European CNRS network (GDR 2503) « Thermal Nanoscience and Nanoengineering »

Scientific School
THERMAL INSTRUMENTATION AND METROLOGY FOR MICRO/NANO: FUNDAMENTALS AND APPLICATIONS

DATE: Sunday 30 November -Friday 05 December 2014

LOCATION: Fréjus in France / Villa Clythia (CNRS center)

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PARTICIPANTS: opened to the QUANTIHEAT Consortium & to scientists/industrials external to the consortium

RHYTHM OF WORKING:

- 6 hours of lectures scheduled / day.
- 2 hours for poster sessions (Monday and Tuesday evenings).
- 1 Roundtable planned on Thursday evening.
- A free half-day scheduled on Wednesday afternoon.

Scientific Committee: S. Gomès (CETHIL-CNRS Lyon, France), B. Hay (LNE, France), N. Trannoy (GRESPI- Univ. Reims, France), G. Tessier (Univ. Paris-Descartes, France), C. Sotomayor (ICN, Spain), J. Weaver (Univ. Glasgow, UK), P. Klapetek (CMI, Czech Republic) and S. Volz (EM2C- EC-Paris, France).

Organization Committee: S. Gomès, S. Rault and P.-O. Chapuis (CETHIL-CNRS Lyon, France).

PROGRAMME

Thematic School's timetable							
TIME	November 30	December 1	December 2	December 3	December 4	December 5	TIME
08:30-09:00				**Optical techniques III**	**Electrical techniques II**	**SThM lecture I**	08:30-09:00
09:00-10:00		**Introduction**	**Near-field infrared optical microscopy and spectroscopy**			**Near field radiation**	09:00-10:00
10:00-10:30		***Break***					10:00-10:30
10:30-11:30		**Conduction in nanomaterials : advanced modelling methods I**	**AFM based nanoscale IR spectroscopy**	**Conduction in nanomaterials : advanced modelling methods II**	**Nanomechanical measurements - AFM**	**Near field radiation**	10:30-11:30
11:30-12:30			**AFM based nanoscale thermal analysis**			**SThM lecture III**	11:30-12:30
12:30-14:00		***Lunch***					12:30-14:00
14:00-15:00		**Electrical techniques I**	**Metrology II**	***Free half- day or Cap Dramont visit***	**Optical techniques IV**		14:00-15:00
15:00-16:00		**Optical techniques I**					15:00-16:00
16:00-16:30		***Break***			**Break**		16:00-16:30
16:30-17:30	***Registration and Welcome***	**Metrology I**	**Optical techniques II**		**SThM lecture II**		16:30-17:30
17:30-18:00							17:30-18:00
19:00							19:00
20:00	***Aperitive***	***Dinner***		***Gala dinner***	***Dinner***		20:00
21:00	***Dinner***	***Poster Session***	***Poster Session***		***Roundtable***		21:00
TIME	November 30	December 1	December 2	December 3	December 4	December 5	TIME

DETAIL OF SESSIONS / LECTURES / LECTURERS

Sessions	Lectures	Lecturers
Optical techniques	(I) Near infrared thermography, Mid-wave IR thermography.	Damien Teyssieux, FEMTO-ST
	(II) Raman thermometry	Emigdio Chávez, ICN
	(III) Photoreflectance, Thermoreflectance	Gilles Tessier, PARIS 5
	(IV) Time Domain ThermoReflectance	Stefan Dilhaire, Bordeaux University
Electrical techniques	Resistance thermometry	John Weaver, GU
	Thermal property measurement by electrical means	Olivier Bourgeois, Néel Institute
SPM techniques for thermometry, thermophysical and mechanical characterization of materials	(I) Scanning Thermal Microscopy	Nathalie Trannoy, URCA
	(II) Scanning Thermal Microscopy	John Weaver, GU
	(III) Scanning Thermal Microscopy	Séverine Gomès, CNRS
	AFM based nanoscale IR spectroscopy and Local Thermal Analysis	Eoghan Dillon, Anasys Instruments
	Near-field infrared optical microscopy and spectroscopy	Yannick De Wilde, Langevin Institute, ESPCI
	Nanomechanical measurements - AFM	Rafaël Barbattini, Asylium Research
Modelling in support for the interpretation and for the analysis of Measurement	Conduction in nanomaterials : advanced modelling methods I	Olivier Chapuis, CNRS
	Conduction in nanomaterials : advanced modelling methods II	Petr Klapetek, CMI
	Near-field thermal radiation	Rodolphe Vaillon, CNRS
Metrology	Metrology I : definitions, methods, traceability and standards	Tony Maxwell, NPL
	Metrology II : Measurement uncertainty	Alexandre Allard, LNE

