



E-MRS 2005 Spring Meeting
May 31 – June 3, 2005

SYMPOSIUM O

Synchrotron radiation and materials science

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E-MRS 2005 Spring Meeting

SYMPOSIUM O

Tuesday, May 31, 2005
Mardi 31 mai 2005

Morning
Matin

9:00

WELCOME
F. Boscherini

Session I : Nanostructures Session chair : T. Schmidt

O-I.01 9:05 -Invited-

TOMONAGA-LUTTINGER LIQUID STATE IN CARBON NANOTUBES: PHOTOEMISSION STUDY

H. Kataura, NRI, AIST, Tsukuba 305-8562, Japan, H. Shiozawa, IFW, Dresden, Germany, H. Ishii, Tokyo Metropolitan University, Tokyo 192-0397, Japan

Electronic structure of single-wall carbon nanotubes was investigated by high-resolution photoemission spectroscopy using synchrotron radiation as a light source. Oscillations in electronic density of states due to one-dimensional van Hove singularities were clearly observed near the Fermi level, which can be well reproduced by simple tight-binding calculation with assumptions a broadening and 100 meV energy shift probably due to a charge transfer from the metal substrate. Furthermore, in the vicinity of the Fermi level, two kinds of typical power-law dependences were observed. This can be regarded as evidence of Tomonaga-Luttinger liquid states. Both exponents show very similar values 0.46 and 0.48 good agreement with theoretical prediction. Photoemission spectra of single-wall carbon nanotubes encapsulating fullerenes were also measured. After a careful subtraction of the spectrum of empty nanotube, very sharp π -band spectra of fullerenes were obtained. This suggests a well isolated one-dimensional fullerene chain inside carbon nanotubes.

O-I.02 9:35

HIGHLY-ORDERED ULTRA-THIN COLLOIDAL NANOPARTICLE FILMS

J.I. Flege, Th. Schmidt, G. Alexe, T. Clausen, J. Falta, Institute of Solid State Physics, University of Bremen, Otto-Hahn-Allee, 28359 Bremen, Germany, I. Randjelovic, V. Aleksandrovic, A. Kornowski, H. Weller, Institute of Physical Chemistry, University of Hamburg, Grindelallee 117, 20146 Hamburg, Germany, S. Bernstorff, Sincrotrone Trieste, Strada Statale 14, km 163.5, 34012 Basovizza/Trieste, Italy

The preparation and investigation of ordered arrays of nanoparticles has attracted a tremendously increasing interest in the last years due to potential applications e. g. in optoelectronics, magnetic storage devices and catalysis. Here, we present an extensive study using grazing-incidence small-angle x-ray scattering (GISAXS), atomic force microscopy (AFM) and scanning electron microscopy (SEM) in order to characterize the structure and morphology of self-assembled metallic CoPt₃ nanoparticle films on silicon substrates. CoPt₃ nanoparticles of 6 nm in diameter were synthesized [1] with a well-defined size and shape surrounded by organic ligands. Subsequently, the nanoparticles were deposited using Langmuir-Blodgett (LB) and spin-coating (SC) techniques.

The GISAXS experiments were performed at the synchrotron light source ELETTRA using a CCD area detector to record the diffuse scattering pattern. A quantitative data analysis within the distorted-wave Born approximation reveals the formation of well-ordered self-assembled quantum dot superlattices exhibiting a local hexagonal symmetry, in very good agreement with the SEM results. Furthermore, details about the inter-particle distances and the particle-size distribution as functions of specific preparational parameters related to the LB [2] or SC technique are deduced. Moreover, AFM investigations corroborate the formation of nanoparticle monolayers. Additionally, the influence of different organic ligands on the ordering properties and the possibility of using polymeric buffer layers to modify the particle-substrate interaction are discussed.

[1] E. V. Shevchenko et al., J. Am. Chem. Soc. 124, 11480 (2002).

[2] J. I. Flege et al., Mat. Res. Soc. Proc. 2004, accepted.

O-I.03 9:55

CHEMICAL PROPERTIES OF NANOSTRUCTURES FABRICATED BY LOCAL ANODIC OXIDATION ON N-GAAS

G. Mori(a,b), M. Lazzarino(a), S. Heun(a), D. Ercolani(a,c), G. Biasiol(a), A. Locatelli(d), and L. Sorba(a,c), (a)Laboratorio Nazionale TASC-INFM, 34012 Trieste, Italy, (b)Università degli Studi di Trieste, 34127 Trieste, Italy, (c)Università degli Studi di Modena e Reggio Emilia, 41100 Modena, Italy, (d)Sincrotrone Trieste S. c. p. a., 34012 Trieste, Italy

Local anodic oxidation (LAO) is an effective tool for patterning the surface of a conductive sample with nanometer size features. The LAO-oxide formation takes place when in a humid atmosphere the tip of an AFM microscope is negatively biased with respect to the sample.

The chemical properties of the LAO nanostructures grown on n-GaAs have been investigated with microscopic X-ray photoemission spectroscopy (XPS). We found that the LAO-structures desorb under irradiation with soft X-rays (130 eV). We analyzed the desorption process by time-resolved photoelectron spectroscopy. We observed that even in the first stages of X-ray exposure the LAO-oxide is mainly composed of Ga_2O_3 , with a small fraction of Ga_2O_3 and As-oxides. The As-oxides are located only in the surface layers of the LAO-oxide where they account for 10 % of the oxide. Moreover, we found evidence for the presence of unoxidized GaAs embedded in the LAO-oxide. We studied LAO-oxides grown on a GaAs-AlAs layered structure to test the commonly accepted model for LAO process. The growth of the LAO-oxide inside the substrate is explained in terms of diffusion through the oxide of the OH- groups produced by the water electrolysis induced by the huge electric field (10^8 V/cm) present in the tip-sample junction. We recorded a strong Al signal in correspondence of the LAO structures while the Al signal is absent elsewhere. In order to explain this observation a more complicated dynamics of the oxide constitutes, beyond the simply OH- groups diffusion, should be considered.

O-I.04 10:15

STRUCTURAL CHARACTERIZATION OF ION IMPLANTED AU NANOCRYSTALS USING SYNCHROTRON BASED ANALYTICAL TECHNIQUES

P. Kluth, B. Johannessen, S.M. Kluth, Department of Electronic Materials Engineering, The Australian National University, Canberra ACT 0200, Australia, G.J. Foran, Australian Nuclear Science and Technology Organization, Menai, Australia, D.J. Cookson, Advanced Photon Source, Argonne National Laboratory, 9700 S. Cass Avenue, Argonne IL 60439, USA, M.C. Ridgway Department of Electronic Materials Engineering, The Australian National University, Canberra ACT 0200, Australia

Synchrotron based analytical techniques provide powerful tools for structural characterization of nanocrystalline materials. We have utilized extended x-ray absorption fine structure (EXAFS) spectroscopy and small-angle x-ray scattering (SAXS) combined with conventional analytical methods such as x-ray diffraction (XRD) and transmission electron microscopy (TEM) to investigate Au nanocrystals (NCs) formed in thin SiO_2 using ion implantation and annealing. Though the face-centred-cubic structure present in bulk material is retained for the NCs, EXAFS measurements reveal a significant bondlength contraction as a function of NC size [1]. Increased disorder was observed for NCs annealed below the viscosity temperature for SiO_2 and the melting temperature of Au, possibly related to stress induced plastic deformation of the NCs. We have also studied the structural evolution of NCs irradiated with ions subsequent to formation. In contrast to bulk elemental metals, which cannot be rendered amorphous by ion irradiation, EXAFS and XRD measurements of irradiated NCs indicate the loss of long range order consistent with amorphous material. SAXS measurements yield additional important information about NC size and shape both before and after ion irradiation. In particular the evolution of the size distribution during ion irradiation caused by collisional mixing was evaluated.

[1] P. Kluth, B. Johannessen, V. Giraud, A. Cheung, C. J. Glover, G. de M. Azevedo, G. J. Foran, and M. C. Ridgway, Appl. Phys. Lett. 85 (2004) 3561

10:35

BREAK

Session II : Magnetic nanostructures

Session chair : S. Cherifi

O-II.01 11:00 -Invited-

RECENT RESULTS IN TIME-RESOLVED XMCD-PEEM

Gerd Schönhense, Institut für Physik, Johannes Gutenberg-Univ., 55128 Mainz, Germany

Stroboscopic imaging using photoemission electron microscopy and X-ray magnetic circular dichroism (XMCD-PEEM) gives direct access to fast remagnetization processes. At high temporal field gradients ($>1\text{mT}/500\text{ps}$) the micromagnetic response on an external magnetic field pulse deviates substantially from the quasi-static behaviour. The fast formation of blocking domains (incoherent magnetization rotation [1]) and rapid coherent rotation of the domain magnetization [2] occur along with a field-induced magnetic flux traversing the microstructure.

High time resolution in the 15 ps-range became possible in the low-alpha (bunch compression) mode of BESSY II (Berlin) providing photon pulses of $< 2\text{ps}$ width. Domain images with small time increments revealed rapid oscillations of the visible position of domain boundaries. Micromagnetic simulations give evidence of the excitation of plane-wave like spin waves travelling perpendicular to the wall thus giving rise to an apparent oscillation of the wall position with a frequency in the 10 GHz range. The direction of magnetization inside the domains oscillates with large amplitudes in the non-linear regime at lower frequencies. Supported by BMBF (grants 05KS1UM1/5 and 05KS1BDA/9) and DFG (SPP 1133). [1] C. M. Schneider et al., Appl. Phys. Lett. 85 (2004) 2562 [2] D. Neeb et al., J. Phys.: Cond. Matt., 17 (2004) in print

O-II.02 11:30

SPIN AND ORBITAL MAGNETIC MOMENTS FROM SINGLE ATOMS TO SMALL CR CLUSTERS

C. Boeglin, H. Bulou, F. Scheurer, I. Chado, IPCMS, 67034 Strasbourg, France, P. Ohresser, LURE, 91405 Orsay, France, S.S. Dhesi, ESRF, BP 220, 38043 Grenoble, France, E. Gaudry, LMCP Jussieu, 75252 Paris, France, B. Lazarovits, T.U. 1060 Vienna, Austria
Reduced dimensionality systems are studied in order to trigger their magnetic properties. Interesting meta-stable magnetic states are sought by adjusting the structure and/or the morphology of the objects. In this class magnetic properties of Cr show highly atomic structure dependent properties. At room temperature Scanning Tunneling Microscopy, Low Electron Energy Diffraction and Auger Electron Spectroscopy show that Cr atoms show preferential nucleation at the kink sites on the reconstructed Au(111) surface forming monolayer high self-organized clusters. At low temperature (5 - 50 K) low surface diffusion provides in the very low coverage limit random atomic distribution of Cr on Au(111). We studied by means of X-ray Magnetic Circular Dichroism (XMCD) the magnetic properties of Cr isolated atoms, dimers and larger Cr clusters deposited on the Au(111) surface at various temperatures. The orbital and the effective spin magnetic moments of Cr are extracted as a function of the cluster size. We show that the atomic coordination of Cr is directly provided by the shape and fine structure of the XAS and XMCD spectra. Monte Carlo simulations performed as a function of coverage explain the Cr configuration and correlates the experimental magnetic spin moments to the Cr cluster sizes. We will discuss the effect of surface diffusion on the magnetic properties of Cr / Au(111).

O-II.03 11:50

EVOLUTION OF Co/Pt6COVERED NANOLINES UNDER MAGNETIC FIELD USING COHERENT SOFT X-RAYS RESONANT MAGNETIC SCATTERING

G. Beutier(a), F. Livet(b), A. Marty(a), G. van der Laan(c), S. Stanesco(d), V. Chamard(b), V. Baltz(e), (a)CEA-SP2M, 17 av. Des Martyrs, 38054 Grenoble cedex 9, France, (b)LTPCM-INPG, ENSEEG-Domaine Universitaire, BP 75, 38402 Saint-Martin d'Hères Cedex, France, (c)Magnetic Spectroscopy Group, Daresbury Laboratory, Warrington WA4 4AD, U.K., (d)ESRF, Grenoble, France, (e)CEA-Spintec, 17 av. Des Martyrs, 38054 Grenoble cedex 9, France

We report on the soft x-ray resonant magnetic scattering (SXRMS) from etched lines in silicon covered by a Co/Pt multilayer with perpendicular magnetization. A similar system was previously studied with incoherent [1] and coherent [2] radiation. Using a 10- μ m diameter pinhole at 7 mm in front of the sample, we increased the transverse coherence of the incident x-rays while illuminating only a small sample area. These conditions allow us to obtain a speckle pattern by using a CCD camera [3]. The experiment was performed in reflection geometry on beamline ID8 at the ESRF.

The speckle pattern is related to the local magnetic configuration of the lines that are illuminated by the beam through the pinhole. Using a pulsed perpendicular magnetic field, we studied the reversal of the lines through the changes in the speckle pattern [4]. The comparison between major and minor magnetization loops shows that saturating the sample erases its magnetic memory, whereas a smaller field does not prevent to retrieve the original magnetic state. The main challenge arising from magnetic scattering using coherent x-rays is the reconstruction of the real-space magnetic configuration within the illuminated area. Important results have been obtained recently in transmission geometry, using a holographic method [5]. In the non holographic case, phase retrieval algorithms must be used. For this purpose, our system presents several advantages. A monte carlo algorithm will be presented and its first results discussed.

[1] Chesnel et al., Phys. Rev. B 66, 024435 (2002).

[2] Chesnel et al., Phys. Rev. B. 70, 180402(R) (2004).

[3] Chesnel et al., Phys. Rev. B 66, 172404 (2002).

[4] Pierce et al., Phys Rev. Lett. 90, 175502 (2003).

[5] Eisebitt et al., Nature 432, 885 (2004).

O-II.04 12:10

THREE-DIMENSIONAL MAGNETIZATION STRUCTURES IN Fe/Mo(110) ISLANDS

R. Hertel, IEE-IFF, Research Center Jülich, 52425 Jülich, Germany and O. Fruchart and S. Cherifi, Laboratoire Louis Néel, CNRS Grenoble, BP166, 38042 Grenoble Cedex 9, France, and P.-O. Jubert, IBM Zürich Research Laboratory, 8803 Rüschlikon, Switzerland and A. Locatelli, Sincrotrone Elettra, Basovizza, 34012 Trieste, Italy and S. Heun, TASC-INFM Laboratory, Basovizza, 34012 Trieste, Italy

Contrary to magnetic thin-film elements, not much is known about the magnetization distribution in mesoscopic three-dimensional (3D) magnetic particles. We have investigated 3D magnetization structures in numerous mesoscopic Fe/Mo(110) islands by means of x-ray magnetic circular dichroism combined with photoemission electron microscopy (XMCD-PEEM). The self-organized growth yields particles of characteristic hexagonal shape with a size of up to about 2.5 μ m and thickness of up to 250 nm. The XMCD-PEEM studies reveal unexpected, asymmetric magnetization distributions at the surface. Micromagnetic simulations are in excellent agreement with the observed magnetic structures and provide information on the internal structure of the magnetization which is not accessible in the experiment. It is shown that the magnetization is influenced mostly by the particle size and thickness rather than by its shape. Hence, the hexagonal samples can be regarded as model systems for the study of the magnetization in thick, mesoscopic ferromagnets.

12:30

LUNCH

Tuesday, May 31, 2005
Mardi 31 mai 2005

Afternoon
Après-midi

Session III : Semiconductors
Session chair : P. Fons

O-III.01 14:00 -Invited-

IN-SITU STUDIES OF SEMICONDUCTOR GROWTH BY SYNCHROTRON X-RAY DIFFRACTION

Klaus H. Ploog and **Wolfgang Braun**, Paul Drude Institute for Solid State Electronics, 10117 Berlin, Germany

We established an experimental setup at BESSY to study surfaces and interfaces of epitaxial layers during MBE using x-ray diffraction. The advantages compared to RHEED are (i) the penetration deep into the layers to study also properties and interfaces, and (ii) the potential to analyse the results using x-ray theory. We first present our kinetic studies of homoepitaxy on GaAs(001), InAs(001), and GaSb(001). The kinetics of the reconstruction domains on GaAs is independent of the kinetics governing the morphological island evolution. GaAs exhibits a unique recovery behavior with linear coarsening of the islands/pits as a function of time. The coarsening process consists of 2 stages, the annihilation of islands/pits on different levels until a 2 level system is formed, followed by lateral coarsening. Despite their similarity, InAs and GaSb differ significantly from GaAs in their growth and coarsening behavior. Whereas InAs shows a coarsening exponent of $n=0.5$, the coarsening is frozen on GaSb for substrate temperatures required for layer-by-layer growth. Second, we discuss the heteroepitaxy of hexagonal MnAs on cubic GaAs(001) where a distinct interplay between film and substrate structure at the interface occurs. At appropriate conditions, MnAs grows with the c-axis of the film along the [110] azimuth of the substrate. Growth proceeds in the Volmer-Weber mode with initially small, disconnected islands that undergo rapid strain relaxation until they coalesce. At the interface, the system develops an array of misfit dislocations with a distinct periodicity along the a-axis (visible by additional superstructure peaks). Along the orthogonal c-axis, the interaction with the substrate is weaker, and a coincidence lattice with two different periods is formed.

O-III.02 14:30

STRAIN, SIZE AND COMPOSITION OF SEMICONDUCTOR NANOSTRUCTURES DETERMINED VIA X-RAY ANOMALOUS DIFFRACTION AND DIFFRACTION ANOMALOUS FINE STRUCTURE

A. Letoublon, V. Favre-Nicolin, H. Renevier, Commissariat à l'Energie Atomique, Département de Recherche Fondamentale sur la Matière Condensée, SP2M/Nanostructures et Rayonnement Synchrotron, 17 rue des martyrs, 38054 Grenoble Cedex 9, France, M.G. Proietti, Departamento de Física de la Materia Condensada, Instituto de Ciencia de Materiales de Aragón, CSIC-Universidad de Zaragoza - c. Pedro Cerbuna 12, 50009 Zaragoza, Spain, C. Monat, M. Gendry, LEOM, UMR-CNRS 5512, Ecole Centrale de Lyon, 69134 Ecully, France, O. Marty, LENAC, Université Lyon I, 69621 Villeurbanne, France, C. Priester, Institut d'Electronique, de Microélectronique et de Nanotechnologie, dep. ISEN, 59652 Villeneuve d'Ascq, France

Self-assembled nanostructured materials are one of the very important topics in materials science nowadays. In this contribution we show that anomalous diffraction and Diffraction Anomalous Fine Structure (DAFS) are determinant to recover structural properties of the nano-objects, such as strain, size and composition.

We report on the study of encapsulated semiconductor nanostructures: Quantum Sticks (Qs) of InAs/InP and GaN/AlN Quantum Dots (QDs).

In the case of Qs, we show that the partial structure factor of As atoms (FAs) that belong to the InAs stick-like nanostructures (Quantum Sticks), can be directly recovered by means of grazing incidence anomalous x-ray diffraction. It gives access to the QDs size and the average strain. Fixed-Q anomalous diffraction, measured as a function of the energy, at the As K-edge, in grazing incidence, provides a clear evidence of pure InAs Qs.

Further information about composition and strain accommodation can be obtained by quantitative analysis of the DAFS oscillations above the edge. The data analysis shows that the local structure values are in agreement with strained InAs.

We also applied grazing incidence anomalous diffraction and DAFS to GaN quantum dots embedded in GaN/AlN multilayer. The goal is to recovering the size and strain accommodation of the GaN quantum dots as a function of the number of GaN/AlN layer and the spacer thickness.

In summary, we show that application of anomalous diffraction and DAFS together with reciprocal space mapping provide a new breakthrough on nanostructures structural properties.

[1] A. Létoublon, V. Favre-Nicolin, H. Renevier, M.G. Proietti, O. Marty, C. Monat, M. Gendry, C. Priester, Phys. Rev. Lett., 92, (2004), 186101.

O-III.03 14:50**SHAPE, STRAIN, AND ORDERING OF LATERAL INAS QUANTUM DOT MOLECULES**

B. Krause and T.H. Metzger, ESRF, BP 220, 38043 Grenoble Cedex, France, A. Rastelli, R. Songmuang and O.G. Schmidt, MPI-FKF, Heisenbergstr. 1, 70569 Stuttgart, Germany

Semiconductor quantum dots have received much interest due to their unique structural and electronic properties. They can be thought of as artificial atoms since their electrons are confined in 3 dimensions. In analogy to the natural hierarchy in a solid (from the single atom to the molecule, and finally to the crystal), the complexity of a quantum dot system can be tailored from single quantum dots to molecules, and finally to 3D quantum dot crystal. For InGaAs and InAs on GaAs(100), it has been shown that lateral quantum dot molecules, i.e. closely placed groups of quantum dots, can be produced by molecular beam epitaxy exploiting a self-organization process: the quantum dots nucleate preferentially around nanoholes in the substrate.

Here, the results of a grazing incidence X-ray diffraction (GID) study on self-assembled InAs/GaAs quantum dots are presented. GID gives information about the strain, the shape, the lateral ordering, and the composition of the quantum dots. The samples studied here are increasingly complex, ranging from statistically distributed single quantum dots over ordered and oriented arrangements of 2 quantum dots (bimolecules) up to 4 quantum dots (quadmolecules). Their shape, the lattice parameter distribution and the composition are quantified.

O-III.04 15:10**MORPHOLOGY AND COMPOSITION OF Ge ISLANDS EMBEDDED IN Si**

F. d'Acapito(a), M. de Seta(b), L. di Gaspare(b), G. Capellini(c), M. Giorgi(b), (a)GILDA CRG, INFN - OGG c/o ESRF; (b)Universita' Roma Tre; (c)INFN and Universita' Roma Tre, Italy

Ge on Si self assembled islands have attracted a wide interest since they could potentially be applied as self-assembled quantum dots in optoelectronic. Because the optical and electronic properties of the resulting devices depend strongly on the island shape and composition the study of these island characteristic is of paramount importance. In this work the morphology and composition of Ge islands on Si(001) substrate during the capping with a silicon layer were investigated by means of Atomic Force Microscopy (AFM) and X ray Absorption Fine Structure (XAFS).

