

# NanoIR Spectroscopy and Imaging: Recent Developments and Applications

Wednesday, March 24th, 2021 - 13:00 (CET)



## Program – Wednesday, March 24<sup>th</sup>, 2021

- 13:00**            **Welcome address**  
*Dr Miriam Unger, Applications Manager EMEA, NanoIR, Bruker Nano GmbH*
- 13:10**            **Photothermal AFM-IR Spectroscopy and Imaging: When AFM meets Infrared**  
*Prof Alex Dazzi, University Paris-Saclay, France*
- 13:40**            **Revealing the nanoscale infrared properties of strained graphene bubbles with scattering SNOM**  
*Tom Vincent, NPL and Royal Holloway, University of London, UK*
- 14:10**            **Live Demo: Performing s-SNOM Measurements on a hBN sample**  
*Dr Miriam Unger und Dr. Hartmut Stadler, Bruker Nano GmbH*
- 14:40**            **NanoIR Product Update**  
*Dr Miriam Unger*
- 14:50**            **Break**
- 15:00**            **Infrared Nanospectroscopy at the Single Molecule Scale**  
*Dr Francesco Simone Ruggeri, Wageningen University, Netherlands*
- 15:30**            **Epoxy-Amine / Iron oxide Interphase Formation and Oxidation**  
*Dr Suzanne Morsch, Manchester University, UK*
- 16:00**            **Live Demo: AFM-IR Measurements on a polymer sample**  
*Dr Miriam Unger und Dr. Hartmut Stadler, Bruker Nano GmbH*
- 16:30**            **Introduction of a new AFM-IR mode**  
*Prof Alex Dazzi*
- 16:40**            **Closing**  
*Dr Miriam Unger*

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## Talk abstracts

### Photothermal AFM-IR Spectroscopy and Imaging: When AFM meets Infrared

*Prof Alex Dazzi, Institut de Chimie Physique, Université Paris-Saclay, France*

The invention and development of the AFM-IR technique began because of a strong wish to go beyond resolution and push the limit of infrared microscopy in the Free Electron Laser Center at Orsay in 2004. The idea of AFM-IR is based on the coupling of a tunable infrared laser and an AFM (Atomic Force Microscope). The sample was irradiated with a pulsed nanosecond tunable laser in total reflection configuration to avoid tip illumination. If the IR laser is tuned to a wavenumber corresponding to sample absorption band, the absorbed light is directly transformed into heat. This fast heating results in a rapid thermal expansion, localized only in the absorption region. The thermal expansion is then detected by the AFM tip as a shock as the cantilever will oscillate on its own resonance modes. Because of the damping with the surface contact this oscillation will decrease in function of time (ring down). The 4-quadrants detector of the AFM records these oscillations. Thus, the detection scheme is analogous to photo-acoustic spectroscopy, except that the AFM tip and cantilever are used to detect and amplify the thermal expansion signal instead of a microphone in a gas cell. As oscillations amplitude detected by the AFM is rigorously proportional to the local absorption, recording for one tip position, the oscillations maximum as a function of laser wavenumber allows a local IR absorption spectra to be built up. This spectra correlates very well with conventional IR absorption spectra collected in FT-IR. In addition, mapping oscillations amplitude versus tip position for one specific wavenumber gives a spatially resolved map of IR absorption that can be used to localize specific chemical functions. After 14 years of development and improvement, the AFM-IR technique has now become a robust and efficient tool for infrared analysis at nanometer scale. The AFM-IR system works in contact or tapping mode with a sensitivity and resolution of around 5-10 nm and a spectra bandwidth of about  $0.5 \text{ cm}^{-1}$  (linked to the pulsed laser properties). The range of applications is huge covering diverse research areas like materials science, life science, and astrochemistry.



*Alexandre Dazzi is a tenured Professor of Physics at Université Paris-Saclay and works at the Institut de Chimie Physique. His research focuses on the infrared and nanoscience domain. After inventing and developing the AFM-IR technique, he has worked on improving AFM-IR instrumentation and focused on biological applications. He now has a user facility and collaborates with various groups in different domains like astrophysics, culture heritage, polymer science, and microbiology. He was the 2009 laureate for France's national instrumentation prize from the Société Française Division de Chimie Physique and received the Ernst Abbe Award in 2014 from the New York Microscopical Society.*

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## Revealing the nanoscale infrared properties of strained graphene bubbles with scattering SNOM

*Tom Vincent, NPL and Royal Holloway, University of London, UK*

Graphene is a promising material for nanophotonic devices which manipulate light at the nanoscale. Traditional optical microscopy, however, lacks the resolution needed to measure such subwavelength features. This talk will show how scattering SNOM was used to directly image the nanoscale light-matter interaction from bubbles in graphene heterostructures. This revealed pronounced domains within the bubbles, with strongly enhanced absorption. Colocalised measurements of topography, strain and charge doping from the same bubbles suggest that these domains are caused by strain. This shows an interplay between graphene's strain and its optoelectronic properties, which could form the basis for future tunable nanophotonic devices.