Optical techniques

(I) Visible-Near Infrared Thermography (Vis-NIR thermography) (FEMTO-ST):

1. Thermal radiation of solids
   a) Planck's law
   b) Wien's law
   c) Stefan-Boltzmann law
   d) Emissivity of solids
2. Infrared thermography/Vis-Near IR thermography comparison
   a) Disturbance radiation
   b) Error on the temperature value due to the error on emissivity
   c) Thermal sensitivity
3. CCD matrix sensor
   a) CCD sensor architectures
   b) Full-Frame CCD noises
4. Limit of temperature detection
5. Calibration of Vis-NIR thermography
6. Experimental measurements
7. Thermal detectivity enhancement of Vis-NIR thermography

Scientific background required: no specific background required.

(II) Introduction to Raman thermometry (ICN):

The experimental measurement of the thermal conductivity involves two steps: the introduction of thermal energy into the system, heating, and the detection of the change of temperature or related physical properties due to the increase of the thermal energy, i.e., sensing. Both, heating and sensing, can be measured mainly by electrical or optical methods and/or a combination of both.

At nanoscale, the introduction of electrical contacts is challenging due to the complexity of the fabrication process. In consequence, novel contactless characterization techniques for thermal conductivity (or thermal diffusivity) determination have been developed such as, e.g., time-domain thermoreflectance (TDTR), frequency-domain thermoreflectance (FDTR), thermal transient grating (TTG), the photoacoustic method and Raman thermometry.

In this lecture we will focus in the use of Raman thermometry as a novel contactless technique for the determination of the thermal properties in nanostructures. As a mode of example we will focus the lecture in the determination of thermal conductivity and thermal field distribution in single-crystal free-standing silicon nanomembranes. This lecture will be divided as follow:

1. Introduction to Raman scattering:
   - What is Raman scattering?
   - Phonon band structure in crystals
   - The scattering process: elastic and inelastic scattering.
   - Selection rules.
2. Raman spectroscopy: Instrumentation and measurement
   - Description of the equipment
   - Laser, filters and gratings
   - Measurement and analysis
3. Raman thermometry:
   - Raman as a thermometer
   - Single Laser Raman thermometry: experimental description
   - Thermal field distribution and two-laser Raman thermometry

- Analysis of the experimental results and theoretical approach.

Scientific background required: this course assumes basic knowledge of Raman spectroscopy and solid state physics. There is a brief introduction to Raman light scattering processes and phonon spectroscopy, but emphasis will be on the use of Raman as effective contactless technique for the measurements of thermal properties of nanostructures.

(III) Photoreflectance, Thermoreflectance (PARIS 5):

1. Thermal dependence of the refractive index. Role of the probe wavelength.
2. Measurement techniques : single point or full field imaging
3. Thermoreflectance : temperature measurements on active devices
4. Photoreflectance : thermal properties measurements on passive samples

Scientific background required:
- basics of heat diffusion (in the modulated or continuous regimes)
- basic optics (microscopy and lasers)

(IV) Time Domain ThermoReflectance (Bordeaux University):

1. Time and space scales for thermal transport
   1.1 Validity of Fourier law and assumptions coming from Macroscopic scales
   1.2 New approach for nanoscale heat transport
2. Optical Metrology of thermal conductivity
   2.1 laser material interaction (pump)
   2.2 Optimal temperature measurement (optical sampling, role of the wavelength
   2.3 Thermal properties identification (Non linear least mean square, sensitivity function)
3. Case studies
   3.1 Bulk materials
   3.2 Thins films
   3.3 Alloys

Electrical techniques

(I) Resistance thermometry (GU):

1. Temperature measurement: ITS90 and thermodynamic temperature
2. Resistance thermometry using precision platinum resistors
3. Johnson noise: derivation. Noise as a thermodynamic thermometry technique
4. Practical noise thermometry
5. Resistance thermometry with non ideal platinum resistances (thin films)
6. Self calibration of resistance thermometers using noise thermometry

