EUROPEAN
MATERIALS RESEARCH SOCIETY

Strasbourg (France)

E-MRS Spring Meeting 2003
June 10 - 13, 2003

SYMPOSIUM M

Optical and X-ray metrology for advanced device materials characterization

Symposium Organizers:

Jürgen Schreiber, Martin-Lüther University Halle-Wittenberg, Germany,

Mircea Modreanu, NMRC, Cork, Ireland

Martin Murtagh, Optical Metrology Innovations Ltd., Cork, Ireland

Daniel Chateigner, CRISMAT-ISMRA, Caen, France

Jesús Ricote, ICMM-CSIC, Madrid, Spain

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E-MRS 2003 SPRING MEETING

SYMPOSIUM M

Tuesday, June 10, 2003 — Morning
Mardi 10 juin 2003 — Matin

Session I: Optical characterisation: modulation spectroscopy
Session chair: Jürgen Schreiber and Mircea Modreanu

M-I.1 9:00 -Invited-

ADVANCES IN MODULATION OPTICAL SPECTROSCOPY

T.J.C. Hosea, Advanced Technology Institute, University of Surrey, Guildford, Surrey GU2 7XH, U.K.

Modulated reflectance spectroscopy is a simple, straightforward technique in which the reflectivity of a semiconductor is perturbed by an external periodic influence - most usefully nowadays a chopped laser beam, i.e. photo-modulated reflectance (PR). Since samples need no special mounting, can be studied in air at room-temperature, and can be full-sized pre-fabrication wafers, PR is truly non-destructive. In contrast to conventional techniques such as low-temperature photo-luminescence, PR spectra are often replete with sharp, detailed derivative-like features arising from ground-state, and other possible higher-energy optical transitions within a sample, from which can be extracted many material parameters crucial to successful and efficient device operation, such as compositions, layer thicknesses, built-in electric fields and band line-ups. Modulation spectroscopy has now proven to be an extremely useful and unique tool for characterising optoelectronic structures and materials. Despite this, main-stream science still seems to regard the technique as rather obscure, sometimes even with suspicion. The talk will discuss the advances in the application and interpretation of modulation spectroscopy, as pioneered at Surrey, and which have generated interest in the growth industry. The focus will be on the assessment of vertical- cavity surface-emitting lasers and resonant-cavity LEDs, which so far have defied analysis by the more conventional non-destructive techniques.

M-I.2 9:40 -Invited-

PHOTOMODULATED REFLECTANCE AND TRANSMITANCE: OPTICAL CHARACTERISATION OF NOVEL SEMICONDUCTOR MATERIALS AND DEVICE STRUCTURES

J. Misiewicz, G. Sek, R. Kudrawiec, P. Sitarek, Institute of Physics, Wroclaw University of Technology, Wybrzeze Wyspianskiego 27, 50-370 Wroclaw, Poland

Photomodulation spectroscopy, in reflection and transmission modes, is presented as a powerful nondestructive optical technique for the investigation of fundamental physical properties of new semiconductor materials, including multinary compounds, and complex micro- and nanostructures. We will focus on several representative examples:

- optical transitions, hence band structure and band gap temperature parameters of materials as: GaN, GaAsN, GaInAsN, GaAsSbN etc.
- electric fields in different parts of multilayer heterostructures by pump beam wavelength selection
- electric field in d-doped field effect structures
- optical transitions in quantum confinement systems (like quantum wells) including parity forbidden ones and above barrier resonant state transitions
- selection rules investigation by three beam photoreflectance method - nanostructures: quantum dot lasers and coupled dots The abilities of photoreflectance and phototransmitance in application to practically every kind of semiconductor system will be discussed.

M-I.3 10:20

APPLICATION OF PHOTOREFLECTANCE SPECTROSCOPY TO OPTOELECTRONIC MATERIALS AND DEVICES

L. Long, J. Schreiber, Fachbereich Physik, Martin-Luther-Universität Halle-Wittenberg, Friedemann-Bach-Platz 6, 06108 Halle, Germany

Typical photoreflectance lineshapes related with simple and complex wafer structures are reviewed, and transient behavior of photoreflectance spectrum components is analyzed in detail in respect of amplitude and phase delay of the different PR spectrum features as function of modulation frequency. A fitting procedure for photoreflectance spectrum with special consideration for Seraphin coefficients is developed to extract the physical parameters for materials and device characterization. It applies very well to photoreflectance experiment carried out on GaAs wafer materials, QWIP, HBT and VCSEL devices.

10:40 **BREAK**

Session II: Methodological advances in X-ray
Session chair: Davor Balzar and Olivier Durand

M-II.1 11:00 -Invited- GRAZING INCIDENCE X-RAY DIFFRACTION FROM SELF-ORGANIZED EPITAXIAL STRUCTURES

V. Holy, Institute of Condensed Matter Physics, Masaryk University, Brno, Czech Republic, J. Stangl and G. Bauer, Institute of Semiconductor Physics, Kepler University, Linz, Austria

X-ray scattering of self-organized semiconductor nanostructures (quantum wires and dots) yield information on their shapes and chemical composition, however the experiments are complicated by a very small useful signal, since the objects under investigation are extremely small. Therefore it is necessary to suppress the substrate signal and enhance the intensity stemming from the wires or dots. This can be achieved by a surface-sensitive x-ray scattering geometry, where the angle of incidence and/or the angle of exit are comparable with the critical angle of total external reflection. In this grazing-incidence arrangement, the information depth, where the scattered wave is collected from, can be reduced down to few nm. The grazing incidence method can be used in a small-angle scattering arrangement (grazing-incidence small-angle x-ray scattering - GISAXS) or in a diffraction arrangement (grazing-incidence diffraction - GID). The former method is a variant of the well-known conventional small-angle scattering method (SAXS) and it is sensitive to local variations of electron density. Therefore GISAXS investigates the shapes and the positions of the nano-objects below the sample surface. The GID is mainly sensitive to strains in and around the objects, so that it allows us to estimate non-destructively the chemical composition of the objects. In the talk, several experimental examples demonstrating the application of the grazing-incidence methods will be presented.

M-II.2 11:40 -Invited- RECENT ADVANCES IN CHARACTERIZATION OF ULTRA THIN FILMS USING SPECULAR X-RAY REFLECTIVITY TECHNIQUE

S. Banerjee(a), S. Ferrari(b),D. Chateigner(c) and A.Gibaud(d), (a)Saha Institute of Nuclear Physics, 1/AF Bidhan Nagar, Calcutta 700 064, India, (b)Lab. MDM - INFM via Olivetti 2, 20041 Agrate Brianza, Milano Italy, (c)Laboratoire CRISMAT-ISMRA, UMR 6508 CNRS, bd. M. Juin, 14050 Caen, France, (d)Laboratoire de Physique de l'Etat Condens, UPRES A 6087 CNRS, Faculté des Sciences, Université du Maine,Le Mans, France

We present different approaches to analyze the grazing incidence x-ray reflectivity (GIXR) data to characterize ultra thin films. The analysis of the GIXR data yields structural parameters such as surface and interfacial roughness, density profiles and thickness of the film and its individual layers if the film consists of many layers. We shall describe three schemes (1) a model dependent method based on dynamical scattering, which is generally known as recursive formalism, (2) a model independent method based on distorted wave Born approximation and (3) an inversion technique based on Born approximation. We will point out the problem of the non-uniqueness of the solutions, which are generally encountered in analyzing the x-ray reflectivity data based on the recursive formalism when the experimental data is fitted using non-linear least square fitting technique to obtain the fit parameters. We will demonstrate the above formalism on few systems as case studies. We will also show how to converge to a realistic solution using the above mentioned formalisms.

M-II.3 12:00 STRAIN PROFILES IN EPITAXIAL THIN FILMS DETERMINED BY HIGH-RESOLUTION X-RAY DIFFRACTION

A. Boulle, O. Masson, R. Guinebretière, A. Dauger Science des Procédés Céramiques et de Traitements de Surface UMR 6638, ENSCI, 47-73 avenue Albert Thomas 87065 Limoges Cedex, France

Strain plays a crucial role in the growth of thin films and multilayers as it can affect the morphology as well as the electronic properties of the films. The knowledge of the strain distribution across the film thickness is therefore of primary importance.

In this work, we present a new approach for the determination of the strain profiles using high-resolution X-ray diffraction. The method is based on the kinematical scattering theory and takes into account the effect of film thickness and thickness fluctuation. The shape of the strain profile is expressed in terms of uniform B-splines basis functions. Modelling of the diffraction profiles directly yields the shape of the atomic displacement field across the film thickness. The X-ray diffraction experiments have been carried out both at the SPCTS laboratory (Limoges, France) and at the BM2 beamline at the ESRF (Grenoble, France). This model is very versatile and can be applied to a wide range of epitaxial systems among which oxide epitaxial thin films. Its potential will be demonstrated by applying this approach to the case of sol-gel derived zirconium oxide layers epitaxially grown onto (11-20)-cut sapphire substrates.

M-II.4 12:20 IMAGING THE SENSITIVITY INHOMOGENEITIES OF DIAMOND DETECTORS USING FOCUSED X-RAY BEAMS AT ESRF

P. Bergonzo, D. Tromson, C. Mer, E. Snidero, DIMRI/ SIAR, CEA/Saclay, 91191 Saclay, France, R. Barrett, ESRF, 38043 Grenoble, France

Diamond is a semiconducting material that can withstand high temperatures, a wide range of corrosive environments and exhibits high radiation hardness. This combination of properties makes it extremely attractive for use as particle and photon detectors. Such detectors can be fabricated using either certain type IIa-type natural gems or by using CVD diamond films. The rarity of suitable natural diamonds means that the CVD diamond is particularly attractive for industrial use with the added benefits of a reproducible growth process, low cost and size.

The ESRF mono-chromatised and focused X-ray source (ID21) has been used to image the spatial variations of detector sensitivity over a range of both natural and CVD diamond devices. The measurements are performed by mapping the photon-induced current flow of the biased detectors. Evaluation of the sensitivity inhomogeneities are of dual interest: for detection applications involving relatively small beams it is important that the device response be as uniform as possible; furthermore, from a fundamental aspect, the understanding of the origin of the sensitivity variations can be a route, for example in CVD materials, to modifying the growth and processing parameters in order to produce improved devices. This paper demonstrates an application of alternative detection modes in X-ray microscopy for a problem of technological interest. Moreover, it shows how the flexible control of the primary X-ray penetration depth by use of probe energies ranging from 3 to 7keV permits an evaluation of the relative influence of the surface and bulk materials.

12:40 **LUNCH**

Session III: Applications: optical metrology
Session chair: Martin Murtagh and Jan Misiewics

M-III.1 14:00 -Invited-

NEXT GENERATION OPTICAL SPECTROSCOPY TECHNOLOGIES FOR ADVANCED DEVICE MATERIALS CHARACTERISATION

P.V. Kelly and M.E. Murtagh, Optical Metrology Innovations Ltd., 2200 Cork Airport Business Park, Cork Airport, Co. Cork, Ireland

The field of optical spectroscopy applied to the characterisation of crystalline thin-film and their surfaces and interfaces, sometimes referred to as epioptics, is maturing towards adoption in the advanced device materials industry. Advanced device material challenges motivate and commercially justify the adoption of new metrology. The next generation of optical spectroscopy characterisation tools is surveyed, with emphasis on modulation spectroscopies for the compound semiconductor industry, and on potential developments for the nanoelectronic materials sector. Developments in photoreflectance spectroscopy, as well as reflectance anisotropy spectroscopy and optical second harmonic generation, and their current and potential future applications are surveyed. Among the material applications of these techniques surveyed are the qualification of InGaP/GaAs heterojunction bipolar transistor (HBT) wafers, and of vertical cavity surface emitting laser (VCSEL) wafers by photoreflectance spectroscopy. Applications of photoreflectance to strain measurement and plasma etch damage monitoring in compound semiconductor and silicon epiwafers and substrates are discussed. Metrology tool concepts embodying the scientific progress in optical spectroscopy are presented which bridge the technology from laboratory to industrial production line.

