Aalto University
School of Chemical
Technology

Properties and characterization

CHEM-E5125Thin Films Technology

2019

Jari Koskinen

Contents

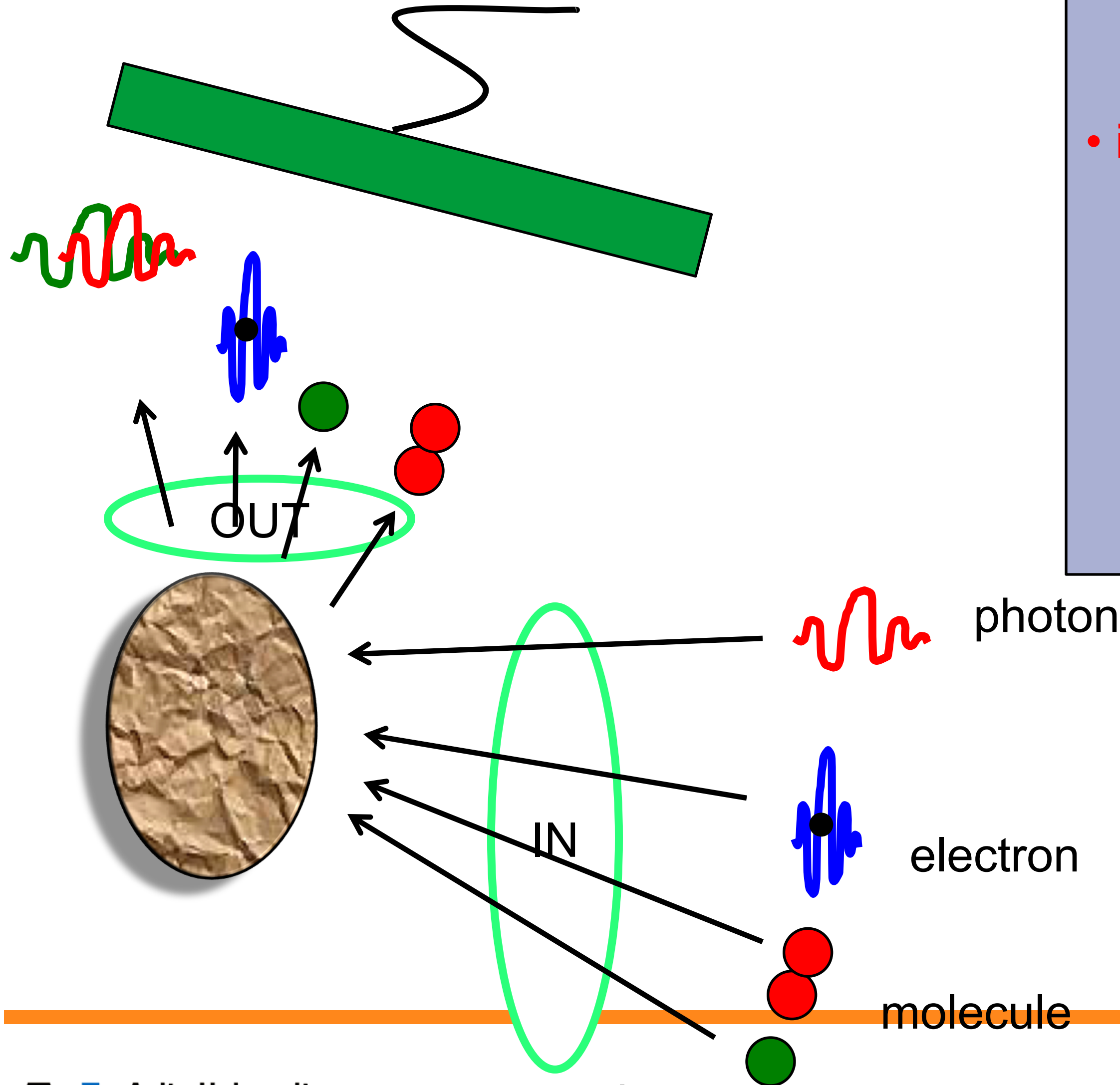
- Thin film properties
- Wealth of methods – MATRIX
- Scattering
- Thickness - profilometry
- Composition – EDS, WDS, SIMS, RBS, ERDA, GDOES
- Microstructure –XRD, TEM
- Bonding – ESCA, RAMAN
- Topography - ADM
- Electrical conductivity – four point probe
- Mechanical properties - indentation
- Optical transmittance