(II) Thermal properties measurements at the nanoscale using electrical methods (Néel Institute):

1. Thermometry down to the nanoscale
   - 1.1.1 Resistive thermometry, 4 wire geometry, different materials (semiconductors, metal, metal to insulator transition), different temperature ranges.
   - 1.1.2 Scalability of these thermometries at nanometer length
   - 1.1.3 DC and AC measurements, limit in bias current, over heating effect, error in measurements
   - 1.1.4 Specificity of low temperature measurements (adapted thermometry, Kapitza resistance, T^5 law, electron phonon interaction)
2. Thermal measurements
   - 2.1 Principle, basic concepts of thermal relaxation time (C/K)
   - 2.2 Thermal conductance measurement, DC method
   - 2.3 Dynamic technique: the 3 omega method (thermal model for semi-infinite geometry, for nanowires with longitudinal heat flow)
   - 2.4 Extension to the measurement of specific heat: 1D and 2D thermal model.
3. Adapted nanofabrication for thermal measurements
   - 3.1 Clean room process for downscaling the thermometers
   - 3.2 Nanofabrication of suspended sensors based on SOI, GOI, and SiN (chemical etching, KOH, XeF_2, CH_3CF_4, HF)
   - 3.3 Some examples of suspended sensors and nano SThM tip
4. Application to the nanoscale
   - 4.1 Limitation of thermal measurements at low dimension (handling of sample, sensitivity and resolution, accuracy etc…), thermal problems linked to 1D or 2D systems.
   - 4.2 Measurement of individual nano-objects, measurement of very low energy, low noise measurement chain (3 omega, measurement platform, electrical set-up)
   - 4.3 Thermal transport at the nanoscale specificity of low temperature measurement (Casimir regime, Ziman regime, ballistic transport)
   - 4.4 Various examples of extreme measurements: membranes, nanowires, local probe etc…

SPM techniques for thermometry and thermophysical characterization of materials

(I) Scanning Thermal Microscopy (URCA)

1 - Short history of the Scanning Thermal Microscopy Some technics for measurement of local temperature and thermal conductivity

2 - Principles of functioning and imagery of scanning thermal microscopy

Principle of AFM

Principle of thermal imagery, Influence on imagery of different parameters as the probe shape, …

3 - Thermal probes with theirs principles of functioning and examples of applications for thermal conductivity

Wollaston probe, mode DC and AC

KNT probe

Silicon probe of Anasys

Probe of Wielgoszewski mode DC and AC….

4 - Probe-Sample heat Transfer

Mechanisms of heat transfer between a thermal probe and a sample (state of art) participating to measurement

Phenomena at long distance

Phenomena at short distance

5 - Limitations, precautions for the use of these technics

(II) Scanning Thermal Microscopy (GU)

1. Development of probes for Scanning Thermal Microscopy:
2. Choice of sensor type and cantilever material
3. Cantilever design. Compensation of differential expansion.
4. Stability and failure mechanisms: wear, electromigration and ESD
5. Isolation of the sensor using transformers
6. What are the free parameters in the production of a novel thermal probe?
7. Calibration: What is the temperature of the sensor? How does this relate to the temperature of the sample?
8. Null point SThM thermometry. Critique of the single sensor technique
9. Optimised probe design for null-point SThM

(III) Scanning Thermal Microscopy : approach of the measurement (CNRS)

After reminding the fundamentals of thermal metrology by contact, the lecture describes the approaches currently used to characterize measurements. In many cases, the link between the nominal measured signal and the investigated parameter is not yet fully understood 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.

I. Introduction: SThM Measurement Approaches

- temperature measurement
- thermal conductivity Measurement

II. Specification of SThM measurement through modeling

- Probe parameters
- Probe losses to the environment: h coefficient
- Effective parameters describing the probe-sample thermal interaction
- Approaches for smaller probes

III. Review of probe-sample heat transfer modeling

- Gas heat transfer
- Heat conduction at the tip-sample mechanical contact
- Water meniscus

Scientific background required: basic course of physics needed, some solids state physic knowledge helpful for better understanding.