The samples were deposited at 750°C by using the Chemical Vapor Deposition technique. AFM images of the Ge island layer show that before the silicon capping most of the islands are strained-dome shaped. In the early stage of the capping the islands clearly show a dramatic flattening with an increasing of the island base and a simultaneous height reduction, transforming progressively into square-based pyramids. After 45 s growth time the Si cap layer start to cover the islands and after 180 s a planar surface is established. The observed island shape changes are induced by a strong strain driven SiGe intermixing enhanced by the high growth temperatures, as confirmed by an XAFS analysis at the Ge-K edge. XAFS data show a Si45Ge55 alloy prior the Si deposition and an increasing Si content up to Si70Ge30 obtained after a 45 s growth. For longer depositions the composition saturates at this level. This can be interpreted with the reduced Ge-Si diffusion in the bulk with respect to the surface diffusion of the same elements.

O-III.05 15:30**IN-SITU INVESTIGATION BY GISAXS AND GID OF THE GROWTH MODE, STRAIN STATE AND SHAPE OF Ge ISLANDS DURING THEIR GROWTH ON Si(001)**

M.-I. Richard(a), T.-U. Schüllli(a), E. Wintersberger(b), G. Renaud(a), G. Bauer(b), (a)CEA-Grenoble, DRFMC/SP2M/NRS, 17 av. des Martyrs, 38054 Grenoble, France, (b)Johannes Kepler Universität Linz, Institut für Halbleiterphysik, 4040 Linz, Austria

We used a combination of grazing incidence small angle scattering (GISAXS) and X-ray diffraction (GID) measurements to analyze the growth mode of Ge on Si(001). The measurements were performed on beamline BM32 at the ESRF.

The deposition was followed monolayer (ML) by monolayer. GISAXS measurements provide the detailed evolution of the shape of the grown QDs. GID allows monitoring the island nucleation by the beginning of lattice relaxation. The 2x1 surface reconstruction was also monitored by GID and in-plane reciprocal space maps were recorded. These last GID measurements allow tracking the 2D-3D transition of the Stranski-Krastanow growth. Layer-by-layer growth is observed for up to 3 deposited MLs at 600°C. Then, a variation of the relaxation behaviour between 4 and 5 deposited MLs reveals a change of the island morphology. At 5 ML, rods of scattering by {113}-type facets appear in the GISAXS images. For increasing deposition, these streaks get narrower, indicating an increase in size of these facets. Recording GISAXS for several azimuths also shows the appearance of {15 3 23} facets, which confirm recently published STM results. The diffusion of the wetting-layer into the islands has also been studied by GID. For each added monolayer, the intensity scattered by the partially relaxed Ge islands was integrated over all three directions of the reciprocal space maps. After the onset of islands formation, this intensity should just increase linearly with the deposited amount, if no material from the Ge wetting layer is transformed into 3D islands. For a deposition temperature of 550°C, a deviation from this linear behaviour was observed, which shows that the equivalent of about one atomic layer is transported from the 4 ML thick wetting layer into the islands.

15:50

BREAK

Session IV : Semiconductors and optical materials

Session chair : F. D'Acapito

O-IV.01 16:20 -Invited-

NANOSCALE MECHANISM OF PHASE-CHANGE OPTICAL RECORDING REVEALED BY XAFS

A.V. Kolobov(a), P. Fons(a), J. Tominaga(a), A.I. Frenkel(b), A.L. Ankudinov(c), T. Uruga(d), (a)Center for Applied Near-field Optics Research, National Institute of Advanced Industrial Science and Technology, Japan, (b)Department of Physics, Washington University, USA, (c)Department of Physics, Yeshiva University, USA, (d)Spring8, Japan

Recent advances in multimedia require new faster and denser recording media. One of the most promising media types to date is phase-change materials when bits of information are recorded as amorphous marks against a crystalline background. The materials used are Ge₂Sb₂Te₅ (in DVD-RAM) and a Ag-In-Sb-Te alloys (in DVD-RW). Commercially available re-writable DVDs ensure repeatable switching over a million cycles. Strangely, the details of the structural changes behind the utilized transition are unknown.

To investigate the structure of the media in both crystalline and laser-amorphized states we have performed XAFS experiments. To ensure better statistics and maximum confidence in the results, concurrent three-edge data analysis has been performed. Our results demonstrate that Ge₂Sb₂Te₅, the material of choice in DVD-RAM, does not possess the rocksalt structure, as generally believed, but is distorted with the Ge and Sb atoms being off centre. The laser-induced amorphization results in drastic shortening of covalent bonds and a decrease in the mean-square relative displacement, demonstrating a substantial increase in the degree of short-range ordering, in sharp contrast to amorphization of covalently bonded solids. EXAFS and XANES analysis lead us to a conclusion that this novel order-disorder transition is due to a reversible flip of Ge atoms within the Te fcc sublattice from octahedral to tetrahedral symmetry sites without rupture of strong covalent bonds [1]. Our results demonstrate the importance of synchrotron radiation for modern material science.

[1] A.V. kolobov et al., Nature Mater. 3 (2004) 703

O-IV.02 16:50 -Invited-

PHOTOEMISSION SPECTROMICROSCOPY INVESTIGATION OF THE MECHANISM OF DARK-SPOT DEGRADATION OF ORGANIC LIGHT-EMITTING DEVICES

Luca Gregoratti(a), P. Melpignano(b), A. Baron-Toaldo(b), V. Biondo(b), S. Priante(b), R. Zamboni(c), M. Murgia(c), S. Caria(c), A. Barinov(a), M. Kiskinova(a) (a)Sincrotrone Trieste ScPA, Area Science Park, SS14-Km163.5, 34012 Basovizza-Trieste, Italy, (b)Centro Ricerche Plast-Optica, Via Jacopo Linussio, 1, 33020 Amaro (UD), Italy, (c)Istituto per lo Studio dei Materiali Nanostrutturati del CNR, via P. Gobetti 101, 40129 Bologna, Italy

With respect to the other photoelectron microscopy techniques a Scanning PhotoEmission Microscope (SPEM) uses the most direct approach to photoelectron spectromicroscopy which is the use of a small focused photon probe to illuminate the surface. The SPEM at the Elettra synchrotron light source can operate in two modes: imaging and spectroscopy. In the first mode the sample surface is mapped by synchronized-scanning the sample with respect to the focused photon beam and collecting photoelectrons with a selected kinetic energy. The second mode is photoelectron spectroscopy from a microspot. The SPEM on the ESCAmicroscopy beamline at Elettra has a lateral resolution of 150 nm; and an overall energy resolution which is now better than 200 meV. Utilization of Organic Light Emitting Devices (OLEDs) as low-cost and high efficiency flat displays and illuminators has attracted the interest of optoelectronics industries in the last decade. A major obstacle for vast commercialisation is their relatively short lifetime. This synchrotron study focuses the investigation on one of the most evident failures of an organic LED, the formation of dark spots. The OLEDs were operated in air or in UHV with voltages up to 32 V, in order to provoke faster degradation. The changes in morphology of the initially smooth Al cathode surface during operation evidence a creation of 'domelike' structures, followed by local disruption of the Al cathode, exposing micro-areas of the ITO anode and the organic layer. The images taken at the energies of the different chemical species and micro-spot spectra identify a release of volatile In-, Sn- and C-containing species, including metallic In, clear evidence that the degradation is driven by local decomposition of the anode/organic interface.

O-IV.03 17:20

STUDY OF THE MODIFICATIONS OF THE MICROSTRUCTURE OF GaN AFTER HIGH-DOSE Si IMPLANTATION

M. Katsikini, F. Pinakidou, E.C. Paloura, Aristotle University of Thessaloniki, Department of Physics, 54124 Thessaloniki, Greece and F. Boscherini University of Bologna, Department of Physics and Istituto Nazionale per la Fisica della Materia, 40127, Bologna, Italy

The microstructure of epitaxially grown GaN, that was subjected to Si implantation, was studied using X-ray absorption fine structure (XAFS) spectroscopy. The implantation dose was $1 \times 10^{18} \text{ cm}^{-2}$, which is high enough to cause amorphization of the sample. The energy of the implanted ions was 100keV resulting to a damaged region with a depth of about 200nm, which is higher than the information depth of the N-K-edge XAFS spectra recorded at the BEAR beamline of ELETTRA, Trieste. Crystalline GaN and amorphous Si₃N₄ were used as reference compounds. The short-range-order of the sample can be deduced from the Fourier Transform of the $k^3(k)$ spectrum. After ion implantation the N-Ga distance increases while N-Si bonds are also detected. Although, the absence of long or mid-range order is also obvious in the NEXAFS spectra, the presence of a resonance line, with small width at half maximum, that appears at about 1eV above the absorption edge, indicates the existence of defects strongly localized on the N atoms, most probably N-dangling bonds. The NEXAFS spectra are compared with simulations using the FEFF program.

O-IV.04 17:40**SEGREGATION OF Si DOPANT SPECIES IN GaN REVEALED BY SPECTRO-MICROSCOPY**

Th. Schmidt, M. Siebert, S. Gangopadhyay, A. Pretorius, S. Figge, S. Einfeldt, D. Hommel, and J. Falta, Institute of Solid State Physics, University of Bremen, Otto-Hahn-Allee 1, 28359 Bremen, Germany, L. Gregoratti, A. Barinov, and M. Kiskinova, ELETTRA Synchrotron Light Source, Strada Statale 14, 34012 Basovizza, Italy

GaN based structures have a great application potential in electronic and opto-electronic devices, e. g. in blue light emitting diodes and laser diodes. In this context, doping of the nitride material is indispensable. Most commonly, Mg and Si are used for p-type and n-type doping, respectively. Large Mg concentrations are known to lead to defect formation in GaN [1], which is attributed to a strong surface segregation tendency [2]. No such dramatic effects have been reported for Si so far. Si-doped GaN(0001) films of about 3 microns thickness were grown by metal-organic vapor pressure epitaxy on sapphire substrates. ESCA microscopy was performed at the ELETTRA spectro-microscopy beamline, on samples with different Si dopant concentrations. From the contrast observed in images recorded at several core level energies, the presence of steep facets is proven which form elongated cracks running in high-symmetry crystal directions. Since the incorporation of Si is known to result in tensile stress [3], such cracks could be considered a strain relaxing mechanism. In addition to the morphological information, the investigation of the Si 2p and Ga 3p signals clearly show that there is a strong Si enrichment at the crack facets. Together with our observation that the density of these cracks is almost independent of the dopant concentration and thus on the strain imposed on the GaN films, it seems more likely that Si incorporation at the crack facets is energetically favoured and the facets are stabilized by Si segregation.

O-IV.05 18:20**SOFT X-RAY XAFS OF N IN ZnO:N -- WHY IS DOPING SO DIFFICULT?**

P. Fons(a), H. Tampo(a), F. Boscherini(b), R. Carboni(b), M. Ohkubo(a), F.S. Niki(a), S. Friedrich(b), (a)National Institutes of Advanced Industrial Science & Technology, Tsukuba 305-8562, Japan, (b)University of Bologna, 40127 Bologna, Italy

In the last few years, much research effort has been directed towards ZnO based optoelectronics. ZnO is a 3.4 eV direct bandgap semiconductor that holds great promise for optoelectronics applications due in part to its large 60 meV excitonic binding energy as well as the availability of bulk substrates. The key problem now preventing widespread application of ZnO to device applications is the difficulty in reliably doping ZnO p-type.

One of the most promising candidates for p-type doping of ZnO is thought to be Nitrogen (N), however, reliable p-type doping using N has yet to be achieved. ZnO is naturally n-type due to the presence of intrinsic defects and this in conjunction with the large difference in mobilities (hole mobility is about a factor of 100 less than electron mobility), use of Hall effect measurements alone to optimize doping conditions has proven difficult.

We have applied soft x-ray XAFS at the N K-edge to directly investigate the local atomic environment of N in the ZnO lattice as a function of processing conditions. Samples were grown by molecular beam epitaxy using a N RF-radical source for nitrogen. Near-edge XAFS (XANES) data were taken at Elettra using the undulator beamline ALOISA and a Ge solid state detector as well as a superconducting detector employed at the Advanced Light Source at LLBL. In conjunction with first-principle calculations using feff 8, we have determined that while for low temperature, metastable growth conditions, N substitutes onto oxygen sites, thermal processing results in the formation of molecular nitrogen and thus effectively passivates the dopant effect of N. Optimization of growth conditions to enhance the doping effectiveness of N will be discussed.

Wednesday, June 1, 2005
Mercredi 1er juin 2005

Afternoon
Après-midi

Session V : Mesoporous materials
Session chair : F. Schüth

O-V.01 14:00 -Invited-

TIME-RESOLVED X-RAY DIFFRACTION EXPERIMENTS ON MESOPOROUS THIN FILMS

P-A. Albouy, Laboratoire de Physique des Solides, Bât.510, Université de Paris-Sud, 91405 Orsay, France

The synthesis of mesostructured materials involves the formation of organic-inorganic composites, whereby the organic phase is organised on a mesoscopic scale and serves as a template for the inorganic skeleton. A large variety of organic surfactants or metal oxide precursors can be used, and both the composition of the starting solution and the conditions for material formation determine the final structural organisation. We presently focus on thin-films as obtained by the dip-coating process (evaporation-induced self-assembly). As a result of the rapid film formation, it is impossible to obtain time-resolved structural information with conventional X-ray equipment. For this reason, measurements were conducted at the Austrian SAXS beam line of the synchrotron Elettra, using two specially built apparatus: First a dip-coater that allows SAXS patterns to be taken during film formation. Measurement time ranges from 100ms (linear detector) to 1s (CCD camera), to compare with a time-scale for film formation of the order of 1'. Transient phases were revealed in some cases and the influence of the deposition conditions (withdrawing speed, humidity and temperature) could be real-time checked. Secondly, the final film treatment involves removal of the organic matrix and intensive condensation of the metal oxide walls by heating. We used a small oven (max. temperature : 900°C ; adjustable heating path) that allows simultaneous SAXS and WAXS measurements. The evolution of the mesostructure (shrinkage, deterioration) and the occurrence of crystallisation within the oxide matrix were monitored.

O-V.02 14:30

SYNCHROTRON MICRO-SAXS AND MICRO-DIFFRACTION SCANNING TECHNIQUES FOR THE STUDY OF COMPLEX (BIO-)MATERIALS

H.C. Lichtenegger, Institute for Materials Science & Technology, Vienna University of Technology, Favoritenstr. 9-11, 1040 Wien, Austria

The study of complex materials such as hierarchically structured biomaterials usually requires the combination of several investigation methods. Position-resolved synchrotron microfocus techniques are non-destructive and can be combined to map the structure of complex materials such as wood, teeth and artificial nanostructures in detail. The following examples shall illustrate the potential of such combinations: -Microdiffraction-mapping of single wood cells: imaging of the helical orientation of cellulose fibrils (ESRF, Grenoble) 1. - combination of micro-SAXS and WAXD on worm teeth: reinforcement of teeth with copper-mineralized fibers (APS, Argonne) 2. - x-ray fluorescence and microdiffraction mapping on worm teeth: mapping crystallinity and local texture (APS, Argonne). -SAXS on mesostructured surfactant-water mixtures: preferential orientation of liquid-crystalline domains at capillary surface (Elettra, Trieste) 3.

[1] H. Lichtenegger, M. Müller, O. Paris, C. Riekel, P. Fratzl, J. Appl. Crystallogr. 1999, 32, 1127.

[2] H. C. Lichtenegger, T. Schöberl, M. H. Bartl, J. H. Waite, G. D. Stucky, Science 2002, 298, 389.

[3] C. Fritscher, S. Bernstorff, N. Hüsing, D. Brandhuber, T. Koch, S. Seidler, H. C. Lichtenegger, J. Synchrotr. Radiat. 2005, in print.

O-V.03 14:50

NUCLEATION AND GROWTH OF MESOPOROUS SPHERICAL SILICA NANOPARTICLES STUDIED BY TIME-RESOLVED SMALL ANGLE X-RAY SCATTERING

A. Hahn(a), D. Kumar(a), G. Büchel(c), H. Amenitsch(b), T. Narayanan(c), K.K. Unger(a), (a)Institut für Anorganische Chemie und Analytische Chemie, Johannes Gutenberg-Universität, Duesbergweg 10-14, 55128 Mainz, Germany, (b)Institute of Biophysics and X-ray Structure Research, Austrian Academy of Sciences, Schmiedlstr. 6, 8043 Graz, Austria, (c)ESRF, BP 220, 38043 Grenoble Cedex, France

Porous spherical silica particles with a mean diameter between 100 nm and 2 µm were synthesized in our group via modified Stöber processes [1,2].

Previous investigations to elucidate the particle formation mechanisms by conventional off-line methods failed and gave only limited insight into the growth mechanisms.

This paper reports on attempts to monitor the nucleation process from the formation of the first primary particles up to the final particles via in-situ time resolved ultra small angle X-ray scattering experiments performed at the ID2 (ESRF, Grenoble). Various synthesis procedures leading to particles up to 2 µm were investigated at different temperatures using a flow-through-cell. The particle growth was recorded using a CCD-camera and also with a Bonse-Hart-camera setup.

Using the Bonse-Hart-camera setup it was possible to observe and measure the growth of the main silica particles up to the final size. One synthesis procedure showed during the whole course of the growth process nanoparticles with a constant diameter of 22 nm providing evidence for a growth process via an agglomeration mechanism. All experiments clearly show that there is a constant number of growing particles throughout the whole growth process until the final particle size is reached.

This indicates a nearly instantaneous formation of the nuclei in the first seconds of the reaction followed by an agglomeration of nanoparticles, with no further formation of nuclei. Finally, the variation of reaction temperatures showed the influence on the particle size distribution during the reaction, providing valuable information for improvements on the synthesis procedure

[1]C. Kaiser et al., Proceedings of the COPS-IV symposium, Bath, UK (1997) 406

[2]G. Büchel et al., J. Supramol. Sci., 5 (1998) 253

O-V.04 15:10

IN SITU SAXS/XRD STUDIES OF THE FORMATION OF MESOSCOPICALLY ORDERED SURFACTANT-SILICA MESOPHASES

Mika Lindè(a), Cilaine V. Teixeira(a), Heinz Amenitsch(b), Viveka Alfredsson(c), Freddy Kleitz(d), (a)Dept. Phys. Chem., Åbo Akademi University, Porthansgatan 3-5, 20500 Turku, Finland, (b)Austrian Academy of Science, Schmiedlstrasse 6, 8043 Graz, Austria, (c)Phys. Chem. 1, Lund University, 221 00 Lund, Sweden, (d)Dept. Heterogeneous Catalysis, Max-Planck Institute for Coal Research, Kaiser-Wilhelm-Platz 1, 45470 Mülheim/Ruhr, Germany The formation of a variety of different surfactant-silicate mesophases has been studied by in situ synchrotron SAXS/XRD. The syntheses were carried out having the surfactant, the pH of the solution and the temperature as parameters, resulting in the formation of hybrid materials having a 2D hexagonal or one of several cubic structures. Qualitative data analysis of both the scattering and diffraction patterns will be presented, which allows the different stages of the formation to be described in some detail. The results are compared with literature data (if available) obtained with complementary techniques. It is shown that the formation mechanism is drastically different depending on the initial structure of the surfactant aggregates in solution. Thus, new synthesis approaches can be foreseen that allow the structure of the final hybrid mesophase to be rationally controlled.

O-V.05 15:30

IN SITU SAXS WAXS INVESTIGATIONS OF CRYSTALLISATION IN METAL OXIDE MESOPOROUS ORDERED THIN LAYERS

D. Grosso(a), C. Boissiere(a), P.A. Albouy(b), H. Amenitsch(c), and C. Sanchez(a), (a)Chimie de la Matière Condensée, UMR UPMC-CNRS 7574, 4 place Jussieu, 75252 Paris 05, France, (b)Laboratoire de Physique des Solides, Université Paris-Sud, 91405 Orsay, France, (c)Institute of Biophysics and X-ray Structure Research, Austrian Academy of Sciences, Steyrergasse 17/VI, 8010 Graz, Austria

Mesoporous crystalline metal oxide thin layers combine the physicochemical properties of nanoscale crystalline particles with the homogeneity of mesostructured porosity into coatings, which is one of the preferential shapes used for integration into high technology devices. Therefore, these layers additionally require a perfect tuning of particle and pore respective dimensions in the desired mesoorder. An in situ time-resolved SAXS - WAXS experiment, adapted to the 8KeV Elettra SAXS beam line, was used to assess the various phenomena taking place during thermal treatment and crystallisation of cubic-derived mesoporous photoactive Anatase TiO₂ and tetragonal MgTa₂O₆, spintronic Illmenite CoTiO₃, and ferroelectric Perovskite SrTiO₃. Because of the very low quantity of materials, the high flux only available in third generation synchrotron facilities is required. The geometry used for such simultaneous low and wide angles analyses of thin films is described in details. We show that depending on the heating regime, ordered mesoporous nanocrystalline frameworks can be obtained through a rigorous control of the various steps involved in the stabilisation of the inorganic network (i.e. dehydration, template thermal decomposition, inorganic nucleation, growth and diffusive sintering). We also show that the thermal treatment induces a porosity opening with the formation of an exotic grid like mesostructure.