M-III.2 14:40

THREE BEAM PHOTOREFLECTANCE AS A POWERFUL METHOD TO INVESTIGATE SEMICONDUCTOR HETEROSTRUCTURES

R. Kudrawiec, G. Sek, J. Misiewicz, Institute of Physics Wroclaw University of Technology, Wybrzeze Wyspianskiego 27, 50-370 Wroclaw, Poland, A. Forchel, Institute of Physics, Wuerzburg University, Am Hubland, 97074 Wuerzburg, Germany

Three beam photoreflectance (TBPR) has been proposed to investigate the influence of built-in electric field changes on the optical properties of low-dimensional semiconductor structures. To change the internal electric field, we have used third beam continuously illuminating the sample and causing changes of the built-in electric field due to the photovoltage effect. It works as a contactless forward bias and allows for a change of the field, even down to the flat band conditions. We have applied TBPR to study heterostructures for which the band-to-band transitions with Franz-Keldysh oscillations (FKO's) are observed. In this case, PR spectrum is very often a superposition of signals from different regions (depths) of the sample (from surface and interfaces) and the analysis of PR spectrum becomes complicated and difficult. Three beam PR combined with the Fourier analysis allows us to determine and distinguish between the built-in electric fields existing at different parts of the InGaAsP/InP laser structures.

The possibility of contactless changes of the electric field allows also investigating the intensity of parity forbidden transitions in quantum wells which are usually observed in modulation spectra due to the built-in electric field induced breaking of the selection rules. We propose to use TBPR as an experimental method to examine the nature of the PR lines. We have shown that changes of built-in electric field by amount of a few tenths of kV/cm can modify the intensity of forbidden transitions for InGaAs/GaAs double quantum wells.

M-III.3 15:00

UV PHOTOREFLECTANCE FOR WIDE BAND GAP NITRIDE HETEROSTRUCTURES

C. Bru-Chevallier, S. Fanget, G. Guillot, Laboratoire de Physique de la Matière, UMRCNRS5511 - INSA de Lyon, Bâtiment Blaise Pascal, 7 avenue Jean Capelle, 69621 Villeurbanne Cedex, France, O. Briot, S. Ruffenach, GES, UMRCNRS Université Montepellier II, 34095 Montpellier Cedex5, France

Group III nitride semiconductor compounds have demonstrated their capacity for light emission and have recently gained increasing interest in high power electronics. Most of the work usually addresses the hexagonal phase of GaN which is thermodynamically stable, but in which very high spontaneous and piezoelectric fields are present. Such internal fields strongly influence optical as well as electronic properties of nitride heterostructures and then device performance : either light emitting devices or high electron mobility transistors.

Therefore, the experimental determination of such internal electric fields is unavoidable for the design of devices, and photoreflectance (PR) in the Franz Keldysh regime is demonstrated to be one of the best technique to this goal, as it is a direct all-optical measurement. In the paper several GaN/AlGaN quantum wells with different thickness are studied using photoluminescence (PL), PL excitation and PR in the UV spectral range. From the analysis of Franz Keldysh oscillations of PR spectra, internal electric field values are derived, which are in good agreement with confined state energy in the wells measured by PLE, and calculated taking into account the quantum confined Stark effect.

M-III.4 15:20

THE METROLOGY OF A MINIATURE FT SPECTROMETER MOEMS DEVICE USING WHITE LIGHT SCANNING INTERFERENCE MICROSCOPY

P.C. Montgomery(a), D. Montaner(a), O. Manzardo(b), M. Flury(b) and H.P. Herzig(b), (a)Laboratoire PHASE, CNRS, 23 rue du Loess, BP 20 CR, 67037 Strasbourg cedex 2, France, (b)Institute of Microtechnology, Applied Optics Laboratory, Rue Breguet 2, 2000 Neuchâtel, Switzerland

Micro-optical electro-mechanical systems (MOEMS) technology, making use of existing silicon based fabrication techniques shows great potential for making complete miniaturized hybrid devices. Such technology has been used to make a Fourier transform spectrometer based on a time-scanning Michelson interferometer. An electrostatic comb drive actuator moves the scanning mirror, which has a maximum driving distance of 40 m. The measured resolution of the spectrometer is 6 nm at a wavelength of 633 nm. The dimensions of the device are 5 mm x 5 mm x 0.5 mm, and the depth of features is 75 µm.

During quality control of such devices it is necessary to check the dimensions of micron wide structures that are tens of microns deep, over areas of tens of square millimeters. In this work we have used white light scanning interference (WLSI) microscopy to measure the different structures present on the device, in order to determine some of the challenges in making precision 3D measurements non-destructively. We investigate such problems as shadowing produced by the deep narrow structures used in the comb actuator, the effect of using different envelope determination algorithms on the measured shape of narrow structures and the need to measure with a high lateral resolution over large areas. The results of this work demonstrate that WLSI, with certain modifications, shows great potential for the rapid and precise quality control of MOEMS devices.

M-III.5 15:40

METHODOLOGY FOR CONTACTLESS CHARACTERIZATION OF HIGH ELECTRON MOBILITY TRANSISTOR STRUCTURES USING SURFACE PHOTOVOLTAGE SPECTROSCOPY

S. Solodky, A. Kramtsov, T. Baksht, M. Leibovitch and Yoram Shapira, Tel-Aviv University, Electrical Engineering, 69978 Tel-Aviv, Israel

AlGaAs/InGAs/GaAs pseudomorphic HEMT (PHEMT), AlGaN/GaN high electron mobility transistor (HEMT) and InAlAs/InGaAs metamorphic HEMT (MHEMT) epitaxial structures have been characterized using surface photovoltage spectroscopy (SPS). The interplay between two opposite direction signals coming from the regions with opposite direction of electric fields define the shape of the spectra. The shape of the spectra is interpreted using self-consistent numerical simulations. The effects of the transistor delta-doping levels dtop, dbot and surface charge Qsur on the spectrum features have been studied using numerical simulations. Based on the latter, an empirical model has been developed, which allows extraction and comparison of dtop, dbot and Qsur and is applicable for both double-sided and single sided delta-doped structures. Prediction of the final device performance by the model is shown for two MHEMT structures, demonstrating the sensitivity of the methodology. Applying the model shows a relative difference of 7.5% in dtop of the two structures. Devices produced on these structures show a relative difference of 8.2% in their maximum drain currents, which correlates well with the dtop value calculated using the model.

16:00 **BREAK**

Session IV: Applications: X-ray metrology
Session chair: Sandro Ferrari and René Guinebretière

M-IV.1 16:20 -Invited-

TEXTURE, RESIDUAL STRESS AND STRUCTURAL ANALYSIS OF THIN FILMS USING A COMBINED X-RAY ANALYSIS

L. Lutterotti(a,b), D. Chateigner(c), S. Ferrari(d) and J. Ricote(e), (a)Dipartimento di Ingegneria dei Materiali, Univ. di Trento, 38050 Trento, Italy, (b)Earth and Planetary Science Department, Univ. of California, Berkeley CA 94720, USA, (c)Laboratoire de Cristallographie et Sciences des Matériaux-ISMRA, 14050 Caen, France, (d)Materials and Devices for Microelectronics Laboratory, INFM. Via C. Olivetti 2, 20041 Agrate Brianza, Milano, Italy, (e)Instituto de Ciencia de Materiales de Madrid, CSIC, Cantoblanco, 28049 Madrid, Spain

Advanced thin films for today industrial and research needs require highly specialized methodologies for a successful quantitative characterization. In particular, in the case of multilayers and/or unknowns phases a global approach is necessary to obtain some or all the required information.

A full approach has been developed integrating novel texture and residual stress methodologies with the Rietveld method (for crystal structure analysis) and it has been coupled with the reflectivity analysis. The complete analysis can be done at once and offers several benefits: the thicknesses obtained from reflectivity can be used to correct the diffraction spectra, the phase analysis help to identify the layers and to determine the electron density profile for reflectivity; quantitative texture is needed for quantitative phase and residual stress analyses; crystal structure determination benefits of the previous. To achieve this result, it was necessary to develop some new methods, especially for texture and residual stresses. So it was possible to integrate them in the Rietveld, full profile fitting of the patterns. The measurement of these spectra required a special reflectometer/diffractometer that combines a thin parallel beam (for reflectivity) and a texture/stress goniometer with a curved large position sensitive detector. This new diffraction/reflectivity X-ray machine has been used to test the combined approach. Several spectra and the reflectivity patterns have been collected at different tilting angles and processed at once by the special software incorporating the aforementioned methodologies. Some analysis examples will be given to shown the possibilities offered by the method.

M-IV.2 17:00

IN-LINE MONITORING OF ADVANCED MICROELECTRONIC PROCESSES USING COMBINED X-RAY TECHNIQUES

D. Delille(d), J.P. Gonchond(c), F. Heider(e), L. Kwakman(d), S. Marthon(a), I. Mazor(b), A. Michallet(a,c), D. Muyard(a), L. Perino Gallice(d), J.C. Royer(a), A. Tokar(b), C. Wyon(a,c) (a)CEA-LETI, CEA Grenoble, 17 rue des Martyrs, 38054 Grenoble, France, (b)Jordan Valley, 103, Ramat Gavriel Industrial zone, 23100 Migdal Haemek, Israel, (c)STMicroelectronics, 850 rue Jean Monnet, 38926 Crolles, France, (d)Philips Semiconductors, 850 rue Jean Monnet, 38926 Crolles, France, (e)Infineon Technologies, Siemensstrasse 2, 9500 Villach, Austria

Accurate and reliable in-line monitoring of the various layers thickness involved during the whole integrated circuits manufacturing process is mandatory to develop and produce advanced microelectronic devices.
X-Ray Reflectivity (XRR) is a quite relevant metrology technique to precisely determine the thickness of both transparent and metallic thin and ultra-thin films. Furthermore X-Ray Reflectivity is very sensitive to surface and interface roughness, and also provides information about the layers density. X-Ray Fluorescence (XRF) is currently used as a metrology technique to control the thickness and the elemental composition of relatively thick films. In the frame of an European project, performance of a new in-line metrology tool, which gathers XRR and XRF to monitor thickness of films deposited on 200 and 300mm silicon wafers, have been assessed. Preliminary results on the monitoring of HfO2 thin films, SiGe and SiGeC epilayers, nickel, cobalt and tungsten silicide layers, copper barrier and copper seed layers using this tool will be presented.

M-IV.3 17:20

NEW DEVELOPMENTS IN X-RAY FLUORESCENCE METROLOGY

L.M. van der Haar, C. Sommer and M.G.M. Stoop, PanAlytical, XRF Semiconductor Metrology, PO Box 13, 7600 AA Almelo, The Netherlands

In the recent past, X-ray fluorescence (XRF) has proven to be a reliable, non-destructive technique for investigating thickness and composition of thin films and multilayers. Combined with fundamental parameter analysis, XRF has become a powerful and versatile technique that has found a broad range of applications in the semiconductor industry. Amongst others, applications include the characterization of B an P doped silicon glasses, metals, silicides/salicides, nitrides, barrier layers, oxides, ferroelectrics, polysilicon, GMR stacks and advanced low- and high-k dielectrics. State-of-the-art XRF techniques allow concentration monitoring of light elements like B in silicon glass with a relative precision better than a few tenths of a percent. However, as in-line metrology is increasingly adopted throughout the industry and metrology is moving from monitor to product wafers, new XRF metrology challenges arise. This has given a boost to the development of micro-focus X-ray equipment with respect to optics and detectors that enable fast, on-product measurements. An overview will be given of a number of the above-mentioned XRF applications and the issues that have to be addressed for on-product measurements. Results will be shown from micro-spot XRF experiments on films and film stacks. Important factors that govern the system performance are described and an outlook for future developments is given.

M-IV.4 17:40

X-RAY MATERIAL CHARACTERIZATION BY USING OF RADIATION BEAMS FORMED BY PLANAR AIR WAVEGUIDE-RESONATORS

V.K. Egorov, E.V. Egorov, IPMT RAS, Chernogolovka, Moscow district 142432 Russia

The work is described TXRF spectrometer and X-ray diffractometer equipped by waveguide-resonance formers of an radiation beam. There is the analysis of the metrology peculiarities of these instruments and the detail description of experimental diffraction patterns and TXRF spectra obtained at using of waveguide-resonators. Application perspectives of these devices for the chemical analysis of target surface layers and phase and structure control of bulk targets and planar nanostructures are discussed.
Surprising analytical possibilities of TXRF spectrometers and diffractometers had in their arrangement the planar X-ray waveguide-resonators (PXWR) are defined by unique characteristics of beams formed by the resonators. The beam after PXWR has a standard height h=10 mm, a very small width s~50 nm, low divergence \Delta\theta~0.2° and very high radiation density. At the power of a conventional X-ray source P~200 W the copper characteristics beam after quartz PXWR shows the radiation density near 1*10^12 quant/cm^2sec. Small size of the beam width is the beautiful prerequisite to elaborate high resolution X-ray tomographs. PXWR can be put into practice of the X-ray reflectometry with success [1]. [1] V.K. Egorov, E.V. Egorov, MRS Proceedings, v716, 2002, pp. 189-195.

