Nanobeams for Nanoelectronic Devices; the Importance of ERL for Characterization of the Optoelectronic Device Structures

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ERL X-ray Science Workshop 6: "Workshop on New Science Opportunities with Nanometer-Sized ERL X-Ray Beams"







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Outline

Introduction Nanoscale Selective Area Epitaxy Advantages of monolithic integration Nanoscale Growth and Nano-hetero-integration X-ray Diffraction with submicron spatial resolution Experiments at CHESS and APS - Challenges to maximize $N_{ph}/(A^{-}\Delta th)$ Future experiments with ERL nanobeams HRXRD for optoelectronics RSM + real space mapping; XSW - Nanotechnology requirements for $N_{nh}/(A \Delta th)$

ERL and High Angular Resolution XRD technique

Nanometer-Sized ERL X-Ray Beams





Focusing Devices

The Nobel Prize in Physics, Chemistry, ...

nology

Figure of merit: N_{ph}/A



Nano science and technology



Semiconductor Planar technology



Nano-optoelectronics

High density integration in silicon technology





1947

Simulated HRXRD diffraction intensity for a 10 nm thick layer of Si_{0.8}Ge_{0.2} on Si



Future Electro-Optic Chips

No place for III-V optoelectronic components?



Limitations of high density integration in silicon technology

FEATURE	LIMIT	REASON
Oxide Thickness	2.3 nm	Leakage (I _{GATE})
Junction Depth	30 nm	Resistance (R _{SDE})
Channel Doping	V _T =0.25 V	Leakage (I _{OFF})
SDE Under Diffusion	15 nm	Resistance (R _{INV})
Channel Length	0.06µm	Leakage (I _{OFF})
Gate Length	0.10µm	Leakage (I _{OFF})



Where is optoelectronic integration on this scale?



Note that active optoelectronic devices should have dimensions close to the wavelength of light

Junction

Issues with integration of Optoelectronic circuits

While electronics manufacturing has evolved from physical assembly of discrete components to high volume manufacture of multi-milliontransistor devices, optical devices are generally still assembled from discrete components, a comparatively slow, laborious, costly, and lowyield process. 90% of the cost is in packaging of individual telecom components

The two approaches currently used for optical component Hybrid Integration and Monolithic Integration; both display distinct limitation

Advanced Monolithic Integration Architecture : Example

RZ, Tandem EA Modulator



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New substrates and integration with Si III-V Semiconductor Optoelectronics:

Strain $\Delta a/a$

53E1

Si World:

 $Si + SiO_2 + Ge$

GaAs: GaAlAs InP: InGaAs, InGaAsP, InGaAlAs

GaN: B, Sb, In, Al Alerrative Substrates sapphire, SiC, Si, LiNbO₃, ...

Si or SOI platform

Nano-Hetero-Epitaxy (NHE)

Motivation: Recent results show that small diameters of the heterostructures and the absence of lateral confinement strongly enhance lateral relaxation and allow lattice and thermal expansion mismatched materials to be combined without propagation dislocations

Applications: Lattice mismatched hetero-integration of highly integrated systems combining superior proprieties of Si-based electronic and III-V based Photonic devices

Collaborations: NJIT, CHESS, Georgia Tech, Lucent, SUPELEC, Uni Metz, France



NHE (3D strain relief)





(a) Conventional Heteroepitaxy

Strain at growth interface remains constant and strain energy grows linearly with epilayer thickness. Dislocations eventually created.

(b) Stress relief in Nanosize Nucleus

3-D strain in nanosize nucleus gives exponential stress/strain decay (Luryi and Suhir, 1986). Decay length is proportional to (and of similar magnitude to) island diameter. Strain energy saturates at a maximum value.





Subwavelength Optical Elements (SOEs)

Taking integrated optical-component design to the **next level of density**, cost, and reliability will require new optical **« building blocks »** with:

 Broad range of optical functionality
Easily integration with other optical/electronic materials





SOEs are the realization of nano-technology applied to optical elements. Through manipulation of the size, shape, type and the period of nanostructures SOEs deliver a broad range of useful optical effect and functionality.

