# **Multiscale study of the thermophysical properties using the 3omega technique**

#### **ESIEE Paris coordinators:** Georges HAMAOUI

**Laboratory:** ESYCOM lab, ESIEE Paris - Université Gustave Eiffel, France

## **National partner :** Nicolas Horny

**Laboratory:** Institut de Thermique, Mécanique, Matériaux (IThEMM), Université de Reims Champagne-Ardenne, France

#### **International partner:** Sebastian Volz

**Laboratory**: Laboratory for Integrated Micro-Mechatronic Systems (LIMMS), Institute of Industrial Science, University of Tokyo, Japan

**Master Programs:** CODS Connected Objects, Devices and Systems **ESIEE Programs:** ENE, SEI, SE

# **A. Master internship proposal**

#### **Context and motivations:**

Climate change is a paramount global challenge stemming from centuries of carbonbased fossil fuel consumption. While the energy sector is both a contributor to and a potential remedy for this issue, thermal energy management remains pivotal. Approximately 90% of worldwide energy processes involve heat manipulation across diverse temperature ranges. Simultaneously, achieving energy efficiency and transitioning to low-carbon technologies are critical components of global decarbonization efforts. Researchers are exploring technologies like thermal rectification,<sup>1</sup> thermoelectricity, radiative/conductive cooling, and thermophotovoltaic converters (TPV) to recover thermal energy efficiently.<sup>2</sup> Yet, these technologies often suffer from low efficiencies, necessitating a fundamental shift in materials design. Engineered metamaterials at the micro- and nanoscale, facilitated by advances in micro- and nano-fabrication techniques, offer unparalleled prospects for enhancing heat management and energy efficiency across a spectrum of applications.

## **Motivations: needs and challenges for accurate characterization of thermophysical properties**

Accurate thermal measurement is crucial for deriving energy-related parameters like heat flux and energy exchange. However, at the nanoscale, current thermal measurement techniques lack the required accuracy and resolution, hindering progress in science and engineering. For example, with modern nanofabrication techniques, thermoresistive and thermoelectric temperature sensors in the 100 nm range have been designed, $3$  breaking the 500 nm barrier of far-field photothermal techniques<sup>4</sup> and pushing sensor spatial resolution down to the sub-30 nm range.<sup>5</sup> Nevertheless, achieving accurate and reliable measurements of thermophysical properties presents several needs and challenges, including the selection of appropriate measurement techniques, sensitivity to environmental conditions and sample geometry, high spatial and temporal resolution requirements, and standardization for reproducibility. Addressing these challenges involves developing i) new measurement techniques, ii) sample preparation methods, and iii) data analysis approaches to characterize thermophysical properties at nanoscale and microscale. Establishing measurement standards and protocols ensures reproducibility and comparability, supporting the development and optimization of materials and technologies for various heat management applications.

#### **Scientific and technical barriers to be overcome**

Various experimental methods<sup>6</sup> have been developed to measure thermal transport at the nanoscale, such as thermoreflectance (FDTR, SDTR, TDTR), 3ω, scanning thermal microscopy (SThM), photothermal radiometry (PTR), laser flash method, Raman spectroscopy, and hot plate, among other methods.<sup>6</sup> However, further refinement is needed to enhance accuracy. Many methods are limited when measuring uncorrelated thermophysical properties (thermal conductivity k, density  $\rho$ , specific heat  $C_p$ , and thermal boundary resistance TBR). Overcoming these limitations requires using different frequency/time excitation ranges, such as FDTR/TDTR, which can measure independent thermophysical properties. Furthermore, in the nanoscale realm, the definition of heat transfer needs to be extended, as the notion of temperature difference only applies under local thermodynamic equilibrium, known as the diffusive regime. The mean free path, of energy carriers is considered the limiting length scale for heat transfer, but if one broadens the view of transfer as the rate of energy flow, non-equilibrium conditions need to be taken into account, known as the ballistic regime.<sup>7</sup>

**Hence, this project seeks to update our 3omega experimental setup, coupled to a 3D heat diffusion model, as a practical instrument to achieve metrological measurements of new materials applied in novel heat management applications.**

#### **Objectives**

This project focuses on multiscale characterization of innovative nanomaterials through the development of instrumentation (**Task 1**) and analysis protocols (**Task 2**), dedicated to such characterization, in order to meet the needs of observation and detection metrology currently missing in many French laboratories and industries including at ESYCOM lab. The objective is to extend the experimental characterization capabilities of the laboratory, the University Gustave Eiffel, by complementing the existing techniques by a new advanced 3omega setup (Task 1). With this new setup, thermal properties, such as thermal diffusivity  $a$ , thermal effusivity  $e$ , and TBR, will be measured for advanced carbon-based materials, such as diamond, silicon carbide (SiC), and 2D layers like graphene (**Task 3**). <sup>8</sup> These materials exhibit exceptional thermal conductivity properties and offer a unique opportunity to enhance heat dissipation in microelectronic devices, for example, like shown in **Figure 1**. 9,10



**Figure 1** (taken from refs. <sup>11</sup> and <sup>12</sup>, respectively): (a) Comparative thermal performance model (input power versus maximum package temperature)<sup>11</sup> for selected thermal management substrates in 20-pin SOIC, overlap construction geometry**; (b)** Thermal performance of an electronic chip with and without graphene-based film (GBF) and a silanefunctionalized graphene oxide (FGO). Inset: basic schematic of thermal evaluation chip structure.<sup>12</sup>

By updating this setup, the team aims to optimize heat transport in microelectronic devices and develop innovative and tunable thermal properties for carbon-based metamaterials.