Wednesday, June 11, 2003 — Afternoon
Mercredi 11 juin 2003 — Après-midi

Session V: Optical characterisation of organic materials
Session chair: Pat Kelly and Ravin Ginige

M-V.1 14:20 -Invited-

OPTICAL CHARACTERISATION OF FULLERENES AND CARBON NANOTUBES: IN SITU MEASUREMENTS AND CORRELATION WITH ELECTRONIC PROPERTIES

Eleanor E.B. Campbell, S. Dittmer, M. Sveningsson, A.V. Gromov, V. Popok, A. Lassesson, Dept. of Experimental Physics, School of Physics and Engineering Physics, Göteborg University and Chalmers, 41296 Göteborg, Sweden

Carbon nanotubes and fullerenes are among the most promising materials for possible future applications in the area of nanoelectronics and nanoelectromechanics. This is partly a consequence of their dimensions (diameters typically on the order of 1 nm) but most especially because of the range of interesting properties they show. Carbon nanotubes are especially fascinating because of their extremely favourable mechanical properties as well as their wide range of electronic properties depending on the geometry of the carbon tube. In this contribution we will present results on combining the optical characterisation of nanotubes and fullerenes with direct measurements of thier electronic properties. In particular, we want to focus on the in situ Raman characterisation of individual carbon nanotubes integrated as devices in circuits where it is possible to directly correlate the transport properties with the structural information obtained from single nanotube micro-Raman spectroscopy. Raman spectroscopy is capable of providing information on nanotube diameter and chirality. We will also discuss optical characterisation and transport measurements of endohedral fullerenes.

M-V.2 15:00 -Invited-

BARRIERLESS ELECTRON-HOLE CAPTURE IN POLYMER BLEND HETEROJUNCTION LIGHT-EMITTING DIODES INVESTIGATED WITH TIME-RESOLVED PHOTOLUMINESCENCE SPECTROSCOPY

Arne C. Morteani, Anoop S. Dhoot, Ji-Seon Kim, Neil C. Greenham, Richard H. Friend and **Carlos Silva**, University of Cambridge, Cavendish Laboratory, Madingley Road, Cambridge CB3 0HE, U.K.

We discuss the application of time-resolved optical spectroscopy to the characterisation of novel organic optoelectronic devices. Organic semiconductors, for example, fabricated as thin-film light-emitting diodes (LEDs) now provide a promising new display technology. We have fabricated distributed heterojunction structures that are formed by de-mixing of two polymers co-deposited from common solution. We have used hole-accepting and electron-accepting derivatives of polyfluorene, and have optimized these structures to achieve high efficiency diodes (>19lm/W) that operate at very low voltages (100cd/m2 at 2.1V). This is achieved because electron-hole capture across the heterojunction is arranged to be a barrier-free process to form a bound interface state (an exciplex) which has significant charge-transfer character and is lower in energy than the charge-separated state. With respect to the bulk exciton, the exciplex is red-shifted (here between 140 and 360meV) and its radiative lifetime is strongly increased (between 68 and 118ns at low temperatures). The barrier for thermal excitation of the exciplex to allow it to move away from the heterojunction is small (100-250meV), and this process gives efficient bulk exciton emission at room temperature. We will provide an overview of femtosecond and picosecond-resolved spectroscopies and their applications in organic semiconductor physics.

M-V.3 15:40

OPTICAL SPECTROSCOPIC METHODS APPLIED TO LUMINESCENT ORGANIC MATERIALS

B. Servet, M. Vergnolle, P. Le Barny, Thales R&T Fr, Domaine de Corbeville, 91404 Orsay cedex, France, J. Schreiber, Li Long, Fachbereich Physik,, Martin-Luther-Universität Halle-Wittenberg, Friedemann-Bach-Platz 6, 06108 Halle, Germany, M. Modreanu, B. O'L'oney, NMRC, Lee Marltings, Prospect Row, Cork, Ireland

Luminescent organic materials including conjugated polymers and small molecules provide a wide range of new active semiconductors designed for organic optoelectronic devices and particularly for organic light emitting diodes (OLEDs) with potential integration in flat panel displays. The understanding of their optical properties in both materials and devices is crucial for such applications and has required the development of non-destructive optical methods.

We report here the progress in optical spectroscopic methods applied to luminescent organic materials. Optical absorption in the UV-visible range provides the bandgap energy from the low-energy absorption band edge. Photoluminescence (PL) spectra consist of multiple optical transitions due to exciton-phonon coupling, which can result in a broad PL signal with an apparent red shift of its maximum below the bandgap. These PL features are similar to those of electroluminescence (EL) performed on OLED structures using ITO as a transparent electrode, with small differences in peak position and FWHM depending on the material. In addition to conventional spectroscopies, we demonstrate that photoreflectance (PR) can be applied to OLED structures so as to provide the accurate bandgap energy of the organic layer, in good agreement with optical absorption results.

M-V.4 16:00 STRUCTURAL AND OPTICAL PROPERTIES OF BOTH PURE POLY(3-OCTYLTHIOPHENE) (P3OT) AND P3OT/FULLERENE FILMS

Tobias Erb, Sofiya Raleva, Uladzimir Zhokhavets, Gerhard Gobsch, Bernd Stühn, Institute of Physics, Ilmenau Technical University, 98684 Ilmenau, Germany, Matthias Spode, Oliver Ambacher, Centre of Micro- and Nanotechnology, Ilmenau Technical University, 98684 Ilmenau, Germany

P3OT and P3OT/fullerene composites are promising materials for plastic solar cells.

Films of these materials were prepared by spin coating from chloroform, chlorbenzol+dichlormethan and toluol solutions onto silicon substrates. Their structural properties were studied using X-ray reflection. The results provide information on film thickness, surface roughness and electron density variation perpendicular to the film surface. The degree of anisotropy of the films depends strongly on the film composition, film thickness, solvent type and polymer concentration in the solvent. In addition, the optical properties of the samples were studied by means of spectroscopic ellipsometry within the spectral range 1.2 ... 5.0eV, which provides the anisotropic dielectric function of the films. A close correlation between the results obtained by both methods could be found. Especially, the strong optical anisotropy of the films can be explained in terms of a preferable orientation of the polymer chains parallel to the substrate. The influence of the optical anisotropy on the performance of optoelectronic devices is discussed.

16:20 **BREAK**

16:45-17:30 WORKSHOP

MODULATION AND ANISOOTROPIC REFLECTANCE SPECTROSCOPIES

Organized by EU project GRD-1999-10535-MARS

Thursday, June 12, 2003 — Morning
Jeudi 12 juin 2003 — Matin

Session VI: Material characterisation: spectroscopic ellipsometry
Session chair: Jeff Hosea and Mircea Modreanu

M-VI.1 9:00 -Invited-

GENERALIZED ELLIPSOMETRY FOR MATERIALS CHARACTERIZATION

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Ellipsometry experiments normally measure 2-4 parameters, which are converted to the ellipsometric parameters Psi and Delta. This is usually sufficient for many samples, but more complicated situations (such as anisotropic or depolarizing samples) require more sophisticated measurements.

Over the last 7 years, we have developed the two-modulator generalized ellipsometer (2-MGE), which measures 8 elements of the sample Mueller matrix simultaneously either in reflection or transmission. In reflection, the 2-MGE totally characterizes light reflection from anisotropic samples, measuring the normal ellipsometry parameters, as well as the cross-polarization and depolarization effects. Applications include the determination of the spectroscopic optical functions of uniaxial materials, such as TiO_2, ZnO, and BiI_3, and the measurement of cross-polarization from diffractive structures. In transmission, the 2-MGE completely characterizes a general linear diattenuator and retarder. Applications include the measurement of the retardation and diattenuation of film polarizers and internal electric fields in $LiNbO_3$ and CdZnTe under bias.

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M-VI.2 9:40

OPTICAL CHARACTERIZATION OF INDIUM-TIN-OXYNITRIDE FABRICATED BY RF-SPUTTERING

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Indium-Tin-Oxynitride (ITON) thin films have been fabricated by rf-sputtering from an Indium-Tin-Oxide target in plasma containing a mixture of Ar-N_2 gases. ITON films fabricated under different sputtering conditions were characterized employing different optical characterization techniques, like XRD, FTIR, ellipsometry and Reflectance- Transmittance in the whole DUV-IR spectrum range. The optical properties of ITON films were compared with those of Indium-Tin-Oxide (ITO) films fabricated in pure Ar plasma under the same sputtering conditions. The microstructure of ITON films was found to be dependent on the nitrogen concentration in plasma and plasma conditions. The absorption edge of the ITON films was shifted to shorter wavelengths and showed reduced infrared reflectance, when compared to the respective properties of ITO films. The potentialities of indium-tin-oxynitride films for use as a new transparent optical material for opto-electronic devices will be addressed.

M-VI.3 10:00

OPTICAL CHARACTERIZATION OF ILSCVD HfO_2 HIGH-k DIELECTRICS

M. Modreanu, P.K. Hurley, B.J. O'Sullivan, B. O'Looney, NMRC, University College, Lee Maltings Prospect Cork, Ireland, J.P. Senateur, H. Rousell, F. Rousell, M. Audier C. Dubourdieu, C. Jimenez, LMGP - ENSPG, INPG - UMR CNRS 5628, BP 46, 38402 Saint Martin d'Heres, France, I.W. Boyd, Q. Fang, Dept. Electronic and Electrical Eng., University College London, U.K., T.L. Leedham, H. Davies, Epichem Oxides and Nitrides, Mildenhall, Suffolk, U.K., S. Rushworth, A.C. Jones, Epichem Group, Bromborough, Wirral, U.K., H. Guillon, J.M. Decams, Qualiflow-Jipelec, 350 Rue Alfre Nobel, BP7, 34935 Montpellier Cedex 9, France

HfO_2 thin films have been deposited by Thermal and UltraViolet assisted Injected Liquid Sources CVD (ILSCVD) techniques. HfO_2 thin films with thickness varying from 2-40 nm were formed over a range of temperatures (300-425oC). After deposition the samples were annealed by Rapid Thermal Annealing (RTP) at 800oC in an oxygen/argon ambient or by UV annealing at 400oC in oxygen. The films were analysed physically using variable angle spectroscopic ellipsometry (VASE), XRD, FTIR and High Resolution TEM. The XRD results show that as-deposited HfO_2 films microstructure strongly depends on deposition temperature. Both polycrystalline (T>365oC) and amorphous films (T<320oC) were formed. The polycrystalline structure is identified as monoclinic. After annealing, due to the solid phase crystallisation, as-deposited amorphous-like HfO_2 thin films become crystalline and the film porosity is strongly reduced. Optical properties of the as-deposited polycrystalline HfO_2 are also improved after annealing: an increase of the refractive index and a decrease of the film thickness is obtained. The dispersion of the refractive index for the HfO_2 thin films were characterised using Wemple and Di Domenico the single effective oscillator model. An optical bandgap around 5.77eV was found in good agreement with the literature.

M-VI.4 10:20 KINETICS OF INTERFACIAL LAYER FORMATION DRUING DEPOSITION AND POST-ANNEALING OF HIGH K DIELECTRICS ON SILICON

V. Craciun, C. Essary, N. Bassim, J.M. Howard and R.K. Singh, Materials Science and Engineering, University of Florida, Gainesville FL 32611, USA

One of the critical problems associated with the use of high-k dielectrics as replacements for SiO2 gate oxides is the formation of an interfacial layer with the Si substrate that has detrimental effects on the overall capacitance of the structure. This presentation explores the oxygen source and kinetics of formation of this interfacial layer. ZrO2, HfO2, and Y2O3 films were deposited on Si substrates using a pulsed laser deposition technique and were post-annealed under various atmospheres, temperatures and times. Using several characterization techniques such as x-ray reflectivity and diffraction, angle-resolved x-ray photoelectron spectroscopy, variable angle spectroscopic ellipsometry and, high-resolution transmission electron microscopy with an EELS attachment, the thickness, density, roughness and chemical bonding of the interfacial layer were determined. These results showed that the interfacial layer mainly consists of a physical mixture of SiO2 and the deposited oxide and that silicates do not form at temperatures below 600C. It also appeared that the kinetics of the interfacial layer growth do not follow similar models developed for the oxidation of silicon. Further comparisons were made with samples that contained a nitride pretreatment of the Si substrate to study the nitridation effectiveness in slowing the interfacial layer growth.

