

**MS25 O1**

**Simultaneous microRaman spectroscopy and X-ray microdiffraction** Richard J. Davies, Manfred Burghammer, Christian Riekel. *European Synchrotron Radiation Facility, Grenoble, France.*  
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Raman spectroscopy and X-ray scattering are complementary techniques. Whilst one provides information relating to molecular bond energies and orientations, the other probes a sample's crystallographic structure and morphology. Thus, not only do they provide differing information, but this relates to quite different length scales within a materials' hierarchical structure. However, the two techniques also share several similarities. They are non-contact and non-destructive for many materials, and neither requires modified or coated samples. As a result, both are used extensively for in-situ studies such as investigating the effects of deformation, temperature or pressure, and are frequently reported together. Over recent years Raman spectroscopy and X-ray scattering have independently evolved microfocus capabilities. With smaller laser and X-ray beams, smaller structural heterogeneities can be resolved within an individual sample. Microfocussing also allows confined sample volumes to be studied and reduces averaging when performing in-situ experiments. However, microscopic sampling can be problematic for combinatorial studies. In particular, many materials are heterogeneous over such length scales. This makes it difficult to ensure that data collected sequentially is from the same point on the specimen. Meanwhile, for in-situ studies, it is very difficult to reproduce dynamic behaviour in sequential experiments. Many of these difficulties can be overcome by collecting both data sets simultaneously in-situ. This capability is available at the ID13 beamline of the ESRF. Its combined  $\mu$ Raman and  $\mu$ XRD setup delivers the laser and X-ray beams to the same point on the sample simultaneously using an on-axis geometry, [1]. At the common focal position both beams have a spot size of approximately 1  $\mu$ m, [1]. This novel tool for materials' characterisation ensures a common sampling location on the specimen and consistency during dynamic studies. In addition, it also reduces experimental time, and is beneficial for rare and valuable samples. The system's specifications and capabilities have been demonstrated using laboratory-produced specimens, [1]. Meanwhile, its novel ability to access both the crystalline and amorphous fraction has already provided a new insight into structure-property relationships in high-performance fibres, [2].

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**MS25 O2**

**Multiwavelength Anomalous Diffraction (MAD) and Diffraction Anomalous Fine Structure (DAFS) in the study of structural properties of nanostructures** H. Renevier<sup>a</sup>, J. Coraux<sup>b</sup>, M.G. Proietti<sup>c</sup>, V. Favre-Nicolin<sup>b</sup>, B. Daudin<sup>b</sup> <sup>a</sup>Institut Polytechnique de Grenoble, *Laboratoire des Matériaux et du Génie Physique, UMR5628 CNRS-INPG, INPG-MINATEC, 3 parvis L.*

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The knowledge of strain, chemical composition, intermixing at the interfaces, are of great importance to understand the growth mechanism as well as the electronic and optical properties of hetero and nanostructures. Moreover, to be suitable for devices, the nanostructures are encapsulated or embedded in a superlattice and capping plays a decisive role in the modification of the optical properties by modifying the strain and possibly inducing atomic diffusion. Strain is closely related to composition, shape and aspect ratio of the nanostructures, and on the mutual stress which nanostructures, substrate and the matrix apply to each other. X-ray diffraction is known to be a powerful tool for measuring strain fields. The combination of MAD which allows to extract the scattering amplitude of the resonant atoms, and DAFS, which allows to determine the local environment of atoms located in an iso-strain volume selected by diffraction, is a very powerful approach to disentangle strain and composition. A MAD or a DAFS experiment consists in recording the scattered intensity as a function of the incoming x-ray beam energy in the vicinity of absorption edges. Like X-ray Absorption Fine Structure (XAFS), DAFS provides information about the local environment of the resonant atom (also known as the anomalous atom). We will give a brief insight on the basic principles of the MAD and DAFS methods. Then we will report examples of *grazing incidence* MAD and *grazing incidence* DAFS studies of the structural properties of GaN Quantum Dots and InAs Quantum Wires (embedded or not) [1,2,3,4,5]. We will also give a comparison with glancing angle x-ray absorption (XAFS) studies and show the complementarity of XAFS and DAFS.

