1st International Workshop on

Nano-scale Spectroscopy and its Applications to Semiconductor Research

December 11 - 14, 2000
Trieste, Italy
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Abstract Book

1st International Workshop on
Nano-scale Spectroscopy and its Applications to Semiconductor Research

Program

Sunday, 10 December 2000

18:00 - 20:00	Welcome reception at the ICTP

Monday, 11 December 2000

8:45 - 9:00	Opening remarks by the workshop chairman G. Salviati
Session Synchrotron radiation techniques I, chair: Y. Watanabe	
9:00 - 9:45	T. Kinoshita, University of Tokyo: WHAT CAN WE DO BY PHOTOELECTRON SPECTROMOCROSCOPY?
9:45 - 10:10	S. Di Fonzo, Sincrotrone Trieste: NON-DESTRUCTIVE DETERMINATION OF LOCAL STRAIN UNDER BURIED INTERFACES WITH 100-NANOMETER SPATIAL RESOLUTION
10:10 - 10:35	K. C. Prince, Sincrotrone Trieste: Surface chemistry of lead sulphide
10:35 - 11:30	coffee break
Session Synchrotron radiation techniques II, chair: N. Ueno	
11:30 - 11:55	M. Kiskinova, Sincrotrone Trieste: Scanning Photoemission Microscopy: recent developments and applications
11:55 - 12:20	R. J. Phaneuf, University of Maryland: Spatially Resolved Spectra and Energetically Resolved Photoelectron Images

	of Reactive Surface Phases and PN-Junctions
12:20 - 12:45	R. Larciprete, Sincrotrone Trieste: VISIBLE AND UV PULSED LASER PROCESSING OF THE Ti/Si(001) INTERFACE STUDIED BY XPS MICROSCOPY WITH SYNCHROTRON RADIATION
12:45 - 13:10	E. Di Fabrizio, INFM-TASC: Zone Plate for x-ray applications
13:10 - 14:30	lunch break
Session Synchrotron radiation techniques III, chair: M. Kiskinova	
14:30 - 15:15	N. Ueno, Chiba University: Imaging of organic thin films using electron emission microscope
15:15 - 15:40	M. Cinchetti, Johannes Gutenberg Universität Mainz: The Effect of Surface Defects on the Efficiency of GaAsP Photoemitters for Polarised Electron Sources
15:40 - 16:05	M. Mynar, Delong Instruments: X-ray photoemission and low energy electron microscope
16:05 - 16:30	B. Kaulich, ESRF: X-RAY MICROSCOPY AND SPECTROMICROSCOPY TECHNIQUES USING MULTI KEV PHOTONS
16:30 - 17:00	coffee break
Session Synchrotron radiation techniques IV, chair:T. Kinoshita	
17:00 - 17:45	S. Heun, Sincrotrone Trieste: PHOTOELECTRON SPECTROSCOPY FROM INDIVIDUAL HETEROEPITAXIAL NANOCRYSTALS ON GaAs(001)
17:45 - 18:10	M. Bertolo, Sincrotrone Trieste: Cross-sectional photoemission microscopy of semiconductor heterostructures

Tuesday, 12 December 2000

Session Synchrotron radiation techniques V, chair: S. Heun	
9:00 - 9:45	Y. Watanabe, NTT Basic Research Laboratories: Synchrotron radiation photoelectron spectroscopy of nanostructures
9:45 - 10:10	A. Barinov, Sincrotrone Trieste: Formation stages and thermal stability of metal/n-GaN interface
10:10 - 10:35	C. Teichert, Montanuniversität Leoben: Self-organized semiconductor nanostructures and their applications
10:35 - 11:30	coffee break
Session Scanning probe techniques I, chair: S. Ushioda	
11:30 - 12:15	R. Cingolani, Universita' di Lecce: Wavefunction mapping in single quantum dots
12:15 - 12:40	J. Wiebe, Hamburg University: An Ultra High Vacuum-300 mK-Scanning Tunneling Microscope for Local Measurements of the Density of States on InAs(110)
12:40 - 13:05	F. Rosei, Università di Roma Tor Vergata: Growth and characterization of Ge/Si(111) quantum dots by Scanning Tunneling Microscopy
13:05 - 15:00	lunch break
15:00 - 18:00	Excursion to Sincrotrone Trieste

Wednesday, 13 December 2000

Session Scanning probe techniques II, chair: M. Sakurai	
9:00 - 9:45	S. Ushioda, Tohoku University: Probing of electronic transitions with atomic scale spatial resolution
9:45 - 10:10	T. K. Johal, Universita di Lecce:

	IMAGING OF QUANTUM DOT STATES BY SCANNING TUNNELING SPECTROSCOPY
10:10 - 10:35	K. Kanisawa, NTT Basic Research Laboratories: IMAGING OF ZERO-DIMENSIONAL STATES IN SEMICONDUCTOR NANOSTRUCTURES USING SCANNING TUNNELING MICROSCOPY
10:35 - 11:00	B. Ressel, Sincrotrone Trieste: ELECTRONIC STRUCTURE OF THE Pb/Si(111) MOSAIC PHASE
11:00-11:30	coffee break
Session Scanning probe techniques III, chair: M. Colocci	
11:30 - 12:15	M. Sakurai, RIKEN: STM-INDUCED LIGHT EMISSION FROM NANOSTRUCTURES ON A SI(001)-(2X1)-D SURFACE
12:15 - 12:40	L. Ottaviano, INFM Unità dell'Aquila: On the spatially resolved electronic structure of polycristalline WO3 films investigated with Scanning Tunneling Spectroscopy
12:40 - 13:05	J. P. Singh, Nuclear science Centre New Delhi: Electronic excitations induced enhancement in metallicity on HOPG and Si surfaces: in-situ STM/STS studies
13:05 - 14:30	lunch break
Session Scanning probe techniques IV, chair: K. Yagi	
14:30 - 15:15	S.-W. Hla, Freie Universität Berlin: Single Molecule Engineering with a Scanning Tunneling Microscope
15:15 - 15:40	H. Iwasaki, Osaka University: STM Nanolithography on SiO2/Si
15:40 - 16:05	N. Jug, Institute Josef Stefan Ljubljana: Surface structure of some transition-metal trichalchogenides

16:05 - 16:50	A. Armigliato, CNR-Istituto LAMEL Bologna: STRAIN ANALYSIS IN SUBMICRON ELECTRON DEVICES BY CONVERGENT BEAM ELECTRON DIFFRACTION
16:50 - 17:00	coffee break
Session Scanning probe techniques V, chair: S.-W. Hla	
17:00 - 17:45	M. Colocci, INFM Firenze: New trends in the optical investigation of semiconductor heterostructures at the nanoscale
17:45 - 18:10	A. Crottini, Swiss Federal Institute of Technology Lausanne: A novel tip-surface distance control for low temperature scanning near-field photoluminescence spectroscopy
18:10 - 18:35	L. Sirigu, Swiss Federal Institute of Technology-EPFL Lausanne: OPTICAL PROPERTIES OF V-GROOVE QUANTUM WIRE LASERS AND LED
20:00 - 23:00	Social dinner

Thursday, 14 December 2000

Session Electron microscopy techniques I, chair: G. Salviati	
9:00 - 9:45	N. Yamamoto, Tokyo Institute of Technology: TEM-Cathodoluminescence Study of Microstructures and Defects in Semiconductor Epilayers
9:45 - 10:10	M. Ivanda, Rudjer Boskovic Institute Zagreb: COMPARISON OF HIGH RESOLUTION TRANSMISSION ELECTRON MICROSCOPY AND LOW FREQUENCY RAMAN SCATTERING IN DETERMINATION OF PARTICLES SIZE DISTRIBUTION OF NANOSIZED TiO2
10:10 - 10:35	A. Patnaik, Indian Institute of Technology Madras: STRUCTURE AND CHARGE TRANSPORT IN N+ BEAM INDUCED pi-BONDED NANOCRYSTALLINE CLUSTERS IN POLY PHENYLENE OXIDE

10:35 - 11:00	J. Swiatowska-Mrowiecka, University of Mining and Metallurgy: INFLUENCE OF THE CRYSTALLOGRAFIC ORIENTATION ON THE MECHANISM OF ZINC DISSOLUTION IN ORGANIC SOLVENTS
11:00 - 11:30	coffee break
Session Electron microscopy techniques II, chair: N. Yamamoto	
11:30 - 12:15	K. Yagi, Tokyo Institute of Technology: Surface Imaging with use of an Energy Filter
12:15 - 12:40	V.Grillo, Universitaet Erlangen-Nuernberg: COMPOSITIONAL FLUCTUATIONS AND LUMINESCENCE PROPERTIES OF InGaN QUANTUM WELLS GROWN ON GaN AND SAPPHIRE SUBSTRATES: A COMPARISON
12:40 - 13:05	M. Starowicz, University of Mining and Metallurgy, Krakow: ELECTROCHEMICAL DEPOSITION OF SI(IV) COMPOUNDS ON THE METAL SURFACE FROM ANHYDROUS ORGANIC SOLVENTS
13:05 - 14:30	lunch break
Session Electron microscopy techniques III, chair: T. Sekiguchi	
14:30 - 15:15	G. Salviati, MASPEC-CNR Institute Parma: Cathodoluminescence submicrometric depth profiling of optical emissions in semiconducting heterostructures and devices
15:15 - 15:40	O. Martinez, Dpto. Física de la Materia Condensada. E.T.S.I.I. Valladolid: MICROCHARACTERIZATION OF CONFORMAL GaAs AND AlGaAs ON Si LAYERS BY SPATIALLY RESOLVED OPTICAL TECHNIQUES
15:40 - 16:25	T. Sekiguchi, National Research Institute for Metals Tsukuba: Low-energy Cathodoluminescence Study of Semiconductor Nanostructures and Nanoparticles
16:25 - 16:40	Concluding remarks by the workshop vice-chairman N. Yamamoto

Friday, 15 December 2000

8:00 - 22:00	Excursion to CNR-MASPEC in Parma

List of Invited Speakers

A. Armigliato, CNR-LAMEL, Bologna: Strain analysis in submicron electron devices by Convergent Beam Electron Diffraction

R. Cingolani, Universit di Lecce

M. Colocci, Universit di Firenze: New trends in the optical investigation of semiconductor heterostructures at the nanoscale

S. Heun, Sincrotrone Trieste: Photoelectron spectroscopy from individual heteroepitaxial nanocrystals on GaAs(001)

Saw-Wai Hla, Freie Universit t Berlin: Single Molecule Engineering with a Scanning Tunneling Microscope

T. Kinoshita, University of Tokyo: What can we do by photoelectron spectromicroscopy

M. Kiskinova, Sincrotrone Trieste: Scanning Photoemission Microscopy: recent developments and applications

M. Sakurai, RIKEN, Wako-shi: STM-induced light emission from nanostructures on a Si(001)-(2x1)-D surface

G. Salviati, CNR - MASPEC, Parma: Cathodoluminescence submicrometric depth profiling of optical emissions in semiconducting heterostructures and devices

T. Sekiguchi, National Research Institute for Metals, Tsukuba: Low-energy Cathodoluminescence Study of Semiconductor Nanostructures and Nanoparticles

N. Ueno, Chiba University, Chiba: Imaging of organic thin films using electron emission microscope

S. Ushioda, Tohoku University, Sendai: Probing of electronic transitions with atomic scale spatial resolution

. Watanabe, NTT Basic Research Labs, Atsugi: Synchrotron radiation photoelectron spectroscopy of nanostructures

K. agi, Tokyo Institute of Technology: Surface Imaging with use of an Energy Filter

N. amamoto, Tokyo Institute of Technology: TEM-Cathodoluminescence Study of Microstructures and Defects in Semiconductor Epilayers

WHAT CAN WE DO BY PHOTOELECTRON SPECTROMOCROSCOPY?