10:40 **BREAK**

Session VII: Materials- X-ray characterisation (I)
Session chair: Daniel Chateigner and Lourdes Calzada

M-VII.1 11:00 -Invited- STRUCTURAL CHARACTERIZATION OF MULTILAYERED MATERIALS FOR OPTOELECTRONIC COMPONENTS BY HIGH-RESOLUTION X-RAY DIFFRACTOMETRY AND REFLECTOMETRY: CONTRIBUTION OF NUMERICAL TREATMENTS

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For recent years, the market of multilayered optoelectronic devices is growing rapidly and, therefore, some innovative approaches are made on novel semi-conductors-based optical components. Complementary to the knowledge of their physical properties, realization of all this devices implies the mastering of the material elaboration. Therefore, it is of great importance to be able to characterize the stacking structure of these multilayered optoelectronics materials. It is given here an approach of the structural analysis of thin-films materials by both x-ray reflectometry and diffractometry with a scope centered on applications to real cases from optoelectronic domain. To this end, we propose methods to extract information on individual layer thicknesses contained in the x-ray reflectivity profiles and the diffractometry spectra. Since the complexity of multilayered materials for optoelectronics is increasing dramatically, leading to complex x-ray diffraction and x-ray reflectometry spectra, the extraction of information using simulation and fitting procedures is becoming very time-consuming. We show that the assessment to precise and fast determination of individual layer thicknesses appears possible using FFT-based tools, even in the case of complicated stacking. Actual and non-exhaustive examples, from studies developed in the Thales research lab, will illustrate all these different methods.

M-VII.2 11:40 AUTOMATED METROLOGY SYSTEM INCLUDING VUV SPECTROSCOPIC ELLIPSOMETRY AND X-RAY REFLECTOMETRY FOR 300MM SILICON MICROELECTRONICS

P. Boher, P. Evrard, O. Condat, C. Dos Reis, C. Defranoux SOPRA, 26 rue Pierre-Joigneaux, 92270 Bois-Colombes, France, E. Bellandi, STMicroelectronics, Central R&D, Via C.Olivetti 2, 20041 Agrate Brianza (MI), Italy

Characterization of complex structures including very thin layers is now a quite common need in different fields of microelectronics. The use of more than one characterization technique becomes mandatory in numerous cases to get a clear picture of the structural properties of the samples. In the proposed paper, we use vacuum ultraviolet spectroscopic ellipsometry (VUVSE) and x-ray reflectometry (XRR) on the same instrument. VUVSE is more sensitive than conventional ellipsometry to surface and interface roughness because of the wavelength reduction and also of the increase absorption of all the materials in this region. XRR is also very sensitive to roughness and is capable to measure layer thickness of very thin films without any need of model. So, the two techniques are very complementary.

A new automated metrology system dedicated to 300mm silicon microelectronics has been developed. It is working in purged environment to reduce the oxygen and water moisture in the part per million range. The optical set-up includes a pre-monochromator in the polariser arm to avoid photo-bleaching. The wavelength range of the instrument is 145-720nm. The system works in rotating analyser configuration to minimize the parasitic residual polarisation. A x-ray reflectometer is also included on the same set-up. A load lock capable to introduce a complete cassette, a robot to handle automatically the wafers, a rho/theta mapping stage and automatic adjustment of the samples are also included. The proposed paper will present in details the new system and experimental results on high k dielectrics and epitaxial layers.

M-VII.3 12:00

METROLOGY ISSUES IN THIN ONO STACKS MEASUREMENT BY SE AND XRR

A. Elbaz, R. Piagge, E. Bellandi, S. Spadoni, C. Coccorese, G. Pavia, M. Alessandri, STMicroelectronics, via C. Olivetti 2, 20041 Agrate Brianza (MI) Italy, S. Ferrari, Laboratorio MDM-INFM, via C. Olivetti 2, 20041 Agrate Brianza (MI) Italy, S. Banerjee, Saha Institute of Nuclear Physics,1/AF Bidhan Nagar, Calcutta 700064, India, P. Boher, SOPRA, 26 rue Pierre Joigneaux, 92270 Bois Colombes, France

Thin oxide-nitride-oxide (ONO) structures are used in non-volatile memories as an inter-poly dielectric. 2002 ITRS Roadmap forecasts an aggressive scaling down of the thickness, especially for NOR Flash Memories. Accuracy and repeatability of thickness measurements for the three layers are key points for device development and in-line process control.

We performed X-ray reflectivity (XRR) and Spectroscopic Ellipsometry (SE) analysis of several ONO stacks with different deposition conditions. TEM and deep UV SE (PUV-SE) were used to validate SE and XRR measurements. In the energy range from 1.5 to 6.5eV, SE analysis shows a correlation between bottom and top oxide thickness. The small electron density difference between Si and SiO2 makes the SiO2/Si interface determination difficult to estimate by means of XRR. Comparing the results of both techniques, we have observed that SE total thickness is higher respect to XRR and TEM. A small shrinkage and a higher density of bottom oxide are detected by XRR after a densification treatment. Physical phenomena of the process have been also discussed.

M-VII.4 12:20

X-RAY REFLECTIVITY AND SPECTROSCOPIC ELLIPSOMETRY AS METROLOGY TOOLS FOR THE CHARACTERIZATION OF INTERFACIAL LAYERS IN HIGH-K MATERIALS

S. Ferrari, Laboratorio MDM – INFM, Via Olivetti 2, 20041 Agrate Brianza (Mi), Italy, M. Modreanu and P.K. Hurley, NMRC, University College, Lee Maltings Prospect Cork, Ireland, G. Scarel and M. Fanciulli, Laboratorio MDM – INFM, Via Olivetti 2, 20041 Agrate Brianza (Mi) Italy

Metal oxides (MO) of high dielectric constant are candidates to substitute SiO2 as gate dielectric in complementary metal oxide semiconductor (CMOS). Recently microelectronic industry focused his interest manly on ZrO2, HfO2 and his silicates. The interfacial layer between the MO and silicon formed either during the deposition or during post-deposition processes should be controlled very carefully. X-ray reflectivity (XRR) and spectroscopic ellipsometry (SE), are excellent tools for the characterization of the interfacial layer, but several metrological challenges arise when one considers the complexity of this system. In this paper we study the growth of interfacial SiO2 upon annealing in O2 by means of XRR and SE. The analysis of SE data is made with a common optical model adjusting the layer thickness and the surface and interface roughness, while thickness and interface composition are extracted from XRR data by means of the matrix method. The poor electron density contrast between SiO2 and Silicon as opposed to the strong one between HfO2/ZrO2 and Si makes the extraction of structural data from XRR data extremely difficult. Yet we show that interfacial oxide growth can be measured by XRR, that in addition provide information on interdiffusion phenomena occurring between the high-k layer and the interfacial SiO2. The result shows that tby combining XRR and SE techniques a comprehensive microstructural and optical characterization of high-k thin films can be made.

12:40 **LUNCH**

Session VII: Materials- X-ray characterisation (II)
Session chair: Sangam Banerjee and P. Evrard

M-VIII.1 14:00 -Invited- ELASTIC-STRAIN TENSOR AND INHOMOGENEOUS STRAIN IN THIN FILMS BY X-RAY DIFFRACTION

D. Balzar, Department of Physics and Astronomy, University of Denver, 2112 E Wesley Ave, Denver CO 80208, USA and N.C. Popa, Frank Laboratory of Neutron Physics, Joint Institute for Nuclear Research, 141980 Dubna, Moscow region, Russia

Strains and defects have an important role in shaping materials and device properties. X-ray diffraction is a nondestructive, reliable, and quick technique to study both residual strains (as determined from the diffraction-line shifts) and inhomogeneous strains associated with defects (as determined from the diffraction-line broadening and shift).

Residual strains are customary determined from the line shifts of a single diffraction line by using the so-called sin2Ψ method. We present a modified approach that is based on the Rietveld refinement of the whole diffraction pattern. The method allows for the determination of the texture-weighted complete strain and stress tensors as a function of crystallite orientation, as well as the average values of the macroscopic strain and stress tensors. We also report on the studies of residual elastic and inhomogeneous strain in BaSrTiO3 thin films, which are affected by different preparation conditions and doping. To explain the induced changes in permittivity and Curie-Weiss temperature, we model the dielectric and ferroelectric properties of interest in the framework of Landau-Ginsburg-Devonshire thermodynamic theory and obtain a good agreement with the measurements.

M-VIII.2 14:40 APPLICATION OF THE X-RAY COMBINED ANALYSIS TO THE STUDY OF LEAD TITANATE BASED FERROELECTRIC THIN FILMS

J. Ricote(a), D. Chateigner(b), M.L. Calzada(a), C. Wiemer(c) and L. Lutterotti(d), (a)Instituto de Ciencia de Materiales de Madrid, CSIC, Cantoblanco, 28049 Madrid, Spain, (b)Laboratoire de Cristallographie et Sciences des Matériaux-ISMRA, 14050 Caen, France, (c)Materials and Devices for Microelectronics Laboratory, INFM, Via C. Olivetti 2, 20041 Agrate Brianza, Milano, Italy, (d)Dipartimento di Ingegneria dei Materiali, Univ. di Trento, 38050 Trento, Italy

Lead titanate based ferroelectric thin films are well known for their excellent properties that make them good candidates for several applications in microelectromechanical systems, dynamic and ferroelectric random access memories (DRAM and FeRAM) and also in high frequency components. In order to obtain a complete characterisation of the structural parameters, texture and stress state of these films, a recently developed combined analysis of the X-ray diffraction data is carried out. The advantages of this approach reside in the fact that we obtain simultaneously quantitative and more reliable information of the microstructural parameters of the films, as a cyclic Rietveld, quantitative texture analysis and stress analysis is performed. The method avoids artefacts like bias coming from refining independently one or some given parameters (i.e. unit-cell) when these are intrinsically correlated to others (i.e. residual stresses), since the refinement converges to the best solution of the whole ensemble of parameters of importance. Results obtained for the ferroelectric films are analysed and compared to others obtained with more conventional techniques.

The study of a series of films with the same composition deposited on different platinised substrates will shed light onto the influence of the stress developed during the processing on the microstructure of the resulting film. The small structural differences introduced by the different amount of Ca doping of the PbTiO3, which are usually difficult to detect with high accuracy in thin films, will also be analysed with this method.

M-VIII.3 15:00 COMBINING GRAZING INCIDENCE X-RAY DIFFRACTION AND X-RAY REFLECTIVITY FOR THE EVALUATION OF THE STRUCTURAL EVOLUTION OF HFO2 THIN FILMS WITH ANNEALING

C. Wiemer, S. Ferrari, M. Fanciulli, MDM Laboratory INFM, via C. Olivetti 2, Agrate Brianza (Milano) 20041, Italy, G. Pavia, STMicroelectronics, via C. Olivetti 2 Agrate Brianza (Milano) 20041, Italy, L. Lutterotti, University of Trento, Department of Materials Engineering, via Mesiano 77, Trento 38050, Italy

Metal oxides of high dielectric constant are candidates to substitute SiO2 as gate dielectric in complementary metal oxide semiconductor (CMOS) technology. A lower equivalent oxide thickness and a higher physical thickness are necessary in order to maintain the gate capacitance and reduce the leakage currents. Surface and interfacial roughness, as well as crystallinity and thickness are fundamental characteristics that influence the electric properties of the gate oxide. For atomic layer deposited (ALD) oxides, properties such as degree of crystallographic order and interface morphology are extremely difficult to investigate. In this contribution we present the application of x-ray techniques to the study of thermal stability of 10 nm thick HfO2 deposited by ALD. Grazing incidence x-ray diffraction spectra are refined by Rietveld analysis. X-ray reflectivity data are fitted in order to evaluate the thickness, the surface roughness and the interface stability upon thermal processing between 300 and 1050 °C. The electronic density values obtained from the scattering coefficients of the crystallographic phase are compared to the values related to the critical angle of the x-ray reflectivity spectra. The thickness and the electronic density of the as grown layer are not affected by the thermal treatment. The surface and interfacial roughness are also very stable, although the SiO2 native oxide layer increases with annealing. The crystallographic ordering evolves starting from the amorphous structure of the as deposited film towards the polycrystalline monoclinic HfO2 films obtained by annealing at high temperatures. The results are discussed and compared to plan view and cross sectional transmission electron microscopy images.

