



# SyNeW 2015

Dutch-Belgian Synchrotron/Neutron/FEL Workshop  
and satellite mini-schools

Plenary sessions:  
Jaarbeurs, Utrecht  
June 1, 2015

Mini-schools  
Buys-Ballot building,  
The Uithof, Utrecht  
June 2, 2015



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# Preface

SyNeW 2015 brings together Dutch and Belgian users of synchrotron, neutron, and free-electron-laser facilities. It provides a forum to exchange scientific ideas, discuss our participation in large facilities, and reflect on the needs of frequently employed experimental stations & beamlines. The meeting will follow the traditions set by meeting in The Hague in 2006, Brussels in 2009, Amsterdam in 2011, and Antwerp in 2013. On June 1 2015 the stage is set for an overview of the latest scientific results and most recent developments in various research fields that use modern x-ray and neutron sources.

Synchrotron, neutron and free-electron laser (FEL) facilities have become versatile and indispensable tools in physics, chemistry, and biology including many interdisciplinary fields such as soft matter, novel smart materials, cultural heritage, and environment. They are crucial for many topics of fundamental research and essentially contribute to the solution of major societal challenges.

Belgium and The Netherlands participate together in the European Synchrotron Radiation Facility (ESRF) as the Benesync consortium. A very important and successful example of Dutch-Flemish collaboration is the joint beamline BM-26 DUBBLE. Since its opening 15 years ago on June 5, 2000, it has contributed to over 1000 publications (scientific papers + PhD theses). The celebration of this outstanding result is a part of the programme of SyNeW-2015.

Moreover, this meeting is meant not only to follow the old traditions and to celebrate results achieved in the (recent) past. The scope of the meeting is further developed by introducing three satellite mini-schools, which will be take place on June 2, the day after the plenary sessions and poster presentations on June 1.

This booklet provides information about the programme of the SyNeW-15 meeting and presents abstracts of the lectures and poster contributions on June 1 at the Jaarbeurs in Utrecht. Moreover, it gives the summary of the programme of the satellite mini-schools and gives other useful practical information.

We hope you enjoy the meeting and the satellite mini-schools,

**Andrei Petukhov** and **Martin Feiters**, on behalf of the organization committee

# Committee Members

## Scientific Advisory Committee

**Chair:** Martin Feiters (RU Nijmegen)

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Renko de Vries (WUR Wageningen)

Hans Hilgenkamp (UT Twente)

Thom Palstra (RUG Groningen)

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Mark Golden (UvA Amsterdam)

Kees de Kruif (UU Utrecht)

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Loes Kroon-Batenburg (UU Utrecht)

Wim Bouwman (TUD Delft)

Wim Bras (DUBBLE Grenoble)

Burak Eral (UU Utrecht)



# Programme

Monday, June 1, 2015

9:30	Registration + coffee	
<b>Session 1</b>	Chair: <b>Henk Schenk</b> (Amsterdam, NL)	
10:10	<b>Wim Bras</b> (DUBBLE/NWO)	The DUBBLE project: a historical note
	<b>Elisabeth Monard</b> (FWO)	Dutch/Belgian collaboration in large-scale facility projects: present & future
	<b>Jos Engelen</b> (NWO)	
	<b>Harald Reichert</b> (ESRF)	Role of CRG beamlines & Phase II upgrade programme
<b>Session 2</b>	Chair: <b>Bart Goderis</b> (Leuven, BE)	
10:40	<b>Tony Ryan</b> (Sheffield, UK)	Synchrotron studies of Polymer Motility
11:10	<b>Carlo Lamberti</b> (Torino, IT)	Understanding activation, working conditions and deactivation of catalysts: an overview of experiments at DUBBLE
11:40	<b>Cedric Gommès</b> (Liège, BE)	Small-angle scattering in porous materials: a review highlighting data analysis challenges
12:10	Lunch + posters	
<b>Session 3</b>	Chair: <b>Katja Pappas</b> (Delft, NL)	
14:00	<b>Ekkes Brück</b> (Delft, NL)	Cool magnets and hot electrons
14:30	<b>Alexei Kimeľ</b> (Nijmegen, NL)	Femtosecond Optical Control of Magnetism: from fundamentals to nanoscale dynamics revealed by X-rays
15:00	<b>Jos Oomens</b> (Nijmegen, NL)	Infrared ion spectroscopy
15:30	Coffee break	
<b>Session 4</b>	Chair: <b>Bauke Dijkstra</b> (Groningen, NL)	
15:50	<b>Thomas Barends</b> (Heidelberg, DE)	Biological Crystallography with XFELs
16:20	<b>M. Jayne Lawrence</b> (London, UK)	Neutron scattering studies of the molecular mechanism of the anti-fungal drug, amphotericin B
16:50	<b>Serena de Beer</b> (Mülheim, DE)	X-ray spectroscopic studies of biological dinitrogen reduction in molybdenum and vanadium nitrogenases
17:20	Closing	
17:30-18:00	Reception / drinks	







# **Abstracts of Invited Lectures**

## i01 Synchrotron studies of Polymer Motility

**Anthony J Ryan**

Faculty of Science, The University of Sheffield, Brookhill Sheffield, S10 2TN, UK

The cartoons routinely used in newspapers and on the TV to describe nano-therapeutics are causing serious damage to our credibility. There is a lack of visually arresting but scientifically accurate images of soft matter and nanotechnology. Aside from the technical difficulty of finding pictures of things that are literally too small to be visualised, one of the biggest problems is the prevalence of the “Fantastic Voyage” theme. Many excellent pieces on nanotechnology are let down by being illustrated with library pictures of nanoscale submarines and devices that are scientifically quite absurd, but which because of their high visual impact appeal to picture editors. But why is the “Fantastic Voyage” dream so enduring? Will it be possible to build a device that swims around the body? Will it be the shrunk submarine envisaged by Hollywood and Microsoft Encarta? I don’t think so. But what will it be? We think something soft and wet, just like biology, and have built a series of biomimetic devices. Our progress in the development of generic molecular devices based on responsive polymers will be discussed.

Spatially separating regions of different chemical potential is one of the principal mechanisms of biology. The formation of vesicles from block copolymers will be reviewed and the effects of polymer molecular weight and composition on the membrane structure and properties discussed in terms of their applications.

A scalable synthetic muscle has been constructed that transduces nanoscale molecular shape changes into macroscopic motion. The working materials, which deform affinely in response to a pH stimulus, are self-assembled block copolymers comprising nanoscopic hydrophobic domains in either a weak polyacid or a weak polybase matrix. Devices have been assembled where the muscle does work on a cantilever and the force generated has been measured. When coupled to a chemical oscillator this provides a free running chemical motor that generates a peak power of 20 mW/kg by the serial addition of 10nm shape changes that scales over five orders of magnitude. It is the nanostructured nature of the gel that gives rise to the affine deformation and results in a robust working material for the construction of scalable muscle devices.

A “push-me pull-you” device has been constructed such that the simultaneous expansion and contraction of polyacid and polybase materials are coupled in response to an oscillating reaction. The electrospinning of nanofibres with an increased surface to volume ratio has also been investigated to biomimetically improve muscle design.

## i02 Understanding activation, working conditions and deactivation of catalysts: an overview of experiments at DUBBLE

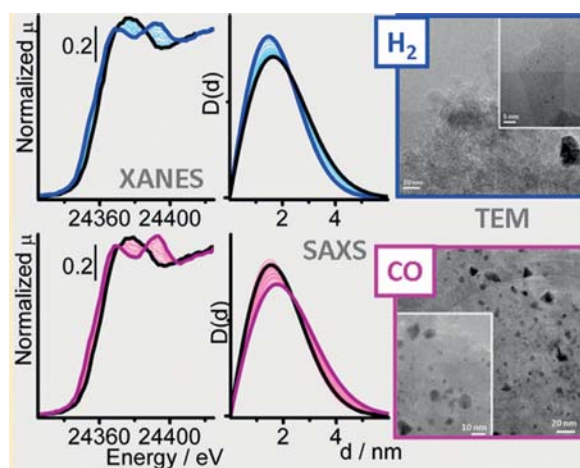
**C. Lamberti**,<sup>1,2</sup> G. Agostini,<sup>3</sup> E. Borfecchia,<sup>1</sup> F. Giannici,<sup>4</sup> G. Portale,<sup>5</sup> A. Longo,<sup>5</sup> W. Bras,<sup>5</sup> R. van Silfhout,<sup>6</sup> K.A. Lomachenko,<sup>1,2</sup> R. Pellegrini,<sup>7</sup> G. Leofanti,<sup>8</sup> E. Groppo<sup>1</sup>

1) Department of Chemistry, Turin University, Italy, 2) Southern Federal University, Rostov-on-Don, Russia, 3) ESRF, Grenoble France, 4) Dipartimento di Fisica e Chimica, Università di Palermo, Italy, 5) Netherlands Organization for Scientific Research, 6) The University of Manchester UK 7) Chimet SpA, Catalyst Division, Vicomaggio, Italy, 8) Consultant, Canegrate (Milano), Italy.

Starting in December 2006 the Turin group have been regular users of the DUBBLE beamlines. In collaboration with the *Chimet* company we have followed aspects of the preparation of industrially relevant Pd catalysts supported on active carbons and on oxidic matrices for selective hydrogenation reactions, till the analysis of spent catalysts extracted from industrial plants. We also performed a methodological study on the particle size distribution combining TEM, STEM, temperature dependent EXAFS and SAXS on Rh/carbon systems and investigated Pd supported on high-surface area, cross-linked polymer systems via standard XAS and with the unique XAS/SAXS combined techniques only available on DUBBLE, see Figure 1.

To optimize our results on low energy edges (Ti and Cr) we were involved in a three way collaboration with beamline staff and Manchester University to implement a feedback system aimed to stabilize beam position and flux along an EXAFS scan. The data thus obtained is of unsurpassed quality. This allowed us to determine the isomorphous substitution of Ti into CHA and STT zeolitic frameworks for selective oxidation reaction in  $H_2O_2$ . We finally performed an ongoing research on polymerization catalysts studying (i) different versions of a  $Cr/SiO_2$  Phillips catalyst, (ii)  $Cp_2Cr$  hosted in different matrices, (iii) Ti-based Ziegler-Natta systems.





**Figure 1** | Combined XAS/SAXS data collected on under *in-situ* temperature programmed reduction in  $H_2$  (top) and CO (bottom) fluxes on Pd catalysts supported on porous polymeric hosting matrices. XANES monitors the  $Pd^{2+} \rightarrow Pd^0$  reduction, while the evolution of the particle size distribution is followed by SAXS. Right parts report the *ex-situ* TEM study. Groppo et al. *J. Phys. Chem. C* 118 (2014) 8406–8415.

Collaborators: DUBBLE staff, OSLO Univ., CPE Lyon, Chimet-, ENI- and Versalis-companies.

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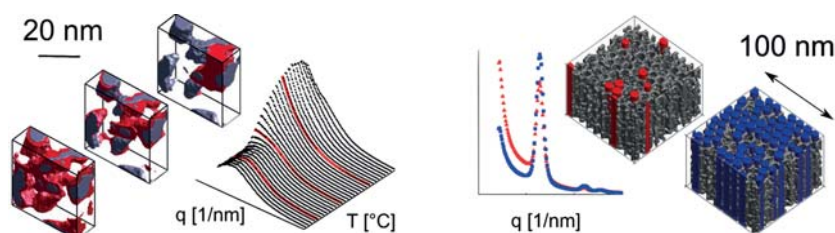
## i03 Small-angle scattering in porous materials: a review highlighting data analysis challenges

**Cedric J. Gommès**

Department of Chemical Engineering, University of Liège, Belgium

Countless technologies and chemical processes make use of nanoporous materials: heterogeneous catalysis, including electrochemical reactions in fuel-cell electrodes, adsorption separation processes, kinetically selective membrane processes, are but a few examples. Nanopores are also relevant to natural processes as diverse as the weathering of rocks and ion transport through biological membranes.[1] Small-angle scattering of x-rays (SAXS) or neutrons (SANS) is one of the few experimental methods currently available for the in situ analysis of phenomena in this type of materials at the mesoscopic scale.[e.g. 2,3]

In this presentation, we briefly review some recent applications of small-angle scattering to the in situ analysis of phenomena inside mesoporous solids. A particular focus is put on the data analysis challenges, whereby the scattered intensity is converted to real-space structures with nanometer resolution.



**Figure 1 |** Examples of porous nanostructures reconstructed from small-angle scattering data. Left: temperature-dependent morphology of confined nitrobenzene [3]; Right: metal loading in copper catalysts supported on silica, displaying mesoscale heterogeneity [4].

[1] O. Coussy, *Mechanics and Physics of Porous Solids*, Wiley, 2010.

[2] P. Huber, *J. Phys.: Condens. Matter* 27 (2015) 103102.

[3] C.J. Gommès, *J. Appl. Cryst.* (2013) 493.

[4] C.J. Gommès, G. Prieto, J. Zecevic, M. Vanhalle, B. Goderis, K.P. de Jong, P.E. de Jongh, *in preparation*.

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## i04 Cool magnets and hot electrons

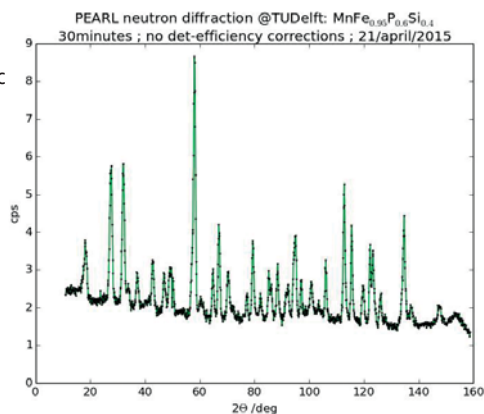
**Ekkas Brück**,<sup>1</sup> Hargen Yibole,<sup>1</sup> Xuefei Miao,<sup>1</sup> Maurits Boeije,<sup>1</sup> N. Van Thang,<sup>1</sup> Niels van Dijk,<sup>1</sup> Lambert van Eijck,<sup>1</sup> Katja Pappas,<sup>1</sup> Wim Bras,<sup>2</sup> Frank de Groot<sup>3</sup> and Rob de Groot<sup>4</sup>

1) Department of Radiation Science & Technology, Delft University of Technology, the Netherlands, 2) European Synchrotron Radiation Facility, DUBBLE CRG, Grenoble, France 3) Debye Institute for Nanomaterials Science, Utrecht University, the Netherlands 4) Institute for Molecules and Materials, Radboud University, Nijmegen, the Netherlands

Magnetic refrigeration is a promising alternative for current compressor based heat pump technology. The dominant advantages of this emerging technology are energy efficiency, no greenhouse gas emissions and less noise. The most advanced magnetocaloric materials display a magnetoelastic phase-transition of first order. To better understand this phase transition in MnFePSi we employ neutron and X-ray studies combined with electronic structure calculations. The crystal structure and position occupancies were determined by neutron powder diffraction, as Mn and Fe and P and Si, respectively display very similar X-ray scattering cross-sections, while the neutron scattering factors are varying significantly. These structural data have been used for the electronic structure calculations. Additionally, neutron diffraction has been used to determine the thermal evolution of the magnetic moments in the ferromagnetic phase. The experimental low-temperature magnetic moments agree well with those determined from first principles.

A salient feature revealed from the electronic structure calculations is the significant reduction of the Fe magnetic moment in the paramagnetic state. This loss of moment comes along with a significant spatial redistribution of electronic density. We employ X-ray magnetic dichroism and X-ray absorption spectroscopy to trace these electronic effects.

Neutron diffraction pattern of MnFePSi recorded on the PEARL diffractometer at RID.





