 100 nm. The thermal conductance determined from the experimental measurements was on the same order than the ones estimated using Eq. (11).

Using the same configuration of probe and sample, Shi and Majumdar [32] later estimated that the heat transfer through meniscus is negligible in front of the heat transfer through air. The authors did not account for the thermal conductances at the probe–water and water–sample surfaces in their first work [6] and that was probably the reason behind such difference [32].

Taking into account these thermal conductances, Assy et al. [122] have recently shown that conduction through water meniscus is not dominant in the probe–heat transfer interaction for the Wollaston probe, whatever the probe temperature. The values of the thermal conductances are lower than $0.2 \,\mu \text{WK}^{-1}$ which is one order of magnitude smaller than the values of the thermal conductance due to the heat transfer through air ($G_{air} \simeq a \text{ few } \mu \text{WK}^{-1}$ [69, 70, 125]). The study was performed at various Wollaston probe temperatures on a sample of hydrophilic nature. The thermal conductance describing the heat transfer through the meniscus could be estimated through the analysis of the capillary forces.

Since the thermal conductance of the water meniscus depends on the dimensions of the meniscus, many factors might influence this heat path: hydrophilic or hydrophobic nature of the surfaces [152, 153], roughness of surfaces [154, 155], relative humidity [156].

4.4 Air heat transfer Heat can be transferred from the hot tip to the sample (or from the hot sample to the cold



tip) through the air when the microscope is not located in vacuum. Indeed, a large area of the probe is in contact with the air lying around and can heat it. Hot air can then heat the sample. It is important to note that the hot part of the tip can extend over tens of micrometres in some SThM probes. As a consequence, the heated part of the sample could potentially be as large, which raises questions about the spatial resolution of SThM. It has been shown that the heat flux deposited by the heated Wollaston probe, of micrometre size, can indeed extend over few microns [125, 157]. As a consequence, this heat transfer mechanism is extremely efficient. 65% of the heat transferred from the Wollaston probe to the sample is carried by air at room temperature [158]. When the heater is far from the tip end and therefore from the sample (such as for the silicon tip), strong probe-sample air heat transfer also occurs, with air being heated at the area of the conical tip or directly by the cantilever. When the tip-sample distance is large, heat convection can occur: the heat flux will be carried by the motion of air generated by the temperature difference [157]. At shorter distances (few micrometres and below), heat diffusion will be the main mechanism of heat transfer through the air. The heat transfer can then be modelled with standard finite-element modelling (FEM) tools. When the distance reaches few hundreds of nanometres, ballistic heat transfer will appear. The mean free path of the heat carriers, here air molecules (mostly nitrogen ones), Λ_{air} becomes comparable with the tip-sample distance ($\Lambda_{air} \sim 70 \text{ nm}$ at P = 1 bar). In principle, FEM tools cannot be used anymore and more advanced methods have to be considered [32, 159, 160]. Direct simulation Monte Carlo (DSMC) [161, 162] and a quasi-ballistic heat transfer model [160] have indeed been developed to better assess the subcontinuum conduction. However, if the area involved in ballistic heat transfer is much smaller than the one involved in diffusive heat transfer, the ballistic regime can be safely neglected. This may be the case in very-conical tips of micrometre sizes.

A large part of the heat can be lost in the air because heat lost by a hot tip does not necessarily flow towards the sample. For the Wollaston tip, this part of the Joule power is close to 40% [163, 164]. It is therefore very important to precisely know the heat transfer coefficient h that describes the air-wall heat losses, in particular because its value can be different at micro or nanoscale than what is expected from macroscale correlations [164]. h can be identified by comparing the probe signal while working under ambient environment and under vacuum conditions where no transfer to the air is assumed. For the Wollaston tip, $h \sim 3000$ $Wm^{-2}K^{-1}$ [158, 160]. For the palladium wire, Puyoo [132] identified a value close to $6600 \text{ Wm}^{-2} \text{ K}^{-1}$. Kim and King [164] used numerical simulations and FE model to determine the heat losses to the air from the silicon cantilever of the silicon probes. The estimated coefficient h around the leg was found about $2000 \,\mathrm{W}\,\mathrm{m}^{-2}\,\mathrm{K}^{-1}$ and around $7000 \,\mathrm{W}$ $m^{-2}K^{-1}$ to the heater in the case of steady state heating.