Toyohiko Kinoshita[1]

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Usually, the spatial resolution of photoelectron spectromicroscopy is poorer than that of conventional electron microscopy such as TEM, STM etc. The advantage of the photoelectron spsectromicroscopy is the possibility of element specific imaging and spectroscopic studies for very small samples and/or small area of surfaces. Discussion of the studies, especially towards mesoscopic effects, using these advantages will be performed. The design plan of the high-brilliant light source of University of Tokyo will also be introduced, which may be a most powerful light source for photoelectron spectromicroscopy.

NON-DESTRUCTIVE DETERMINATION OF LOCAL STRAIN UNDER BURIED INTERFACES WITH 100-NANOMETER SPATIAL RESOLUTION

S. Di Fonzo[1], W. Jark[1], S. Lagomarsino[2], C. Giannini[3], L. De Caro[3], A. Cedola[2,4], & M. Müller[4]

1) *SINCROTRONE TRIESTE, S.S.14 km 163.5 in Area Science Park, I-34012 Basovizza - Trieste, Italy*
2) *Istituto Elettronica Stato Solido (IESS) - CNR, V. Cineto Romano 42, 00156 Roma, Italy*
3) *Centro Nazionale Ricerca e Sviluppo Materiali (PASTIS-CNRSM), S.S. 7 Appia km 712, I-72100 Brindisi, Italy*
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In nano-fabrication with x-ray or electron lithography state-of-the-art structure sizes approach the level of only few tens of nm, and 180 nm features are already standard in microelectronics [1]. The strain induced under interfaces with these widths can locally distort the crystal lattice and may unpredictably affect the performance of the devices. uality control with nanometer resolution has thus mandatorily to be developed. The deeply buried strain fields need to be characterized with a non-destructive technique, which provides high sensitivity of at least 10^{-4} for relative lattice spacing variations. Only x-rays and electron beams can provide the requested spatial resolution. However, the latter do not provide sufficient penetration depth in reflection geometry, while the necessary sample preparation for transmission may irreversibly affect the original strain. -rays instead are non-destructive and have high penetration depth also in reflection. They can be focused by different means even to about 0.1 micron spot size. However, good strain sensitivity requires a high beam collimation, which these micro-spot techniques cannot provide, and thus until now strain analysis was made with spot sizes of only 10 microns [2]. Here we introduce a magnifying diffraction imaging scheme, which has as the principle optical component an x-ray waveguide (see fig 1.). It supplies in the vertical direction a source size of only about 100 nm, while it keeps the horizontal beam collimation unchanged [3]. These properties are well suited for the study of strain under interconnect lines. For our prototype study we choose an industrially relevant topic, the strain under oxidised lines on a silicon crystal. We put the structures, as indicated in fig. 1, such that their short dimension is in the vertical direction, assuming that their properties are homogeneous along the lines, and succeeded to measure the strain across narrow lines (width about 1 micron) with unprecedented spatial resolution of 100 nm and with competitive sensitivity of 10^{-4} for the relative lattice variations [4].

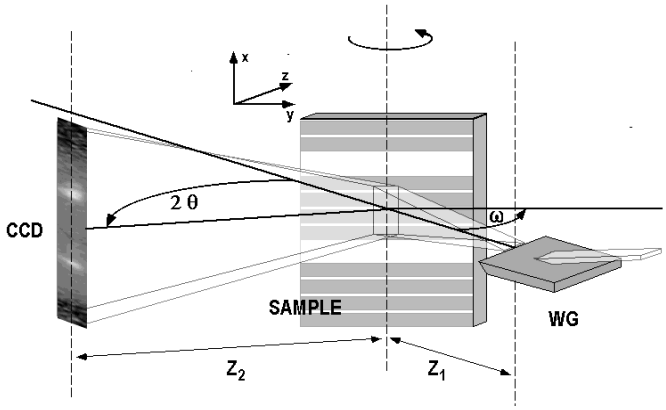


Figure 1: Set-up of the magnifying diffraction imaging scheme.

References:

[1] ESPRIT, Microelectronics Advanced Research Initiative (MEL-ARI), **Home page:** http://www.cordis.lu/esprit/src/melop-rm.htm
[2] P. C. Wang et al, *Appl. Phys. Lett*. **72**, 1296-1298 (1998).
[3] W. Jark et al., *J. Appl. Phys.* **80**, 4831-4836 (1996).
[4] S. Di Fonzo et al, Nature **403**, 638-640 (2000)

Surface chemistry of lead sulphide

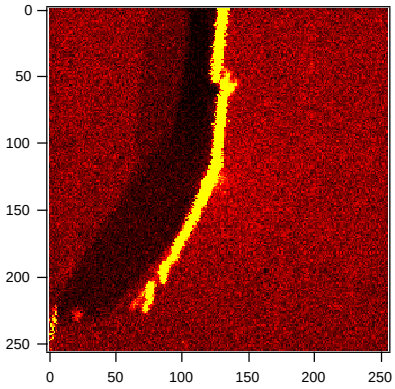
K.C. Prince, S. Heun, L. Gregoratti, A. Barinov, M. Kiskinova,

Sincrotrone Trieste, Area Science Park, km 163.5, Strada Statale 14, Basovizza (Trieste), Italy

Many minerals of economic interest are semiconductors and their surface chemistry is of fundamental importance in processes such ore concentration and treatment. In the present work we have examined the surface oxidation of one of the prototypical material, lead sulphide, in two states ”oxidised” in air for one year; and “clean”, cleaved in air and inserted into the vacuum system within about 10 minutes. The elements detected on the surfaces were Pb, S, O, C and Cl (the last two being natural impurities). Images showed topographic strong contrast (fig. 1) but no obvious chemical contrast. Spectra taken at different points show energy shifts up to about 0.5 eV, due to band bending and pinning of the Fermi level by defects or impurities.

Sulphur is present on the oxidised sample in a single chemical state with the same binding energy as for the clean sample, and is due to the native PbS below the oxide film. No oxy-sulphur oxidation products (sulphite, sulphate, thiosulphate) were present although these had been hypothesised. From the O 1s and Pb 4f binding energies, it is concluded that the species present are PbS, Pb_3O_4 and $Pb(OH)_2$, but no PbO was present.

Fig. 1. Topographical contrast from oxidised PbS. The bright feature is a step and the dark feature to the left is a shadow due to the detection of electrons at grazing emission.



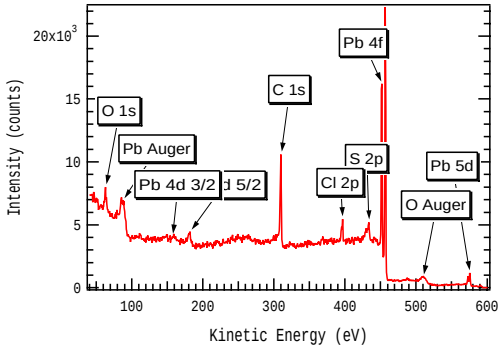


Fig. 2. Wide energy photoelectron spectrum at photon energy 600 eV.

Scanning Photoemission Microscopy: recent developments and applications

Maya Kiskinova

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Probed length scales of sub-micrometer dimensions have been achieved in photoemission spectroscopy thanks to the high flux and brightness of the soft x-rays provided by the third generation synchrotron sources. The operation principle, the present status of synchrotron radiation scanning microscopy and the advantages and disadvantages of the scanning photoemission microscopes will be presented and discussed.

The present performance level of synchrotron radiation scanning microscopes will be demonstrated using selected results obtained at ELETTRA.[1-4] The novel findings that will be discussed include identification of sub-micron area chemical phases at the surface and near surface regions, mapping of morphologically complex surfaces and interfaces and examination of mass transport phenomena.

1. M. Kiskinova, Surf. Int. Anal.30 (2000) 464 and references therein.
2. A. Schaak et al, Phys. Rev. Lett. 83 (1999) 1882 and unpublished data
3. L. Gregoratti et al, Phys. Rev. B57, L2134 (1998), ibid. B59, 2018 (1999).
4. S. G nther et al, J. Chem. Phys. B 101 (1997) 10004 and unpublished data

Spatially Resolved Spectra and Energetically Resolved Photoelectron Images of Reactive Surface Phases and PN-Junctions*

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We present results of investigations using the scanning photoelectron microscope (SPEM) at Elettra to study variations in structure and stoichiometry for the reactive Co/Si(111) and Ni/Si(111) systems, and variations in near-surface energy band bending on Si pn-junction device structure. For both Co and Ni adsorbed on Si(111) a competition exists between surface and silicide phases [1-3]. Si 2p maps or "images" acquired with the SPEM show contrast between phases due to different chemical shifts of the core-level energy. Measured spatially resolved Si 2p spectra allow us identify the stoichiometry of different silicide islands and correlate this with the island shapes. SPEM images of lateral silicon pn-junction arrays, fabricated on a Si(001) surface, show energy-dependent image contrast which varies continuously across the space charge region between regions of different doping [4]. The use of a 16 channel electron analyzer allowed us to image *the variation of the near surface band-bending across a pn-junction.* Combined with measurements of the changes in the spectra across the pn-junction, we are able to characterize variations in dopant concentration, the width of the charge depletion zone, and variations in band bending with oxide preparation.

*Work supported by the Laboratory for Physical Sciences, a NSF-MRSEC, and by Synchrotron Trieste.

[1] R. J. Phaneuf and P. A. Bennett, Surface Rev. and Lett., **5**, 1179, (1999).
[2] R. J. Phaneuf, P. A. Bennett, M. Marsi, S. Günther, L. Gregoratti and M. Kiskinova, Surface Sci., **431**, 232 (1999).
[3] L. Gregoratti, S. Günther, J. Kovac, R. J. Phaneuf, M. Marsi and M. Kiskinova, Phys. Rev. B **59**, 2018, (1999).
[4] R. J. Phaneuf, H.-C. Kan, M. Marsi, L. Gregoratti, S. Günther and M. Kiskinova, J. Appl. Phys., **88**, 863 (2000).

VISIBLE AND UV PULSED LASER PROCESSING OF THE Ti/Si(001) INTERFACE STUDIED BY XPS MICROSCOPY WITH SYNCHROTRON RADIATION

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2 ENEA, Div. Fisica Applicata, via E. Fermi 45, 00044 Frascati (RM), Italy

The interest for titanium silicides is related to the importance of the C-54 phase of $TiSi_2$, which has the lowest resistivity of all the refractory metal silicides and thus provides a good electrical contact in very large scale integrated devices. Among the several advantages of using laser processing to form $TiSi_2$, the most important one is that laser heating is localized in the area where the silicide formation and growth occurs, without necessity of annealing the entire wafer. In order to achieve compound selectivity and process optimization and control, detailed characterization of the lateral distribution of the silicide phases produced in the laser annealed areas is of fundamental importance.