## i05 Femtosecond Optical Control of Magnetism: from fundamentals to nanoscale dynamics revealed by X-rays

**Alexey V. Kimel**

Radboud University, Institute for Molecules and Materials, Nijmegen, the Netherlands

The demand for the ever-increasing speed of information storage and manipulation has triggered an intense search for the fastest possible way to control magnetization in media. Controlling magnetism by light is one of the promising approaches to this problem, as femtosecond laser pulses are among the shortest stimuli in contemporary science [1]. However, realization of the opto-magnetic recording requires answering several fundamental questions. In my lecture I would like to discuss how X-ray spectroscopy has led to a breakthrough in understanding femtosecond optical control of magnetism. In particular, I will show that mechanisms of magnetic switching triggered by femtosecond optical excitation are very counterintuitive [2,3]. Peculiarities of ultrafast optical control of magnetism in magnetic nanostructures will be discussed [4]. It will be also shown that employing plasmonic nano-antennas it should be possible to confine opto-magnetic excitation in a spot well below diffraction limit, making ultrafast all-optical magnetic data recording achievable [5].

- [1] A. Kirilyuk, A.V. Kimel and Th. Rasing, *Rev. Mod. Phys.* **82**, 2731 (2010).
- [2] I. Radu, K. Vahaplar, C. Stamm, T. Kachel, N. Pontius, H. A. Durr, T. A. Ostler, J. Barker, R. F. L. Evans, R. W. Chantrell, A. Tsukamoto, A. Itoh, A. Kirilyuk, Th. Rasing, and A. V. Kimel, *Nature* **472** 205-208 (2011).
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- [4] L. Le Guyader, M. Savoini, S. El Moussaoui, M. Buzzi, A. Tsukamoto, A. Itoh, A. Kirilyuk, T. Rasing, A.V. Kimel and F. Nolting, *Nature Communications* **6**, 5839 (2015).
- [5] T. M. Liu, T. Wang, A. H. Reid, M. Savoini, X. Wu, B. Koene, P. Granitzka, C. Graves, D. Higley, Z. Chen, G. Razinskas, M. Hantschmann, A. Scherz, J. Stöhr, A. Tsukamoto, B. Hecht, A. V. Kimel, A. Kirilyuk, Th. Rasing, H. A. Dürr, *arXiv:1409.1280*.

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## i06 Infrared ion spectroscopy

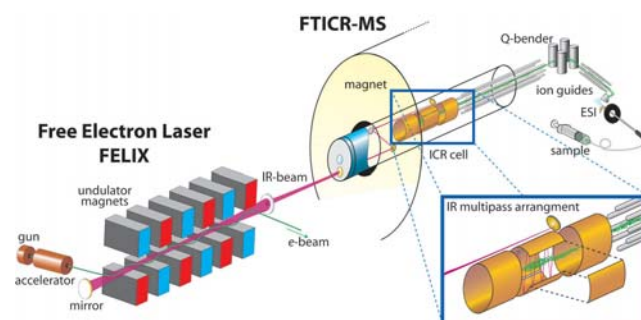
Jos Oomens,<sup>1,2</sup> Britta Redlich<sup>1</sup>

1) Radboud University, Institute for Molecules and Materials, FELIX Laboratory, Nijmegen, the Netherlands,  
2) van 't Hoff Institute for Molecular Sciences, University of Amsterdam, Amsterdam, the Netherlands

As a consequence of Coulombic repulsion, the density of ions in any type of mass spectrometer is extremely low, many orders of magnitude lower for instance than the density of residual air in its vacuum system. Clearly, obtaining an IR spectrum of such a dilute sample presents a substantial challenge. Yet, an IR spectrum could reveal detailed structural information on the molecular ion – isomeric, tautomeric or conformeric – that is simply not contained in the mass spectrum.

Using the continuously tunable IR radiation from the Free Electron Laser for Infrared eXperiments (FELIX), we developed a method for the routine recording of IR spectra of mass selected ions in an ion storage mass spectrometer [1]. Irradiation of the stored ion cloud with the intense pulsed radiation of the FEL induces dissociation of the ions whenever the photon energy matches an infrared transition in the ionic species. Dissociation is readily detected in the mass spectrum, so that an IR spectrum can be reconstructed from a series of mass spectra recorded with the FEL wavelength being tuned over an appropriate range of the ion's IR spectrum.

We will present an overview of applications of this novel methodology relating to structural questions in ion chemistry, such as what are the product ion structures in dissociation reactions involved in peptide sequencing and how do metal ions coordinate to organic ligands?



**Figure 1** | IR beam from the FELIX source is coupled into the ion storage cell of a Fourier Transform mass spectrometer with external electrospray ionization (ESI) source.

## i07 Biological Crystallography with XFELs

**Thomas R.M. Barends**

Department of Biomolecular Mechanisms, Max Planck Institute for Medical Research, Jahnstrasse 29, D-69120 Heidelberg, Germany

X-ray Free Electron Lasers are pushing back the limits of possibility in biological structure determination. By providing extremely bright pulses of femtosecond duration, X-ray FELs have enabled structure determination from micron-sized crystals [1-6] and promise novel studies with unprecedented time resolution. We will discuss recent advances in XFEL-based biological crystallography with an emphasis on radiation damage effects [7-9] and sub-picosecond time-resolved studies.

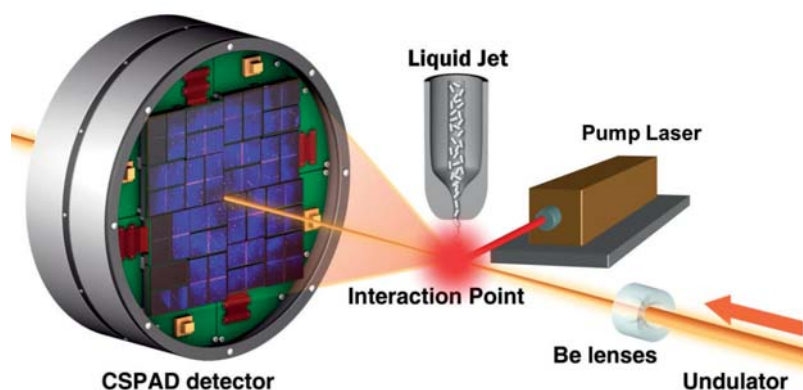


Figure is adapted from [4]

- [1] H.N. Chapman et al., *Nature* 470, 73 (2011).
- [2] L.C. Johansson et al., *Nature Methods* 9, 263 (2012).
- [3] L. Redecke et al., *Science* 339, 227 (2013).
- [4] S. Boutet et al., *Science* 337, 362 (2012).
- [5] W. Liu et al., *Science* 342, 1521 (2013).
- [6] T.R.M. Barends et al., *Nature* 505, 244 (2014).
- [7] L. Lomb et al., *Physical Review B* 84, 214111 (2011).
- [8] A. Barty et al., *Nature Photonics* 6, 35 (2012).
- [9] K. Nass et al., *J. Synchrotr. Rad.* accepted (2015).

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## i08 Neutron scattering studies of the molecular mechanism of the anti-fungal drug, amphotericin B

**M. Jayne Lawrence**,<sup>1</sup> D.J. Barlow,<sup>1</sup> F. Foglia,<sup>1</sup> A.E. Terry, S.E.<sup>2</sup> Rogers,<sup>2</sup> L. Clifton,<sup>2</sup> B. Deme,<sup>3</sup> G. Fragneto<sup>3</sup>

1) Institute of Pharmaceutical Science, King's College London, London, UK, 2) ISIS, Rutherford Appleton Laboratories, Didcot, UK, 3) Institute Laue Langevin, Grenoble, France.

For over 50 years, amphotericin B has been widely employed in treating life-threatening systemic fungal infections. Its usefulness is now becoming compromised by an increasing incidence of pathogen resistance, requiring the development of new anti-fungal agents. However the rational development of new anti-fungal agents requires a detailed knowledge of amphotericin's molecular mechanism. In order to shed light on this problem, a combination of small angle neutron scattering (1), neutron reflectivity (2), and neutron diffraction (3) experiments have been performed to understand amphotericin's interaction and incorporation into model biological membranes. These studies show that amphotericin disturbs the structure of both fungal and mammalian membranes, and co-localises with the membrane sterols in a manner consistent with trans-membrane pore formation. The differences seen in the membrane lipid ordering and in the distributions of the drug-ergosterol and drug-cholesterol complexes within the membranes are consistent with amphotericin's selectivity for fungal vs. human cells.

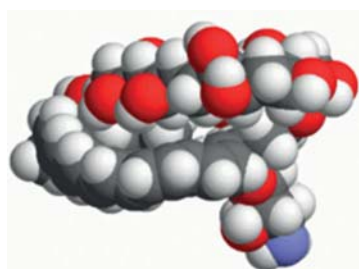


Figure 1 | Amphotericin.

- | 1 | Foglia F et al., (2011) Small-angle neutron scattering studies of the effects of amphotericin B on phospholipid-sterol membrane structure. *Biochim Biophys. Acta*, 1808, 1574-1580.
- | 2 | Foglia F et al., (2014) Interaction of amphotericin B with lipid monolayers. *Langmuir* 30, 9147-9156.
- | 3 | Foglia F et al., (2012) Neutron diffraction studies of the interaction between amphotericin B and lipid-sterol model membranes. *Scientific Reports* 2, Article Number: 778.

## i09 X-ray spectroscopic studies of biological dinitrogen reduction in molybdenum and vanadium nitrogenases

Serena de Beer

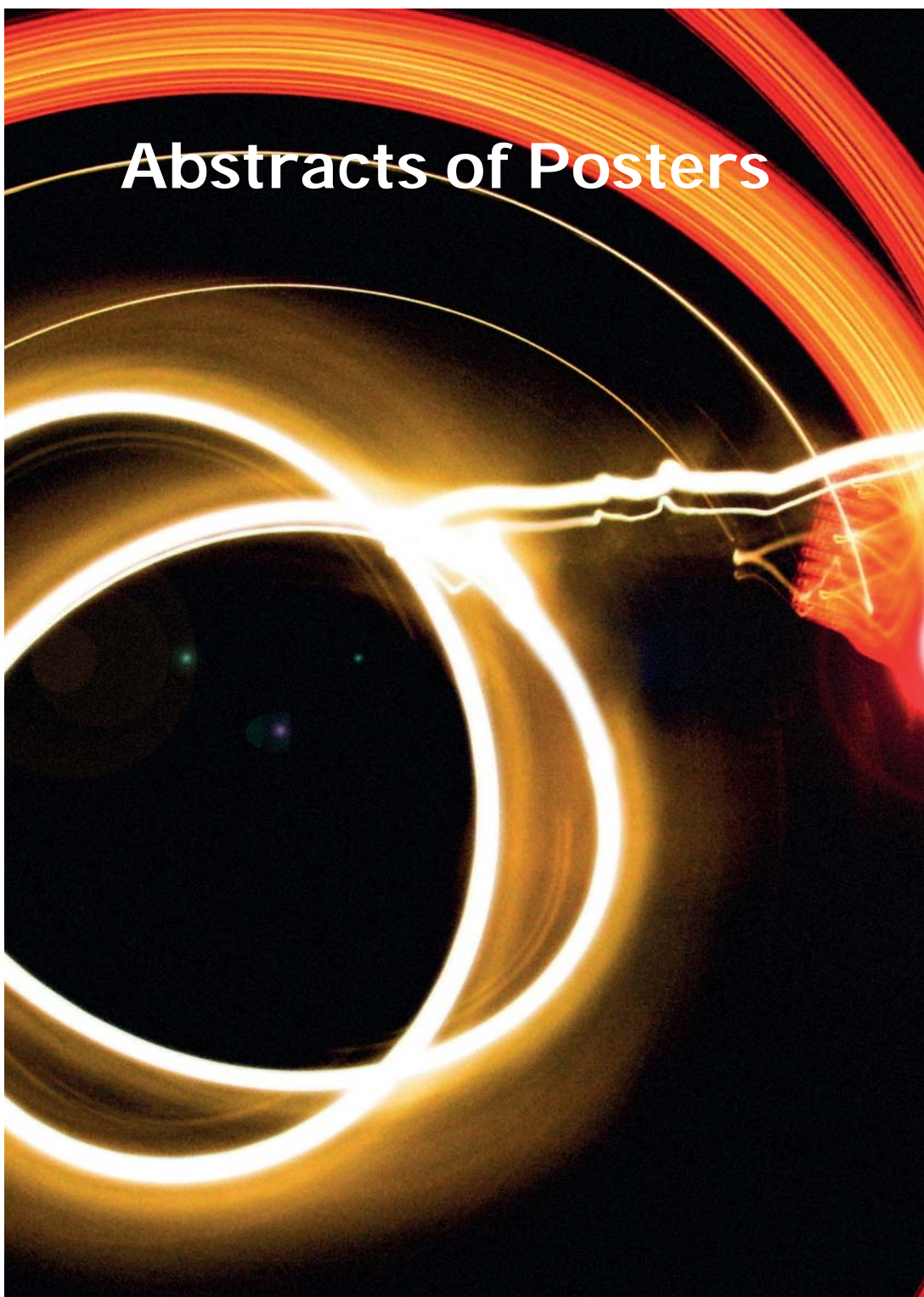
Max Planck Institute for Chemical Energy Conversion, Stiftstr. 34-36, 45470 Mülheim an der Ruhr, Germany

The conversion of dinitrogen to ammonia is a challenging, energy intensive process, which is enabled biologically by the nitrogenase family of enzymes. By far the most studied nitrogenases are the Mo-dependent forms, which contain a MoFe<sub>7</sub>S<sub>9</sub>C cluster, the so-called FeMoco active site, where N<sub>2</sub> to NH<sub>3</sub> conversion occurs. The presence of a central carbon in this cluster was revealed by a combination of valence-to-core X-ray emission spectroscopy (VtC XES), high-resolution crystallography and pulsed EPR methods. However, numerous questions about the electronic structure of this cluster and how it enables such remarkable reactivity remain. Herein, a brief overview of the recent contributions of X-ray spectroscopy to our understanding of biological nitrogen reduction will be given. This will include high-resolution Mo X-ray absorption (XAS) studies, which have revealed the presence of an unusual spin-coupled Mo(III) site in the FeMoco cluster. In addition, Mössbauer and high-resolution Fe XAS studies, which have been used to establish the oxidation state distribution of the iron atoms will be presented. Finally, very recent VtC XES and high-resolution Fe XAS studies of the vanadium-dependent nitrogenases will be presented. While the molybdenum-dependent forms of the enzymes are far better nitrogenases, the vanadium-dependent enzymes have been shown to effectively enable carbon-carbon bond coupling. The differences in the FeMoco and FeVco electronic structures will be discussed, with an emphasis on the potential role of the heterometal in modifying the enzymatic selectivity.