SUMMARY for the INTRODUCTION

 NHE is one of the key technologies for Monolithic Integration of Optical Devices

 Progress in NHE depends on Submicron and nano characterizations

MOVPE: Nanoscale Selective Growth



Selective Growth on Nanoscale (Simplified Picture)

Lateral gas phase diffusion D/k ~ $30 \div 200 \ \mu m$

Vertical gas phase diffusion



Characterization of the Nanoscale Selectively Grown structures



Sufficient surface migration



Selective Growth on Nanoscale Insufficient surface migration



Oxide Mask

Oxide Mask

Substrate

Analytical Characterization

HRXRD RSM XSW Composition change Strain △d/d Relaxation Effects Sidewall and interface quality Impurity incorporation and activation







High angular resolution but low spatial resolution



High spatial resolution but low angular resolution





the collimator / analyzer crystal: e.g., Si(004) – 2 arc sec at 12 keV







High resolution optics for microbeam XRD



High resolution optics for microbeam XRD



X-ray optics for microbeam RSM



Takes ~ 15 hours to measure this MapMax intensity:50,000 cpsSatellites:300 cps

MQW peaks









Experimental setup for the microbeam high resolution diffraction and RSM measurements at CHESS based on one-bounce imaging capillary with the working distance of **30 mm** and the beam size at the focal spot of **10 \mum**. Ge (1 1 1) channel cut crystal is embedded between the capillary optics and the investigated structures providing higher angular resolution of **13 arcsec**. Single-bounce Ge crystal analyzer provides high angular resolution in the detection arm. Wavelength: **0.1 nm**.



beam size: 0.35 μ m (vert.) x 0.24 μ m (hor.) analyzer: Si(004) perfect crystal angular resolution is determined by Si(004) rocking curve: 2.2 arc sec $\Delta\theta/\Delta 2\theta$ scans: $\Delta 2\theta = 2 \Delta \theta$ flux $\approx 7.10^6$ ph/sec

Samples: multiple quantum well waveguides grown by MOCVD selective area growth technique



InGaAsP identical masks

TABLE I. Parameters of the investigated samples.

	$In_xGa_yAl_{1-x-y}As$		$In_xGa_{1-x}As_yP_{1-y}$	
	Open wafer	Ridge	Open wafer	Ridge
Well width (nm)	5.0	6.5	7.0	8.9
Barrier width (nm)	10.3	12.9	9.9	13.2
Global strain (%)	0.22	1.15	0.49	0.75
Well composition	x = 0.65		x = 0.85	
	y = 0.19		y = 0.57	
Barrier composition	x = 0.48		x = 0.87	
	y=0.19		y=0.29	



goal:

determine MQW composition and thickness variations:1) across the ridge2) as a function of ridge width (mask geometry)

Comparison of P-based and Al- based structures





conclusion:

Al-based SAG MQW is superior in uniformity compared to traditional P-based devices

Conclusions for submicrobeam studies of InP-based SAG

 \Box microbeam diffraction with arc sec angular resolution and lateral 0.24 μ m resolution has been demonstrated by using a combination of phase zone plate and perfect analyzer crystal

post-focusing perfect crystal optics is an efficient way to condition microbeam for a variety of high angular resolution microbeam diffraction applications

this setup has been applied to study compositional and thickness variations in multiple quantum well waveguides grown by MOCVD in selective area growth regime

Al-based SAG MQW is much more superior in uniformity compared to traditional P-based devices which is promising for development of one-growth-step SAG technology

MQW 0th peak





GaN/InGaN MQW SAG structures

SEM Image





FIG. 3. SEM photograph of a stripe in $\langle 1\bar{1}00 \rangle$ direction with trapezoidal cross section. The Mg-doped GaN cover layer results in a brighter SEM contrast. The five QWs are resolved only on the top facet.

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GaInN quantum wells grown on facets of selectively grown GaN stripes

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Positioning of the SAG structures in the X-ray beam





40 µm

HRXRD in InGaN/GaN MQW SAG structures



1: 2µm SiO, mask, 6µm Opening, field width 400µm 2: 4 3: 6µm SiO, mask, 6µm Opening, field width 600µm 4: 6 5: 10µm SiO, mask, 6µm Opening, field width 800µm 6: 7: 14µm SiO, mask, 6µm Opening, field width 1000µm 8 ______ 9: 20µm SiO, mask, 6µm Opening, field width 1300µm 0 2

4m SiO, mask, 6μm Opening, field width 500μm 8 m SiO, mask, 6μm Opening, field width 700μm 9 μm SiO, mask, 6μm Opening, field width 900μm 8 16μm SiO, mask, 6μm Opening, field width 1100μm



Simulations using RadsMercury BEDE dynamic diffraction model

	d	Low	High	Material	x	Low	High
17	1524	1000	5000	Ga(x)In(1-x)N [hexagonal] ■	0.9965	0.985	1
16	1269.36	1000	5000	Ga(x)In(1-x)N [hexagonal] ▼	0.9986	0.985	1
15	3672.72	1000	5000	Ga(x)In(1-x)N [hexagonal] ▼	0.9980	0.985	1
14	2708.83	1000	5000	Ga(x)In(1-x)N [hexagonal] ▼	0.9897	0.985	1
13	2270.44	1000	5000	Ga(x)In(1-x)N [hexagonal] ▼	1.0000	1	1
12	229.83	100	300	Al(x)Ga(1-x)N [hexagonal] ■	0.2228	0.21	0.23
11	125	125	125	GaN [hexagonal] 🛛 🛛 🔻	0.0000	0	0
10	80.41	77	82	Ga(x)in(1-x)N [hexagonal] 🛛 🔻	0.8723	0.85	0.89
1	121.17	119	125	GaN [hexagonal]	0.0000	0	0
Sub.	00	00	00	GaN [hexagonal] 🛛 🔍 🔻	0	0	0



FIG. 3. SEM photograph of a stripe in $\langle 1 \overline{1} 0 0 \rangle$ direction with trapezoidal cross section. The Mg-doped GaN cover layer results in a brighter SEM contrast. The five QWs are resolved only on the top facet.



