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Phase and Stress Analysis of Porous Titania Layer with Two-dimensional XRD

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The surface modification of titanium by micro-arc oxidation under various voltages was performed to form a porous titania layer which may improve the biocompatibility of titanium implants. The phases and residual stresses of the porous layers were measured with two-dimensional X-ray diffraction. The results show the porous layers contain anatase (TiO_2) and rutile (TiO_2). The content of rutile (TiO_2) increases with increasing voltage. At 450V, anatase peaks almost disappear and many new peaks appear in the profile, some of them are identified as polymorphous $CaTiO_3$. The residual stresses in the porous layers are compressive and increase with increasing voltage.

This presentation also introduces the recent progress in two-dimensional X-ray diffraction using the above application as an example for microstructure and residual stress analysis. The two-dimensional X-ray diffraction provides far more information than the conventional X-ray diffraction. Phase identification can be done by integration over a selected range of diffraction rings. The integrated data gives better intensity and statistics, especially for those samples with texture, large grain size, thin film or small quantity. Stress measurement using two-dimensional detector is based on a direct relationship between the stress tensor and the diffraction cone distortion.

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Keywords: porous materials, stress, two-dimensional XRD

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Three-Beam Diffraction Anomalous Fine Structure of Thin Films

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Different from the usual two-beam DAFS (diffraction anomalous fine structure), we have recently developed the multi-beam DAFS (MDAFS) for observing the local structural environment of resonant atoms. With three-beam diffraction data for different photon energies, the visibility *Rv* of the intensity asymmetry related to the phases of structure-factor triplets involved in the three-beam diffraction can be determined. Analysis based on the dynamical diffraction theory and XAFS gives fine structures of DAFS spectra. In this paper, the three-wave diffractions of (100) *CdTe* thin films epitaxially grown on the (100) *InSb* substrates are measured for different photon energies covering all the *L* edges of the constituent atoms. The crystallographic phase of structure-factor triplets and the resonance phase shifts influenced by the substrate could be analyzed to give the interface structures in relation to the *CdTe* and *InSb*. Using this MDAFS technique, we have also extracted the information about the fine structures of *Cd* and *Te* around the interface.

Keywords: structure, thin film, DAFS

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X-ray Study of Titanium Coatings Made in Shaped Charge Jet Condition

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To improve hardness and corrosion resistance of a titanium surface, one technique of its treatment is the introduction of C, N and B into the metal crystal lattice. A technique for the application of coatings to titanium surfaces by a cumulative jet is suggested [1].

Some coatings were studied on a DRON-RM4 diffractometer. X-ray phase analysis was performed using the POWDER CELL 2.4 software [2]. The atomic coordinates and thermal parameters were assigned according to the literature data. The characteristics, such as quantitative phase relations, unit cell parameters, profile parameters (u, v, w), etc. were refined. The cubic and hexagonal modifications TiX, were X= C, N, B, were revealed in the samples investigated. The unit cell parameters a=4.23 – 4.31 Å, a=2.97 – 2.98 and c=4.75 – 4.80 Å were obtained for the cubic and hexagonal phases, respectively. The coating phase compositions appeared to be dependent on geometric characteristics of the cumulative device. The mean microhardness of the coating is 18 GPa, while the maximum value 39.5 GPa.

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Keywords: X-ray powder analysis, coatings, titanium compounds

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X-ray Investigation of Hydrogen Implanted GaAs

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Smart-cut is a layer transfer technique, which offers a route to the monolithic integration of dissimilar materials. The technique exploits hydrogen implantation-induced exfoliation and wafer bonding to transfer thin layers of a semiconductor onto another material, which may have a different lattice constant. However, it is still unclear exactly how smart-cut is affected by different ion implantation parameters, especially for III-V materials such GaAs. This work aims to investigate the role of the implant temperature and the dose rate on blistering in GaAs, in order to reconcile these findings, and to further the understanding of the smart-cut process.

Semi-insulating GaAs wafers were implanted with 190 keV H_2^+ ions, to a fluence of $5x10^{16}$ H/cm^2, at sample temperatures of 180K, 300K, 470K and 570K. The distribution of hydrogen and the implantation damage in the samples were studied by ion beam analysis and X-ray high-resolution diffraction. Information concerning the ion implantation damage and the strain distribution in the film was obtained by simulating the X-ray diffraction pattern. It was found that at higher temperature, hydrogen is mobile in the lattice and can rearrange into the plates, microcracks and bubbles, which are presented in blisted material, thus relieving the strain in the lattice. The dose rate was also found to be significant for the smart-cut process, as blistering and exfoliation are inhibited at low dose rates.

Keywords: smart-cut, X-ray diffraction, ion implantation

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Grazing Incidence X-ray Diffraction Studies of Pharmaceutical Tablets

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Grazing incidence diffraction (GID) is a technique not yet being used in the field of pharmaceutical physics widely. However, GID is a very potential alternative to the other surface sensitive techniques, e.g. various spectroscopic methods, used in the pharmaceutical materials analysis. With GID it is possible to monitor phase transitions on the surface of tablet as a function of time and depth, for example.

In the present study GID has been utilized to study the disorder of the tablet surface after the compaction. Three active pharmaceutical ingredients, namely tolbutamide, carbamazepine and chlorpropamide, were chosen to act as model tablet compounds. Several tablets were compacted using different compaction pressures. The prepared tablets were then analysed with GID with various incident angles in order to

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

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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