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# Abstracts of Posters



## p01 Simultaneous Synchrotron WAXD and Fast Scanning Chip Calorimetry

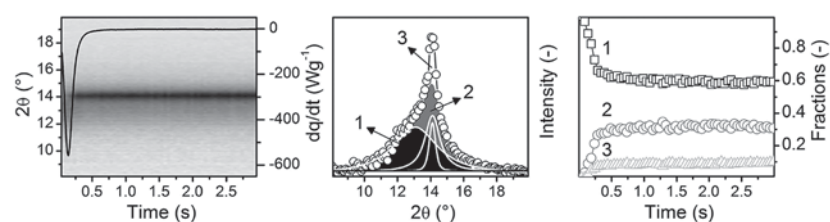
**Dorien Baeten**,<sup>1</sup> Vincent B. F. Mathot,<sup>1</sup> Thijs F. J. Pijpers,<sup>1</sup> Olivier Verkinderen,<sup>1</sup> Giuseppe Portale,<sup>2</sup> Peter Van Puyvelde,<sup>3</sup> Bart Goderis<sup>1</sup>

1) Polymer Chemistry and Materials, KU Leuven, Belgium, 2)DUBBLE CRG BM26@ESRF, Netherlands Organization for Scientific Research, Grenoble, France, 3)Soft Matter, Rheology and Technology, KU Leuven, Belgium

In the last decades, different fast scanning calorimetry (FSC) techniques have been developed, able to operate at scanning rates up to  $40000^{\circ}\text{C s}^{-1}$  and higher [1-2]. A major challenge in the nanocalorimetric study of materials is to relate calorimetric temperature / time dependent events to structural changes of the system. Recently, our lab developed a unique device, combining FSC with *in-situ* synchrotron Wide Angle X-ray Diffraction (WAXD) [3]. In this contribution, the melting and crystallization of high-density polyethylene at scanning rates up to  $200^{\circ}\text{C s}^{-1}$  is shown to illustrate the capabilities of the combined approach and to address some FSC instrumental issues (thermal lag and baseline curvature). Furthermore the isothermal crystallization of polyamide 11 (PA11) at very high supercooling is discussed, thereby shedding new light on its supercooling dependent polymorphism. At high supercoolings ( $70^{\circ}\text{C}$ ), only  $\delta'$  mesophase is formed while at intermediate supercoolings ( $100$  and  $130^{\circ}\text{C}$ ) both  $\delta'$  mesophase and  $\delta$ -crystals are observed in which the mesophase/crystalline ratio increases with increasing supercooling. In Figure 1 FSC and WAXD data are shown for the isothermal crystallization at  $100^{\circ}\text{C}$ . In general, by simultaneously recording FSC and synchrotron WAXD data, high quality calorimetric and structural information on sub  $\mu\text{g}$  samples at very short time scales under well controlled thermal conditions is obtained.







**Figure 1** | Left: PA11 WAXD and FSC data during isothermal crystallization at 100°C; Middle: WAXD pattern after 3 s decomposed into its amorphous (1), mesophase (2) and crystalline (3) fraction; Right: fractions as a function of crystallization time (same numbers).

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## po2 Overview of the DUBBLE project

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The Dutch-Belgian Beamline (DUBBLE) project operates two beam lines at the European Synchrotron Radiation Facility (ESRF). 70% of the beamtime is available for Dutch and Flemish users and is thus part of the national research infrastructure. Besides this, 30% of the beam time is available for general ESRF users, which has a side benefit that there are often experimental cross fertilizations between Dutch/Flemish groups and top European research groups. BM26-A is dedicated to time-resolved X-ray absorption spectroscopy (XAS) and BM26-B dedicated to combined small angle X-ray scattering (SAXS) and wide angle X-ray scattering (WAXS). These lines operate simultaneously and cater to a diverse user community. Due to the fact that infrastructural developments have been focussed upon the special requirements of the Dutch-Flemish user community DUBBLE is now a highly productive platform which offers this community a set of unique experimental opportunities. These range from industrial polymer processing equipment to catalysis under operando conditions as well as fundamental physics questions in high magnetic fields or other extreme conditions. The first experiments were performed in 1997 and output has risen continuously, from ten peer-reviewed publications in the year 2000 to over one hundred papers and PhD thesis in the last number of years. The average impact factors of the journals in which publications appear has also constantly increased with many papers appearing in for instance Physical Review Letters, JACS and Angewandte Chemie.

## po3 Loading and Unloading Kinetics of Hydrogen in Magnesium based thin films

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Hydrogen storage in metal hydrides have recently been studied as a possible way to store energy. One of the possible options is magnesium, which is abundant and cheap and it has a hydrogen storage capacity of 7.6 wt%, three times more than conventional high pressure cells. However, due to its high hydrogen desorption temperature of around 600 K, high thermodynamic stability ( $\Delta H = -75 \text{ kJ mol}^{-1}$ ) and slow kinetics, its ability to release the hydrogen is limited. To overcome these problems, alternative approaches have been developed that include the use of nanostructures or thin film systems.



**Figure 1 |** Four layered system as studied in this research.

A four layered system as depicted in Figure 1 is used to study the (un)loading kinetics of magnesium. The Pd layer functions as a catalyst in the hydrogen dissociation reaction and prevents the Mg layer from oxidising, whereas the Ti layer prevents the Mg layer from mixing with the Pd layer. Previous research using hydronography showed that a nucleation and growth mechanism during hydrogen loading governs the formation of insulating  $\beta$ -Mg phase in the metallic  $\alpha$ -Mg phase [1,2].



In this research, we employ a combination of neutron reflectivity, hydronography and Density Functional Theory. Hydronography is used to distinguish between the metallic  $\alpha$ -Mg phase and insulating  $\beta$ -Mg phase. Neutron Reflectivity measurements were performed at the ROG Reflectometer of the Reactor Institute Delft and at ISIS' OFFSPEC. Preliminary results show that the Ti-layers are loaded first and rapidly, before loading of the Mg-layers occurs. Furthermore, substantial changes in reflectivity upon loading and unloading are observed after full conversion of the hydronography signals suggesting that the insulating  $\beta$ -Mg phase and the metallic  $\alpha$ -Mg can contain various degrees of hydrogen loading. This is in accordance with the DFT calculations.

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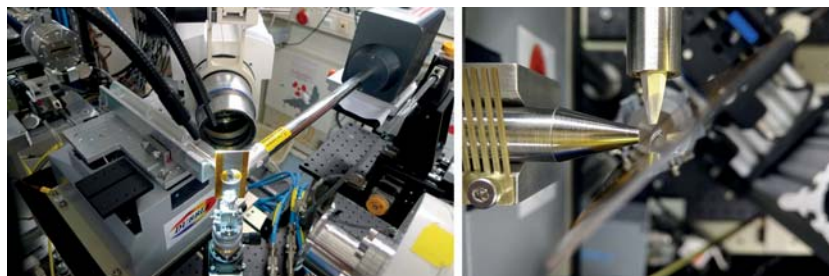
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## po4 Three-Dimensional Microfocused Confocal X-ray Absorption Spectroscopy: a DUBBLE polycapillary XANES/EXAFS setup

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The ESRF-CRG BM26 DUBBLE beamline has had a long-standing collaboration with the Ghent University XMI group regarding micro-focus spatially resolved confocal XRF and XANES/EXAFS using polycapillary optics.[1] In an effort to make these methods available to a larger user base we have developed a plug-and-play style setup, resulting in a macro to micro-focus conversion of the beamline in a matter of only a few hours.



**Figure 1 |** Overview of the polycapillary-based confocal setup at the ESRF-CRG BM26 DUBBLE beamline. A burnt piece of papyrus (left) and fish scale (right)

Together with the easy convertibility to a sub-micron surface-sensitive grazing incidence/exit setup, a wide range of extra research possibilities is now available for users at DUBBLE, some of which will be presented on the poster such as depth-resolved examinations of ion-exchange glass, toxicological studies on biological samples, cultural heritage preservation, etc.

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## po5 Reaction of sulfide with lepidocrocite studied by online X-ray absorption spectroscopy

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Sulfidization of iron oxides is an important process in environments which are characterized by spatial redox gradients or temporally fluctuating redox conditions. For example: microbial sulphate reduction in deeper parts of aqueous sediments can lead to the formation of dissolved S(-II) which can diffuse upwards into sediment layers which contain iron oxides. The reaction of S(-II) with iron oxides is relevant as it prevents the emission of toxic H<sub>2</sub>S into the overlying water column and because the reaction of S(-II) with iron oxides is typically accompanied by the release of previously bound nutrients or trace elements.

Due to its environmental importance, the reaction between S(-II) and iron oxides has been extensively investigated in view of its mechanism, kinetics, and the inherent solid phase transformations [1,2]. Recent findings suggest that the reaction of lepidocrocite ( $\gamma$ -FeOOH) with S(-II) at circumneutral pH leads to the formation of metastable intermediates which, in turn, control the formation of stable end products such as mackinawite (FeS) or pyrite (FeS<sub>2</sub>) [3]. Here, we performed on-line X-ray absorption spectroscopy at the K-edge to monitor the change in Fe redox state and coordination environment during the reaction of lepidocrocite with S(-II).

The progress of the reaction was reflected in the change of XANES spectra.

Component analysis revealed that four components are required to reproduce all the spectra. Those components can be assigned to lepidocrocite, dissolved Fe<sup>2+</sup>, mackinawite and an unknown reaction product. Analysis of EXAFS spectra, collected after acid consumption has ceased, indicates that Fe in the unknown product remains coordinated to oxygen even when S(-II) is present in excess. Furthermore, Fe-Fe distances are longer than in mackinawite and resemble those in Fe<sub>2</sub>S<sub>2</sub> clusters. The





unknown product might reflect the reactive intermediate, which was proposed in previous studies. This intermediate might consist of parts of the lepidocrocite structure in which Fe centres remain connected by Fe-O-Fe bonds and which are interconnected by sulphide bridges.

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## p06 Electric field polarity dependent modification of the metal/ferro-electric oxide interface chemistry

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Multiferroic materials with magnetoelectric (ME) coupling are rare in nature and the coupling is rather weak, therefore we investigate an alternative approach by coupling the ferromagnetic and the ferroelectric phases via an interface in a multilayer structure. Our aim is to study in detail the magnetic spin structure at the interface. Based on the longitudinal magneto-optic Kerr effect (MOKE), we observed the electric-field modulation of the magnetic properties of Fe or  $\text{Co}_{50}\text{Fe}_{50}$  films on barium titanate (BTO) and lithium niobate (LNO) substrates. A change in the coercive field ( $H_c$ ) of the ferromagnetic layer was observed as a function of the electric field applied across the thickness of the piezoelectric substrate. These changes were irreversible which can possibly be due to electric field induced structural changes at the interface, causing the interfacial atoms to become non-magnetic (magnetically dead layer). The ME coupling in such systems is via interface due to which a detailed understanding of the chemistry and the structure of the interface is necessary. Hence, we study the evolution of the metal/oxide interface of Fe/BTO and Fe/LNO system with the applied electric field using Mössbauer spectroscopy and Nuclear Resonant scattering of the synchrotron radiation on the samples with 1 nm  $^{57}\text{Fe}$  probe layer at the interface. The use of two complementary characterization techniques (MOKE and NRS) is a beneficial combination for the investigation of the ME coupling since they provide both a macroscopic and microscopic probe of magnetism. We proposed a model for the electric field- induced oxidation of ferromagnetic/ferroelectric interface based on MOKE and NRS results. Mössbauer spectroscopy results confirmed the hypothesis and showed a systematic trend of evolution of the interface with the electric field depending on the direction of the field [1].

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## po7 Molecular nitrogen in N-doped TiO<sub>2</sub> Nanoribbons: Nanoscale NEXAFS

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Nitrogen doping titanium dioxide (TiO<sub>2</sub>), with N at substitutional sites has been reported to be indispensable for enhancing the use of TiO<sub>2</sub> as a visible-light photocatalytic material. The main effect of the N-doping is the narrowing of the energy band gap of TiO<sub>2</sub> due to the mixing of N 2p and O 2p states. Additionally, an isolated narrow band responsible for the visible light photoactivity is formed above the valence band. Nitrogen doping into the TiO<sub>2</sub> lattice is rather challenging as N atoms must be accommodated geometrically and electronically. To address the fundamental issues we explored a chemical route and report here a comprehensive investigation of N-doped TiO<sub>2</sub> nanoribbons. As a precursor material for N-doped TiO<sub>2</sub> nanoribbons we used hydrogen titanate nanoribbons (HTiNRs) because upon heating HTiNRs easily transform first to the monoclinic TiO<sub>2</sub> β-phase (TiO<sub>2</sub>-B) and then to anatase (tetragonal phase) while retaining the morphology of the parent nanostructure intact.

The nitrogen doping of TiO<sub>2</sub> nanoribbons during the thermal transformation of hydrogen titanate nanoribbons (HTiNRs) between 400 and 650 °C in a dynamic ammonia atmosphere was investigated using X-ray photoelectron spectroscopy (XPS), transmission X-ray microscopy combined with near-edge X-ray absorption fine structure spectroscopy (NEXAFS-TXM), X-ray diffraction (XRD) and electron paramagnetic resonance measurements (EPR). Comprehensive structural characterizations have revealed that for a calcination temperature of 400°C, the HTiNRs transform into pure monoclinic TiO<sub>2</sub>-B whereas at higher calcination temperatures (580 and 650°C) a mixture of TiO<sub>2</sub>-B and anatase is obtained. XPS and EPR results clearly reveal the nitrogen doping of TiO<sub>2</sub> nanoribbons and that, depending on the calcination temperature, nitrogen atoms occupy interstitial and substitutional sites. Moreover, in samples calcined at 580 and 650°C the presence of N<sub>2</sub>-like species in the HTiNRs was detected by NEXAFS-TXM. These species are trapped in the HTiNRs structure. EPR measurements upon light illumination have disclosed the generation of photoexcited states which implies that nitrogen has an important effect on the electronic structure of N-doped TiO<sub>2</sub>.

## po8 Coordination and Catalysis of $\text{Zn}^{2+}$ in Epoxy-Based Vitrimers

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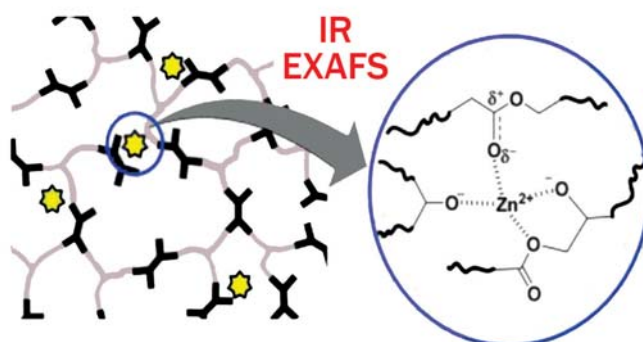
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Vitrimers [1,2,3] are networks in which the topology can be changed while keeping constant the number of bonds and crosslinks. This unique feature lies in their chemical structure, characterized by the presence of exchangeable bonds. The prototype of an exchange reaction is transesterification, which follows an associative mechanism in basic conditions.

To elucidate the catalytic action of  $\text{Zn}(\text{II})$  in the exchange reactions taking place in epoxy-acid vitrimers, extended X-ray absorption fine structure (EXAFS) spectroscopy has been used as well as infrared (IR) spectroscopy on vitrimers and model systems. The catalytic action of  $\text{Zn}(\text{II})$  in vitrimers is characterized by a double activation, nucleophilic activation of hydroxide groups into more nucleophilic alkoxide groups and electrophilic activation of the ester carbonyl bond. EXAFS analysis shows that the zinc cation is coordinated by four oxygen atoms, two of them being negatively charged, i.e. alkoxide groups to satisfy the electroneutrality whereas IR analysis demonstrates the lengthening of the ester carbonyl bond. The main roles of  $\text{Zn}^{2+}$  can be stated as follows: 1) serving as counterion for alkoxide groups; 2) activating the carbonyl bonds; 3) bringing reacting centres close to each others.  $\text{Zn}^{2+}$  coordinated in the epoxy-based network is therefore ideally suited to catalyze exchange reactions by transesterification and to bring vitrimer properties.

We gratefully acknowledge helpful discussions with J. G. P. Goossens, from Eindhoven University of Technology. We acknowledge ESRF (European Synchrotron Radiation Facility) for provision of X-Ray beam time and thank Dr. D. Banerjee for his help on Dutch-Belgian Beamline (DUBBLE).





