

Course name:
Growth and Characterization of Nano-electronic Materials (EE728)

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Metrology: Semiconductor Materials and Devices



Outline

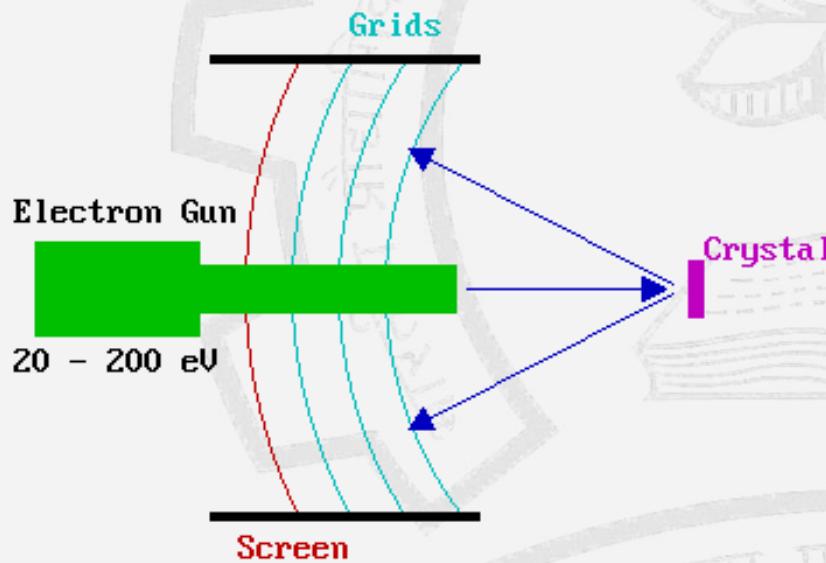
- In-situ and Ex-situ diagnostic tools
- Low Energy Electron Diffraction (LEED)
- Reflection High Energy Electron Diffraction(RHEED)
- Ellipsometry
- X-Ray Diffraction (XRD)
- X-Ray Photoelectron Spectroscopy (XPS)
- Ultra-violet Photoelectron Spectroscopy (UPS)
- Atomic Force Microscopy (AFM)
- Scanning Electron Microscopy (SEM)
- Transmission Electron Microscopy(TEM)

In-situ and Ex-situ diagnostic tools

In situ: A Latin phrase that translates literally to 'In position'.
It is used in many different contexts

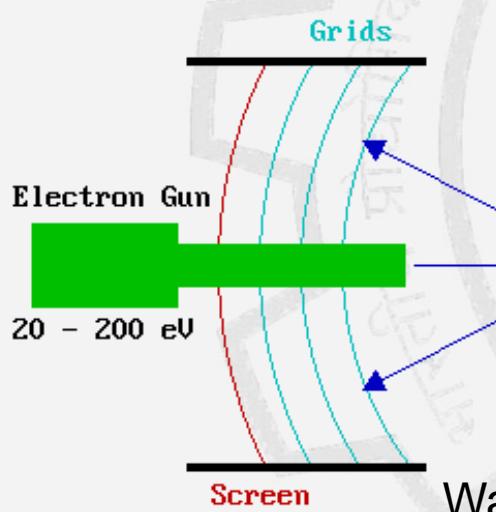
In-situ diagnostic tools

Low Energy Electron Diffraction (LEED)



LEED

A beam of electrons of a well-defined low energy (typically in the range 20 - 200 eV) is incident normally on the sample. The diffracted electrons can be observed by a fluorescent screen after energy-filtering grids, which selects only the electrons with the same kinetic energy as the primary electrons.



$$p = m.v = (2mE_k)^{1/2} = (2m.e.V)^{1/2}$$

Where

m - mass of electron [kg]

v - velocity [m s⁻¹]

E_k - kinetic energy

e - electronic charge

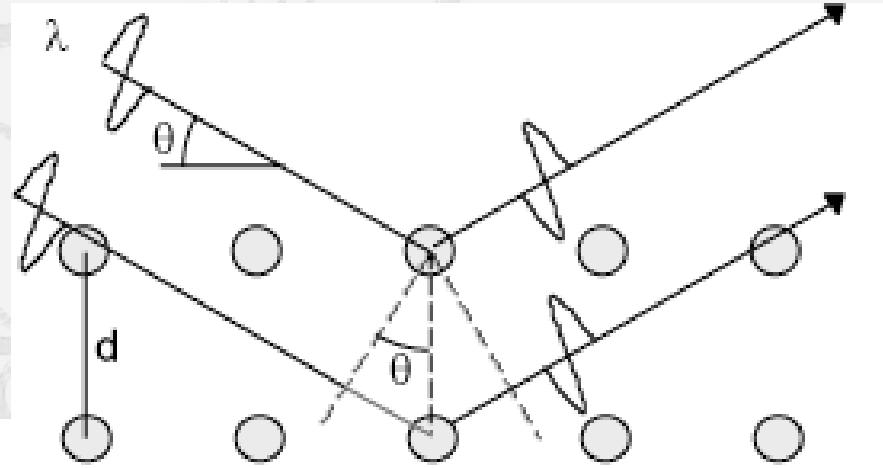
V - acceleration voltage (= energy in eV)

Wavelength, $\lambda = h/(2m.e.V)^{1/2}$ for 20 eV electrons, λ is 2.7 Å

Remember that this is order of the crystal lattice parameter

Real space consideration

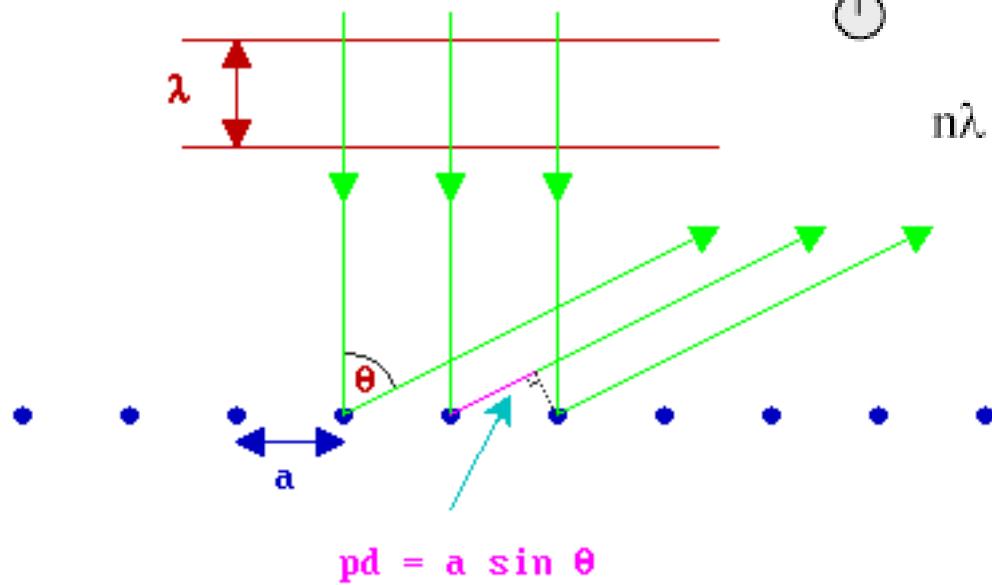
For 3D case



$$n\lambda = 2d \sin \theta$$

Bragg Equation

For 2D case



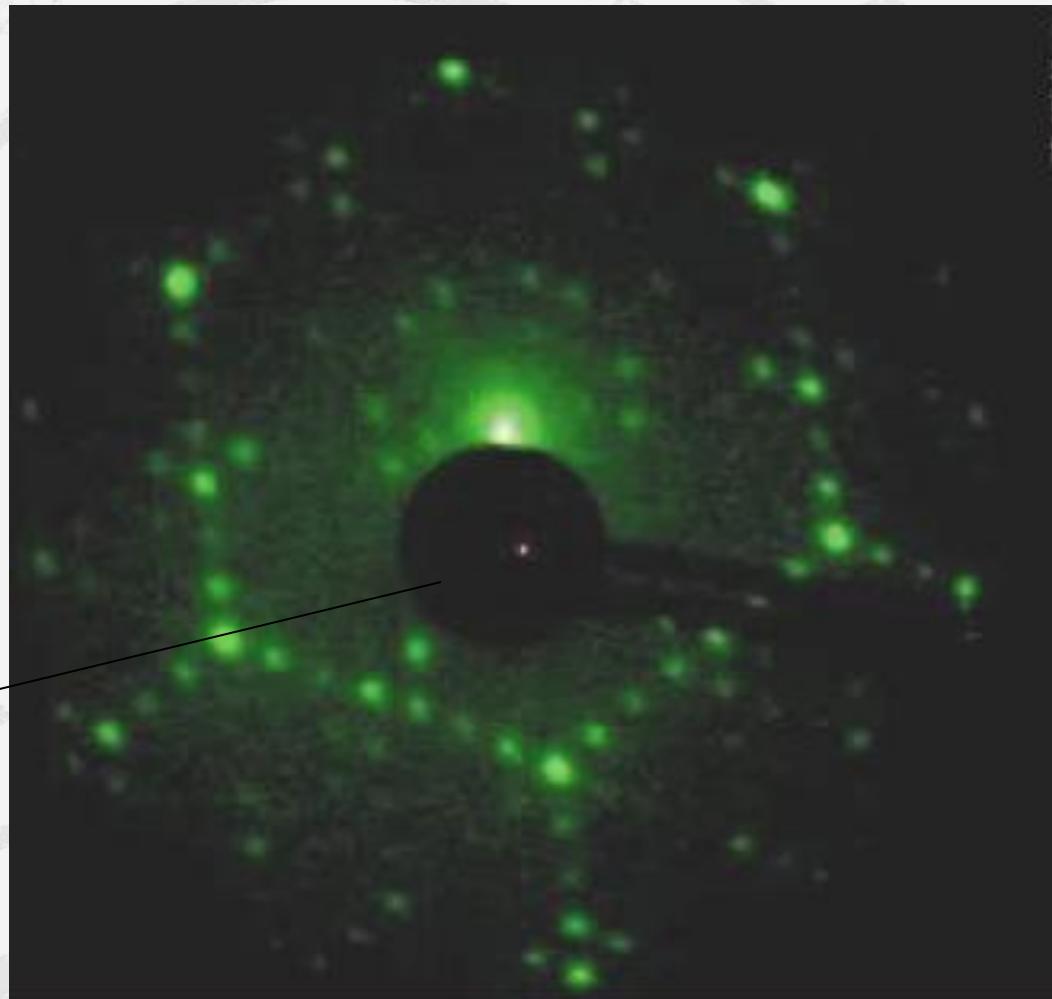
Normal LEED case with
incident e beam normal to the
surface, along one direction

$$\underline{A \sin \theta = n\lambda.}$$

Experimental data

LEED pattern
obtained from
 $\text{Si}(111)7\times7$
reconstructed
surface

Electron gun



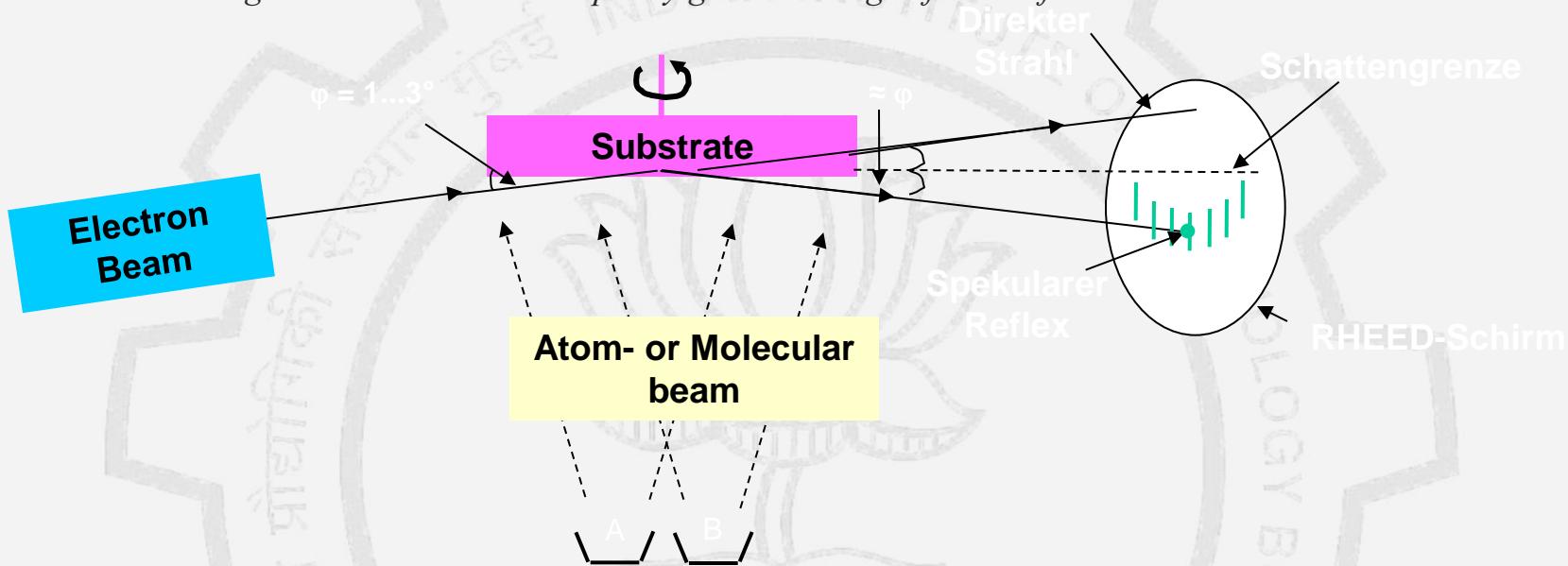
In situ Epitaxial growth control

in situ as the film grows

RHEED: Reflection High-Energy Electron Diffraction

In situ Epitaxial growth control

done along with molecular beam epitaxy gives coverage of the surface



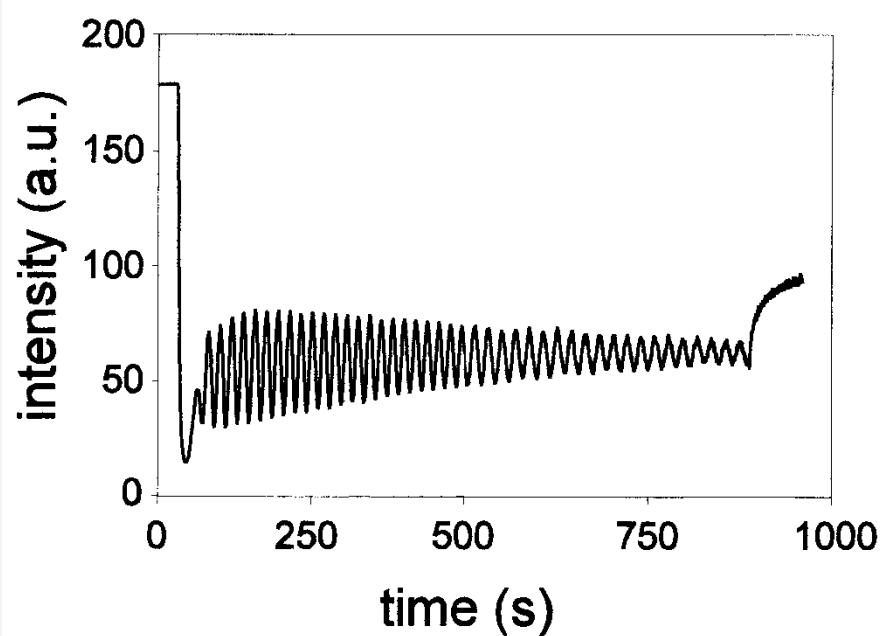
RHEED: Reflection High-Energy Electron Diffraction

- Diffraction of electrons with energies of 10 ... 40 keV at grazing incidence ($\theta = 1 \dots 3^\circ$). At the growing surface of the crystal
Information Depth: 1-5 monolayers
Control of surface coverage or reconstruction

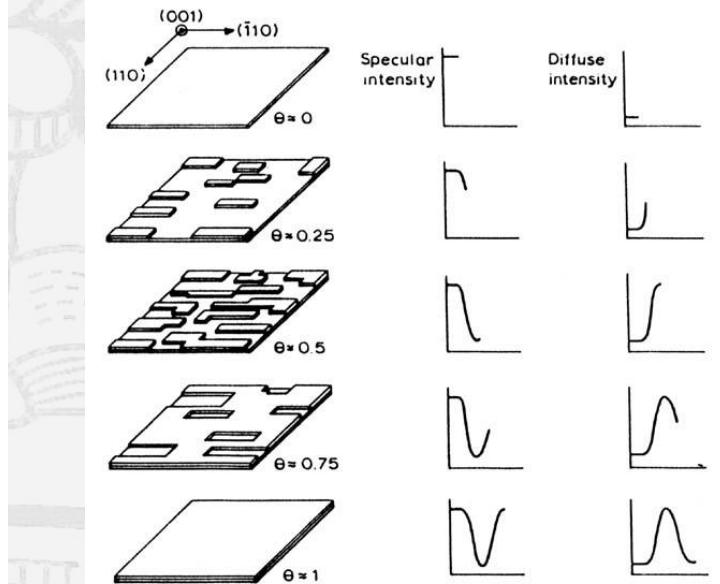
RHEED-Oscillation

Measuring Intensity of Spelcular Reflected Beam from Grown Layer.