AFM based nanoscale IR spectroscopy: Technology and Applications (ANASYS INSTRUMENTS)

Abstract: Infrared spectroscopy is a critically important technique for chemical composition but suffers from the major limitation that its spatial resolution is limited by optical diffraction to around 10 um. A new technique is now available that allows IR spectroscopy with spatial resolution below 50 nm. This technique, called AFM-IR, combines the complementary techniques of atomic force microscopy (AFM) and infrared (IR) spectroscopy to achieve this spatial resolution improvement by over 2000x. AFM-IR allows for detailed studies of nanoscale structure-chemical composition correlations on a uniquely broad range of application spanning physical and life sciences.

In this lecture, we will review the fundamental aspects of AFM-IR and draw examples from applications in:

- polymers, polymer blends/composites/laminates;
- materials for energy, that is, organic photovoltaics, biofuels, fuel cells;
- self-assembled monolayers and other thin films;
- materials physics, that is, semiconductors (joint work with Intel), plasmonics;
- life sciences, that is, amyloid fibrils, cells, bacteria, viruses, bone and tissue

AFM based nanoscale thermal and thermo-mechanical analysis: Technology and Applications (ANASYS INSTRUMENTS)

Abstract: Nanothermal analysis (nano-TA) enables highly localized thermal analysis using a self-heating probe of an Atomic Force Microscope (AFM). AFMs are ubiquitous imaging tools for samples at the nanoscale. One of the biggest drawbacks of AFM, however, is its inability to identify features or domains from the image. Recent breakthroughs in probe fabrication technology have resulted in the availability of nanoscale thermal probes which can measure phase transition temperatures of materials, allowing for localized characterization and identification of materials at the nanoscale. In nano-TA, the conventional AFM probe is replaced by one which can be heated, allowing thermo-mechanical measurements (including transition temperatures and thermal expansion) to be made on selected regions of the surface of a sample. This mode has found a range of applications, especially in the polymer and pharmaceutical sciences, including analysis of composites, polymer blends, and defect analysis to name a few.

The nanoscale thermal probes are silicon-based micromachined thermal probes (with a tip radius of a few 10s of nm) and can also be used for high resolution AFM imaging, including AFM measurements at variable tip tem¬peratures. Also due to the small thermal volume of these probes the rate of change of the temperature of the probe can be significantly higher than traditional thermal analysis. These nanothermal probes also offer dramatic potential for original research as the nanothermal probes act as a highly localized heat source can be used to raise the temperature of selected na¬noscale regions. These probes have been used to locally modify surfaces, initiate chemical reactions, induce and measure the thermoelectric effect, for example. This presentation will describe heated tip technology, nano thermal analysis and other heated tip applications.

Lorentz Contact Resonance for nanoscale mechanical spectroscopy:

Lorentz Contact Resonance (LCR) allows for the clean excitation of the resonance modes of a ThermaLever™ AFM cantilever. The resonant frequency and amplitude of these resonances are dependent on the stiffness of the material in contact with the probe. When tuned to a particular resonant frequency and scanned across a sample, the probe can obtain a qualitative map of the varying stiffness of each component on the surface of a sample. Each individual component can then be highlighted by tuning to its resonant frequency and scanning the surface. Another advantage of using a Thermalever™ probe is that the temperature of the probe can be ramped and corresponding changes in stiffness can be seen. This allows for the measuring of thermal transitions on materials that have traditionally been difficult to measure, such as, thin films and highly filled epoxys. The contact frequency between tip and sample will change as a sample undergoes a thermal transition, with the resonance shifting to a lower frequency as softening occurs. When LCR is combined with AFM-IR and nanoTA, samples can be characterized chemically, mechanically and thermally with nanoscale resolution.

Near-field infrared optical microscopy and spectroscopy (Langevin Institute, ESPCI)

Classical optical microscopy allows one to detect propagative fields and is limited in spatial resolution to approximately half the wavelength of observation. The latter prevents one in principle to perform nano-optical observations in the infrared, where the wavelength is typically in the range of tens of micrometers. For the same reason, Fourier transform infrared spectroscopy cannot achieve a spatial resolution better than tens of micrometers, which often restricts the technique to global investigations of large size samples.