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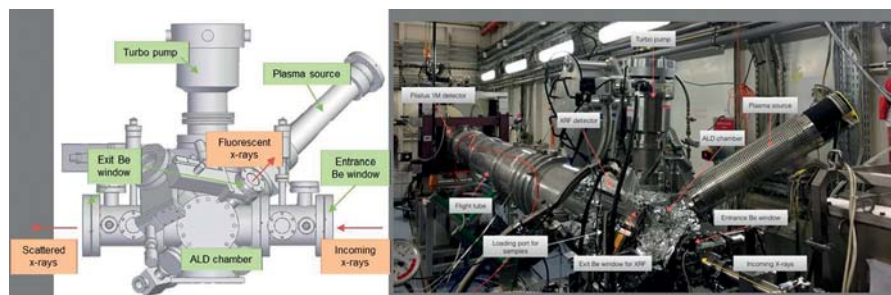


## p9 *In-situ* GISAXS Study of the Initial Island Growth During Atomic Layer Deposition of Platinum

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Atomic Layer Deposition (ALD) processes for metals are often characterized by a nucleation controlled growth mode. Islands are initially formed that coalesce in a continuous film after a sufficient number of ALD cycles. This work demonstrates the unique suitability of synchrotron-based grazing incidence small angle x-ray scattering (GISAXS) for in situ monitoring of the nanoscale surface morphology during ALD of Pt. GISAXS allows to quantify the evolution in size, shape and spacing of the Pt nuclei. To enable GISAXS measurements during ALD, we developed a dedicated ALD setup that can be installed at the end station of the DUBBLE beamline at the ESRF (Figure 1).



**Figure 1** | Dedicated ALD setup for in situ GISAXS measurements installed at the DUBBLE beamline at the ESRF.

Here, we have studied the influence of the reactant type ( $O_2$  gas vs.  $N_2$  plasma) on the nucleation of Pt ALD using the  $MeCpPtMe_3$  precursor. GISAXS indicated that, for the  $O_2$ - process, the spacing between the particles increases with progressing





Pt deposition, suggesting that atom and cluster surface diffusion phenomena play a major role during particle nucleation and coalescence. On the other hand, for the  $N_2$  plasma process, the average particle distance increases during the initial nucleation phase, but remains constant during the particle growth process. The particle dimension analysis revealed that the  $N_2$  plasma process results in elongated particles, while the  $O_2$ -process gives rise to flattened particles. Based on this analysis, it is concluded that  $O_2$  induces atom and cluster surface diffusion and promotes the ripening of the Pt nanoparticles, while diffusion phenomena seem to be suppressed during  $N_2$  plasma based ALD leading to smoother Pt coatings.

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## p10 Nature of deactivation for $\text{Fe}_2\text{O}_3\text{-CeO}_2$ materials in $\text{CO}_2$ utilization processes

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A series of  $\text{Fe}_2\text{O}_3\text{-CeO}_2$  materials were prepared to investigate the types of deactivation during repeated  $\text{H}_2$ -reduction and  $\text{CO}_2$ -reoxidation. The effect of cycling was investigated in Ce rich (10wt% $\text{Fe}_2\text{O}_3\text{-CeO}_2$ ) and Fe rich (80wt%  $\text{Fe}_2\text{O}_3\text{-CeO}_2$ ) materials. Sintering, phase transformation and extraction of Fe are the forms of deactivation observed. The crystallographic changes during reaction leading to sintering and phase transformation were identified using in situ XRD. In addition to the increase in particle size due to sintering, the repeated cycling at 650°C lead to solid-solid transformation of oxide structures to  $\text{CeFeO}_3$ , resulting in an overall lower oxygen storage capacity. The  $\text{CeFeO}_3$  phase remained inert during reduction and reoxidation. Further, TEM and XAS were used to investigate the local structure around Ce and Fe before and after reaction. By modelling the XAS Ce-K signal in the 10wt% $\text{Fe}_2\text{O}_3\text{-CeO}_2$  sample, the amount of Fe in  $\text{CeO}_2$  was determined as 21% of total iron oxide. The local structure analysis around Ce-L<sub>III</sub> and Fe-K edges revealed the location of the nearest neighbours. TEM showed nanoparticles of  $\text{CeO}_2$  with iron both incorporated and at the surface of 10wt% $\text{Fe}_2\text{O}_3\text{-CeO}_2$ . In the Fe rich material (80wt% $\text{Fe}_2\text{O}_3\text{-CeO}_2$ ), 5 to 10 nm  $\text{CeO}_2$  nanoparticles show incorporated Fe, while decorating the 100 nm iron oxide clusters.

After 100 redox cycles, TEM images of 10wt% $\text{Fe}_2\text{O}_3\text{-CeO}_2$  indicated the appearance of iron oxide nanoclusters at ceria grain boundaries. These clusters originated from the strong sintering of the surface iron oxide particles. The increase in the lattice parameter calculated from XRD could indicate that an amount of Fe was extracted from the  $\text{CeO}_2$  lattice.

**Keywords:** Deactivation, Sintering, phase transformation, in situ XRD, XAS modelling.



## p11 A new neutron powder diffractometer PEARL at the TU Delft

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Reactor based constant-wavelength neutron powder diffractometers follow the same generic design since the 1950's. However, according to L. Cussen [1] different options exist that may lead to higher performance. The diffractometer PEARL at the 2MW reactor of the TU Delft has now been realised along these ideas. The instrument, that entered commissioning in 2015, has a fixed monochromator take-off angle and operates with 3 wavelengths (1.3, 1.7, 2.5 Å) covering d-spacings from 0.7 to 7 Å with  $\delta d/d \geq 2 \times 10^{-3}$  without the use of any collimators. The neutrons are detected by a scintillator banana detector, that covers 148° with 2 mm resolution and is based on a technology developed at ISIS, UK. The detector combines high efficiency for thermal neutrons with gamma discrimination sufficient for the use at a steady-state neutron source [2]. The design of the instrument and the first experimental results will be presented that illustrate the performance of the concept: a rather competitive instrument is now in operation at the TU Delft.

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## p12 Extended X-ray Absorption Fine Structure Investigation of Sn Local Environment in Strained and Relaxed Epitaxial $\text{Ge}_{1-x}\text{Sn}_x$ Films

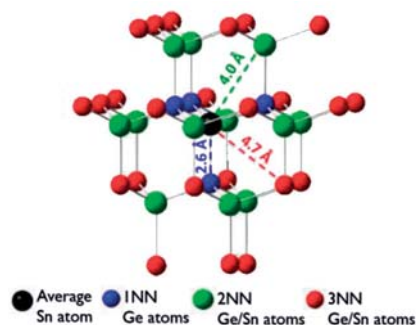
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We present an extended X-ray absorption fine structure investigation of the local environment of Sn atoms in strained and relaxed  $\text{Ge}_{1-x}\text{Sn}_x$  layers with different compositions. We show that the preferred configuration for the incorporation of Sn atoms in these  $\text{Ge}_{1-x}\text{Sn}_x$  layers is that of a  $\alpha$ -Sn defect, with each Sn atom covalently bonded to four Ge atoms in a classic tetrahedral configuration. Sn interstitials, Sn-split vacancy complexes, or Sn dimers, if present at all, are not expected to involve more than 2.5% of the total Sn atoms. Within the accuracy of the measurements, the degree of strain relaxation of the  $\text{Ge}_{1-x}\text{Sn}_x$  layers is not reflected on the local atomic surrounding of the Sn atoms. The calculated topological rigidity parameter  $a^{**}=0.69\pm0.29$  indicates that the strain due to alloying in  $\text{Ge}_{1-x}\text{Sn}_x$  is accommodated via bond stretching and bond bending, with a slight predominance of the latter, in agreement with ab initio calculations reported in literature. Finally, a comparison between the  $\text{Ge}_{1-x}\text{Sn}_x$  lattice constants measured by X-ray diffraction and the ones estimated from these EXAFS measurements suggests that the Ge-Ge, the Ge-Sn and the Sn-Sn bond lengths exhibit a similar composition dependence.

**Figure 1** | Typical model of the probed local structure around an average Sn atom.

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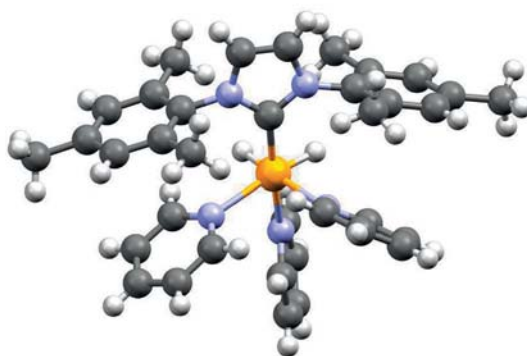


## p13 EXAFS and DFT Studies on Ir Catalysts for SABRE

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The intrinsic insensitivity of Nuclear Magnetic Resonance (NMR) can be over-come by hyperpolarization techniques to produce non-Boltzmann spin-state distributions. In Signal Amplification By Reversible Exchange (SABRE) [1] this is achieved by the temporary association of parahydrogen and a substrate such as pyridine (py) in the coordination sphere of a transition metal (Figure 1). Polarization is transferred from the parahydrogen-derived hydride ligands to the bound substrate via scalar coupling, followed by dissociation of the hyperpolarized substrate into the bulk solution.



**Figure 1** | SABRE catalyst based on an iridium *N*-heterocyclic carbene complex, [IMesIr(py)<sub>3</sub>H<sub>2</sub>]<sup>+</sup>. Yellow, Ir; grey, C; blue, N; white, H.

At substrate concentrations lower than that of the catalyst, it is necessary to add a co-substrate such as methyltriazole (mTz) to reduce the interference of solvent molecules with SABRE [2]. Several mixed Ir complexes can be considered to exist,





which are not all observed by NMR. Density Functional Theory (DFT) calculations were performed on these complexes to better understand this phenomenon. Extended X-ray Absorption Fine Structure (EXAFS) studies in organic solutions provide interesting complementary information on the complexes involved [*Chem. Eur. J.*, accepted].

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## p14 Reactive Self Assembly of PbSe Nano-crystals into Two-Dimensional Super-lattices Studied *in-situ* with GISAXS/WAXS

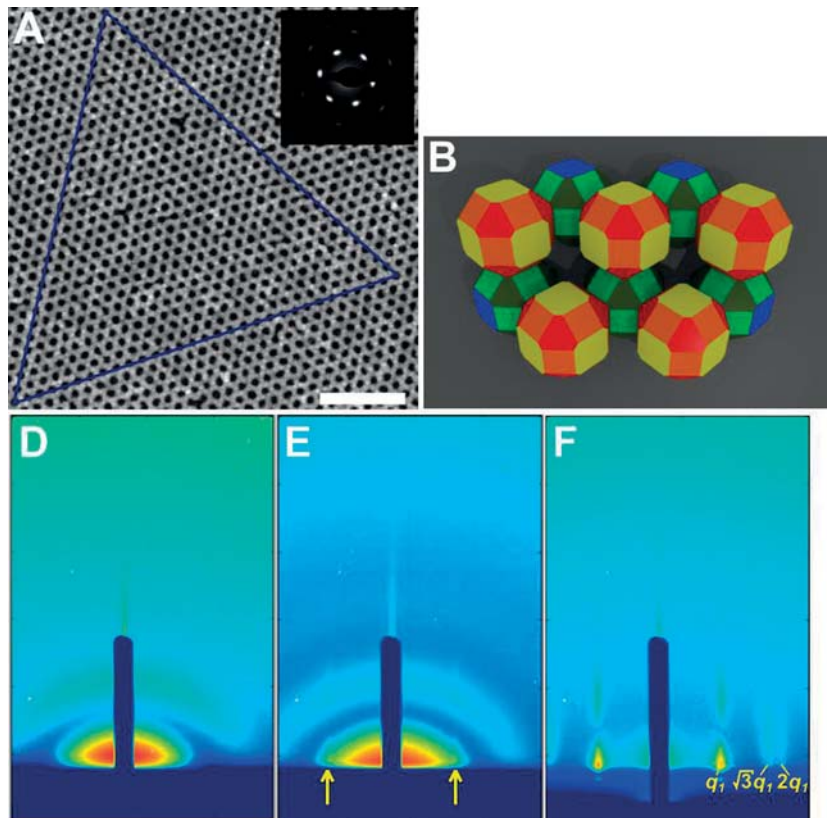
**J.J. Geuchies**,<sup>1</sup> C. van Overbeek,<sup>1</sup> J.L. Peters,<sup>1</sup> F.T. Rabouw,<sup>1</sup> J. Hilhorst,<sup>2</sup> O. Konovalov,<sup>2</sup> A.V. Petukhov<sup>1</sup> and D. Vanmaekelbergh<sup>1</sup>

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Two-dimensional (2D) semiconductors with nanoscale periodicity are a subject of great interest in modern solid state physics. We have recently developed a bottom-up method that can produce such materials with much finer nanoscale features than possible even with the most advanced lithography techniques [1]: PbSe nanocrystals (NCs) can self-assemble at a liquid/air interface and fuse into various superlattices (SLs) through oriented attachment. The final structures possess atomic coherence between atomic lattices of neighboring NCs. The geometries can be linear or branched wires of NCs, or 2D sheets of square symmetry. Most importantly, by choosing the right experimental parameters the result is a graphene-like “honeycomb” structure. Its electronic properties are predicted to be exotic as in graphene, but controllable as with semiconductors [2,3].

Here we study the mechanism of formation of these SLs *in-situ* by using Grazing Incidence Small-and-Wide-Angle X-ray Scattering (GIS&WAXS). In this way, we are able to follow the temporal evolution of SL formation not only on the nanocrystal scale, but also the atomic scale. It is found that (1) the NCs first self-assemble into a close-packed colloidal structure at the liquid-air interface, after which (2) there is a lattice contraction where the particles are stripped of their ligands and approach each other [4].





**Figure 1** | (A) TEM image of the honeycomb SL, scalebar 50 nm. Inset shows the corresponding electron diffraction pattern indicating the atomic alignment of the NCs in the superlattice. (B) Block model of the honeycomb superlattice. (D),(E),(F) GISAXS patterns with increasing time, showing the self-assembly of the PbSe building blocks. Eventually a hexagonal structure is formed at the liquid-air interface.

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## p15 Dynamic Exfoliation and Restacking of colloidal 2D materials from Layered Titanates

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3D Layered metal oxides like  $K_{0.8}[Ti_{1.73}Li_{0.27}O_4]$  can be exfoliated into  $Ti_{1.73}O_4$  'nano-sheets', the 2D oxide equivalents of graphene. The 3D parent oxide crystals consist of a stack of negatively charged anionic layers which are separated from each other in water via cationic interlayers (e.g.  $K^+$ ).

In the first step of an exfoliation process, the counter-ions in the 3D crystal are replaced by protons via ion exchange. In the second step, the interlayer protons  $H^+$  are replaced by bulky organic ions, leading to either an exfoliated state of colloidal 2D nanosheets in solution, or to a hybrid state in which the cationic interlayers are replaced by bulky organic cations. In this study, several quaternary ammonium ions and aminoacids were chosen as organic cations. These ions are part of the same chemical family, and were used to investigate the influence of parameters such as charge, molar mass or size of the molecules in the intercalation of layered titanates. The mechanisms of intercalation, exfoliation and re-stacking of this layered titanate system were studied by *in-situ* time-resolved SAXS experiments in the Dutch-Belgian Beam Line (DUBBLE) at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France. The intercalation of bulky quaternary ammonium ions was generally believed to consist of a direct intercalation and formation of a swollen layered phase. However, our experiments proved that the process consists of a very fast two-step reaction, in which the quaternary ammonium ions exfoliate the parent crystal within seconds, after which the system re-stacks, forming a swollen phase. The entire process takes place on a time scale of seconds to minutes.

In the case of long aminoacid chains, it was seen that the amino group reacts with the protons in the interlayer, leading directly to a stable swollen phase within several minutes. The final result is a well-crystallized hybrid organic-inorganic composite. These colloidal systems are good examples of new 2D materials that are soft and ductile, materials that can withstand substantial deformation (i.e. beyond the ~0.5% upper limit for normal ceramics) without -brittle- fracture.