MBE of Si on Si(111)



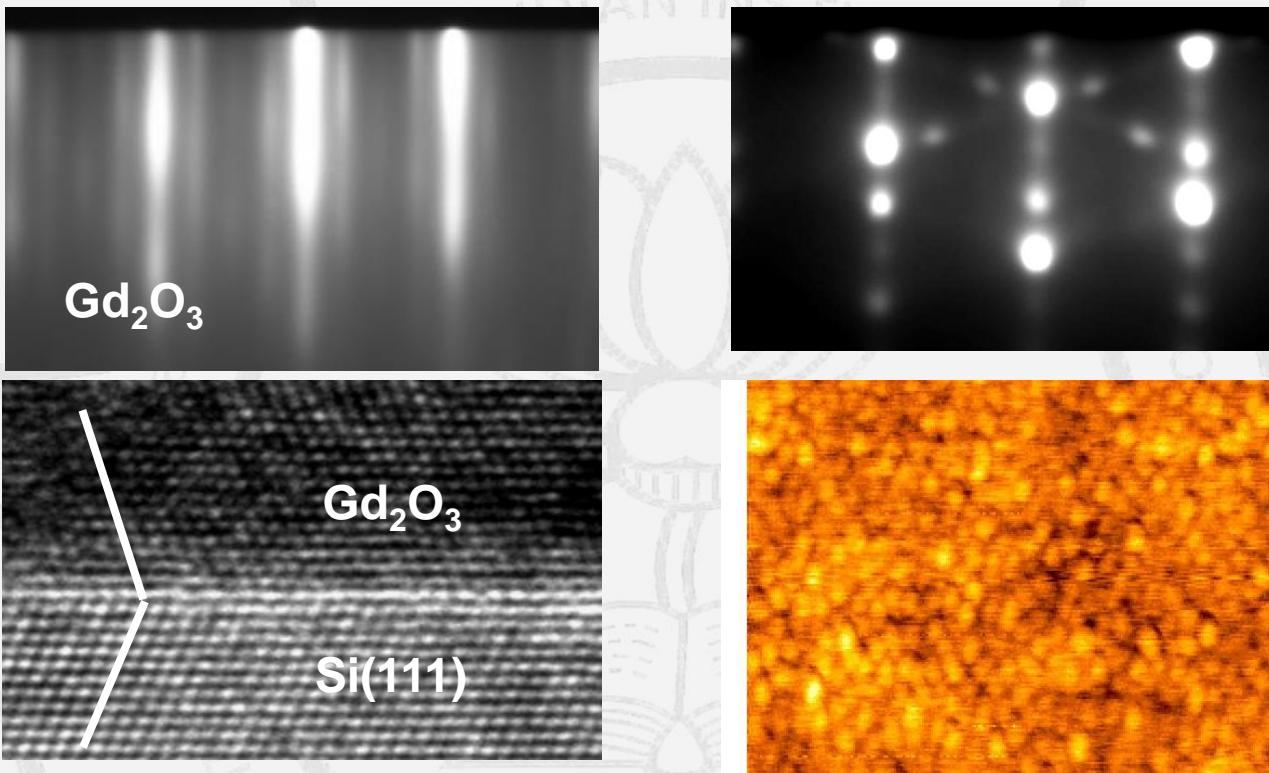
Model of the formation of RHEED oscillations



flat surface will result in no constructive and destructive interference but as the mbe starts the surface will slowly develop so it will be non uniform at the middle which will result in

J.H. Neave, B.A. Joyce, P.J. Dobson, N. Norton,
Appl. Phys. A 31 (1983) 1

RHEED Example



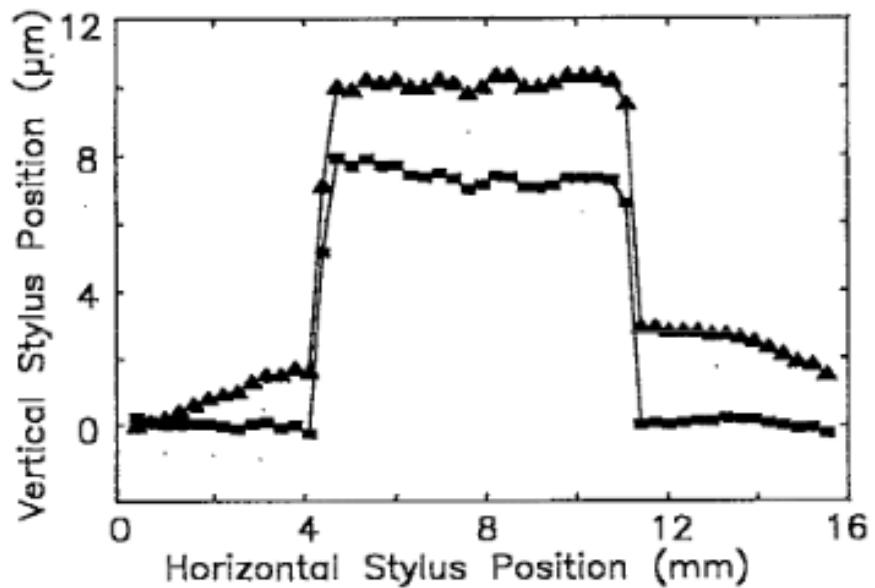
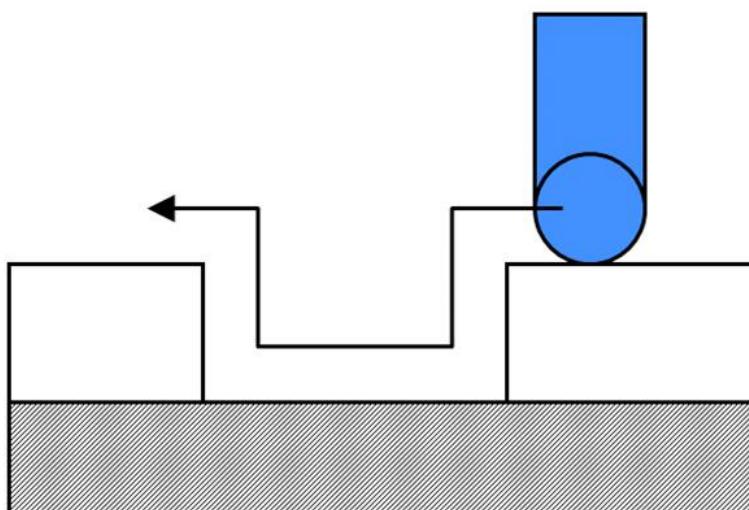
→ Single-crystalline Gd_2O_3
growth of the cubic phase

→ Single-crystalline Si
Nanocrystals on single
crystalline Gd_2O_3

Ex-Situ Diagnostic Tools

Profilometer for film thickness measurement

With the profile method, a surface is sampled with a fine diamond tip along a line, recording a height profile.

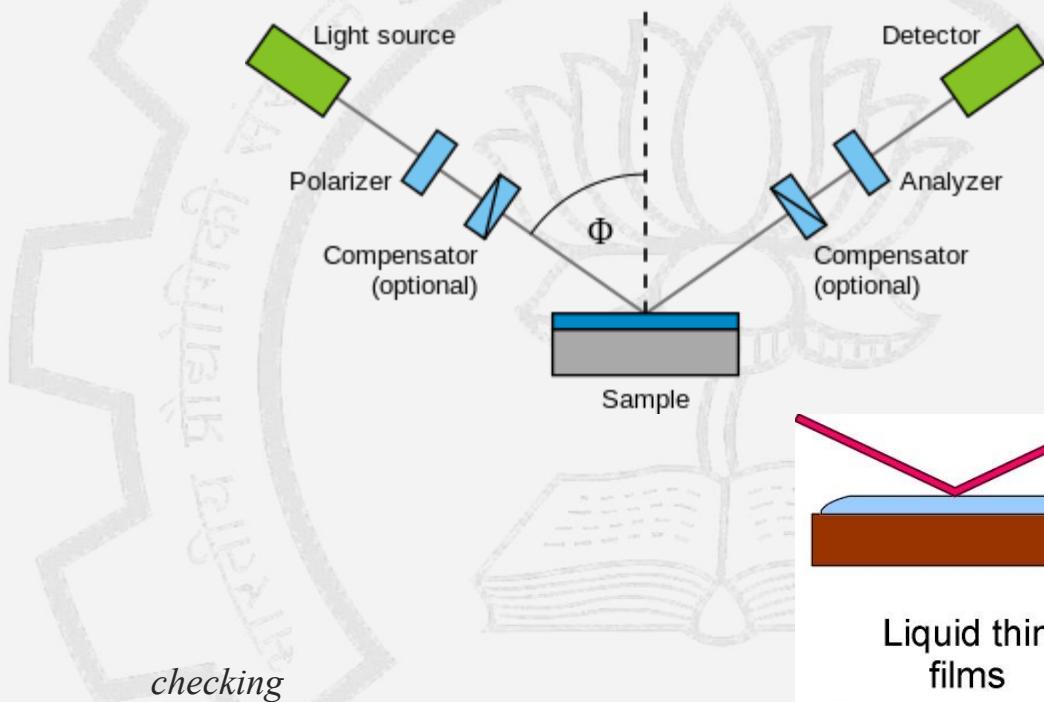


Example: Profilometer

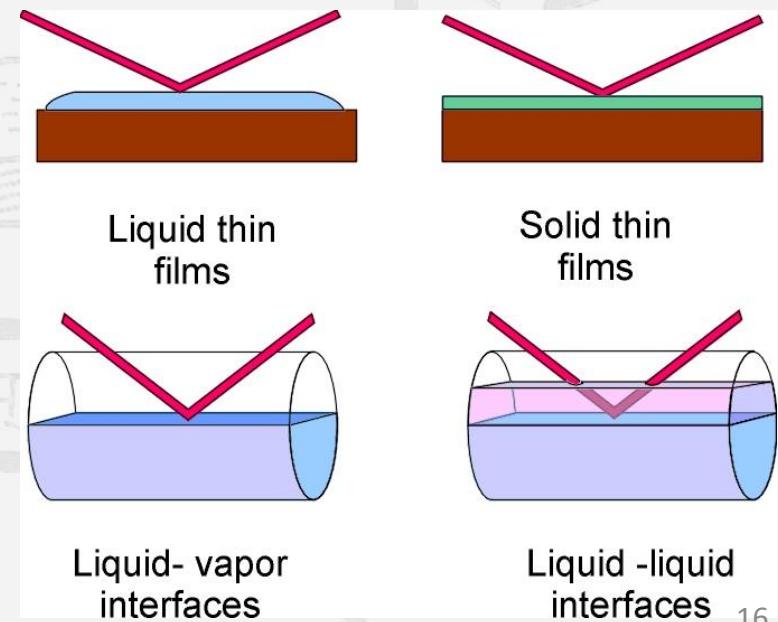
PROFILOMETERS	Dektak XT (Bruker)	Talysurf (Taylor Hobson)
Vertical resolution	0.5 nm	15nm
Lateral resolution	~ 0.2 and 1.25 µm	0.25 µm
Vertical measuring range	1 mm	1mm
Tip radius	2 and 12,5µm	2.5µm
Load pressure	1 – 15 mg	~70mg
Measurement length	55 mm (+3D scan)	1 – 120 mm (+3D scan)

- Several surface morphology parameters can be determined from the height profile, such as roughness, waviness, the height histogram, skewness and slope of the height distribution, mean local surface pitch, contact ratios and so on

Ellipsometry: Ellipsometric Analysis of Thin Films



A method of probing surfaces
with light.



History

- Fresnel derived his equations which determine the Reflection/Transmission coefficients in early 19th century. Ellipsometry used soon thereafter.
- Ellipsometry became important in 1960's with the advent of smaller computers.

The reflectance for s-polarized light is

$$R_s = \left| \frac{Z_2 \cos \theta_i - Z_1 \cos \theta_t}{Z_2 \cos \theta_i + Z_1 \cos \theta_t} \right|^2,$$

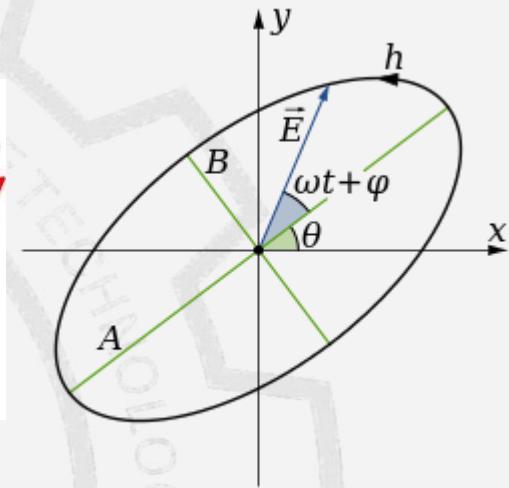
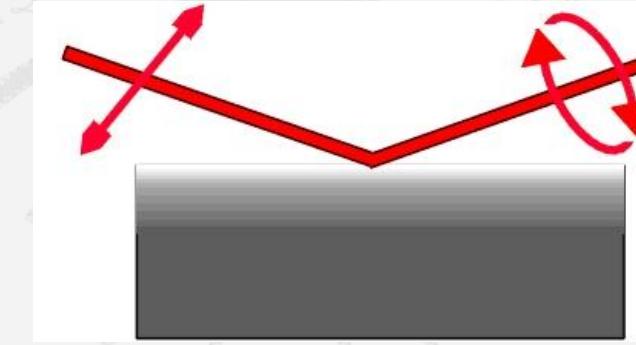
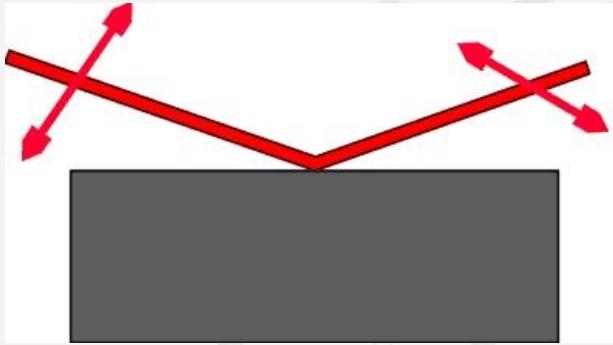
while the reflectance for p-polarized light is

$$R_p = \left| \frac{Z_2 \cos \theta_t - Z_1 \cos \theta_i}{Z_2 \cos \theta_t + Z_1 \cos \theta_i} \right|^2,$$

where Z_1 and Z_2 are the wave impedances of media 1 and 2, respectively.

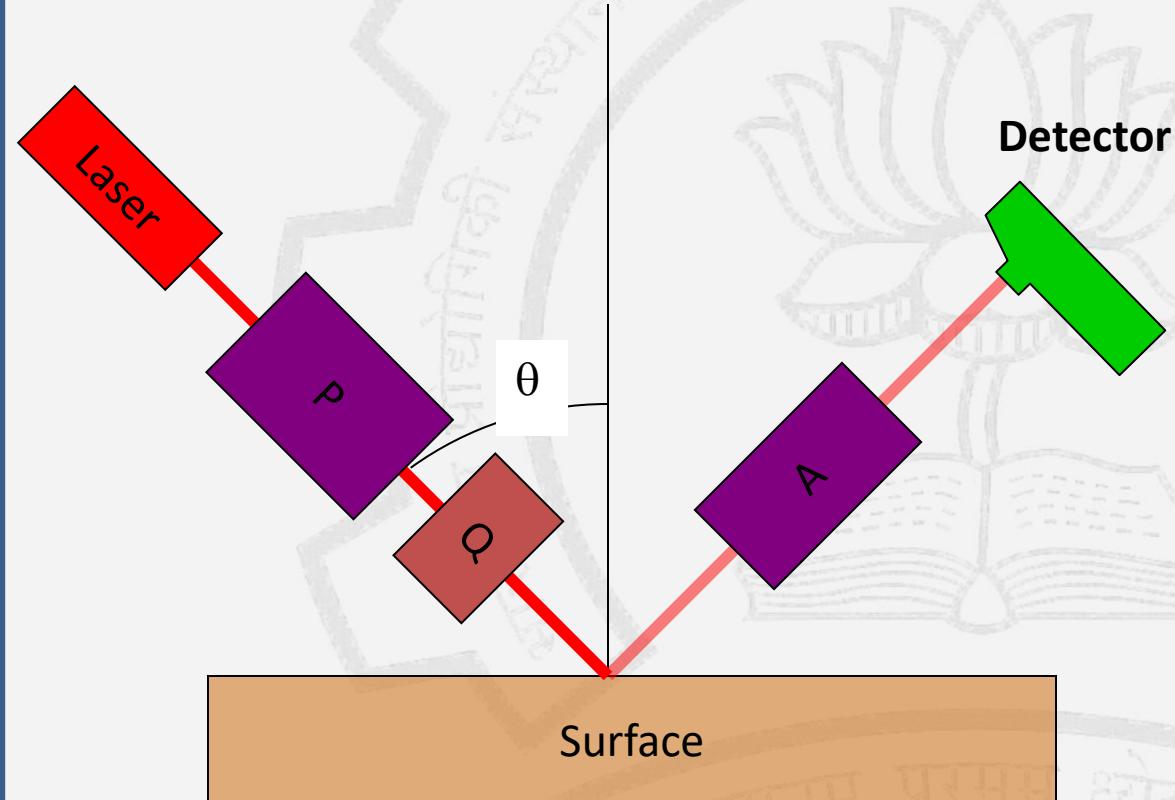
$$Z = \frac{E_0^-(x)}{H_0^-(x)}$$

Methodology



- Polarized light is reflected at an oblique angle to a surface
- The change to or from a generally elliptical polarization is measured.
- From these measurements, the complex index of refraction and/or the thickness of the material can be obtained. ($n = n + ik$)

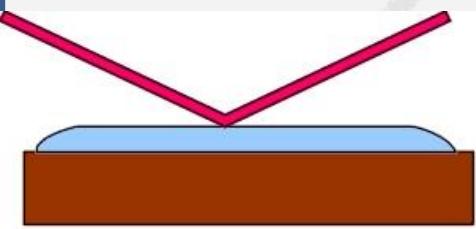
Methodology: Contd.



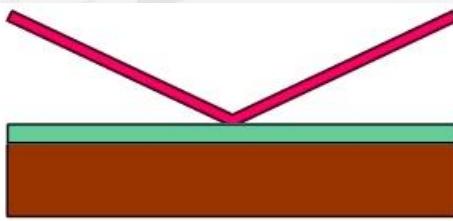
An ellipsometer measures the changes in the polarization state of light when it is reflected from a sample. If the sample undergoes a change, for example a thin film on the surface changes its thickness, then its reflection properties will also change.

Measuring these changes in the reflection properties can allow us to deduce the actual change in the film's thickness.