*Tom Vincent joined the Quantum Materials and Sensors group at the UK's National Physical Laboratory (NPL) as a research student in 2017, after finishing his undergraduate degree in physics at the University of Surrey. His studentship is held jointly between NPL and Royal Holloway, University of London. His work studies 2D materials, with a particular focus on the interplay between light-matter interactions and the physical features of real 2D materials, such as fractures, strains and bubbles.*

## Infrared Nanospectroscopy at the Single Molecule Scale

*Dr Francesco Simone Ruggeri, Wageningen University, Netherlands*

Here, we will demonstrate the application of infrared absorption nanospectroscopy (AFM-IR) as a real breakthrough for the analysis of heterogeneous biological samples at the nanoscale. AFM-IR exploits the combination of the high spatial resolution of AFM (~10 nm) with the chemical analysis power of IR spectroscopy. We first prove that AFM-IR is a versatile technique to study biomolecular processes bridging multiple biological scales to unravel the structure of functional and pathological protein self-assemblies, the physical-chemical organization of chromatin and structure of protein in single cells. Then, as a major advance in the field, we will demonstrate the achievement of single protein molecule detection of infrared absorption spectra and maps by introducing off-resonance, low power and short pulse ORS-nanoIR. The technique enables the accurate determination of the secondary structure elements of single proteins in the amide band I region. Then, we will show the application of this single molecule sensitivity to unravel the molecular interaction fingerprint between a small molecule and its target, to study the surface properties of artificial model membranes and the crystal state of protein aggregates.



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*Dr Francesco Simone Ruggeri joined the chair groups of Organic Chemistry and Physical Chemistry of Wageningen University as Assistant Professor in 2020. Before this, he completed his independent Junior Research Fellowship at the Darwin College and post-doctoral research at the Department of Chemistry & Centre for Misfolding disease at the University of Cambridge, UK. He holds a PhD in biophysics obtained in 2015 at the École Polytechnique Fédérale de Lausanne (EPFL) in Switzerland, where he acquired a strong expertise on scanning probe microscopy and single molecule methods.*

*Dr Ruggeri has developed and applied single molecule infrared nanospectroscopy to study the chemical and structural properties of biological systems that are challenging to access using conventional bulk biophysical methods. Furthermore, he was the first to demonstrate the application of peak-force tapping mode and infrared nanospectroscopy (AFM-IR) to correlate the nanomechanical, chemical and structural properties of biological samples at the nanoscale in air and liquid environment. As a major advance in the field of microscopy and spectroscopy, he has also demonstrated that infrared nanospectroscopy is capable of acquiring the chemical fingerprint and secondary structure of biological samples in native liquid environments and at the single biomolecule scale. His approach has led to new insights into the formation and structural characterization of the misfolding of proteins and their correlation with the onset of neurodegenerative disorders, as well as unravelling the chemical and structural properties of biomaterials.*

## **Epoxy-Amine / Iron oxide Interphase Formation and Oxidation**

***Dr Suzanne Morsch, Corrosion and Protection Centre, School of Materials, The University of Manchester, UK***

The physicochemical properties of network polymer / metal oxides interphase regions determine the performance of many advanced composites, adhesives and protective coatings. Historically, extensive interphase structures within epoxy-amine networks have been proposed on the basis of under-curing in thin films. Recent infrared microspectroscopy studies have, however, concluded that for epoxy-amine networks, the chemistry of thin films does not accurately represent the buried interphase region. The nature and origins of the buried interphase structure have thus remained elusive, since these nanoscale regions are inaccessible to most organic analysis techniques. Here we describe the use of AFM-IR analysis to unambiguously confirm the presence of nanoscale off-stoichiometric interphase regions in epoxy-amine / iron oxide interphase regions for the first time, and to demonstrate that at > 100 nm, no chemical gradient exists towards the interface. Furthermore, diffusion limited oxidation (DLO) is shown to initiate at the buried polymer-metal interface and progress slowly, resulting in chemical gradients of <400 nm after 28 days exposure to mild thermal aging conditions (70 °C, 14% RH).

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*Dr Suzanne Morsch obtained her PhD in Surface Science from the University of Durham in 2013, for research focused on AFM lithography and plasma polymer films. Since 2013 she has held a role first as the lead Research Associate, now as Research Fellow, in the AkzoNobel Laboratory for Corrosion Protection in the Department of Materials at the University of Manchester. Her research focuses on the relationship between nanostructure, small molecule transport and degradation of polymer networks.*

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