M-VIII.4 15:20

APPLICATION OF X-RAY SCATTERING METHODS TO THE ANALYSIS OF SI BASED HETEROSTRUCTURES

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High resolution X-ray diffraction, X-ray reflectivity and X-ray diffuse scattering have been applied to study various device type SiGe heterostructures. For device structures the tolerances on thickness and composition are tight. Therefore there is a high demand for reliable and accurate characterization techniques. The main purpose of the present study is to combine the complementary information content of the different X-ray scattering techniques in order to obtain a sample model that applies sufficiently to all data sets. Due to the versatility of modern laboratory X-ray equipment all measurements using these non-destructive techniques can be easily performed on one single instrument just by exchanging optical modules. Quantitative information about compositions, thicknesses, interface layers, interface roughnesses and lateral correlation lengths is extracted from the measured data. This almost complete structural picture of the samples is obtained by full pattern simulation of the corresponding experimental profiles. Starting from a model based on the growth sequence, the layer parameters are adjusted until a close match with the experimental data is achieved. The availability of fast and reliable automatic refinement algorithms speeds up and simplifies the analysis process significantly. In this paper the experimental issues and the evaluation process of the structural characterization are discussed for some typical layer structures. All steps of the analysis were performed by means of commercially available software and hardware.

M-VIII.5 15:40

RESIDUAL STRESS ANALYSIS OF THIN FILMS AND COATINGS THROUGH XRD2 EXPERIMENTS

M. Gelfi, E. Bontempi, R. Roberti and L.E. Depero, INSTM and Dipartimento di Ingegneria Meccanica, Università di Brescia, Via Branze 38, 25123 Brescia, Italy

Residual stresses are one of the crucial parameters determining the performances of structural as well as functional materials. In the case of coatings and films, the substrate and the deposition process may determine very high residual stress fields which can affect both the in service behaviour and the coating or film integrity, as their adhesion or cracking resistance may be fundamentally modified. The accurate and reliable assessment of residual stress is thus mandatory.

In this paper we propose a non destructive method for the evaluation of the residual stress by means of the analysis of a single 2D diffraction image collected by a laboratory X-ray microdiffractometer equipped with an image plate detector (XRD2). The diameter of the incident beam can be changed by collimators ranging from 800 to 10 microns, allowing the mapping of the stress field on the surface. Examples will be discussed to show the advantages and the potentialities of this approach.

16:00 **BREAK**

16:20 POSTER SESSION

M/P.01 CONTROLLABLE FABRY-PEROT INTERFEROMETER BASED ON DYNAMICS VOLUME HOLOGRAMS

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Fabry-Perot interferometers are widely known and used in different applications. The presented scheme of the interferometer is based on a volume hologram recorded in photorefractive BaTiO3 crystal.

A volume holograms exhibit only one single spectral response and therefore interferometer based on a volume hologram consequently contains also only one single response and the problems connected with the "free spectral range" are not exist anymore. The dynamic nature of the phase hologram in BaTiO3 allows to control the transfer function of the interferometer in the on-line regime via the transformation of the recording beams phase profile. The demonstrated technique of the two-phase shifted holograms is an efficient tool to increase the angular and the spectral selectivity and provides the additional technical possibilities. The achieved angular and spectral selectivity are of the order of 10-7 ... 10-8. The presented theory is in the perfect agreement with the experimental data. We discuss and demonstrate different possible applications of the presented interferometer.

M/P.02 XRD2 EXPERIMENTS IN THIN FILMS AND MULTILAYERS ANALYSIS

E. Bontempi(a), F. Casoli(b), F. Albertini(b) and L.E. Depero(a), (a)INSTM and Laboratorio di Strutturistica Chimica - Università di Brescia, Via Branze 38, 25123 Brescia, Italy, (b)IMEM - CNR, Parco Area delle Scienze 37/A, 43010 Fontanini, Parma, Italy

In these years, a lot of efforts have been devoted in the development of new innovative materials. Among them, thin films and multilayers show an enormous potentiality in different and strategic fields.

For example sensors, protective coatings, and magnetic devices are usually based on these materials. Thus, the development of non destructive analysis for their characterisation is required. It is well known that structure and microstructure strongly influence the functional as well as the structural properties of these materials. The structural characterisation is usually based on glancing incidence X-ray diffraction (GIXRD) and transmission electron microscopy (TEM). The evaluation of the preferred orientation is generally neglected and rarely pole figures experiments have been performed. In this communication we show that by the analysis of 2D images collected by means of a laboratory X-ray microdiffractometer (XRD2) allows to non destructively evaluate the structure, the microstructure, and the preferred orientation of the samples. The spatial resolution is given by collimators with diameter ranging from 800 to 10 microns, allowing the mapping of the surface and the quality control of the layer(s). Co/Au multilayer on Si(100) substrates deposited at different Ar pressure has been analysed and the relationship among Ar pressure, structural properties, and magnetic properties will be discussed.

M/P.03 SMALL-ANGLE X-RAY SCATTERING STUDY OF THE MnO2 SEMICONDUCTOR THIN FILMS POROUS STRUCTURE

L. Skatkov, PCB "Argo", Israel, P. Cheremskoy, V. Gomozov, National Technical University, Kharkov, Ukraine

MnO2 smiconductor films is an important material for application in thin films microelectronics device such as MDS capacitors. For practical application of MnO2 (prepared by thermal deposition of Mn(NO3)2) the characterization of their submicropores (SMP) is critically important.

In the present work the surface morfology, concentration and size distribution of SMP were investigated by small-angle X-ray scattering (SAXS) and scanning electron microscopy (SEM) methods. Since the size distribution to be polymodal, we classified the pores into 4 groups, according their size. By the absence of niticiable anisotropy of SAXS the character of the dispersion indices, which asymptotically obey the Porod law, one can judge that scattering SMP have no domineering orientation, and they are more equiaxial than macro- and micropores, which revealed by SEM. It should be noticed that, unlike macro- and micropores, some equiaxial SM_P's are closed and filled with gas. Their formation is due both clustering oxygen vacancies shaped in MnO2 to the fact that the solidifying rate during the thermal deposition largely exceeds the gas emission rate, thus a part of the gas emitted is trapped in SMP's.

M/P.04 SMALL-ANGLE X-RAY SCATTERING INVESTIGATION OF NEUTRON-IRRADIATED 6H-SiC AND DIAMOND

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Deep understanding of irradiation-induced defects is of paramount importance for application of SiC in electronics and for future fusion reactors. Ion-induced damage of SiC is relatively well understood, but effects of neutron irradiation are much less clear. Mechanisms of the damage and evolution of created defects are different. Large penetration depth of neutrons permits to create uniform damage in the sample crossection. This allows one to use bulk techniques for characterisation of defects. Small-angle X-ray scattering (SAXS) is a powerful tool for investigation of structure of materials on mesoscopic scale, e.g. clusters of point defects, initial stages of defects agglomeration etc., which are difficult object for other techniques. SAXS study of as-grown 6H SiC and CVD diamond films shows existence of relatively large defects with diameter of few tens of nanometers. Several repeating irradiations with reactor neutrons were performed at nominally room temperature. The fluence of each irradiation was (1-3)x10E16 n/cm2/sec. After the irradiation the size of scattering defects increased and their size distribution changed. Most likely these defects are clusters of intrinsic and extrinsic point defects. Models of the defects, evolution of their size distribution with progressive irradiation steps and thermal recovery are discussed.

M/P.05 MODULATION SPECTRA OF SEMICONDUCTING FERROELECTRIC SbSBr

Sh.M. Efendiev, A.A. Akhmedov and E.R. Mustafaev, Azerbaijan Technical University, H. Javid prosp. 25, Baku 370073, Azerbaijan

Photoreflectance spectra of semiconducting ferroelectric compound SbSBr is presented at the wavelenghths around the energy gap region, and at the tem-perature near the Curie point. The temperature dependence of spectra is also investigated. The mechanism for photoreflectance effect in given compounds is discussed. In order to explain the mechanism of photoreflectance in SbSBr, a comparison of the experimental data has been been made both with electroreflectance spectra with the theoretical curve expected under the assump-tion of a periodical change of free carrier density. Temperature dependence of the energy gap position and the ƒ´R/R lineshape is also presented.

M/P.06 VR TRANSMISSION AND CATHODOLUMINESCENCE SPECTRA OF Fe,Co,Ni,Cu:SnO2-BASED OXIDES

V. Brinzari, G. Korotcenkov, N. Syrbu, M. Nazarov, Y. Boris, Technical University of Moldova, bld. Stefan cel Mare 168, 2004 Chisinau, Moldova and J. Morante, A. Cornet, University of Barcelona, Barcelona, Spain

Thin (30-100nm) nano-sized SnO2 films doped by one of the metals from following rank – Fe, Co, Ni, Cu were deposited on quartz or silicon substrates by spray pyrolysis method from mixed aqueous solutions of metal chlorides with stannic chloride as a base component. Structural characterization (SEM, AFM, XRD) has showed the impossibility of determining the second oxide phase due to transition metal except tin dioxide in the doping range up to 16at.%. Study of such spectral optical properties of given films in visible region as transmission and cathodoluminescence allows to obtain new interesting information about the nature and character of incorporation of doping atoms. Introducing already low concentration levels of dopants (<1%) drastically changes the shape of cathodoluminescence spectra and absorption edge in transmission spectra as compared with undoped SnO2. It appears to be the spectra are very sensitive to the film nanostructure, type and amount of doping by transition metal and demonstrate different shapes and nonmonotonic changes due to the level of doping. From our opinion these methods may be used for supplementary characterization of nano-sized oxide films. The attempt of explanation of such behavior and some details of oxide formation are discussed.

M/P.07 BURIED INTERFACE CHARACTERIZATION BY INTERFERENCE MICROSCOPY

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White light interferometry is normally used for analyzing the morphology of single air/material surfaces. Being a far field optical technique, and therefore non-contact, it is possible to measure interfaces buried under a transparent layer which are inaccessible by near field scanning probes. Buried interfaces play a significant role in several fields such as in micro-electronic and in optoelectronic applications. The techniques used to inspect them are often destructive and time consuming.

In this work, interference microscopy has been modified for the direct analysis of the buried interface. We have developed our own test samples, consisting of step structures (< 2 µm) etched in silicon and then covered by a layer of photo resist. We measured the height of the steps before (by stylus, AFM, confocal microscopy and interference microscopy) and after (using only interference microscopy) the deposition of the resist layer. Initial measurements of the profile of the buried silicon step through the transparent layer were made with a precision of 18 %. This figure can be improved by considering the different contributions to the measurement errors. For example, the roughness and the geometry of the air/layer surface and inhomogeneities in the refractive index of the layer can contribute to a degradation of the wavefront reflected from the interface. This work has allowed the development of a new non-destructive technique with great potential for analyzing buried interface structures.

M/P.08 SPECTROSCOPIC CRITERIA OF COMPOSITION OF LITHIUM NIOBATE CRYSTALS OF A STOICHIOMETRIC COMPOSITION

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The commercially used crystals usually have a congruent composition as the most macroscopically uniform in composition along the growth axis. However, for a series of practical applications LiNbO3 crystals of a stoichiometric composition (Li/Nb=1) have essential advantages over the crystals with a congruent composition. The application of stoichiometric lithium niobate in optic devices calls for creating methods of control of the degree of structural perfection and the crystal composition. When studying Raman spectra we performed a thorough analysis of structural peculiarities of the cation sublattice of real lithium niobate single crystals of varied chemical compositions (both nominally pure and doped). The maximum in the Raman spectrum of the lithium niobate crystal in the region 100-120 cm-1, corresponding to two-particle states of acoustic phonons with the summary wave vector equalling zero was found to be sensible to fine peculiarities of structural ordering of the cation sublatttice. It was established that in the spectrum of a crystal with a stoichiometric composition of a high degree of structural perfection the Raman lines in the region 100-120 cm-1 are absent altogether. The absence of maximum in the Raman spectrum may be assumed as an experimental criterion of the lithium niobate crystal's conformity to stoichiometric composition of a high degree of structural perfection. Any deviation from stoichiometry results in the appearance of this maximum in the spectrum.

M/P.09 EFFECT OF SB+ DOPING ON THE FORMATION OF CRSI2 SILICIDE: USE OF IN SITU X-RAY DIFFRACTION

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Chromium disilicide (CrSi2) is a semiconductor material with suitable properties for optoelectronic applications. The formation mechanisms of this compound depend on several parameters: annealing treatment, impurities, etc.

