In the framework of this approach, results of recent studies of local atomic and electronic structure for several types of nanostructures: free and supported Cu nanoclusters, magnetic nanoclusters, and irradiated by C and Si ions ZnO thin films are reported. The parameters of local atomic structure obtained from the XANES spectra analysis have been controlled by using theoretical optimization of the atomic structures on the basis of density function theory. The research is supported by RFBR 10-02-92658-IND_a and the President of Russian Federation MK-4283.2010.2 grants.

[1] Grigory Smolentsev, Alexander V. Soldatov *Computational Materials Science* **2007**, *39*, 569.

Keywords: XANES

MS.56.3

Acta Cryst. (2011) A**67,** C131

Graphene, electrons, plasmons, and quantum: A perfect match

F. Javier Gacía de Abajo, *Instituto de Óptica-CSIC, Serrano 121, 28006 Madrid*, *(Spain).* E-mail: J.G.deAbajo@csic.es

Electron beams constitute excellent tools to probe both propagating and localized plasmons with outstanding spatial resolution. The inelastic events recorded in electron energy-loss spectroscopy (EELS) and in light emission during electron-plasmon interaction (cathodolumisnescence, CL) have been recently used to resolve plasmon excitations and to yield maps with detailed spatial distributions [1]. Here, we will illustrate several recent examples of plasmon mapping via EELS and CL in both extended metallic nanostructures and in nanoparticles. We will also discuss plasmons in graphene as an emerging powerful framework to study the interaction between photon, plasmons, and electrons at the single particle level, with potential applications to areas as varied as ultrasensitive biosensing, nonlinear optics, and quantum information processing.

[1] F.J. García de Abajo, *Reviews of Modern Physics* **2010**, *82*, 209-275.

Keywords: plasmon, EELS, cathodoluminescence

MS.56.4

Acta Cryst. (2011) A**67,** C131

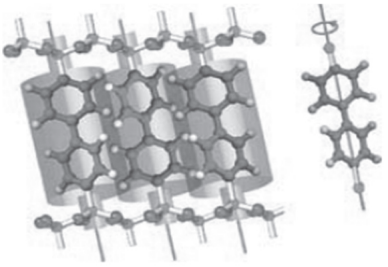
Amphipathic and amphidynamic crystalline materials: an XRD and MAS NMR study

Angiolina Comotti, Silvia Bracco, Mario Beretta, Piero Sozzani, *Department of Materials Science, University of Milano Bicocca, Milan, (Italy).* E-mail: angiolina.comotti@mater.unimib.it

Molecular self-assembled crystalline materials are promising in several fields, including gas storage, selective recognition and modulation of functions of active molecules. The tandem X-ray diffraction and solid state NMR approach allowed us to study amphipathic or amphidynamic materials i.e. crystalline structures that exhibit an intrinsic duality within the same periodic architecture [1-4]. In particular, we realized self-assembled crystalline architectures with guest molecules compartimentalized in two amphipathic nanospaces with distinct geometries and polarities [5]. The effect of these distinct environments on the NMR properties of the guest molecules is evident from chemical shift data and 2D heterocorrelated NMR techniques that could discriminate identical guest molecules embedded in distinct structural environments - one highly polar and the other nonpolar. The large magnetic susceptibility effect, due to ring currents of the aromatic host, enabled the determination of the host-guest distances and corroborated the variable-temperature crystal structure resolution. A dual behavior was also highlighted in a block copolymer. The molecular recognition of specific blocks of triblock copolymers by a host molecule led to the formation of hierarchical periodic structures [2]. The end blocks of the triblock copolymer were locked into the inclusion crystals whilst the central block was excluded, creating a new material of assembled nanocrystals regularly superimposed on one another. The formation of the supramolecular architectures was followed *in situ* synchrotron X-ray diffraction while fast-^{1}H MAS NMR provided direct evidence of selective inclusion of the blocks.

Notably, amphidynamic materials could be recognized in hybrid organic-inorganic crystalline materials [1]. The precise engineering of highly-organized porous materials containing organic elements pivoted on inorganic layers enabled the fabrication of fast molecular rotors entirely exposed to the guest molecules exploring the cavities. Powder X-ray diffraction highlighted the crystalline order on both the meso- and molecular scales. Spin-echo deuterium NMR gave direct proof of the reorientation rate of the aligned rotors and demonstrated the active role of the guests in modulating the rotor dynamics.