We have studied the composition and spatial distribution of different Ti-Si phases formed by irradiation of a 5 ML Ti film deposited on Si(100) with visible and UV short laser pulses. The experiment was performed using SPEM at the Esca Microscopy beamline at Elettra. Laser irradiation of the Ti/Si(001) interface was performed in-situ, the mildly focussed beams of a Nd:Yag (λ=532 nm and 355 nm, FWHM=10 ns) and of an excimer laser operating with ArF (λ=193 nm FWHM=18 ns) entering normal to the sample through a quartz window. All experiments were carried out with photon energy of 545 eV, lateral resolution of 0.12 μm and overall energy resolution of 0.4 eV.

The lineshape of the Ti 2p and Si 2p spectra and the Ti 2p and Si 2p maps of the laser processed area have revealed that TiSi is produced in the external region of the laser spots, where the surface temperature does not exceed 500 °C, whereas in the central area $TiSi_x$ (x>=2) forms. The synthesis of more diluted alloys is consistent with the thermal gradient existing on the Ti/Si(001) surface during laser irradiation. The similarity in the surface composition and phases distribution observed at the 193 nm and 355 nm confirms that laser heating is the only driving force for the silicide formation process.

Zone Plate for x-ray applications

Enzo Di Fabrizio[a], Stefano Cabrini[b], Filippo Romanato[a], Anna Massimi[c] , Alessandro Nottola[b]
Lisa accari[a], Matteo Altissimo[a], Luca Businaro[a], Dan Cojoc[a]

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ABSTRACT

-ray microscopy is living a period of fast development. This progress was triggered by the performances of the focusing optical elements for -rays, which have recently pushed their resolution limit to the nanometer region, thus fully exploiting the optical characteristics of the third generation synchrotron radiation sources. In the fabrication field the fast progress is essentially due to the improvements of electron beam and -ray lithography techniques. The Zone Plate (ZP) can now be considered the main optical devices for high resolution beam focusing. For some applications, the efficiency is more important than the resolution, in this case, it is possible to fabricate a new type of multilevel zone plate. This geometry offers the double advantage of increasing the efficiency and of introducing selection rules that minimise parasite high diffraction order. Finally, a further progress toward an ideal continuos profile can be done by using both e-beam and -ray lithography even at -ray energy as will be described in the next sections. High resolution and high efficiency Zone Plate for -rays in the energy range of 300 e and 12 Ke fabricated by means of electron beam and -ray lithography will be presented. Regarding the high resolution regime zone plate with 40 nm outermost zone and thickness of 0.2 μm are fabricated. For high efficiency performances, multilevel zone plate and continuous profile were fabricated to provide an increase of efficiency at the first diffraction order and to suppress higher ones. The combination of the two lithography allows a powerful design flexibility at several energy regimes.

Imaging of organic thin films using electron emission microscope

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Imaging of surface electronic states offers new information on solid surfaces, which can not be obtained by usual surface-sensitive spectroscopy. Recent progress of photoelectron emission microscopy (PEEM) and low-energy electron emission microscopy (LEEM) has given various new informations on the surfaces including dynamics of the molecular adsorption. We have been trying to observe surface images of organic thin films using metastable-atom electron emission microscope (MEEM) which is selectively sensitive to the outermost surface. In this workshop, our recent results obtained by PEEM, MEEM and LEEM will be shown. By comparing these images, we can deeply understand the growth of organic thin films and organic/substrate interfaces.

The Effect of Surface Defects on the Efficiency of GaAsP Photoemitters for Polarised Electron Sources

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Institut für Physik, Johannes Gutenberg Universität, 55099 Mainz

Photoemitters on the basis of GaAs are worldwide in use as the most efficient sources of polarised electrons [1]. The electron polarisation is induced by optical spin orientation using circularly polarised light [2], usually from a red or infrared laser. The photon energy is matched to the band gap at the Γ point. Negative electron affinity of the emitter is induced by activating the surface with a Cs or Rb suboxide. Normally, the bands corresponding to the heavy and light holes are degenerate at the Γ point, leading to electron spin polarisation values of typically 30%. If the material is exposed to mechanical strain, the degeneracy at the Γ point can be lifted leading to a splitting of the bands at the Γ point. Provided proper matching of the photon energy to the gap between the topmost valence band and the bottom of the conduction band, much higher polarisation values (principally up to 100%) can be obtained. In several sources of polarised electrons such strained photocathodes are in use with the strain being induced via molecular beam epitaxy of thin photoemitter films on substrates with a certain lattice mismatch.

Due to the metastable nature of the strained film, these types of photocathode are rather delicate with respect to cleaning procedures and thermal treatment. In this contribution we present a study of strained GaAsP photocathodes by means of photoemission electron microscopy (PEEM). The aim of the study was to probe whether the photoelectron intensity and polarisation obtainable in a polarized electron source can be correlated with the structure of the surface as seen in the PEEM.

The strained film investigated was $GaAs_{0.95}$ $P_{0.05}$ (thickness 150 nm) epitaxially grown on $GaAs_{0.7}$ $P_{0.3}$ serving as substrate material. The lattice mismatch is about 1%. Such films can deliver a photoelectron yield in the order of 10^{-3} and spin polarisation values exceeding 70%. The PEEM investigation reveals that "good" cathodes are characterised by rectangular-shaped domains with sizes up to 10µm x 10µm that are almost undistorted. The domain boundaries are parallel to [100] crystallographic directions. In addition, a few point defects per terrace are visible. For a cathode which delivered a much lower yield and only half of the maximum polarisation, the surface appeared strongly distorted and rough. Characteristic small islands with elongated shape along the [100]-direction indicate that the strain has been released by generating an irregularly striped pattern with microdomains of widths smaller than 1 µm. Obviously, this structural change is the origin of the significant drop in efficiency, which made this cathode unuseable as photoemitter. A more detailed analysis of the PEEM images using a Fourier analysis procedure revealed an angular deviation of 1.5±0.5° with respect to the [100]-direction. A possible origin of this deviation will be discussed.

The experiment was funded by Materialwissenschaftliches Forschungszentrum (MWFZ) Mainz.

References:

[1] see, e.g., K. Aulenbacher et al., Nucl. Instr. and Methods A **391** (1997) 498
[2] F. Meier, B.P.Zakharchenya (Eds.), *Optical Orientation*, North Holland, Amsterdam (1984)

X-ray Photoemission and Low Energy Electron Microscope

Radovan Vašina, Martin Mynář and Vladimír Kolařík

Delong instruments, s.r.o., Bulharská 48, Brno, CZ-612 00, Czech Republic

An X-ray Photoemission and Low Energy Electron Microscope (XPLEEM) has been developed for use in Sincrotrone Trieste, Italy. To produce an image, XPLEEM can use several excitations of the specimen surface, such as electron beam, UV light, synchrotron monochromatized soft X-

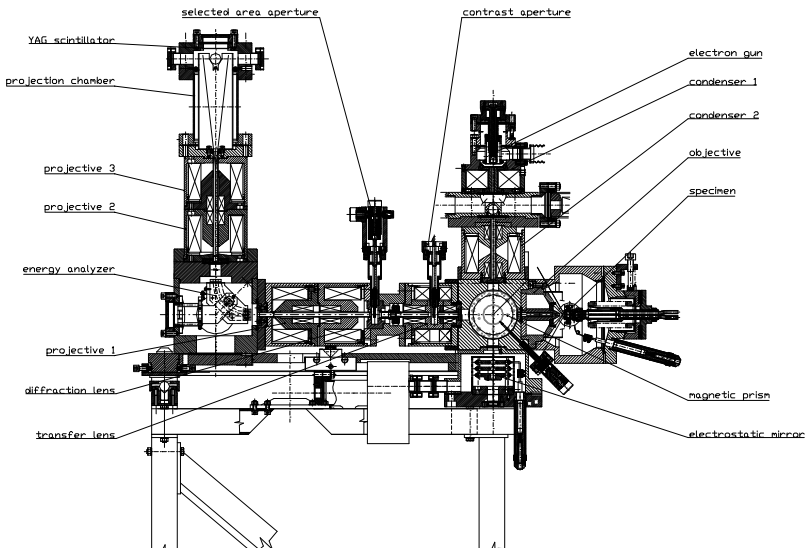

Fig. 1 Cross section of XPLEEM with the energy analyzer.

rays and hard X-rays. The electron optics column of XPLEEM [1] consists of an electron gun with a Schottky field emitter, two-fold symmetry magnetic prism with 90° deflection, objective, Wien filters, electrostatic tetrode mirror, projective and imaging analyser. Fig. 1 shows XPLEEM design. XPLEEM is now being tested and commissioned with other excitation sources than synchrotron light. Methods for alignment of XPLEEM have been proposed.

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X-RAY MICROSCOPY AND SPECTROMICROSCOPY TECHNIQUES USING MULTI KEV PHOTONS

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Multi-keV transmission X-ray microscopy and spectro-microscopy with full-field imaging (TXM) and scanning type (SXM) techniques take advantage of high penetration power, high optical resolution, and chemical sensitivity with having access to K absorption edges of materials. Thus, such a technique is suited for studies of bulk properties and gives complementary information to surface sensitive techniques like atomic force, scanning electron and photo-emission microscopy.

At the ID21 beamline at the ESRF, currently two complementary types of microscopes for photon energies of 2-7 keV are in use: (1) A full-field imaging or transmission x-ray microscope (TXM) working similar to a visible light transmission microscope gives highest resolution due to its static design. The use of incoherent illumination and parallel image formation allows relatively short exposure times in the second or minute range. Thus, the TXM is best suited for morphological studies, but is limited in acquiring spectroscopic information due to its full-field detection mode. (2) In the SXM, the image is formed by scanning the sample over a micro-probe. This allows simultaneous detection of different signals like transmission or fluorescence and is therefore best suited for spectro-microscopy analysis. The SXM requires spatially coherent illumination for diffraction limited imaging with zone plates. Thus, the limited usable part of the X-ray beam often results in increased exposure time.

Both microscopes use zone plates as focusing elements and work at sub-100nm spatial resolution. The microscopes are employed in a broad field of applications in life sciences, geochemistry and materials sciences like for example semiconductor research.

PHOTOELECTRON SPECTROSCOPY FROM INDIVIDUAL HETEROEPITAXIAL NANOCRYSTALS ON GaAs(001)

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In the last decade, semiconductor systems with reduced dimensionality have attracted great interest regarding their basic physical properties as well as possible electronic and optoelectronic device applications. Growth in the Stranski-Krastanov mode is widely used to fabricate self-organized nano-scale quantum dots without the need for expensive lithography processes.

However, while the quantum dot concept sounds very promising, there are some serious problems that limit its possibilities. Applications will clearly depend on the control of the quality, composition, size, and uniformity of the dots. For example, size fluctuations lead to large inhomogeneous broadening of the photoluminescence spectra of ensembles of dots. To overcome this problem, nano-scale spectroscopic techniques have been developed which permit the study of the properties of individual quantum dots.

We measured core level spectra of individual heteroepitaxial InAs nanocrystals on a Se-terminated GaAs-substrate with the spectroscopic photoemission and low energy electron microscope (SPELEEM) at ELETTRA which allows laterally resolved photoemission spectroscopy. The nanocrystals were obtained by depositing nominally 2 and 4 monolayers (ML) of InAs on a Se-terminated GaAs surface. The Se-termination of GaAs(001) results in the formation of a 2-3 ML-thick film of Ga_2Se_3 on top of GaAs(001). During heteroepitaxy the InAs reacts with the Ga_2Se_3. A phase separation takes place on the anion sublattice, while an alloying takes place on the cation sublattice. During the initial stages of growth, a submonolayer-thick wetting layer of $In_xGa_{1-x}As$ is formed which is capped by $(In_yGa_{1-y})_2Se_3$. $(In_yGa_{1-y})_2Se_3$ covered InAs nanocrystals are formed on this surface. The implication of our results is that significant mass transport has occurred from the Se-terminated GaAs surface to the nanocrystals.