The aim of the present work is to study the effect of antimony doping on the silicidation. For this purpose, prior to the Cr deposition, Si(111) was implanted with Sb+ ions at 120 keV to 5e14 or 5e15 Sb+cm-2 . The annealing (30 min. at 900°C) of the implanted targets and the subsequent Cr deposition were performed in situ in ultra high vacuum. Thereafter, the obtained Cr/Si samples were heat treated under vacuum in a furnace attached to an X-ray diffractometer. The analysis by in situ X-ray diffraction (XRD) was carried out as the ramped annealing was performed. This technique is beneficial to provide information on the growth mechanisms using only one sample. The final thickness of the silicide, was obtained by Rutherford backscattering spectrometry. For all samples, the CrSi2 compound was the only formed silicide. The reaction started at 425°C for the virgin, and for the 5e14 Sb+cm-2 implanted specimens. However, for the high dose implants (i.e. 5e15 Sb+cm-2), the reaction was detected at 450°C. The formation of CrSi2 , progressed up to approximately 500°C, at which all Cr was converted into chromium disilicide. This behavior was noticed only for the virgin specimens and for the low dose implants (i.e. 5e14 Sb+cm-2). However, in case of the high dose, some Cr remained even at 500°C. The conclusion of this work is that the presence of antimony ions in Si(111) substrates, delayed the formation of CrSi2 silicide but did not inhibit it.

M/P.10 APPLICATION OF MODULATION SPECTROSCOPY FOR DETERMINATION OF RECOMBINATION CENTER PARAMETERS

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Optoelectronic properties of semiconductor materials and devices are defined in general by complex of intrinsic and impurity point defects, which could be rather complicated in binary and ternary compounds. It complicates the analysis and interpretation of the spectral data and definition of generation-recombination mechanisms involved. These difficulties could be avoided using modulation spectroscopy. In the given paper the authors considered the possibility to use l-modulation to define the structure and parameters of luminescence centers in wide-band A3B5 and A2B6 compounds as a promising semiconductors for optoelectronic applications. The spectra of the materials investigated show the characteristic stripes, which is formed by superposition of several recombination mechanisms, such as interband, exciton recombination and transitions through single shallow centers and donor-acceptor pairs for the case of the edge irradiation. Luminescence in low-energy of the spectrum is determined by deep levels. We developed special differential measurement methods using main and doubled modulation frequency, which are able to separate the recombination mechanisms and to determine the transition type, structure and energy parameters of the recombination centers as well. Our methodology was tested on well studied semiconductors and showed good correlation with reference data.

M/P.11 ANISOTROPIC SIZE BOADENING ANALYSIS OF TEXTURED NANOCRYSTALLINE SILICON THIN FILMS PROBED BY X6RAY DIFFRACTION

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Nanocrystalline silicon thin films are challenging materials for large scale microelectronics prepared at low temperature processing. However their complete microstructural and structural characterisation is a hard task, mainly because of their thin structure and the simultaneous appearance of texture, cell parameters and anisotropic crystal shapes. We used in this work a newly developed X-ray technique which is able to combine quantitatively the texture, structure and anisotropic shape determination. Silicon films were grown by reactive magnetron sputtering in a plasma mixture of H_2 and Ar around 200°C, for controlled target to substrate distances, on amorphous SiO_2 and single-crystal (100)-Si substrates. Films were measured using four-circle diffractometry and a curved position sensitive detector.

Whatever the substrate used, preferred orientations are observed with texture strengths around 2 to 3 times a random distribution, with a tendency to achieve lower strengths for films grown on SiO_2 substrates. A strong anisotropic broadening of the diffracted lines is observed due to anisotropic shapes of individual crystallites. Anisotropic shapes and textures are strongly correlated with, as a global trend, longest crystallite sizes along the <hkl> direction of the texture. Cell parameters change both with the stabilised orientation and the crystallite size, and are always refined larger than the value for bulk silicon, by around 0.005 to 0.015 Angstroms.

M/P.12 BAND STRUCTURE AND OPTICAL PROPERTIES OF TlGaSe2

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The band structure of the monoclinic TlGaSe2 crystals has been calculated by the pseudopotential method. The nonlocal pseudopotentials calculated by procedure offered G.B. Bachelet, D.R.Hamann and M.Schluter (Phys. Rev. B, v.26, p.4199 (1981)) were used. The band structure of TlGaSe2 was calculated taking into account 2700 plane waves. Results of our calculations shows, that maximum valence band and the bottom of the conduction band are located at the center of the Brillouin zone. Therefore this crystal belongs to the direct transition semiconducting crystals, with the band gap of 2.5 eV. This result larger than that from local pseudopotential calculations 2.1 eV (Abdullaeva S.G., et al., Phys. Status Solidi B 1986,133,171) and experimental results (Abdullaeva S.G., et al., Phys. Status Solidi B 1983,103, K61) 2.25 eV. Also the optical functions are calculated and compared with experimental data received from reflectance modulation spectra.

M/P.13 X-RAY AND ELLIPSOMETRIC STUDY OF MISFIT STRAIN ANISOTROPY IN PARTIALLY RELAXED III-V EPITAXIAL LAYERS

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We have investigated two types of III-V heterostructures, GaAs/InGaAs and InP/InAlAs/InGaAs, with a small lattice-mismatch that contained a net of well-resolved misfit dislocations lying along two orthogonal <110> directions at the (001) interface. Different dislocation densities in both crystallographic directions led to anisotropic relaxation of misfit strain in the epitaxial layers. This, in turn, resulted in distortion of the unit cell, which we have revealed using high-resolution X-ray diffractometry. On the other hand, the distortion of the zincblende lattice and the unresolved strain in the layers caused energy shifts of semiconductor optical gaps and biaxial changing in their dielectric functions, which were studied by means of generalized and anisotropic ellipsometry. X-ray diffraction analysis was used to verify the ellipsometric results. Our findings are interpreted in terms of different glide velocities of two types of misfit dislocations lying along two different <110> directions, which differ in their core structures.

M/P.14 RESEARCHES CONCERNING THE EXTENSION OF Ni SOLUBILITY IN Al BY MELT QUENCHING

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Melt-spinning and melt-extraction methods of rapid solidification have been applied on Al-Ni alloys in the low composition. The main structural effect obtained was an extension of the solid state solubility of Ni in Al up to 4 wt% Ni. The degree of extension may be estimated by taking into account the equilibrium solubility of Ni in Al that is extremely low, namely 0.006 wt% Ni at 5000C. Such an effect is of high interest, both scientifical and practical, because it opens the possibility to enlarge the number of Al alloys by including the transition metals as alloying elements. In normal solidification these elements are ruled out (Mn being an exception) because of their extremely low solid state solubility. For the transition metals, including Ni, the solid state solubility extension promoted by rapid solidification is the only way to transform them in useful alloying elements, resulting in new high strength Al based alloys, for which very efficient hardening mechanisms are put at work, namely solution hardening and precipitation hardening. The solid state solubility extension of Ni in Al has been ascertained in our experiments by electron microscopy and X-ray diffraction. The SEM images for the slowly solidified alloys have been compared to those obtained by rapid solidification. The structural transformation induced by rapid solidification was confirmed by X-ray diffraction, The melt spun fibbers of the richest alloy (Al + 4wt% Ni) have shown only the intensity peaks of the face centred cubic phase (the supersaturated ? solid solution of Ni in Al), while additional intensity peaks indicating the presence of a secondary phase (Al3Ni intermetallic compound) were seen in the diffraction pattern of the slowly solidified alloy.

M/P.15 OPTOELECTRONIC SENSING SYSTEM FOR SMART POLYMER PROCESSING

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In the last decade, thermoset and thermoplastic polymers have been widely used in many industrial areas including structural and coating applications. As metter of fact, this calss of materials offers unique advantages over conventional materials, however their final properties are strongly dependent on processing stage expecially with regards to thermoset polymers. In order to improve quality and reliability of this kind of materials, an integrated optoelectronic sensing system have been designed and successfully tested. Basic idea involves the use of optical fibre technology in light of their unique advantages over conventional characterization techniques (DSC, ultrasonics, dielectric and spectroscopic). In particular, the distal end of a standard optical fibre has been used to retrieve infoemation about refractive index strongly related to polymer density. In addition, the integration with fibre Bragg gratings ensures multifunction capability including temperature and residiual strain measurements. A properly designed optoelectronic unit hase been designed to perform in situ and on line simultaneous measurements. In particular, for the first time, curing, phase transitions such as gelification and glass transition, and curing induced residual strain have been characterized by proper data analysis. Experimental results demonstrate sensing system capability to perform on line measurements providing the full state vector for the implementation of real Smart Polymer Processing.

M/P.16 GIANT LO OSCILLATION IN THE MULTI-PHONONS Zn1-xBex(Se,Te) PERCOLATIVE ALLOYS

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We finalize a percolation-based picture1 for the basic understanding of the atypical two-modes behavior observed by Raman scattering in the Be-VI optical range of Zn1-xBex(Se,Te) alloys. These open the class of mixed crystals with contrasted bond stiffness. In the transverse symmetry, a double-branch picture based on the MREI model substitutes for the single-branch standard. The low- and high-frequency branches refer to Be-VI vibrations in the Be-rich hard-like (H) and Zn-rich soft-like (S) regions, respectively. These are tied up at separate ends of the composition range and overlap in the percolation regime ($0.19<x<0.81$), corresponding to a clear two-mode behavior. Latter frequency-aspect is completed on the strength side by taking the amount of Be atoms in the H region ideally equal to the Be-content in the alloy, as a result of random atomic substitution. The experimental and theoretical TO-lines are in excellent agreement at any x; no adjustable parameter is needed. In contrast a more sophisticated approach based on discrete decomposition of the apparently-single H- and S-modes is needed to achieve full contour modeling of the more complicated LO lines in the percolation regime, where the host matrices have a fractal geometry. Basically intra- (S→S, H→H) and inter- (H→S) component transfer of oscillator strength towards the more ionic ends in the series results in the building up of a quasi-unique giant LO oscillation.

[1] O. Pagès et Al. Phys. Rev. B 65, 35213 (2001).

M/P.17 OPTICAL CHARACTERISATION OF GERMANIUM THIN FILM GROWN BY LOW ENERGY PLASMA ENHANCED CHEMICAL VAPOUR DEPOSITION FOR GAAS/GE SOLAR CELL APPLICATIONS

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We report the optical quality of germanium (100%) thin films (1µm) grown using Low Energy Plasma Enhanced Chemical Vapour Deposition (LEPECVD) on a silicon substrate with a graded SiGe intermediate layer. With the high cost of optoelectronic material compared to silicon and also due to size limitation of the available III-V optical graded substrates, there is much interest in growing high optical quality epitaxial layers on large size silicon substrates. Such a Si/SiGe/Ge substrate would form a virtual substrate for GaAs solar cells and also allows one to combine optoelectronics with microelectronics. We have investigated such substrates using UV-Visible spectroscopic ellipsometry (SE) and modulated photoreflectance (PR) techniques.

The optical interband transitions observed from analysis of the complex dielectric function of Ge thin films (obtained from SE) and from PR spectra are in good agreement with (100) 6° off towards [111] Germanium substrates used for GaAs/Ge solar cells.

M/P.18 THE DIELECTRIC FUNCTION OF A GaAs/AlGaAs SINGLE QUANTUM WELL: CALCULATION AND COMPARISON WITH MODULATION SPECTROSCOPY AND PHOTOLUMINESCENCE EXCITATION DATA

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The dielectric function (DF) of modulation doped (001) GaAs/AlGaAs (Al content of x=0.33) single quantum well (SQW) structures containing a two-dimensional electron gas (2DEG) was calculated as a function of the dc bias voltage applied to the Schottky gate. We solved the problem in three steps. First a self-consistent calculation of the conduction band potential relief, as well as the wavefunctions and the energy spectrum of electrons in the conduction band was carried out by solving the Poisson and Schrödinger equations. The Schottky barrier height, the doping profile, the conduction band discontinuity, and the bias voltage were treated as fixed parameters. Then the valence band structure and corresponding wavefunctions were deduced by the kp method using a 4x4 Hamilton operator in the axial approximation. Finally the DF was obtained which was used to simulate electroreflectance, photoreflectance and photoluminescence excitation spectra. At this stage the band gap renormalization has to be taken into account. The results were found to be in good agreement with low-temperature (5 to 80 K) experimental data for SQW structures in a whole range of applied bias voltages corresponding to the 2DEG density varying from nearly zero up to 5x10^11 cm^-2.

M/P.19 HETEROJUNCTION BIPOLAR TRANSISTOR (HBT) CHARACTERISATION USING NON-CONTACT OPTICAL SPECTROSCOPY

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We report the application of optical spectroscopic techniques including photoreflectance (PR), ellipsometry and photoluminescence for qualification of InGaP/GaAs multi-layer HBT material. These techniques reveal important information regarding the quality of the different InGaP and GaAs layers for the emitter, base, collector and surface cap regions.