[1] A. Comotti, S. Bracco, M. Beretta, P. Valsesia, P. Sozzani, *Angew. Chemie Int. Ed.* **2010**, *49*, 1760-1764. [2] S. Bracco, A. Comotti, L. Ferretti, P. Sozzani, *J. Am. Chem. Soc.* **2011**, *in press*. [3] S. Bracco, A. Comotti, P. Valsesia, M. Beretta, P. Sozzani, *Cryst. Eng. Commun.* **2010**, *12*, 2318-2321. [4] A. Soegiarto, A. Comotti, M.D. Ward, *J. Am. Chem. Soc.* **2010,** *132*, 14603-14616. [5] A. Comotti, S. Bracco, P. Sozzani, S. Hawxwell, C. Hu, M. Ward, *Cryst. Growth Des.* **2009**, *9*, 2999-3002.



Keywords: NMR, inclusion, dynamics

MS.56.5

Acta Cryst. (2011) A**67,** C131-C132

Metal-semiconductor surface phase transitions: A photoelectron – diffraction study

Jose Avila, Maria Carmen Asensio, *Synchrotron SOLEIL, BP-48, 91192 Gif sur Yvette Cedex (France).* E-mail: avila@synchrotron-soleil.fr

The origin of surface phase transitions has been a matter of intense dispute among theoreticians and experimentalists, during the last years. Concurrently, inside surface science, systematic studies of simple model systems has successfully provided remarkable advances during the last 40 years. In spite of this, the emergence of novel powerful techniques has made attainable a more truthful representation of those "well-known" traditional systems. The improved new pictures confirm that complex phenomena take place at surface originally described as simple model systems. In particular, notions as Peierls distortions, Fermi surface nesting, Jahn-Teller distortions, metal-insulator fluctuations, disproportionation and charge and spin density waves, have been pointed out as responsible mechanisms of reported Surface Phase Transitions. In this sense, pioneer studies have focused their attention on the traditional Pb or Sn adsorbed on Ge(111) surfaces,

where several transitions have been reported and related to a wide range of diverse driving forces. In this talk, a comprehensive review of those phenomena will be presented.

Concerning the instrumentation, a special attention will be paid to microscopic techniques able to identify the role of heterogeneities at the surface phase transitions. It is manifest that the elevated flux of the third generations synchrotron radiation sources are essentially profited in the area of the X-ray microscopy. Now a day, those light sources are able to provide high brightness at micrometric or even nanometric beam sizes. In such context, photoemission microscopes will be introduced as tools capable to fill up the existing emptiness between the STM spectroscopy and the low-spatially resolved traditional ARPES and NEXAFS in the area of Surface Phase transitions.

Keywords: phase transition, microspectroscopy, surface

MS.57.1

Acta Cryst. (2011) A**67,** C132

An attempt to prepare membrane proteins using the wheat cell-free protein production system

Yaeta Endo, Tatsuya Sawasaki, Lassale Michael W., *Cell-Free Science and Technology Research Center, Ehime University, Matsuyama 790-8577, (Japan); Systems and Structural Biology Center, RIKEN, 1-7-22 Suehiro-cho, Tsurumi-ku, Yokohama, Kanagawa 230-0045, (Japan).*

The cell-free protein production system we developed from wheat embryos has the significant advantage of producing eukaryotic multidomain proteins in a folded state. In this paper, I will briefly introduce the characteristics and capability of this system together with some results of our ongoing work on the biochemical drawing of protein networks and examples of sample preparation including multiprotein complexes for structural analysis. I will then focus on our recent attempt to develop a versatile methodology for preparing membrane proteins using the wheat cell-free system. Performing translation in the presence of liposomes, we could produce all of the 30 membrane proteins chosen for the test, which included GPCRs and ion channels, each in the form of a protein-liposome complex. These complexes were isolated by brief centrifugation without any purification tags, and were solubilized with a detergent solution. Each protein was separated by Superdex200 gel-filtration column using a buffer solution containing fos choline-14. The purified membrane proteins all exhibited mono-disperse peaks in chromatogram. Three proteins HRH2, DRD1, and HTR3A, were selected for ligand binding assay. The Kd values determined by Biacore using respective defective mutants as a reference confirmed specific binding activity retained in each of the three purified proteins. Although the binding affinity of proteins were not high enough at the moment, further optimization of the solubilization conditions may bring the protocol as a useful HT-methodology for preparing membrane protein samples.