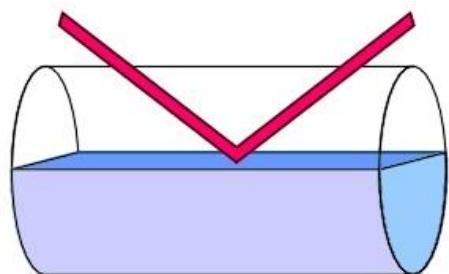
Applications



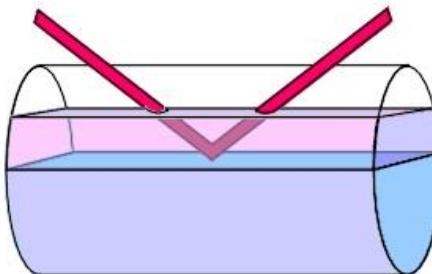
Liquid thin films



Solid thin films



Liquid-vapor interfaces



Liquid-liquid interfaces

- Determining the thickness of a thin film

Applications - Continued

- Research
 - Thin films, surface structures
 - Emphasis on accuracy and precision
- Spectroscopic
 - Analyze multiple layers
 - Determine optical constant dispersion relationship
 - Degree of crystallinity of annealed amorphous silicon
- Semiconductor applications
 - Solid surfaces
 - Industrial applications in fabrication
 - Emphasis on reliability, speed and maintenance
 - Usually employs multiple methods

Ellipsometry

- Allows us to probe the surface structure of materials.
- Makes use of Maxwell's equations to interpret data.
- Is often relatively insensitive to calibration uncertainties.
- Accuracies to the Angstrom
- Can be used in-situ (as a film grows)
- Typically used in thin film applications

Reference Ellipsometry

1. Riedling, K. (1988). Ellipsometry for Industrial Applications. New York, Springer-Verlag Wein, p.1-21.
2. Tompkins, H. G. (1993). A User's Guide to Ellipsometry. New York, Academic Press, Inc.
3. Tompkins, H. G., McGahan, W. A. (1999). Spectroscopic Ellipsometry and Reflectometry: A User's Guide. New Your, John Wiles & Sons, Inc

X-ray diffractometry (XRD) and reflectometry (XRR) and their application in semiconductor material research

X-ray techniques

photographic registration
of intensities

X-ray photography

Angular dependent
registration of diffracted
(reflected) X-ray intensities

X-ray diffractometry

registration of X-ray
fluorescence radiation

X-ray fluorescence

X-ray techniques

photographic registration of intensities

Angular dependent registration of diffracted (reflected) X-ray intensities

registration of X-ray fluorescence radiation

X-ray photography

absorption contrast

material analysis

medical application

tomography

X-ray topography

diffractometry

X-ray diffractometry

X-ray fluorescence

Berg-Barrett topography

Lang topography

Section topography

and many others

Example:
seed

Dislocations in the growth
of a 300 mm Si crystal

X-ray techniques

photographic registration of intensities

X-ray photography

absorption contrast

material analysis
medical application
tomography

X-ray topography
dифрактометрия

Angular dependent registration of diffracted (reflected) X-ray intensities

X-ray diffractometry

powder diffractometry
(wide angle diffract.)

high resolution XRD

reflectometry XRR

Standing wave techniques

Scattering techniques

registration of X-ray fluorescence radiation

X-ray fluorescence

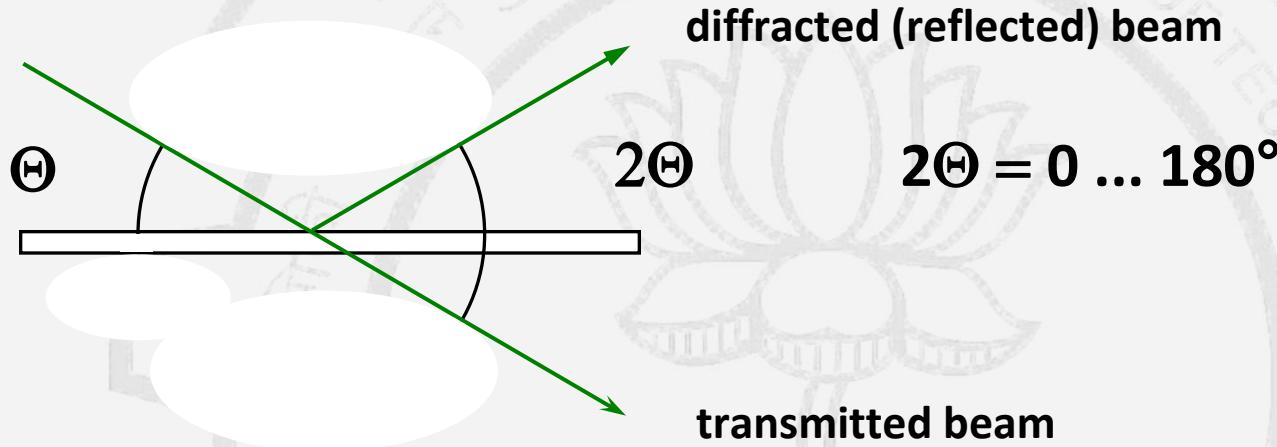
XRF

- element analysis
- layer thickness meas.

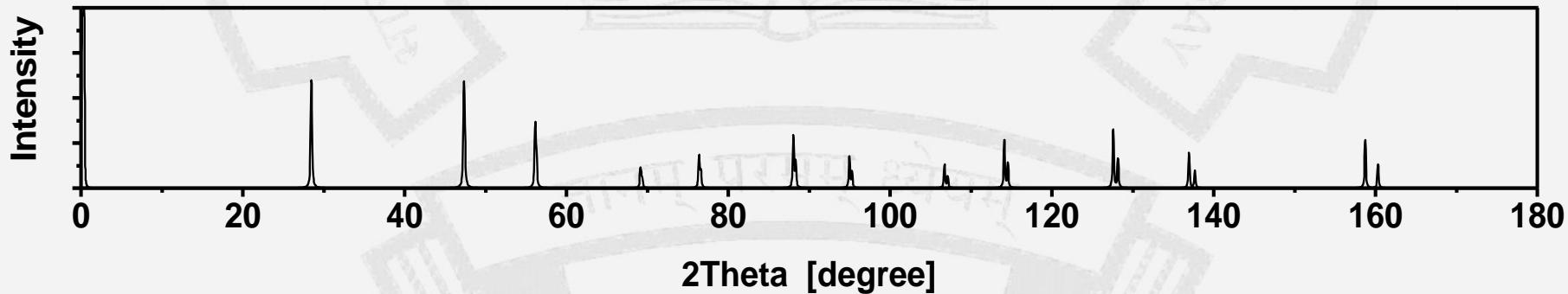
TXRF (total refl. XRF)
- surface contamination

Spectroscopic techniques
e.g. EXAFS

Angular range of XRD



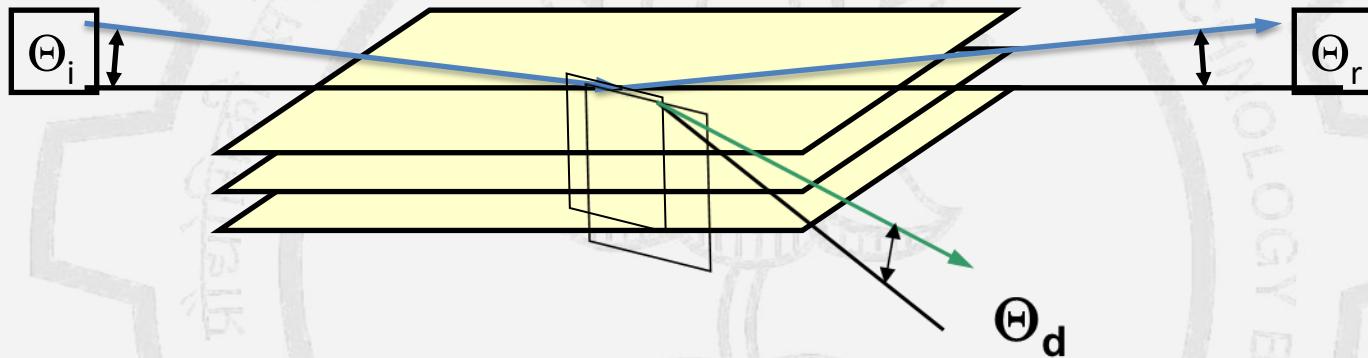
180° diffraction pattern of Si with CuK_α radiation



X-ray reflectometry (XRR)

Introduction

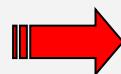
Experimental geometry for scattering under grazing angles:



Various techniques to probe different properties:

- Specular reflectivity (forward scattering, $\Theta_i = \Theta_r$) \rightarrow density profile)
- Diffuse reflectivity (small angle scattering, $\Theta_i \neq \Theta_r$) \rightarrow inhomogeneities)
- Grazing incidence diffraction GID (wide angle scattering \rightarrow crystallinity)

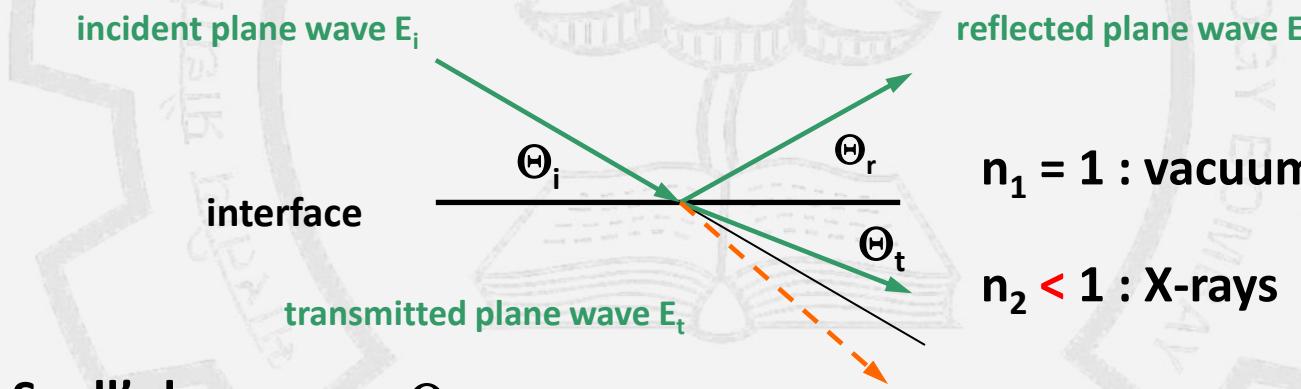
Elementary X-ray optics under grazing angles



There is NO difference between X-ray and visible light optics !

The only difference is the index of refraction !

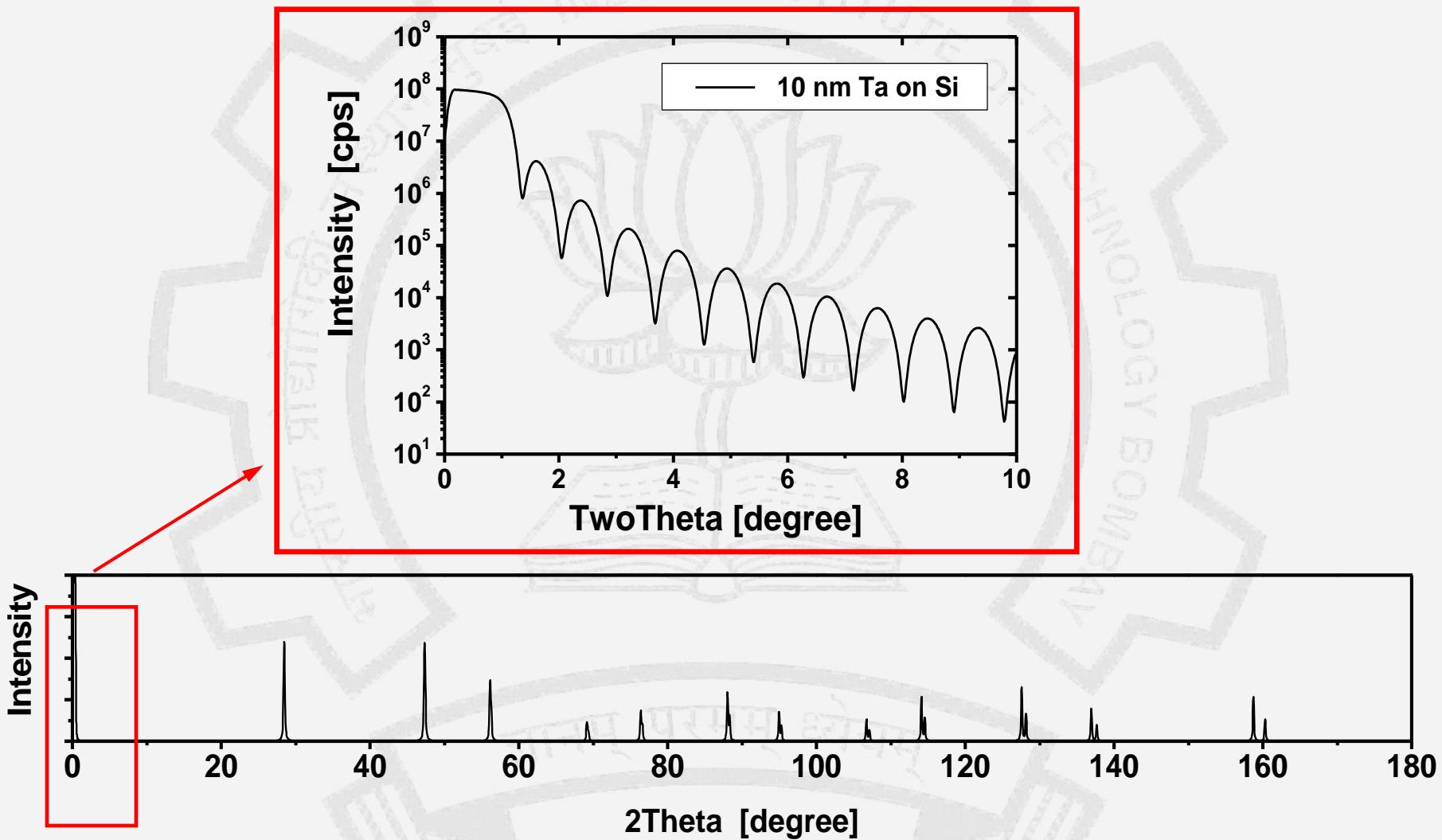
Reflection and refraction of electromagnetic waves at an interface:



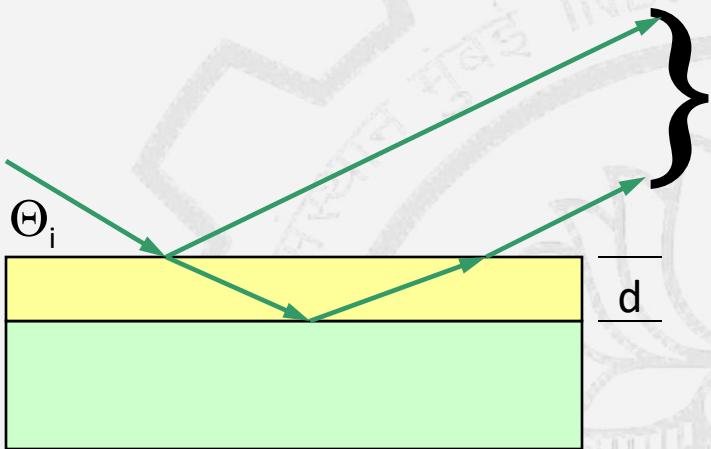
Snell's law: $n_1 \cdot \cos\Theta_i = n_2 \cdot \cos\Theta_t$

Fresnel equations

X-ray reflectometry (XRR)



Specular reflectivity: layer on a surface

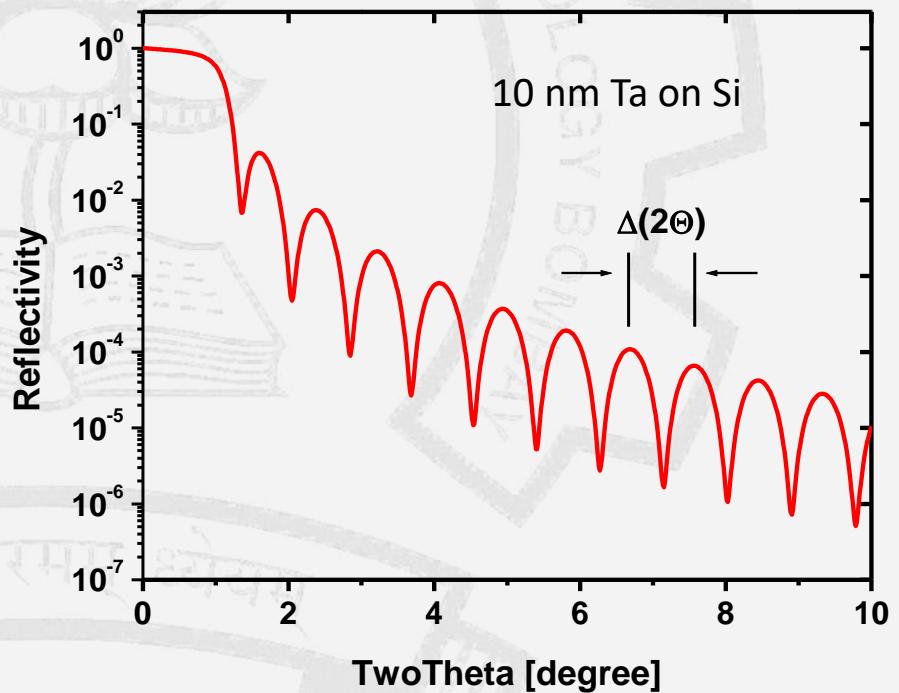


Constructive / destructive interference
as a function of Θ_i

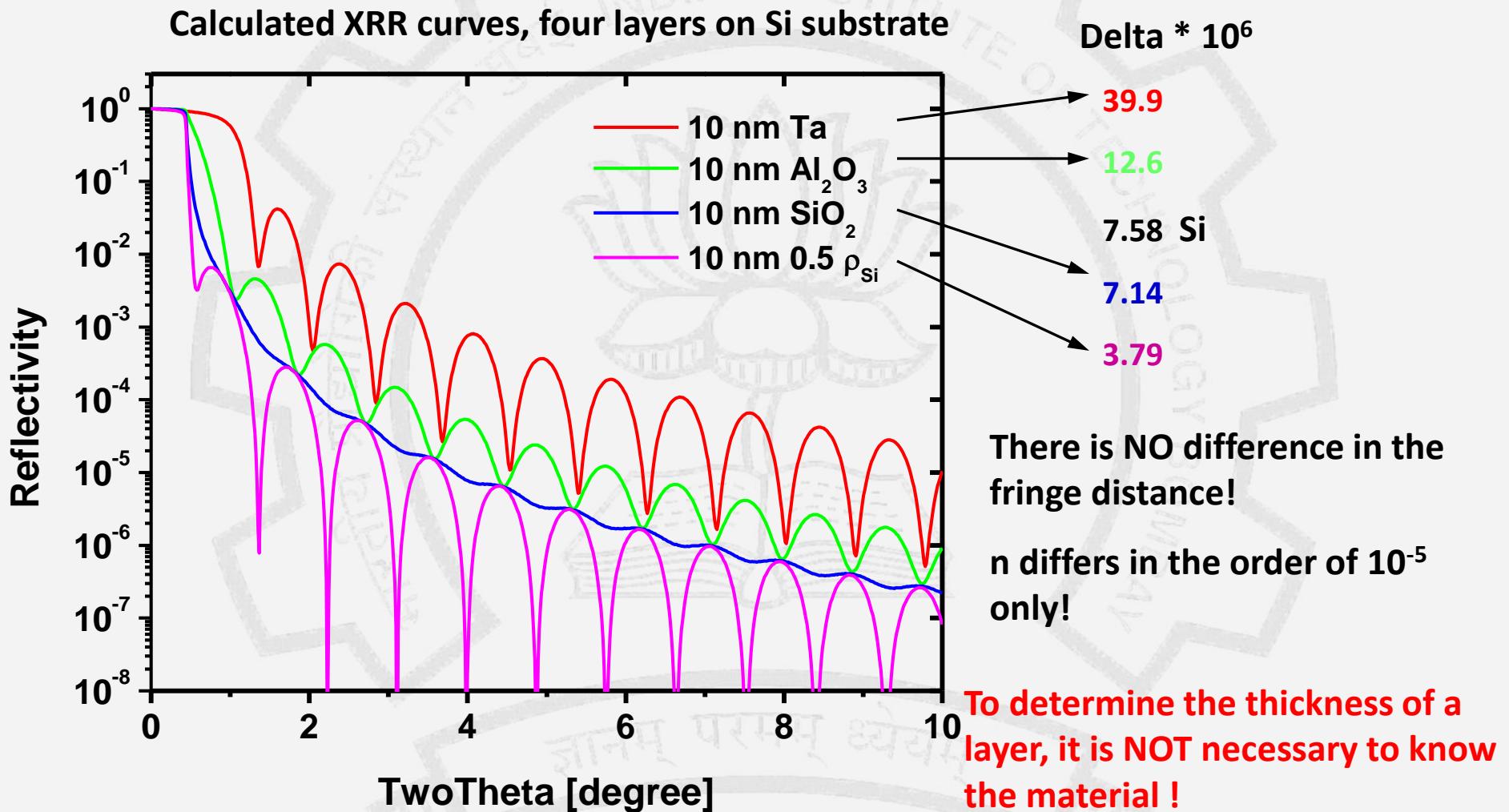
“thickness oscillations”

$$\Delta(2\Theta_i) \approx \frac{\lambda}{d}$$

? NO material dependence ?