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**MS25 O3**

**RDF analysis, Positron Annihilation and Raman Spectroscopy of  $x\text{TiO}_2$ -(60-x)  $\text{SiO}_2$ -40 $\text{Na}_2\text{O}$  nonlinear Optical Glasses: III. Non-bridging oxygen bonds tracing and structural analysis** A. Abou Shama<sup>1</sup>, M.S. Abd El-Keriem<sup>1</sup>, M. Abdel Baki<sup>2</sup>, F. El-Diasty<sup>1</sup> <sup>1</sup>Physics Department, Faculty of Science, Ain Shams University, 11566 Abbassia, Cairo, Egypt <sup>2</sup>National Research Center, Glass Department, Dokky, Giza, Egypt.  
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**Keywords:** nonlinear optical properties, RDF analysis, Positron annihilation.

$x\text{TiO}_2$ -(60-x) $\text{SiO}_2$ -40 $\text{Na}_2\text{O}$  glasses have proven an interesting nonlinear optical properties [1]. The investigated glasses show one order of magnitude

enhancement over some TiO<sub>2</sub> silicate glasses. In this work, we continue studying these glasses using three different techniques to analyze the glass structures seeking to provide a deep insight for the relation between structure compositions and the optical characteristics of these glasses. The Radial Distribution Function analysis (RDF) combined with Raman spectroscopy is used to study these glasses. Positron annihilation life time spectroscopy and Doppler broadening measurements are carried out to investigate the change in the glass structure as the incorporation of TiO<sub>2</sub> concentration into glass. The origin of the non-bridging oxygen (NBO) bonds has been traced correlating their existence with the measured nonlinear optical properties of the investigated glasses.

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#### MS25 O4

**Neutron diffraction, inelastic neutron scattering (INS) and DFT study of levoglucosan (1,6-anhydro-β-D-glucopyranose)** Mariana Sládkovičová, *Institute of Inorganic Chemistry, Slovak Academy of Sciences, Dúbravská cesta 9, SK-845 36 Bratislava, Slovak Republic.* E-mail: [Mariana.Sladkovicova@savba.sk](mailto:Mariana.Sladkovicova@savba.sk)

**Keywords: neutron diffraction, INS, DFT**

The study reported here is part of an ongoing project focused on obtaining an accurate description of hydrogen bonding in selected compounds using a combination of diffraction and spectroscopic methods with quantum chemical calculations. Neutron diffraction data are used to provide accurate information on the geometry of hydrogen bonds, while INS spectra are applied to obtain information on the dynamics of hydrogen atoms in the structure. Such information is then compared to theoretical calculation done for an isolated molecule with simulated surroundings and for crystal. The aim of the present study is to provide a full and accurate description of the hydrogen bonds in the structure of a monosaccharide levoglucosan (LG). The

structure of LG was accurately determined by refinement of time-of-flight neutron single crystal diffraction data taken at 30 and 100 K [1,2]. Molecules of LG are held together by a hydrogen bond array formed by a combination of strong O-H...O and supporting weaker C-H...O bonds. The strong hydrogen bonds link molecules in finite chains, with hydroxyl oxygens acting as both donors and acceptors of hydroxyl hydrogen atoms. The accurate specification of hydrogen bond geometry from neutron diffraction experiment allowed the structure to be correlated with DFT calculations and INS experiment. Hydrogen bonds influence the geometry of LG both, in vacuum [2] and in crystal structure [1] and their formation is reflected in the INS spectrum of crystalline LG [3]. The INS spectrum of LG is rather complex, characteristic for highly mixed modes. Most of the peaks are observed below 1500 cm<sup>-1</sup>. Here, characteristic CH and OH bending, torsional, and skeleton regions were defined, although there are no strict borders between them. In the higher parts of the spectrum only badly resolved CH and OH stretching modes appear (2800-3500 cm<sup>-1</sup>), mostly buried in intensive phonon wings. Wavelet analysis helped to localize the OH stretchings. Detailed interpretation of observed spectra and the assignment of peaks were accomplished by analyzing theoretically obtained spectra from periodic DFT calculations. Molecular dynamics calculations in solid state helped to interpret peaks in the lowest part of the experimental spectrum (<200 cm<sup>-1</sup>). Influence of hydrogen bonds on the spectrum was studied using molecular DFT approach. The calculations indicate strong frequency shifts due to O-H...O bonds and to noticeable extend due to weaker C-H...O contacts in bending and stretching regions. In the stretching region red shifts (~250 cm<sup>-1</sup>) of O-H and blue shifts (~30 cm<sup>-1</sup>) of C-H frequencies are predicted. The highest blue shifts (~300 cm<sup>-1</sup>) are predicted for OH torsional modes.

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