depth profile the surface disorder and possible pressure induced phase transitions.

The results indicate that all of the studied compounds were changed due to the compression. The GID analysis shows that the surface regions of the compacted tolbutamide, carbamazepine and chlorpropamide tablets were disordered. The manifestations of the disordering in the diffractographs are the increased peak intensity and height and the decreased peak width. Moreover, a polymorphic phase transition was observed in chlorpropamide tablets. The biggest changes took place at the very surface of the tablets. The transitions were also dependent on the used compaction pressure.

Keywords: pharmaceuticals, grazing incidence diffraction, pressure-induced disordering

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Structural and Compositional Investigation of Semiconductor Quantum Materials

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The size, shape, strain distribution, compositional profile and spatial distribution are the critical factors determining the electronic level and thus the physical properties of semiconductor nano-structures. For those MBE-grown mesoscopic objects, lattice mismatch, surface segregation, interface diffusion and various kinetic effects make their formation mechanism very complicated. In fact, the structure and the formation mechanism of these self-assembled nano-structures are still not well understood. In this work, we applied grazing incidence X-ray scattering methods including reciprocal space map and small angle X-ray scattering to study the strain field, shape and spatial distribution of III-V semiconductor nano-structures. In particular, we will focus on the application of resonant X-ray scattering technique to accurately determine the compositional distribution within the nano-structures with high resolution.

Keywords: surface X-ray scattering, semiconductor epitaxy, nanostructures

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Structure Analysis of Crystal Grain Nearby Surface using X-ray Scattering at Small Glancing Angle of Incidence

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When X-rays are applied to the material surface at a grazing angle of incidence, the intensity of X-rays scattered on the surface is the sum of the X-rays that scattered by the atoms only on the surface, ca. several ten atomic layers deep, and the contribution of the atoms of each depth to the X-rays intensity varies on the incidence angles.

Since the penetration depth of X-rays changes by changing an incidence angle, a structural change of the depth direction of a material surface layer can be known in analyzing incidence angle dependence of the information that the scattered X-rays have.

We derived the x-ray intensity propagating during the surface layer materials that are characterized with complex refractive index, which changes continuously in depth, and studied analyzing method for evaluating the distribution of grain size of the crystal in the surface layer of material by using x-ray diffraction at small glancing angle incidence.

Intensities of the diffracted x-rays on polycrystalline iron surface were measured continuously at the various incidence angles, and the dependency of the incidence angles was investigated.

Keywords: surface X-ray scattering, polycrystalline X-ray diffraction, grazing incidence diffraction

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Probing Interface Strain With X-ray Bragg-Surface Diffraction

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Epitaxially grown Au films on semiconductor substrates, especially on GaAs single-crystals, have a wide variety of applications in the semiconductor industry. It has been yet very difficult to apply modern electron microscopy such as scanning tunneling microcopy and transmission electron microcopy in studying the interface structure since the interface is buried under an over-layer film. Moreover, the grazing incidence X-ray diffraction frequently used for characterization of surfaces/interfaces may encounter difficulties when the incident X-rays propagate from a lower refractive index medium into a higher one.

To overcome this difficulty, we adopt the three-wave Bragg-surface diffraction technique to investigate the effects of interface on the formation of diffraction images. From the angular positions of the diffracted images the variation of lattice constants parallel and normal to the interface can be determined. The experiment is carried out at NSRRC. The Bragg- surface diffraction used is the GaAs(006)/(1-13), where (006) is a symmetric Bragg reflection and (1-13) is a surface diffraction. The photon energy employed is 11.07 keV. Details about the analysis of strain will be reported.

Keywords: X-ray multiple diffraction, interface, strain

P.12.02.1

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Chemical Preparation of GaAs (100), (110), (111) and (112) Substrates with HF:H_2O_2: Citric Acid:H_2O

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Chemical preparation of GaAs (100), (110), (111) and (112) substrates was performed by HF:H_2O_2: Citric acid: H_2O solution. The removed layer thickness was evaluated as a function of the constituent concentrations, temperature and the etching time. HF concentration was varied from 0.065 to 5.2 mol, H_2O_2 was varied from 1.28 to 3.23 mol and citric acid ($C_6H_8O_7.H_2O$) concentration was maintained constant (1.3 mol) to obtain the etching rate. The temperature of etching was varied of room temperature to 75 °C for the same constituent concentration. The rate of etching and the surface quality were controlled by high resolution optical microscope.

Keywords: surface quality, chemical preparation, rate of etching

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The X-ray Reflectometry and the Phase Contrast Methods for Crystal Analysis

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The X-ray phase contrast method [1] provides high resolution visualization of the internal structure of low absorbing substances with flat density gradient. This method can also be used for the study of refraction index changing processes, e.g. crystal growth. NaCl solution, where the same crystals are grown, has been used as an investigated object. The results of experiments were the density gradient of the near-surface region around the growing crystals and the width of the intermediate layer.

The X-ray reflectometry methods provide estimating the physical and geometrical properties of the near-surface region of the crystals with a high accuracy. These methods are based on the measurement of

the reflectance within the small angles of the incidence area (in and near the complete external reflection area). NaCl monocrystals have been used as investigated objects. Crystal faces quality after splitting, short and long time processes of solution and growth have been estimated.