This is confirmed by differences in valence band structure and work function between the nanocrystals and the Se-terminated GaAs substrate which we measured with the SPELEEM. The samples showed differences in the valence band edge position and work function both between nanocrystals and substrate as well as between 2 ML and 4 ML. We suggest that a Se-termination of the InAs nanocrystals caused by the $(In_yGa_{1-y})_2Se_3$ surface layer is the reason for these differences.

Cross-sectional photoemission microscopy of semiconductor heterostructures

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Photoemission spectroscopy has been traditionally used to monitor chemical, structural and electronic changes upon interface formation. [1] Measurements are usually performed at different growth stages and the junction parameters are inferred from the coverage-dependence of the core emission. With the emergence of spectromicroscopy, a natural application of photoemission to the study of semiconductor interfaces is to directly determine the heterojunction parameters by measuring the device in cross section.

Initial test systems examined were GaAs p-n doping superlattices with different periods produced by molecular beam epitaxy (MBE). After growth, wafers were mechanically thinned-down to below 100 μm, pre-notched and transferred in vacuum to the photoemission spectrometer. *In-situ* cleaving was used to expose {110} cross-sections of the buried interfaces. Several efforts were devoted to obtaining flat bands conditions on both the p- and the n-side of the junction. Such conditions were inferred from an analysis of the Ga 3d core lineshape with energy and spatial resolution better than 0.15 eV and 0.5 μm, respectively, having reduced to a negligible spectral contribution the diffuse background typical of any Schwarzschild microscope.

The methods developed were then applied to Al/n-GaAs(001) Schottky junctions, also fabricated by MBE. Spatially-resolved studies of the core-level emission in cross-section were used to produce a map of the semiconductor depletion layer near the metallurgical junction. Our data show that a systematic study of this system as a function of doping type and level can provide unique information of the static surface dielectric constant, which is relevant to many aspects of surface physics, including core-hole relaxation and electron/surface interactions. [2]

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Synchrotron radiation photoelectron spectroscopy of nanostructures

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Nano-structured materials, such as semiconductor nanostructures and carbon nanotubes, with a dimension less than the de Broglie wavelength of electrons, are expected to exhibit quite different electronic properties from those of common three dimensional materials. Our research aim is to understand the formation mechanism of semiconductor nanostructures and the electronic structure of nano-structured materials by means of soft x-ray photoelectron spectroscopy. The recent results on nanoscale crystal formation through surface modification, the electronic structures of these nanocrystals, and the electronic properties of single-walled carbon nanotubes will be presented [1,2]. Furthermore, newly-developed time-resolved and submicrometre-area photoelectron spectroscopy systems will be presented [3,4].

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Formation stages and thermal stability of metal/n-GaN interface

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The interfaces between some metals and n-GaN surface have been investigated by means of synchrotron radiation scanning photoelectron microscopy. The first stages of the interface formation at room temperature and the evolution of the interfaces after stepwise annealing have been studied. Our results reveal temperature dependence of the Schottky barrier heights, which in case of Au/GaN interface are caused by structural rearrangements below 500 ^{0}C and by formation of an Au gallide alloy above 500 C. The effects of the spatial heterogeneity and the presence of GaN growth defects on composition of the interface and the barrier heights are examined (fig.1, fig2).

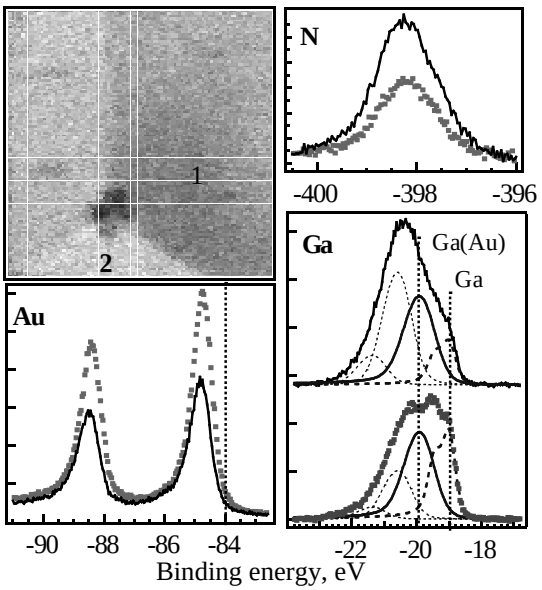

Fig.1. 22 μm image of Au $4f_{5/2}$ photoemission line with sets of spectra taken at points 1 and 2 which reveals different concentration of $AuGa_2$ on different grains after annealing above 750^{0}C but the same barrier height on different features.

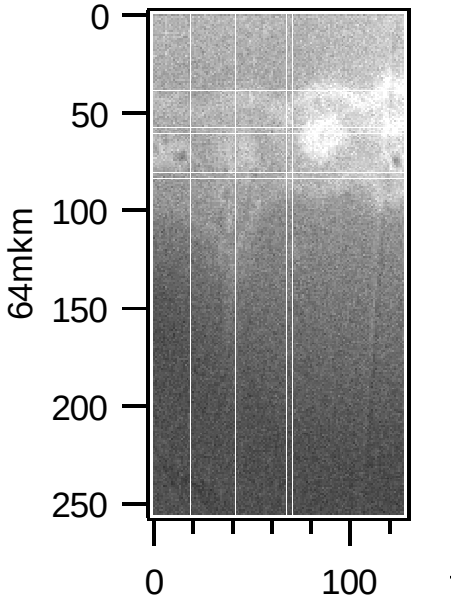

Fig.2 Ni3p image showing Ni concentration variations around carbon defects on GaN grown on SiC after annealing to 300^{0}C.

Self-organized semiconductor nanostructures and their applications

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Within the trend towards miniaturization in high-technology applications there are increasing demands for effective and inexpensive ways to fabricate nanostructured surfaces. Exploitation of strain-induced self-organization phenomena during semiconductor heteroepitaxy is an elegant route towards large-scale arrays of uniform nanostructures as it will be demonstrated for the Stranski-Krastanov growth of SiGe films on Si(001) substrates.

Due to the lattice mismatch, the growth front of SiGe films on vicinal Si(001) undergoes a series of elastic strain-relief mechanisms. These include surface reconstruction, step bunching of the preexisting substrate steps, subsequent formation of {105}-faceted three-dimensional crystallites, and the formation of a dislocation network. Using atomic-force microscopy it is shown that one can use strain relief to create a variety of periodic nanoscale structures by tuning substrate miscut, alloy composition, and layer thickness [1]. Among these structures are ripple patterns with a periodicity ranging from a few tens of nm to 1 µm (Fig. 1a) as well as two-dimensional arrays of self-organized crystallites. (Fig. 1b).

Since these self-organized semiconductor nanostructures cover the entire wafer on which the films were grown, they can be used as large-area nanopatterned templates for subsequent deposition of various material. We will demonstrate this novel application of semiconductor nanostructure for the case of magnetic roughness determination of nanopatterned Co films [2].

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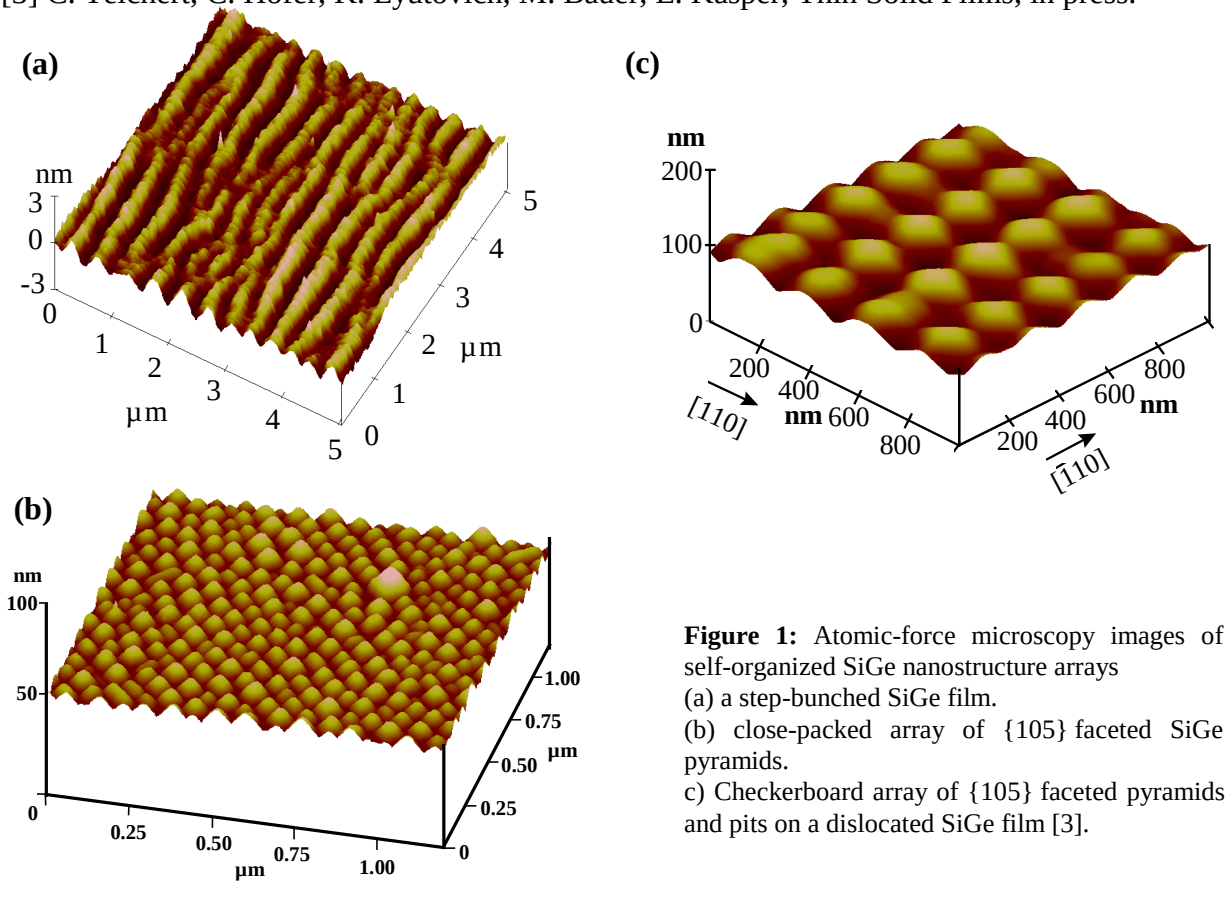

Figure 1: Atomic-force microscopy images of self-organized SiGe nanostructure arrays
(a) a step-bunched SiGe film.
(b) close-packed array of {105} faceted SiGe pyramids.
c) Checkerboard array of {105} faceted pyramids and pits on a dislocated SiGe film [3].