15:50

BREAK

Session VI : Cultural heritage materials (joint session with symposium Q)

Session chair : M. Menu

O-VI.01 16:20 -Invited-

APPLICATIONS OF SYNCHROTRON RADIOATION TO ARCHAEOLOGY

Mario Vendrell, Dept. Crystallography and Mineralogy, University of Barcelona, Spain

Since the beginning of the application of experimental research to the ancient materials, the accuracy of the results has been improved following the challenges of the needs arising from the study of such materials. Thus, deeper information has been obtained in order to better know the techniques used to built-up or produce a particular material, how and why ancient goods were used, who used them, when, etc.

From the very beginning, the scientific equipments also improved their accuracy while the amount of sample need were reduced in size and/or weight. This open a wide range of opportunities to the research of ancient materials which can be deeper studied and analyzed.

A significative step in this evolution of the experimental equipments has been the development of synchrotron radiation which allows a high brilliance and intensity of light to be applied as radiation source to any of the already known experimental techniques such as X-ray diffraction, infrared spectroscopy, X-ray fluorescence, imaging, among others. This allows to get information of very small samples, to obtain data of very small parts of a sample, to go deeper in the information, including to map the spatial distribution of data of very small thin films, samples, sections or whatever the sample were prepared.

Several examples of SR applications to cultural heritage will be shown, basically on the fields of ancient ceramics (lustre decorated Islamic pottery) and ancient paintings. In the examples proposed, the problem to be solved will be identified, as well as the limitations found using classical experimental equipments and the results obtained with SR.

O-VI.02 17:00

COPPER NANO-CLUSTERS IN ANCIENT OPAQUE GLASS FROM POMPEII AND SICILY: A XAS STUDY

R. Arletti, G. Vezzalini, Earth Science Department, L.go S. Eufemia 19 41100 Modena, Italy, and S. Quartieri, G. Sabatino, M. Triscari, Earth Science Department, Salita Sperone 31, 98166 Messina S. Agata, Italy and M.C. Dalconi, Earth Science Department, Corso Ercole I° d'Este, 44100, Ferrara, Italy

In recent years, the opaque and coloured archaeological glass has been the object of considerable interest. Nevertheless, some aspects of the ancient production technology are still almost unknown. The aim of this work was to investigate the configuration and the oxidation state of copper, and its influence on colour and opacity, in red and green opaque Roman glass (mosaic tesserae, game counters, and glass artefacts) from Sicily and Pompeii excavations. High-resolution Cu-K edge XANES and EXAFS spectra were collected at the GILDA beamline (ESRF, Grenoble). The detailed data analysis allowed us to conclude that, in red samples, copper is mainly present as metallic nano-clusters, accompanied by minor amounts of Cu⁺ coordinated to the oxygen atoms of the glass framework. These clusters are responsible for both colour and opacity of the glass. It is noteworthy that similar metallic nano-clusters were also found in Renaissance luster decorations. In green samples both Cu⁺ and Cu²⁺ were detected, both incorporated in the glass matrix, the second being responsible for the colour of the artefact.

O-VI.03 17:20

MODELLING THE SIZE OF RED-COLOURING COPPER NANOCCLUSERS IN ARCHAEOLOGIC GLASS BEADS

M.O. Figueiredo, J.P. Veiga, CENIMAT, Fac. Sci. Technol., New Univ. Lisbon, 2829-516 Caparica, and J.P. Mirão, Geophysics C., Évora Univ., 7002-554 Évora, Portugal

Ancient red glasses have been studied to ascertain the origin of colour, attributed either to the presence of both copper clusters and cuprous oxide in Celtic enamels and Gallo-Roman tesserae or to copper alone in Satsuma glasses. A study on valence state and local atomic structure of copper implanted in silica glass assigned the presence of Cu⁰ and Cu^I species. Therefore, the red colour of opaque ancient glasses still calls for further study.

X-ray absorption experiments at Cu K-edge were conducted at the ESRF beam line ID22 on the red layer in a "rosette"-type bead found during excavations at Lisbon Old-City and on selected model compounds (metallic copper and the red oxide, Cu₂O - the mineral cuprite, with linear Cu-O linkage). To model Cu 1s XANES spectra, a multiple scattering formalism was applied using the FEFF code. A hypothetical tetragonal structure was postulated for Cu₂O to release constraints from linear O-Cu-O bonds. Assuming clusters of 42 atoms in metallic copper and 56 atoms in modified cuprite, the general trend of calculated spectra compare well to experimental data collected from the red glass. A full discussion is presented on the obtained results.

O-VI.04 17:40

CHEMICAL ANALYSIS OF ANCIENT PHARMACEUTICAL PRACTICES

M. Cotte, B. Fayard, J. Susini, ESRF, BP 220, 38043 Grenoble Cedex, France; P. Dumas, Synchrotron SOLEIL, L'Orme des Merisiers, Saint-Aubin, BP 48, 91192 Gif-sur-Yvette Cedex, France; Ph. Walter, (C2RMF), UMR 171 CNRS, Palais du Louvre, Porte des Lions, 14 quai F. Mitterrand, 75001 Paris, France

Our work aimed at a better understanding of the chemistry involved in the synthesis of lead plasters, which were pastes made of lead salts and fat, frequently used in the past as pharmaceutical products. Two non destructive synchrotron based techniques, X-ray micro-fluorescence and infrared micro-spectroscopy were used.

First, ancient Egyptian cosmetics were analysed by synchrotron infrared micro-spectroscopy. This technique allows both organic and mineral components to be identified and located. Furthermore, information about the supra-molecular organisation of the molecules can be obtained. Lead soaps were shown to be highly concentrated within the cosmetic. Second, about 100 recipes of lead plasters, mentioned in ancient texts, were assessed and compared. A selection of formulae has been reconstituted and the kinetics was followed by infrared spectroscopy. The primary chemical reaction involved in the preparation is the saponification of fat by the lead salts. The effect of some crucial parameters such as addition of water or temperature was studied. This chemical viewpoint provided a better insight on the technical evolutions developed over centuries. Finally, the question of the potential toxicity of such lead based products was addressed by analysing the interactions between these lead compounds and skin.

In conclusion, this study shows that although lead plasters exist since more than 30 centuries they yet appear as highly advanced preparations.

19:00

AWARD CEREMONY

The symposium organizers and the candidates to the graduate student award are requested to attend.

CONFERENCE RECEPTION

Thursday, June 2, 2005
Jeudi 2 juin 2005

Morning
Matin

Session VII : Surfaces and interfaces
Session chair : B. Müller

O-VII.01 9:00 -Invited-

RESULTS AND PERSPECTIVES IN HARD X-RAY PHOTOEMISSION SPECTROSCOPY (HAXPES) FROM SOLIDS

Giancarlo Panaccione, Istituto Nazionale per la Fisica della Materia INFN- Lab.TASC S.S. 14 Km 163.5, in AREA Science Park, I-34012 Basovizza (Trieste), Italy

PhotoEmission Spectroscopy (PES) is one of the most powerful tools to investigate solid state properties. A strong surface sensitivity, i.e. a few atomic layers, can be achieved by tuning the kinetic energy of photoelectron to the minimum of the so called 'universal curve' of escape depth. The state of the art in high quality surface science experiments can be summarized by an energy resolution of 5 - 30 meV for valence band spectra with $h\nu < 40$ eV, and of 30-200 meV at $h\nu > 100$ eV for core level spectra. On the other hand, there is a lack of corresponding experimental information on truly bulk properties in important materials such as high temperature superconductors and heavy fermions systems as well as in the case of low dimensional artificial solids.

We report of PES experiments performed at ESRF (Beamline ID16), within the frame of the European VOLPE (VOLUME PhotoEmission from solids) project, where we were able to achieve high energy resolution as well as good statistics spectra up to 6-8 keV. We demonstrate that, at these kinetic energies, one may obtain information from >100 Angstrom of depth, corresponding to a maximum surface contribution $< 3-5$ %. We present recent results obtained on transition metals and highly correlated systems, and we discuss the relevant technical and scientific aspects of this new field of research.

O-VII.02 9:30

BURIED INTERFACES CHARACTERIZATION USING HARD X-RAYS

F. Rieutord, B. Bataillou, H. Moriceau, CEA-Grenoble, 17 rue des Martyrs, 38054 Grenoble Cedex, France

X-ray scattering using hard X-rays under small incidences is a unique tool for the characterization of interfaces between dense materials e.g. the bond interface between two solids. Very few techniques have indeed the capability of penetrating large distances in materials yet keeping a nanometer scale surface sensitivity [1]. In the frame of studies on adhesion at nanometer scale, we have developed the technique of "interfacial" X-ray reflectivity. In addition to standard adjustment methods for extracting the interfacial density profile, several direct inversion techniques applicable to the interfacial reflectivity case will be described on practical wafer bonding examples [2].

O-VII.03 9:50

DRIVING FORCES OF ADSORBATE-INDUCED FACETING

Y. Garreau(a), A. Coati(a), O. Robach(b) and J. Creuze(c), (a)LURE, Bât. 209D, BP 34, Centre Universitaire Paris Sud, 91898 Orsay Cedex, France, (b)ESRF, BP 220, 38043 Grenoble, France, (c)LEMHE, Bât. 410, Centre Universitaire Paris Sud, 91405 Orsay Cedex, France

The self-organisation of nanometric systems has been intensively studied in the last years due to their potential application for the fabrication of magnetic, optoelectronic or catalytic nano-objects. The faceted surfaces represent a promising way for the realisation of nanostructured templates, in particular in the case of the faceting induced by adsorption, which allow one to control the system dimensions by tuning the adsorbate coverage.

Driving forces of adsorbate-induced faceting of vicinal surfaces are investigated through a coupling between Grazing Incidence X-Ray Diffraction (GIXD) experiments and atomic scale simulations. The approach is applied to the adsorption of Ag onto Cu vicinal surfaces, for which a submonolayer deposition induces a periodic faceting with the formation of Ag-covered facets and bare Cu facets. To obtain the driving forces of this phenomenon, we have developed a thermodynamical modelling in which the energetical quantities (adsorption energies of Ag atoms on the different Cu faces and surface energies of the clean Cu faces) are evaluated on atomic-scale simulations. We find that Ag adsorption on facets with high step densities is energetically favoured and that a faithful description of the system evolution, measured by GIXD, is obtained only if a proper value of the step interaction energy is included within the model. Moreover, we have measured by GIXD the periods of the nanostructure as a function of the Ag coverage. They vary between 10 and 100 nm in all the explored range. This variation can be explained using a model, which affirms that the ordering of the system is due to the elastic relaxations propagating deep into the substrate. GIXD measurements give evidence of this type of bulk relaxations.

O-VII.04 10:10

STRUCTURAL STUDY OF THE CuPc/Au(110) INTERFACE BY MEANS OF X-RAY ABSORPTION AND DIFFRACTION OF X-RAY, ELECTRONS, He ATOMS

L. Floreano, A. Cossaro, F. Bruno, A. Verdini, A. Morgante, Laboratorio TASC-INFM, Basovizza, 34012 Trieste, Italy, D. Cvetko, G. Bavdek, Dep. of Physics, Univ. Ljubljana, Ljubljana, Slovenia, F. Evangelista, A. Ruocco, Dep. of Physics, Univ. Roma3, Roma, Italy
We exploited the different degree of surface sensitivity of Grazing Incidence X-Ray Diffraction (GIXRD), Near Edge X-ray Absorption Fine Structure spectroscopy (NEXAFS), Helium Atom Scattering (HAS) and Electron Diffraction (LEED) to study the structure of Copper-Phthalocyanine, CuPc, overlayers on Au(110). CuPc is ideally suited to UHV studies for its thermal and chemical stability, while the missing row reconstructed (1x2)-Au(110) is expected to drive the orientation of the overlayer. All the data have been taken at the ALOISA-HASPES beamline at ELETTRA Synchrotron.

We followed in real time the CuPc deposition by using the HAS and LEED techniques to monitor the ordering of the CuPc overlayer and the rearrangement of the Au atoms, respectively. At the 1st ML saturation coverage, HAS showed a (5x7) phase, whereas LEED displayed a (5x3) phase with faint traces of a (5x7) phase, due to the larger sensitivity of LEED to high-Z Au atoms.[1] We studied GIXRD rod scans of two ordered phases to determine the CuPc induced reconstruction of the substrate atoms, whereas the orientation of the molecules was determined by NEXAFS spectra taken with different surface to electric field orientations. We envisage a model where CuPc molecules line up in chains along the [1-10] direction, locally inducing a x3 substrate reconstruction. The molecules result to be tilted by 30°; along the [001] direction with respect to the surface, in agreement with the two-domains, asymmetric cell found by XRD. Depending on the coverage, different periodicities are found along the [001] direction transverse to the chains. The highest coverage ordered phase is the (5x7) phase formed by two CuPc chains separated by one Au row.

[1] A. Cossaro et al., J. Phys. Chem. B 108 (2004) 14671.

10:30

BREAK

**Session VIII : Magnetism and spintronics materials
(joint session with symposium B)**

Session chair : R. Hertel

O-VIII.01 11:00 -Invited-

FUNDAMENTAL MAGNETIC STATES OF DISC AND RING ELEMENTS

C.A.F. Vaz(a), M. Klauui(b), J.A.C. Bland (a), L.J. Heyderman(c), F. Nolting(d), (a) University of Cambridge, Cavendish Laboratory, Cambridge, U.K., (b)Fachbereich Physik, Universitaet Konstanz, Konstanz, Germany, (c)Laboratory for Micro- and Nanotechnology, Paul Scherrer Institut, Villigen PSI, Switzerland, (d)Swiss Light Source, Paul Scherrer Institut, Villigen PSI, Switzerland

Currently, one topic of particular interest is the study of the equilibrium magnetic states and magnetisation reversal mechanisms in small elements, which are determined by the interplay between the magnetic anisotropy and exchange interaction with the shape of the element. Although a general understanding is now emerging about the importance of the different energy terms into the switching behaviour and the magnetic states that are attainable at remanence, many issues remain open, e.g., the existence of accessible metastable states in a given geometry, and how the stability of those states evolve with the geometrical parameters of the element. This is critical for device applications, memory elements, in particular in the context of spintronics, and for current induced magnetic switching processes, where well defined and reproducible magnetic configurations are required. Here, we present a systematic study of the remanence magnetic states in Co and Ni80Fe20 disks 1.65 µm in diameter with varying film thickness (5-38 nm), imaged by photoemission electron microscopy (PEEM). Circular elements with small holes (310 nm width) were also imaged. The type of observed magnetic configurations range from vortex configurations to other well defined multidomain states, namely states with two vortex cores ('Z-state' and 'diamond' configuration) and a 'triangle' state, which are mapped according to material and thickness range. In many instances, more than one magnetic state coexist in different disks of the same array, subjected to the same field history. These observations are correlated with magneto-optical Kerr effect (MOKE) measurements and micromagnetic simulations to show that this wealth of magnetic metastable states are stabilised by defects or attained in the nucleation process following the removal of the applied magnetic field.

ANTIFERROMAGNETIC DOMAIN STRUCTURE OF HEMATITE THIN FILMS INVESTIGATED BY X-PEEM MICROSCOPY

R. Belkhou(a), A. Barbier(b), P. Ohresser(a), M. Gautier-Soyer(b), J.-B. Moussy(b), A. Bataille(b), M.-J. Guittet(b), J. Raabe(c), F. Nolting(c) and C. Quitmann(c), (a)LURE, bat 209d, Université Paris-Sud, B.P. 34, 91405 Orsay, France, (b)CEA/DRECAM/SPCSI, 91191 Gif-Sur-Yvette, France, (c)Paul Scherrer Institut –SLS, 5232 Villigen PSI, Switzerland

Exchange bias arises from the direct exchange coupling at the interface between a ferromagnet (FM) and an antiferromagnet (AFM). It gives rise to a hysteresis loop shift and/or an increase of the coercitive field of the FM. This effect is extensively exploited for the development of advanced devices such as magnetic read heads, spin valves and permanent magnetic memory cells. It appeared recently that the AF oxides play a major and active role in the onset of the exchange coupling. Unfortunately, AF thin films are very difficult to investigate because of their zero net magnetic moment. Through magnetic linear dichroism, X-ray photoelectron microscopy (X-PEEM) experiments are a new and powerful tool to evidence the AF magnetic domain structure of FM and AFM layers.

We present recent results obtained using X-PEEM and magnetic dichroism experiments to determine the onset and structure of the magnetic AFM domain structure of hematite at a sub-micron scale. This study is believed to be a mandatory step toward a complete description of the exchange bias phenomenon in such systems. The X-PEEM experiments reveal very rich and complex AFM domain structures made by large domains (10-30 μm) mainly delimited by dislocation lines which are associated with the morphological features. Within each large domain the AFM domains adopt a dendritic fine structure (1-5 μm width). Thanks to the implementation of a variable linear light polarization, allowing identifying the orientation of the magnetic anisotropy in the AFM, we could show that up to 15 nm hematite thickness the AF structure is not bulk like and the anisotropy is not, as intuitively expected, in the basal plane. This genuine feature may explain the unusual properties of thin hematite films with respect to exchange coupling.

SOFT X-RAY RESONANT MAGNETIC SCATTERING STUDY OF PERPENDICULAR EXCHANGE COUPLED [Fe/Co]_n/FeMn FILMS

J. Camarero(a), J. Miguel(b), F. Garcia(c), J. Vogel(d), S. Pizzini(d), F. Romanens(d), J. Sort(c), N.B. Brookes(e), B. Dieny(c) and J.B. Goedkoop(b), (a)Dpto. de Física de la Materia Condensada, Universidad Autónoma de Madrid, 28049 Madrid, Spain, (b)Van der Waals-Zeeman Institute, University of Amsterdam, 1018 XE Amsterdam, The Netherlands, (c)CEA/Grenoble, DRFMC/SPINTEC, 38054 Grenoble, France, (d)Laboratoire Louis Néel-CNRS, 38042 Grenoble, France, (e)European Synchrotron Radiation Facility (ESRF), 38043 Grenoble, France

We present a microscopic magnetization reversal study of room temperature perpendicular exchange coupled ferromagnetic/antiferromagnetic (F/AF) films by means of polarization dependent soft X-ray resonant magnetic scattering. The motivation for this study is to understand the mechanisms that govern the magnetization reversal as well as to study the role of the interfacial coupling strength on it. Element selective magnetic field-dependent measurements in transmission geometry with the field applied perpendicular to the film surface have been performed. Diffraction patterns during the reversal have been measured using a 2D detector. The size of the domains, the amplitude of the magnetic modulation, and the domain wall width has been monitored along the forward and backward branches of the magnetization curve up to fields of 0.3 T. Different nucleation processes and domain size distributions along the forward and backward branches of the reversal are shown. The size of the magnetic domains during reversal is dependent on the strength of the coupling, decreasing when the latter increases. Evidence of the exchange bias being induced by pinned uncompensated AF interfacial spins is also presented.

ELECTRONIC AND MAGNETIC PROPERTIES OF SINGLE-CRYSTALLINE X/MgO(001) INTERFACES (X=Mn,Fe,Co)

M. Sicot and S. Andrieu, Laboratoire de Physique des Matériaux, UMR 7556 CNRS/Univ.Nancy I, BP 239, 54506 Vandoeuvre, France; F. Bertran and F. Fortuna, L.U.R.E., BP 34, 91898 Orsay, France

Spin dependent tunnelling transport from a ferromagnetic layer (F) to another one through an insulating barrier (I) depends on the structural and the electronic properties of the ferromagnetic materials and on the F/I interface. Very large tunnel magnetoresistance were thus predicted on single-crystalline Fe/MgO/Fe and Co/MgO/Co(001) tunnel junctions. On the experimental point of view, the chemical stability and magnetic properties of the F/I interface has to be studied in order to test the theory and improve the understanding of the tunnelling transport. For that purpose, we used X-Ray spectroscopies applied on ultrathin magnetic films (in the range of 1-3 monolayers thick) covered with the barrier. Co, Fe and Mn/MgO(001) single-crystalline bilayers were thus grown by MBE and analysed by using X-ray photoemission spectroscopy (XPS), X-ray absorption spectroscopy (XAS) and X-ray magnetic circular dichroism (XMCD) measurements performed at LURE and ESRF. The XPS and XAS analysis clearly demonstrate the weak hybridization between the MgO barrier and Fe or Co. On the contrary, a net oxidation of the Mn layer in contact with the MgO layer was observed. The Co and Fe ferromagnetic behaviour was observed to be conserved at the interface. Moreover, spin-resolved XPS experiments performed on a Fe layer covered by MgO clearly confirmed that the bulk Fe polarization is not killed at the Fe/MgO(001) interface. Finally, no average magnetization was detected at the Mn/MgO interface, which was assumed to come from some Mn oxidation.

Thursday, June 2, 2005

Jeudi 2 juin 2005

Afternoon

Après-midi

Session IX : Oxides

Session chair : A. Kolobov

O-IX.01 14:00

LOCAL BEHAVIOUR OF NEGATIVE THERMAL EXPANSION MATERIALS

P. Fornasini, G. Dalba, R. Grisenti, J. Purans, and M. Vaccari, INFN and Dept. of Physics, Univ. of Trento, 38050, Povo (Trento), Italy; F. Rocca and A. Sanson, IFN-CNR Section of Trento, 38050 Povo (Trento), Italy

Materials with negative thermal expansion (NTE) over large temperature intervals are raising strong interest, due to potential technological applications. Understanding the physical origin of NTE is still a challenging research topic, due to the different possible causes and the inadequacy of standard techniques. EXAFS can give peculiar insights on the local dynamical behaviour of NTE materials, thanks to the possibility of directly measuring the thermal expansion of selected inter-atomic distances and the intensity of relative vibrations perpendicular to the bonds [1]. After the first work on Ag₂O [2], a refined study of NTE cuprites Cu₂O and Ag₂O, while confirming a positive 1st-shell expansion, has revealed a rather complex 2nd-shell behaviour, inconsistent with a simple rigid unit modes (RUM) model. The differences in macroscopic NTE between the two compounds can be traced back to local dynamical differences. EXAFS studies have been recently extended to zincblende structures, like CuCl, characterized by NTE over a limited low-temperature interval; a positive 1st-shell expansion is observed.