Characterization matrix (part of it)

		Strucutre/composition		Strucutre/composition				Surface				Interface	Mechanical				Optical			Electrical	Magnetic	Tribological	Functional X
		Thickness	Density	Microstructure	Composition	Defects	bonding (chemistry)	surface structure	surface energy	surface topography	surface contamination	misfit, strucutre, compos., strength	Elastic modulus	internal stress	Hardness	Fracture toughness	Transmittance	refractive index	extinction coeff.				
Microscopy	TEM			X	x	X	X				X	X											
	EELS				X		X																
	e-diffraction	x		X	X	X																	
	SEM	x	x	X		x		x		x		X											
	EDS				X																		
	WDS				X																		
Diffraction	XRD			X	x	x		x						X									
	HAS							X		x	X												
	LEED							X		x	X												
X-ray spectr.	XPS				x	x	X	x			X												
	AES				X			x			X												
	UPS				x	x	X	x			X												
	EAFS				x	x	X				x	x											
	SEXAFS				x	x	X	x			x	x											
	XRR	X	X							x													
	XR fluoresence				x						x												
Ion spectr.	SIMS				X	X		x			X	x											
	RBS				X	x		x			x	x											
	ERDA				X	x		x			x	x											
	PIXE				x						x												
	GDOES				X	X		x			X	x											
Optical	RAMAN			x	x	x	x				x	x											
	FT-IR				x	x	x					x											
	Fluoresence				x	x					x												
Microprobing	AFM					x				X													
	STM					x				X										x			
	profilometry	x				x				X													
Mechanical	Nanoindenter												X		X								
	MEMS												X		X								
Stress	bending													X									
Adhesion	scracth											X											
	pull test											X											
	indentation																						

Scattering experiment

- particles → in vacuum !!
- elastic scattering
  - mass, density
  - diffraction
- inelastic scattering
  - information on material
    - DOS
    - bonding
    - identification of elements
    - …



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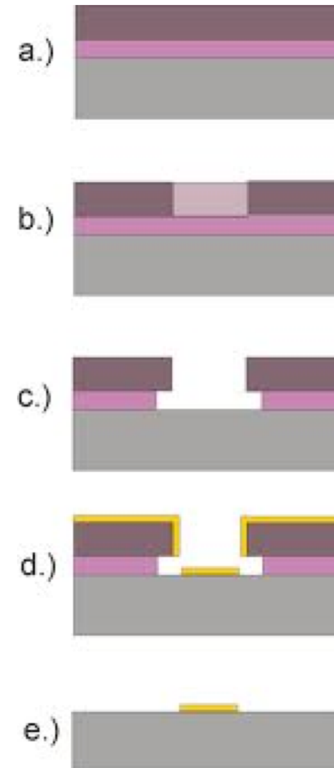
- Mechanical properties - indentation
- Optical transmittance- FTIR (???)