Simulations using RadsMercury BEDE dynamic diffraction model





Parameters of InGaN/GaN MQW structures



Reciprocal Space Map of GaN @CHESS

RSM 0002, GaN/InGaN MQW, Sam1, 6um SAG - 4um Mask, 10 um x-ray beam, S591



Total time for these measurements: 15 hours

SAG: 6 μm Mask: 4 μm

MQW: Period 20 nm, Strain: 0.9 %



Total time for this project: 7 days

Conclusions for microbeam studies of GaN-based SAG structures



 Micro-beam HRXRD setup developed for RSM studies of nitride-based MQW's

- Accurately measured global strain and the thickness in the SAG structures
- Well/barrier thicknesses are determined from the fit using RadsMercury simulation software
- Mosaic structure in MQW is the same as that for the GaN substrate layer

Nondestructive approach to investigate Zn incorporation in InP using X-ray Standing Wave Technique (XSW)

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$Zn^{(i)+} + V_{In}^{-} \rightarrow Zn^{(s)-} + 2h$



Nondestructive XSW "Ångstrøm ruler"

interstitial diffusion



XSW measurements at A2 beamline at CHESS



XSW for 0 0 4 reflection; range: 0.02°; 81 points



fit is using the dynamical diffraction theory in layered crystal structures.

Effect of RTA on Zn incorporation XSW for 0 0 4 reflections



Summary for XSW part

 Using a combination of XSW technique, SIMS, and "CV" we have studied Zn incorporation and activation in InP epilayers

Plans for the Future:

- Zn activation in device structures grown selectively on patterned wafers with sub μm spatial resolution
- Other impurities: Mg in GaN



Summary

• We combined focusing optics and post-focusing optics for high-resolution XRD studies of SAG structures.

 Using micro-beam X-ray diffraction we analyzed strain and composition and thickness enhancement in a variety of MQW SAG structures based on InGaAsP, InGaAlAs, and InGaN/GaN

<u>Plans for the Future:</u> Nitride-based (InGaN, GaBN) and Si-Ge NSAG structures: growth and characterization

HIGH ANGULAR RESOLUTION BEAMLINE @ ERL

Figure of merit: $N_{ph}/(A:\Delta th)$

APS: 7×106 photons/(240 nm \cdot 350nm \cdot 2arcsec) \approx 50 photons/(nm²·arcsec)

Need: 50 photons/(nm2·arcsec) For RSM's + Real Space mapping and for XSW's

(a) Schematic of two coherent beam interference and (b) SEM micrograph of photoresist patterns produced by the technique of interferrometric lithography. The patterns are formed on a 100 nm-thick layer of thermally grown SiO2 on Si.

Cross-sectional schematic of MOVPE growth of nitrides on Si nanopillars for different mechanisms of the precursor surface migration: conventional (a) NSAG mask, (b) Si nanopillar with a free-standing sidewalls SOI mask. and (C)Deposition of GaN on the top of the SOI mask is prevented by using in situ chloride-assisted growth.

ERL nanobeams and NSAG structures

Nanometer-Sized ERL X-Ray Beams

Main Linac Kriays Krisecor Lipector

Nano science and technology

Focusing Devices

NSAG structures for area detectors based on X-rays → VIS light converters

XSW: basic principles

X-ray Standing Waves

The X-ray standing wave (XSW) technique, inherently an interferometric method, employs the interference field, which is produced by the superposition of an incoming plane and coherently scattered waves, as a probe to measure distances similar to an Å ngstrøm ruler. The technique which only became routinely applicable with highly collimated synchrotron sources will immediately benefit from increases in brilliance: Much smaller samples as well as thin epitaxial layers can be used so that exotic materials can also be investigated; weakly modulated rather than strongly modulated wavefields can be used which widens the application range of the XSW technique tremendously, highly dilute systems can be studied and very short measuring times become possible. Since a single, 100 fs pulse will contain photons it will even be possible to enter the fs time range.

- The standing wave field is generated by the coherent superposition of the incident and diffracted plane waves. In a selected Bragg reflection geometry the wavelength of the incoming X-rays has to match the distance of the diffraction planes of the substrate.
- As a consequence of inelastic scattering processes within the adlayer photoelectrons as well as Auger electrons are produced. Their intensity variation when scanning through the Bragg condition by changing the photon energy is characteristic for the adsorbate site.