WAVEFUNCTION MAPPING IN SINGLE QUANTUM DOTS

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An Ultra High Vacuum-300 mK-Scanning Tunneling Microscope for Local Measurements of the Density of States on InAs(110)

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To investigate low dimensional semiconductor systems and metal/semiconductor hybrid systems at high magnetic fields with high energy resolution we have developed an ultra high vacuum (UHV)-low temperatur-scanning tunneling microscope (STM).

The STM can be cooled down to 300 mK with a ^{3}He cryostat and a magnetic field up to 14 T can be applied perpendicular to the sample surface. The bakeable insert of the cryostat provides UHV-conditions and is flanged on top of a 3 chamber UHV-system that contains standard surface analysis techniques and preparation techniques for STM-tips and samples: in particular a room temperature STM with molecular beam evaporators, a LEED/Auger unit and a system to measure the magneto optical Kerr effect on magnetic films. From the cryostat, where low temperature measurements are performed, the STM can be moved into a transfer chamber to exchange STM-tips and samples under UHV-condition.

To demonstrate the capability of the method, we will present two recent results obtained with a (6K / 6T)-UHV-STM on InAs(110). First, the electronic structure of the three dimensional electron system (**3**DES) of the narrow gap semiconductor is investigated by scanning tunneling spectroscopy (STS) and magnetotransport measurements in the extreme quantum limit. The well-known oscillations of the Hall coefficient [1] are reproduced and the last, most pronounced oscillation is shown to be correlated with the appearance of corrugations in the local density of states (LDOS) at the Fermi level. Second, STS-measurements of the LDOS of the adsorbate induced **2**DES will be presented and compared to photo electron spectroscopy measurements of the same system.

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Growth and characterization of Ge/Si(111) quantum dots by Scanning Tunneling Microscopy

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We have studied by Scanning Tunneling Microscopy the formation of quantum dots obtained by Reactive Deposition Epitaxy of Ge on Si(111) at T=450-550 °C. The height and shape of these islands depend on the coverage and on the thermal treatment (deposition rate, substrate temperature or post-growth annealing). The 3D coherent islands nucleation start at a Ge coverage between 3-5 ML, depending on the Ge flux and on the substrate temperature. At T=500° C the 3D islands (average lateral dimensions 150-200 nm) start to appear as truncated tetrahedra; the top of the islands is 7x7 reconstructed, showing a substantial Ge-Si intermixing or at least a modification of the classical Ge(111) reconstruction caused by the stress fields on the island. These islands evolve firstly by introducing new faces and subsequently by including dislocations at the island border, which help to release the misfit strain. Finally an erosion of the top face and of the substrate around the islands occurs, probably connected to a substantial Ge-Si intermixing.
We have also analyzed the distribution of the islands on the surface, which is greatly influenced by the step bunching arising from the flash-annealing procedure. Large flat areas form, separated by groups of steps, so that the deposited material is attracted towards the steps. Two different growth regimes arise: initially the islands form and evolve only on the steps, up to complete ripening; subsequently the same happens on flat areas of the sample. The distance between the islands and the steps is nearly constant, forming initially a single row of equally spaced dots, which is followed by other rows in between. The exploitation of this phenomenon, governed by the surface diffusion coefficient of Ge on Si and by the step distance, is one of the possible paths to achieve the self-organization of quantum dots.

Probing of electronic transitions with atomic scale spatial resolution

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By spectroscopically analyzing the light emitted from the tip-sample gap of the scanning tunneling microscope (STM), we have investigated the electronic transitions in individual quantum wells (QW) of semiconductors and at different atomic sites on a metal surface. The examples of AlGaAs/GaAs QW's and the reconstructed Au(110)-(2x1) surface will be discussed.

IMAGING OF QUANTUM DOT STATES BY SCANNING TUNNELING SPECTROSCOPY

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Advances in epitaxial growth techniques have made electronic wave function engineering a reality. Material systems can be constructed with nanometer dimensions where the nature of the concomitant bound states can be controlled and tuned to the desired electronic and subsequent optical properties. The present challenge facing nanotechnology research is the ability to characterize the nature of a nanostructure material system on a level as fundamental as the electronic wave functions. In the past 15 years there has been widespread use of scanning tunneling microscopy (STM) and spectroscopy (STS) techniques to study individual surfaces atoms and molecules on surfaces. In this abstract we attempt to demonstrate the ability of STS imaging to characterize single artificial atoms in the form of InGaAs quantum dots.

The study of self-assembled InGaAs quantum dot system is a topic of current actuality primarily for advances in opto-electronic technology. In this work we examine intact, un-capped InGaAs quantum dots of device quality material grown by epitaxial, strain-driven self-assembly on a GaAs matrix. These are complex systems where the confining potential is defined by the shape, size, compositional gradient and the strain-field (which are controlled by the growth conditions and are generally not known precisely) of the quantum dot and results in discrete, atomic-like eigenstates. As a consequence of their δ-function-like density of states and the ability to finely tune the wavelength of emission by adjusting the growth conditions, intense emission at 1.3μm has been achieved which is of importance for opto-electronics.

We report, by a combination of scanning tunneling microscopy (STM) and spectroscopy (STS), the direct imaging of the charge density of the confined electronic states, and hence the wave functions, of $In_{0.5}Ga_{0.5}As$ quantum dots. The *s*- and *p*-like hole states have been identified from voltage-dependent conductance maps and compare well with *ab initio* calculations. In this presentation issues pertaining to the imaging of the quantum dot wavefunctions and their subsequent influence on the electronic and optical properties, such as the Coulomb interaction and the alignment of the electron and hole wave functions shall be discussed.

IMAGING OF ZERO-DIMENSIONAL STATES IN SEMICONDUCTOR NANOSTRUCTURES USING SCANNING TUNNELING MICROSCOPY

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We have characterized the local density of states (LDOS) in semiconductor nanostructures at epitaxially grown InAs on a GaAs(111)A substrate using scanning tunneling microscopy (STM) at 5.3 K. The dI/dV images of the two dimensional electronic states at the surface accumulation layer show a bias-dependent LDOS distribution in triangular nanostructures (Figure 1). Calculations reveal that each dI/dV image corresponds to a discrete zero-dimensional state.

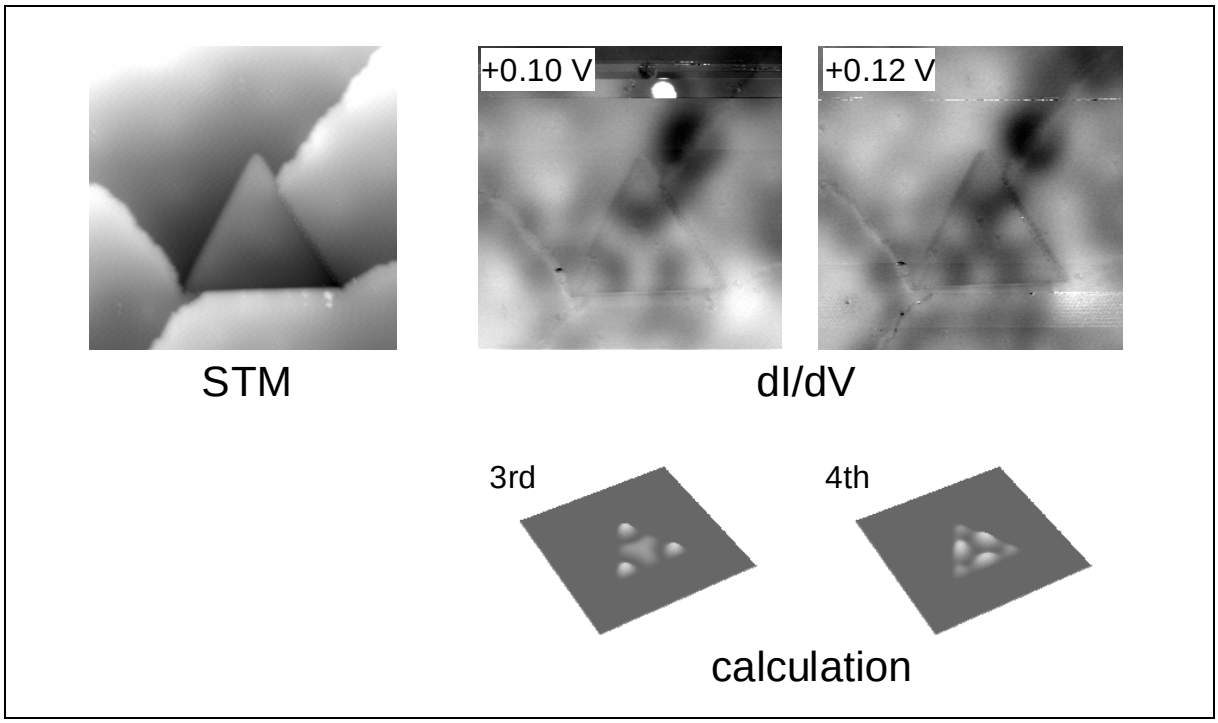

Figure 1: The STM image shows a triangular nanostructure (faultily stacked InAs crystal) at the InAs(111)A surface. The dI/dV images show the bias-dependent LDOS distribution within the nanostructure at 5.3 K. Image size is 67 nm x 67 nm. The dI/dV images correspond to calculated zero-dimensional states of the third discrete and fourth degenerate energy levels respectively. The bias difference of 20 mV is comparable with the energy separation between these energy levels.

ELECTRONIC STRUCTURE OF THE Pb/Si(111) MOSAIC PHASE

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We have used Scanning Tunnelling Spectroscopy and Microscopy to study the $\sqrt{3} \times \sqrt{3}$ R30° mosaic phase of Pb/Si(111). The surface composition is typically $Pb_{0.54}$ $Si_{0.43}$ with a total coverage of approximately 1/3 of a monolayer of lead and silicon atoms [1]. The structure is characterised by short Pb-Si-Pb chains from 2 to about 12 Pb atoms long, which branch at angles of 60 or 120 degrees. Both the Pb and Si atoms are on lattice sites, but the structure contains considerable chemical disorder, and is without long range order.

For tunnelling into empty states both Si and Pb atoms are imaged with about equal contrast. For tunnelling from the filled states, the Pb atoms are much brighter than Si, indicating charge transfer from the Si to the Pb atoms.

In the scanning tunnelling spectra, three clear peaks in the density of occupied states are evident, spaced at intervals of about 300 meV and with a width of about 250 meV. The surface is semiconducting with a gap of about 0.8 eV determined by the zero current region of the curve.

These structures are surprisingly sharp in view of the considerable chemical disorder of the surface, and suggest localised states confined in a linear quantum well.

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STM-INDUCED LIGHT EMISSION FROM NANOSTRUCTURES ON A SI(001)-(2X1)-D SURFACE

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Optical properties of nanostructures and light propagation in a narrow space were studied using the technique of scanning tunneling microscope (STM)-induced light emission. The method was first reported by Gimzewski in 1988 [1]. A system for measuring STM-induced light emission is a combination of an STM and a light detection system. Photon is created as a result of inelastic scattering in the tunneling gap. The light detected at a far field distance includes information of photon creation in the tunneling gap and light propagation from the tunneling gap [2,3].