Film topograpby and thickness

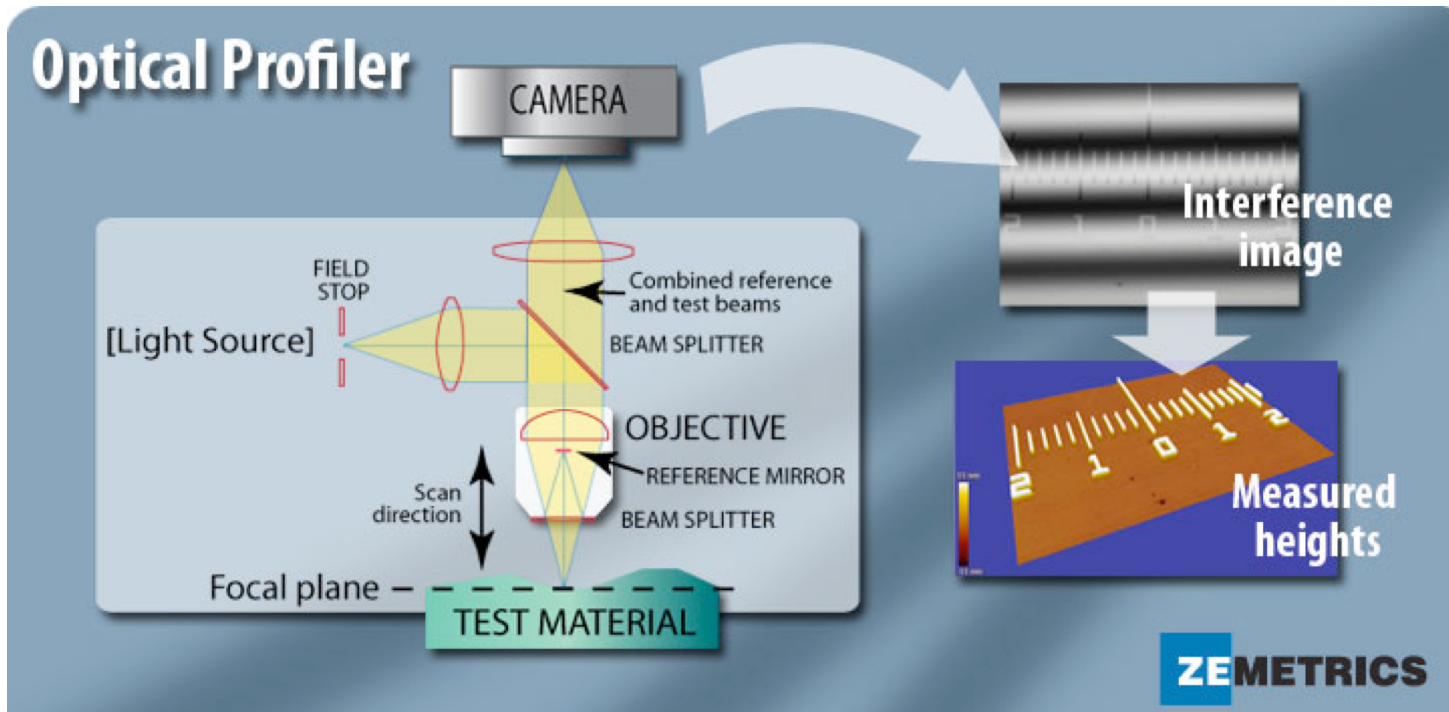
- Film thickness
  - direct measurement by definition
    - profilometer by using masked surface
    - cross section profile + microscopy
    - spectroscopy
- Contact profilometer
  - diamond tip with 1 – 50 mN load
  - tip radius 20 nm – 25 µm (typical 12µm)
  - depth sensitivity/range 0.5 nm/60 µm

