

## Measurements of nanoscale thermal properties of materials via Scanning Thermal Microscopy (SThM): Challenges and solutions.

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Scanning Thermal Microscopy (SThM) is one of the most universal methods for probing heat conductivity, interfacial thermal resistance and local temperature of materials and devices with nanoscale resolution. SThM uses a temperature sensitive heated probe with an apex of lateral dimensions ranging from a micrometre down to few nanometers that can contact a studied material or a nanoscale device at an arbitrary point on its surface. The tip-sample contact results in a heat flow from the heated tip to the sample -  $Q_{ts}$  that reduces a heater temperature  $T_h$ , that is constantly monitored using a sensitive electronic circuit. SThM primary output signal is the heat flow  $Q_{ts}$  or a closely related parameter - total heater-sample thermal conductance  $G_{ts} = Q_{ts}/(T_h - T_s)$  where  $T_s$  is the temperature of the sample. As the tip scanned in a raster way across the sample surface, SThM output produces “thermal” maps reflecting spatial variations in the local sample thermal conductivity  $k_s$  with the lateral resolution down to a few nanometers (1). A major challenge is the quantitative interpretation of SThM “thermal” signal as  $k_s$  is fundamentally entangled with the tip-sample interfacial thermal conductance  $g_{if}$  that directly depends on the geometry of the tip-surface contact, a generally unknown value that can also vary significantly during SThM measurements.

In this paper we describe three linked approaches that allow to eliminate major variabilities in the SThM measurements as well as produce quantitative measurements of nanoscale thin layers of materials. First, we control the temperature of the sample  $T_s$  and the microscope base  $T_m$  via actively controlled Peltier heating/cooling elements with  $\sim 10$  mK precision significantly improving the reproducibility of SThM signal by approximately 10 fold. Secondly, we use simultaneous measurement of shear forces and heat flow between the probe (2). As shear forces directly proportional to the contact area, the correlation observed allowed us to confirm the true ballistic nature of heat transport via nanoscale contacts in such a system, suggesting that even large – sub-micrometer sized contacts are composed by a multiple nanoscale junctions with the size below the mean-free-path of the heat carriers. Shear forces SThM allowed us to eliminate dependence of the SThM output on the most difficult to determine parameter – tip-surface contact geometry. Finally, we present a new paradigm of measurement of thermal conductance in the nanoscale thin layers of materials by producing a nanoscale cross-section of the material or device via SPM-friendly Ar ion polishing producing near-atomically low-angle wedge-shaped flat sections (3), followed by the SThM measurements of total thermal conductance  $G_{ts}$  as a function of the wedge thickness  $t$ . The decrease of the thermal conductance as a function of edge thickness  $dG_{ts}/dt$  allows to exclusively determine thermal conductivity of the sample  $k_{ts}$ , eliminating necessity to know either the tip-sample interfacial thermal conductance  $g_{if}$  or layer-substrate thermal conductance, two notoriously unknown parameters that render majority of SThM measurements to be merely qualitative.

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