In this lecture, the lecturer will present how the use of scattering type scanning near-field optical microscope allows one to go beyond the diffraction limit, and to achieve a resolution in the range of 100 nanometers, while performing the measurements at a wavelength of approximately 10 micrometers. The technique, which allows one to perform both infrared nanoscopic imaging and nano FTIR spectroscopy, may be applied in various fields such as materials characterization, plasmonics, etc. We will show its adaptation to the detection of the near-field thermal emission and show how the presence of surface waves (surface plasmons or surface phonon polaritons) can modify dramatically the coherence properties of near-field thermal emission.

Scientific background required: A general background in physics is necessary to attend this lecture. As it will deal mostly to experiments, there is no other specific background required.

Nanomechanical measurements (Asylium Research)

Understanding nano-scale mechanical properties is of fundamental importance for evaluating the behavior and performance of a wide variety of industrially, biologically and structurally important materials. An Atomic Force Microscope (AFM) tip interacting with a sample experiences forces originating from many different sources – elasticity, viscosity, adhesion, Van der Waals– to name a few.

Hence, it has become increasingly clear that reliable and accurate materials properties measurements require looking at yours ample in more than one way.

AFMs provide a suite of tools to meet the requirements of the nano-mechanics researcher, impressively powerful and rapidly expanding. The various tools are complementary each technique probes and records different responses of your samples.

The talk will approach different of these techniques:

- In details:
  - Force curves and modeling
  - Bimodal AC–AMFM
  - Loss Tangent
  - Contact Resonance
- Overview of other technics:
  - Fast Force Curves
  - Force Modulation
  - Vertical Nano-Indentation
  - Pulsed Force–Peak Force

Modelling in support for the interpretation and for the analysis of Measurement

(I) Heat conduction in nanomaterials: advanced modelling methods I (mesoscopic size) (CNRS):

- Boltzmann transport equation for phonons: theory of thermal conductivity
- Numerical methods: deterministic (Discrete Ordinates, Lattice Boltzmann) and stochastic (Monte-Carlo sampling) techniques
- Reduction of effective and equivalent thermal conductivities in nanomaterials due to confinement
- Phonon scattering at boundaries: specularity and diffusive behaviour
- Thermal boundary resistances: impact of transmission coefficients (Acoustic Mismatch Model, Diffusive Mismatch Model)
- Solids vs amorphous materials: energy-carrying modes (phonons vs extendons/locons)
- Nanocomposites: effective medium theory
- Heat conduction in polymers
- Brief insight into phononics: phonons as waves

Scientific background required:

- classical Fourier's law of heat conduction
- basics in solid state physics

(II) Atomistic modeling and heat transfer phenomena (CMI):

Programme:

- atomistic models of materials
- numerical modeling: classical and quantum physics approaches
- state of the art of atomistic heat transfer calculations
- modeling of irregularities and impurities, realistic models

Scientific background required: basic course of physics needed, some solids state physic knowledge helpful for better understanding.

Near-field thermal radiation (CNRS):

- far-field thermal radiation breakdown: coherence effects and evanescent waves
- stochastic Maxwell equations and the fluctuation-dissipation theorem
- near-field thermal radiation between bodies for plane-plane and sphere-plane configurations
- numerical solutions of near-field thermal radiation exchange between bodies
- an overview on existing near-field thermal radiation experiments and their modeling (link with lecture “Near-field infrared optical microscopy and spectroscopy” from Langevin Institute, ESPCI)
- can we currently model near-field thermal radiation between a SThM tip and a sample?

Scientific background required:

- classical (far-field) thermal radiation between bodies
- basics in electromagnetism

Metrology

(I) Metrology: definitions, methods, traceability and standards (NPL):

The intention is to introduce what the basic ideas are behind metrology, explain why it is important and define basic terms. Then traceability and how all measurements need to be traceable back to fundamental units will be discussed. Finally, standardization process, the structure of standards, their importance and how they are developed (ISO, VAMAS etc) will be described.

(II) Measurement uncertainty (LNE):

1. The concept of uncertainty
2. The different methods of evaluation
    a. The GUM
    b. The GUM-S1 : Monte Carlo
    c. An alternative : interlaboratory comparison
3. Examples
4. Scope of the GUM and GUM-S1 and novel methods of evaluation

Scientific background required: Basic notions in metrology and basic notions in mathematics and statistics (such as mean value and standard deviation).

Contact for more information:

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