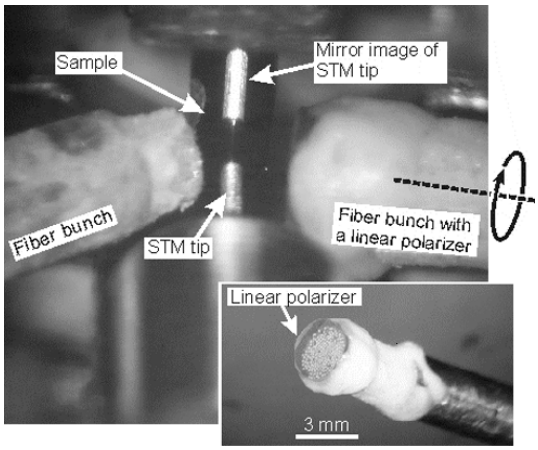

Figure 1: Photon detecting STM

Optical spectra and photon intensity maps of light emitted from nanostructures comprised of Si dangling bonds or Ag clusters on Si(001)-(2x1)-D were measured at room temperature in an ultra high vacuum chamber. Light intensity map obtained by scanning the STM tip on the surface gives an atomic-scale spatial resolution as comparable to that of a topographic STM image. Spectroscopic data give information of the energy position and the orbital symmetry of the surface states. These properties are explained in terms of a model based on optical dipole transitions creating light in the tunneling gap and subsequent light propagation.

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On the spatially resolved electronic structure of polycristalline WO_3 films investigated with Scanning Tunneling Spectroscopy

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The electronic structure of poly-crystalline monoclinic WO_3 has been investigated with unprecedented spatial resolution on this type of systems by means of Scanning Tunneling Spectroscopy. Current Image Tunneling Spectroscopy maps show a clear and systematic increase of the conductance of the surface at the WO_3 crystallite boundaries, with a corresponding marked narrowing of the bulk band gap. This effect is assigned to the occurrence of σ bonded electronic states lying deep in the bulk band gap and due to pairing of reduced W ions at shear planes between adjacent crystallites. STS evidences of π bonding states at the Fermi edge is also presented for the spectra acquired at the crystallite boundaries [1,2].

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Electronic excitations induced enhancement in metallicity on HOPG and Si surfaces: *in-situ* STM STS studies

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Surfaces of highly oriented pyrolytic graphite (HOPG) and hydrogen passivated Si (111) after swift heavy ion (200 Me Au ^{8} and Ag ^{14} ions) irradiation have been investigated by home made *in-situ* high vacuum scanning tunneling microscopy (STM) and scanning tunneling spectroscopy (STS) techniques attached with materials science beam line at 15 M Pelletron accelerator. The virgin surfaces for HOPG were prepared by cleavage of top surface by an adhesive tape, whereas virgin hydrogen passivated Si(111) surfaces were prepared by aqueous HF acid treatment [1]. The STM topographic images (Fig. 1) show the delocalization of the electronic states after swift heavy ion irradiation. The spectroscopy results show approximately linear variation of tunneling current with bias voltage after irradiation. The ohmic behavior of I- curve along with the delocalization of the covalent bonded electronic wavefunctions suggests the metallic nature of these surfaces after irradiation. This is attributed to the electronic excitation induced enhancement in the metallicity of the bonding orbitals [2-3].

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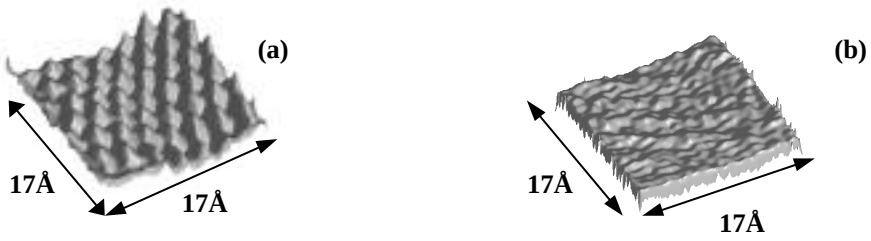

Fig. 1. *In-situ* STM images of (a) pristine HOPG and irradiated with 200 Me Au ^{8} ions at the fluence of $2x10^{13}$ ions cm^{-2}.

Single Molecule Engineering with a Scanning Tunneling Microscope

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By using a variety of STM-tip induced atomic and molecular manipulation techniques, the fundamental reaction steps such as dissociation, diffusion, desorption, re-adsorption and association processes of single molecules have been performed in a controlled step-by-step manner on Cu(111) surface between 8 and 20 K. Based on inherent differences in the bond energies, selective abstraction of iodine atoms from the single iodo- and diiodo- benzene molecules can be succeeded by using tunneling electrons. The necessary threshold voltage to break a single bond inside the molecule was determined by acquiring the I- tunneling spectrum of the single molecules. The diffusion processes of the single molecules and atoms were studied by employing the lateral-manipulation technique where tip-adsorbate interaction forces play a key role. The iodine atom can be moved along any random directions on Cu(111) and the detailed atom movements are decoded from the complex manipulation curves by comparing with simple theoretical simulations. Di-iodobenzene molecules can be followed the STM-tip only along the close-packed row directions on the Cu(111) terrace. Both species show strong attractive interaction toward the STM-tip. To observe the molecular-shapes in STM images, the STM-tip was functionalized by deliberately transferring an iodine atom into its apex where controlled desorption and re-adsorption processes have been applied by inelastically exciting them with the tunneling electrons. Furthermore, rotation and up-right tilting of the di-iodobenzene molecules can be induced at will with the STM-tip which can be explained by inelastic tunneling and di-polar interaction between the tip and the molecule. The adsorbed molecule positions can be altered by applying attractive tip-sample interaction forces and the process can be controlled by corresponding tip-height signals. This can be useful in the repositioning of molecules to the desired adsorption geometry with atomic scale precision. Association between the two molecules can be realized by simultaneously exciting them with the tunneling electrons where the STM-tip induced bond formation process can be explained by rotational excitation of the molecules. By choosing a suitable combination of manipulation techniques, the whole chemical reaction can be created over the single molecules for the first time and as an example, creation of Single-Molecule-Ulmann-Reaction , will be presented where bi-phenyl molecules are individually assembled by using the STM tip. These experiments are the important steps forward for both basic science and future fabrication of nano-devices with the use of a STM.

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STM Nanolithography on SiO_2/Si

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Electron-beam induced reduction of SiO_2 had been known since the early days of SiO_2 Auger spectroscopy studies. Combined with thermal annealing, locally confined low energy electron-beam from a STM tip can be used for nanofabrication of Si oxide film on Si substrate as nano-lithography mask [1-4]; the oxide layer within the e-beam exposed area can be decomposed, and then evacuated from the surface at elevated temperatures above 300C. Nanometer scale patterns of oxide windows on Si substrate such as dot window arrays, lines, and circles can be formed (Fig. 1[3,4]). The sizes of Si oxide windows are estimated against the e-beam exposing time, by which a Gaussian-type e-beam profile is revealed. The results show that the Si oxide windows can be adjusted by varying the e-beam exposure time. We reveal the electron-energy dependence of the quantum yield which suggests the Knotek-Feibelman like mechanism.

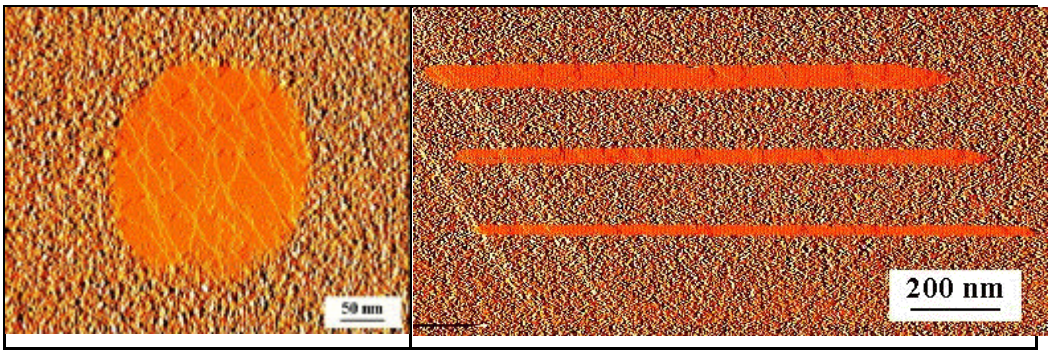

Figure 1: An open window and a line-and-space pattern. The minimum feature size attained is 25nm and rms edge roughness of line patterns is less than 3.5nm.

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Surface structure of some transition-metal trichalchogenides

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The transition-metal trichalcogenides are built of linear trigonal prismatic chains, which form layers separated by van der Waals gaps [1,2]. This structural arrangement is responsible for the one-dimensional character of these compounds and for their physical properties. In the simplest case all [TX_3] chains are equivalent (e.g. $ZrSe_3$), while in some cases there may be two (e.g. $TaSe_3$) or even three (e.g. $NbSe_3$) different types of chains. In addition to its peculiar structure $NbSe_3$ has another unique property, i.e. sliding of charge density waves (CDW), which is a phenomenon not fully understood yet [3].

The composition of the crystals was determined by activation analysis and contrary to the other two it was found that $ZrTe_3$ is always tellurium reach. The crystals were examined in a scanning tunneling microscope (STM) at RT under ultra-high vacuum conditions.

Although high-resolution STM images of all three compounds were recorded during both constant height and constant current mode measurements, a charge instability was constantly present, especially in the triselenides. It is believed that the instability in $ZrSe_3$ and $NbSe_3$ is a result of the peculiar charge distribution between the three chalcogen atoms forming the bases of the trigonal prisms. In $ZrSe_3$ two of these should form a doublet and the third is supposed to carry the same charge alone. According to the STM images the charge seems to switch easily between this three selenium chains under the influence of the local field during scanning. In case of $NbSe_3$ the resolution of the STM images is additionally depended on the direction of scanning. The charge centers were found to be displaced from the expected atomic positions, which is attributed to a precursor effects to CDW sliding.

Contrary to the selenides, there is a structural modulation in the tellurium rich $ZrTe_3$. This modulation shows up clearly in the Fourier transforms of the images. Additional tellurium atoms occupy the only available interstices and form zigzag chains with one of the two chains of the basic structure. Consequently, this column is expected to carry a larger charge as compared to the adjacent one, which stabilizes the charge distribution.

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STRAIN ANALYSIS IN SUBMICRON ELECTRON DEVICES BY CONVERGENT BEAM ELECTRON DIFFRACTION

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Convergent beam electron diffraction (CBED) is an electron microscopy technique which determines the local strain in electron transparent crystalline films through the shift of High Order Laue Zone (HOLZ) lines which form in the central disk of the diffraction pattern. Strains as small as $2 \cdot 10^{-4}$ can be detected by monitoring the shift of the HOLZ lines when compared with the HOLZ pattern of an unstrained region of the sample. CBED is a point-to-point quantitative technique, yielding the components of the strain tensor for each measured point in the region of interest; its spatial resolution is still unsurpassed, as 1 nm electron probes can be routinely obtained with the modern microscopes equipped with field emission guns (TEM/FEG). Recently, TEM/CBED has been applied to the strain field analysis in the active region of sub-micron shallow trench isolation (STI) structures. An example is given in Fig.1, where the CBED patterns have been taken in a number of points along a line parallel to the padoxide/silicon interface ('cutline'). An EU funded research project on the application of TEM/CBED to 0.15 μm CMOS processes for non volatile memories has started this year [1].

