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WIRMS 2017

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WIRMS 2017

WIRMS 2017 delegates at Worcester College, Oxford.

In September 2017, the International Workshop on Infrared Microscopy and Spectroscopy with Accelerator-based Sources (WIRMS) paid its first visit to the UK. Organized by Diamond Light Source, a vibrant international community came together for four packed days among the spires of the historical Worcester College of Oxford University to discuss the latest developments in infrared (IR) microscopy, spectroscopy including Terra Hertz (THz), and imaging by synchrotron radiation (SR) and free electron lasers (FELs).

Day one started with facilities updates. At Brookhaven (USA), three IR beamlines are approved for NSLS-II, with one under construction, the others awaiting funding, and a fourth proposed. A range of extreme environments are planned for material and physical sciences from the start, and first light was expected in May 2018. In Brazil, two years of IR activity at LNLS have shown results in 2D materials and nano-optics. The ultra-low-emittance (ULE) light source SIRIUS is currently under construction, with the first beam expected by the end of 2018. IMBUIA is the planned IR micro- and nano-spectroscopy beamline. In Japan, the proposed upgrades to SPring-8 join the global trend towards ULE rings, which presents some challenges for IR wide front ends, so the key targets are maximizing the brightness at IR beamlines, as well as the THz output, and investigating the potential of low energy rings and alternative sources. At Berkeley (USA), ALS has a new beamline for synchrotron nano-spectroscopy under commissioning and an advanced project for IR spectro-micro-tomography. The first IR beamline at the Indian SR source, Indus-1, has been looking at molecular solids under high pressure and low temperature conditions. Recent additions to the newly operational IR beamline MIRAS at ALBA in Barcelona include new sample compartment setups, such as a temperature-controlled stage, a freeze dryer, and a micro-compression cell.

As for FELs, in the Netherlands, the FELIX laboratory is home to two FELs and three instruments: FELICE, FLARE, and FELIX. The FEL beamlines have a strong connection to the High Field Magnet Laboratory (HFML) for IR experiments in very high magnetic fields. In Italy, TeraFERMI extends the capabilities of the FERMI seeded FEL in terms of peak power and ultrafast temporal structure into THz spectroscopy.

In the afternoon, the focus switched to biomedical applications. Progress in IR microspectroscopy was reviewed in terms of enabled spectroscopic and imaging experiments *in vivo* and at the single cell level at Jagiellonian University, Poland. Wax physisorption combined with microFTIR at Taiwan University could lead to a novel, fast method for early diagnosis of inflammatory kidney disease and cancer tissue recognition. A multicenter study by a group from Barcelona University using synchrotron-based microFTIR on glioma cells was able to disentangle the biochemical processes and variations of biomolecules induced by radiotherapy treatment on a cell by cell basis. Among the capabilities of synchrotron-based IR microprobes, a Manchester University-Diamond (UK) collaboration demonstrated how to monitor the response of drug-resistant leukemic cells to different chemotherapeutic drug doses. A study from SOLEIL revealed that the IR spectrum of a pluripotent

Dr. Ann Fitzpatrick speaking at WIRMS 2017.

stem cell can give information on the position of the cell in the stemness scale, which may help in determining lineage commitment and classifying cells based on the cell lineage. A study led by researchers from Osaka University showed how SR-IR spectroscopic imaging at SPring-8 allows the detection of the spatial distribution with a diffraction-limited resolution, together with the depth information of the racemic region. μSR-FTIR studies have been used to identify the differences between the three layers of a human aortic valve by a research group from the Institute of Nuclear Physics in Poland. An SR-IR microscopy study at ALBA, Spain, of amyloid plaques in the brain, one of the hallmarks of Alzheimer's disease, shows that granular non-fibrillary aggregates cause more oxidation than fibrils. A new methodology combining electrochemical control with SR-IR microspectroscopic imaging to respectively generate and verify well-defined redox states in metalloprotein single crystals was presented by a group from the University of Oxford.

The afternoon concluded with a poster session, sponsored by Novae Lasers, with participants supported by drinks and nibbles toward the end of the long first day.

Day 2 began with sessions on physics, chemistry, and materials. A summary of recent investigations of guest binding kinetics (mostly by UK research groups collaborating with Diamond) in nanoporous metal-organic frameworks (MOFs) was presented, along with a demonstration of a localized photoresponsive effect in the crystals, controlling the motion of guest species. Coupling spectroscopic results in the mid- and far-IR at IRIS (BESSY-II) with small-angle X-ray scattering measurements allowed the identification of the key parameters related to the dynamics of the ionic network in Nafion membranes, an important component in proton-exchange based fuel cells. The AILES beamline at SOLEIL was used to provide the first THz spectroscopic study of the superconducting phase discovered in sulfur hydride, suggesting an essentially conventional mechanism of superconductivity.

At SISSI of ELETTRA-Trieste, the IR signature of a pressure-induced topological Lifshitz transition was identified in the orthorhombic phase in black phosphorus, which is emerging as an important electronic material with high electron mobility and tunable infrared bandgap. Recent work by an Oxford group combining THz spectroscopy (MIRIAM at Diamond) with neutron scattering (ISIS) and density functional (DFT) modelling has provided unambiguous evidence of cooperative THz framework dynamics below 3 THz in a low-symmetry MOF structure. *In situ* SRIR spectroscopic measurements at high pressure, conducted at the Swiss Light Source and SOLEIL, led by researchers from Imperial College London, show that the major types of organic molecules found on Earth have differing resistance to pressure, suggesting that the transfer of organic matter from the biosphere to the geosphere involves selective degradation of some materials which can be used as a diagnostic of deep earth environments. In a natural serpentine sample, one of the abundant hydrous minerals making up the subduction zone of the Earth, and for which the physical properties are known to be strongly dependent on hydrogen bonding, the absorption bands attributable to OH-stretching showed positive pressure dependencies in measurements at the BL43IR beamline of SPring-8.