[1] G. Dalba et al., Phys. Rev. Lett. 82, 4240 (1999).

[2] S. a Beccara et al., Phys. Rev. Lett. 89, 25503 (2002).

O-IX.02 14:20

SHORT-RANGE CHARGE-ORDER AND SPIN TRANSITION AT THE CROSSOVER OF THE METAL-INSULATOR TRANSITION IN RARE EARTH NICKEL PEROVSKITES

Cinthia Piamonteze(a), Hélio C.N. Tolentino(a,b), Aline Y. Ramos(a,c), Frank M.F. de Groot(d), Nestor E. Massa(e), Jose A. Alonso(f), (a)Laboratório Nacional de Luz Sincrotron, C.P. 6192, 13084-971, Campinas, Brazil (d)Laboratoire de Cristallographie, CNRS U.P.R. 5031, Grenoble, France (d)Laboratoire Louis Néel, CNRS U.P.R. 5051, Grenoble, France (d)Dept. Inorganic Chemistry and Catalysis, Utrecht University, Utrecht, The Netherlands (d)Lab. Nac. Investigacion y Servicios en Espectroscopia Optica, UNLP, La Plata, Argentina (d)Instituto de Ciencia de Materiales de Madrid, CSIC, Madrid, Spain

The rare earth (R) nickel perovskites display a thermally driven first-order metal-insulator transition and an unusual antiferromagnetic order at low temperature. Strong electron-lattice coupling suggests the presence of Jahn-Teller-polarons associated to local charge fluctuations. Systems with lighter R were found to crystallize with orthorhombic symmetry, where Ni occupies a very symmetric site. For systems with heavier R, a monoclinic distortion was found in the insulating phase. A long-range charge-ordering associated to the two different Ni sites explain the insulating phase, as well the antiferromagnetic ordering. Nevertheless, a common description for all lanthanides and the electronic changes at the crossover temperature remain as important questions to tackle. We have shown by using x-ray absorption spectroscopy at the Ni K-edge that two Ni sites coexist in all systems, independent on their long range crystallographic symmetry [1]. The site with longer Ni-O distance is weakly hybridized and leads to the electronic localization. Dramatic differences in the Ni L-edge spectral shape have been observed among the insulating and metallic states. Using charge transfer multiplet calculations, we establish the importance of the crystal field and 3d spin-orbit coupling to create a mixed-spin ground state [2]. We explain the transition in terms of modifications in the Ni crystal field splitting that induces a spin transition from an essentially low-spin (LS) to a mixed-spin state. This ground state modification explains the lower hybridization at the insulating state and shed light on the unusual observed antiferromagnetic order.

[1] Piamonteze et al., PRB 71, Jan (2005) Brief Report

[2] Piamonteze et al., PRB 71, Jan (2005) Rapid Comm.

O-IX.03 14:40**QUANTITATIVE STRUCTURAL REFINEMENT OF Mn K EDGE XANES IN LaMnO₃ AND CaMnO₃ PEROVSKITES.**

C. Monesi(a), C. Meneghini(a,b), F. Bardelli(a,b), M. Benfatto(c), S. Mobilio(a,b,c), U. Manju(d), D.D. Sarma(d), (a)Dipartimento di Fisica Università di Roma Tre, Via della vasca navale 84, 00146 Roma, Italy, (b)INFM-GILDA c/o ESRF Grenoble, France, (c)INFN Laboratori Nazionali di Frascati, via E. Fermi 40, 00044 Frascati, Italy, (d)Solid State and Structural Chemistry Unit, Indian Institute of Science Bangalore 560012, India

Recently a large number of theoretical and experimental studies were dedicated to manganese oxides with perovskite structure, such as La_{1-x}Ca_xMnO₃ due to their special magnetic, electronic and transport properties. These properties arise from a strong interplay between ferromagnetic double-exchange interaction, antiferromagnetic super-exchange and strong electron-phonon interaction. The local atomic structure around Mn ions, plays a crucial role in determining these complex phenomena, mainly because the electron-phonon coupling involves a dynamical Jahn-Teller deformation of the Mn local surroundings. For this reason short range order techniques like X-ray absorption spectroscopy (XAS) are very well suited for studying these compounds.

Recently an innovative code, named MXAN, was developed which allows a quantitative full structural refinement of the XANES signal. This work exploits the MXAN approach to analyze the Mn K edge XANES data of LaMnO₃ and CaMnO₃, the two end compounds of the doped manganite series La_{1-x}Ca_xMnO₃, in which the Mn ions are present only in one charge state as Mn³⁺ and Mn⁴⁺ respectively. The Mn local structure obtained resulted to be in very good agreement with those obtained from the EXAFS part of the spectra, showing how a quantitative picture of the local structure can be obtained from XANES in such compounds. In addition XANES provided additional topological information on the bond angle distributions. The very good results obtained on these two end compounds, open the way to extend this approach to the mixed oxide, where Mn³⁺ and Mn⁴⁺ are simultaneously present.

O-IX.04 15:00**X-RAY ABSORPTION SPECTROSCOPY AND RESONANT INELASTIC X-RAY SCATTERING IN Na_{0.3}CoO₂ AND Na_{0.3}CoO₂·yH₂O**

P. Leininger(a), J-P. Rueff(a), M. Four(a), G. Vanko(b), H. Ishii(c), A. Yaresko(d), (a)Laboratoire de Chimie Physique-Matière et Rayonnement (UMR 7614), Université Paris 6, 11 rue Pierre et Marie Curie, 75231 Paris Cedex 05, France, (b)ESRF, 6 rue Jules Horowitz, BP220, 38043 Grenoble cedex 9, France, (c)SPRING-8, 1-1-1 Kouto, Mikazuki-cho, Sayo-gun, Hyogo 679-5198, Japan, (d)Max-Planck-Institut für Physik komplexer Systeme, Nöthnitzer Str. 38 01187 Dresden, Germany

We studied the electronic properties of the novel superconductor Na_{0.3}CoO₂·yH₂O and its parent compound Na_{0.3}CoO₂ by resonant inelastic x-ray scattering (RIXS) and X-ray absorption spectroscopy (XAS) at the cobalt K-edge.

The experiments were carried out at the ESRF on the inelastic x-ray scattering beamline ID16. The spectra were measured in single crystal compounds and in powder. The XAS spectra are characterized by a pre peak and a main peak which are different in both compounds. The RIXS spectra show a peak about 10 eV from the elastic peak in all samples. In order to interpret the experimental results we perform LMTO calculations. We simulate the XAS spectra in the hydrated and unhydrated compounds with two different incident polarizations. By comparing the XAS calculation and the experiments we can explain the different peaks in the spectra. The changes, in the calculation, between the hydrated and unhydrated compounds are coherent with the experimental results. We interpret the 10 eV peak in the RIXS spectra as a charge transfer peak. Such a peak is not observed in the calculated optical spectra.

15:20

BREAK

16:00-19:00

POSTER SESSION

POSTER SESSION
Thursday, June 2, 2005
16:00 – 19:00

- O/P.01** **FUNCTIONAL IMAGING OF CELL CLUSTERS USING SYNCHROTRON RADIATION BASED MICRO COMPUTED TOMOGRAPHY**
B. Müller, P. Thurner, Computer Vision Lab, ETH Zürich, Sternwartstr. 7, 8092 Zürich, Switzerland and M. Riedel, ProBioGen, Goethestr. 54, 13086 Berlin, Germany
Synchrotron radiation based micro computed tomography (SR μ CT) is a superior method to nondestructively measure the local X-ray absorption in 3D space. Since biological cells consist mainly of water as the surrounding medium, higher absorbing agents (stains) have to be incorporated to make visible the structures of interest. With the standard SR μ CT setup, one can uncover the stain distribution with the spatial resolution of 1 μ m or even better. We have investigated human embryonic kidney (HEK293) cell clusters, staining either for cell membranes or RNA/DNA. The retrieved tomograms obtained at the beamlines BW2 and W2 (HASYLAB at DESY, Hamburg, Germany) and 4S (SLS, Villigen, Switzerland) clearly show that membrane staining gives rise to homogeneous absorption throughout the cell clusters, whereas the RNA/DNA-stained cell clusters exhibit a core of higher density and a rim of significantly lower density, which correlate with results of fluorescence microscopy and histology. The inner part of the clusters is associated with necrotic cells as the result of insufficient oxygen and nutrition supply and is surrounded by the rim of (6 ± 1) layers of vital cells. Consequently, SR μ CT can be used for functional imaging essential in tissue engineering.
- O/P.02** **STAINING CAPILLARIES IN THE MYOCARDIUM FOR SYNCHROTRON RADIATION BASED MICRO COMPUTED TOMOGRAPHY**
B. Müller, J. Fischer, P. Thurner, Computer Vision Lab, ETH Zürich, Sternwartstr. 7, 8092 Zürich, Switzerland and F. Beckmann, GKSS at DESY, Notkestr.85, 22607 Hamburg, Germany
Blood vessels formed after medical interventions such as radiofrequency treatment have to be visualized down to the capillaries level (diameter about 5 μ m) in order to validate the procedure. Synchrotron radiation based micro computed tomography provides the necessary micrometer resolution, but since both the vessels and the surrounding tissue consist mainly of water the absorption contrast is too weak to segment the vessel tree. Therefore, appropriate contrast agents have to be selected and suitable staining protocols to be developed. Our experiments proved that among the contrast agents used in medicine lyophilic salts with a mean particle diameter of 1.5 μ m such as CaSO₄, SrSO₄ and BaSO₄ are well suited for the staining of blood vessels. The combination of these salts with the embedding kit JB-4 (Polysciences Inc.) allows tissue fixation and long-term storage in solid state. We present results of heart tissue of pigs demonstrating the feasibility of the approach. Simple intensity-based segmentation algorithms are used to extract the vessel trees in the myocardium. These 3D images show that the vessels are not always connecting, which implies that the stain is not fully homogeneously distributed. This means that the procedure can be improved, e.g. by tailoring the viscosity of the suspension (salt/JB-4) to reduce sedimentation effects. The development of this technique is vital to verify neo-vascularization in the quantitative manner.
- O/P.03** **BIOMECHANICS OF HUMAN ARTERIES STUDIED WITH SMALL ANGLE X-RAY SCATTERING**
F. Schmid(a), H. Amenitsch(a), G. Sommer(b), M. Rappolt(a), P. Regitnig(c), P. Laggner(a), G.A. Holzapfel(b), (a)Institute of Biophysics and X-ray Structure Research, Austrian Academy of Sciences, Graz, Austria, (b)Department of Solid Mechanics, Royal Institute of Technology, Stockholm, Sweden, (c)Institute of Pathology, Medical University Graz, Graz, Austria
The collagen diffraction patterns of different human arteries under conditions of uniaxial tensile stress have been investigated by synchrotron small angle X-ray scattering. Using a recently designed tensile testing device the orientation and d-spacing of the collagen fibers in all arterial layers have been measured in-situ with the macroscopic force and sample stretching under physiological conditions. First results show a direct relation between the orientation and extension of the collagen fibers on the nanoscopic level and the macroscopic stress and strain [1]. This is attributed first to a straightening, second to a reorientation of the collagen fibers, and last to an up-take of the increasing loads by the collagen fibers. Results for all layers, a comparison and interpretation will be presented. [1] Schmid F. et al, Journal of Synchrotron Radiation, 2005, in press
- O/P.04** **EVAPORATION INDUCED SELF-ASSEMBLY OF PHOSPHOLIPIDS STUDIED WITH SURFACE DIFFRACTION**
H. Amenitsch, B. Sartori, M. Rappolt, G. Pabst, P. Laggner, Institute of Biophysics and X-ray Structure Research, Austrian Academy of Sciences, Schmiedlstr. 6, 8043 Graz, Austria and D.Grosso, LCMC, Université Pierre et Marie Curie, 4 place Jussieu, 75252 Paris Cedex 5, France
Phospholipids are the major constituted of biomembranes and form complex morphologies depending on the hydration level. In particular surface deposited structures are widely used on one hand as model systems for structural investigations, like dynamic parameters or phase behavior, and on the other hand in various applications, e.g. biosensor or biomimetic coatings. Evaporation induced self-assembly process (EISA)¹ and particular dipcoating² is a powerful method to create high quality coatings of mesoporous thin films. In this presentation we show that this method applied for phospholipids has enormous advantages to the conventionally used methods and allows the production of thin films under controlled and reproducible conditions. Grazing incidence small angle X-ray diffraction is the method of the choice to check the order, quality of the films and enables even to study in-situ the self-assembly process of the lipids after the dipcoating. Several examples of this process will be given demonstrating the advantage and the time course of the self-assembly. Further the improvement of the film quality is shown in terms of order, morphology and thickness due to the fabrication of the films under controlled and carefully selected conditions.
1. Y. F. Lu et al., Nature 398, 223-226 (1999).
2. F. Cagnol et al., Journal of Materials Chemistry 13, 61-66 (2003).

O/P.05**X-RAY MICRO-DIFFRACTION FOR ENGINEERED BONE ANALYSIS**

A.Cedola(a), M. Mastrogiacomo(b), V. Komlev(c), C. Giannini(d), A. Guagliardi(d), R. Cancedda(b), F. Rustichelli(c), and S. Lagomarsino(a), (a)Istituto di Fotonica e Nanotecnologie - CNR, V. Cineto Romano 42, 00156 Roma, Italy e INFN, Unita' di Ancona, (b)Centro Biotecnologie Avanzate - Laboratorio Medicina Rigenerativa, Largo R. Benzi 10, 16132 Genova, Italy, (c)Istituto di Scienze Fisiche, Università di Ancona, Via Ranieri 65, I60131 Ancona, Italy e INFN, Unita' di Ancona, (d)Istituto di Cristallografia - CNR, Via Amendola 122/o- 70126 Bari, Italy

One of the most recent therapeutic strategies for the reconstruction of damaged large bony segments includes the tissue engineering approach. It takes advantage of the patient's own cells, which are isolated, expanded in vitro, loaded onto a bioceramic scaffold and reimplanted into the lesion site. Bone marrow stromal cells (BMSC) are the most commonly used cell type.

A structural characterization of the engineered bone is largely desirable. An important point is to evaluate if the BMSC extracellular matrix deposition on a bioceramic scaffold recapitulates the ontogeny of the natural bone development. Moreover the investigation of the interaction between the newly deposited bone and the scaffold results particularly interesting. Indeed the chemistry and the geometry of the scaffold used to deliver BMSC in the lesion site determine spatial organization of the new bone and the bone-biomaterial integration. A local structural study of the interface between the newly deposited bone and the biomaterial is therefore expected. For this purpose we applied high spatial resolution x-ray microdiffraction analysis with synchrotron radiation using as optical element an x-ray waveguide at ID13 beamline of ESRF. The engineered bone was obtained applying an in vivo mouse model using ceramic scaffolds and osteogenic precursor cells. The crystal structure of the neodeposited tissue and the distribution and orientation of the mineral bone grains at the interface were studied in detail. Moreover the grain size distribution at the scaffold walls has been evaluated and the orientation of the collagen fibres has been studied. On the basis of the acquired data, we propose a model for the deposition and growth of the engineered bone within the scaffold pores.

O/P.06**EFFECT OF CHAIN LENGTH ON THE LATERAL ORGANIZATION OF CHARGED LIPID MEMBRANES**

Georg Pabst(a), Heinz Amenitsch(a), Michael Rappolt(a), Peter Lagner(a), John Katsaras(b), Karl Lohner(a), and Andrea Hinkel(a), (a)Institute of Biophysics and X-ray Structure Research, Austrian Academy of Sciences, Schmiedelstr. 6, 8042 Graz, Austria, (b)National Research Council, Steacie Institute of Molecular Sciences, Chalk River, Ontario K0J 1J0, Canada

We have studied the hydrocarbon chain packing in the gel phase of well aligned multibilayers composed of phosphatidylglycerol (PG) by grazing incidence x-ray diffraction. The hydrocarbon chain length was varied between 16 hydrocarbons and 18 hydrocarbons per chain. We observe a transition from a orthorhombic packing with a tilt direction towards nearest neighbours for di16PG to a monoclinic chain packing with a tilt direction in-between nearest and next nearest neighbours for di18PG. This leads to an increase of packing density, which might affect the lipids affinity for interaction with membrane active compounds.

O/P.07**HIGH-ENERGY SYNCHROTRON DIFFRACTION STUDY OF STRESS-INDUCED PHASE TRANSITION AND TEXTURE IN A SUPERELASTIC NITI SHAPE MEMORY ALLOY**

W.W. Schmahl, A. Baruj, J. Khalil-Allafi, Th. Reinecke, Shape Memory Research Center, SFB 459, Ruhr-Universität, 44780 Bochum, Germany

Ni-rich NiTi shape memory alloys are the most promising materials for superelastic applications due to their high strength and recoverable strains up to 8%. Superelasticity is based on the stress-induced phase transition from cubic B2 austenite to monoclinic B19' martensite and recovery of the B2 phase upon release of stress. In flat specimens of superelastic NiTi under uniaxial tensile stress the austenite - martensite transformation is localised in shear transformation bands. We investigated the stress-induced phase transformation in-situ in flat Ni50.7Ti49.3 tensile specimens by high-energy diffraction methods in a space-resolved mode at HASYLAB, DESY, Hamburg, Germany. The superelastic properties were optimized by solution annealing at 850°C for 15 min and water quenching; with subsequent cold rolling at -150°C with a thickness reduction of 20 %. Finally, the material was aged at 300°C for 6 minutes. Upon uniaxial loading of the sample, shear bands formed at a critical strain of 0.8 % and spread upon increasing the strain. Inside the shear band, the transformation to martensite was incomplete even at 8% macroscopic tensile strain. Using an area detector, the texture of the stress-induced martensite as well as the strain on the residual austenite grains was recorded and evaluated. The matrix outside the shear band does not show enough martensite to be detectable by diffraction. Between the shear band and the adjacent matrix we observe a boundary region with transitional states of strain.

O/P.08**APPLICATION OF μ -XAFS FOR THE DETERMINATION OF THE CRYSTALLIZATION RATIO IN A SERIES OF VITRO-CERAMIC MATERIALS CONTAINING INDUSTRIAL WASTE**

F. Pinakidou, M. Katsikini, E.C. Paloura, P. Kavouras, F. Komninou and Th. Karakostas, Aristotle Univ. of Thessaloniki, Dept. of Physics, 54124 Thessaloniki, Greece, A. Erko, BESSY GmbH, Albert Einstein Str. 15, 12489 Berlin, Germany

The distribution of Fe in a series of vitrified and annealed Pb- and Fe-rich solid industrial waste is studied by means of x-ray fluorescence mapping (XRF), micro- and x-ray absorption fine structure (μ-XAFS and XAFS) spectroscopies. The under study samples contain toxic ash that is vitrified with appropriate quantities of vitrifying (SiO₂) and flux (Na₂O) agents. The effect of the Fe concentration and annealing temperature on the oxidation state and the local environment around Fe is studied using Fe-K-edge μ-XAFS and XAFS measurements conducted at the KMC-2 monochromator at BESSY. XRF mapping in combination with μ-XAFS applied to samples containing 50% and 60% ash, demonstrated that annealing at temperatures above 600oC induces loss of homogeneity. By appropriate fitting of the EXAFS spectra it is found that X% of the Fe atoms form Fe-rich islands where Fe is octahedrally coordinated in FeO₆ polyhedra (RFe-O=1.93Å). Contrary to that, (100-X)% of the Fe atoms are incorporated in the vitreous matrix (which has a lower Fe content), and are coordinated in FeO₄ tetrahedra (RFe-O=1.88Å). Moreover it can be proposed that the crystallization ratio X/(100-X) increases with the annealing temperature and the loss of homogeneity becomes more extensive.

O/P.09**ORDER AND SEGREGATION IN Co-Pt ALLOY CLUSTERS INVESTIGATED BY IN-SITU X-RAY ABSORPTION**

P. Andreazza, N. Bouet, C. Andreazza-Vignolle, D. Zhao, Centre de recherche sur la Matière Divisée, UMR 6619, CNRS-Université d'Orléans, 1B rue de la Férollerie, 45071 Orléans Cedex 2, France and H. Magnan, A. Traverse, LURE, CNRS-CEA-Université Paris XI, Bât. 209D, 91898 Orsay Cedex, France

With respect to bulk materials, surface-supported clusters offer additional degrees of freedom to tune the magnetic properties by controlled modifications of particle size, shape and interactions with the substrate. These characteristics make nanosized systems attractive for fundamental studies as well as for miniaturized data-storage applications. The use of binary alloy clusters opens new fields to tailor magnetic properties. Using binary 3d transition metal alloys like Co-Pt with a very high magnetic moment it might be possible to enhance the magnetic moments in nanoparticles above values known from pure materials.

The understanding of the magnetic behaviour of these bimetallic particles proceeds though the accurate determination of both the atomic structure and morphology, and of the chemical ordering and surface segregation phenomena that they undergo. Co-Pt nanoparticles (1-3nm) have been elaborated under high vacuum and their internal chemical organisation studied in-situ by Extended X Ray Absorption fine Structure (EXAFS) combined with TEM observations. These investigations have allowed us to determine structural models of the arrangement of the Co and Pt atoms inside the nanoparticles (core-shell, solid solution, ordered structures) versus composition, size and annealing temperature. Then, the correlation between size and structure of nanoparticles was studied according thermodynamic point of view taking into account experimental and calculated data, such as surface energies, effective pair interaction energies.