All the values of h in the studies mentioned above show that heat losses are largely enhanced at small scale comparing to the values at the macroscale. We should mention that these studies identified the values of h when the probe is out of contact with the sample. Such values are susceptible to change when the probe comes to contact with the sample because of a restricted area of exchange between the probe and the environment medium. This effect has been neglected up to now.

4.5 Thermal radiation When not in vacuum, it is very difficult to determine experimentally the fraction of heat that is transferred or lost by thermal radiation. Very often, air heat transfer, by convection, diffusion or by ballistic heat transfer, cannot be distinguished from thermal radiation. As a consequence, it has been customary to neglect thermal radiation or to include it in the evaluation of another heat transfer mechanism [163]. This second way is particularly attractive when dealing with the air heat losses previously mentioned, as thermal radiation towards environment can be embodied in the coefficient h.

However, it should be underlined that thermal radiation mechanisms are very different when the involved sizes are comparable to, or smaller than, the typical wavelength λ defined from Wien's displacement law $\lambda T = 2897 \,\mu m \,\mathrm{K}$. Below few tens of micrometres (at room temperature), not only the classical thermal radiation mechanism (termed farfield) is possible, but also near-field radiative heat transfer due to tunnelling of evanescent surface waves generated by thermal motion [165–166]. This means that the transferred radiative heat flux can be increased by orders of magnitude in comparison to the far-field prediction [167-169]. This has been verified experimentally with bimetallic probes on which large spheres were attached, to increase the area of exchange of thermal radiation [89, 90]. It is also at the heart of the principle of the scanning thermal microscope developed by Kittel and colleagues, which is based on the non-contact heat exchange due to near-field heat transfer. The increase due to near-field is intense when the optical spectra of the material exchanging radiation are similar. When they are not, heat transfer is not as large and may not be very effective [170]. In addition, computation of nearfield heat transfer may be dependent on the geometry while only few academic cases have been considered up to now. A tip-sample interaction model involving electric and magnetic dipolar contribution of the tip and multireflections has been proposed [171, 172]. We note that the effective tip radii reproducing the experimental data are larger than the ones observed by SEM. For large tips, a proximity approximation may be used [173]. A conical-tip calculation is also available [174]. The impact of thermal radiation on SThM can be considered as an open question in many cases and should deserve intense research in the next years.

5 Applications SThM is mostly used for thermometry, for thermal conductivity characterization of materials as well as a way to determine local phase-change transition. It is operated in air, in vacuum, and has been recently extended to liquids [175–176].

5.1 Thermometry Investigations of sample surface temperature have been performed through point studies and mappings of the amplitude of the steady periodic temperature field.

Single to be commercialized for more than 15 years, the Wollaston probe in passive mode was used for microsystem diagnostics [48, 55–57]. It was successfully used to characterize the temperature profile measurements of a PN thermoelectric couple [55] and shown to be useful for failure localization and analysis of integrated circuits [48, 177, 178]. These studies were made in ac regime. However, the active area, the size and shape of the Wollaston probe limited the thermal investigations of ICs due to topography-related artefacts in thermal images [47] and the analysis of nanoscale structures. As a result, applications of SThM to active microdevices and nanodevices have mainly been performed with smaller probes.

The first thermocouple probes with a built-in thermal sensor [179] were used to measure the distribution of the amplitude of the steady periodic temperature field inside a vertical-cavity surface-emitting laser (VCSEL) [179]. Since then, SThM using small probes (thermocouple, resistive probes as well as fluorescent particle probes) has been used to characterize the heat dissipation and transport pathways in various nanocomponents such as multiwall carbon nanotubes MWCNs (about 10 nm in diameter) and single wall carbon nanotubes ~SWCNs (about 1-2 nm in diameter) [180-182], Joule self-heated graphene nanoribbons (GNRs) [183], self-heating silicon nanowire and a self-heating nanowire diode (with doped Si probes) [120], nanoscale constrictions in metallic microwires deposited on an oxidized silicon substrate [119], suspended (with asymmetric type of contacts) current-carrying GaN nanowires [184].