Lift-off mask lithography and contact profilometry



- Simple and reliable step height
- Reflecting surfaces problematic

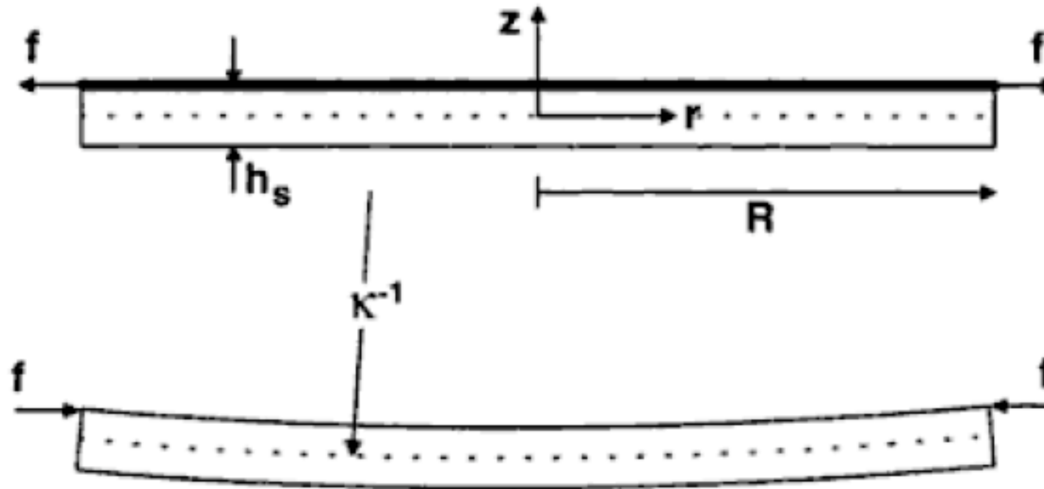
Optical profilometer



- Non contact 2D (3D surface map)
- Fast
- Reflecting surfaces problematic

Optical Profilometer

Internal stress of thin film- substrate curvature by profilometer



Stoney equation substrate modulus

$$\sigma_m = \frac{M_s h_s^2}{6 h_f} \left\{ \frac{1}{\rho_2} - \frac{1}{\rho_1} \right\}$$

radius of curvature after and intially

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Composition

- What elements present in the film
- Depth distribution
- Interface
- Often a direct feedback for deposition parameters
  - gas ratio, target composition etc.

SEM EDS and WDS

- Microanalysis EDS and WDS (material of an other course)

Excerpts from lectures in X-ray microanalysis

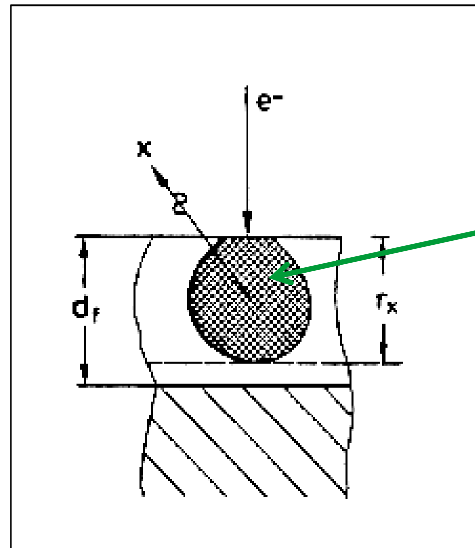
Thin surface layer analysis by SEM + x-ray microanalysis (EDS or WDS)

E. Heikinheimo

Aalto - Dept. of MS & E - 2011

Aalto University
School of Chemical Technology

Thin-film analysis (I)



Excitation volume

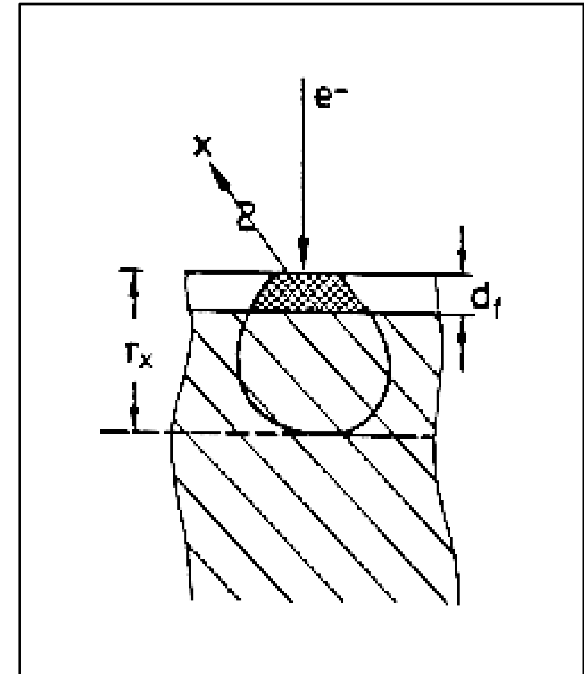
* Thickness of surface film $d_f > r_x$, substrate does not influence

* Film can be processed as bulk, with normal matrix correction program

* d_f = 0.2…2 µm (e.g. by adjusting beam energy)

Thin film analysis (II)

- $d_f << r_x$, $d_f > 1$ nm
- Substrate signal is decisive
- There can be several films on top of each other: "sandwich structure"
- Thin-film software is needed, which is based on calculating $\Phi(\rho z)$- function (amount of generated radiation) as function of depth; a hypothesis of studied film structure is needed
- In principle thickness and composition of film is obtained from both film and substrate signal (checking possibility).
- Non-destructive method, same sample can be analysed by other methods, e.g. RBS.