Reflectivity curves of different layer materials



X-ray reflectivity information

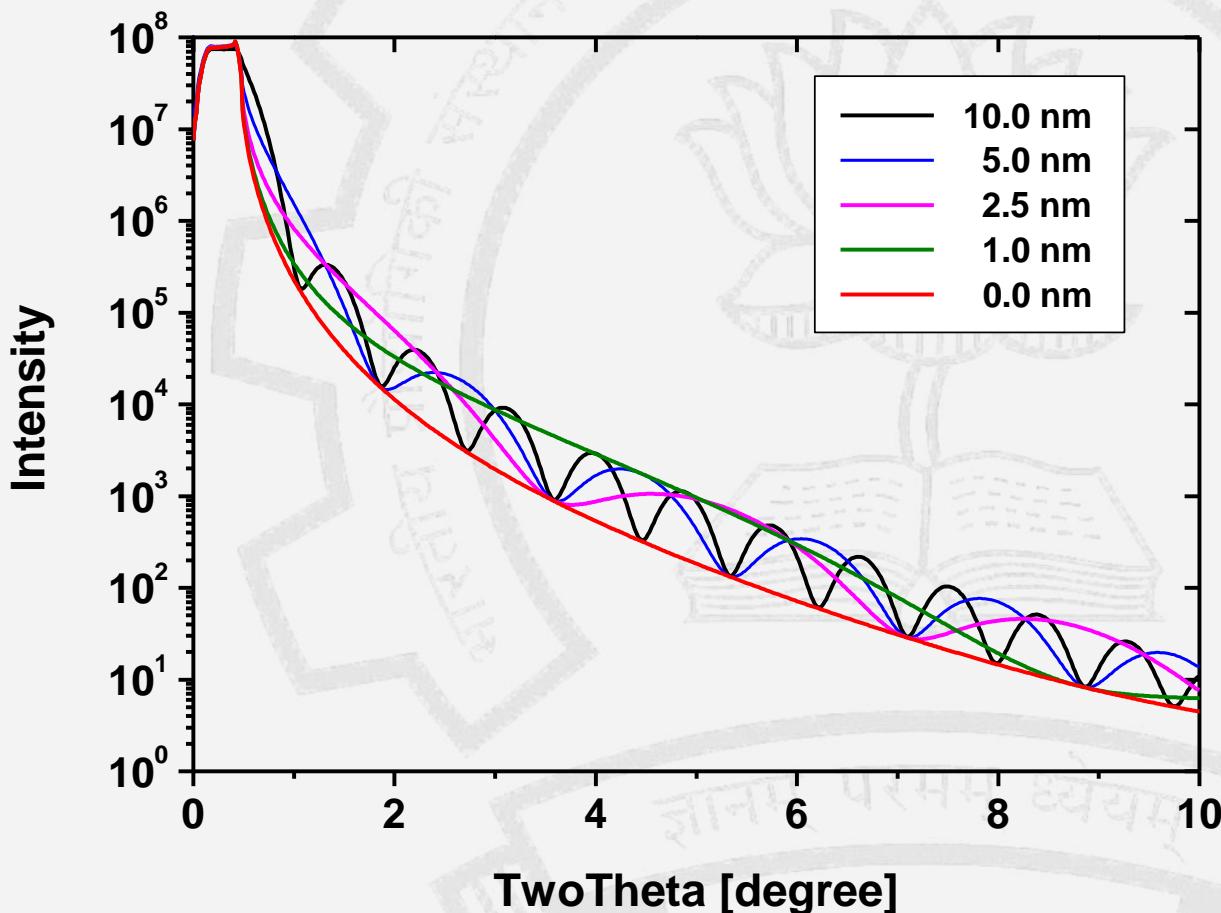
Information from XRR curves:

- layer thickness
- layer density
- interface roughness

Which experimental (or general) limitations are given for these parameters?

Minimum layer thickness (example Al_2O_3)

XRR curve simulation for Al_2O_3 layers of different thickness



$$I_o = 7.5 \cdot 10^7 \text{ cps}$$

$$I_u = 2 \text{ cps}$$

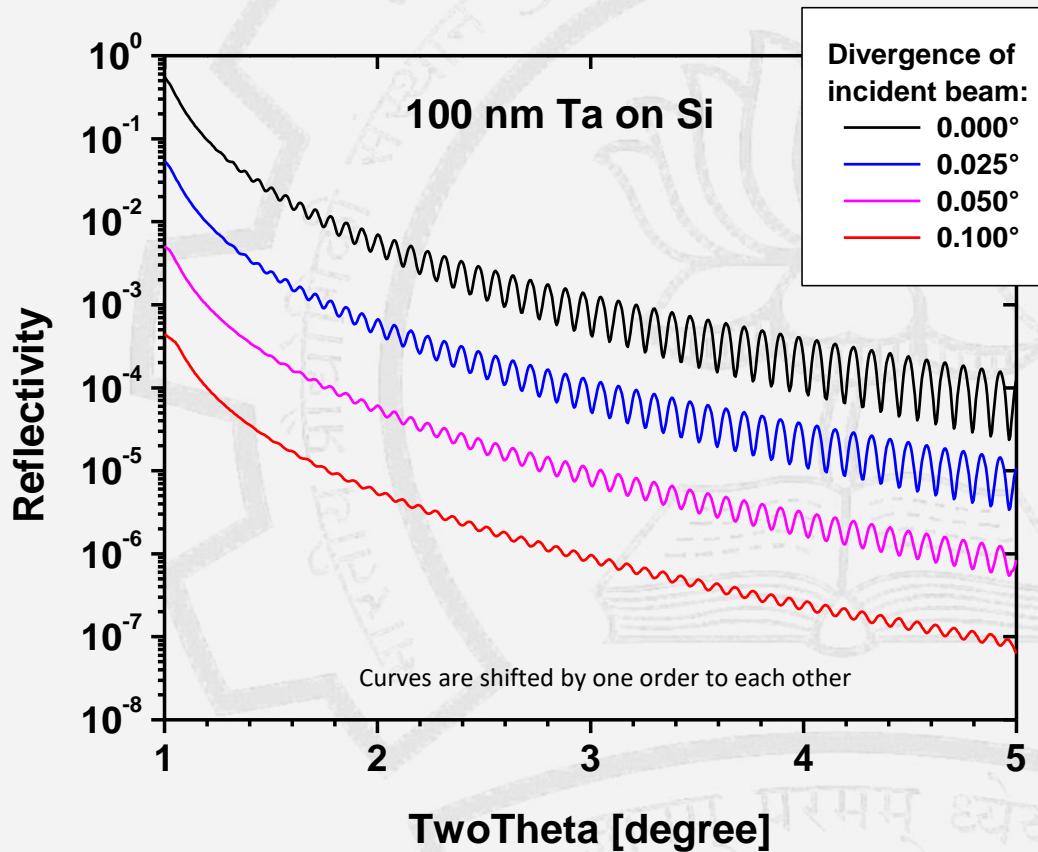
SF & IF roughness: 0.2 nm

The minimum layer thickness depends on

- the INTENSITY of your X-ray source (measuring range)
- the difference in δ between layer and substrate

Maximum layer thickness (example Al_2O_3)

XRR curve simulation for different divergence of the incident beam



$$d = 100 \text{ nm}$$

$$\Rightarrow \Delta(2\Theta)_{\text{osc}} \approx 0.088^\circ$$

The maximum layer thickness depends on

- the DIVERGENCE of the incident X-ray beam

$$d_{\max} \approx \lambda / \Delta(2\Theta)_{\text{div}}$$

Typical layer thickness range : 2 ... 200 nm

Some hints for XRR measurements and curve analysis

1. Try to get as much information about the sample preparation as possible.
2. Adopt the measuring conditions (scan range, step size, counting time, etc.)
3. Extract first information from XRR curve:
 - main oscillation distance → main layer thickness
 - beating oscillations → additional layer thickness
4. Start curve simulation with the simplest possible layer model.
5. Try to improve your model carefully and step by step.
6. Don't overestimate a weak improvement of your fitting parameter!

Always remain skeptical of your own results!

Wide angle (powder) XRD

amorphus

Bragg equation: $2 d \sin\Theta = n \lambda$

d – net plane distance

n – diffraction order, λ - wavelength

Estimation of crystal lattice range to be investigated:

1st diffraction peak:

d = largest existing net plane distance

Si: d = a = 5.43 Å, n = 1 (100-reflection)

$$\sin\Theta = \lambda/2a \Rightarrow 2\Theta = 16.3^\circ$$

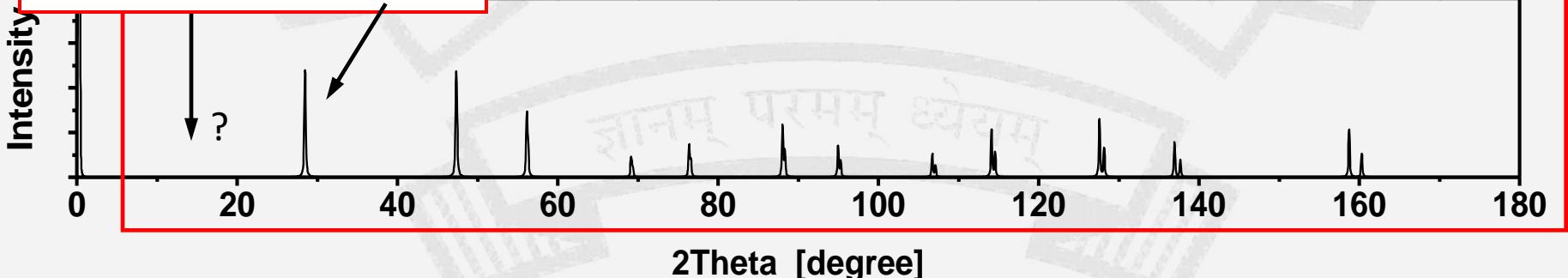
Smallest net plane distance:

$$180^\circ \Rightarrow \sin\Theta = 1, n = 1$$

$$d_{\min} = \lambda/2 = 0.77 \text{ \AA} \text{ for CuK}_\alpha \text{ radiation}$$

100 is forbidden !

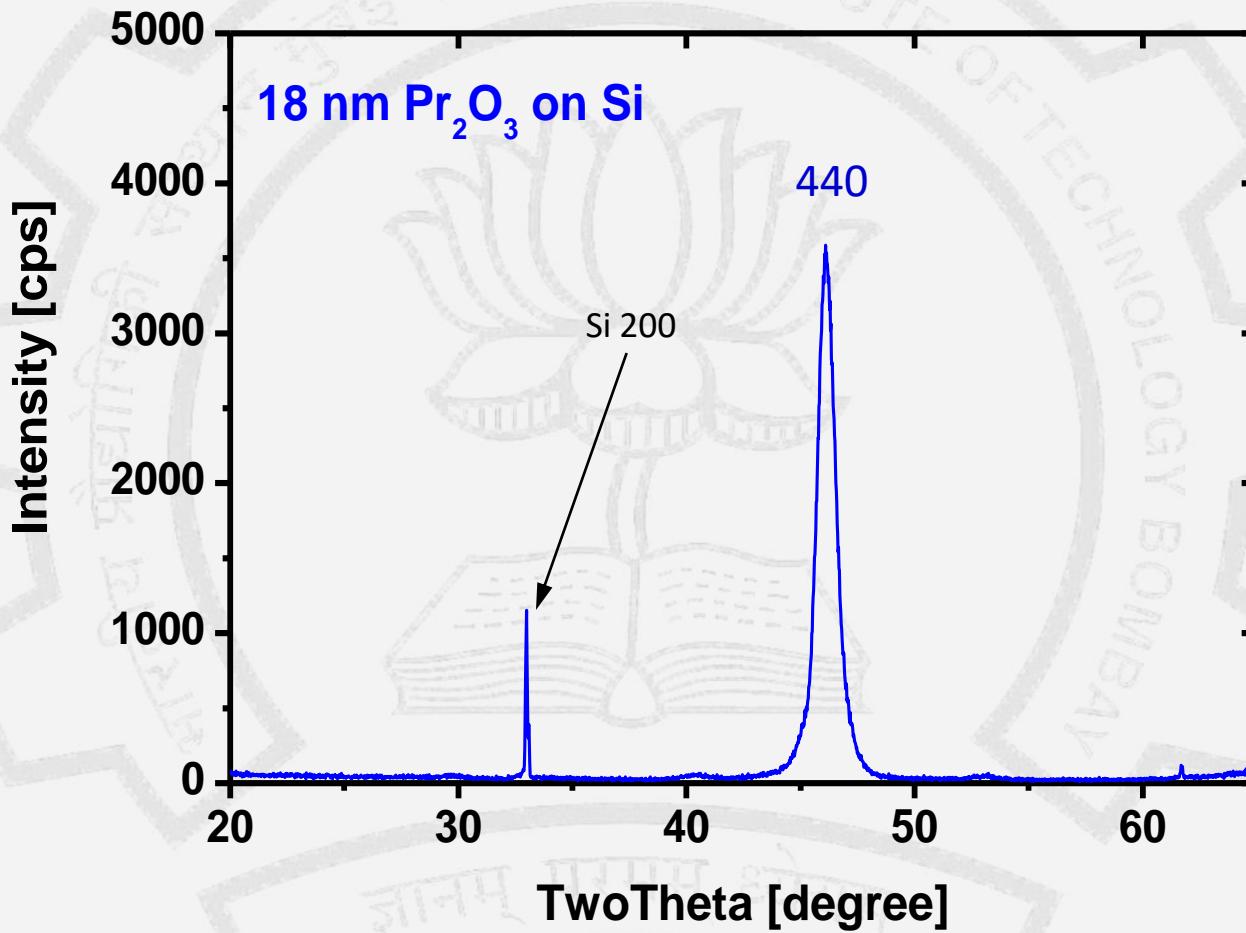
1st allowed reflection: 111



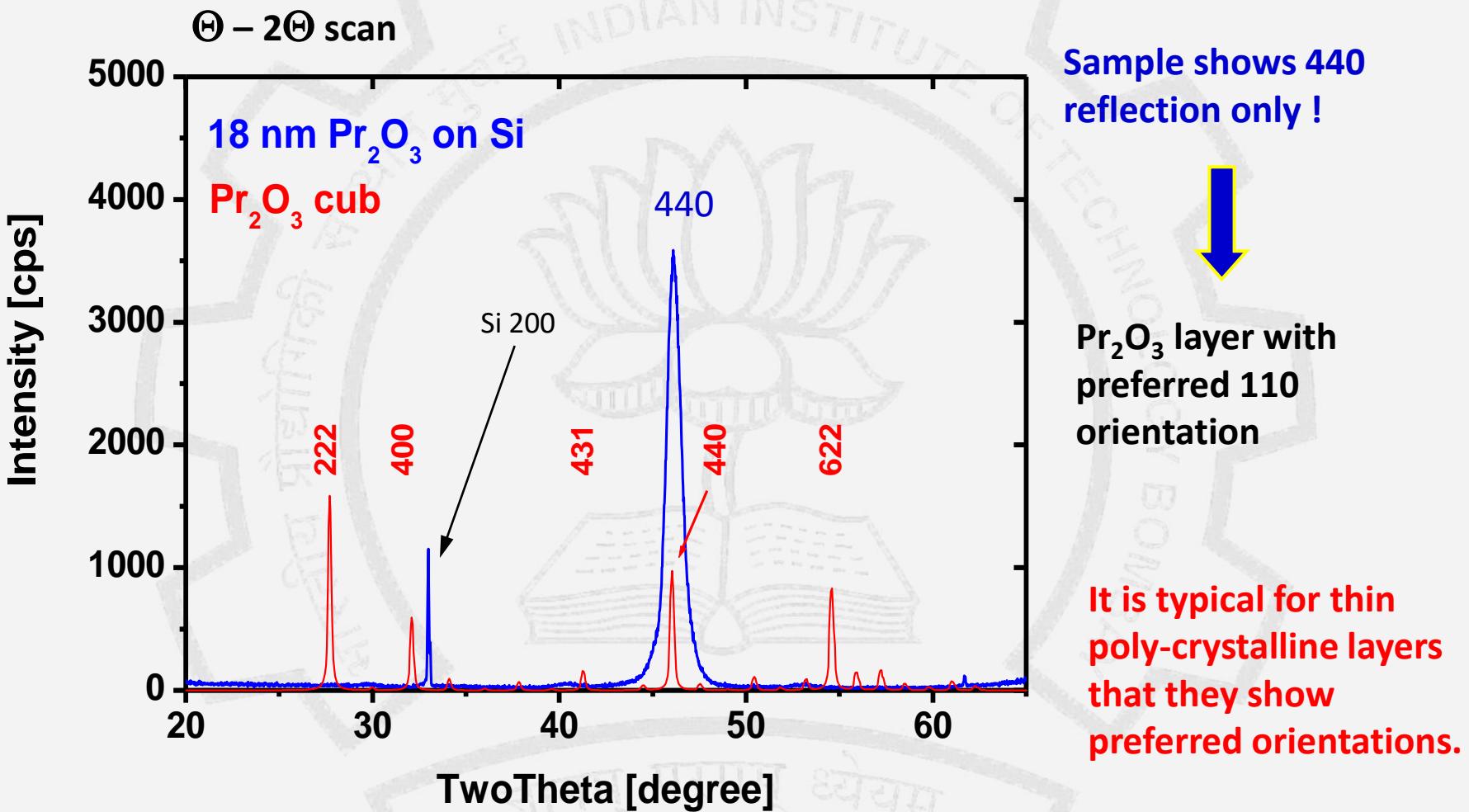
Powder diffraction

- Only that fraction of crystallites contributes to the diffraction, for which the Bragg law $2ds\sin\Theta_B = n\lambda$ and the condition $\Theta = \Theta_B$ is fulfilled.
- Most of the crystallites do not contributes to the diffraction.
- One possibility to increase the number of contributing crystallites is sample rotation.

Diffractogram of a Pr_2O_3 layer on 100 silicon



Diffractogram of a Pr_2O_3 layer on (100) silicon



Diffraction of poly-crystalline layers

- Only those crystallites contributes to the diffraction in a $\Theta - 2\Theta$ scan, for which the diffracting netplanes ($2d \sin \Theta_B = n\lambda$) lie parallel to the surface.
- The angular spread of the crystallite orientation can be investigated by a Θ scan.
- The determination of the azimuthal orientation of crystallites requires additional freedom of sample orientation.

Single crystal diffraction

- A single crystal diffraction ($\Theta - 2\Theta$ scan) shows always the fine structure of the used radiation (usually K_{α} -doublet) due to the limited angular divergence.
- A Θ scan on a single crystal 2Θ peak position analyzes the angular divergence of the incident beam.
- Offset correction is important. But sometimes it can be helpful to measure with a defined offset to suppress strong substrate peaks.

Grain size analysis

⇒ see basics; kinematical theory; lattice factor

- The smaller a diffracting crystal (grain), the wider is the diffraction pattern.