O/P.10

Withdrawn

O/P.11**DEPTH RESOLVED LOCAL STRUCTURE IN THIN FILMS WITH PERPENDICULAR MAGNETIC ANISOTROPY BY GRAZING INCIDENCE X-RAY ABSORPTION SPECTROSCOPY**

Narcizo M. Souza Neto(a,b), Aline Y. Ramos(a,c), Hélio C.N. Tolentino(a,d), Alessandro Martins(e), Antonio D. Santos(b), Flávio Garcia(a), (a)Laboratório Nacional de Luz Síncrotron, Campinas SP, Brazil, (b)Departamento de Física dos Materiais e Mecânica, Univ. de São Paulo SP, Brazil, (c)Laboratoire Louis Néel, CNRS U.P.R. 5051, Grenoble, France, (d)Laboratoire de Cristallographie, CNRS U.P.R. 5031, Grenoble, France, (e)Universidade de Goias, GO, Brazil

Research on thin magnetic films with perpendicular anisotropy has increased greatly in the last years, with the prospect of using the polar magneto-optical effect to increase the density of information stored in magnetic media. Besides this perpendicular magnetic anisotropy, high coercivity is also requested for candidate materials. Both characteristics are found in near-equiatomic FePt(Pd) and CoPt films with the ordered FCT structure. Aiming to understand and optimize the thickness dependence of the magnetic properties in these films, we investigate the local structure in FePt and CoPt films prepared by dc magnetron sputtering over MgO(100) substrate. Long range order and average magnetic anisotropy are measured by XRD and VSM. Grazing incidence x-ray absorption spectroscopy provides a depth sensitive local structural probe, as the penetration depth can be tuned by varying the incident angle close to the critical angle for total reflection (around 0.3deg at 7.7keV, Co Kedge). For 50nm thick films the depth dependence of the local chemical and structural structure is clearly evidenced and associated to a chemical ordering, while for thicker film (200nm), the segregation of the Co atoms into the film seems to be the dominant feature. We also report on preliminary measurements of depth resolved cobalt magnetic moment, by resonant magnetic x-ray scattering at the Co K edge.

O/P.12**LOCAL MAGNETISM IN GRANULAR IRON/IRON OXIDE NANOSTRUCTURES: A PHASE- AND SITE-SELECTIVE X-RAY MAGNETIC CIRCULAR DICHROISM STUDY**

L. Signorini, L. Pasquini, F. Boscherini and E. Bonetti, Department of Physics University of Bologna, V.le Bertoni Pichat 6/2, 40127 Bologna, Italy, I. Letard, ESRF, 6 rue Jules Horowitz, BP 220, 38043 Grenoble Cedex, France, S. Brice-Profeta, ENSEEG/LTPCM, Domaine Universitaire, 1130 rue de la piscine, BP 75, 38402 Saint-Martin d'Hères Cedex, France, M. Marangolo and Ph. Sainctavit, LMCP, 140 rue de Lourmel, 75015 Paris, France

We report an X-Ray Magnetic Circular Dichroism (XMCD) study of the local magnetic properties of the nanostructured granular iron/iron oxide system. This system has been studied for its peculiar magnetic, and magnetoresistive properties, and new size- and disorder driven magnetic effects have been recently found[1]. The magnetic properties depend on the presence of Fe atoms in three distinct phases (α-Fe, magnetite and maghemite); in the oxide phases Fe is present in tetrahedral and octahedral environments. Due to the phase and site selectivity of the technique we were able to study the contribution of different phases and sites to the average magnetic properties.

Core shell nanoparticles with core size ranging from 5 to 13 nm were synthesized using the inert gas condensation technique and 12 hours of controlled surface oxidation; the particles were compacted to form pellets. A previous structural characterization[2] showed that investigated samples consist of α-Fe particles uniformly embedded in a nanocrystalline matrix made of both magnetite and maghemite. The average dimension of the oxide grains is 2-3 nm. XMCD spectra were measured as a function of iron particle dimension and applied magnetic field and were analyzed by fitting them with dichroic signals of reference bulk phases. We detect the presence of a significant spin canting, which predominantly affects the octahedral sites of the oxide matrix, and study its dependence on the degree of structural disorder and applied magnetic field.

[1] L. Del Bianco, D. Fiorani, A. M. Testa, E. Bonetti and L. Signorini, Phys. Rev. B 70, 052401 (2004)

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O/P.13**MAGNETIC DOMAIN CONFIGURATIONS IN THIN RIBBONS WITH COMPETING ANISOTROPIES**

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Innovative film deposition and microfabrication techniques may be applied to prepare systems with tunable magnetic properties, showing exciting competitions between magnetic anisotropies of different origin. The study of such systems is motivated by their potential applications in high-density magnetic data storage or in magneto-electronic devices. However, magnetic nanostructures are also excellent model systems for studying fundamental magnetic properties, since thickness, shape, and size of the structures as well as the distance between them can be varied independently of each other.

In this study we report about the properties of thin Fe and Co stripes prepared by depositing the magnetic materials at oblique incidence on ribbon-shaped pre-patterned silicon substrates. This oblique evaporation (OE) geometry produces films with a dominant in-plane uniaxial character of the magnetic anisotropy. The interplay of shape and OE-induced in-plane anisotropy determines the micromagnetic structure. The magnetic domains are imaged with a photoemission electron microscope using the x-ray magnetic circular dichroism effect (XMCD-PEEM). With decreasing structure size, the demagnetizing field arising from the magnetic changes formed at the ribbons edges becomes important. It induces shape anisotropy with an easy axis parallel to the ribbons' long axis. When this axis is perpendicular to the OE-induced easy axis, a stripe domain configuration of alternating magnetization direction is observed within the ribbons. For the longitudinal case, both the shape anisotropy and the OE-induced anisotropy favor a magnetization along the magnetic stripes. The domain walls density is reported for different anisotropy geometries as a function of the stripes' width and spacing.

O/P.14**MAGNETIC ORDER IN SUBMICROMETRIC PATTERNED MAGNETIC FILMS STUDIED BY RESONANT SCATTERING OF POLARIZED SOFT X-RAYS**

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Submicron sized structures are currently attracting attention especially for potential applications in magnetic storage media and solid-state devices.

In this work we report on the analysis of an array of rectangular (1000nm x 300nm) dots of permalloy (Ni81Fe19), with a varying thickness from 10 to 120 nm. We have used resonant magnetic scattering of polarized soft x rays (XRMS) to investigate their magnetic behavior as a function of the applied field. This technique couples the advantages of structural analysis to the sensitivity to electronic structure which is the consequence of a spectroscopic response to resonant core-electron excitations where optical constants depend upon the local magnetic moment of the scattering ion. Our micro-magnetic simulations confirm experimental findings on both the effect of the large shape anisotropy due to the geometrical asymmetry of the system and the effect of increasing the thickness on the multi-domain formation during the reversal. To investigate the existence of a correlation in the magnetic alignment between pairs of neighboring dots during the reversal we measured the field-dependent scattered intensity at half the value of the first order structural peak of the rocking scan. At the same time, field-dependent measurements at Bragg peaks of different orders show hysteresis which differ from that of the specular reflectivity as they should be sensitive to the spatial distribution of the magnetization inside the dot whereas the zero order simply measures the average magnetization: an interpretation of such hysteresis on the basis of micro-magnetic simulations will be made in analogy with the model successfully used for diffracted magneto-optical Kerr effect (D-MOKE) experiments.

O/P.15**TEMPERATURE DEPENDENT MN MAGNETISATION PROFILE IN A LSMO/STO SUPERLATTICE**

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A clear understanding of the decrease of the spin polarisation of the La2/3Sr1/3MnO3 (LSMO) interface with temperature is still a subject of debate.

Temperature dependent soft X-ray resonant magnetic scattering has been performed in order to investigate the Mn magnetisation profile in a LSMO/STO superlattice. The measurements have been carried out at the SIM beamline at SLS. Angular dependent reflectivity curves were recorded at RT and at 50K close to the Mn L3 edge with circular polarised light for two opposite direction (I+, I-) of a longitudinal applied magnetic field. Photon energy dependent reflectivity curves across the L2,3 Mn edge, at the angular positions of the four first order Bragg peaks, were collected in the same way. An increase of the magnetic contrast at 50 K is observed as well as a strong change in the shape of the asymmetry ratio with regard to RT. This is a direct signature of a change of shape for the Mn magnetisation profile throughout the LSMO layer. Energy dependent reflectivity has been measured at several temperatures at the second order Bragg peak. The result show that the average intensity is comparable for 50,100, 200K and changes at 270 and 300K. (I+ - I-) continuously reduced with T in amplitude as the macroscopic magnetisation. However, rather than vanishing, we observe a change of sign at 270 K and a strong dichroic contrast. Those observations will be discussed from magneto-optic calculations aiming at the quantitative determination of the in-depth change of the Mn magnetic amplitude with T from the interface.

O/P.16**STRUCTURAL CHARACTERIZATION OF A CONJUGATED POLYELECTROLYTE**

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Conjugated polymers are attractive materials both from a theoretical and a practical point of view. However, they are difficult to process since they are insoluble and infusible. By adding charges to these polymers, water-soluble conjugated polyelectrolytes are obtained. It then becomes possible to easily tune the strong Van der Waals attraction due to the p-conjugation of their backbones and the long-range repulsive electrostatic interaction due to the localized electrical charges between chains in solution. The aim of this project is to study first the thermodynamics and the structure of conjugated polyelectrolytes in aqueous solutions. In a second part, we will study the influence of their dispersion state on the conductivity of the thin films obtained from the aqueous solutions by various deposition techniques.

Once the polymers synthesized by oxidative coupling polymerization of 3-thiophene acetic acid, they are characterized by size exclusion chromatography and by UV-visible absorption spectroscopy. Their structure in aqueous solutions was also studied by X-ray and neutron small angle scattering techniques. Such techniques give information not only about the polyelectrolyte character of the synthesized conjugated polymers but also about their aggregation state in solution according to the ionization degree as well as the doping ratio of the chains and the ionic strength of the solutions.

O/P.17**CONTROLLED DRUG RELEASE FROM SILICA-BASED POROUS AND NON-POROUS MATERIALS**

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The aim of this study was to synthesize and evaluate silica-based materials that would provide a slow release of highly water-soluble biphosphonate or the more apolar ibuprofen. The biphosphonate used in this work, etidronate, is a small water-soluble molecule that can be used to inhibit bone resorption in the body, since calcium ions can be complexed to the biphosphonate through a bidentate bond. This kind of silica material could therefore serve as active filler in polymer-based implants. The biphosphonate was incorporated into the silica during the spray-drying step, and the silica could provide a slow release on the month time-scale. The release process is suggested to be controlled by both erosion and diffusion processes. A series of mesoscopically ordered silicas with different pore sizes, pore connectivity, and pore geometry have been studied as carrier matrices for controlled release of Ibuprofen, and the release processes were monitored under in vitro conditions. The adsorption of ibuprofen from hexane was shown to be of Langmuir type in all cases, and the degree of drug loading was dependent on the specific surface area and the pore diameter of the host matrix. The release process is found to be mainly diffusion controlled, but clear differences were observed between the studied materials, which we mainly ascribe to differences in the pore connectivity and pore geometry of the materials, and the aqueous stability of the matrix. The studied nanostructured silicas display a high degree of drug loading, and, depending on the host material, a controlled drug release can be provided for time periods varying from hours to weeks.

O/P.18**ONLINE-STUDIES ON THE FORMATION OF SILICALITE-I MONITORED VIA SMALL ANGLE SCATTERING TECHNIQUES**

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The aim of this study was to monitor the nucleation process of silicalite-I from the dissolving of amorphous Aerosil up to the final stage of fully crystalline silicalite-I via in-situ small angle x-ray-scattering (SAXS) and small angle neutron scattering (SANS) experiments.

A typical silicalite-I synthesis mixture consists of 5.64 g Aerosil 380, 34 ml H₂O, 5.1 g NaOHaq (37 %) and 20.32 g aqueous tetrapropylammoniumhydroxide-solution (20 %). The reaction is carried out at 90 °C in polypropylene beakers. With the help of a 2 mm ID inert tube and a peristaltic pump the reaction mixture is pumped through a measuring cell. The time resolved SANS experiments were performed at the D22 (ILL, Grenoble) and the in-situ time resolved SAXS experiments were performed at the BL 5.2 L (Elettra, Trieste). All data was collected with on-site measurement equipment.

The obtained data show that the first reaction step is the dissolving of the Aerosil. This is shown by the use of pretreated Aerosil which was heated for 24 h at 90 °C with NaOH giving a smaller initial particle diameter. After the first decrease of the average particle diameter down to ca. 40 nm, an increase up to a value of about 120 to 200 nm after 6 to 15 h can be observed - depending on the reaction mixture. This value remains on the same level until at around 48 h Bragg peaks can be observed showing the end of the reaction with the appearance of crystalline silicalite-I. This indicates that the nucleation process occurs inside the agglomerates.

O/P.19**SMALL ANGLE X-RAY SCATTERING INVESTIGATION OF SELF-ORGANIZED CdSe/ZnSe QUANTUM DOT ALIGNMENT**

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For the realization of light emitting devices in the yellow-green spectral region, CdSe/ZnSe is one of the most promising material systems. For laser diodes, well-ordered arrays of quantum dots (QDs) are especially interesting. Apart from a vertical ordering of CdSe QDs embedded in ZnSe, as found earlier [1], the grazing incidence small angle x-ray scattering (GISAXS) investigations presented here clearly show, that a lateral ordering can be achieved as well. Stacked CdSe QD layers separated by strain compensating ZnSe spacer layers were grown by molecular beam (migration enhanced) epitaxy on GaAs(001). GISAXS Experiments were performed at the SAXS beamline at ELETTRA. Depending on the growth parameters, satellite peaks at non-zero lateral scattering components can be observed, the presence of which clearly prove a vertical and lateral correlation of the QDs. The results obtained for different spacer layer thicknesses and different number of stacked QD layers point to a strain-driven, self-organized ordering process similar to other material systems [2-4]. Moreover, an two-fold anisotropy of the satellite spots is found, which can be attributed to anisotropic growth processes related to surface properties. The ordering process is found to be kinetically limited, as found from the dependence of the GISAXS data on growth temperature. The results indicate that the ordering can be significantly improved after optimization of growth parameters [5]. [1] D. Litvinov et al., J. Appl. Phys. 89, 3695 (2001) [2] J. Tersoff et al., Phys. Rev. Lett. 76, 1675 (1996) [3] M. Pinczolis et al., Appl. Phys. Lett. 73, 250 (1998) [4] Q. Xie et al., Phys. Rev. Lett. 75, 2542 (1995) [5] Th. Schmidt et al. Appl. Phys. Lett. 84, 4367 (2004); Elettra Highlights 2003-04, 63 (2004)

O/P.20**ALIGNMENT OF GE NANO-ISLANDS ON Si(111) BY TAYLORED SURFACE SELF-PATTERNING**

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The growth of germanium nano-structures on silicon, especially self-assembly of such structures is of high technological interest. The adsorption of group-V and group-III elements prior to Ge deposition is known to modify the surface energetics and surface kinetics, and thus to change the growth mode [1-3]. Specifically, the adsorption of Ga on Si(111) has been shown to lead to a narrowed 3D island size distribution during subsequent Ge epitaxy [4]. In the present investigation, the evolution of the Si(111) surface after deposition of Ga has been observed by low energy electron microscopy (LEEM) and x-ray photoemission electron microscopy (XPEEM) at the nanospectroscopy beamline at ELETTRA. At elevated temperatures, Ga sqrt3 domains nucleate at step edges and at domain boundaries of the initial 7x7 reconstruction, and these sqrt3 domains grow along high-symmetry crystallographic directions. At submonolayer coverages, a 2D mesoscopic pattern formation is achieved due to the phase separation of Ga sqrt3 and bare Si 7x7. The pattern size can be tuned by choosing different substrate miscut angles, and the shape can be varied through the miscut direction. A pattern formation mechanism is proposed which is based on intrinsic properties of the Si(111) surface. Subsequent Ge deposition leads to nucleation of 3D Ge islands, which takes place at the boundaries between sqrt3 and 7x7 domains. This can be attributed to different diffusion lengths for Ge on either of the surface reconstructions, leading to heterogenous nucleation conditions.

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O/P.21**MAGNETIC DOMAIN WALLS IN T-SHAPED PERMALLOY MICROSTRUCTURES**

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The magnetic domain configuration of 20 nm thick T-shaped permalloy structures has been observed using a photo emission electron microscope and X-ray magnetic dichroism. The shape of the structures allows us to analyse 90 degree Néel walls in the contact region. We deduce the wall from the XMCD images and investigate its dependence on the contact width. The results are compared to micromagnetic simulations. We find a decrease in domain wall width with decreasing contact dimensions as expected by theory and in good agreement with our micromagnetic simulations.

O/P.22**COHERENT X-RAY DIFFRACTION FROM A MICRO-CRYSTAL AT THE ESRF: FIRST RESULTS AND QUESTIONS**

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X-ray scattering techniques are powerful tools to probe the structure of matter on a nanometric length scale, in a non-destructive way. Materials in their own environments can thereby be studied (embedded particles, under extreme pressures/temperatures, etc.). However, the phase retrieval problem leads to model-dependant analysis of the scattered intensity. It has been shown that the oversampling of the diffraction pattern can solve the phase problem [1]. The 3D electron density distribution can be obtained through inversion algorithms without the need of a priori hypothesis. While it is well established for optics, this technique remains challenging for x-rays, where a highly brilliant source, such as the one delivered by third generation synchrotrons, is needed [2].

We will present first coherent x-ray diffraction experiments performed at the ID1 beamline at ESRF (France) [3]. The 3D coherent diffraction pattern from a single micrometric Au crystal is investigated. After the description of the experimental setup and the presentation of the results (diffraction and inversion), we will discuss the problems emphasized by this experiment: (i) use of a beamstop, (ii) source size related contrast, (iii) possible use of KB mirrors for the investigation of nanometric particles.

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O/P.23**GISAXS STUDIES OF THE FORMATION OF Ge NANOCRYSTALS IN SiO₂ IN (Ge+SiO₂)/SiO₂ MULTILAYERS DEPOSITED BY MAGNETRON SPUTTERING**

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Grazing incidence small angle x-ray scattering (GISAXS) was applied to study the synthesis and size evolution of Ge nanocrystals in SiO₂ multilayers deposited by magnetron sputtering on either SiO₂ or Si substrate. The deposited films contained 15 bi-layers, each consisting of a 5 nm-thick layer of pure SiO₂ and a layer (either 5 or 10 nm-thick) of co-sputtered mixture of 40%mol Ge and 60%mol SiO₂. Sputtering was performed either at room temperature or at 700 oC. As-deposited samples were subsequently thermally annealed up to Ta = 1000oC. The 2D GISAXS patterns, besides detecting the bi-layered structure, revealed the formation of nanoparticles (NPs) in the deposited films. The shape and the size of Ge NPs were found to be strongly dependent on the annealing temperature, the best Ta for the formation of spherical Ge-NPs being in the Ta = 700 - 800 oC range. It has been shown that the average size of NPs, at given Ta, can be controlled by the thickness of the Ge-SiO₂ co-sputtered layer. The deposition on the 700 oC heated substrate resulted in formation of non-spherical, faceted nanoparticles.

O/P.24**IMAGING OF MAGNETIC QUANTUM DOTS**

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Arrays of magnetic nanodots have been prepared by grazing-incidence evaporation of Co in ultra-high vacuum onto self-organized templates consisting of nanometric pyramids of SiGe alloy previously grown on Si(001), and imaged in-situ by Low-Energy Electron Microscopy (LEEM) and X-ray Photoemission Electron Microscopy (XPEEM) with X-ray Magnetic Circular Dichroism (XMCD) at the Nanospectroscopy Beamline of synchrotron Elettra. Co nanodots appear correlated with the original substrate relief pattern; their lateral sizes are as small as 200 nm x 25 nm. The dots are ferromagnetic and single-domain at room temperature; they display uniaxial magnetic anisotropy caused by the off-normal deposition. Domains composed of various dots are observed, indicating that the individual magnetic nanoparticles are dipolarly coupled. Strategies to overcome this limitation and obtain arrays of magnetically independent nanodots will be discussed.

O/P.25**STRUCTURAL PERTURBATIONS WITHIN NANOCRYSTALLINE CU PROBED BY TEMPERATURE-DEPENDENT EXAFS**

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Extended X-ray absorption fine structure (EXAFS) spectroscopy has emerged as an invaluable synchrotron radiation technique for determining the short-range atomic structure of a diverse variety of materials. Here, Cu nanocrystals were synthesised in 5 µm glass films (SiO₂) by high-energy ion implantation and thermal annealing. We have previously shown that glass-embedded metallic nanocrystals exhibit subtle structural deviations [1], as compared to their bulk counterparts, which reflect the nonlinear optical properties of such composite materials [2]. In order to properly take advantage of these unique characteristics it is of great importance to understand their fundamental properties. In order to do so, we have performed temperature-dependent EXAFS measurements on Cu nanocrystals of ~ 15 nm diameter. High quality data were collected for nanocrystals and bulk standards up to $k = 18 \text{ \AA}^{-1}$ at the Photon Factory, Japan. We reveal that the nanocrystals develop structural characteristics different to the bulk material as a function of temperature. In particular, the nanocrystal Cu-Cu bondlength increases more than that of the bulk material with increasing temperature. Furthermore, the Debye-Waller factor increases at the same rate for both nanocrystals and bulk material, while as anticipated the coordination number is independent of temperature. The experiments demonstrate that the thermal behaviour of Cu nanocrystals embedded in SiO₂ relates to size effects of nanoscaled crystals and the influence of the surrounding matrix.

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O/P.26**STUDY OF GROWTH MECHANISM IN CADMIUM SULFIDE NANOCRYSTALS**

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In the present work, we report the study of growth kinetics in CdS nanocrystals. The growth kinetics was monitored using UV-absorption and small angle X-ray scattering. We show that the kinetics follow the Lifshitz-Slyozov-Wagner theory for Ostwald ripening. This was verified not only by following the size dependence on time but also the dependence of the size distribution and the rate constant.