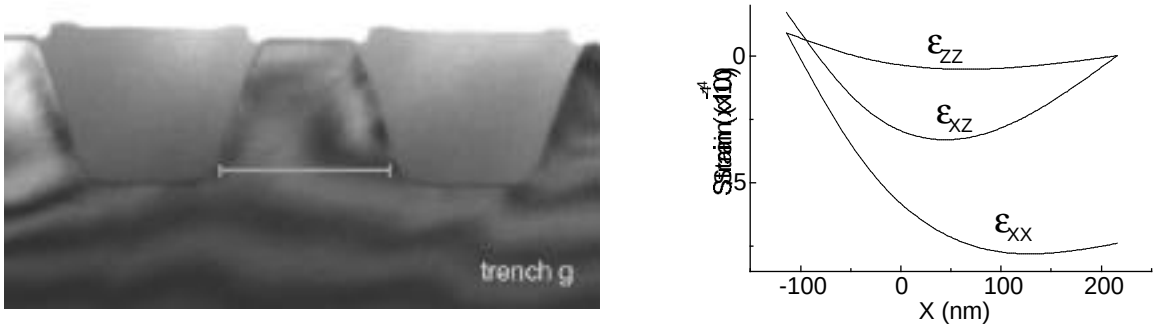

Figure 1. (left) TEM image of a 0.37 μm large active area bounded by two STIs. The horizontal line represents the cutline along which the CBED patterns have been taken, giving rise to the strain field reported on the right.

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New trends in the optical investigation of semiconductor heterostructures at the nanoscale

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Abstract. The continuous shrinking of the active regions of semiconductor heterostructures and devices results in unprecedently demanding characteristics of the laboratory tools for optical investigation of the materials quality and device performances. The recent implementation of Scanning Near-field Optical Microscopy (SNOM) techniques within the broad field of optical spectroscopy has made it possible to achieve a spatial resolution well below the laser wavelength used for excitation. We report on recent results on the recombination and emission dynamics in quantum dots and quantum wire structures, obtained by using near-field luminescence spectroscopies, both continuous wave and time-resolved.

In particular, we have investigated the photoluminescence (PL) properties of GaN / AlN quantum dots, at room temperature, in order to assess the emission patterns from these structures, so highly relevant for device application. Very long recombination lifetimes have been recorded in these structures by implementing, in the SNOM microscope, a time-correlated single photon counting apparatus with a time resolution of the order of 50 ps.

The optical properties of single stressor-induced quantum wires, obtained from electron beam litography followed by chemical etching, have been investigated by means of a low-temperature SNOM under continuous wave excitation. We are able to show that a clear distinction between the presence versus absence of one-dimensional confinemant due to the linear stressors can be obtained in this way and the issue of exciton localization in these structures is discussed.

A novel tip-surface distance control

for low temperature scanning near-field photoluminescence spectroscopy

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Near-field distance controls, based on tuning forks without piezo elements, have been successfully employed in atomic force microscopes[1]. We extended this technique to the case of low temperature SNOM. In our experimental set-up, the fibre is fixed at two points. The first point is glued to a rigid support two centimeters far from the tip, while the very end of the tip is attached to the fork itself (see fig.1). When the system is driven at its resonance frequency, by an alternating voltage applied to the tuning fork, the electrical impedance is reduced to a minimum. Since the oscillatory parameters (resonance frequency, phase difference between voltage and current, width of the resonance) depend on the magnitude of the shear forces acting on the tip, a feedback control of the tip-surface distance is performed by directly monitoring the amplitude and the phase of the electrical current in the fork. The system is stable (several hours), compact and it does not require neither piezo elements nor voltage preamplifiers, thus resulting suitable for a low temperature environment. With a spatial resolution of 150 nm, fine structures in semiconducting heterostructures can be therefore observed at 5K. In the case of V-shaped quantum wires, we show that the photoluminescence emission comes from elongated boxes (fig.2). The spatial distribution and coherence length of 1D excitons in the homogeneous regime is observed for the first time.

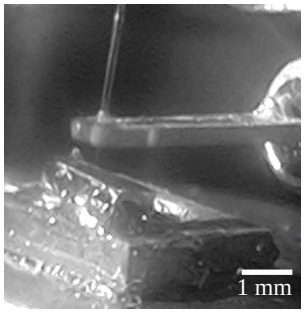

Fig.1 Picture of the low-temperature SNOM sensor. The optical fiber is mounted perpendicular to the quartz fork.

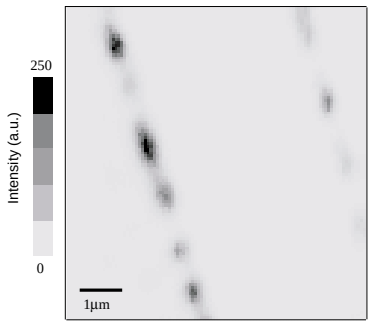

Fig.2 Intensity map of quantum wire PL emission.

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OPTICAL PROPERTIES OF V-GROOVE QUANTUM WIRE LASERS AND LED

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The improved performances of quantum well (W) optoelectronic devices with respect to the equivalent bulk heterostructures devices led to the consideration that the realization of lower dimensionality i.e. quantum wires (WR) and quantum dots (Ds) was the path to follow toward the ultimate luminescent device [1]. So far no such improved performance has been demonstrated mainly due to the neglected impact of Coulomb correlation effects and of structural inhomogeneities of real low dimensional structures.

In this work we will present an overview on recent results obtained on optically pumped - groove GaAs AlGaAs WR lasers We will show that the lasing emission is sustained by excitonic gain arising from a population inversion of localized excitons. Moreover photoluminescence excitation (PLE) and direct absorption measurements will be presented as direct evidence of stimulated emission from the one-dimensional ground subband up to 150K in cw operation. We will also present some recent progress in the fabrication of electrically pumped Ga AlGaAs WR lasers on which anodic oxidation techniques have been used to effectively confine the injected current into the WR active region. Finally experimental results on high quantum efficiency InGaAs based WR LEDs will be presented.

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TEM-Cathodoluminescence Study of Microstructures and Defects in Semiconductor Epilayers

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Optical properties of microstructures and defects in semiconductor epilayers grown on substrates were studied by a cathodoluminescence (CL) detection system equipped with a transmission electron microscope (TEM). The TEM-CL technique is useful for studying optical properties of nano-scale regions, because it gives a CL spectrum from a localized region irradiated by a focused electron beam and reveals a spatial distribution of light emission as a CL image. Here we present a few applications of this technique to the study of InGaAs, ZnSe and GaN epilayers grown on GaAs substrates.

1. InGaAs: Monochromatic CL images taken by the emission from the $In_xGa_{1-x}As$ layers show linear features running parallel to the 110 directions. There is no direct correlation between the misfit dislocations observed in TEM images and the strong line contrasts in the monochromatic CL images. In addition the linear features also appear in a thin region where the misfit dislocations are removed. Those results indicate that the linear features are considered to be due to compositional fluctuations of the In concentration in the $In_xGa_{1-x}As$ layer [1].
2. ZnSe The $_0$ emission from ZnSe has been reported to be concerned with dislocations [2]. Several types of dislocations in the ZnSe GaAs samples were examined by the TEM-CL technique. It was found that some of the threading dislocations with the 1 2 110 type Burgers vectors emit the $_0$ emission, while misfit dislocations at the interface and extended dislocations are not luminescent.
3. GaN Threading dislocations with three types of Burgers vectors show dark contrasts with different intensities in the monochromatic CL image of the free-exciton emission. The relation between the contrast and the character of the dislocation was investigated from the comparison of the dark field TEM images with the CL image using a plan-view thin sample and a cross-sectional sample.

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COMPARISON OF HIGH RESOLUTION TRANSMISSION ELECTRON MICROSCOPY AND LOW FREQUENCY RAMAN SCATTERING IN DETERMINATION OF PARTICLES SIZE DISTRIBUTION OF NANOSIZED TiO_2

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Size distribution of TiO_2 nanoparticles synthesized by sol-gel method are determined by high resolution transmission electron microscopy (HRTEM) and compared with those determined by low frequency Raman scattering (LFRS). The methodology for the determination of nanosized particle distribution by LFRS is described. It is based on ν^{-1} dependence of the Raman light to vibration coupling coefficient and on the fact that each nanocrystallite of diameter D vibrates with its own eigen frequency $\nu \sim 1/D$. The effect of the particle vibrational lifetime on the shape of distribution is analyzed and found to be negligible for the weakly connected TiO_2 particles. Fig. 1 shows comparison of the HRTEM and LFRS TiO_2 size distributions. On the basis of comparison of particle size distributions deduced by HRTEM and LFRS on 15 different samples, the Raman spectroscopy shows to be simple, fast method that has favorable statistic over the macroscopic probe volume and that enables "in situ" measurements.

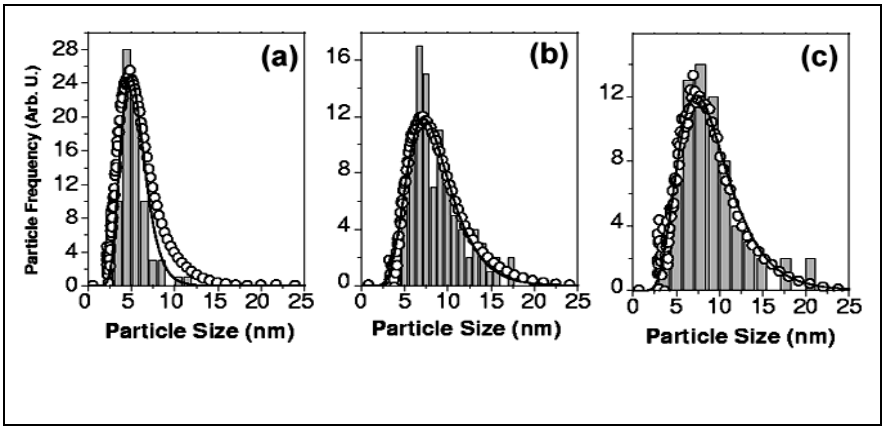

Figure 1: Particle size distributions for the sample S_1 (a), S_1 annealed 1 h at 320 ^oC (b), S_1 annealed 1h at 450 ^oC (c), bars - HRTEM results, circles - LFRS results, line - fit of log-normal distribution on the HRTEM distribution.

STRUCTURE AND CHARGE TRANSPORT IN N^+ BEAM INDUCED π-BONDED NANOCRYSTALLINE CLUSTERS IN POLY PHENYLENE OXIDE

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The method of ion implantation with a precise dopant concentration and accurate depth profiling of the dopant level in the implanted layer has emerged as a promising technique towards insulator-semiconductor transition in polymers. In the present work, adopting various spectroscopic means, the 100 keV N^+ ion implanted polymer layer in poly(2,6-dimethyl-1,4-phenylene oxide) [PPO]as a function of ion fluence(over a range of $1x10^{15}$ - $5x10^{16}$ ions.cm^{-2}) is characterised with respect to chemical and electronic structures. Above a critical fluence of $1x10^{16}$ ions.cm-^{2}, the energy density transferred to the implanted layer resulted in a molecular reconstruction / self organisation process with evolution of graphite-like clusters of varying size ~2-50 nm. Electron diffraction analysis of these clusters showed the formation of a single crystalline graphite phase. The electronic structure of the fused hexagonally clustered aromatic rings with varying sizes, number of rings varying from ~2-170, was inferred from the optical band gap values. Evidence for fractal pattern formation with a dimension of 1.37 was obtained from TEM profiles. The fluence dependent conductivity in the polyphenylene reached a maximum value of ~10^{-2} S.cm^{-1} for a maximum fluence of $1x10^{17}$ ions.cm^{-2}. Charge transport from the temperature dependent conductivity values in a temperature range 90K to 450K is explained in terms of thermally activated band conduction and 3D variable range hopping.