Scherrer equation (1918):

$$D = K \lambda / \delta\Theta_D \cos\Theta_B$$

with D – grain size

$\delta\Theta_D$ – diffraction broadening

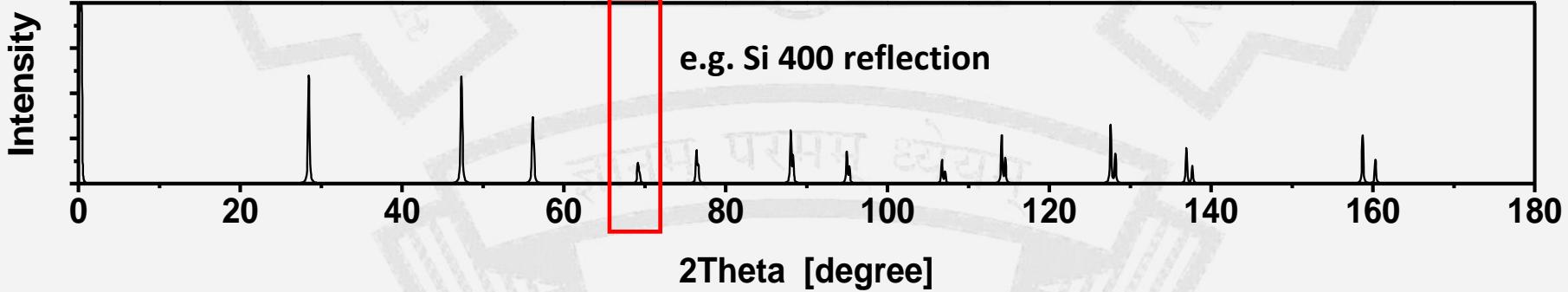
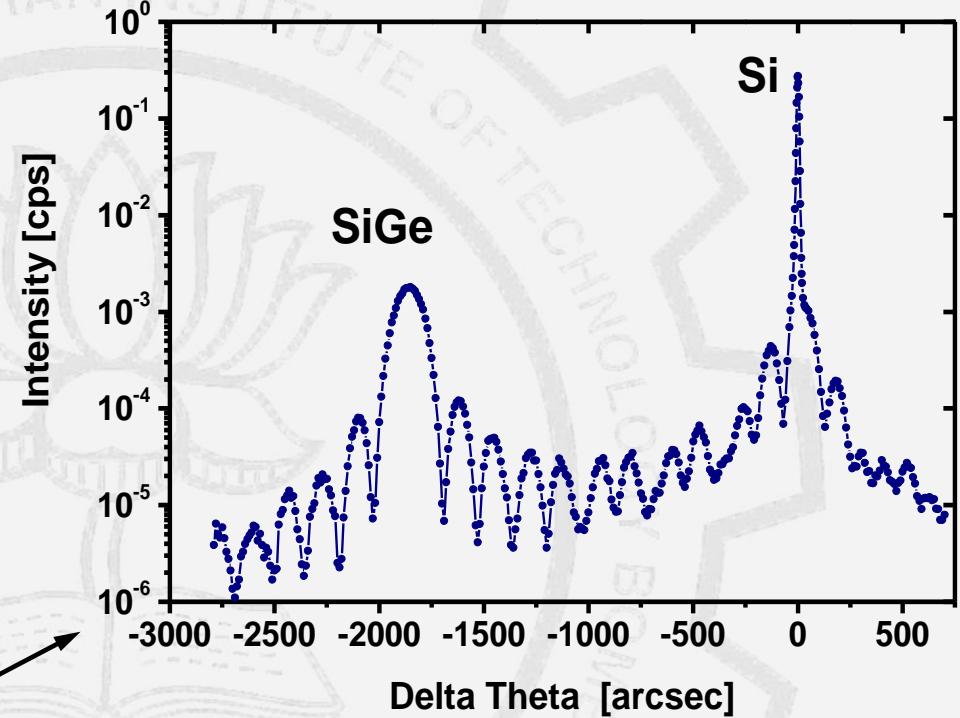
K – constant depending on hkl, etc. (0.7...1.7)

Observed integral line width: $\delta\Theta^2 = \delta\Theta_i^2 + \delta\Theta_D^2$

with $\delta\Theta_i$ – instrumental broadening

High resolution XRD

DCD rocking curve



Summary on XRD

- **X-Ray reflectometry (XRR)**

$2\Theta = 0 \dots 10^\circ$

layer and substrate might be crystalline or amorphous

thickness of thin layers, surface/interface roughness, layer density

- **Wide angle (powder, single crystal) X-ray diffractometry (XRD)**

2Θ : whole angular range

(poly)crystalline material

phase analysis, lattice parameter, grain size, strain, texture

- **High resolution X-ray diffractometry (HRXRD, DCD, TCD)**

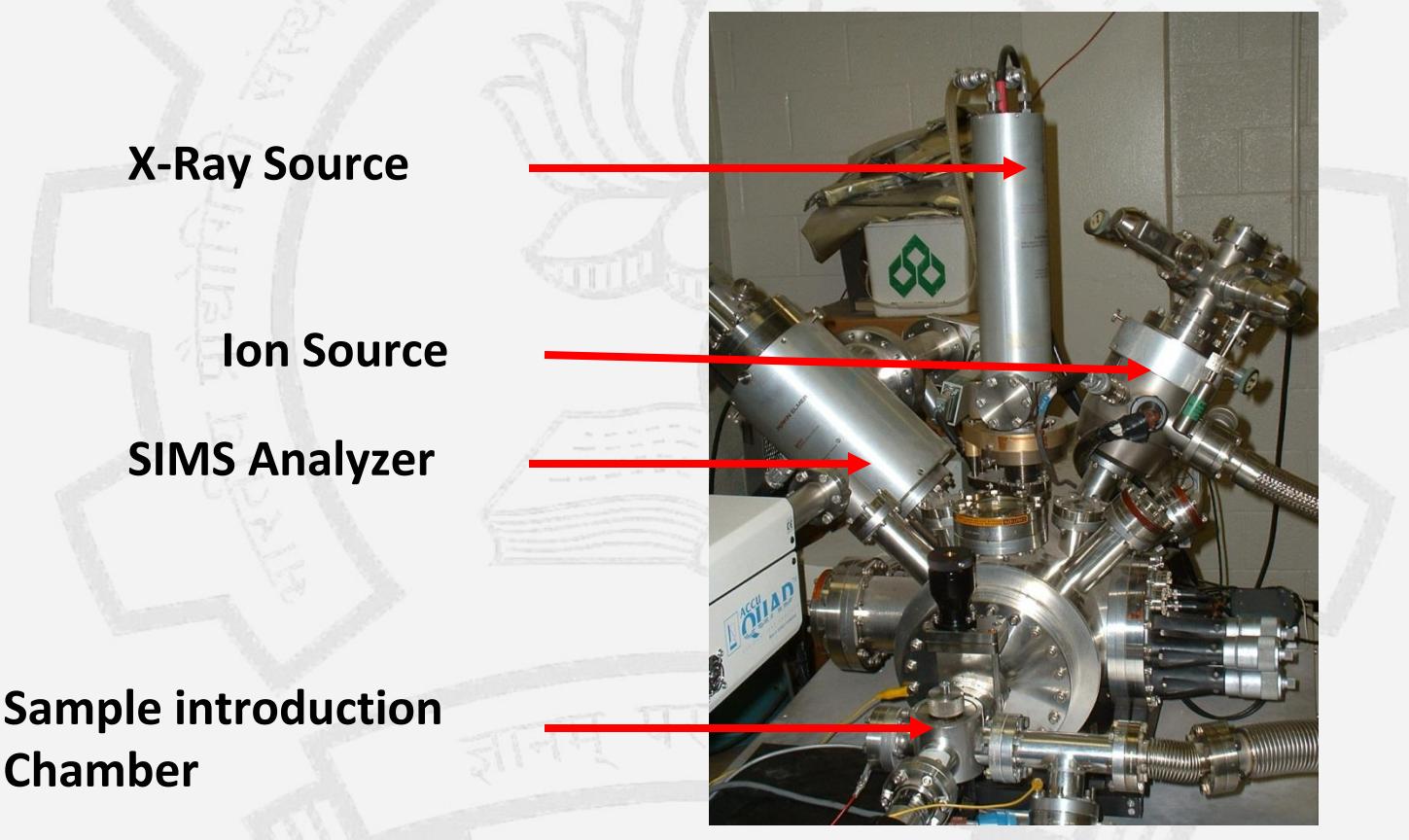
2Θ : near single crystal reflection peak, $\Delta\Theta < \pm 0.5^\circ$

(nearly perfect) single crystalline material, epitaxial layers

analysis of crystal perfection, depth profile of lattice strain

double-crystal diffractometers

X-Ray Photoelectron Spectroscopy (XPS)



XPS Background

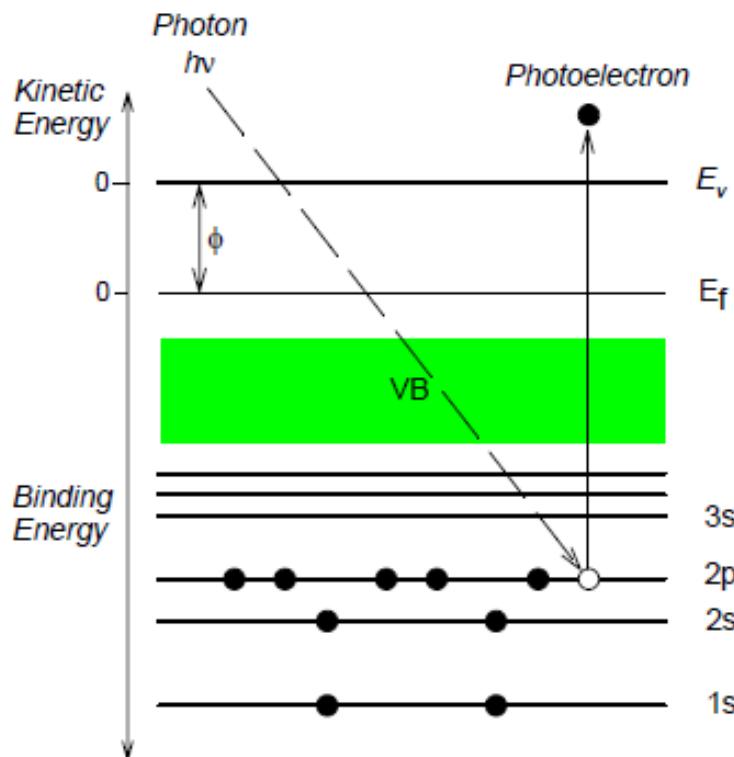
- XPS technique is based on Einstein's discovery about the photoelectric effect, developed around 1905
 - The concept of photons was used to describe the ejection of electrons from a surface when photons were impinged upon it
- During the mid 1960's Dr. Siegbahn and his research group developed the XPS technique.
 - **In 1981, Dr. Siegbahn was awarded the Nobel Prize in Physics for the development of the XPS technique**

XPS Basics

- Irradiate the sample surface, hitting the core electrons (e^-) of the atoms.
- The X-Rays penetrate the sample to a depth on the order of a micrometer.
- Useful e^- signal is obtained only from a depth of around 10 to 100 Å on the surface.
- The X-Ray source produces photons with certain energies:
 - MgK α photon with an energy of 1253.6 eV
 - AlK α photon with an energy of 1486.6 eV
- Normally, the sample will be radiated with photons of a single energy (MgK α or AlK α). This is known as a monoenergetic X-Ray beam.

XPS Basics

--- X-ray Photoelectron Spectroscopy (XPS)



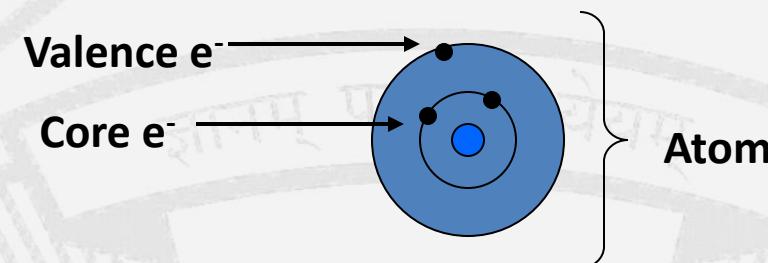
$$KE = h\nu - (E_B + \phi)$$

XPS spectrum:
Intensities of photoelectrons
versus E_B or KE

- Elemental identification and chemical state of element
- Relative composition of the constituents in the surface region
- Valence band structure

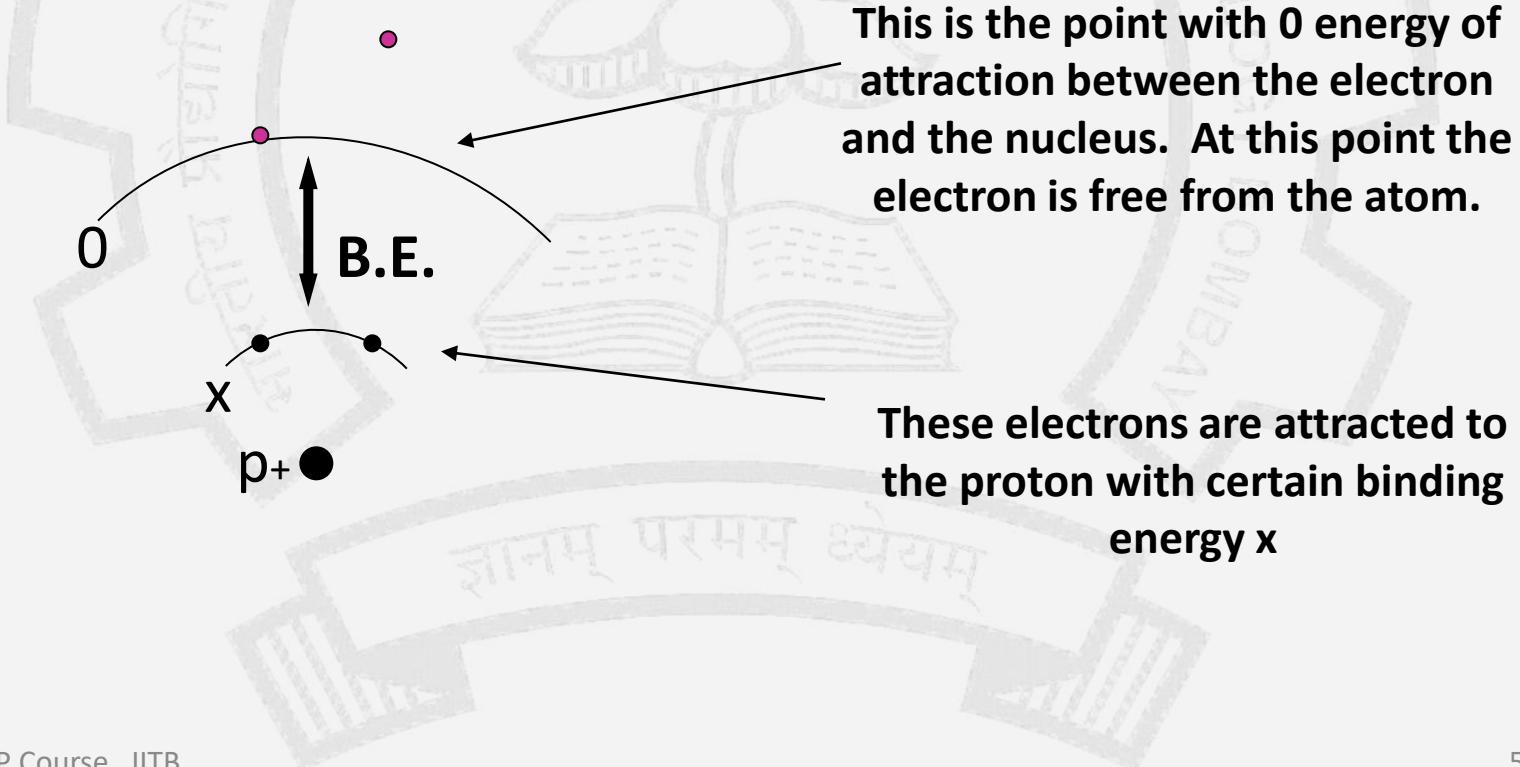
Why the Core Electrons?

- An electron near the Fermi level is far from the nucleus, moving in different directions all over the place, and will not carry information about any single atom.
 - Fermi level is the highest energy level occupied by an electron in a neutral solid at absolute 0 temperature.
 - Electron binding energy (BE) is calculated with respect to the Fermi level.
- The core e⁻s are local, closed to the nucleus and have binding energies characteristic of their particular element.
- The core e⁻s have a higher probability of matching the energies of AlK α and MgK α .

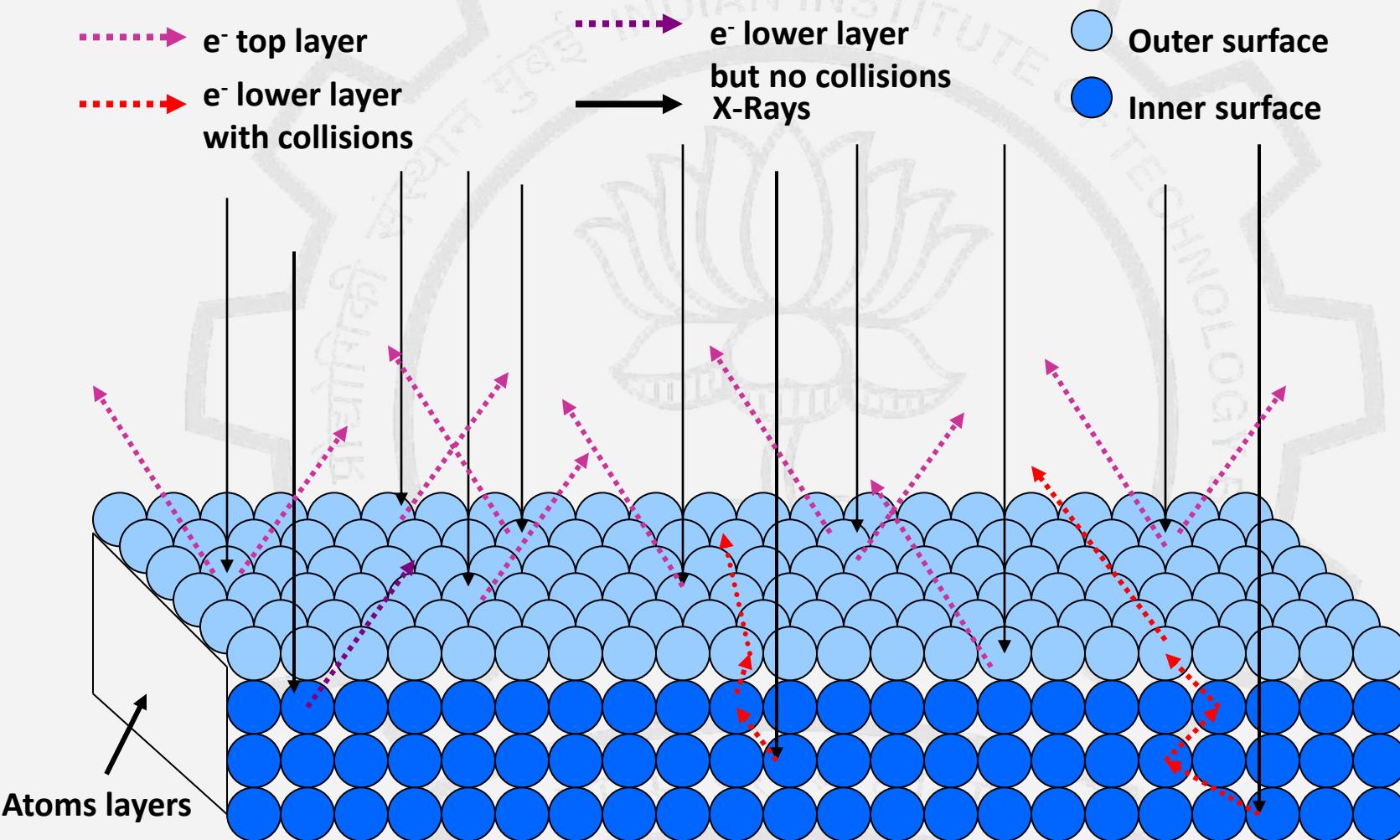


Binding Energy (BE)

The Binding Energy (BE) is characteristic of the core electrons for each element. The BE is determined by the attraction of the electrons to the nucleus. If an electron with energy x is pulled away from the nucleus, the attraction between the electron and the nucleus decreases and the BE decreases. Eventually, there will be a point when the electron will be free of the nucleus.