[1] Bushuev V.A., Petrakov A.P., *Crystallography*, 2001, **46**, N 2, 209-214.
Keywords: X-ray crystal analysis methods, X-ray reflectometry, X-ray crystallography

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Hydrogen-Bonded Structure of Alcohols Adsorbed on Silica Surface in Cyclohexane
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Liquid molecules at the solid-liquid interface often exhibit quite different properties from those in the bulk, which is attributed to the surface-induced structuring of liquids. We recently found a hydrogen-bonded ordered structure, which we call a "molecular macrocluster", of alcohols when we investigated the adsorption of them on silica (glass and oxidized silicon) surfaces from cyclohexane.

Alcohols studied were monohydric alcohol such as methanol and ethanol [1], and dihydric alcohol (ethylene glycol)[2]. A combination of colloidal probe atomic force microscopy, FTIR-ATR spectroscopy, and adsorption excess isotherm measurement was employed. The force measurement revealed the long ranged attraction (e.g. ca. 35 nm for ethanol) between silica (glass) surfaces, which was ascribed to the attraction due to the contact of the opposed adsorption layers bearing the high interfacial energy. FTIR-ATR spectroscopy demonstrated that alcohol molecules adsorbed on the silica (silicon oxide) surfaces formed hydrogen-bonded clusters (polymers), which extended 15~20 nm (for monohydric alcohol) from the surface silanol groups. Practically no cluster was formed on the hydrogen-terminated silicon surfaces. Interesting differences were observed in the mode of adsorption depending on the chemical structures. Dynamic properties of adsorbed alcohols were studied by NMR spectroscopy.

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Keywords: solid-liquid interaction, interfacial liquid structure, hydrogen bonding

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ATSAS 2.1 - A Program Suite for Small-angle Scattering Data Analysis
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A new release 2.1 of the program package ATSAS for small-angle scattering data analysis is presented. The package allows one to perform a complete analysis of the scattering data covering major steps from data reduction to automated 3D modelling. ATSAS is primarily oriented towards macromolecular solutions but can also be used for other types of systems. Its main components are:

1) Primary data processing and reduction package PRIMUS [1], which also computes overall structural parameters and characteristic functions and permits to invoke major data analysis programs from a single graphical user interface.

2) An *ab initio* three-dimensional modelling suite including e.g. programs DAMMIN and GASBOR bead and dummy residues modelling [2,3].

3) A rigid body modelling suite (programs MASSHA [4], GLOBSYMM, SASREF etc) to characterize macromolecular complexes in terms of the structure of sugbunits.

4) A suite for quantitative analysis of interacting systems and mixtures (programs PEAK, SVDPLOT, MIXTURE etc [1]).

[1] Konarev P.V., et.al., *J. Appl. Cryst*., 2003, **36**, 1277. [2] Svergun D.I., *Biophys. J*., 1999, **76**, 2879. [3] Svergun D.I., et.al., *Biophys. J.*, 2001, **80**, 2946. [4] Konarev P.V., et.al., *J.Appl. Cryst.*, 2001, **34**, 527.
Keywords: small-angle scattering, data processing software, macromolecules

P.12.07.2
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Probing Intermediate Filament Structure and Assembly with Small-angle X-ray Scattering
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Intermediate filaments (IFs) together with microtubules and actin filaments, form the cytoskeleton of most living cells. IFs are long macromolecular aggregates, with about 10 nm cross-section. While crystallographic data on the dimer representing the elementary IF 'building block' have recently become available, little structural detail is known on both the mature IF architecture and their assembly pathway.

We have applied small angle X-ray scattering (SAXS) to investigate the *in vitro* assembly of human IF protein vimentin in varying pH and ionic strength conditions. SAXS is a method allowing one to analyze protein structure in solutions at different external conditions and also to quantitatively characterize of mixtures of different oligomeric states. We demonstrate that formation of tetramers, octamers and IFs represent the principal steps along the vimentin assembly pathway. By combining the SAXS data with the atomic structures and additional structural restraints, three-dimensional models of these assembly intermediates are constructed and refined. These results are further confirmed by electron microscopy observations.
Keywords: intermediate filaments, filament assembly, small angle X-ray scattering

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Grazing Incidence Small Angle X-ray Scattering from Nanoparticles : beyond Classical Analysis Approximations
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GISAXS (Grazing incidence small angle X-ray scattering) has emerged in the past years as a powerful technique for probing the morphology of nanostructures elaborated on surfaces by recording the diffuse scattering around the specular reflected beam. A recent experimental breakthrough [1] allowed using this synchrotron technique to record background free scattering patterns during the undisturbed growth of islands in ultra-high vacuum environment.

Contrary to real space techniques, extracting morphological parameters as the shape, sizes and size distributions for a classical Volmer-Weber growth implies a complete data analysis that is hampered by the multiple reflection effects induced by the grazing geometry and by the correlations between the size of the scatterers and their separation. It will be shown that suitable models can improve the analysis by (i) including the gradient of index of refraction seen by the incoming and scattered beams contrary to classical Distorded Wave Born Approximation for substrate only [2] and (ii) by calculating the parallel diffuse scattering within the framework of the paracrystal model, thus going beyond the classical Local Monodsiperse Approximation [2,3].

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Keywords: grazing incidence, SAXS, nanoparticles