#### **Master internship Program:**

The work plan includes the following five main steps.

1. Understanding the project goals through bibliography research at ESYCOM lab at the beginning of the project.

2. Experimental development of the 3omega setup at the ESYCOM lab.

3. Develop the 3D model and the measurement protocol.

4. Perform experiments and measurements at ESIEE Paris, which will be compared to those made at Reims by Pr Nicolas Horny and Pr Sebastian Volz teams using both the PTR and 3omega techniques.

5. Examining the relationship between microstructure and thermophysical properties of samples. This step comprises also bibliography research.

# **B. Brief scientific description of the teams involved**

## **Groups involved:**

The project brings together leading experts in the areas of the fabrication of novel materials using carbonaceous nanomaterials, the thermal characterization and the optical measurement.

# **ESYCOM**, **ESIEE Paris - Université Gustave Eiffel**

[ESYCOM Laboratory](http://esycom.cnrs.fr/) works mainly in the fields of engineering of communication systems, sensors and microsystems for the city, the environment and the person.

The topics covered are more specifically:

- Antennas and propagation in complex media, photonic components - microwaves;

- Microsystems for environmental analysis and pollution control, for health and the interface with living organisms;

- Micro-devices for recovering ambient mechanical, thermal or electromagnetic energy.

## **IThEMM – Université de Reims Champagne-Ardenne**

The scientific interests of the team deal with the development of metrologies and modeling to study physical mechanisms and properties of energy transport in nanostructured objects and at solid-solid interfaces. Nanoscale heat conduction, thermoelectric generation, thermophotovoltaic devices and near-field thermal radiation are the main research topics. The team has extensive skills in Photothermal radiometry (PTR) and thermal analysis at micro/nanoscale.

## **LIMMS, IIS - University of Tokyo**

The team, led by Sebastian Volz (CNRS Research Director - HDR), brings its expertise in the development of simulations and direct characterizations aimed at understanding the roles of phonons, electrons and photons in the thermal behavior of micro and nanostructures. Their involvement in large European projects related to thermoelectric micro-modules and thermal interface materials underlines their contributions. Sebastian Volz has been leading the CNRS's European Thermal NanoSciences and NanoEngineering network for 17 years (Springer, Topics in Applied Physics series, n° 107 and 118) and is currently director of the LIMMS.

## **References:**

- 1. Wong, M. Y., Tso, C. Y., Ho, T. C. & Lee, H. H. A review of state of the art thermal diodes and their potential applications. *Int J Heat Mass Transf* **164**, 120607 (2021).
- 2. Melnick, C. & Kaviany, M. From thermoelectricity to phonoelectricity. *Appl Phys Rev* **6**, 021305 (2019).
- 3. Zhao, S. & Wang, H. W. An Integrated H-type Method to Measure Thermoelectric Properties of Two-dimensional Materials. *ES Energy & Environment* **9**, 59–66 (2020).
- 4. Li, Z., Aleshire, K., Kuno, M. & Hartland, G. V. Super-Resolution Far-Field Infrared Imaging by Photothermal Heterodyne Imaging. *Journal of Physical Chemistry B* **121**, 8838–8846 (2017).
- 5. Zhao, D., Fabiano, S., Berggren, M. & Crispin, X. Ionic thermoelectric gating organic transistors. *Nat Commun* **8**, 1–7 (2017).
- 6. Xian, Y., Zhang, P., Zhai, S., Yuan, P. & Yang, D. Experimental characterization methods for thermal contact resistance: A review. *Appl Therm Eng* **130**, 1530–1548 (2018).
- 7. Hopkins, P. E., Norris, P. M., Stevens, R. J., Beechem, T. E. & Graham, S. Influence of Interfacial Mixing on Thermal Boundary Conductance Across a Chromium/Silicon Interface. *J Heat Transfer* **130**, 062402 (2008).
- 8. Liu, Y., Qiu, L., Liu, J. & Feng, Y. Enhancing thermal transport across diamond/graphene heterostructure interface. *Int J Heat Mass Transf* **209**, 124123 (2023).
- 9. Ekpu, M., Bhatti, R., Ekere, N. & Mallik, and S. Advanced Thermal Management Materials for Advanced Heat Sinks used in Modern Microelectronics. *IOP Conf Ser Mater Sci Eng* **814**, 1–8 (2020).
- 10. Fabisiak, K. & Staryga, E. CVD diamond: from growth to application. *Journal of Achievements in Materials and Manufacturing Engineering* **37**, 264–269 (2009).
- 11. Fabis, P. M. Material property, compatibility, and reliability issues in diamondenhanced, GaAs-based plastic packages. *Microelectronics Reliability* **39**, 1275–1291 (1999).
- 12. Zhang, Y. *et al.* 2D heat dissipation materials for microelectronics cooling applications. *China Semiconductor Technology International Conference 2016, CSTIC 2016* (2016) doi:10.1109/CSTIC.2016.7463960.