Discussion of technique development began in the afternoon of the second day. An initial exploration by the Canadian and Diamond

Opening session by Gianfelice Cinque at WIRMS 2017.

Light Sources found that a new micro-Photo-Acoustic Spectroscopy (microPAS) method could achieve the 100 μm spatial scale for analyses of optically difficult samples such as scattering beads and fibers. A focal plane array (FPA) detector has been coupled to the IR beamline in Melbourne-ALS, using multiple mirrors to improve the homogeneity of the SR illumination. An overview on data analysis/processing via new tools from ELETTRA included IR Orange. Based on the Orange Canvas, which is open-source, user-friendly, and cross-platform, it allows data analysis and machine learning through a visual interface. Supercontinuum IR sources are commercially available from NKT Photonics, now covering the near-IR and partially into the mid-IR.

The second day ended with a boat trip in good weather for all participants on the Thames River, sponsored by Specac, in line with the classic water theme typical of WIRMS events.

On the third day, the near-field IR session featured an overview from the University of Colorado on how IR nanoimaging is now being used to show more details of molecular structure, and for chemical identity of vibrations, correlated analysis, and peak positions for molecular interaction and dynamics. A project at MLS in Germany aimed to find a corrosion-resistant material for a special reactor, via surface analysis using nano-FTIR. The advantages of s-SNOM at the ALS in California versus photothermal nanoIR were discussed: nanometer spatial resolution, wavelength independence, soft and hard matter, amplitude and phase of optical field. Low-temperature SNOM in combination with FEL at Dresden Germany offers resonant excitation, spectroscopy, and imaging and can be used with complementary AFM techniques.

From the new techniques session, a new mid-IR spectrometer has been developed for non-cyclic or slow cycling processes at BESSY in Germany. The aim was to develop a method capable of time-resolved IR in biophysics. 3D chemical imaging is being developed to overcome the current limitations of 3D histology/pathology by sequential sectioning done between Bordeaux University in France and Academia Sinica in Taiwan. Full-field IR microscopy via FPA detector in combination with SR has been experimentally proven via a novel use of adaptive optics for optimal illumination of larger field of view in IR imaging at Diamond.

VERTEX 80V + verTera gives the world's first hybrid FTIR/cw THz spectrometer by Bruker. neaSNOM by Neaspec is a near-field scanning optical microscope offering broadband nano-FTIR, that has been coupled to SR-IR at ALS, MLS, LNLS, and PAL for testing.

The THz research session included a study on ultrafast detectors, room-temperature diode-based systems made at RAL Space in the UK, then synchronous 2D scanning on materials using pulsed THz-FEL in Osaka, Japan, and studies done on the longitudinal bunch dynamics observed at the CLS in Canada, all done using coherent synchrotron radiation. A test accelerator (t-ACTS) is under development at ELPH lab in Japan as a coherent THz source.

Day 3 came to a close with talks on Multidisciplinary Applications, which continued on the next day. An overview from the Polytechnic University of Catalonia was given on how SR-IR investigations can help us understand how and when paintings were created, and the underlying causes of degradation. Collective Enhanced SR-IR Absorption (SR-CEIRA) is a promising method of increasing the sensitivity of FTIR at ELETTRA, to allow conformational studies of proteins of biological and biomedical relevance in an aqueous environment.

A session on technique development started with a discussion by SOLEIL in France of recent developments on other IR sources, which are becoming increasingly available to complement the capabilities of synchrotron IR facilities. Two industrial talks were given, showing the capabilities for high magnification imaging in IR by Agilent, and the latest developments in IR nanoscale imaging by Anasys. A multi-technique study focused on the preparation of microparticles in view of the analysis of, e.g., asteroids was presented from SOLEIL and a Paris and Orsay collaboration.

SR-IR is a powerful technique for characterizing the hydrous and organic phases in water-rich meteorites at high spatial resolution. A study by the Natural History Museum, UK, and Diamond investigated the hypothesis that Earth's water was delivered by an asteroid.

AFM-IR is unravelling the mysteries of historical musical instruments; research by Paris-Saclay University examined the materials used in their construction and the processes involved in their degradation.

The conference dinner took place in the historic setting of the dining hall of Exeter College, the fourth oldest college in the University of Oxford. With attendees gathered around long wooden tables dating back to 1618, Mark Tobin from the Australian Light Source gave his keynote speech, with some official and unofficial highlights from past WIRMS conferences.

With concluding remarks by Mike Martin from ALS, and the announcement that WIRMS 2019 will be held in Brazil, the participants moved to the Rutherford Appleton Laboratory for a site visit. Three facilities were shown—Diamond Light Source, ISIS Neutron and Muon Source, and the Central Laser Facility—to demonstrate the complementary methods offered at the RAL site, and the synergy now necessary among labs to tackle the complexity of new science cases.

Peer-reviewed proceedings of the WIRMS 2017 conference are currently under publication in a virtual special issue of Elsevier's *Infrared Physics and Technology* journal. ■

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