It is important to remind that thermal imaging with SThM was essentially qualitative until 2002. Kwon et al. [185] were the first in 2003 to demonstrate that the amplitude and phase of the steady periodic temperature field can be mapped quantitatively under ambient conditions without the distortion due to the heat transfer through the air gap with an SThM probe. This is possible if the heating frequency of the steady periodic temperature field to be measured is sufficiently high. They explained that the influence of the thermal wave transferred through the air gap and the cantilever of the SThM probe becomes negligible compared to that of the thermal wave transferred to the sensor directly through the tip-sample thermal contact because the penetration depth of the thermal wave decreases with increasing heating frequency. A quantitative measurement of the steady temperature distribution under ambient conditions was recently demonstrated with a spatial resolution around 50 nm bounded by the diameter of the tip-sample thermal contact area [36].

As already mentioned in Section 2.2, UHV-based SThM technique enables to quantitatively map temperature fields with much better spatial resolution. However, temperature uncertainty may be larger in vacuum due to the smaller heat transfer between the tip and the sample.

5.2 Thermal conductivity For thermal conductivity measurements, the tip acts as a thermometer and delivers heat to the sample simultaneously (conventional SThM in active mode either in dc or ac regimes). The thermal investigation of matter by use of a very-localized heat source enables materials to be probed at the level of very small subsurface volumes. Therefore, the technique has quickly appeared to be a promising method to study the thermal conductivity of micro and nanostructures. We mention some examples of analyses in the following.

The Wollaston wire probe was the leading probe during a long time. It has been applied through point measurements to the characterization of various bulk materials and thin films, mainly insulating materials because of its thermal conductivity sensitivity limited for high thermal conductivity materials, such as:

- Bulk ZnO (0001) [59], and Ba₈Si₄₆ that is a simple binary representative of intermetallic clathrates, guest-host systems with a high potential for thermoelectric applications due to their ultralow thermal conductivity even their perfect crystal structure [186].
- Porous [62, 66, 110, 187] and mesoporous [65] bulks or thin films of submicrometric thickness [99, 152]: some results obtained with SThM were compared with values measured on the same material samples by Raman thermometry and can be in good accordance [65, 70, 187, 188]. The ratio of reduction of the effective thermal conductivity of porous silicon due to irradiation with swift heavy ions was experimentally demonstrated [187].
- Thin films on substrate. This includes SiO₂ thin films on Si substrate [67], fully and partially coalesced lateral epitaxial overgrown GaN/sapphire (0001) [60].

The Wollaston wire probe was also used in ac mode to characterize powder materials such as bare diamond crystallites 300 μ m in diameter, and on crystallites coated with Cr, Cu and Cu/Cr layers with thickness in the range 0.5–30 μ m [189]. It was also used to map thermal-conductivity profile of irradiated materials [190].

We underline that as evidence of local quantitative measurement with this probe (and this is the same for all SThM probes), profiling of a sample with a known thermal conductivity distribution should be presented instead of measurement at certain points, because of the possible distortion of the measured profile when measurements are performed in ambient conditions.

The smallest details thermally imaged with the Wollaston probe were found about a few tens of nanometres in size. In some cases, topography-related artefacts may be suspected [71]: carbon contamination-assisted sharpening of the probe was shown [191], or probes were modified [192, 193]. In the two last cases, a better sensitivity to sample thermal property [191, 192] was obtained due to the decreasing of the probe–sample thermal resistance at the probe–sample contact. As a result, a micrometric spatial resolution has generally been agreed by the SThM users for



the Wollaston probe under ambient conditions. For a more complete overview of applications regarding the Wollaston probe, readers are referred to the reviews [58, 194].

Other resistive metallic probes with smaller tips have been used, such as the Pd probe operating in the 3ω method (ac regime) to measure the thermal conductivity of single silicon nanowire [73]. The measurement led to thermal maps of cross-sections of nanowires embedded in a SiO₂ substrate. A spatial resolution of around 100 nm was achieved in thermal imaging performed under vacuum environment. The method was recently adapted for characterizing the thermal conductance of SiGe nanowires [195] and a Sb₂Te₃ phasechange nanowire [196].

A modified SThM technique has also been proposed to image thermally multilayered periodic photonic structures [197]. Thermal boundaries between the layers were revealed by means of an analysis of the topographic and thermal signals, and the periodicity of the structure was analysed. A spatial resolution close to 70 nm was found.

Doped Si probes made it possible to image thermal conductivity contrast of biological materials with a spatial resolution of 10 nm and a thermal resolution of 50 nW [44]. In addition, they enabled the estimation of the thermal conductivity of a 3 nm thick HfO_2 film on a silicon substrate with a spatial resolution of around 25 nm. In this case, experiments were also performed under vacuum environment [45].