O/P.27**STRUCTURAL AND DYNAMICS PROPERTIES OF BARIUM MONOXIDE: A MOLECULAR DYNAMICS STUDY**

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The structural and dynamical properties of crystalline BaO were simulated using isenthalpic-isobaric molecular dynamics technique. It is proposed an effective interaction potential which consists of an effective pair potential taking into account atomic size effects, charge-charge, charge-dipole and dipole-dipole interactions. Two and three body correlations were analyzed through pair distribution function, coordination number, and bond angle distribution and compared with experimental available data. The effect of hydrostatic pressure and temperature on the structural and dynamical properties as volume change, structural phase transitions, vibrational density of states, phonon anharmonicity, dynamic Debye-Waller factor and thermal expansion coefficient are correctly described, in good agreement with the available experimental results.

O/P.28**MODIFICATION OF THE Fe-ENVIRONMENT IN Fe₂O₃ GLASS/GLASS CERAMIC SYSTEMS CONTAINING Pb, Na AND Si**

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The effect of composition and casting temperature in the microstructure and the oxidation state of Fe in a series of glass/glass ceramic materials that contain Fe₂O₃, PbO, Na₂O and SiO₂ is studied by means of X-ray fluorescence mapping (XRF), micro- and conventional X-ray absorption fine structure (XAFS) spectroscopy, ⁵⁷Fe Mössbauer spectroscopy and X-ray diffraction (XRD). The Mössbauer and XRD results revealed that the binary systems Fe₂O₃-PbO and Fe₂O₃-Na₂O are glass ceramic materials that contain (Fe₂O₃)_x-PbO ($x=1,2$ and 4), where Fe is octahedrally coordinated, and Na₃Fe₅O₉ crystallites, respectively. The ternary systems Fe₂O₃-Na₂O-SiO₂ are homogeneous vitreous materials when the concentration of Fe₂O₃ is below 40 at%. The XRF maps and ⁵⁷Fe-XAFS spectra, recorded at the KMC-2 monochromator at BESSY, reveal that Fe is inhomogeneously distributed in the ternary samples that contain 50% Fe₂O₃. More specifically, Fe is octahedrally coordinated in islands with high Fe-content, i.e. participates in the formation of γ -NaFeO₂ and Fe₂O₃ microcrystallites. In the glass matrix, where the Fe concentration is lower, Fe is tetrahedrally coordinated and acts as a glass former.

O/P.29**PHOTOLUMINESCENCE CHARACTERIZATION OF SOL-GEL PREPARED LOW DENSITY SILICA SAMPLES**

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Due to the close resemblance of the photoluminescence (PL) of porous silica (PS) to the one observed in oxidized porous silicon [1], sol-gel synthesized PS is a suitable candidate to study the optical properties of nanometric Si compounds and their relation with the chemical and physical properties of the surface. In this work we investigated the PL properties of PS by exciting with synchrotron radiation in the 4-10 eV range silica samples in the 0.6-1.2 gcm⁻³ low density range. In order to establish the nature of the emissions at 2.8 and 3.7 eV, PL, excitation of PL (PLE) and time decay measurements were performed at different temperature. Beside the previous emissions, a PL band at about 1.9 eV excited in the vacuum ultraviolet (8.9 eV) were recorded.

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O/P.30**ELECTRONIC BAND STRUCTURE AND OPTICAL PROPERTIES OF SOME DOUBLE METAL PHOSPHATE MATERIALS**

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Double alkaline and other metal (aluminum, indium, titanium) phosphates like to MAI(In)P2O7, MTiOPO4, MTi2(PO4)3 (M is Na, K), M3Ti3P5O20 (M is K, Rb) types are well known useable materials. Others of them up today are only perspective multifunctional materials revealing a variety of very useful optical, electro physical (NASICON-structure superionic conductors) properties, etc. Some of noted phosphates can be used for confinement of radioactive waste and fission products, and then own and impure luminescence of these materials, which characteristic are determined by electronic structure of material, can be applied for monitoring of their destroying caused by radioactive irradiation.

No doubts, in all of mentioned cases data about electronic energy structure of these matrixes are very important. Therefore, we worked out investigation of their electronic energy states using radiation in wide region: from visible light to X-rays including synchrotron radiation. So, luminescence of noted above compounds at UV, visible light, synchrotron radiation and X-ray excitations was studied. Excitation and reflection spectra were obtained and analyzed as well. These data are compared with calculated data on electronic band structure which were obtained by us as result of theoretical study by means of full-potential LAPW method using WIEN2k program code [1]. Experiments with synchrotron radiation were made on SUPERLUMI station in HASYLAB (DESY, Hamburg) with support of STCU project No. 2042.

[1] P. Blaha, K. Schwarz, G. Madsen, D. Kvasnicka and J. Luitz, WIEN2k, An Augmented Plane Wave Local Orbitals Program for Calculating Crystal Properties (Karlheinz Schwarz, Techn. Universität Wien, Austria), 2001. ISBN 3-9501031-1-2

O/P.31**RESONANT INELASTIC X-RAY SCATTERING APPLIED TO THE ELECTRONIC STRUCTURE OF STRONGLY CORRELATED SOLIDS**

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Recent progress in material science made available systems having new or unexpected properties such as high-temperature superconductivity or metal-insulator transition. A detailed understanding of the electronic structure of such systems is of primary importance for the physics driving these properties and for technological motivations. Thus innovative techniques allowing a detailed investigation of their electronic structure are needed. X-ray emission spectroscopy has been used in the past to probe the bulk occupied electronic states of solids with element selectivity. The high brilliance of modern synchrotron radiation sources makes it possible to perform resonant X-ray emission spectroscopy, or resonant inelastic x-ray scattering (RIXS), i.e., to study the energy dependence of the radiative decay of an atomic core electron excited into a specific empty state. Although the experiments under high resolution conditions are still difficult, new information concerning the electronic structure is obtained. Emphasis will be placed on the advantages of RIXS to unravel electron correlation effects in complex materials. Metal-insulator behaviour in transition metal oxides or mixed valency in rare-earth compounds will be used to illustrate information gained from resonant inelastic scattering in the intermediate-energy and low-energy X-ray range.

O/P.32**MODELING THE DIFFRACTION PROFILES OF CVD-GROWN PEROVSKITE OXIDE MULTILAYERS**

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A major issue is the integration of electrical/magnetic functions into thin oxide layers and multilayers. Such functional systems can be synthesized by the chemical vapor deposition of the organo-metallic precursors (MOCVD) and pulse laser deposition (PLD). One case of study is the (BaTiO₃/SrTiO₃)_n epitaxial multilayer which exhibits a high dielectric constant and low dielectric losses, important in microelectronics and communications (DRAM memories, condensators...).

We have examined by laboratory and synchrotron X-ray techniques (reflectivity and high angle diffraction) BaTiO₃/SrTiO₃ multilayers deposited on c-oriented LaAlO₃ and SrTiO₃ substrates. The thickness of the single layers is varied between 25 and 200 Å. The periodicity of the multilayers ranges from 70 to 200 Å, with different Sr/Ba ratios. Synchrotron radiation experiments were carried out at the European Synchrotron Radiation Facility at BM2. A key issue in the structural characterization of such systems is the description of the lattice deviations and defects. We have extended a diffraction model (Fullerton et al. Phys. Rev. B45, 9292 (1992)) previously applied on metallic multilayers, to the more complex case of perovskite structures. The entire diffraction profiles of the (BaTiO₃/SrTiO₃)_n superlattices are fitted over 8 orders of diffraction. By the means of a computed refinement procedure, we evaluate the presence and the magnitude of the coherence length, the interface roughness, the discrete thickness fluctuations, and the intra-layer gradients of strain and atomic diffusion. We show that the diffraction profiles are highly sensitive to the interface state, in particular the macro-strain profile (lattice parameter gradient) as well as the atomic composition profile.

O/P.33**LOCAL STRUCTURE AND DYNAMICS AROUND IODINE IN AgI-Ag₂MoO₄ GLASSES**

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Multi-component glasses (borate, phosphate, molybdate,...) containing both Ag₂O (modifier) and AgI (dopant) are quite interesting for their potential utilisation in the field of solid electrolytes. When embedded in the binary glass matrix, silver iodide gives rise to an amorphous network of tetrahedral units where I⁻ anions are surrounded by 4 Ag⁺ cations. The influence of different glass matrices on the conducting properties of AgI is one of the open problems in the field. In this work we present an EXAFS study at the K-edge of Iodine of glasses (AgI)_{0.67}:(Ag₂MoO₄)_{0.33} and (AgI)_{0.75}:(Ag₂MoO₄)_{0.25} as a function of temperature. In both glasses the I-Ag bond length exhibits a contraction as a function of increasing temperature, similarly to recent results found by us in silver borate glasses. At low temperature the I-Ag bond length is about 0.05 Å larger than in crystalline beta-AgI. When the glass transition temperature is achieved (310-350 K), the I-Ag distance shrinks more rapidly in both glasses, leaning towards the crystalline I-Ag distance.

Among the AgI-based silver oxide glasses, silver molybdates are quite different from borates, because of the absence of a continuous network. These results indicate that the anomalous negative thermal expansions of the I-Ag first shell distance is present independently on the components of the glass matrix and may be related to the fast ion diffusion properties of Ag ions in the AgI rich amorphous regions of the glasses. These new experimental results open new perspectives for the study of the local origin of thermodynamic properties of glasses.

O/P.34**RESONANT PHOTOEMISSION AT THE 2p EDGE IN COMPOUNDS CONTAINING Mn WITH DIFFERENT VALENCIES**

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The electronic structure and the electron dynamics in Mn containing systems has to be better understood for fundamental and technological reasons. Useful information can be obtained on both points from resonant photoemission measurements at the Mn 2p threshold [1-3]. In the present experiments, we have investigated the Raman to normal Auger crossover of non-radiative Mn 2p decay in MnO and KMnO₄ which can be considered as having a half-filled and an empty 3d shell, respectively. For the Mn 2p-3p3p decay in MnO, Raman behaviour is observed up to 3.4 eV above the resonant energy (maximum of the L₃ absorption) for the highest kinetic energy transition contained in this feature. A similar situation occurs up to 5.3 eV above resonant energy, which is the highest detuning used in this series of measurements, for the 2p-3p3d and the 2p-3d3d decays. We found that for the latter case the Auger signal far above the resonant energy ($h\nu = 800$ eV) has 2.5 eV less kinetic energy than the decay electrons at 5 eV above resonance, where we find the highest kinetic energy for this decay channel. Thus two main conclusions can be drawn: - the persistence of Raman behaviour far above resonance is rather unusual; - the differences in the Raman to normal Auger crossover for the three decays indicates that the decay process is very sensitive to the final state, being kept in mind that the intermediate state is the same for all the three decay channels studied. For KMnO₄, we observe completely different behaviour: here the Raman-Augur crossing happens 2-3 eV below resonance.

[1] M.C. Richter et al, Phys. Rev. B 63 (2001) 205416.

[2] S. Hüfner et al, Phys. Rev. B 61 (2000) 12582.

[3] M.C. Richter et al, J. Alloys and Compounds 362 (2004), 41.

O/P.35**LOCAL STRUCTURE AND MAGNETO-TRANSPORT OF Sr₂FeMoO₆ OXIDES**

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Sr₂FeMoO₆ are half-metallic ferromagnetic with elevated Curie temperature and high magnetoresistance even at room temperature. In the last few years these compounds have stimulated a large fundamental research interest in the field of heavily correlated systems. The crystallographic structure of Sr₂FeMoO₆ and related compounds belong to the double perovskite system of general formula A₂BB'O₆. Their structure is made of distorted pseudocubic units with Sr ions at the cube centers and B(B')O₆ octahedra on the corners. The arrangement of Fe and Mo ions on B and B' sublattices (chemical order) plays a fundamental role in determining the magnetotransport properties: in ordered compounds Fe and Mo alternates on the B and B' sublattices, this produces huge magnetoresistive response in low fields and high saturation magnetization. In disordered compounds Fe and Mo indifferently occupy B and B' sublattices, this depresses the MR and magnetization. The degree of long range chemical order in these compounds is usually quantified by x-ray diffraction (XRD) as the intensity of superlattice reflection is directly related to the order parameter. In this work the local structure around Fe and Mo ions in chemically ordered and disordered compounds, has been investigated by XAS. The accurate analysis of the EXAFS signals collected at the Fe and Mo k-edges reveals interesting and unexpected results: Fe-O-Mo configurations are largely preferred with respect to Fe-O-Fe and Mo-O-Mo either in the ordered and disordered compounds. These findings prove that the short range order is preserved, despite the long range chemical order features suggesting that the disordered samples are made by chemically ordered regions which short coherence length (few tens Å) prevent the detection by XRD.

O/P.36**CORRELATIONS BETWEEN THE LUMINESCENCE AND STRUCTURAL PROPERTIES OF NANODOTS IN SILICON-RICH NITRIDE BY X-RAY ABSORPTION SPECTROSCOPY**

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The luminescent silicon-rich nitride thin films are successfully deposited on single crystalline silicon wafer substrates at 300°C by using the electron-cyclotron-resonance chemical-vapor-deposition (ECR-CVD) technique with SiH₄-Ar-N₂ mixture gases. The photoluminescence peak energy emitted from these samples can be tuned from the blue to the near-infrared by adjusting the degree of silicon richness. The synchrotron radiation extended X-ray absorption and near-edge absorption spectroscopy measurements demonstrated the formation of the silicon clusters in the silicon-rich nitride films. It is noted that no obvious bond length variation could be found in the samples with different degree of silicon richness. From the high-resolution cross-sectional transmission-electron-microscopy images, some small (<3nm) regions can be seen which are slightly darker, however, does not show lattice fringes. The images in the flimsy edge of samples exhibit clear lattice fringes and the clusters with size about 1-3 nm can be clearly identified. These observations demonstrate the existence of the nano-crystalline silicon clusters in the luminescent silicon-rich samples. The near-edge feature of synchrotron radiation excited visible luminescence yield spectrum shows that the co-existence of Si-Si and Si-N luminescence sites. This observation combined with microstructure characterizations strongly suggests that silicon cluster embedded in the silicon nitride matrices model seems to provide the only viable explanation on the origin of photoluminescence.

O/P.37**ELECTRONIC STRUCTURE IN CHROMIUM ALUMINUM OXYNITRIDE THIN FILM**

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The electronic structure of the chromium aluminum oxynitride has been investigated using resonant photoemission spectroscopy (RPES) and discrete variational (DV)-Xa method. The RPES measurement around the Cr 2p_{3/2} absorption edges exhibited remarkable resonant interference behavior for Cr 3d valence electrons, whereas small resonant interference behavior for N 2p valence electrons. Therefore this RPES method can be useful to analyze the valence band of chromium aluminum oxynitride film. The top of the valence band is consisted of predominant Cr 3d and of small N 2p. The difference between the measured PES spectra of the valence band and DV-Xa calculation of chromium aluminum oxynitride is the range from 0.47 to 3.62 eV. This difference should be caused by the Coulomb interactions between d electrons of chromium and the structure character of amorphous film. Through the experimental and theoretical study, the valence band structure of chromium aluminum oxynitride was figured out in detail.

O/P.38**A NEW NITROGEN-HYDROGEN COMPLEX IN GaAsN REVEALED BY X RAY ABSORPTION SPECTROSCOPY**

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GaAsN alloys belong to a novel class of semiconductors with fascinating physical properties. Indeed, a small amount of nitrogen incorporation in GaAs leads to a counterintuitive and large band gap reduction and to an unexpected sudden increase in the effective mass of electrons. Even more surprisingly, both electronic and structural changes can be reversed fully and in a tunable manner by hydrogen incorporation. In this paper, we combine in an original way X-ray absorption spectroscopy at the nitrogen K-edge with ab initio simulations to investigate the atomic geometry of N-H complexes in hydrogenated GaAsN. In this way, we provide experimental evidence that a dihydrogen-nitrogen complex with C_{2v} symmetry is the most abundant species in hydrogenated GaAsN. This finding contradicts all previous predictions of "in-line" N-H₂⁺ complexes as the predominant species, and accounts for recent infrared absorption experiments.

O/P.39**IMPROVEMENT OF NITROGEN INCORPORATION INTO OXYNITRIDES ON 4H-SiC(0001)**

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Thin films of oxynitrides are grown on 4H-SiC(0001) surfaces as to prepare these surfaces for further growth of gate dielectric layers. Our former investigation indicated two major problems by thermal treatment in N₂O gas (see [1]). First: Even under optimised preparation conditions the nitrogen incorporation is very low (at the best around 7 atom-%). And second: The preparation condition optimised for nitrogen incorporation (high temperature and low N₂O pressure) leads to very thin layers (in the order of 1..2 mono-layers only) and to graphite formation. Here we report on a surface pre-treatment to overcome these problems. The pre-treatment consists of the evaporation of Si onto the SiC surface. On the one hand the additional Si prevents the formation of the graphite by compensation of the loss of Si due to SiC decomposition and Si evaporation. On the other hand the excess Si improves the incorporation of N by acting as a sacrificial layer for the growth of oxynitride. Thereby the same high amount of N can be incorporated like observed on Si wafers [2]. The as prepared SiC surfaces and oxynitride layers were investigated by STM to check the crystallinity of the surface and by photoelectron spectroscopy to examine the chemical composition of both, the SiC surface (graphite formation) and the oxynitride layer (nitrogen incorporation). [1] P. Hoffmann, A. Goryachko, D. Schmeißer: "Oxynitrides on 4H-SiC(0001)", Materials Science & Engineering B, in press. [2] P. Hoffmann, R.P. Mikalo, D. Schmeißer: "Si(001) Surface Oxidation by N₂O" J. Non-Crystall. Solids 303 (2002) 6-11.

O/P.40**ULTRATHIN LAYERS OF GaN ON GaAs(100) AND InN ON InP(100) GROWTH STUDIED BY SYNCHROTRON RADIATION**

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The nitridation of InP(100) and GaAs(100) surfaces has been studied using synchrotron radiation photoemission. In the case of InP(100) surfaces, the nitridation with a Glow Discharge Cell (GDS) produced indium nitride by reaction with indium clusters created during ionic cleaning. In the case of GaAs(100) surfaces, a deposition of one monolayer of metallic gallium was achieved prior the nitridation. We used the In4d, P2p, Ga3d and As3d core levels to monitor the chemical state of the surface and the coverage of the species. A theoretical model based on stacked layers allows to assert that almost two monolayers of indium nitride or gallium nitride are produced. For InN/InP(100) structure, the effect of annealing on the nitridated layers at 450°C has been also analysed. It appears that this system is stable until this temperature, well above the congruent evaporation temperature (370°C) of clean InP(100) : no increase of metallic indium bonds due to decomposition of the substrate is detected as shown in previous works studying the InP(100) surfaces.

O/P.41**LOCAL ATOMIC AND ELECTRONIC STRUCTURE OF HIGH DIELECTRIC CONSTANT ULTRA THIN FILMS ON Si(001)**

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Yb, Lu, and Hf oxides are under consideration as high dielectric constant (k) materials capable of substituting SiO₂ in ultra-scaled complementary metal oxide semiconductor(CMOS)devices; such materials reduce tunneling currents because devices with physically thicker films can be fabricated while keeping an acceptable capacitance. The successful growth of ultra thin high-k oxide films on Si was already reported, however, an understanding of their bulk and interface structures, and of their physical properties is missing. To study the local atomic and electronic structure of Lu₂O₃, Yb₂O₃, and HfO₂ ultra thin films grown by Atomic Layer Deposition[1] (ALD) on Si, we performed X-ray absorption (XAS) experiments at both the cation L and O K absorption edges, also using a grazing incidence geometry[2]. The objective was to study the bulk and interface structures as a function of the deposition parameters and annealing processes. The method was already successfully applied in the study of the Y₂O₃/Si(001)[3].

Ultra thin films (2 to 10 nm thick) were grown using both H₂O and O₃ as oxygen sources. For the Lu₂O₃ films, a newly synthesized complex, the dimeric [(h₅-C₅H₄SiMe₃)₂LuCl]₂, was used as Lu precursor. By a combined analysis of the near edge and extended parts of the XAS spectrum, we obtained a comprehensive description of the film atomic and electronic structures. Bixbyite structure is preserved in the thicker films, but significant disorder is apparent in the thinnest ones. Films prepared using H₂O and O₃ show clear differences in the electronic structure (in particular in the vicinity of the conduction band minimum).

[1]G.Scarel et al,Appl.Phys.Lett.85,630(2004).

[2]F.D'Acapito et al.,J.Synch.Rad.10,260(2003).

[3]M.Malvestuto et al.,accepted on Phys.Rev.B(2005).

O/P.42**THE APPLICATION OF X-RAY MICROPROBE TO PROBLEMS IN HARD, WIDE-BANDGAP SEMICONDUCTORS**

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Diamond, gallium nitride, and aluminum nitride are of great potential in the development of novel electronic devices, owing to their wide-bandgap, polarization-based effects, and superior mechanical, magnetic and thermal properties. Recent advances in the crystal growth techniques have enabled numerous studies on doping effects via X-ray microprobe. Here, we focus on the use of spectroscopic techniques such as X-ray fluorescence (XRF), X-ray absorption near-edge structure (XANES), as well as X-ray diffraction (XRD) on the micrometer scale to characterize various physical aspects. For instance, impurity segregation effects by means of elemental imaging on Si doped AlN. Depending on the initial atomic sequence (N-rich or Al-rich growth conditions), the spatial distribution of Si on AlN presents droplets with diameter in the range of 5 -10 m. In the same way, a combination of XRF mapping and angular dependent-XANES techniques applied to Mn doped GaN enabled us to investigate not only the Mn cluster formation, but also its crystalline structure. Additionally, conventional analytical methods like laser-excited luminescence and Raman scattering are used to support our findings. Finally, the influence of nitrogen on 400 nm ultra-nano-crystalline diamond films is examined by micro-XRD. Using the Scherrer's formula, an estimation of the average grain size is reported as a function of the nitrogen content. To close, a new microprobe approach based on X-ray excited luminescence is introduced, demonstrating that luminescence mapping and spectroscopic analysis on semiconductor layers can be also achieved with elemental sensitivity on a microscopic scale.