## p16 Shining light on hydrotreating catalyst activation

Lennart van Haandel,<sup>1</sup> Emiel J.M. Hensen<sup>2</sup> and Thomas Weber<sup>1</sup>

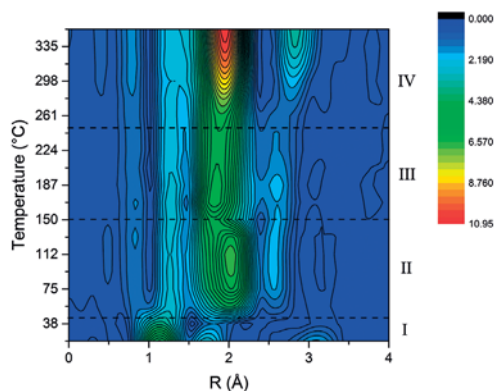
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Two-dimensional transition metal sulfides have been applied in oil-refining for many years to catalytically remove sulfur from oil (HDS). In HDS catalysts, the electronic properties of MoS<sub>2</sub> are tuned by the addition of Co/Ni, improving the intrinsic activity by about one order of magnitude. Studies on model systems, often under reaction conditions deviating substantially from those in industrial practice, have led to the Co-Mo-S model with Co/Ni atoms decorating the edges of MoS<sub>2</sub> nanosheets [1]. A drawback of these model studies is that trends in model HDS reactions using thiophene or dibenzothiophene usually do not match with trends in gas-oil HDS. Consequently, structure-performance relations at the lab scale are not very well suited to predict performance under real HDS conditions (20-60 bar, 350-400°C, gas-oil feed). We developed an X-ray absorption spectroscopic (XAS) cell to monitor the active phase genesis of Co-Mo catalysts under *in-situ* conditions close to industrial practice (20 bar, 350°C). In this way, we demonstrate that the temperature of the oxide-to-sulfide phase transition strongly depends on activation parameters such as pressure and sulfiding agent and that crystallization of MoS<sub>2</sub> proceeds via several (oxy)sulfidic Mo species (Figure 1).

**Figure 1** | Temperature-programmed sulfidation of a Co-Mo catalyst in H<sub>2</sub>S(10%)/H<sub>2</sub> at 20 bar pressure, monitored by time-resolved EXAFS at the Mo K edge.

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## p17 Science on BM26-B at ESRF

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The SAXS/WAXS beamline of DUBBLE, BM-26B, at the European Synchrotron Radiation Facility (ESRF) offers the opportunity to perform extensive studies of a broad range of materials and processes [1,2] over a large size scale range (0.9-250 nm, real space) and time domain (msec time resolution feasible). The beam line is unique in its possibilities to implement complex, and often, large sample handling environments. Due to the availability of state of the art detectors (high count rates and time resolution) it is possible to offer a broad range of experimental technique combinations.

Also the beamsize can be adapted to the requirements of the experimenter from 10 microns to 300 microns by using different post focusing optical systems specifically developed to retain a good low angle resolution. A Kirkpatrick-Baez system (KB mirrors) enables to perform simultaneous SAXS-WAXS experiments with a beamsize of 10x10 microns. Furthermore, compound refractive lenses allows to perform SAXS experiments with a microbeams (50 x 20 micron) with extremely low divergence. Furthermore, microradian scale information can be obtained by a post-focusing system with compound reflective lenses.

The versatility of the beamline is manifest in the many different sample environments that can be mounted on the beamline. This ranges from industrially relevant polymer extruders and film blowers to 30 Tesla pulsed magnets for fundamental research in the physics of materials under extreme conditions.

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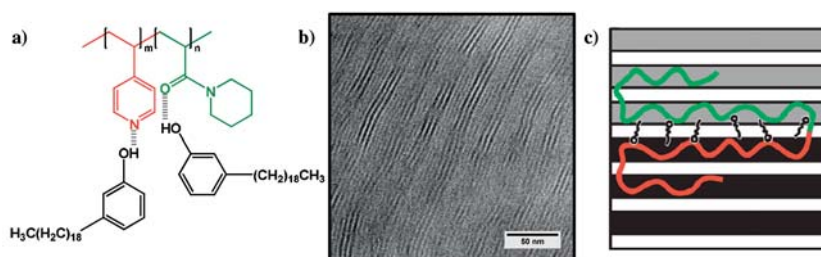


## p18 Hierarchical Self-assembly of Supramolecular Double-comb Diblock Copolymers

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For entropic reasons in particular, diblock copolymers are known to form highly ordered mesomorphic structures (10 to 200 nm) spontaneously. Besides being interesting for their application in membrane technology, compatibilizers, electronic devices and catalysis, block copolymers are also very interesting from a fundamental point of view, since more complex morphologies can be found in more complicated polymeric architectures (e.g. triblock terpolymers, star polymers and comb-shaped copolymers). A very subtle route for avoiding the synthesis of such complicated materials is supramolecular chemistry. For instance, comb-shaped copolymers can be obtained in blends of hydrogen bond donating alkylphenol-based surfactants and a hydrogen bond accepting homopolymer [1], while more complex hierarchical structure formation can be achieved by applying this procedure in block copolymer mixtures.



**Figure 1** | Self-assembly of a [P4VP-**b**-PAPI](3-NDP)<sub>0.5</sub> double-comb diblock copolymer (a) resulted in a unique double parallel lamellar-in-lamellar morphology: TEM (b), schematic (c).



Recently this type of self-assembly was brought to a new level by combining 3-nonadecylphenol (3-NDP) surfactants with a double hydrogen bond accepting diblock copolymer (poly(4-vinylpyridine)-*block*-poly(N-acryloylpiperidine), P4VP-*b*-PAPI, Figure 1a) [2]. Several unique hierarchical morphologies were identified in these supramolecular complexes by using a single P4VP-*b*-PAPI diblock copolymer ( $57 \text{ kg}\cdot\text{mol}^{-1}$ ,  $f_{\text{P4VP}} = 0.47$ ), which had not been observed in block copolymer-based materials before (Figure 1b and 1c). Furthermore, the observed phase behavior as a function of comb density was found to be in excellent agreement with our previously performed theoretical analysis [3].

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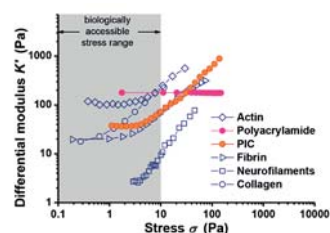
## p19 Mechanics and Structure of Strain Stiffening Hydrogels

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Biopolymers can be found throughout the body, both in the intracellular cytoskeleton, such as actin and intermediate filaments, and in the extracellular matrix, such as collagen and fibrin. These biopolymers have in common that they assemble into stiff bundles or fibers, which form a porous network. This structure gives these biopolymer networks unique mechanical properties, known as *strain stiffening*. Their stiffness increases when they are deformed. In Nijmegen, we developed a thermoresponsive hydrogel based on synthetic polyisocyanides (PICs), which forms a gel upon heating and becomes liquid upon cooling down. [1] This synthetic hydrogel uniquely mimics the strain stiffening response of biopolymer gels (figure 1) and its stiffness can be tuned over several orders of magnitude. [2]

**Figure 1** | Stiffness as a function of applied stress for biopolymer gels and for synthetic polyacrylamide and PIC gels.



We use rheology to study the mechanics of the PIC hydrogels, and small angle X-ray scattering (SAXS) to study the structure of these gels. We found that both the mechanics and the structure of our gel can be tuned over a broad range using simple variables such as polymer concentration, polymer length, temperature and ion concentration. Moreover, we managed to combine rheology and SAXS which allows us to study the structure of the gel under deformation. This combination of two techniques provides fundamental insights in the structural mechanism of the strain stiffening response of our hydrogel.

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## p20 Investigation of Phonon Mediated Phenomena using Nuclear Inelastic Scattering

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The behaviour of collective atomic vibrations, i.e. phonons, influences many material properties, such as thermal and mechanical attributes. Furthermore, phonons have been identified to play an essential role in conventional superconductivity. When reducing the system's dimensions to the nanoscale, the phonon behaviour will deviate from the corresponding bulk phonon behaviour due to restriction of phonon propagation caused by e.g. grain boundaries. Due to the close relationship between critical temperature ( $T_c$ ) and phonon density of states (PDOS) it is imminent that the phonon confinement influences  $T_c$ . Several different nanostructures, such as nano – islands, nano – particles embedded in a matrix and nano – cluster – assembled films have been characterized and their phonon behaviour has been investigated by nuclear resonant inelastic x-ray scattering at the European Synchrotron Radiation Facility (ESRF) and the Advanced Photon Source (APS).

Softening of the high-energy phonon modes, which resulted in an increase in  $T_c$  of up to 11% has been observed.

Not only the superconducting behaviour in Sn is phonon-mediated, also the structural phase transition from the alpha-Sn to the beta-Sn phase is driven by phonons. The semiconducting alpha-Sn phase transforms into the metallic beta-Sn phase above a temperature of 286 K, driven by a change in vibrational entropy [1]. Alpha-Sn thin films can however be stabilized at room temperature by choosing an appropriate substrate [2]. Due to the minimal lattice mismatch between the InSb and the Sn lattice cell parameters, InSb is a good candidate for room temperature stabilization of alpha-Sn thin films. The Sn phase transition has been studied during an in situ experiment at the ESRF, which constitutes a very clean method of probing the PDOS.



Measuring the PDOS at different stages during the phase transformation provides a deeper insight into the mechanisms involved in the phase transition. Furthermore, thermodynamic properties, such as the vibrational entropy, can be extracted from the experimentally obtained PDOS. The transition has been studied for different thicknesses, and an increase in the phase transition temperature with decreasing thicknesses has been observed. Equivalent to bulk, a strong increase of the vibrational entropy during the transition from alpha – to beta – Sn has been found.

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## p21 Core-Shell structures in $\text{Au}_x\text{Ag}_{1-x}$ bimetallic nanoclusters investigated by a combination of HAADF-STEM and GISAXS

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Bimetallic  $\text{Au}_x\text{Ag}_{1-x}$  clusters with various compositions ( $x = 0.8, 0.6$  and  $0.1$ ) were grown in the gas phase and deposited on amorphous  $\text{SiO}_2$  wafers and TEM grids. The clusters were characterized by a high angle annular dark field scanning transmission electron microscopy (HAADF-STEM) and grazing-incidence small-angle x-ray scattering (GISAXS). Clusters with an average diameter of 2.1 nm to 2.6 nm were produced using dual laser ablation of pure Ag and Au metal targets under UHV conditions [1]. Controlling over the composition of the bimetallic clusters in the gas-phase prior to deposition was accomplished by fine tuning of the laser energy densities and the time delay between the laser pulses. Size and composition were monitored by reflectron time of flight mass spectrometry.

The cluster beam was then deposited in soft landing mode on  $\text{SiO}_2$  wafers and TEM grids with a low coverage of approximately 0.1 atomic monolayer (ML) to minimize aggregation. Despite the low surface coverage fraction, GISAXS proved to be extremely sensitive to measure size and cluster organization. Both with HAADF-STEM and GISAXS core-shell structures were observed for all the investigated compositions. A systematic inversion of the structural element in the core and the shell of the clusters was found with the low abundant species constituting the dominant species in the core: in gold rich clusters ( $x = 0.8$  and  $0.6$ ) the core is silver rich and the outer shell is gold rich, while for silver rich clusters ( $x = 0.1$ ) the opposite is found. This core-shell structure is expected to play a significant role in the catalytic properties of these clusters that are currently being tested for CO oxidation reactions in the framework of the Catsense research network [2].

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[2] Catsense – EU FP7 Initial Training Network ‘Design of novel high performance catalysts and biosensors based on deposited mass-selected clusters assisted by computational theoretical screening’ [www.catsense.eu](http://www.catsense.eu)

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## p22 Science and Industrial Application on the Dutch-Belgian EXAFS Beamline (DUBBLE) at ESRF

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The Dutch-Belgian Beamline (DUBBLE) project operates two beam lines at the European Synchrotron Radiation Facility (ESRF). This facility provides beam time for mainly for Dutch and Flemish researchers but 30% of the beam time is available for general ESRF users. The main technique is Extended X-ray Absorption Fine Structure (EXAFS) spectroscopy and simultaneous EXAFS/Small Angle/Wide Angle X-ray Scattering (SAXS/WAXS) in operando condition. EXAFS technique can also be performed in a time-resolved mode.

The EXAFS beam line is specialized in on-line catalysis experiments but users from the fields of environmental geochemistry, biochemistry, archaeology have been increasing in recent years. EXAFS is a powerful technique to investigate the local molecular environment and the chemical states of the atoms of a specific element in almost any type of substance. It is particularly well suited to investigate materials that lack long-range order like:

- Nanomaterials, amorphous and highly disordered solids (clusters, catalysts, etc.)
- Liquids, gels, molecular solutions, liquid crystals (molecular sieves, etc.)
- Molecules and macromolecules containing metallic or heavy atoms (polymers, biomolecules, etc.)

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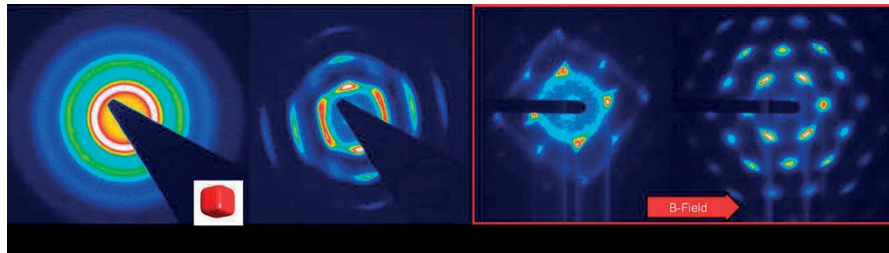
## p23 Self-assembly of Hematite Colloidal Cubes: From Ordered Layers to Single Crystals

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The self-assembly of colloidal particles can be directed in various ways, of which one is changing the building block shape [1]. In this work, we study micron-sized cubic hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ) colloids and their spontaneous self-organization. Colloidal cubes are synthesized with different sizes and the interactions are controlled by solvent composition, surface coating and external fields. Using microscopic techniques and microradian X-ray diffraction we are able to investigate the structure of self-organised densely packed cube sediments in detail (Figure 1). We show that the flat faces of the cubes cause alignment and form short to long-range ordered layered structures. Additional alignment in an external magnetic field causes the formation of a single BCM crystal. The identified 3D structures were found to be similar to those predicted by recent simulations [2]. Our results open up the possibility to assemble colloidal lattices with novel symmetries, which can prove useful for development of new nanomaterials such as photonic crystals.





**Figure 1** | Left to right. SAXS patterns of sediment of attractive cubes, repulsive cubes and cubes in a magnetic field at the liquid-solid interface, both face-centered-like and simple-like cubic phases coexist.

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## p24 Intermediate phases in the formation of 2D superlattices by oriented attachment of nanocrystals

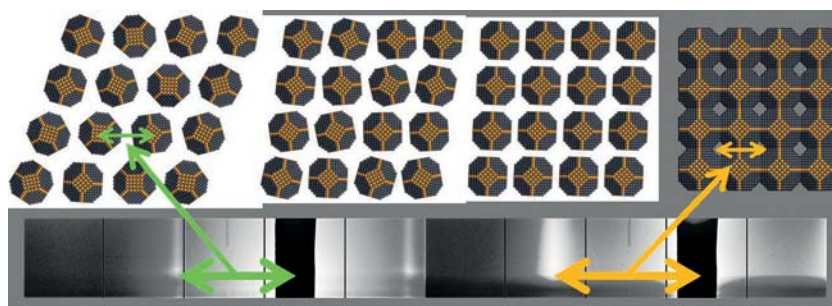
**C. van Overbeek**,<sup>1</sup> J.J. Geuchies,<sup>1</sup> W.H. Evers,<sup>2</sup> F.T. Rabouw,<sup>1</sup> J.L. Peters,<sup>1</sup> J. Hilhorst,<sup>3</sup> A.V. Petukhov,<sup>1</sup> L.D.A. Siebbeles<sup>2</sup> and D. Vanmaekelbergh<sup>1</sup>

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The current interest in materials with a 2D honeycomb geometry is due to their unique optoelectronic properties. We realized semiconductors with this geometry by oriented attachment of colloidal nanocrystals. [1,2] Tight-binding calculations predict that these materials have the conventional band gap of a semiconductor, however with Dirac cones for the conduction electrons and valence holes [3].