Figure 1: Bright field transmission electron micrograph of the PPO film implanted at a fluence $5x10^{16}$ ions.cm^{-2}.
The crystalline microdiffraction pattern of the cluster region is shown in the outset.

INFLUENCE OF THE CRYSTALLOGRAFIC ORIENTATION ON THE MECHANISM OF ZINC DISSOLUTION IN ORGANIC SOLVENTS

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The effect of potential and ageing of two different plane of zinc monocrystal (0001) and (11-20) was investigated. The etching has the anisotropic character.

Anodic dissolution of zinc in anhydrous organic solvents proceeds in two oxidation steps similar to the dissolution of this metal in aqueous media .The stability of monovalent cations and than stability of surface anodic products is much higher in the organic solvents than in aqueous environments. Therefore the participation of Zn surface species in electrochemical reactions on zinc surface is more distinctive in organic media. Our investigations demonstrate that the anodic dissolution of zinc in methanol and acetonitrile solutions is strongly inhibited by the anodic intermediate product. At low overpotentials the catalytic mechanism dominates and the dissolution proceeds on active places of the metal surface (kinks and structural imperfections). In the presented work the influence of crystallographic orientation on the dissolution of zinc monocrystals has been investigated. The creation of Zn_{ad} proceeds faster on the high packed (0001) plane than on the plane (11-20). The desorption of intermediate is however more difficult for the plane with higher atom density and takes place at higher overpotentials.

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Surface Imaging with use of an Energy Filter

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Transmission electron microscopy is powerful for microanalysis of thin films in scanning mode of micro- or nano-beam if it is combined with various techniques such as electron energy loss spectroscopy, spectroscopy of emitted photon analysis from optical regions to X ray regions, energy analysis of emitted secondary electrons.

For surface studies, many works under a UHV specimen condition in reflection mode of observation as well as in transmission mode have been done. However, only few works have been done in combination with analytical techniques. Most of analytical studies have been done with use of conventional scanning electron microscope.

We have recently constructed an ultra-high vacuum electron microscope with an omega type energy filter (accelerating voltage of 200kV, field emission gun, vacuum at the specimen position of 10^{-9}Pa level, attachments of specimen preparation chambers and in-situ evaporators). The present paper shows results obtained by the microscope

Electron energy loss spectrum was obtained in reflection electron microscopy (REM) mode. In the REM mode surface plasmon excitation probability is very high. It was found clealy that image contrast due to elastically scattered electrons is conserved in images by electrons which excited surface plasmon once and twice. Surface plasmon excitation probability depending on diffraction conditions and diffracted beam was studied.

Energy filtered hologram can also be obtained with use of a biprism and coherent lengths of electrons which excited surface plasmon once and twice were measured.

Applications of the microscope to high resolution studies on high index Si surfaces are also presented.

COMPOSITIONAL FLUCTUATIONS AND LUMINESCENCE PROPERTIES OF InGaN QUANTUM WELLS GROWN ON GaN AND SAPPHIRE SUBSTRATES: **A COMPARISON**

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InGaN Quantum wells are the basis for light emitting diodes (LED) in the blue and green spectral region. One of the interesting effects in InGaN alloys with In concentrations x<0.1 are the strong exciton localisation and a strong band gap bowing. Localisation of carriers has been explained (i) by In concentration fluctuations in the alloy or (ii) to be an intrinsic effect due to hole localisation even in an homogeneous alloy. Up to now studies of optical properties and compositional fluctuations have been performed only in highly dislocated material grown on a foreign substrate (e.g. SiC, sapphire). Dislocations due to their strain field may essentially influence decomposition, concentration fluctuations and thus the optical properties of these structures.

We in this work analyse optical, structural and compositional properties of InGaN single and multiple quantum wells by transmission electron microscopy (TEM). The layers have been grown by metal organic chemical vapour deposition onto dislocation free GaN(0001) substrates. By quantitative analysis of high-resolution TEM- and EELS-data we show that compositional and thickness fluctuations occur dependent on growth conditions and In concentrations. In some of the samples three-dimensional growth can be observed. The sizes of islands are around 50 nm. Concentration fluctuations occur on the scale of 5 -10 nm and in the concentration range of 5%. Spectrally and spatially resolved CL in the TEM allows to directly correlate these fluctuations to optical properties. In a number of samples no compositional fluctuations can be revealed in the quantum wells within the limits of resolution. We discuss these results in the framework of the work proposed by Bellaiche and Zunger (Phys.Rev.B **57**, 4425 (1998)).

ELECTROCHEMICAL DEPOSITION OF SI(IV) COMPOUNDS ON THE METAL SURFACE FROM ANHYDROUS ORGANIC SOLVENTS

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Cathodic deposition of silicon compounds on metallic surfaces has been performed in anhydrous deaerated solution of lithium chloride in methanol [1]. The investigations of composition and morphology of the film were performed by means of SEM, PS, AFM and -ray techniques. The amorphous deposits obtained on the surface of platinum, copper and austenitic 18 10 stainless steel consist of Si(I) metoxy compounds [2].

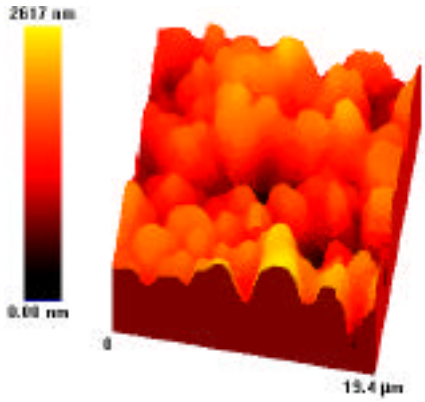

Figure 1. Layer of silicon compounds deposited on copper surface

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Cathodoluminescence submicrometric depth profiling of optical emissions in semiconducting heterostructures and devices

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In addition to high spectral and lateral resolution and monochromatic imaging, depth resolution is one of the advantages of the Cathodoluminescence (CL) technique in the SEM-CL mode. Depending on the sample characteristics, it is possible to achieve lateral and depth resolutions of about 200-300 nm and 100-200 nm respectively.
Here it will be shown how the low temperature spectrally resolved SEM-CL (LTSCL) can be employed to give a reliable insight on the mechanisms of failure analysis in GaAs and InP based HBTs and HEMTs and to determine dopant diffusion in InP/InGaAsP based MQW Fabry-Perot lasers. Further the influence of accelerating voltage and irradiaton time on the in-depth yellow emission in hexagonal GaN epilayers will be also briefly discussed.
As for AlGaAs/GaAs based Be-doped HBTs, CL spectroscopy has been used to detect the base-dopant outdiffusion induced by current stress. CL measurements were carried out on at a temperature of 5K and in the energy range between 1.44 eV and 1.55 eV before and after a bias aging stress. Depth resolved CL investigations were performed at various electron beam accelerating voltages in the range between 14 kV and 10 kV. CL spectra collected at Eo =10 keV presented a main peak is at 1.504 eV with a shoulder at 1.493 eV. The CL spectra for Eo=14 keV exhibit a main peak at 1.493 eV and a shoulder at 1.504eV. The CL measurements after the stress revealed that the main effect of the stress is to increase the CL integrated intensity for all the spectra, independent on the accelerating voltage range. This suggested that the mechanism of recombination-enhanced impurity diffusion iswas responsible for the dopant outdiffusion. The Be diffusion found by CL studies was confirmed by SIMS analyses.

Concerrning InP based HEMTs, a clear reduction of the CL emission collected from the gate-drain region of stressed devices has been found, indicating a modification of the trap density inside the device.
Depth resolved analyses from the gate-drain region before and after stress evidenced that the electric field due to the traps induced by the hot-electron-stress mainly influenced a device region between the highly doped InAlAs and InGaAs cap layers and the n+ doped donor and undoped spacer_ InAlAs layers. The effect of the hot electron stress has been evidenced mainly on the catodoluminescence transitions from the InAlAs layers. No evidence of a possible influence on the intrinsic InGaAs channel has been found.

The strain field distribution around misfit dislocations (MDs) in MQW based InGaAs/GaAs solar cells has been also studied. The CL spectra presented a main peak at 917 nm (MQW) and a satellite one at 930 nm. Monochromatic CL micrographs at 930 nm showed luminescence emitted only by a narrow region around MDs, the effect of non-radiative recombination being a weak dark contrast in the centre of the bright region. The localised emission was due to the impurity atmosphere surrounding the dislocation core.
Monochromatic CL intensity profiles across MDs were analysed after eliminating spurious effects due to the non uniform light collection efficiency along the scan line. This was achieved by normalising the profiles acquired at different wavelengths to a reference one. At 930 nm (maximum of the satellite peak) the profile was a gaussian centred at the dislocation core; everywhere else the CL emission was zero. Moving toward the MQW peak the background signal increased while the satellite peak decreased (921 nm) until a new maximum appeared at about 1 micrometer to the left of the original one (918.2 nm). Going beyond the MQW peak, at 916.2 nm, a roughly specular profile was obtained with a maximum at about 1 μm to the right. This was explained by considering the calculated strain field distribution generated by the dislocation line.

Concerning GaN epilayers, long time dependent cathodoluminescence investigations have shown different kinetics among the near-band-edge, yellow, blue and donor-acceptor-pair transitions in the range of thousands of seconds. The results are discussed on the basis of variations of the point defects concentration induced by the electron beam irradiation, as a function of the beam current and accelerating voltage. Finally, depth resolved CL has also shown an anticorrelation between the emissions underneath the broad yellow band centered at 2.2 eV.

MICROCHARACTERIZATION OF CONFORMAL GaAs AND AlGaAs ON Si LAYERS BY SPATIALLY RESOLVED OPTICAL TECHNIQUES

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The growth of III- layers on Si substrates has the interest to combine the high performance of these semiconductors with the mature technology of Silicon. However, the large mismatch and the difference in the thermal expansion coefficients prevent the obtention of layers with low enough defect concentration for device applications.

Recently, a growth technique, the so-called conformal growth, allows the obtention of nearly defect free GaAs and AlGaAs layers on Si substrates. This method is basically a confined lateral growth. Layers of submicrometric thickness with very low defect concentration are obtained.

We present herein a study of some GaAs and AlGaAs conformal layers on Si. The layers are characterized by spatially resolved techniques as PhotoLuminescence Imaging (PLI), Cathodoluminescence (CL) and microRaman. Different properties of these layers are studied. In particular, the stress distribution, the alloy composition or the free electron or hole density in doped layers are determined. Some others aspects as the influence of the seed orientation, the existence of nucleations and the crystalline order are also discussed.

Low-energy Cathodoluminescence Study of Semiconductor Nanostructures and Nanoparticles

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We have developed a cathodoluminescence (CL) system with high spatial resolution (about 50 nm) operating under low electron beam energies like 1 ke . A scanning elecreon microscopy with a field emission gun was customized for this purpose. We have applied this system for the characterization of semiconductor nanostructures, such as InGaAs GaAs quantum dots and ZnO nanopowders. These observations indicate that nano-charactrization is indispensable for the development of semicondiucting materials and devices.

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