X-Rays on the Surface



X-Rays on the Surface

- The X-Rays will penetrate to the core e^- of the atoms in the sample.
- Some e^- s are going to be released without any problem giving the Kinetic Energies (KE) characteristic of their elements.
- Other e^- s will come from inner layers and collide with other e^- s of upper layers
 - These e^- will be lower in lower energy.
 - They will contribute to the noise signal of the spectrum.

Equation

$$KE = h\nu - BE - \phi$$

KE → Kinetic Energy (measure in the XPS spectrometer)

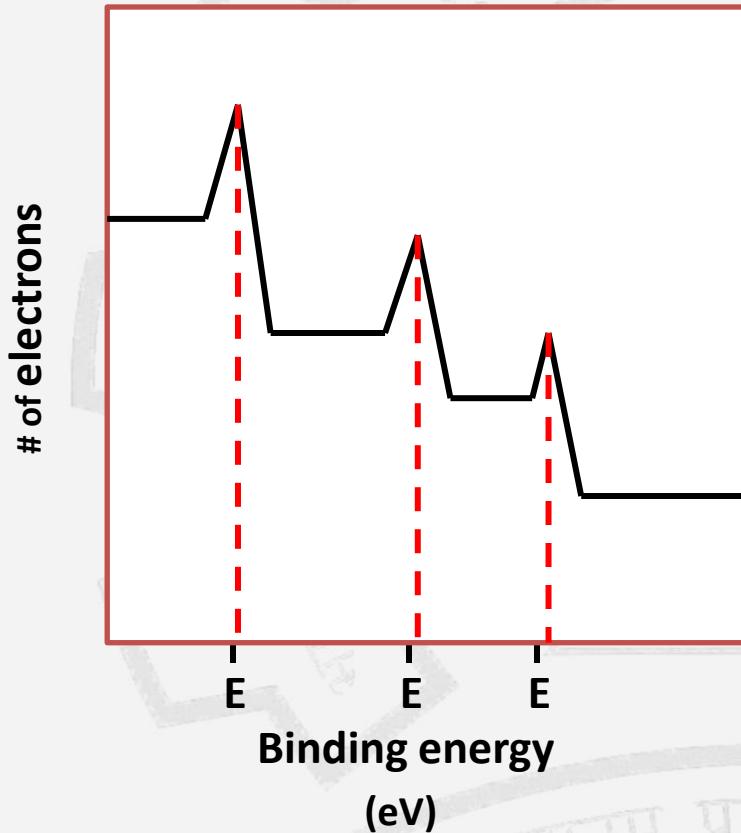
$h\nu$ → photon energy from the X-Ray source (controlled)

ϕ → spectrometer work function. It is a few eV, it gets more complicated because the materials in the instrument will affect it. Found by calibration.

BE → is the unknown variable

- The equation will calculate the energy needed to get an e^- out from the surface of the solid.
- Knowing KE, $h\nu$ and ϕ the BE can be calculated.

KE versus BE



KE can be plotted depending on BE

Each peak represents the amount of e⁻s at a certain energy that is characteristic of some element.

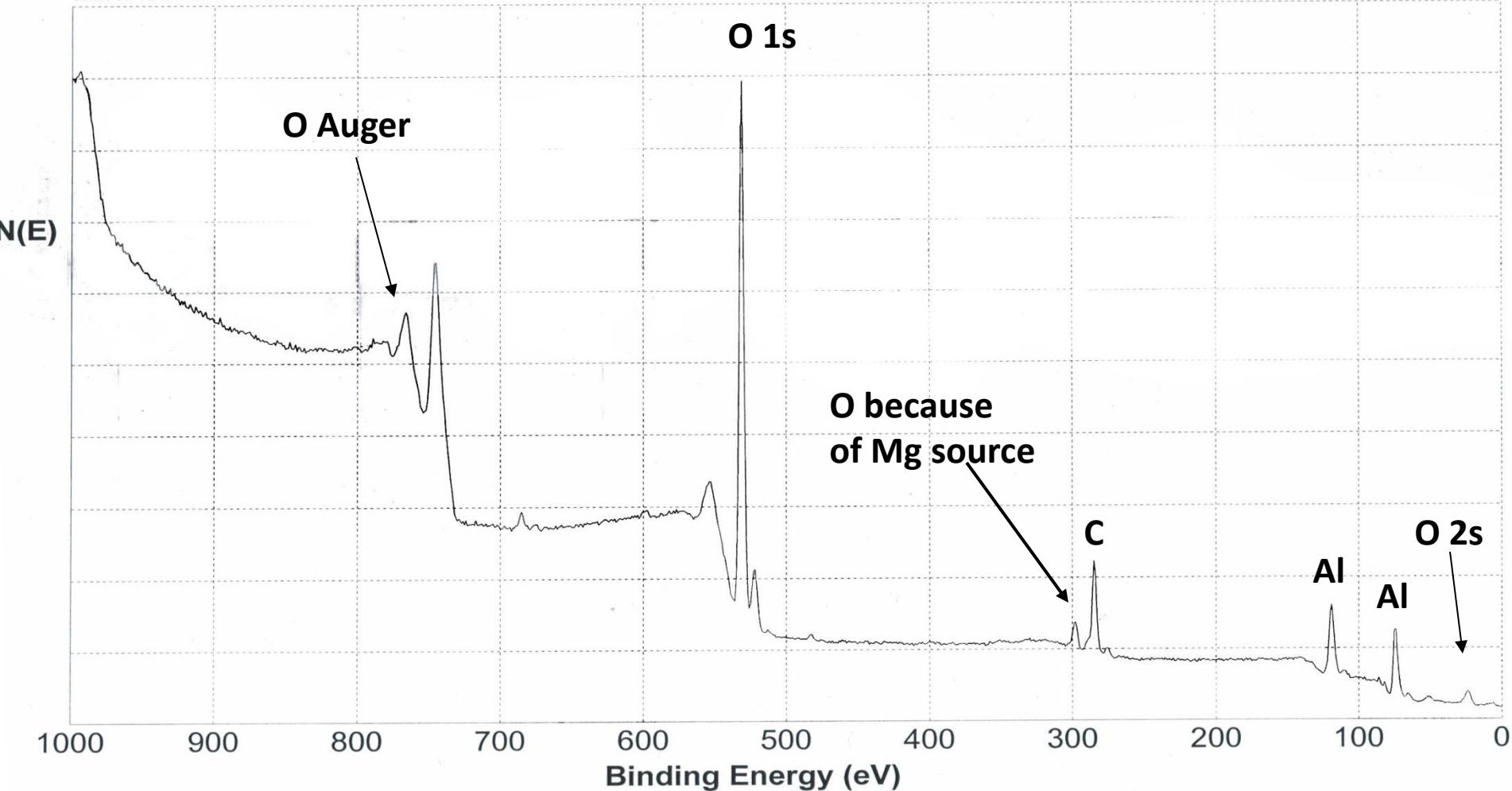
BE increase from right to left

1000 eV 0 eV

KE increase from left to right

XPS Spectrum

Min: 2132 Max: 130996



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Department of Physics at the University of Texas at El Paso

Identification of XPS Peaks

- The plot has characteristic peaks for each element found in the surface of the sample.
- There are tables with the KE and BE already assigned to each element.
- After the spectrum is plotted you can look for the designated value of the peak energy from the graph and find the element present on the surface.

Chemical Effects in XPS

Chemical shift: Change in binding energy of a core electron of an element due to a change in the chemical bonding of that element.

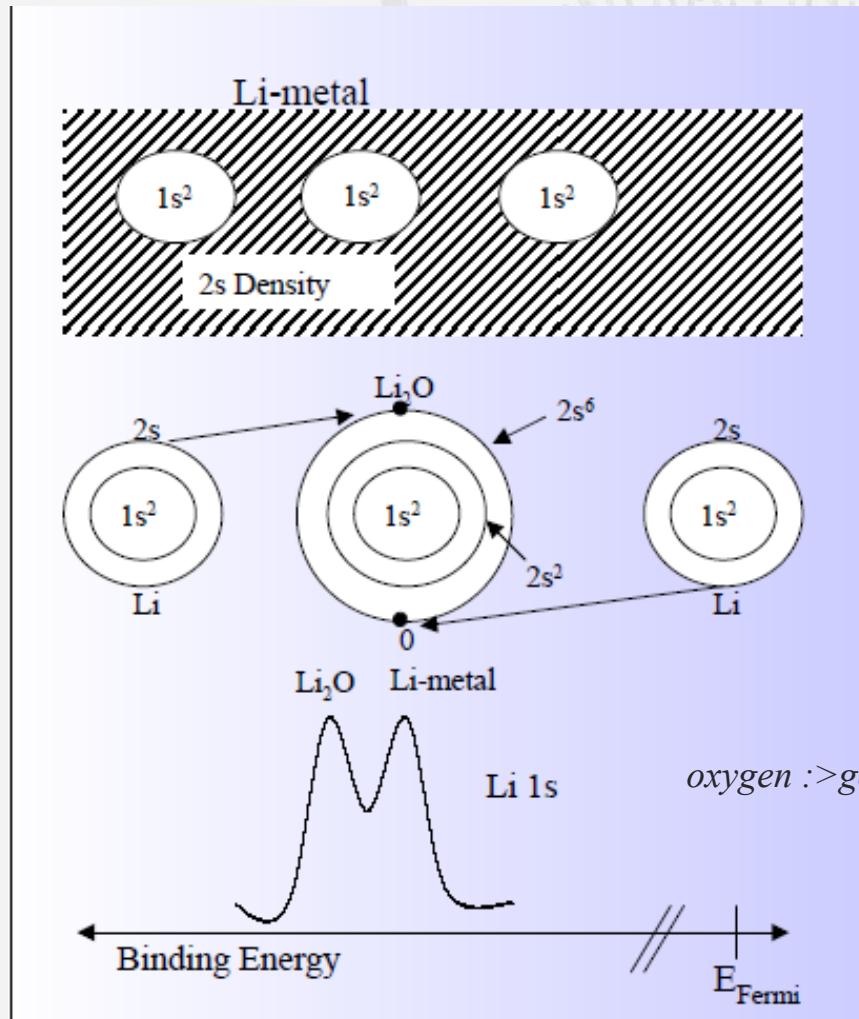
Qualitative view: Core binding energies are determined by:

- electrostatic interaction between it and the nucleus, and reduced by:
- the electrostatic shielding of the nuclear charge from all other electrons in the atom (including valence electrons)
- removal or addition of electronic charge as a result of changes in bonding will alter the shielding

Withdrawal of valence electron charge → increase in BE (oxidation)

Addition of valence electron charge ← decrease in BE

Chemical Shifts: Oxide Compared to Metal



➤ Binding Energy is lower due to increased screening of the nucleus by 2s conduction by 2s electrons

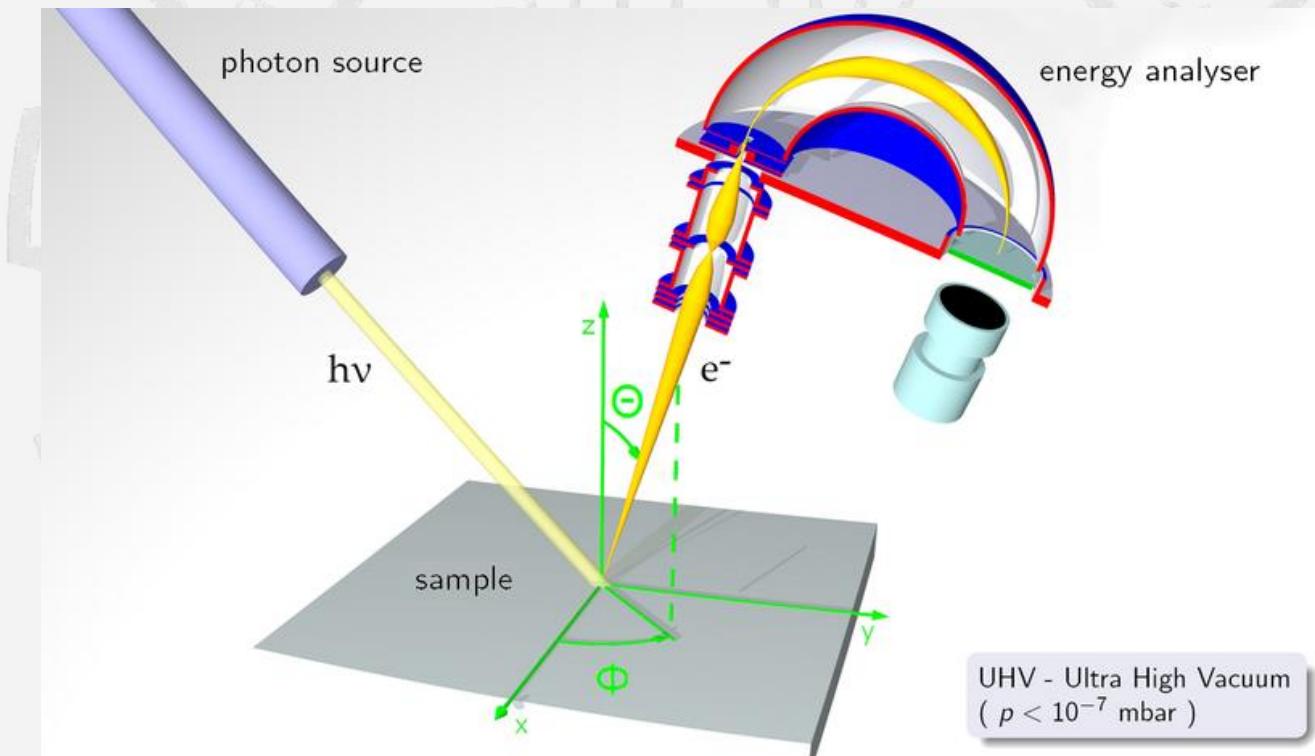
➤ Binding Energy is higher because Li 2s electron density is lost to oxygen

oxygen :> gets electron so BE reduces in oxide

References: XPS

- *Handbooks of Monochromatic XPS Spectra* -, B.V.Crist, published by XPS International LLC, 2005, Mountain View, CA, USA
- *Handbooks of Monochromatic XPS Spectra, Volumes 1-5*, B.V.Crist, XPS International LLC, 2004, Mountain View, CA, USA
- *Surface Analysis by Auger and X-ray Photoelectron Spectroscopy*, ed. J.T.Grant and D.Briggs, IM Publications, 2003, Chichester, UK
- *Practical Surface Analysis by Auger and X-ray Photoelectron Spectroscopy*, 2nd edition, ed. M.P.Seah and D.Briggs, Wiley & Sons, 1992, Chichester, UK
- *Practical Surface Analysis by Auger and X-ray Photoelectron Spectroscopy*, ed. M.P.Seah and D.Briggs, published by Wiley & Sons, 1983, Chichester, UK
- *Handbook of X-ray Photoelectron Spectroscopy*, J.F.Moulder, W.F.Stickle, P.E.Sobol, and K.D.Bomben, Perkin-Elmer Corp., 1992, Eden Prairie, MN, USA

Ultra-violet photoelectron spectroscopy (UPS)



Energy range: Spectroscopy

- Different probes are possible:
- Auger -> electrons (2 – 10 keV)
- XPS (or ESCA) -> X rays (0.2 – 2 keV) (x-rays photoelectron spectroscopy)
- UPS -> Ultraviolet photons (10 – 50 eV) (UV photoelectron spectroscopy)

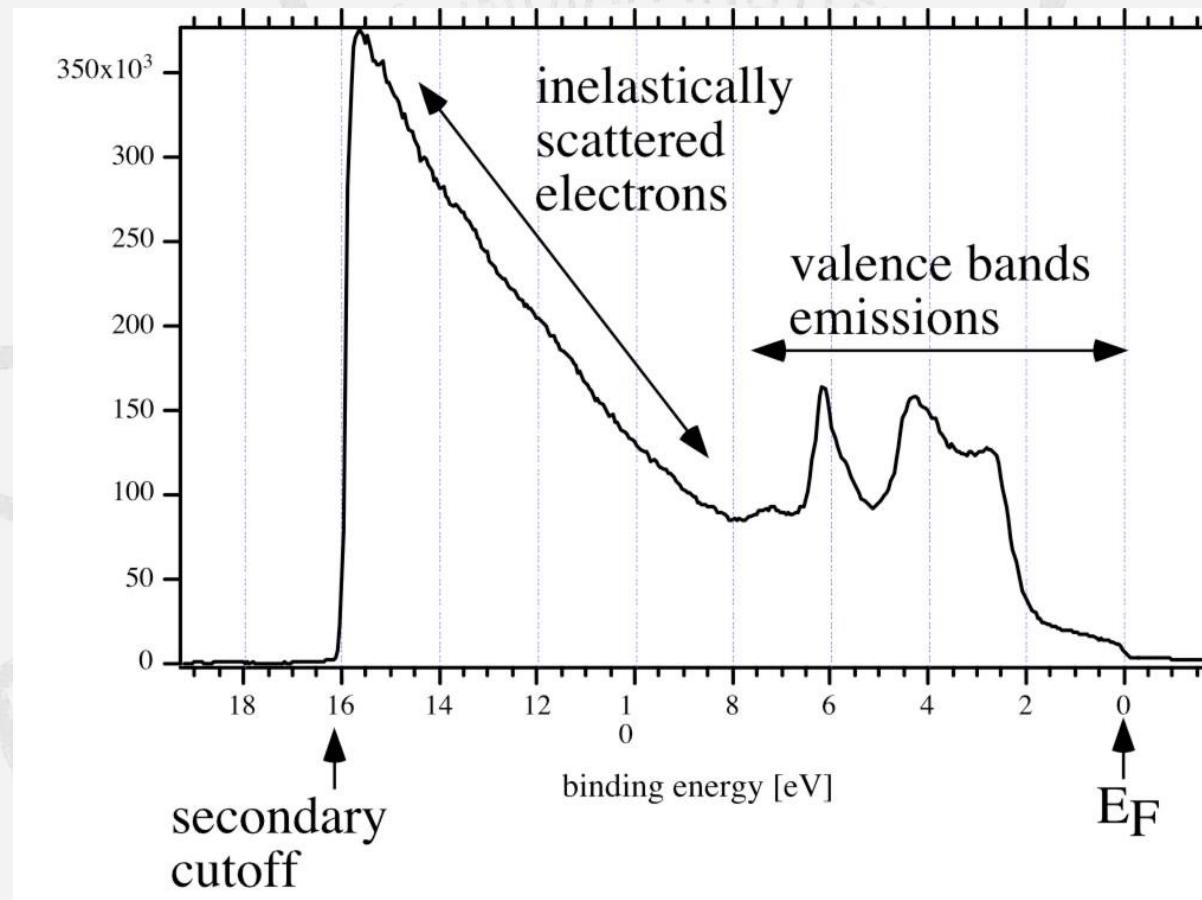
Photoemission from Valence Bands

Photoelectron spectroscopy is the most powerful and versatile technique to study the **electronic structure** of the valence bands in atoms, solids and molecules (ionization energy of molecules, HOMO).