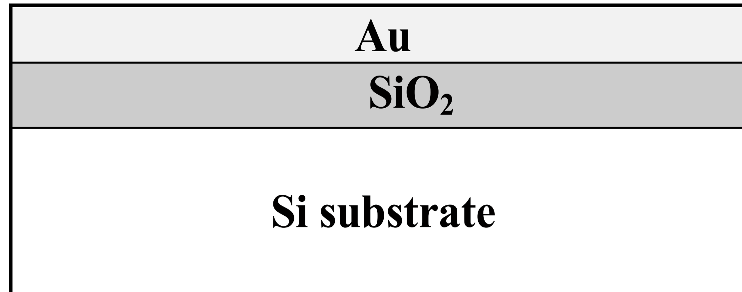
Thin-film analysis (IV)
Detection limit (DL)

El.	Line	Substr.	E_0	P/B	B	DL (nm)
Cr	K	Si	20	268	90	0.20
Ti	K	Si	20	400	54	0.33
Ag	L	Si	20	186	25	0.46
C	K	Fe	10	151	36	1.60
Mg	K	Fe	20	1001	26	0.29
Al	K	Fe	20	1786	24	0.18
Si	K	Fe	20	1538	22	0.26

Even single atom films (monolayers) can be observed!

NB. Detection limit $\neq$ analysable limit

Thin-film analysis (V)
Example 1



2 samples

Measuring k-ratios with 5, 7, 10 and 15 keV (to increase accuracy)
To define thickness measurement at one voltage is enough

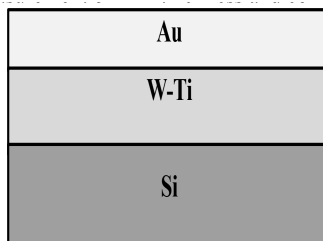
Layer	Film thickness / nm (sample #1)	Film thickness / nm (sample #2)
Au	11.1	10.9
SiO_2	18.0	67.6

k-ratio measurements with EPMA,
calculations with PC-based software (StrataGem)

NB. Au-layers are equally thick in reality (were coated simultaneously)

Thin-film analysis (VI)
Example 2

Simultaneous measurement of composition and thickness, two films on top of each other on Si-substrate