In particular this paper summarizes a full lineshape reproduction of the HBT GaAs PR response, including simulation effects of emitter/base layer intermixing upon the PR Seraphin coefficients. Both emitter/base and base/collector interfaces were also examined with respect to the complex PR phase response as well as laser pump excitation conditions, with results also compared with electrical current gain data. This paper demonstrates the application of non-destructive and rapid techniques for evaluation and control of compound semiconductor materials for HBT technology.

M/P.20 NONLINEAR-OPTICAL PROBING OF NANOSECOND FERROELECTRIC SWITCHING

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Switching properties of thin ferroelectric films are intensively studied regarding to their main applications, random access memories and electro-optical modulators and as microwave electronic components. These devices should operate at frequency range 1-100 GHz and therefore the dynamic response of the film parameters should be studied on a time scale from nanoseconds down to picoseconds

In this work we study the switching process in ferroelectric thin films with temporal resolution of 5 ns by optical second harmonic generation (SHG). In 70-nm BaSrTiO3 (BST) film two types of switching/relaxation processes are observed. When the normal (positive or negative) voltage pulse is applied for the film, the SHG transient is quite similar to voltage transient. Such temporal behavior of the SHG signal can be described in the frame of Landau theory. If the voltage is kept constant and then drops to zero following by a back increase (“zero” pulse), oscillations of the SHG signal are observed, extended far beyond the applied voltage pulse. At least two mechanisms might be responsible for the increase of the SHG oscillations: (i) direct oscillating dielectric post-polarization of the film and (ii) oscillating piezoresponse. Temporal behavior of the polarization SHG diagrams shows that on the different stages of the switching/relaxation process different domain variants are involved. Our studies show that thin BST films can be used for GHz-range devices and SHG technique gives efficient tool for study the processes of fast polarization switching.

M/P.21 CHARACTERIZATION OF ELASTIC STRAINS, Cd COMPOSITION AND LAYERS THICKNESS IN ZnSe/Zn1-XCdXSe QUANTUM WELLS STRUCTURES BY X-RAY DIFFRACTION MEASUREMETS

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The system ZnSe/Zn1-XCdXSe is of special interest because of the possible application for opto-electronic devices operating in the blue-green spectral range. The structural perfection of such materials with high lattice mismatch very fast deteriorates with increasing of Cd concentration that leads to increasing of local deformation and cadmium diffusion and/or segregation.

The influence of different growth parameters on structural parameters of ZnCdSe QWs and values of deformations of QWs and ZnSe barriers have been investigated by high-resolution X-ray diffraction (HRXRD) measurements. The investigated Zn1-xCdxSe/ZnSe multi-quantum wells structures with high molar fraction of cadmium (30-50 %) and wide ZnSe barriers (50, 100 and 500 nm) were grown by molecular beam epitaxy (MBE) on semi-insulating (001) GaAs substrates. The structures were grown on 1000 nm thick ZnSe buffer layer and capped by 30 nm ZnSe layer. The presence of superlattice was confirmed by the appearance of corresponding satellites in (004) diffraction profiles. The evaluation of QW thickness and Cd composition was made from the experimental diffraction rocking curve. The measured w/2q-scans were evaluated using simulations based on semi-dynamical diffraction theory. It was found the fluctuations of cadmium up to 3-5 % within the QW layer. The Zn1-xCdxSe QWs have compressive deformation for any x and ZnSe layers have tensile deformation.

M/P.22 PHOTOELECTRICAL MEASUREMENTS OF THE LOCAL VALUE OF THE CONTACT POTENTIAL DIFFERENCE IN THE METAL INSULATOR SEMICONDUCTOR MIS STRUCTURES

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Metal Insulator Semiconductor (MIS) structures are the basic building blocks of modern integrated circuits. Some of the MIS structure parameters are most accurately determined by photoelectric methods. One of such parameters being the contact potential difference (CPD) which determines MIS transistor's threshold voltage. In this work the local values of CPD and their distributions in the plane of the MIS structure's gate have been determined for the first time. This has been achieved by application of a focused beam of UV radiation from a laser source. The less than 10mm diameter of UV radiation beam allows determination of distributions of local CPD values in the plane of the gate. The CPD distributions obtained have been confirmed by a series of independent measurements by other methods. It is believed that the CPD distributions obtained (as well as distributions of local values of other parameters) are due to the mechanical stress distributions under the gate of a MIS system. In this paper the method and the optical setup used to determine local values of CPD are described.

M/P.23 A METHODOLOGY TO REDUCE ERROR SOURCES IN THE DETERMINATION OF THIN FILM CHEMICAL COMPOSITION BY EDAX

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EDAX is commonly used to determine the chemical composition of thin films. The correct determination of the stoichiometric ratio of binary compound thin films is of great importance because it is related to the density of intrinsic lattice defects (vacancies). In our group we have been using this technique to know the stoichiometric ratio in FeS2 from several years ago (1). We have identified three main error sources which modify the obtained results and can yield mistaken conclusions. These three error sources are the following: a) environmental oxidation of the thin film surface, b) carbon thin film used to protect against environmental corrosion the reference sample, and c) the EDAX signals from the substrate. In general, the contribution of these error sources decreases as the film thickness increases and it is strongly dependent on the incident electron beam energy.

Here, we present the methodology to investigate the stoichiometry of thin films by EDAX reducing the error sources. We have used FeS2 thin films deposited on glass by sulfuration of previously evaporated iron layers. FeS2 films with thicknesses from 0.1 to 1.0 mm have been investigated. Different incident beam energies from 3 to 30 keV have been used. Finally, we establish a measurement protocol, which allows selecting the adequate incident energy to each film thickness (2). In this manner, we have determined the stoichiometric ratio of FeS2 thin films with different thickness. The stoichiometric values obtained are in well agreement with those reported from single crystals.

[1] I.J. Ferrer, C. Sánchez. J. Appl. Phys 70, 2641-2647 (1991).

[2] J.R. Ares, Formation mechanism and conductivity type in pyrite thin films. Ph. D. Thesis, UAM, Spain (2002), p. 107-128.

M/P.24 ROTATIONAL EFFECTS ON PARTICLE SIZE DETERMINATION AND REFLECTIVITY DEPENDENCE ON HAZE MEASUREMENTS IN OPTICAL SCATTEROMETRY

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Optical scatterometry measurements such as those performed by the KLA-Tencor Surfscan are widely used for determining particulate contamination, particle size and 'haze' in virtually every branch of semiconductor wafer production. Particle sizing is determined by Rayleigh's law of light scattering and assumes particles to be perfectly spherical. However, many surface defects present on semiconductor wafers are not spherical, but have a large aspect ratio. Thus conventional scatterometry measurements are dependent on the relative angle between the scanning beam and the major axis of the particle. It has also been observed that haze is not always related to the surface roughness of a wafer as measured by AFM, contrary to popular belief.

We present results demonstrating these effects on apparent particle sizing and haze measurements.

M/P.25 OPTICAL PROPERTIES OF LPCVD Si/SiO2 STACKED LAYERS DEPOSITED ON SILICON

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The stacked films of alternated LPCVD poly-Si and SiO2 layers deposited on crystalline Si has been investigated by means of optical spectroscopy and electrical methods as voltage dependence of the structure capacitance. The refractive index considered, as a complex number and the width of the films are the main parameters, which are extracted by using a fitting procedure of experimental data. The approach is based on the simulation of the spectra in the range of 250-900 nm. The calculation is related to light propagation theory in non-homogeneous media taking into account the optical transition in silicon. A matrix multiplication is used for the computation of both the transparency and absorbency of each stacked layer, which is considered, made by many slices of width smaller than 20nm. The IR spectroscopy has been used as an optical complementary technique to investigate the profile of stretching Si-O-Si band of the stacked layers. The shift of the IR vibration mode related to the chemical environment of Si-O bonds has been revealed by quantum calculations. The study has been analysed the specific contribution of the oxide / poly-Si interfaces to IR absorption spectra of the multiple SiO2/Si film sample. The C(V) characteristics were related to the spectroscopic data.

M/P.26 ENERGY BANDS AND EXCITON STATES IN MIXED Zn(P1-x Asx)2 AND CdxZn1-xP2 CRYSTALS

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In present work we have studied the semiconductor mixed Zn(P1-xAsx)2 crystals in full range of x: 0<=x<=1, and crystals CdxZn1-xP2 at . Low-temperature (1.8 K) absorption, reflection and photoluminescence spectra of excitons in these mixed crystals have been studied. Likely to "pure" ZnP2 crystal, in mixed crystals the same excitonic series (C, B, and A) are observed. With increase of x in Zn(P1-x Asx)2crystals the energy gap decreases. The dependence is slightly sublinear. At the increase of x, there is also decrease of the excitonic series rydbergs. The dependences of excitonic rydbergs on x are rather remarkable. These dependences are strongly superlinear at x close to 0, and most linear at x close to 1. And stronger superlinearity takes place at . Let us note that the rydbergs of B- and A-series decrease considerably: at the crossing from ZnP2 to ZnAs2 the rydbergs decrease in more than 3 times. Meanwhile, the rydberg of A-series decreases sufficiently less: it decreases in 1.4 times. At the increase of x from 0 to 0.05 the decrease of the energy gap takes place both in Zn(P1-x Asx)2 and CdxZn1-xP2 crystals. At the increase of x from 0 to 0.05 the rydbergs of series decrease in crystals of both types as well. The dependences of Eg and Ry on x are considerably stronger in Zn(P1-x Asx)2 than in CdxZn1-xP2 crystals.

M/P.27 COMPARATIVE STUDY OF PbTiO3 BULK CERAMICS AND FILMS WITH HIGH Ca DOPING

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Substitution of Pb by large percentages of Ca (close to 50%) in the PbTiO3 perovskite introduces a shrinkage in the lattice along the c-axis direction of the tetragonal phase. This is due to the replacement of Pb by an ion as Ca with its same valence, but smaller size. This produces a change in the structure, from tetragonal to orthorhombic when Ca doping increases from 0% to 50%. PbTiO3 bulk ceramics with high contents of Ca show a reduction of the transition temperature as well as an increase of the dielectric constant at room temperature. If materials with these properties would be fabricated as thin films, they could be of interest in Dynamic Random Access Memories (DRAMs). But also, their high permittivity values joined to the non-linearity of the permittivity with the voltage, would make them promising for their use in high frequency components.

In this work, Ca-modified PbTiO3 films with Ca~50% have been prepared by chemical solution deposition (CSD) onto Pt/(100)MgO substrates. Structure and texture of the films have been determined by x-ray diffraction (XRD), observing a preferred growth of the Ca-PbTiO3 films when compared with the random structure of bulk ceramics. Comparison of the x-ray patterns of both materials, ceramics and films, has also permitted to make an approximation to the stresses supported by the films on the substrate. Data obtained from XRD are related with the dielectric properties of the films and these are compared with those measured in bulk ceramics.

Session IX: Optical metrology: devices
Session chair: Bernard Servet and Pat Kelly

M-IX.1 9:00 -Invited-

APPLICATION OF OPTICAL METROLOGY TO THE PRODUCTION OF INGAP/GAAS HBT EPITAXIAL WAFERS

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Optical metrology is critically important to the manufacture of compound semiconductor epitaxial wafers. An optical based characterisation technique provides fast and non-destructive analysis, both during the epitaxy phase (in-situ) and after growth is completed (ex-situ). The most commonly used class of tool is based on laser light scattering and is used in the detection and quantification of defects/particles on the wafer surface (e.g. Tencor Surfscan). A second class of tool uses optical interference/reflectance to analyse 'transparent' multilayer structures (e.g. ellipsometry or IR reflectometry). A third class of tool is based on the interaction of light with the various epitaxial layers, either separately or in combination, through the generation of luminescence (photoluminescence) or changes in reflectivity (photoreflectance). The applicability and usefulness of these techniques depends on the details of the layer structure being investigated and whether the analysis is performed in-situ or ex-situ.

We will discuss the application of ex-situ optical metrology to the monitoring and control of the InGaP/GaAs HBT production process. We will demonstrate the usefulness of laser light scattering, photoreflectance and IR reflectometry for routine production, and the use of spectroscopic ellipsometry and (potentially) photoluminescence for calibration.