O/P.43**ANALYSIS OF ORDERING IN ALXGA1-XAS USING X-RAY DIFFRACTION**

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In the present study the quantity of the variant III CuAu-I-type ordering of Al_xGa_{1-x}As grown on (110), (111)A and (001) GaAs substrates is determined for different temperatures and Al fractions. For this purpose Al_xGa_{1-x}As films were produced by metal organic chemical vapour deposition and analysed using X-ray diffraction at the European Synchrotron Radiation Facility in Grenoble.

It is found that the ground state of Al_xGa_{1-x}As is given by the CuAu-I-type structure, followed by the metastable disordered phase. The samples with an Al fraction of $x = 0.5$ typically have the highest ordering degree. This agrees well with the fact that the CuAu-I-type is a superstructure of AlAs and GaAs monolayers. Generally, a high degree of order is observed for high growth temperatures and low growth rates. With respect to the substrate orientation the ordering is found to be strongest for samples grown on (110) substrates with a maximum long-range order parameter S of 0.07 ± 0.01 , followed by the samples grown on (111)A and (001) substrates with an S of 0.019 ± 0.004 and 0.006 ± 0.003 , respectively. Because of symmetrical considerations, a total S of 0.057 ± 0.007 is expected for the (111)A samples. This is close to the ordering degree of the (110) samples, reflecting the (110)-like surface configuration as a result of the vacancy (2x2) surface structure of the (111)A face at higher temperatures.

O/P.44**GRAZING INCIDENCE SYNCHROTRON X-RAY DIFFRACTION INVESTIGATION OF ORGANIC THIN FILMS USED AS ACTIVE LAYERS FOR FETs**

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Organic thin films (made up of small molecules or polymers) are interesting as active layers in electronic and optoelectronic devices such as field-effect transistors (FETs), light-emitting diodes (LEDs) and photovoltaic cells thanks to their multifunctionality, i.e. the possibility to combine good electrical and optical properties. It has been demonstrated that in organic film-based FETs, the transport properties depend strongly on the orientations of the ordered microcrystalline domains. Moreover, the charge-carrier transport occurs within a thin layer next to the organic-gate dielectric (i.e. the substrate where the film is deposited) interface.

To shed light onto charge-carrier transport in this class of devices, both the study of film growth from the early stages and the control of the quality of the organic-substrate interface are therefore of primary importance. We focused our attention on tetracene thin films, vacuum-sublimed on a SiO₂ substrate, employed as active layers in organic FETs. By means of grazing incidence synchrotron X-ray diffraction we investigated the structural properties of films obtained with increasing nominal thickness at different deposition conditions (deposition rate, substrate surface treatment). We investigated a possible correlation between texture of the thin films and transistor electrical properties.

O/P.45**SEMICONDUCTOR MATERIALS BEHAVIOR UNDER IONIZING CONDITIONS**

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Using semiconductor materials under ionizing conditions for complex structures which use different types of logical elements needs to analyze the reliability of active elements and integrated circuits for different type of irradiation doses. The main problem consists in determination of maximum level of continuous irradiation for a stabile behavior of integrated circuits. The design of electronic structures based on semiconductor devices needs a prediction for parameters variation of active elements during irradiation process. The usual approximation of this prediction consists in combination of laboratory testes with electrophysical models, the results may be used for prediction of all integrated systems.

O/P.46**CORE-LEVEL PHOTOABSORPTION STUDY OF Si-INCOPORATED TETRAHEDRAL AMORPHOUS CARBON THIN FILMS**

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This report investigates the C *K*-edge x-ray absorption fine structure, NEXAFS, spectra of Si-incorporated hydrogenated tetrahedral amorphous carbon (ta-C:Si:H) films (up to 23 at.% Si) deposited by filtered cathodic vacuum arc (FCVA) system under varying tetramethylsilane (Si(CH₃)₄; TMS) precursor gas pressure. The relative change in the area ratio of π^*/σ^* resonance absorption peaks of NEXAFS spectra, located respectively at 285.3eV and 289eV, has been used for determining sp²-bonding in the films. The results point out a slight increase in sp² sites with Si-addition up to 15 at.% Si before it saturates with further increase in Si%.

Moreover, an *in-situ* rapid thermal annealing, RTA, has confirmed a graphitisation process caused by Si- and hydrogen-additions. The π^*/σ^* peak ratio increased by thermal annealing at 700°C from 0.25, 0.3, and 0.35 to 0.35, 0.5, and 0.6 for ta-C:Si:H samples with silicon content of 0.5, 9.8, and 21.5 at.%, respectively. It is believed that the evolution of hydrogen during thermal annealing process, as revealed by ERDA measurements, was responsible for the observed thermal instability.

The presence of local order in ta-C:Si:H films was observed in angle dependent NEXAFS. Rotation of the specimens with respect to the incident photon beam, showed a measurable angular dependence suggesting that the π orbitals are partially oriented. The π_{C-C} resonance intensity decreases from normal to glancing incidence, while the σ_{C-H}^* exhibits an opposite trend. The observed opposite angular dependencies reveal that the σ_{C-H}^* orbitals are orthogonal to the π orbitals.

- O/P.47** GROWTH OF AG ON MGO(001) STUDIED IN SITU BY GRAZING INCIDENCE SMALL ANGLE X-RAY SCATTERING
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Nanometric size islands on a substrate or buried particles display fascinating properties for both basic and applied research. They include catalysts and semiconductor quantum dots whose properties are driven by the particle morphology. In this context, Grazing Incidence Small Angle X-ray Scattering (GISAXS) is a unique technique that allows to determine the morphology of such particles in a non-destructive way. Performing in situ GISAXS is particularly well suited to investigate the morphology of a deposit that grows 3D on a substrate, when the use of imaging techniques is difficult, which is the case for many metal/oxide interfaces (the AFM tip often moves the metal clusters, and STM can not be used on insulating substrate). The Ag/MgO(001) system is characterized by a Volmer-Weber (3D) growth mode, and has been thoroughly studied to investigate the elementary processes of heterogeneous nucleation, growth and coalescence. In situ GISAXS patterns of Ag/MgO(001) were recorded on a 2D detector during growth in ultra high vacuum at the European Synchrotron Radiation Facility (ESRF). The growth of Ag on MgO(001) surfaces was studied at different temperatures, in order to determine the growth laws. A precise analysis of the GISAXS data will be presented as well as the analysis of the growth by kinetic Monte-Carlo.
- O/P.48** CHANGE OF HETEROSTRUCTURES CONTACT POTENTIAL DIFFERENCE INDUCED BY X-RAY IRRADIATION
Z.D. Kovalyuk(a), V.M. Katerynychuk(a), O.A. Politanska(a), O.M. Sydor(a), A.I. Savchuk(b), M.D. Raransky(b), and M.Yu. Palamaryk(b), (a)Chernivtsi Department of the Institute of Materials Science Problems, The National Academy of Sciences of Ukraine, 5, Iryna Vilde St., 58001 Chernivtsi, Ukraine, (b)Chernivtsi National University, 2, Kotsyubynsky St., 58012 Chernivtsi, Ukraine
The characteristic X-ray radiation influence on the intrinsic oxide-p-InSe and p-n-InSe heterostructures photoelectric parameters is established. This influence results in increasing the potential barrier between the contacting semiconductors. It is shown that with the dose radiation increasing the barrier magnitude approaches to the maximal possible value – a half of the energy gap of the base material.
The oxide - p-InSe heterostructures were formed by thermal oxidation of InSe substrates. P-n-InSe structures were prepared by the optical contact method between thin layer plates from n- and p-InSe separately grown crystals. The peculiarity of these layered crystals is a possibility to cleave substrates of micron thickness with mirror-like quality of the surfaces. The current-voltage, capacitance – voltage and spectral characteristics of the prepared heterostructures as well as the current transfer mechanisms through the potential barrier before and after X-ray irradiation were investigated. The obtained results are explained by a change of the acceptor concentration in InSe after irradiation.
- O/P.49** IN SITU X-RAY SYNCHROTRON STUDY OF ORGANIC SEMICONDUCTOR ULTRATHIN FILMS GROWTH
J.-F. Moulin, F. Dinelli, M. Massi, C. Albonetti, R. Kshirsagar, F. Biscarini, Research Section on Nanotechnology of Multifunctional Materials, CNR-ISMN - Bologna Division (former Istituto di Spettroscopia Molecolare), Via P. Gobetti 101, 40129 Bologna, Italy
We present here the results of a X-Ray diffraction study of the evolution of the structural organization of an organic semiconductor (sexithiophene, T6) during the early stages of growth of a high vacuum sublimed thin film. We have studied, both by specular reflectometry and grazing incidence x-ray diffraction, the formation of T6 films on substrates of varying hydrophilicity (silicon oxide and H-passivated silicon). Our results show that T6 grows as a crystalline layer from the beginning of the evaporation. The reflectometry analysis suggests that, in the range of rates and temperatures studied, the growth is never layer by layer but rather 3D in nature. In plane GIXD has allowed us to observe for the first time a thin film phase of T6 formed of molecules standing normal to the substrate and arranged in a cell that displays b-c parameters that are contracted with respect to the bulk. We have followed the dynamics of formation of this new phase and identified the threshold of appearance of the bulk phase, which occurs above c.a 5-6 monolayers. These results are highly relevant to the problem of organic thin film transistors (OTFTs), for which we have previously demonstrated experimentally that only the first two monolayers of T6 films are involved in the electrical transport. The layers above the second one do not effectively contribute to charge mobility, either because they are more "disordered" or because of a screening of the gate field.
- O/P.50** CHARACTERIZATION OF THERMALLY TREATED MO/SI MULTILAYER MIRRORS WITH STANDING-WAVE-ASSISTED EXAFS
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In the last years EUV multilayer mirrors have reached high reflectivity performances, leading such optics suitable for new generation lithography and astrophysical applications. One of the most studied systems is the Mo/Si but it is still challenging to understand the composition of the interlayers. This work aims to test the standing-wave-assisted EXFAS as a possible technique to investigate interface composition.
Samples are 40- and 10-periods Mo/Si multilayers deposited on silicon substrates by RF magnetron sputtering and ion assistance characterization. Thermal annealing in vacuum at temperatures between 240°C and 400°C permits to obtain interface thickness in the range of 1-2nm whereas annealing at 600°C dissolves the multilayer structure. XAFS measurements are carried on these samples. Different data collection techniques are compared: standing-wave excitation, total reflection, fluorescence and total electron yield. Particular attention is given to standing-wave-assisted EXAFS since it allows in principle depth-resolved investigation of the short-range order of Mo/Si multilayers. In fact, changing the angle of incidence in the vicinity of the first-order multilayer Bragg reflection varies the contribution of different regions. The study clearly shows the presence of molybdenum silicide (MoSi₂) interdiffusion layers. Significant structural changes are observed increasing the annealing temperature and the MoSi₂ percentage rise from 14% (as deposited) to 70% (600°C). The results are confirmed by XRR and μ -XRD patterns.

O/P.51**THE Fe/NiO INTERFACE STUDIED BY XAS**

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Coupled ferromagnetic (FM) - antiferromagnetic (AFM) films constitute one of the active elements of new magneto-electronic devices, exploiting the electron spin rather than its charge for information processing. The exchange interactions at the interface between the FM and the AFM layer result in a unidirectional magnetic anisotropy, the so called exchange bias, a quantitative explanation of which has yet to be attained. The theoretical models of different FM-AFM systems often assume an abrupt interface, whereas some experimental studies show that the magnetic behaviour is extremely sensitive to the details of the interfacial roughness, diffusion, chemical state and geometrical structure.

We present here an X-ray absorption study of the epitaxial Fe/NiO(001) interface, performed at the Fe K edge. Taking advantage of the linear polarization of the photon beam it was possible to study the interface structure in the directions parallel and perpendicular to the substrate; 2 ML and 10 ML samples were studied. The data show an epitaxial growth of the iron film which implies a tetragonal distortion of the crystal structure. The first atomic plane of the iron film in contact with the substrate is found to be oxidized, with a crystal structure similar to antiferromagnetic FeO. At increasing Fe thickness the film keeps a strained structure without further oxidation of the metal. At a thickness of 10 ML strain release occurs. This work reports a complete picture of the chemistry and structure of the Fe/NiO interface at the atomic scale, and provides the structural basis necessary to model the magnetic couplings of the system. In particular, the structure of the very first stage of the film growth is elucidated.

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O/P.52**SYNCHROTRON RADIATION-INDUCED PHOTOEMISSION AS A PROBE TO INVESTIGATE ORGANIC ADSORBATES ON SEMICONDUCTOR SURFACES**

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In this work the potentialities of synchrotron radiation-induced photoemission as a probe to investigate the interaction of organic molecules and silicon surfaces are briefly reviewed. The adsorption of a selected number of organic molecules with increasing structural complexity on Si(100)2x1 and Si(111)7x7 is here reported as representative.

The combination of high resolution monochromators at reasonable flux and tunable photon energy allows a detailed analysis of the photoemission spectral features related to surface atoms. The core-level line-shape analysis provides useful information on the adsorption nature (physisorption/chemisorption) and type (molecular/dissociative), the site-selectivity and the nature of adsorbed species. Examples are given concerning the adsorption of hydrocarbons, alcohols and bi-functional molecules on Si(100)2x1. Valence band photoemission provides information on the surface states, the adsorption-induced features and the adsorbate molecular orbitals, showing whether possible energy shifts or even a removal of orbital degeneracy occur. The transition from benzene physisorption to chemisorption on Si(111)7x7 as a function of the temperature is reported as firstly evidenced by photoemission spectroscopy. The photon beam polarization allows to address another topic of great current interest, i.e. the identification of chiral environments on surfaces. The study of chiral molecules on surfaces (e.g. 2,3-Butanediol on Si(100)2x1) using circular dichroism in core level photoemission has considerable potential to provide an atoms-specific identification of chiral centers in molecules.

O/P.53**TEMPERATURE DEPENDENT ABSORPTION BEHAVIOUR OF DIETHYNYL-THIOPHENE ADSORBED ON Cu(100) INVESTIGATED BY NEXAFS AND XPS**

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Penta atomic aromatic heterocyclic compounds are the starting base for the synthesis of several organic conducting polymers. Among others, conjugated poly-ynes containing bonded transition metals in the main chain, show interesting optical and electronic properties due to the pi-conjugation of the π -bonded acetylenic spacer that is preserved through the metal. Pt-DET (Pt-diethynyl thiophene) has been synthesized by some of us and was investigated as sensitive membrane for chemical sensors. The here reported investigation is related to the polymer/metal interaction, important for the study of the performance of electronic devices.

The study of the constituent building blocks is a reliable approach to better understand the electronic properties of polymers. In the framework of this research program, the penta atomic aromatic heterocyclic ring DET was previously investigated by XPS and NEXAFS at the C1s and S2p edges in gas phase and experimental results have been compared with calculated data. Here we report data concerning the electronic and molecular structure of multilayer and monolayer films of DET adsorbed at 90 K on Cu(100) and their temperature-dependent modifications, investigated by angular dependent NEXAFS spectroscopy at the C-K edge. The temperature was increased step by step and the NEXAFS spectra were recorded at 90K, 150K, 200K, 245K and 298K. Comparison with XPS data collected on DET at similar temperatures was also accomplished. The achieved results suggest an interaction between Cu and the C atom bonded to S, with a charge transfer from the Cu and a partial desulfurization reaction that takes place at about 298K; temperature dependent measurements recorded during the desorption process suggest an increase in molecular packing above 130K.

- O/P.54** ELECTRONIC STRUCTURE OF Be(0001) SURFACE AS SEEN BY ARPES WITH VARIABLE POLARIZATION SYNCHROTRON RADIATION
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We measured the electronic structure of the Be(0001) surface using high-resolution angle resolved photoemission (ARPES) with variable polarization synchrotron radiation. We used photons of 32.5 eV and 86 eV delivered by the beamline APE at Elettra, with the two orthogonal linear polarizations. The electronic band structure along the two (GK and GM) high symmetry directions was probed and the Fermi surface cuts were measured. As a function of photon polarization either states of even or odd symmetry with respect to the scattering plane (defined by the direction of light and the propagation direction of the measured outgoing electrons) can contribute to the photoemission intensity. We observe a peculiar change in the symmetry of states when probing different regions of the Brillouin zone with different polarizations. We will discuss the photoemission intensity for Be(0001) by comparing data to the available theoretical results.
- O/P.55** MOLECULAR ORIENTATION OF NEW FUNCTIONAL MIXED AROMATIC MOLECULES DEPOSITED ONTO Au/Si(111): COMPARISON BETWEEN ANGULAR DEPENDENT NEXAFS ANALYSIS AND THEORETICAL CALCULATIONS
C. Battocchio, Department of Chemistry, University of Rome "La Sapienza" P.le A. Moro 5, 00185, Rome, Italy, G. Polzonetti, Department of Physics and unità INFN, INSTM and CISDiC, University "Roma Tre", Via della Vasca Navale 84, 00146 Rome, Italy, A. Licciardello, N. Tuccitto, L. Gambino, G. Marletta, Department of Chemical Sciences, University of Catania, V.le A. Doria 6, 95125 Catania, Italy
Self assembled monolayers (SAM) containing molecules with functionalised tail-groups that can mediate the surface binding of specific molecules, are suitable of different applications, such as active layers in sensors or molecular electronic devices. Among these, terpyridine-based ligands are promising functional species because of their versatility in co-ordinating metal cations. The shape and charge of the species the ligands have to accommodate make their lateral spacing mandatory. A good approach is to alternate the ligand moieties with molecules acting as lateral spacers. In a previous paper some of us have characterised a mixed component system, made out of a substituted terpyridine (MPTP) and mercaptobenzene (MB) as a spacer. The MPTP/MB layer on Au assume a surface composition independent of the ratio of the two constituents in the solution used for the self assembling. In this framework, angular dependent NEXAFS investigation on the same functional mixed monolayer deposited on Au/Si(111) was carried on. The individual systems MPTP and MB, deposited on gold as well, have been used as standards. MB forces the active MPTP to an organized molecular arrangement. Analysis of the angular dependence of few selected resonances of both the C K-edge and N K-edge spectra, the $\pi^* \text{C}=\text{C}$, $\pi^* \text{C}=\text{N}$ and $\pi^* \text{N}-\text{C}$, for MPTP/MB and for the separated, suggested that in the MPTP/MB the phenyl rings are more tilted towards a geometry in which they stand up respect to the Au surface and nearly coplanar between each other, whilst in MPTP itself the rings are more lying down. Comparison between our results and calculations relative to the adsorption geometry gave good agreement, concerning either the tilting of the pyridine rings induced by MB and the close coplanarity between the rings.
- O/P.56** ENVIRONMENTAL FRIENDLY FLUOROCARBONS FOR PLASMA PROCESSING
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Constant emission of pollutants and especially in plasma-processing technology industries has lead to the concern for the effect of climate change. Political pressures have therefore forced the industry to seek more environmental friendly gases. Traditional feed gases (e.g., SF₆, CF₄) are greenhouse gases that absorb strongly in the infrared and have long residence times in the terrestrial atmosphere (SF₆ is of the order of 1000 years). We make use of synchrotron radiation to study the VUV electronic state spectroscopy of CF₃I and C₂F₄ in order to obtain absolute photoabsorption cross sections and thence estimate the photolysis rates and local lifetimes in the Earth's atmosphere, up to the stratopause. FTIR spectroscopy has been also used to evaluate the radiative forcing. These molecules under electron bombardment are a source of plasma etching radicals (CF_x, x = 1, 2, 3) in SiO surfaces. At the present we are developing a novel experiment where these gases will be used to perform plasma etching and evaluate their role as environmental friendly molecules.
- O/P.57** LUCIA, A MICROFOCUS SOFT XAS BEAMLINE
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The beamline "LUCIA" (Line for Ultimate Characterisation by Imaging and Absorption) is a "tender" (0.8-8keV) X-ray microprobe with capabilities for chemical speciation by x-ray absorption spectroscopy (mXAS) and for elemental mapping by x-ray micro-fluorescence (mXRF). It allows the possibility to measure heterogeneous samples at a micronic size and to combine these two element-specific and non-destructive techniques. A monochromatic beam of mm size is incident on a sample which is carried on a scanning x-z stage. mXRF shows the location of the elements, their relative abundances, and with which other elements they are associated. One can take advantage of the monochromatic beam which allows to separate out different elements by their absorption edges. After mapping the fluorescence, interesting spots can be analysed by XAS to determine the speciation (local chemistry, quantitative determination of the local geometric structure around the absorbing atom) of the elements and how it depends on the different components.
Installed at first on the SLS at the Paul Scherrer Institute (Suisse), the LUCIA beamline will be transferred to SOLEIL by beginning 2008. The energy range offered by the beamline corresponds to the best performances of SLS and SOLEIL in terms of brilliance. It allows XAS experiments at the K edge of elements from Na to Fe, L edges from Ni to Gd, and M edges of rare earths and actinides.

O/P.58**DIFFABS (SOLEIL): A BEAMLINE DEDICATED TO THE STRUCTURAL STUDY OF MATERIALS BY COMBINATION OF XRD, XRF AND XAS**

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The DiffAbs (Diffraction-Absorption) bending magnet beamline of Soleil is constructed by the partial transfer of H10 from LURE. It is dedicated to the structural study of materials combining X-ray diffraction (XRD), X-ray fluorescence (XRF) and X-ray absorption spectroscopy (XAS) using macro- or micro-beam.