A hot issue is how hundreds to thousands of nanocrystals can form highly ordered superstructures through a non-reversible process like oriented attachment. With in-situ GISAXS/WAXS and temporal evolution TEM experiments we observed 2D phases with unusual long-range ordering on atomic and nanocrystal scale, but with the nanocrystals unbound, see Figure 1. Hence, we observed for the first time intermediate phases governed by directional specific interactions. The consecutive reversible phase transitions before facet-to-facet binding cause the observed large-scale ordering in the formation of atomically coherent 2D superlattices.





**Figure 1** | The upper part of the Figure shows the nanocrystals gradually transforming their superlattice from quasi-hexagonal unattached to square attached. Unattached nanocrystals have a larger next-nearest-neighbor distance, because of their corona of stabilizing ligands. The lower part of the Figure shows two stills from in-situ GISAXS data measured during the synthesis of 2D superlattices by oriented attachment of nanocrystals. The peak position in the left still exactly corresponds to lattice distances for nanocrystals in a hexagonal lattice separated by a corona of ligands. This peak gradually moved outwards and finally assumed a distance as seen in the right still. This distance exactly corresponds to attached nanocrystals in a square lattice.

- | 1 | W.H. Evers et al; *Nano Lett.* 13, 2317–2323 (2013). doi:10.1021/nl303322k
- | 2 | M.P. Boneschanscher et al; *Science* 344, 1377 (2014). doi:10.1126/science.1252642
- | 3 | E. Kalesaki et al; *Phys. Rev. X* 4, 011010 (2014). doi:10.1103/PhysRevX.4.011010

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## p25 LARMOR: A Multipurpose Polarised Neutron Instrument for Looking into Materials

J. Plomp,<sup>1</sup> R. Dalgliesh,<sup>2</sup> A.A. van Well,<sup>1</sup> Wim Bouwman,<sup>1</sup> Jilt Siestma,<sup>1</sup> Ilja Voets,<sup>3</sup> Graeme Blake,<sup>4</sup> Thom Palstra<sup>4</sup> and **C.Pappas**<sup>1</sup>

1) Delft University of Technology, 2) ISIS, STFC, UK, 3) Eindhoven University of Technology, 4) Zernike Institute for Advanced Materials, University of Groningen

One of the new neutron scattering instruments at the second target station at ISIS, UK, is LARMOR a Small-Angle Neutron Scattering (SANS) instrument that has the flexibility to accommodate a rich flavour of Larmor labelling techniques. ISIS is designing and constructing the basic Time-Of-Flight (TOF) polarized SANS instrument, which is now in its commissioning phase. A series of options that take advantage of the Polarised neutron Larmor labelling capabilities have been conceived and built in the frame of a project financed by NWO-Groot and which involves a collaboration between the TU Delft, TU Eindhoven and the University of Groningen. The foreseen options include; SANS, Polarized SANS, Spin Echo SANS (SESANS), Diffraction and High Resolution Larmor Diffraction, Neutron (Resonance) Spin Echo, MIEZE, Modulated Intensity SANS (MISANS) and Time Of Flight Larmor precession (TOFLAR). These techniques combined on a single beam line cover a large area in the structure and time domain and it should be possible to combine several complementary techniques during one measurement.

LARMOR will thus be a highly effective analytical tool in the hands of Dutch scientists from universities and industry, who through this project will gain rapid and direct access to it. Indeed, in exchange for the investment ISIS guarantees proprietary beam time for the Dutch scientific community on LARMOR over several years. The team of applicants is responsible for organising the access of Dutch scientists to the instrument in a transparent way and for establishing a continuous scientific exchange among the users.

**The deadline for the first Dutch Call of Proposals is July 15, 2015.**

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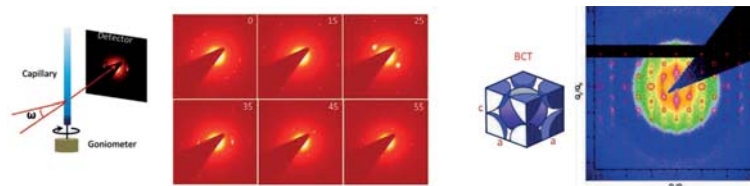


## p26 Microradian X-ray Diffraction Study of Novel Colloidal Architectures

Antara Pal,<sup>1</sup> Janne-Mieke Meijer,<sup>1</sup> Irina Snigireva,<sup>2</sup> Anatoly Snigirev<sup>2</sup> and **Andrei V. Petukhov<sup>1</sup>**

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Hard colloidal spheres are known to be able to self-assemble into periodic close-packed structures such as face-centred cubic or random-stacking hexagonal close-packed crystals. It is interesting to find ways to manipulate the crystal symmetry and to generate a library of colloidal-scale assemblies with different architectures [1]. Here we present a few recent results of synchrotron diffraction studies of crystals of magnetic spherical particles with strong dipole-dipole interactions [2] and asymmetric dumbbells [3]. In this research we have used the microradian x-ray diffraction setup [4] at the Dutch-Belgian beamline BM-26B at the ESRF, Grenoble, France.



**Figure 1** | Microradian diffraction data collected at different sample orientations revealing (i) dominating face-centred-cubic structure with 1.5% stacking faults (very small for colloidal crystals). It also shows that the sample contains a texture of smaller crystallites with a [111] direction perpendicular to the capillary walls.

**Figure 2** | Microradian diffraction pattern from self-organised core-shell silica@magnetite magnetic particles, which provides a clear proof of the body-centred tetragonal (bct) structure rather than the face-centred cubic (fcc) that is usual for hard colloidal spheres.

- [ 1 | J.-M. Meijer, PhD thesis, Utrecht University, January 2015. See also her poster presented at SyNeW-2015.
- [ 2 | A. Pal, J.-M. Meijer, J.R. Wolters, W.K. Kegel and A.V. Petukhov, , *J. Appl. Cryst.*, 48, 238 (2015).
- [ 3 | A. Pal, V. Malik, L. He, B.H. Ern , Y. Yin, W.K. Kegel, A.V. Petukhov, *Angew. Chem. Int. Ed.*, 54, 1803 (2015).
- [ 4 | A.V. Petukhov, J.H.J. Thijssen, D.C. 't Hart, A. Imhof, A. van Blaaderen, I.P. Dolbnya, A. Snigirev, A. Moussa d, I. Snigireva, *J. Appl. Cryst.*, 39, 137 (2006).

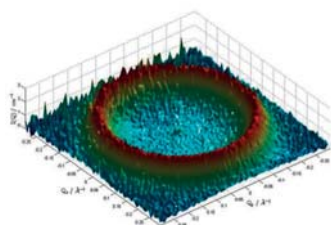
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## p27 LARMOR: Current status

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1) Delft University of Technology, 2) ISIS, STFC, UK

One of the new neutron scattering instruments at the second target station at ISIS, UK, is LARMOR, a Small-Angle Neutron Scattering (SANS) instrument that has the flexibility to accommodate a rich flavour of Larmor labelling techniques. ISIS has designed and constructed a Time-Of-Flight (TOF) polarized SANS instrument, which is now in its commissioning phase. A series of options that take advantage of the Polarised neutron Larmor labelling capabilities are being conceived and built in the frame of a project financed by NWO-Groot and which involves a collaboration between the TU Delft, TU Eindhoven and the University of Groningen. There are many foreseen modes for this instrument and these will be commissioned in steps over the coming years. At this moment the most basic mode of the instrument is available which is the SANS mode.



The SANS scattering from a bulk lamellar structure used for calibration in 3D, measured early May 2015 (picture by ISIS LARMOR team, <https://mobile.twitter.com/larmorisis>)

The next phase would be polarised SANS which is expected to operate end of summer 2015. The first full Delft contribution to the instrument would be the Spin Echo SANS (SESANS) mode. This is expected to be installed end of 2015 and operational early 2016.

At this moment the prototype of the SESANS components has almost been tested completely and we are ready for the production of all components. We will present an overview of the technical innovation and challenges in this project.

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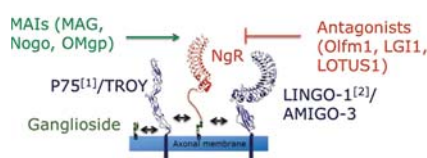
## p28 Ligand Oligomerization in the Central Nervous System

### Structural studies on the Myelin-Associated Glycoprotein dimer and the Olfactomedin-1 tetramer

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1) Crystal and Structural Chemistry, 2) Biomolecular mass spectrometry and proteomics, Bijvoet Center for Biomolecular, 3) Section Electron Microscopy, LUMC, Leiden, 4) Physical and Colloid Chemistry, Debye institute for nanomaterials sciences, Utrecht University

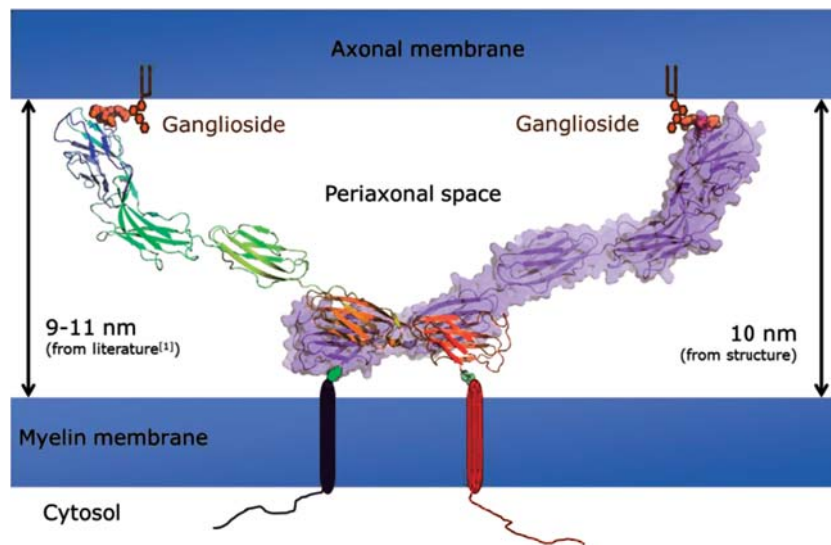
Myelin-associated inhibitor (MAI) signaling via the Nogo receptor (NgR) is a well-studied system in axonal regeneration and plasticity of the central nervous system. Whereas myelin-associated glycoprotein (MAG) and other MAIs inhibit plasticity and regeneration, this signaling can be reversed by antagonists such as Olfactomedin-1 (Olfm1). Using X-ray crystallography and biophysical techniques, we set out to determine the structural basis of these contrasting effects, focusing on MAG and Olfm1.



#### MAG

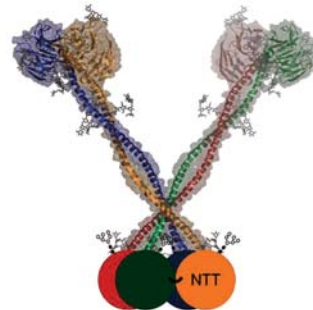
We determined the crystal structure of MAG, which reveals a homodimeric arrangement. We confirmed this dimer by biophysical techniques and mutations. By binding neuronal gangliosides in trans and dimerizing in cis, MAG can restrain the periaxonal diameter as a molecular leaf spring. Furthermore, dimerization upon cell contact might be important for axon-to-myelin signaling during myelin formation.





### Olfm1

Using limited proteolysis we determined the structure of a homodimeric disulfide linked dimer of the C-terminal  $\beta$ -propeller and part of the coiled coil. The combination of the crystal structure, SAXS and electron tomography data as well as the available knowledge about the disulfides and the primary sequence, allows us to reconstruct a model for the full-length Olfm1 tetramer, which suggests a role in receptor clustering.



## p29 Chiral Magnetism in Multiferroic Insulator $\text{Cu}_2\text{OSeO}_3$ : A Polarised Neutron Scattering Study

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1) Delft University of Technology, Mekelweg 15, 2629 JB Delft, the Netherlands, 2) Diamond Light Source Ltd., Chilton OX11 0DE, UK, 3) Institut Laue-Langevin, 6 rue Jules Horowitz, 38042 Grenoble, France, 4) Max Planck Institute for Chemical Physics of Solids, Dresden, Germany

Magnetic chirality is the key ingredient to stabilize skyrmion textures and lattices, which combine fascinating properties with the potential of becoming the spintronic devices of the future. These new states of matter are generated by the anti-symmetric Dzyaloshinskii-Moriya interaction resulting from the absence of a center of symmetry in the crystal structure, as this occurs in thin films, metallic compounds or insulators crystallizing in the B20 structure type (space group  $P2_1$ ). In this topical field of research polarized neutrons provide unique insight on the structure and topology of the magnetic correlations, due to the specificities of the neutron spin. The presentation will focus on the case of a multiferroic insulator  $\text{Cu}_2\text{OSeO}_3$  which orders at  $T_c = 58$  K into a chiral helical state with a period of  $\sim 650$  Å along the (100) direction. Polarised neutron scattering and neutron spin echo spectroscopy were used to investigate the chiral properties and dynamics in  $\text{Cu}_2\text{OSeO}_3$ . We will discuss the chiral magnetic correlations and fluctuations in the very close vicinity of  $T_c$ . And then focus on a systematic study of the scattering under a magnetic field, which leads to a complex temperature-field phase diagram with a well-defined skyrmion lattice phase. We will also report the dynamic phase transitions where the Skyrmion lattice phases appear and then vanish spontaneously after switching off the magnetic field. The results will be discussed in the general context of chiral magnetism.

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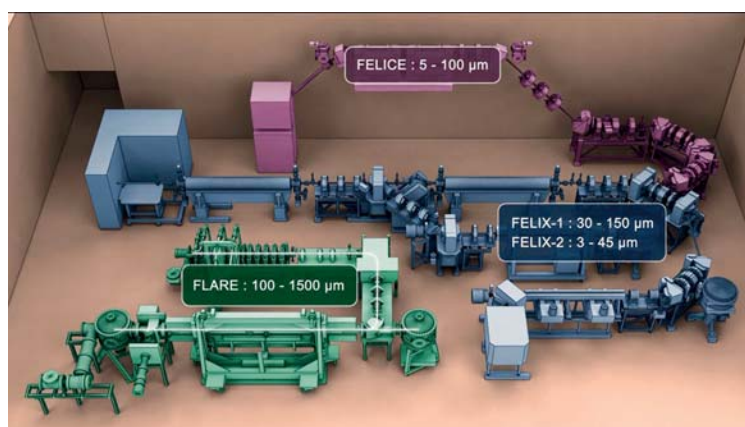
## p30 Investigating Structure and Dynamics using the Infrared and THz Facility FELIX

Joost Bakker, Giel Berden, Jan Kees Maan, Lex van der Meer, Jos Oomens,  
**Britta Redlich**, Anouk Rijs

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The international user facility FELIX (Free Electron Lasers for Infrared eXperiments) at the Radboud University Nijmegen offers a unique tuning range in the infrared and THz regime from 3-1500 micron ( $3300\text{-}6\text{ cm}^{-1}$  or  $100\text{-}0.2\text{ THz}$ ) to perform experiments in various areas of science. It comprises two free electron lasers and three beamlines – FLARE, FELIX and FELICE – that can be operated simultaneously. The layout of the facility and the versatile user laboratories, as well as some special features, such as the intra-cavity operation and the connection of the FEL beam lines to the High Field Magnet Laboratory will be introduced.