This PE process depends on parameters suchas:

- Emited electron parameters
- Kinetic energy
- Emission angles
- Spin polarization
- Incident photon parameters
- Photon energy($h\nu$)
- Angle of incidence
- Polarization

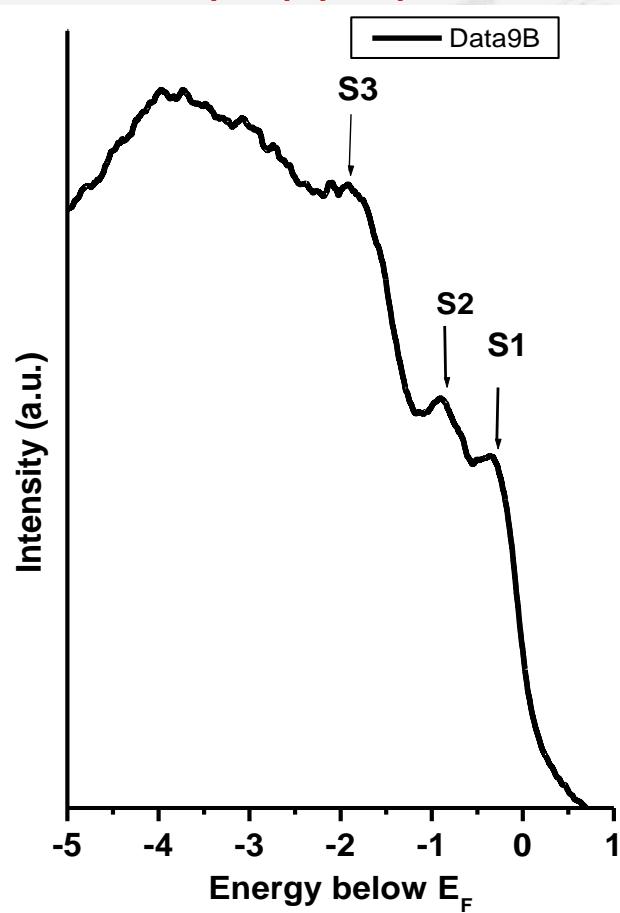
UPS study: Example



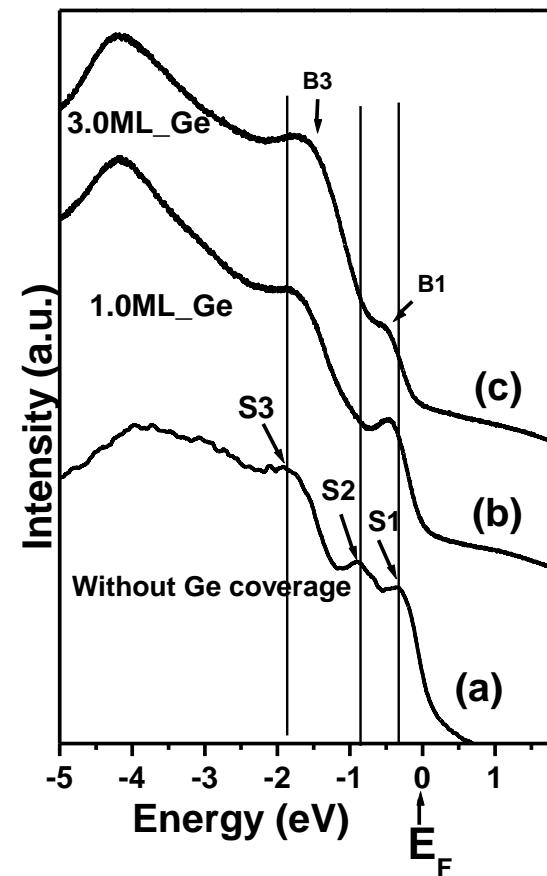
UP-spectrum of Au surface.

Effect of Ge coverage on Si(111)

Si(111)-(7x7)



Si(111)-Ge



S1= adatom state S2= Rest atom state, S3= Back-bond state

References: UPS

- Carlson T.A., "Photoelectron and Auger Spectroscopy" (Plenum Press, 1975)
- Rabalais J.W. "Principles of Ultraviolet Photoelectron Spectroscopy" (Wiley 1977)
- Turner D. W. Molecular Photoelectron Spectroscopy (Wiley, 1970)
- Baker A.D. and Betteridge D. "Photoelectron Spectroscopy. Chemical and Analytical Aspects." (Pergamon Press 1972)

Microscopy

**Atomic Force Microscopy (AFM),
Scanning Electron Microscopy (SEM)
Transmission Electron Microscopy (TEM)**

Types of Microscopy

Type	Probe	Technique	Best Resolution	Penetration	Uses and Constraints
Optical Microscopy	Visible Light	Detect reflected light (opaque samples) or transmitted light (transparent samples). Light focused using lenses.	~200 nm	Surface or volume (can probe through transparent materials)	
Near-Field Optical Microscopy (NSOM)	Visible Light	Detect reflected light (opaque samples) or transmitted light (transparent samples). Uses an aperture very close to the sample surface.	~10 nm	Surface or volume (can probe through transparent materials)	Biological samples.
X-Ray Microscopy (TXM, SXM, STXM)	X-Rays	Image derived from x-ray scattering or interference patterns. X-rays focused using a “zone plate” (Fresnel lens).	~20 nm	Surface or volume (x-rays can penetrate some materials)	Can be tuned to specific frequencies to provide element identification and mapping.
Scanning Electron Microscopy (SEM)	Electrons	Detect electrons back-scattered by the sample. Electrons focused using electromagnets.	~1 nm	Surface	Sample must be in a vacuum.
Transmission Electron Microscopy (TEM, STEM)	Electrons	Detect electrons scattered as they move through the sample. Electrons focused using electromagnets.	~0.05 nm	Volume	Samples must be <100 nm thick.
Focused Ion Beam (FIB)	Ions	Detect ions back-scattered by the sample. Ions focused using electromagnets.	~10 nm	Surface	Due to the large masses of the ions, this probe can be destructive to the surface of the sample. Therefore, it can also be used to etch the sample.
Scanning Tunneling Microscopy (STM)	Cantilever Tip	Detect the quantum tunneling current of electrons from the sample to the probe tip.	~0.1 nm	Surface	Sample must be conductive material and must be in a vacuum. Can be used to manipulate atoms on the sample surface.
Atomic Force Microscopy (AFM)	Cantilever Tip	Detect the electrostatic force between the sample and the probe tip.	~0.1 nm	Surface	Can be used to manipulate atoms on the sample surface.
Magnetic Force Microscopy (MFM)	Cantilever Tip	Detect the magnetic force between the sample and the probe tip.	~10 nm	Surface	Sample must be ferromagnetic or paramagnetic.

Imaging by Radiations

- Imaging with light
- Imaging with Electrons

Common wavelengths :

- i) Light 0.4 -0.7 micron used in optical microscope
- ii) Electrons SEM : 10 - 30 keV
TEM : 100 - 400 keV

Wavelength of electrons λ_e :

- i) Low energy electrons ($v \ll c$):

$$\lambda_e = 12.3 / \sqrt{V} \text{ A, where } V \text{ is in Volts,}$$

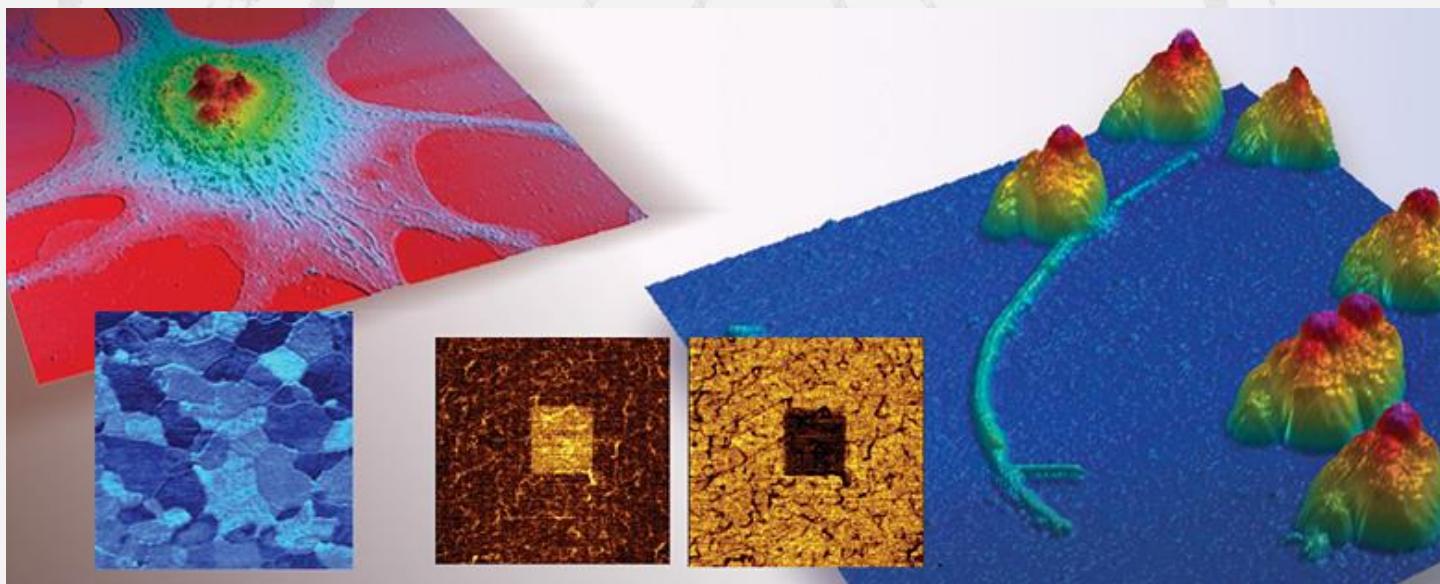
$$\lambda_e = 0.123 \text{ A for } V = 10,000 \text{ V}$$

- ii) High energy electrons ($v \sim c$):

$$\lambda_e = \left[\frac{h}{\sqrt{2m_0T}} \right] \left[\frac{1}{1 + \frac{T}{2m_0c^2}} \right]^{1/2} \text{ For electrons } m_0c^2 = 511 \text{ keV}$$

$$\lambda_e = [12.3/\sqrt{V}] [1 / \{ 1 + (T/1022) \}]^{1/2} \text{ A, where } T \text{ is in keV and } V \text{ is in volts}$$

Atomic Force Microscopy (AFM)

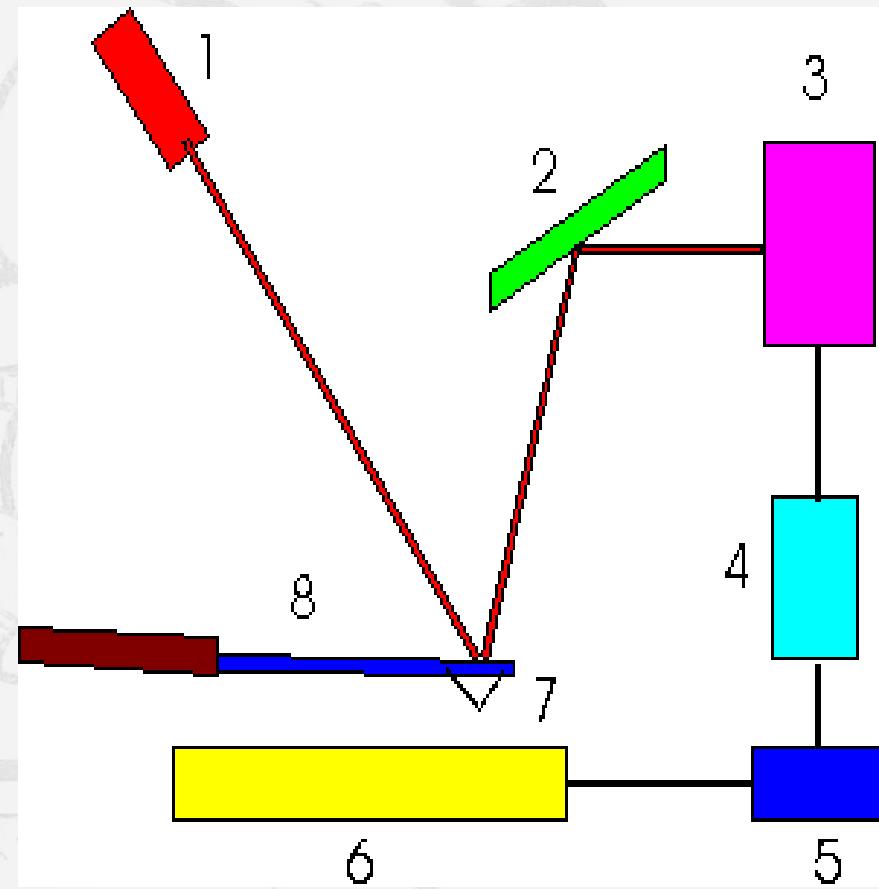


General Applications

- Materials Investigated: Thin and thick film coatings, ceramics, composites, glasses, synthetic and biological membranes, metals, polymers, and semiconductors.
- Used to study phenomena of: Abrasion, adhesion, cleaning, corrosion, etching, friction, lubricating, plating, and polishing.
- AFM can image surface of material in atomic resolution and also measure force at the nano-Newton scale.

Parts of AFM

- 1. **Laser** – deflected off cantilever
- 2. **Mirror** –reflects laser beam to photodetector
- 3. **Photodetector** –dual element photodiode that measures differences in light intensity and converts to voltage
- 4. **Amplifier**
- 5. **Register**
- 6. **Sample**
- 7. **Probe** –tip that scans sample made of Si
- 8. **Cantilever** –moves as scanned over sample and deflects laser beam



Non-Contact Mode

- Measures attractive forces between tip and sample
- Tip doesn't touch sample
- Van der Waals forces between tip and sample detected
- Problems: Can't use with samples in fluid
- Used to analyze semiconductors
- Doesn't degrade or interfere with sample- better for soft samples

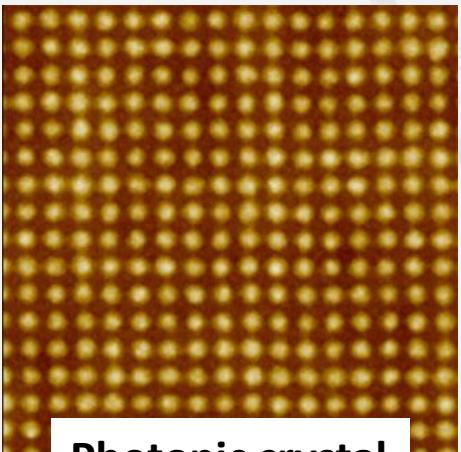
Tapping (Intermittent-Contact) Mode

- Tip vertically oscillates between contacting sample surface and lifting off at frequency of 50,000 to 500,000 cycles/sec.
- Oscillation amplitude reduced as probe contacts surface due to loss of energy caused by tip contacting surface
- Advantages: overcomes problems associated with friction, adhesion, electrostatic forces
- More effective for larger scan sizes

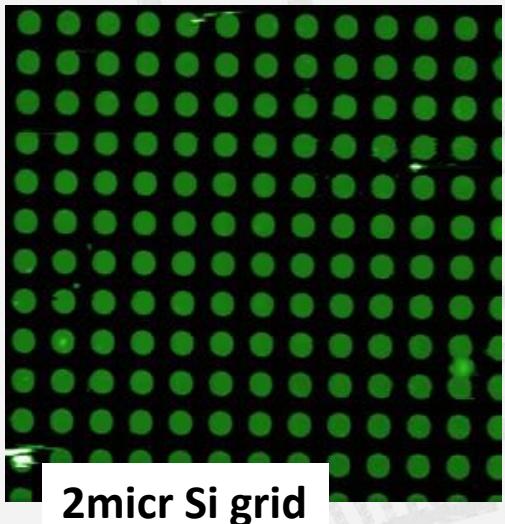
Figures of Merit

- Can measure surface features with dimensions ranging from inter-atomic spacing to 0.1mm
- Resolution limited by size of tip (2-3nm)
- Resolution of imaging 5nm lateral and .01nm vertical

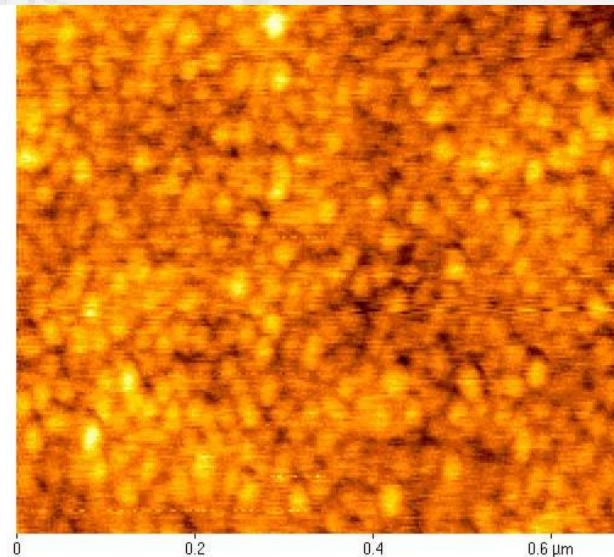
Some results



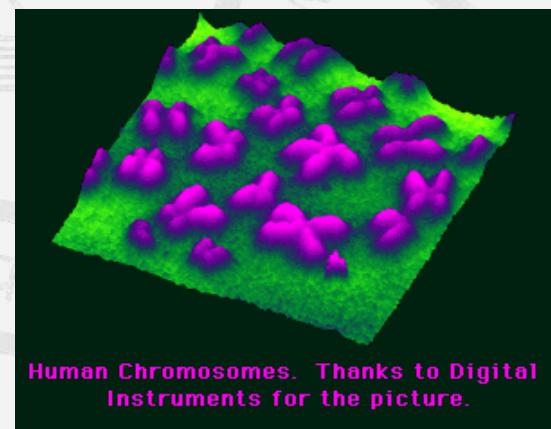
Photonic crystal



2micr Si grid

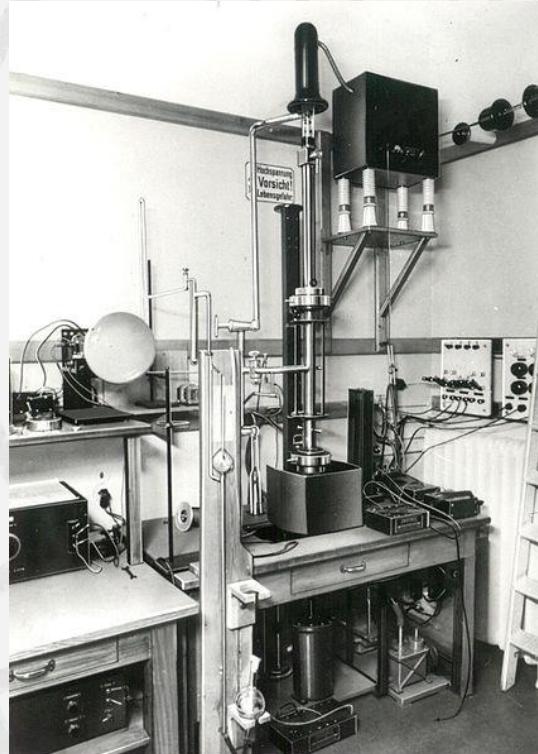


Si Nanocluster on epi- G_2O_3

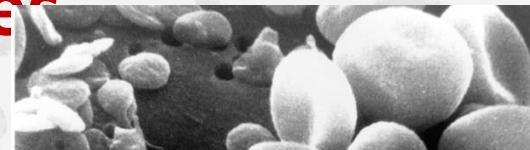


Human Chromosomes. Thanks to Digital Instruments for the picture.