Keywords: preparation of proteins, cell-free, difficult protein

MS.57.2

Acta Cryst. (2011) A**67,** C132

Structural biology of G protein-coupled receptors

Vadim Cherezov, Beili Wu, Ellen Chien, Wei Liu, Fei Xu, Raymond C. Stevens, *Department of Molecular Biology, The Scripps Research Institute, La Jolla (USA).* E-mail: vcherezo@scripps.edu

G protein-coupled receptors (GPCR) constitute the largest family of integral membrane proteins that transmit signals inside cells in response to a variety of extracellular stimuli. Despite the great significance of GPCRs in cell physiology and human health, structural information for this family of receptors is limited, and many essential details related to the mechanism of signal transduction and ligand specificity and selectivity are just beginning to emerge.

Recent breakthroughs in GPCR structural biology have been made possible by the progress in protein engineering, as well as the development and automation of crystallization technologies using lipidic matrices. Structures of 7 different GPCRs have been solved to date, 3 of which were captured in both inactive and active states.

Here we present recently determined structures of the human CXCR4 chemokine G protein-coupled receptor bound to a small molecule It1t and a cyclic peptide antagonist CVX-15 [1]; the structure of the human dopamine D3 receptor in complex with the antagonist eticlopride [2]; and the structure of the human adenosine A_{2A} receptor bound to an agonist UK-432097 [3]. The CXCR4 structures reveal a consistent set of receptor homodimers and provide insights into chemokine signaling and HIV-1 recognition. Structural details of the dopamine D3 receptor help us to better understand the pharmacological specificity between the dopamine D2 and D3 receptors. Comparison of A_{2A} structures bound to antagonist and agonist sheds light on the mechanism of GPCR activation.

The GPCR Network has been established to work with the GPCR community scientists interested in obtaining structural data on different receptors. Interested researchers should visit http://gpcr.scripps.edu for more information.

Supported by the NIH grants PSI:Biology U54 GM094618 (for structure production), Structural Biology Roadmap P50 GM073197 (for technology development) and R21 RR025336 (for development of pre-crystallization assays).

[1] B. Wu, E.Y. Chien, C.D. Mol, G. Fenalti, W. Liu, V. Katritch, R. Abagyan, A. Brooun, P. Wells, F.C. Bi, D.J. Hamel, P. Kuhn, T.M. Handel, V. Cherezov , R. C. Stevens *Science* **2010**, *330*, 1066-1071. [2] E.Y. Chien, W. Liu, Q. Zhao, V. Katritch, G.W. Han, M.A. Hanson, L. Shi, A.H. Newman, J.A. Javitch, V. Cherezov, R.C. Stevens *Science* **2010,** *330*, 1091-1095. [3] F. Xu, H. Wu, V. Katritch, G.W. Han, K.A. Jacobson, Z.-G. Gao, V. Cherezov, R.C. Stevens *Science* Published online **2011**, doi: 10.1126/science.1202793.

Keywords: receptor, signal, transduction

MS.57.3

Acta Cryst. (2011) A**67,** C132-C133

Application of split fluorescent proteins to challenges in crystallography: present and future

Pawel Listwan, Hau Nguyen, Thomas C. Terwilliger, Geoffrey S. Waldo, *Bioscience Division, Los Alamos National Laboratory, Los Alamos, New Mexico, 87544, (USA).* E-mail: plistwan@lanl.gov

The "GFP ToolBox" developed at LANL for the Protein Structure Initiative (PSI) includes spontaneously assembling engineered fragments of fluorescent proteins that can be used to tag and label proteins in living cells and cell extracts. These tools can be used to address challenges in protein expression, screening for complex formation, and crystallization. (1) Using actual examples from challenging multi-domain proteins, we show how this technology can be used in library screens to find soluble protein modules that are well-suited for crystallographic study. (2) We show how the technology can be used to screen for stable protein complexes that can be co-purified. (3) Finally we describe recent experiments and show a preliminary structure in which a small beta hairpin fragment of the GFP scaffold has been inserted into loops and turns of a target protein. The remainder of the GFP scaffold is added, binding to the displayed fragment and reconstituting the GFP barrel. This paves the way to a 'mix-and-