XSW: basic principles

X-ray Standing Waves / Surface Studies.

An XSW measurement of one Fourier component of a monolayer of an adsorbate distribution function takes about 500s (for a strong Bragg reflection) at a second generation SR source with about incident photons. Choosing different reflections usually requires changing the monochromator to realize a non-dispersive crystal setting which is fairly time consuming, since it is crucial to keep the surface clean and stable during the measurements. A thorough analysis of a surface structure by XSW would preferably involve a sizeable number of measurements employing a set of different reflections (H-values) to determine a sufficient set of Fourier components of the system. XSW measurements at third generation sources are just starting. Given the right detector for the photoelectric scattering, measuring times could be cut by a factor of and a large set of H-values could be scanned with a single monochromator (vertical divergence of the source). Already on 3rd generation SR sources the traditional mode of performing XSW measurements will often be abandonned. A single scan of the sample reflection curve can provide enough signal for the XSW analysis within seconds or less. With the divergence of the XFEL is sufficient without additional collimation for practically all XSW measurements (for comparison, FWHM of Si(220) at is about). From this aspect further collimation by a crystal arrangement would hardly be needed. The energy spread of the XFEL which is in the range of would, however, make a monochromator still necessary for many applications. For total reflection XSW or weakly modulated wavefields, which will be discussed below, bandwidth would be sufficient. Measuring times could be reduced by another factor of about , i.e. a monolayer could be measured in about . Piezoelectric devices driven at resonance should make energy scans with a monochromator at a comparable speed possible. These kinds of measuring times fall well into the short time domain which will be discussed later.

XSW: basic principles

X-ray Standing Waves / Extremely Dilute Systems.

Just as the measuring times scale with the brilliance of the sources it scales with the dilution of the system under study. Consequently, extremely dilute systems could be studied with the XFEL. This is a very interesting perspective in the study of impurity diffusion in and on solids or adsorption sites on surfaces in the limit of vanishing adsorbate-adsorbate interactions. A typical question which remains to be answered is whether there is a dominant ionic bond with a different bonding geometry in the adsorption of alkali metals at coverages below 1/100 ML. Diffusion paths of a non-interacting, 2D lattice gas, i. e. a very dilute adsorbate at elevated temperature, could also be analyzed. These are just two examples of systems which cannot be analyzed by diffraction techniques. Just scaling intensities, 1 ppm of a ML (i.e. atoms/)could be analyzed in much less than a second, provided however, given a detection system which can discriminate sufficiently against background signals. Typical ways to enhance surface signals are grazing incidence, which is only applicable in exceptional cases for XSW, and grazing exit fluorescence detection either of which is obtained at the cost of large loss in signal. The latter scheme is possible for 3rd generation sources and perfectly feasible for an X-ray FEL.

The XFEL will deliver between and photons per bunch with a duration of about 100 fs. In principle these are enough photons for a complete XSW measurement - about incident photons are needed for an XSW measurement for one H-value. The proposed bunch spacings are 3 ns (SBLC) and 93 ns (TESLA) and thus offer the possibility of XSW experiments in the fs time domain. The adsorbate would first be sampled by the XSW. A pump pulse from a fast laser in the eV range would then hit the adsorbate covered surface leading to excitation and desorption (in the range if the scheme is to work). Sufficiently fast and powerful pumps are available [23]. The probe, the XSW, is switched on by the XFEL pulse with a desired time delay between 0 and . The total spatial distribution of atoms sampled by the XSW consisting of adsorbed, excited adsorbed and desorbing atoms will thus change with time. Length scales of up to , the interference field spacing are accessible at time scales >100 *fs*. Note that can be varied between fractions of an Å (for Bragg reflection from single crystals) and several hundred Å (for total reflection). To overcome statistical noise (The FEL pulses will also display some intensity fluctuations), a signal averaging technique will have to be used. Several sample-pump-probe cycles have to be run for the same location of the wavefield and this in turn has to be done for several locations of the wavefield.

On a longer time scale (s-ms) the bunches of one bunch train could be used to stroboscopically sample a changing distribution. This could be employed to study e.g. adsorption desorption processes at a solid-electrolyte interface following a step-like change of the applied potential.

Project: Zn diffusion in InP Mechanism of Zn diffusion:

interstitial Zn diffuses till it is trapped by In vacancy.

The ease with which an impurity can diffuse through the lattice is reflected in the value of E_A ; E_A for vacancy diffusion is in the range 3 - 5 eV, while E_A for interstitial diffusion is between 0.5 - 1.5 eV. Impurities which diffuse through a semiconductor via vacancies diffuse more slowly (slow diffusant) than impurities which diffuse interstitially (fast diffusant).