The primary applications are found in the areas benefitting either from the high brightness or the high fluence the lasers offer. Selected experiments from these fields including time-resolved measurements in condensed matter systems and action spectroscopy of molecular systems in the gas phase will be presented.



Schematic overview of the Free Electron Laser beamlines at the FELIX Laboratory.

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## p31 *In-situ* X-ray Scattering During the Formation of $\text{Cu}_{2-x}\text{S}$ nanosheets

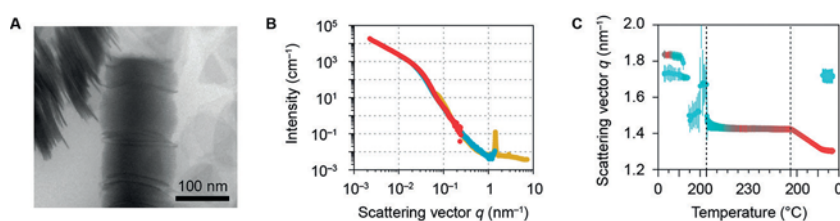
**Ward van der Stam**,<sup>1</sup> Freddy T. Rabouw,<sup>1</sup> Jaco J. Geuchies,<sup>1</sup> Anne C. Berends,<sup>1</sup> Stijn O. M. Hinterding,<sup>1</sup> Robin G. Geitenbeek,<sup>1</sup> Joost van der Lit,<sup>1</sup> Sylvain Prévost,<sup>2</sup> Andrei V. Petukhov<sup>1</sup> & Celso de Mello Donegá<sup>1</sup>

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The electronic properties of nanocrystals depend not only on the chemical composition, but also on size and shape. The desired size or shapes are obtained by choosing the reaction conditions and chemicals used in the synthesis. For example, we have recently demonstrated that the shape of  $\text{Cu}_{2-x}\text{S}$  nanoparticles formed when dodecanethiol decomposes in the presence of a Cu-salt, changes from spherical nanocrystals to ultrathin nanosheets when halides are added [1]. However, little is known about the precise reaction mechanisms that govern the formation of nanocrystals, in particular for the case of non-spherical, anisotropic shapes. In this work we perform *in-situ* small-angle x-ray scattering (SAXS) and wide-angle x-ray scattering (WAXS) experiments during the formation of spherical  $\text{Cu}_{2-x}\text{S}$  nanocrystals and  $\text{Cu}_{2-x}\text{S}$  nanosheets. Figure 1 shows an example of one of our experiments, in which triangular ultrathin nanosheets of 2 nm thickness were obtained in the presence of chloride ions [1]. The intermediate and precursor stages of the reaction could be monitored *in-situ* using SAXS and WAXS, revealing the formation of nanosheets and nanosheet stacks (Figure 1b,c).



$\text{Cu}_{2-x}\text{S}$  has proven to be a robust system to study with *in-situ* x-ray scattering. The desired nanocrystal geometries are obtained in a capillary, despite the difficulties to exactly reproduce the reaction conditions of the lab. Therefore, our results give meaningful insights in the formation of 2-dimensional nanomaterials.



**Figure 1 |** The formation of  $\text{Cu}_{2-x}\text{S}$  nanosheets.  $\text{Cu}_{2-x}\text{S}$  nanosheets form when a solution of CuCl in dodecanethiol, octadecene, and trioctylphosphine oxide is heated to 230°C in a capillary. (A) The final product isolated and imaged with transmission electron microscopy. (B) The SAXS pattern of the reaction mixture after ~10 min at 230°C, measured at three detector to sample distances. (C) The fitted position, width, and intensity (indicated by color) of structure factor peaks as a function of reaction time.

| 1 | van der Stam, W. et al., *Chem. Mater.* 27, 283–291 (2015)

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## p32 The SLcam and its Application Towards Full-field Emission Mode X-ray Absorption Spectroscopy

**P. Tack**,<sup>1</sup> J. Garrevoet,<sup>1</sup> S. Bauters,<sup>1</sup> B. Vekemans,<sup>1</sup> D. Banerjee,<sup>2</sup> A. Longo,<sup>2</sup> W. Bras<sup>2</sup> and L. Vincze<sup>1</sup>

1) X-ray Microspectroscopy and Imaging Group (XMI), Ghent University, Krijgslaan 281 S12, B-9000 Ghent, Belgium, 2) European Synchrotron Radiation Facility (ESRF), DUBBLE-CRG, FR-38043 Grenoble Cedex, France

X-ray Absorption Fine Structure (XAFS) spectroscopy is feasible routinely only at synchrotron radiation facilities. Access to these facilities is high in demand and as a result the experimental planning of XAFS experiments is often time constrained. Because of this time constraint and the time consuming nature of XAFS experiments, chemical state imaging was previously practically unfeasible and limited to measuring separate points of interest determined previously by coupled techniques such as X-ray Fluorescence (XRF) spectroscopy.

The very recent introduction of a state-of-the-art full-field energy-dispersive CCD detector, the SLcam, allows the simultaneous recording of 69,696 XAFS-spectra of the sample, corresponding to 264 by 264 energy-dispersive pixels having microscopic pixel resolution [1]. When combined with an energy tuneable broad-beam excitation, the use of the SLcam allows the collection of X-ray Absorption Near Edge Structure (XANES) image sets in emission mode, capable of studying dilute systems down to trace concentration levels for elements from Ti to U. During a recent pilot experiment at the DUBBLE beamline (ESRF), this novel methodology was successfully tested, resulting in iron chemical state images of a 2 by 2 mm<sup>2</sup> sample area with a spatial resolution down to 8 micron in less than 8 hours of measuring time [2]. Additionally, it is possible to virtually slice the sample, resulting in 3D chemical state information of cubic millimetre sized sampled volumes with microscopic resolution.





The new methodology described is believed to significantly enhance the way and the speed by which fluorescence mode XAFS experiments are performed today. Difficulties lay in further improving the used detector optics, as well as developing and optimising data processing routines that can efficiently handle the large amount of data that is created using this technique.

- | 1 | O. Scharf, S. Ihle, I. Ordavo, V. Arkadiev, A. Bjeoumikhov, S. Bjeoumikhova, G. Buzanich, R. Gubzhokov, A. Gunther, R. Hartmann, M. Kuhbacher, M. Lang, N. Langhoff, A. Liebel, M. Radtke, U. Reinholz, H. Riesemeier, H. Soltau, L. Struder, A.F. Thunemann, R. Wedell, *Anal Chem* 83 (2011) 2532.
- | 2 | P. Tack, J. Garrevoet, S. Bauters, B. Vekemans, B. Laforce, E. Van Ranst, D. Banerjee, A. Longo, W. Bras, L. Vincze, *Anal Chem* 86 (2014) 8791.

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## p33 Unusual Melting Behavior in Flow Induced Crystallization of LLDPE: Effect of Pressure

**E.M.Troisi**, G. Portale and G.W.M. Peters

Flow plays a key role in the crystallization of polymers since it can enhance crystallization kinetics order of magnitude through the formation of point-like nuclei or change the crystalline morphology from spherulitic to highly oriented structure as shish-kebab. In the same way, pressure can influence crystallization increasing the nucleation density and promoting the formation of certain crystals polymorphs, like the  $\gamma$ -form of isotactic polypropylene and the *hexagonal-form* in polyethylene. During injection molding, the most widespread technology to process polymeric materials into products of various sizes and geometries, the crystallizing polymer melt is subjected to complex flow fields and high pressures, leading to orientation and anisotropic properties in the final products.

In this study we focus on the crystallization behavior for processing-like conditions of a linear low density polyethylene (LLDPE). Flow induced crystallization (FIC) was investigated performing step shear experiments in a confined slit flow geometry within a modified Multi-Pass Rheometer, able to mimic flow and pressure conditions as experienced in injection molding (wall shear rates up to 1000 s<sup>-1</sup> and pressures up to 500 bars). Experiments were combined with time resolved in-situ wide-angle X-ray diffraction (WAXD), small angle X-ray scattering (SAXS).

Formation of shish-kebab structures was detected during the shear pulse and the upcoming isothermal crystallization was monitored to follow the development of oriented crystals.

The results showed the unusual melting of part of the oriented lamellae (kebabs) immediately after flow. Use of a structural model to capture the SAXS data permitted us to relate this behavior to pressure driven crystallization/relaxation and to gain understanding on the effect of pressure rise in confined geometry on structure formation.



## p34 X-ray powder diffraction microtomography identifies plumbonacrite as degradation intermediate of minium, a pigment used by Van Gogh

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Faculty of Sciences, University of Antwerp, Belgium

Red lead, a semiconductor pigment used by artists since antiquity, is known to undergo several discoloration phenomena. These transformations are either described as darkening of the pigment caused by the formation of either plattnerite ( $\beta\text{-PbO}_2$ ) or galena ( $\text{PbS}$ ) or as whitening by which red lead is converted into anglesite ( $\text{PbSO}_4$ ) or (hydro)cerussite ( $2\text{PbCO}_3 \cdot \text{Pb(OH)}_2$ ;  $\text{PbCO}_3$ ).

X-ray powder diffraction tomography, a powerful analytical method that allows visualization of the internal distribution of different crystalline compounds in complex samples, was used to investigate a microscopic paint sample from a Van Gogh painting.  $\mu\text{-XRPD}$  2D mapping can be used to identify, visualize and to a certain extent quantify the different crystalline components that are present in complex heterogeneous paint systems. At state-of-the-art synchrotron radiation facilities this can be done on the (sub)micrometer scale. With  $\mu\text{-XRPD}$  tomography the inner distribution of the crystalline components present in such samples can be visualized without physically cross-sectioning the material under investigation.

A very rare lead mineral, plumbonacrite ( $3\text{PbCO}_3 \cdot \text{Pb(OH)}_2 \cdot \text{PbO}$ ), was revealed to be present in a degraded paint sample, containing minium, obtained from a painting of Vincent Van Gogh, called *Wheat Stack Under a Cloudy Sky* by Vincent van Gogh (October 1889, oil on canvas, Kröller-Müller Museum, Otterlo, NL). This is the first reported occurrence of this compound in a painting dating from before the mid-20th century. It constitutes the missing link between on the one hand the photo-induced reduction of red lead and on the other hand (hydro)cerussite, and thus sheds new light on the whitening of red lead.

| 1 | F. Vanmeert et al., *Angewandte Chemie International Edition*, 54 (2015) 1-5; DOI: 10.1002/anie.201411691.



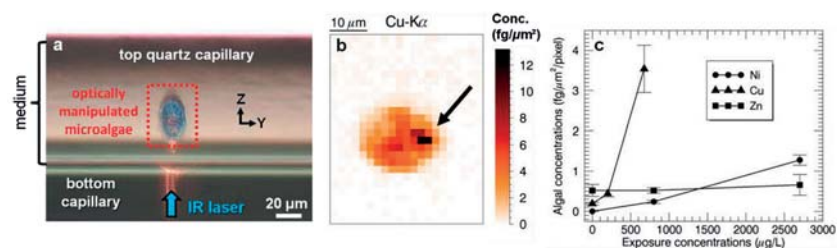
## p35 Non-contact Optical Tweezers-based Single Cell Analysis through *in vivo* X-ray Elemental Imaging

**Eva Vergucht**,<sup>1</sup> Toon Brans,<sup>2,3</sup> Filip Beunis,<sup>2,3</sup> Jan Garrevoet,<sup>1</sup> Stephen Bauters,<sup>1,4</sup> Maarten De Rijcke,<sup>5</sup> David Deruytter,<sup>5</sup> Colin Janssen,<sup>5</sup> Christian Riekkel,<sup>4</sup> Manfred Burghammer<sup>4,1</sup> and Laszlo Vincze<sup>1</sup>

1) X-ray Microspectroscopy and Imaging Group, Ghent University, Belgium, 2) Department of Electronics and Information Systems, Ghent University, Belgium, 3) Center for Nano and Biophotonics, Ghent University, Belgium, 4) European Synchrotron Radiation Facility, Grenoble, France, 5) Laboratory of Environmental Toxicology and Aquatic Ecology, Ghent University, Belgium

We report on a radically new elemental imaging approach for the analysis of biological model organisms and single cells in their natural, *in vivo* state. The methodology combines optical tweezers (OT) technology for non-contact, laser-based sample manipulation with synchrotron radiation confocal X-ray fluorescence (XRF) microimaging for the first time. The main objective of this work is to establish a new method for in situ elemental imaging of free-standing living biological microorganisms or single cells in their aqueous environment. Using the model organism *Scrippsiella trochoidea*, several successful test experiments focussing on applications in environmental toxicology have been performed at ESRF-ID13, demonstrating the feasibility, repeatability and high throughput potential of the OT XRF methodology. We expect that the OT XRF methodology will significantly contribute to the new trend of investigating microorganisms at the cellular level with added in vivo capability [1,2].





**Figure 1** | OT XRF-based in vivo experimental results. (a) Optically manipulated *S. trochoidea* microalgae in natural environment. (b) Cu-K $\alpha$  elemental distribution of a scanned microalgae showing a localized aggregation. (c) Tendency towards the bio-accumulation of Ni, Cu and Zn.

- [ 1 ] E. Vergucht, T. Brans, F. Beunis, J. Garrevoet, M. De Rijcke, S. Bauters, D. Deruytter, M. Vandeghechuchte, I. Van Nieuwenhove, C. Janssen, M. Burghammer, L. Vincze, *Sci. Rep.* (Nature Publishing Group), 5, 9049 (2015).
- [ 2 ] E. Vergucht, T. Brans, F. Beunis, J. Garrevoet, S. Bauters, M. De Rijcke, D. Deruytter, C. Janssen, C. Riekkel, M. Burghammer, L. Vincze, *J. Synchrotron Rad.* (2015, in press).

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## p36 The Semicrystalline Structure of Polyamide 12: A Combined SAXS, WAXD and Solid State NMR Study

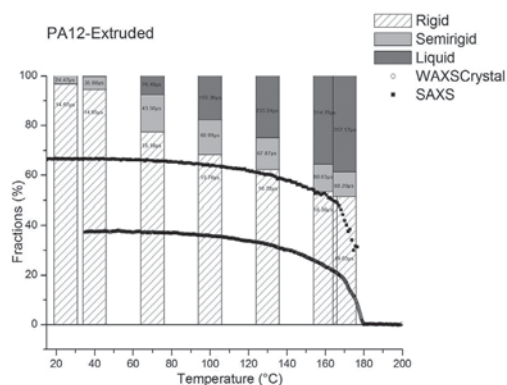
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The semicrystalline morphology of polyamide 12 (PA12) and of many other polyamides is complex because of crystalline polymorphism, the presence of mesomorphic phases and rigid amorphous material besides fully amorphous, liquid-like matter. Quantifying the amount and topology of these phases is challenging and requires the combination of different techniques that are sensitive to specific properties of the phases. SAXS as well as solid state NMR are density-dependent. NMR is very strong in quantifying the phase fractions whereas SAXS is able to reveal their topology, based on a suitable structural model. However, such models quite often do not uniquely cover the SAXS data, unless realistic constraints are used, such as the phase fractions provided by NMR. In that respect SAXS and NMR are highly complementary. WAXD data analysis yields the mass fraction crystallinity, information on polymorphism and mesomorphism as well as estimates on the crystal size and perfection. Again, WAXD can be complementary to SAXS when the WAXD based crystal size or crystallinity is used as input to model the morphology behind the SAXS patterns. Combining synchrotron SAXS and WAXD with solid state NMR allowed compiling a morphological picture for PA12, a snapshot of which is illustrated in Figure 1.