Scanning electron microscope



SEM features



Topography

The surface features of an object or "how it looks", its texture; direct relation between these features and materials properties

Morphology

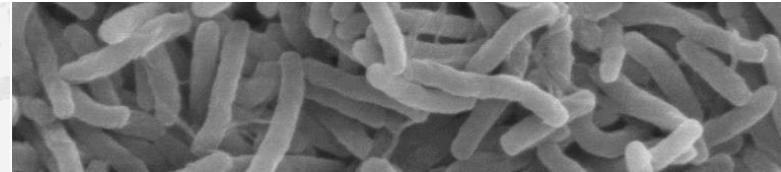
The shape and size of the particles making up the object; direct relation between these structures and materials properties

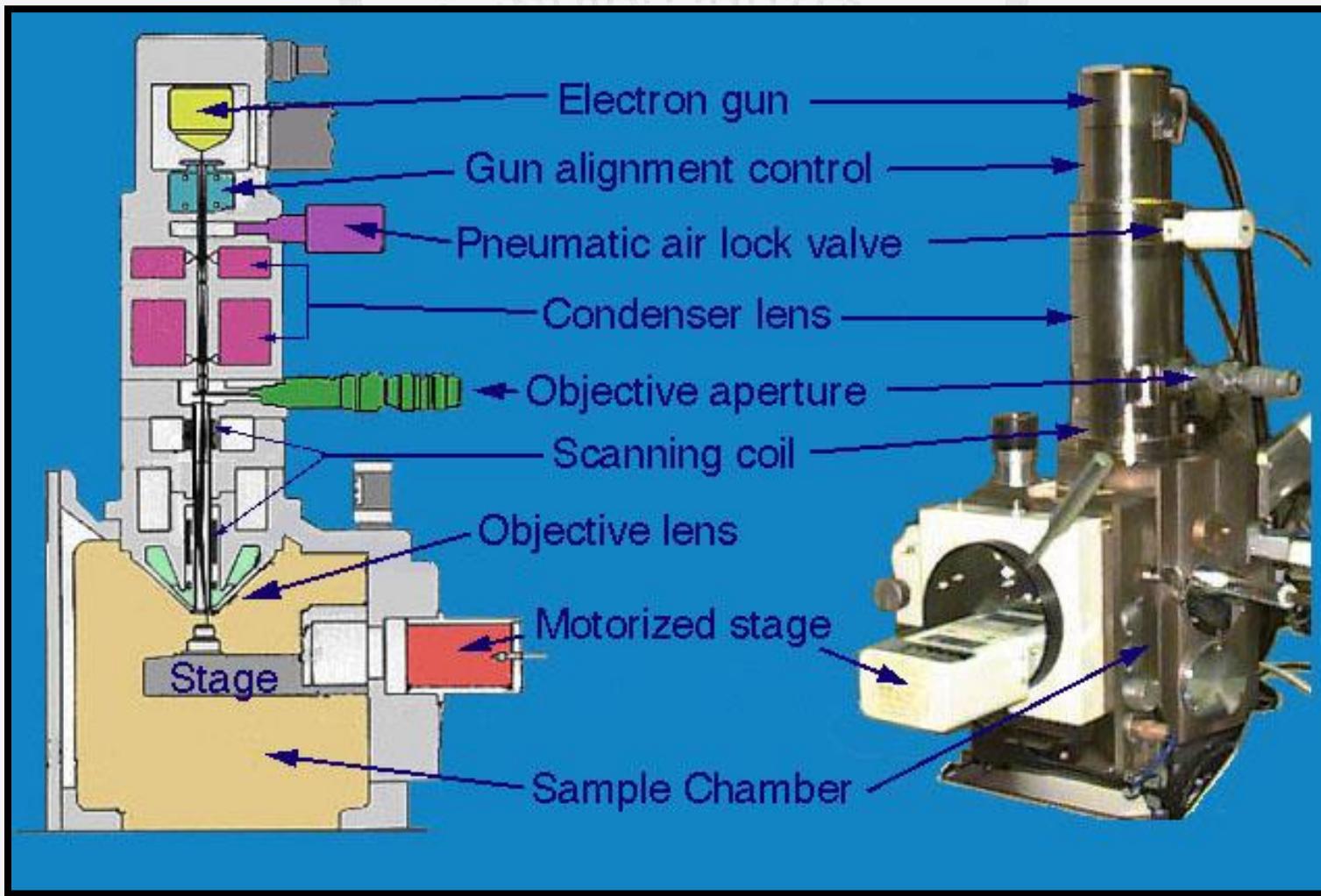
Composition

The elements and compounds that the object is composed of and the relative amounts of them; direct relationship between composition and materials properties

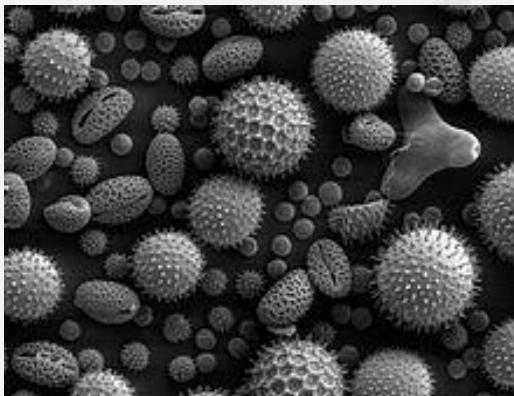
Crystallographic Information

How the atoms are arranged in the object; direct relation between these arrangements and material properties

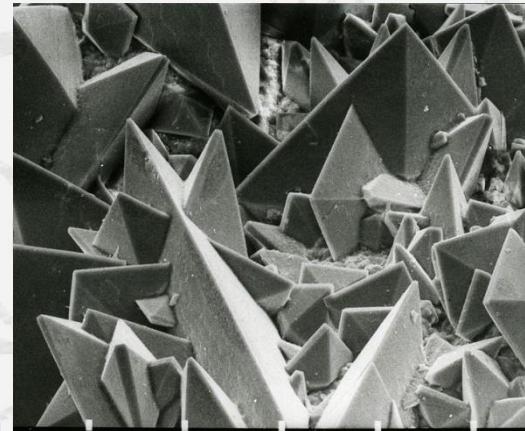




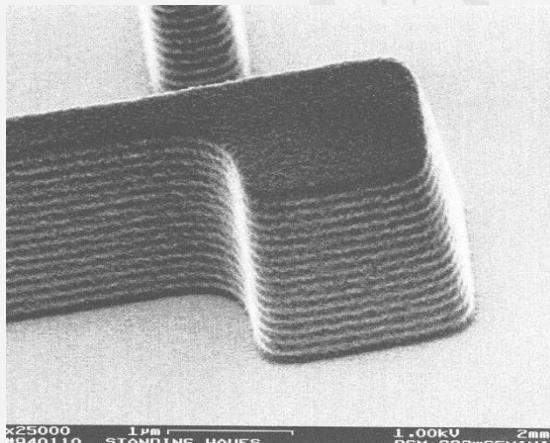
Some SEM results



These pollen grains taken on an SEM show the characteristic depth of field of SEM micrographs.



SEM image of the surface of a kidney stone showing tetragonal crystals of Weddellite



SEM image of a photoresist layer used in semiconductor manufacturing

Transmission electron microscopy



The first practical TEM, Originally installed at I. G Farben-Werke and now on display at the Deutsches Museum in Munich, Germany

TEM - transmission electron microscopy

Instrument components

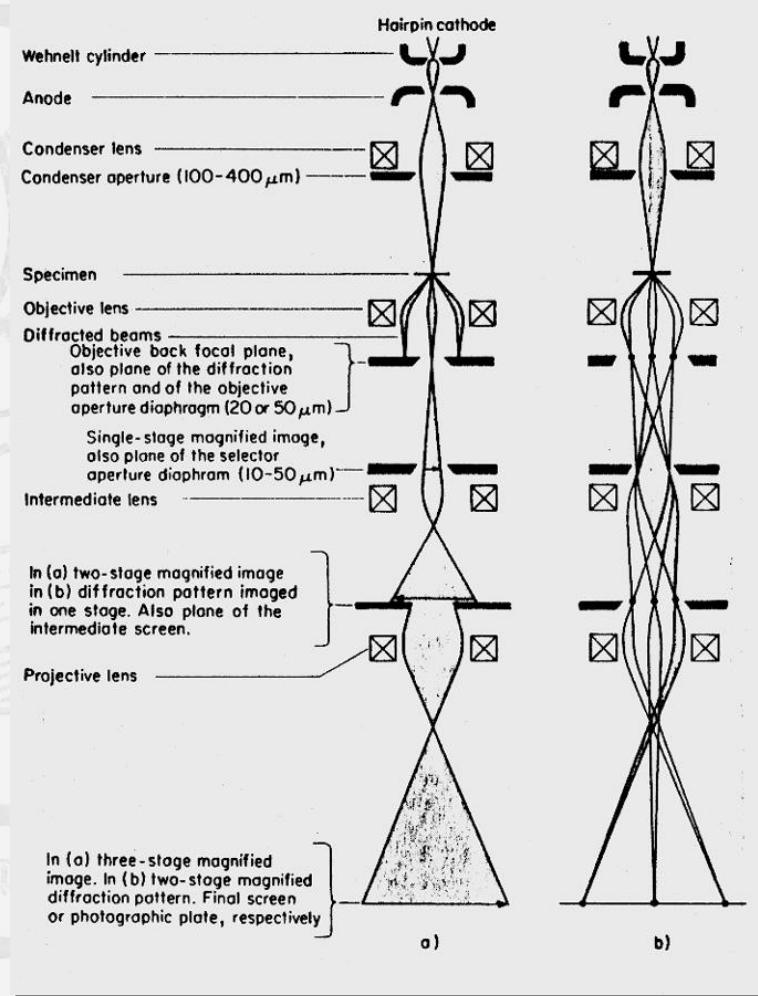
Electron gun

Condenser system (lenses & apertures for controlling illumination on specimen)

Specimen chamber assembly

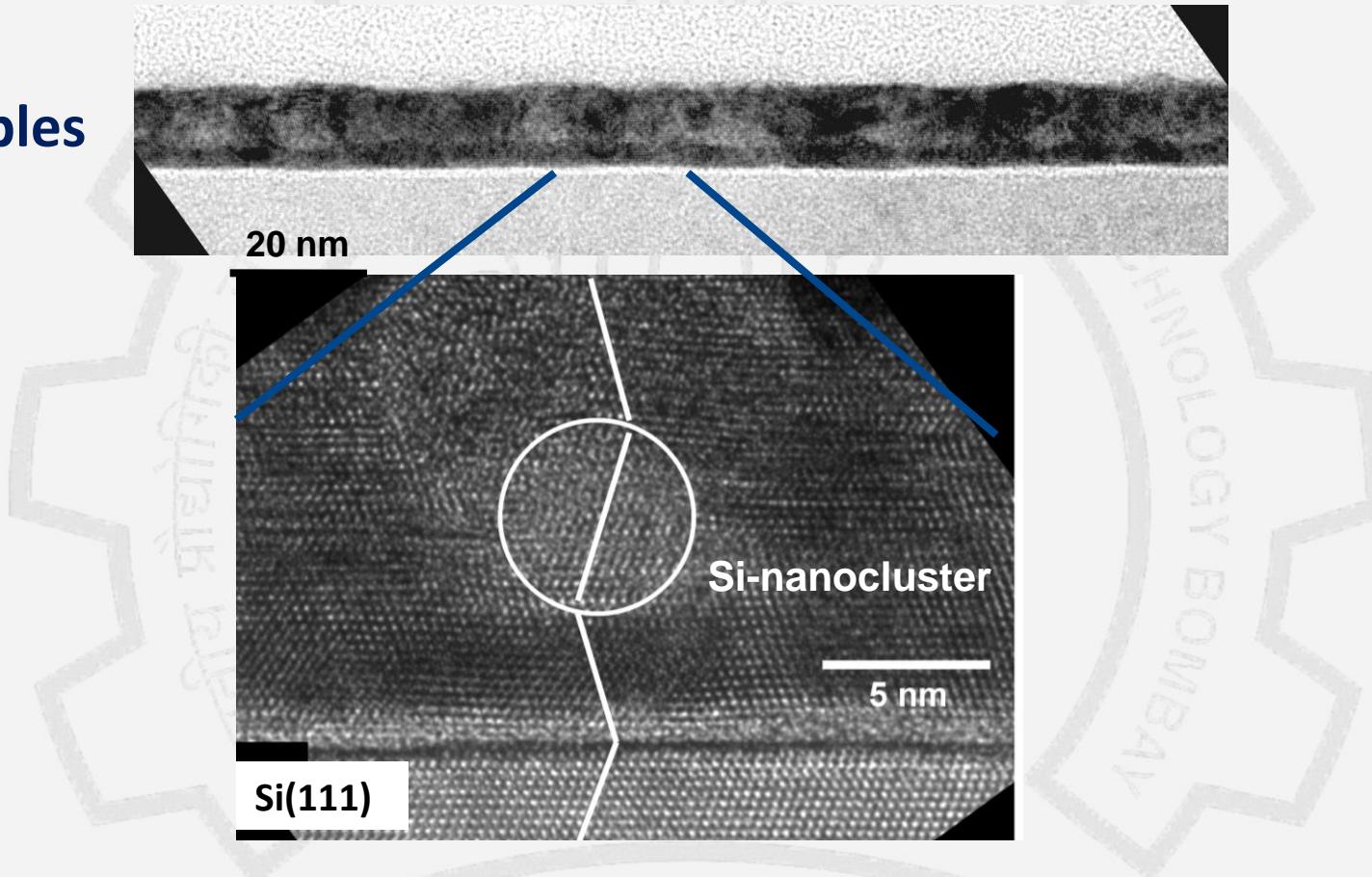
Objective lens system (image-forming lens - limits resolution; aperture - controls imaging conditions)

Projector lens system (magnifies image or diffraction pattern onto final screen)



TEM - transmission electron microscopy

Examples

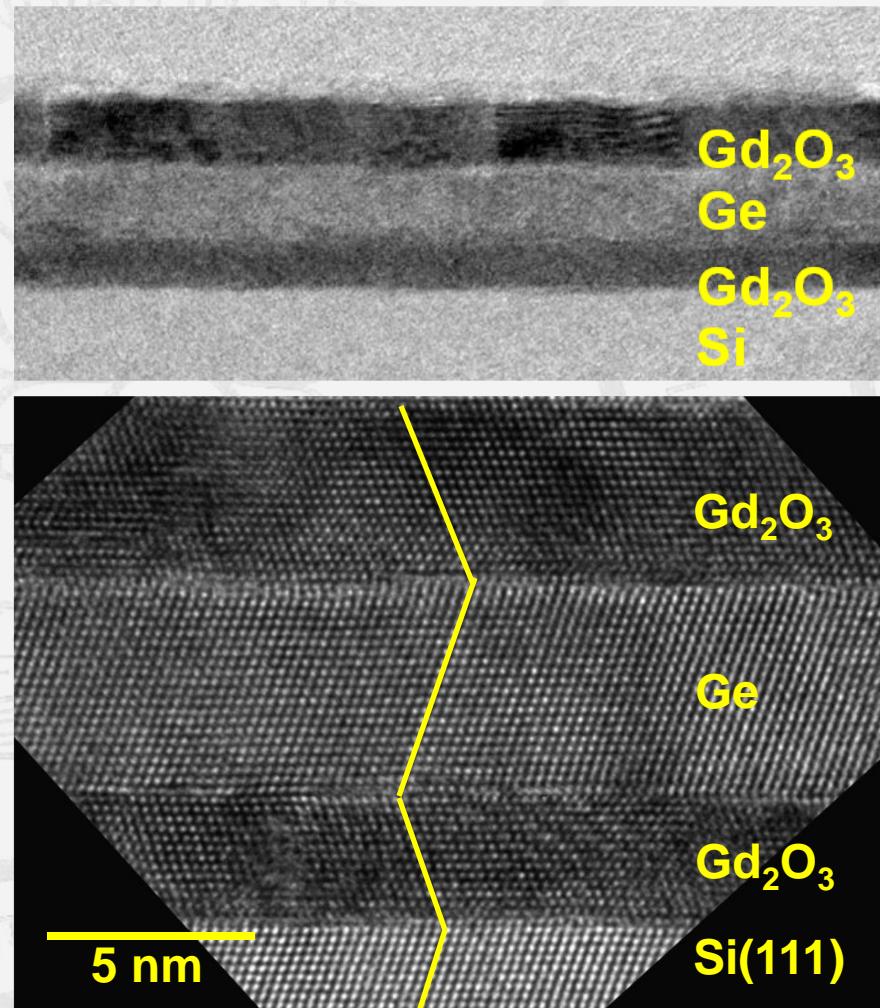


Crystalline Si Nanocluster embedded into crystalline Gd_2O_3

TEM - transmission electron microscopy

Example:

Epitaxial $\text{Gd}_2\text{O}_3/\text{Ge}/\text{Gd}_2\text{O}_3$
Heterostructure on Si
substrate



A. Laha, Nanotechnology 20, (2009) 475604

References (Microscope:

- McMullan, D. (1995) Scanning electron microscopy 1928–1965. *Scanning* 17, 175–185
- Smith KCA, Oatley, CW (1955) The scanning electron microscope and its fields of application *J Appl Phys Vol. 6*, pp. 391-399.
- Fultz,B and Howe, J (2007). *Transmission Electron Microscopy and Diffractometry of Materials*. Springer.
- Egerton, R (2005). *Physical principles of electron microscopy*. Springer.
- http://en.wikipedia.org/wiki/electron_microscope