M-IX.2 9:40

PHOTOREFLECTANCE SPECTROSCOPIC STUDIES OF VERTICAL CAVITY SURFACE EMITTING LASER DEVICES

M.E. Murtagh, S. Ward, D. Nee, V. Guenebaut and P.V. Kelly, OMI Ltd., Cork Airport Business Park 2200, Cork, Ireland, B. O'Looney, F. Murphy and M. Modreanu, NMRC, Lee Maltings, Prospect Row, Cork, Ireland, S. Westwater and S.W. Bland, IQE, Cypress Drive, St. Mellons, Cardiff CF3 OEG, U.K.

This paper summarises the application of the laser based electro-absorptive technique of photoreflectance (PR) for the study of vertical cavity surface emitting lasers (VCSEL). PR results are shown to reveal the technologically important cavity resonance mode and ground state quantum well exciton structures.

In this study GaAs/AlGaAs based quantum well VCSELs were examined with and without quantum well active layers as well as without top distributed Bragg stack mirror. Structures were examined with respect to the complex PR phase response as well as laser pump excitation conditions, with results compared with angle dependent PR data. Cavity mode and quantum well alignments were also studied with reference to the un-modulated reflectance signal as well as correlated with photoluminescence data. The results demonstrate the importance of PR metrology for state-of-art VCSEL characterisation.

M-IX.3 10:00

PHOTOREFLECTANCE SPECTROSCOPY FOR THE DESIGN OF HETEROJUNCTION BIPOLAR TRANSISTORS

C. Bru-Chevallier, H. Chouaib, T. Benyattou, Laboratoire de Physique de la Matière UMR CNRS 5511, INSA de Lyon, Bâtiment Blaise Pascal, 7 avenue Jean Capelle, 69621 Villeurbanne Cedex, France, P. Bove Picogiga, Les Ulis, France

Use of GaAsSb material in the base of InP heterojunction bipolar transistors (HBT) has recently proved to allow favourable conduction- and valence-band discontinuities with InP and therefore potentially leads to high transition frequency HBTs; moreover C-doped GaAsSb features high doping efficiencies. However, lot of antimonide material parameters remain to be measured. In the paper we present photoreflectance spectroscopy of low doped GaAsSb layers on n+ doped InP, which exhibit many Franz Keldysh oscillations. From this work, we show that the Fermi level at the alloy surface lies near the valence band. Relation between the Fermi level pinning and the base resistance of the final device will be adressed.

A second part of the paper will be devoted to the application of PR spectroscopy to the characterization of complex epitaxial structures for HBT. Electric field at the EB and/or at the BC junctions will be derived and tentatively related to device electrical performance such as static I(V) characteristics. Finally, micro-PR characterization on a 5µm diameter spot will also be presented and analysed : this will open the door for micro-PR characterization of operating devices.

M-IX.4 10:20

PHOTOREFLECTANCE STUDIES OF Ga- AND N-FACE AlGaN/GaN HETEROSTRUCTURES CONFINING A POLARIZATION INDUCED 2DEG

A.T. Winzer, R. Goldhahn, C. Buchheim, G. Gobsch, O. Ambacher, Institute of Physics and Center for Micro- and Nanotechnologies, TU Ilmenau, PF 100565, 98684 Ilmenau, Germany, A. Link, M. Eickhoff, M. Stutzmann, Walter Schottky Institute, TU Munich, Am Coulombwall 3, 85748 Garching, Germany

Recently, AlGaN/GaN-based polarisation induced high electron mobility transistors (HEMTs) have become a subject of intense investigations because they are attractive candidates for high voltage, high power operation at microwave frequencies as well as for ion sensitive detectors. For all of these applications detailed knowledge of layer and surface properties is of fundamental importance. Photoreflectance spectroscopy (PR) represents a unique method allowing the contactless determination of electric field strengths in HEMTs with highest accuracy. We report on systematic PR studies of both N- and Ga-face heterostructures grown by plasma-induced MBE. For N-face samples, the main information is extracted by analysing the Franz-Keldysh-Oscillations (FKO) arising from the topmost GaN channel layer. Their energetic splitting indicates a reduced electric field strength at lower temperatures. For the Ga-face samples, the AlGaN barrier field strength depends strongly on the alloy composition, the layer thickness, and temperature. For both types of samples a surprisingly strong temperature dependence of the electric field strength is observed which can be attributed to the change in piezoelectric polarization, while spontaneous polarization remains nearly constant, as will be shown by calculations. Additionally, a comparison of experimental data with self-consistent potential calculations allows the determination of the surface barrier height.

10:40 **BREAK**

Session X: Optical metrology: materials and devices

Session chair: Steve Bland and Martin Murtagh

M-X.1 11:00

CHARACTERIZATION OF InP AND GaAs FILMS BY CONTACTLESS TRANSIENT PHOTOCONDUCTIVITY MEASUREMENTS

M. Kunst (a), H.-C. Neitzert(b), A. Sanders(a) and F. Wünsch(a), (a)Hahn Meitner Institut, Bereich Solare Energetik, 14091 Berlin, Germany, (b)Universita degli studi di Salernob Dipartimento di ingegneria dell´informazione ed ingegneria elletttrica, 84084 Fisciano, Italy

The application of III-V semiconductor films lies mainly in (opto)electronic devices. This makes the monitoring of the photoconductivity an outstanding tool for the characterization of these films. In this contribution InP and GaAs films are studied by contactless transient photoconductivity measurements in the microwave frequency range (TRMC measurements) in particular in view of in-line and in situ characterization by TRMC measurements.

n-doped and p-doped (1017cm-3) InP films were deposited by chemical beam epitaxy on semi-isolating InP substrates. n-doped GaAs films were deposited by molecular beam epitaxy on semi-isolating GaAs substrates. It is shown that the TRMC signals of the InP films are mainly due to the majority carriers because of very fast minority carrier trapping. The shape of the TRMC signal depends strongly on the surface as it is shown by the influence of surface treatments as reactive ion etching and annealing treatments.Information on the surface space charge region can be extracted from the measurements. This is still more clear for the GaAs films where the storage of excess charge carriers in the surface space charge region is unambiguously observed. The GaAs films can be characterized by the amplitude of the TRMC signal that is shown to be proportional to the electron mobility. The experimental data suggests that this is also possible for the InP films but more experimental information is required. The in situ characterization of InP and GaAs films by TRMC measurements is discussed taking into accout the experience gained by in-situ TRMC measurements during the deposition of a-Si:H.

M-X.2 11:20

TEMPERATURE-DEPENDENT ELECTRIC FIELDS IN GaN AND AlGaN SCHOTTKY DIODES STUDIED BY ELECTROREFLECTANCE

S. Shokhovets(a), D. Fuhrmann(a), R. Goldhahn(a), G. Gobsch(a), O. Ambacher(b), Institute of Physics(a) and Center for Micro- and Nanotechnologies(b), Ilmenau Technical University, 98684 Ilmenau, Germany, M. Hermann and M. Eickhoff, Walter Schottky Institute, Technical University Munich, 85748 Garching, Germany

Electroreflectance (ER) of GaN and AlGaN (Al content of x=0.2) Schottky diodes with a semitransparent Pt gate was investigated as a function of the dc bias voltage in a temperature range between 5 and 300 K. The diodes were fabricated on Si-doped layers with Ga- and N-face polarity grown on (0001) sapphire substrates by MBE. We observed a peculiar influence of the bias voltage on the ER spectra, the so-called “rotation”. For the excitonic spectral region, this behaviour is due to quenching of discrete exciton states and the formation of an exciton dead layer underneath the gate while the linear electro-optic effect dominates below the band gap. The data analysis was carried out using electric field-dependent dielectric functions of epitaxial GaN and AlGaN layers. Field inhomogeneities were taken into account by a multi-layer formalism. A comparison with experimental data yielded the electric field strength, the exciton energies and the broadening parameter for the zero-field limit, as well as other parameters of the depletion region, such as the surface band bending and the ionised impurity concentration. We found significant differences in the values of these quantities and their temperature dependences for samples with different polarity. The results are discussed in terms of temperature- and polarity-dependent concentrations of ionised impurities and surface charges and polarity-dependent microstructure of the investigated samples.

M-X.3 11:40

THEORETICAL CONCEPT OF STRAIN EFFECT ON REFLECTANCE ANISOTROPY AND PHOTOREFLECTANCE SPECTRA OF SEMICONDUCTOR MULTILAYER SYSTEM

L. Long, J. Schreiber, Fachbereich Physik, Martin-Luther-Universität Halle-Wittenberg, Friedemann-Bach-Platz 6, 06108 Halle, Germany

Employing Jone's matrix method the optical properties of semicomductor pseudomorphic multilayer system (AlGaAs/GaAs/InGaAs multilayers) are investigated. The theoretical calculation for VCSEL structure agrees with experimental results. In addition, the effect of strain induced anisotropy of reflectance spectrum (RAS) in these systems is analysed. Furthermore, polarisation dependent PR spectrum resulting from strain related electron state variation is reviewed. Potential applications of both methods to diagnosis of optoelectronic materials and devices are discussed.

M-X.4 12:00

OPTICAL PROPERTIES OF SILICON THIN FILMS RELATED TO LPCVD GROWTH CONDITIONS

M. Modreanu, B. O'Looney, F. Murphy, NMRC, Lee Maltings, Prospect Row, Cork, Ireland, M.E. Murtagh and P.V. Kelly, OMI Ltd., Cork Airport Business Park, Kinsale Rd, Cork, Ireland, M. Gartner, Institute of Physical Chemistry, Spl. Independentei 202, Bucharest 77208, Romania, M. Bercu, Faculty of Physics, University of Bucharest, PO Box 11 Mg, 5600, Bucharest, Romania, C. Cobianu, Valahia" Univ. from Târgoviste, Electrical Engineering Faculty, ", P.O. Box 27-17, 77550 Bucharest, Romania

The properties of silicon thin films have been investigated with respect to the deposition parameters (pressure and temperature) of the low pressure chemical vapour deposition (LPCVD) technique. We have investigated optical properties of the LPCVD thin films using UV-Visible spectroscopic ellipsometry (SE), UV reflectance (R) spectrometry as well as modulated photoreflectance (PR) techniques. The microstructure of the silicon films has been correlated with optical spectroscopy data. Investigation on PR and R spectra in the range of 250-400 nm indicates a definite dependence on temperature and growth rate for LPCVD poly-Si films. Information about the phase transition of as-deposited LPCVD silicon from amorphous to crystalline state via a mixed phase (grains embedded within amorphous Si matrix) obtained from SE analysis. Local disorder in LPCVD silicon was also measured from the behaviour of both 370nm and 280 nm peaks in the PR and R spectra and correlated with the degree of crystallisation obtained from SE. Highest disorder in LPCVD poly-Si samples was recorded close to 550oC. From SE, R and PR measurements the E0', E1 and E2 critical-point energies in LPCVD silicon films were determined.

M-X.5 12:20

INFRARED SPECTROSCOPIC ELLIPSOMETRY APPLIED TO THE CHARACTERIZATION OF NANO-STRUCTURES OF SILICON IC MANUFACTURING

P. Boher, M. Bucchia, C. Guillotin, C. Defranoux, SOPRA, 26 rue Pierre-Joigneaux, 92270 Bois-Colombes, France

Different steps of the microelectronic fabrication process involve patterning of a numerous sub micron features in a repetitive way at different locations on the silicon wafer. Precise characterization of this kind of feature becomes difficult due to their very small size and destructive ways like scanning electronic microscope are often the unique solution to optimize the process parameters. In the proposed paper, we will show that infrared spectroscopic ellipsometry offers a non destructive way to obtain valuable information in this kind of situation. Indeed, the IR wavelength is generally much larger than the feature size. So, diffraction effects can be neglected and effective medium approximation models can be applied. In addition most of the materials become transparent in the infrared region so that deep trenches can be addressed.

A new metrology tool based on an infrared spectroscopic ellipsometer and dedicated to 300mm silicon metrology will be used. The measurement spot is smaller enough to be focused on the front face of the wafer so that the back face reflected beam can be optically eliminated. The measurement speed is about 30s per point for a throughput higher than 15 wafer/hour. The measurement head is included in an automatic system including 300mm wafer handling and orientation, XYZ table, pattern recognition and auto focusing. The proposed paper will present experimental results on contact trenches in TEOS layers and deep trench capacitors for DRAM technology.

12:40 **LUNCH**

Friday, June 13, 2003 — Afternoon
Vendredi 13 juin 2003 — Après-midi

14:00-16:00 **ROUND TABLE**
ADVANCES IN OPTICAL AND X-RAY METROLOGY
Pat Kelly, Jürgen Schreiber, Daniel Chateigner