Investigations of non-diffusive thermal transfer are also possible with resistive probes. As an example, measurements on graphene samples have also been recently performed, showing the peculiar nature of heat transfer in this material [198–199].

5.3 Phase change temperatures SThM probes operating in active mode have also been largely used to characterize the phase change temperatures (glass transition or melting temperature) of polymer materials. More details on this SThM application may be found in Refs. [43, 46, 58, 64, 200].

6 Conclusions and perspectives In this review, we have presented many of the techniques involving SPM and thermal sensors, broadly recognized as part of the SThM field. Various thermal effects such as electrothermal ones (Joule and Seebeck effects) or thermoacoustic ones (bimetallic effect, infrared absorption, etc.) have been considered and implemented in SThM. We have underlined the crucial step of calibration. We have also described the tip–sample heat transfer, which is complex, and explained why its deep understanding is required in order to obtain the best achievements of the techniques. Finally, we have presented various modes of applications related to thermometry and thermal property measurements such as local thermal conductivity and mentioned local phase change temperature determinations.

SThM can be of interest for both material science, e.g. when local thermal conductivity is measured, and the microelectronic industry, e.g. when hot spots on surfaces are to be detected as a sign of device failure. While its first steps happened more than 25 years ago, we notice that its spreading is quite limited in comparison to other scanning probe techniques. One issue is that the spatial resolution is still not very well known, with a variety of definitions depending on the application and on the particular focus it is intended for. A unification of the terminology should be useful.

In comparison to optical techniques, the spatial resolution of SThM, at least in vacuum, is extremely promising. However, it has been mostly used until now in standard environment where it competes with the micrometric resolution of usual far-field non-contact optical techniques. Impressive resolution has been detected [120, 182] and could certainly be improved, for example to achieve both sub-100 nm spatial resolution and sub 0.1 K temperature resolution.

An arising question is then as follows: what are the limits of SThM? Since the current works aim at measuring parameters, which require local thermal equilibrium (LTE), such as temperature or thermal conductivity, it is certain that the current approach will break down at some scale. It is well known that there is no LTE below the scale of heat carriers' mean free path Λ , typically 300 nm in silicon, considered as a paradigmatic thermally-conductive dielectric material, and \sim 25 nm in metals. When a SThM instrument with a tip radius of curvature equal to tens of nanometres is brought to contact with a silicon sample, it may be difficult to measure the thermal conductivity of silicon k_{Si} . We have already underlined that the usual approach of considering that there is a thermal diffusion spreading resistance $R_{\text{th,s}} \propto 1/(k_{\text{Si}}b)$ inversely proportional to k_{Si} in the silicon breaks down. Instead, the thermal resistance will include a component linked to the ballistic transfer with sub-mean free path dimension. In the illusory case where the nanometre-scale contact is perfectly determined, the measured signal should be linked to $k_{\rm Si}/\Lambda \approx c_{\rm p} \upsilon$ (where $c_{\rm p}$ and υ are, respectively, the specific heat and the mean velocity of phonons) instead of thermal conductivity k_{Si} and involve the square of the characteristic dimension b of the contact instead of the first power [130].

Now, since the channel to transfer heat becomes very small, the quantity of transferred heat may also be small. It is interesting to note that it actually may become very close to physical limits such as the typical thermally activated energy $k_{\rm B}T$ or to the quantum of thermal conductance, which is $g_0 = (\pi^2 k_{\rm B}^2 T)/3h$ for all usual heat carriers such as electrons, phonons and photons [151]. SThM may open new avenues for the investigations of physical phenomena in the quantum regime, at least at low temperatures. In addition, the heat transfer before contact, due to vibrations of both acoustic and optical modes, is also not very well understood on a theoretical basis [201-203]. Peculiar results may be observed [204]. We observe that thermoelectric heating in junctions is a way to generate heat at atomic contact [205, 206] and that many interesting features have been predicted for atomic [207] and molecular contacts.

Heat transfer in polymer chains has already been investigated [208, 209].

On the practical side, we have also mentioned that multifunctional probes may be developed in the next few years. In additions to applications involving metrology and patterning [210, 211], other types of applications may be added to the thermal ones. As a final conclusion, we anticipate hot future for SThM.

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Review

Article

493

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