**Figure 1** | A combination of SAXS, WAXD and solid state NMR (shaded bars) data describing the PA12 semicrystalline structure

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## p37 Gravitation-induced Quantum Phase Shift in a Spin- Echo Neutron Interferometer

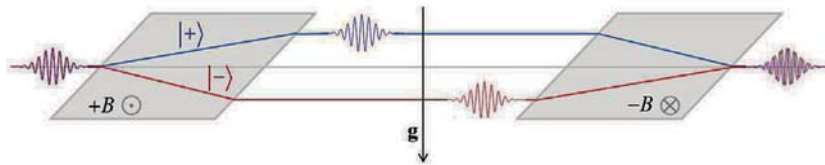
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Several fundamental concepts in physics, such as particle-wave duality, equivalence of inertial and gravitational mass, and the description of a gravitational field in quantum mechanics, play an important role in this research. In an interferometer, the neutron De Broglie wave is coherently split and later recombined. The difference in the Earth's gravitational field in the two paths results in a phase shift of the combined wave. In earlier experiments, a silicon single-crystal Bonse-Hart type interferometer was used, resulting in a 1% discrepancy between the theoretically predicted and experimentally determined phase.

We conducted an experiment [1] in a neutron interferometer where the neutron beam is coherently split in two wave packets, representing the plus and minus spin state of the neutron. By entering a tilted magnetic field both spin states experience opposite Zeeman potential energy and refract differently. Due to this, the corresponding wave vectors will change both in direction and magnitude (see Figure 1). We performed experiments with splitting in both the horizontal and vertical plane and measured a gravitation induced phase that agrees with an accuracy of 0.1% with the theoretical one. Moreover, we showed that an inclination angle of the interferometer with respect to the horizontal of 1 degree leads to a deviation of 1%.





**Figure 1** | In our spin-echo neutron interferometer the magnetic fields are 4 m apart, the energy of the incoming neutrons is  $10^{-2}$  eV, the Zeeman energy is  $10^{-9}$  eV resulting in a distance between the two paths of 1 micrometre, leading to a difference in gravitation potential of  $10^{-13}$  eV.

| 1 | V.O. de Haan, J. Plomp, A.A. van Well, M.T. Rekveldt, Y.H. Hasegawa, R.M. Dalglish, N.J. Steinke, *Phys. Rev. A* 89 (2014) 063611

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## p38 Organic/Inorganic Hybrid Multiferroic Nanocomposites based on Block Copolymer Templates

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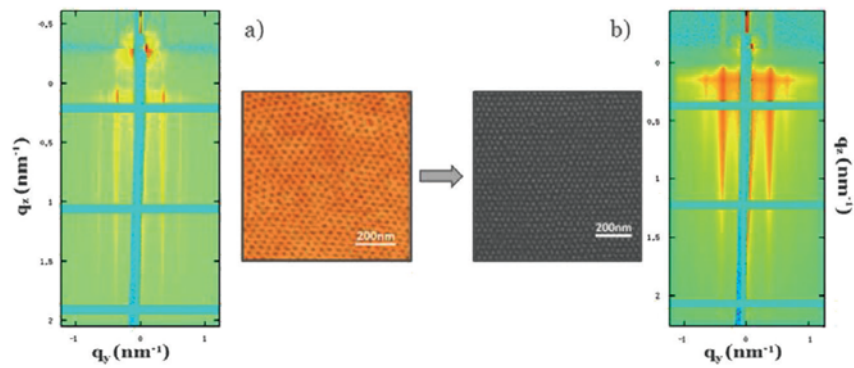
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Magnetoelectric (ME) materials exhibit coupling between magnetic and electric polarizations. While most ME single-phase compounds perform as such only at low temperatures, ME composites open the door to a new technology generation of memory devices and sensors, whose key property is the coupling at room temperature [1]. Shrinking the structural scale down to nanometers makes ME composites compatible with integrated devices.

A promising attempt to obtain ME nanocomposites is to prepare multiferroic nanocomposites, considering the possibility of coupling between ferroelectric and ferromagnetic (or ferrimagnetic) materials [2]. Current fabrication approaches of ordered multiferroic nanocomposites such as pulsed laser deposition or pulsed electron deposition are usually costly and only work with small samples. A low-cost fabrication method for large-area production is highly demanded.

We address this demand by developing a solution based synthesis route, using self-assembled block copolymers (BCP) as templates (Figure 1a), metal oxides as the ferrimagnetic phase (Figure 1b) and poly[(vinylidene fluoride)-co-trifluoroethylene] [P(VDF-TrFE)] as the ferroelectric phase. Highly ordered nanostructures of BCP templates are well transferred to the nanocomposites. The multiferroic property of the composites are demonstrated by electric and magnetic studies. Such organic/inorganic hybrid composites integrate the good processability and low cost of ferroelectric polymers with the solid magnetic property of inorganic ferrimagnets.





**Figure 1** | a) GISAXS and AFM results of BCP template; b) SEM and GISAXS results of metal oxide nanodots .

- | 1 | W. Eerenstein, N.D. Mathur, J.F. Scott, *Nature* 442 (2006) 759.
- | 2 | R.O. Cherifi, V. Ivanovskaya, L.C. Phillips, A. Zobelli, I. C. Infante, E. Jacquet, V. Garcia, S. Fusil, P.R. Briddon, N. Guiblin, A. Mougin, A.A. Ünal, F. Kronast, S. Valencia, B. Dkhil, A. Barthélémy, M. Bibes, *Nature Mater.* 13 (2014) 345.

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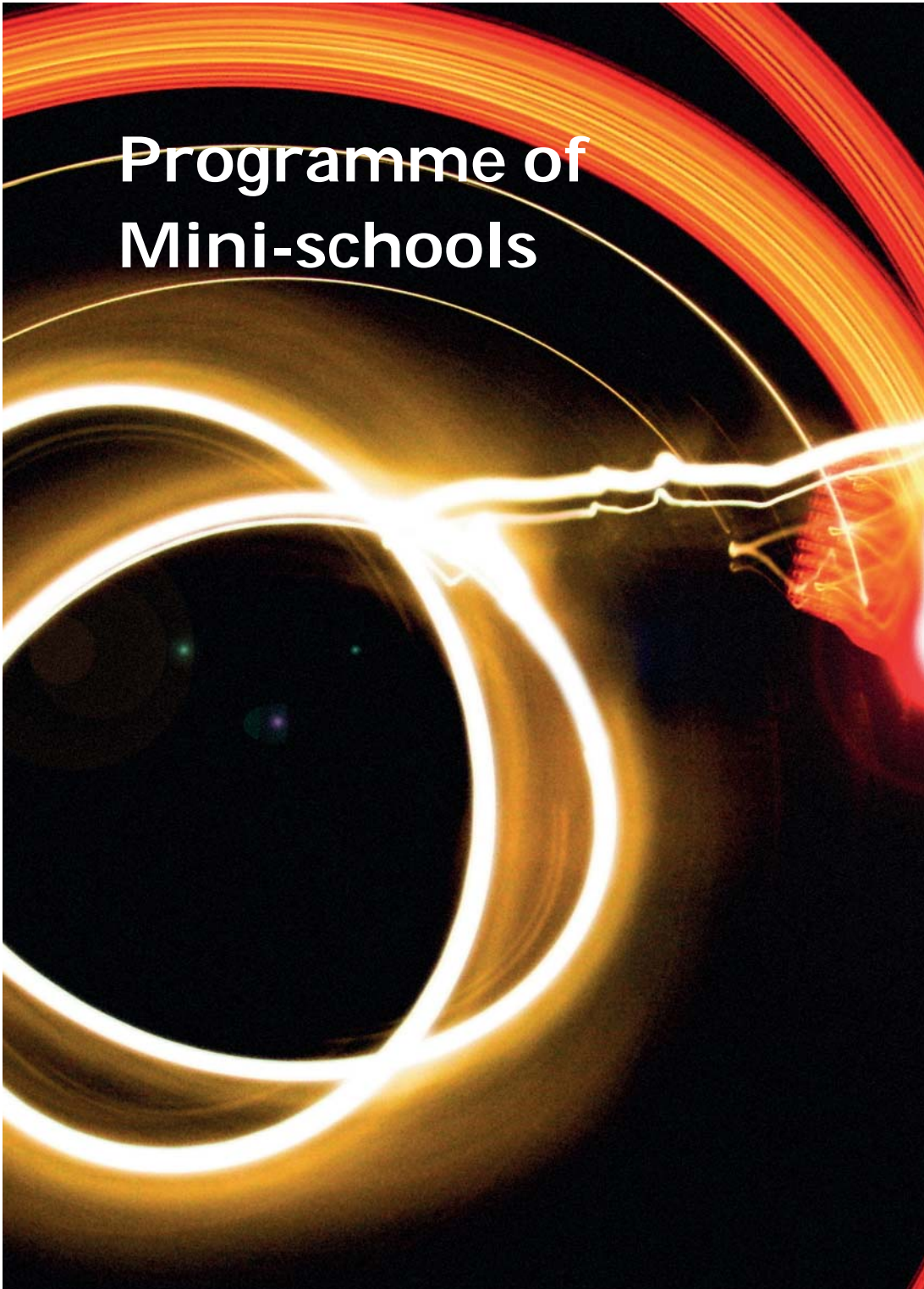
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# Programme of Mini-schools





## X-ray Techniques in Biology

Tuesday, June 2, 2015

Organizers	<b>Piet Gros, Loes Kroon-Batenburg</b> (Utrecht)
Venue	9:00-10:45 Buys Ballot Building 165 10:45-17:00 Buys Ballot Building 112
9:00 – 9:45	<b>Tassos Perrakis</b> (NKI Amsterdam) <i>Microfocus beamlines for macromolecular crystallography: from µmicro towards nano</i>
9:45 -10:30	<b>Cornelius Gati</b> (CFEL Hamburg) <i>Serial Femtosecond Crystallography of G-Protein Coupled Receptors</i>
10:30-11:00	Coffee Break
11:00-11:45	<b>Albert Guskov</b> (RUG Groningen) <i>SFX on membrane transporters</i>
11:45-12:30	<b>Loes Kroon-Batenburg</b> (UU Utrecht) <i>Estimating partialities in serial crystallography with EVAL</i>
12:30-13:30	Lunch Break
13:30-14:15	<b>Arjen Jakobi</b> (EMBL Hamburg) <i>In cellulo crystallisation and X-ray diffraction with yeast peroxisomes</i>
14:15-15:00	<b>Thomas Barends</b> (MPI Heidelberg) <i>De Novo Phasing with XFEL Data</i>
15:00-15:30	Coffee Break
15:30-16:15	<b>Adam Round</b> (ESRF Grenoble) <i>Small angle scattering of biological macromolecules in solution (BioSAS): What can be done and how to do it.</i>
16:15	<i>Drinks</i>



## Small Angle Scattering

Tuesday, June 2, 2015

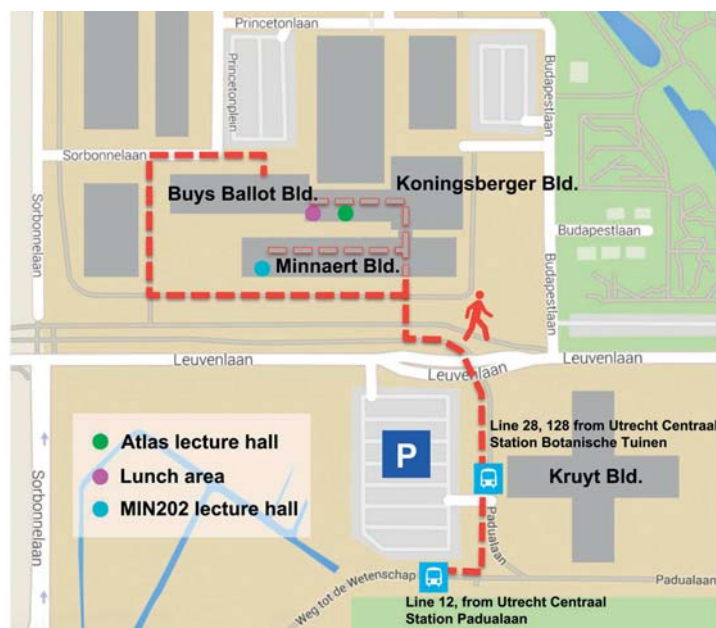
Organizers	<b>Wim Bras</b> (DUBBLE@ESRF) <b>Wim Bouwman</b> (Delft)
Venue	9:00-13:00 Minnaert 202 13:00-17:00 Atlas, Koningsberger
9:00-9:45	<b>Luigi Balzano</b> (DSM) <i>Introduction SAS, SAS on polymers</i>
9:45-10:30	<b>Adam Round</b> (ESRF) <i>Solution Scattering</i>
10:30-11:00	Coffee Break
11:00-11:45	<b>Otto Glatter</b> (Graz) <i>Data Analysis</i>
11:45-12:30	<b>Giuseppe Portale</b> (DUBBLE@ESRF) <i>Grazing Incidence</i>
12:30-13:30	Lunch Break
13:30-14:15	<b>Wim Bras</b> (DUBBLE@ESRF) <i>Technique combination and sample environment</i>
14:15-15:00	<b>Niels van Dijk</b> (TUD) <i>Hard Matter</i>
15:00-15:30	Coffee Break
15:30-15:40	<b>Wim Bras</b> (DUBBLE@ESRF) <i>How and where to get beam time?</i>
15:40-16:15	<b>Wim Bouwman</b> (TUD) <i>SESANS</i>
16:25	<i>Drinks</i>

## X-Ray Absorption Spectroscopy

Tuesday, June 2, 2015

Organizers	<b>Martin Feiters</b> (Nijmegen) <b>Florian Meirer</b> (Utrecht) <b>Frank de Groot</b> (Utrecht)
Venue	<i>Buys Ballot Building 115</i>
9:00-9:45	<b>Martin Feiters</b> (RU) <i>Introduction to X-ray Absorption Spectroscopy, Extended X-ray Absorption Fine Structure</i>
9:45-10:30	<b>Frank de Groot</b> (UU) <i>X-ray absorption spectroscopy near the edge. (aka XANES)</i>
10:30-11:00	Coffee Break
11:00-11:45	<b>Dipanjan Banerjee</b> (DUBBLE@ESRF) <i>X-ray sources, optics, and detectors for absorption spectroscopy beamlines</i>
11:45-12:30	<b>Alessandro Longo</b> (DUBBLE@ESRF) <i>Introduction to EXAFS data analysis.</i>
12:30-13:30	Lunch Break
13:30-14:10	<b>Martin Feiters</b> (RU) <i>Coordination Chemistry and Trace Element Biology</i>
14:10-14:55	<b>Florian Meirer</b> (UU) <i>X-ray micro-spectroscopy, tomography, and operando conditions</i>
14:55-15:20	Coffee Break
15:20-15:50	<b>Mario Delgado</b> (UU) <i>Holistic data analysis with Blueprint XAS</i>
15:50-16:30	<b>Frank de Groot</b> (UU) <i>The future of X-ray absorption spectroscopy</i>
16:30	Drinks

## Map to reach mini-schools on June 2



### Suggested bus services Utrecht CS <-> The Uithof

#### Bus 12

Utrecht CS Centrumzijde	08:26	08:30	08:35	08:39
Utrecht Padualaan	08:39	08:43	08:48	08:52

Utrecht Padualaan	16:31	16:35	16:39	16:44	16:48	16:52	16:57	17:01	17:05	17:10	17:14
Utrecht CS Centrumzijde	16:47	16:51	16:55	17:00	17:04	17:08	17:13	17:17	17:21	17:26	17:30

#### Bus 128

Utrecht CS Centrumzijde	08:14	08:29
Utrecht Botanische Tuinen	08:30	08:45

Utrecht Botanische Tuinen	16:20	16:35	16:50	17:05	17:20
Utrecht CS Centrumzijde	16:40	16:55	17:10	17:25	17:40



# Notes

A series of horizontal dotted lines for taking notes.



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