The available 3-23 keV energy range ensures the XAS study of K-edges between K and Ru and L-edges between Ru and U. The combination of XAS, wide angle scattering (WAXS) and XRD complements local structure information by data on the long range order. Site- and chemistry specific information is obtained by resonant diffraction spectroscopy (DAFS). In general, the study of anomalous diffraction/scattering will be available. Fast kinetics e.g. phase transitions or self-propagating high temperature synthesis, is investigated by time-resolved X-ray diffraction (ms time-scale). Micro-XRF, micro-XRD and micro-XAS are also offered at DiffAbs with ~10 micron resolution. Scanning micro-XRF gives information about the elemental distribution and inter-element correlations. Combined micro-XRF and micro-XRD gives insight into the correlation between the elemental composition and crystalline structure. The combination of XAS with XRF and/or XRD provides the micro-scale variation of the chemical speciation of an element in the function of the elemental composition and crystalline structure. Two main scientific fields of the beamline are dedicated to the study of the behaviour of materials at high and very high temperatures (from room temperature up to 3000°C) and to the determination of strain and stress in thin films and interfaces. In general, DiffAbs will provide useful research tools in the fields of materials sciences, cultural heritage, geology and environmental science.

O/P.59**TRACE ELEMENT ANALYSIS IN PRESOLAR METEORITE GRAINS USING THE NANO-ESCA**

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Trace elements with very small concentrations have been detected in presolar meteorite dust via their local XPS spectra using the full-field imaging nano-ESCA instrument. Its key feature is high transmission up to 1.6 keV start energy (owing to correction of the analyser's spherical aberration [1]) allowing ultimate chemical sensitivity. Small area spectra as well as energy filtered images in the binding-energy range between 0 and 750 eV have been taken for a sample from the Murchison meteorite that consists essentially of submicron SiC grains. These incorporate a number of minor and trace elements as known from mass spectrometry [2]. Our energy resolution of 110 meV along with a spatial resolution in the 100 nm range allow a detailed analysis of 3d and 4d lines of various transition metals and several rare earth elements besides the Si and C lines of the main constituents. Significant core level shifts give access to the chemical state of the trace elements.

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[1] M. Escher et al., J. Phys.: Cond. Matt. 17 (2005) in print

[2] P. Hoppe and U. Ott, in AIP Conf. Proc. 402 (1997) 27

O/P.60**SELF-ABSORPTION EFFECT IN FLUORESCENCE DETECTION OF XANES AND EXAFS**

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The study of structural properties of thin films and coatings by X-ray Absorption Spectroscopy is of wide interest in material science. For that purpose, one of the more appropriated collecting methods is to detect the fluorescence yield. However, the linearity of the fluorescence detection in X-ray absorption spectroscopy depends on the experimental geometry and on the film thickness and it may allow an erroneous coordination number analysis.

ZrO₂ is an important material having a wide range of applications. ZrO₂ films are used to fabricate Thermal Barriers Coatings to insulate turbine gas components because of its low thermal conductivity coefficient. Its application may be so different that ZrO₂ coatings are used to prevent further corrosion of zirconium alloy in nuclear installations. In this work, we study the self-absorption effect in the L-edges of ZrO₂ films when detecting EXAFS and XANES and we propose a method to correct the obtained signal for moderate thick films. The correction will be applied to ZrO₂ thin films prepared by two different methods radio frequency magnetron sputtering and electron beam physical vapour deposited (EB-PVD) covering a large thickness range.

O/P.61**STATUS OF THE WERA SOFT X-RAY BEAMLINE AT ANKA**

Eric Pellegrin, Peter Nagel, Christian Pinta, Bernd Scheerer, and Stefan Schuppler

After its commissioning in early 2005, the WERA beamline at the 2.5 GeV ANKA synchrotron radiation facility within the Forschungszentrum Karlsruhe is to be used for classical as well as for advanced electron spectroscopy in the photon energy range between 80 and 1400 eV (with a later extension down to about 15 eV photon energy). The main objective of WERA is to provide the user with most of the presently available electron spectroscopy tools in situ in order to allow a complete characterization of the electronic as well as the magnetic structure of the sample under investigation. Thus, versatility (instead of specialization) is the primary goal for WERA.

The following experimental stations will be available (incl. an in situ sample transfer between the individual experimental setups) together with the corresponding sample preparation chambers: - photoemission electron microscopy - photoemission spectroscopy, resonant photoemission spectroscopy, near-edge X-ray absorption spectroscopy - soft X-ray magnetic circular dichroism (planned) - pulsed laser deposition of epitaxial thin film samples The technical specifications, the status and the future developments of the WERA soft X-ray beamline project will be presented.

Friday, June 3, 2005
Vendredi 3 juin 2005

Morning
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Session X : Engineering materials 1

Session chair : W. Ludwig

O-X.01 9:00 -Invited-

PHASE TRANSFORMATIONS IN STEEL STUDIED BY 3DXRD-MICROSCOPY

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A long-standing problem in the field of materials science is the understanding of the grain nucleation and growth kinetics during the formation of polycrystalline materials like metals and ceramics. The most important reason for this is given by the limitations of the conventional experimental techniques that cannot give local information about the grain boundary properties of the individual grains in the bulk of the material. Recently, the continuous development of the three-dimensional x-ray diffraction (3DXRD) microscope at the European Synchrotron Radiation Facility (ESRF) has created an opportunity to study the grain nucleation and growth rates, the shape, the crystallographic orientation, and the position of the individual grains in the bulk of the material. We used the 3DXRD-microscope to study the phase transformation of the high temperature austenite phase to the low temperature ferrite phase in medium carbon steel. This process takes place during cooling, and can be distinctly divided in the nucleation stage, in which very small clusters of atoms reorganise into nano-sized grains of the new crystalline structure, and the growth stage, in which these grains gradually grow. A comparison between the measurements and the classical nucleation and growth theories confirms the conventional nucleation and growth mechanisms and in addition shows new nucleation and growth mechanisms.

O-X.02 9:30

FOOTPRINTS OF DEFORMATION MECHANISM DURING IN-SITU X-RAY DIFFRACTION NANOCRYSTALLINE AND ULTRA-FINE GRAINED METALS

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It is generally accepted that plasticity in metals can not be carried anymore by a conventional dislocations mechanism when the grain size is reduced to the nanometer regime. Molecular dynamics simulations of nanocrystalline metals have demonstrated the ability of grain boundaries to emit and absorb dislocations in the absence of dislocation sources within the grains, in other words a dislocation mechanism that does not leave behind dislocation debris.

Driven by these results, a new type of in-situ experiment has been developed at the Swiss Light Source to follow the peak shift and peak broadening during tensile deformation. This is possible thanks to the development of the microstrip detector covering an angle of 60° with an angular resolution of 0.0037°. Tests were performed on electrodeposited nanocrystalline Ni with a mean grain size of 30nm and ultrafine grained HPT-Ni (synthesized by High Pressure Torsion) with a mean grain size of 300nm. Results for electrodeposited nanocrystalline Ni demonstrates a reversible peak broadening during plastic deformation validating the suggestions of molecular dynamics whereas the same type of experiment performed on HPT-Ni demonstrates the development of an additional permanent dislocation network in the ultra-fine grained structure. (Science, 304(2004) 273). These types of in-situ experiments also reveal other intriguing differences in the footprint left behind by the deformation mechanism when comparing electrodeposited Ni and HPT-Ni, such as strain accommodation in the elastic region and the softening observed after the maximum flow stress has been reached.

O-X.03 9:50

STUDY OF THE TEXTURAL EVOLUTION IN Ti-Rich NiTi USING SYNCHROTRON RADIATION

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Nickel-Titanium (NiTi) shape memory alloys (SMA) are successful actuating materials due to the combination of good functionality properties with high strength and good ductility. Shape Memory Effect is strongly influenced by the texture of the material leading to the anisotropic response of the recoverable strain. It is thus very important to study the effect of different thermomechanical treatments on the final texture, by evaluating its evolution during high temperature holding. The goal of the present work is to investigate the textural evolution in high-temperature phase (austenite - B2) of Ti-Rich NiTi SMA (Ni-51at% Ti). This alloy was processed with different thermomechanical treatments involving various heat treatments and cold rolling. The Quantitative Texture Analysis was performed at BM20 (ROBL-CRG) at the European Synchrotron Research Facility (ESRF) using in-situ high temperature X-Ray Diffraction technique with a wavelength set at 1.54 Å. In-situ high temperature texture determination has been carried out for the first time for Ni-Ti alloys, by annealing at different temperatures up to 800 °C under a vacuum better than 10⁻⁶ mbar. The discussion highlights the evolution of texture during annealing at high temperatures and the change in the microstrains/coherence domain size.

O-X.04 10:10

LATTICE DISTORTION IN g' PRECIPITATES OF SINGLE CRYSTAL SUPERALLOY SC16 UNDER CREEP-DEFORMATION

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Specimens of single crystal superalloy SC16 were creep deformed at 1273 K under tensile and compressive stress to strains of $\pm 0.5\%$ using a creep stress of ± 150 MPa. After creep deformation the lattice parameters of g' precipitates were measured in-situ between room temperature and 1173 K by means of high resolution X-ray diffraction at the 6-axis diffractometer at KMC-2 at BESSY. Measurements have been performed parallel and perpendicular to the stress axes. A tetragonal distortion of the ordered L12 crystal lattice was observed for both kinds of creep deformation. The distortion and its temperature dependence are ascribed to dislocations and/or to changes in chemical composition induced during creep-deformation.

10:30

BREAK

Session XI : Engineering materials 2

Session chair : **S. Offerman**

O-XI.01 11:00 -Invited-

MAPPING GRAINS AND THEIR DYNAMICS IN 3 DIMENSIONS.

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3D X-Ray Diffraction (3DXRD) microscopy is an emerging tool for fast and non-destructive characterization of the individual grains and sub-grains inside bulk materials [1]. The method is based on diffraction with hard x-rays ($E \geq 50$ keV), enabling 3D studies of millimetre - centimetre thick specimens. Ray tracing with several detectors is applied. The position, volume, orientation, elastic and plastic strain can be derived for hundreds of grains simultaneously. Furthermore for coarse-grained materials grain boundary maps can be generated. With the present set-up at the 3DXRD microscope at the European Synchrotron Radiation Facility, the spatial resolution is ~ 5 μ m, while diffracting units of size 100 nm can be observed. The methodology will be presented with focus on a set of novel reconstruction algorithms for generation of grain maps. These have enabled the acquisition of 3D movies of the growth of individual grains during annealing processes [2].

In this talk focus will be on fundamental issues in plastic deformation. Based on 3DXRD a set of unique tools for characterization of the dynamics of the individual grains and subgrains have been established. The rotation of 100 deeply embedded grains has been determined as function of tensile deformation in Al [3,4]. As a result "state-of-the-art" models for polycrystal plasticity were proven to be wrong, and a new slip-based model has been proposed [5]. First *in situ* results for the dynamics of the dislocation structures – made very recently at the Advanced Photon Source - will also be summarised.

As a complementary tool a method has been pursued for visualizing the 3D plastic strain field in metals [6]. Based on x-ray tomography with hard x-rays, the shifts in position of embedded marker particles is followed as function of external strain. As examples of work, compression studies of simple geometries will be presented.

[1]H.F. Poulsen. Three-Dimensional X-Ray Diffraction Microscopy. Springer Tracts in Modern Physics 205 (Springer, Berlin, 2004).

[2]S. Schmidt et al. Science **305**, 229-232 (2004).

[3]L. Margulies et al. Science **291**, 2392-2394 (2001).

[4]H.F. Poulsen et al. Acta Mater. (2003), **51**, 3821-3830.

[5]G. Winther, et al. Acta Mater. (2004) **52**, 2863-2872.

[6]S.F. Nielsen et al. Acta Mater. (2003) **51**, 2407-2415.

O-XI.02 11:30

X-RAY TOMOGRAPHY OF POWDER INJECTION MOLDED MICRO PARTS USING SYNCHROTRON RADIATION

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Powder injection molding is one of the most promising replication methods for the production of metal and ceramic micro parts. The material for injection molding a so-called feedstock consists of thermoplastic binder components and inorganic filler with approximately equal volume fractions. Injection molding of the feedstock leads to a green part that can be processed to a dense metal or ceramic micro part by debinding and sintering. During the injection molding process extremely high shear rates are applied. This promotes the separation of powder and binder leading to a particle density variation in the green part causing anisotropic shrinkage during post-processing. The knowledge of introducing density gradients and defects like pores would consequently allow the optimization of the feedstock, the molding parameters as well as the validation of a simulation tool which is currently under development. Conventional x-ray tomography with a spatial resolution of about five μ m does not allow the analysis of micro-parts with dimensions of several tenth of a millimeter. Additionally the effect of beam hardening prevents a quantitative interpretation of intensity variations. Both obstacles can be overcome by using synchrotron radiation. For measurement, bending bars consisting of dispersed silica particles in a polymeric matrix were used. The results concerning density variations and defect distribution as well as the experimental approach will be presented.

O-XI.03 11:50

IN SITU ANALYSIS OF CRACKS IN STRUCTURAL MATERIALS USING SYNCHROTRON X-RAY TOMOGRAPHY AND DIFFRACTION

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The structural integrity of many components and structures is dominated by cracks and hence the study of cracked bodies study is of major economical and social importance. Despite nearly 30 years of study, there is still no detailed consensus regarding either the fundamental parameters that drive cracks or the precise mechanisms of their growth in most materials. Thus. Virtually all crack life prediction models currently in engineering use are largely phenomenological rather than physically based. Historically, a major hindrance to our understanding of crack initiation and propagation has been the inability to measure either the crack tip stresses or the crack morphology deep within materials. The development of very high resolution strain and tomography mapping on Third Generation Synchrotron sources such as the ESRF has opened up the possibility of developing complimentary techniques to monitor the entire plastic/process zone growth mechanisms and the accompanying crack tip field and crack wake field around growing cracks. If realised, such techniques would produce unique information that would be invaluable both in validating present Finite Element simulations of fatigue crack growth and in developing the future high accuracy simulations necessary for the development of physically realistic fatigue life prediction models. Recent work at the ESRF developing technology enabling the imaging of cracks and crack strains are presented together with preliminary results obtained from an aerospace aluminium alloy.

O-XI.04 12:10

NANOMETRIC INTERGRANULAR LIQUID LAYER PENETRATION IN THE Al/Ga SYSTEM: AN IN SITU X-RAY IMAGING INVESTIGATION

E. Pereiro-López, ESRF, B.P. 220, 38043 Grenoble, France, W. Ludwig, GMPPM, INSA de Lyon, 69621 Villeurbanne Cedex, France, P. Cloetens, ESRF, BP 220, 38043 Grenoble, France and D. Bellet, GPM2, ENSPG, INPG, BP 46, 38402 Saint Martin d'Hères Cedex, France

The penetration of liquid Ga along the grain boundaries of Al bicrystals [1, 2] is analysed by synchrotron X-ray projection microscopy. Using Kirkpatrick-Baez focussing optics, a secondary X-ray source is produced of about 100(100 nm² spot size and typical divergences of a few milli-radians [3]. By positioning the sample downstream this source the spatial resolution detector limitation is overcome and the ultimate resolution is rather given by the secondary source size. The present investigation deals with one of the very first applications of such a microscope, currently under commissioning at the ID19 beamline of the European Synchrotron Radiation Facility (ESRF, Grenoble, France).

In situ observations of the Ga penetration process reveal linear propagation of the penetration front accompanied by a continuous thickening of the Ga intergranular wetting layer. By combining sub-micron spatial resolution and in situ imaging capabilities it has been possible to characterize simultaneously the presence of nanometric penetration layers and, for the first time, associated continuous relative movement of the grains of final amplitude in agreement with the value of the saturated Ga layer thickness. The measured deformation of the bicrystal is compared to the predictions of elastic-plastic crack propagation under mode I loading conditions.

[1] E. Pereiro-López, W. Ludwig, D. Bellet, *Acta Materialia* 52, no.2, 321 (2004).

[2] W. Ludwig, E. Pereiro-López, D. Bellet, *Acta Materialia* 53, no.1, 151 (2005).

[3] O. Hignette, P. Cloetens, W. Ludwig, G. Rostaing, *J. Physique IV* 104, 231 (2003).

12:30

LUNCH

Session XII : Complex and biomaterials
Session chair : H. Amenitsch

O-XII.01 14:00

DEPTH-RESOLVED STRAIN MEASUREMENTS IN POLYCRYSTALLINE THIN FILMS BY ENERGY-VARIABLE X-RAY DIFFRACTION

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An energy-variable synchrotron diffraction technique is established as a novel method for depth-resolved measurements of lattice spacings and residual strains in polycrystalline films. The depth sensitivity is achieved by the controlled changing the x-ray energy and, hence, the x-ray penetration into the sample. In this paper, the energy-dependent analytic expression for the shape of diffraction peak, taken from a thin film by using quasi-parallel synchrotron x-rays, is given. We show that the maximum diffraction intensity recorded in the detector is coming from a certain depth, which depends on energy and is composed of the film thickness and x-ray penetration length. This finding opens a way for strain measurements with high depth resolution by changing the x-ray energy in small enough steps. The developed technique is applied to characterize thin film metal-metal multilayers produced by electrodeposition. Measurement of lattice spacings across the film thickness allowed us to detect periodic strain variations on a 100 nm scale due to forces acting at interfaces between individual sub-layers. An ability to collect experimental data in steps of 10 nm, when working not far away from absorption edges, is demonstrated.

O-XII.02 14:20

X-RAY DIFFRACTION CONTRAST TOMOGRAPHY: A NEW TOOL FOR 3D CHARACTERIZATION OF UNDEFORMED POLYCRYSTALS

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We present here two possible extensions of X-ray microtomography, capable to characterize the shape and the orientation of individual grains in the bulk of *undeformed* polycrystalline materials: (i) diffraction contrast tomography in "topo-tomography" alignment [1] and (ii) diffraction contrast tomography in conventional alignment. In both cases, Bragg diffraction (transmission case) gives rise to an additional contribution to the local attenuation coefficient, which in turn can be exploited by means of analytic or algebraic tomographic reconstruction techniques in order to reconstruct the three dimensional grain outlines. The second approach offers in addition the possibility to determine grain orientations.

The imaging principle and related data analysis strategies will be illustrated for both methods with the help of first experimental data, obtained from a coarse grained Al (1050) multicrystal. A comparison of diffraction contrast tomography with respect to the more widely applicable 3DXRD approach [2] will be given. Based on this, we will discuss possible future developments and applications for combined tomographic imaging and diffraction experiments.

[1] W. Ludwig et al, J. Appl. Cryst. 34, (2001), p. 602

[2] H.F. Poulsen, Three-dimensional X-ray diffraction microscopy, Springer (2004)

O-XII.03 14:40

SAXS OF SELF-ASSEMBLED NANOCOMPOSITE FILMS WITH ORIENTED 2D CYLINDER ARRAYS: AN ADVANCED METHOD OF EVALUATION

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The present work shows that the SAXS of oriented 2D hexagonal silica mesostructures of cylinders, measured in symmetrical reflection, can be effectively evaluated using well chosen scattering functions. The method requires a high accuracy of intensity measurements in reflection over several decades. Up to 8 independent structural parameters can be extracted from the SAXS curves by the fitting with suitable scattering functions of arrays of cylinders of infinite length. Highly oriented nanocomposite silica films were prepared by Evaporation-Induced Self-Assembly, using non-ionic Brij surfactants as structure-directing agents, leading to films with the cylinders being oriented parallel to the substrate. The SAXS data showed pronounced interference maxima ("Bragg interferences") and minima, the latter attributable to the form factor of cylinders. Together with SAXS in grazing incidence geometry, the method results in a comprehensive characterization of the 2D hexagonal mesostructure by providing accurate values for the cylinder radius, the lattice parameter, their variances and the average stack height. Also, suitable approaches are introduced to quantitatively take into account the effects of absorption and preferred orientation on the SAXS of oriented cylinders. As an important result, it is seen that with increasing preferred orientation the lattice reflections are enhanced much stronger than the Laue scattering. The results of this study show that fitting procedures with chosen scattering functions are preferable to the use of Fourier transform methods. The method was applied to mesoporous films with cylindrical mesopores, and the pore sizes were in good agreement with independent physisorption measurements.

O-XII.04 15:00

VISUALIZATION OF FATIGUE CRACKS IN DENTAL MATERIALS USING 3D
DATA OF SYNCHROTRON RADIATION BASED MICRO COMPUTED
TOMOGRAPHY

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Fatigue cracks are usually characterized by surface sensitive techniques after decomposition. High-resolution tomography using synchrotron radiation (SR μ CT) allows the non-destructive evaluation of fatigue crack formation. The problem, however, is the visualization of the crack with its characteristic features out of the set of slices. Inhomogeneities of the ceramics and noise in the tomogram further complicate the segmentation of the crack. We have applied different approaches to make visible a crack in a homogeneous poly(methylmethacrylate) and an inhomogeneous opaque dental ceramic. For the application of the elastically deformable contour model (Snakes) a reasonable estimate of the plane has to be extracted, which is performed by the determination of mass center points in the hollow space and the minimum intensity search in parallel projections. To segment the hollow space, for the region of interest a threshold was extracted from the histogram. The search for the appropriate parameters for the Snakes algorithm works much better for the homogeneous material than for the inhomogeneous one. For the dental ceramic, the areas, where the Snakes approach provides reasonable results, are restricted. Nevertheless, SR μ CT combined with sophisticated visualization tools assist to characterize the crack formation in a unique manner.

15:20

THE END