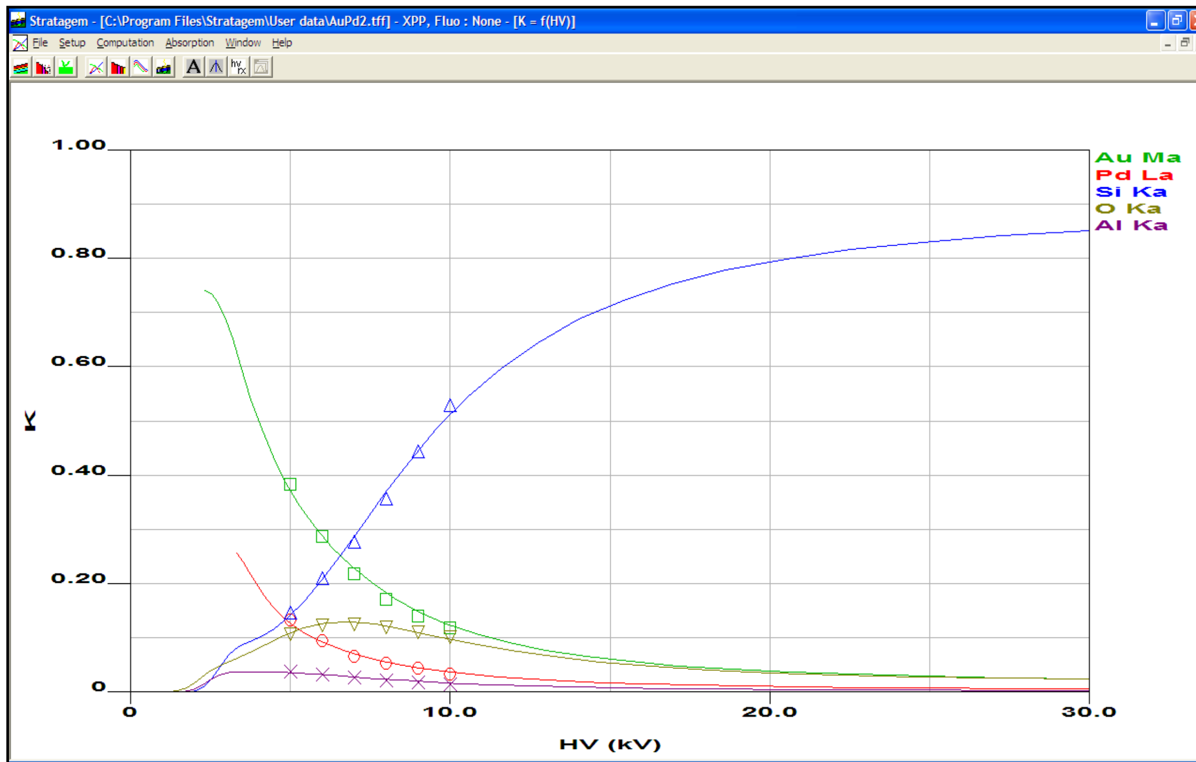


4 measurings 12 keV Au-M, W-M, Ti-K, Si-K, massive pure element standards
→ StrataGem

ρz (Au)	ρz (W-Ti)	Au wt.-fract/	W wt.-fract/	Ti wt.-fract/	Si wt.-fract/
22.7	120.3	1.0000	0.9520	0.0480	1.0000
20.3	122.8	1.0000	0.9492	0.0508	1.0000
25.9	119.7	1.0000	0.9484	0.0516	1.0000
20.7	118.5	1.0000	0.9474	0.0526	1.0000

Thicknesses of films: Au 11.6 nm (14), W-Ti 64.7 nm (60). In parentheses values from manufacturer who did not know the composition of W-Ti film. Reproducibility is good.

Thin-film analysis (VII) Example 3



Microcircuit: Contact pad of current pump

Layer configuration: Au-Pd/Al_2O_3/Al/SiO_2/Si

Measurements at 10, 9, 8, 7, 6 ja 5 kV → k-ratios for all elements present → StrataGem

StrataGem-software:

1. Au 79.0 p% (77 p%)

Pd 21.0 p% (23 p%)

Thickness = 14.3 nm (30 nm)

2. Al_2O_3 = 14.9 nm (1-2 nm)

3. Al = 0.6 nm (20 nm)

4. SiO_2 = 332 nm (?)

(supposed values in parentheses)

Thin film analysis (VIII) Requirements for samples

- Planar
- Homogenous in lateral direction (0.5...5 μm)
- Endures effect of e-beam
- Endures light vacuum (10^{-5} mbar)
- *Bulk matter can be used for standards; this is an advantage compared to others*
- Preferably a conductive substrate (if not, experiment!)
- Combined thickness of surface layers < r_x (≈1 μm), i. e. substrate signal must be clearly seen

Thin-film analysis Hands-on difficulties

- Diffusion between layers, oxide layers, formation of compounds between layers = > difficulty of making correct hypothesis of real structure
- Unknown structure → very laborious, lots of measurements

  $k = f(E_o)$
- Concentration gradient can be estimated by dividing structure into several films on top of each other
- Same element present in several films on top of each other → measurements at several E_0 values (is not always enough)
- Calculations require estimating density of film, which can be difficult and accuracy suffers
- (Requirement of conductivity of substrate) Try!
- Requires its own software, e.g. StrataGem (SAMx, F)

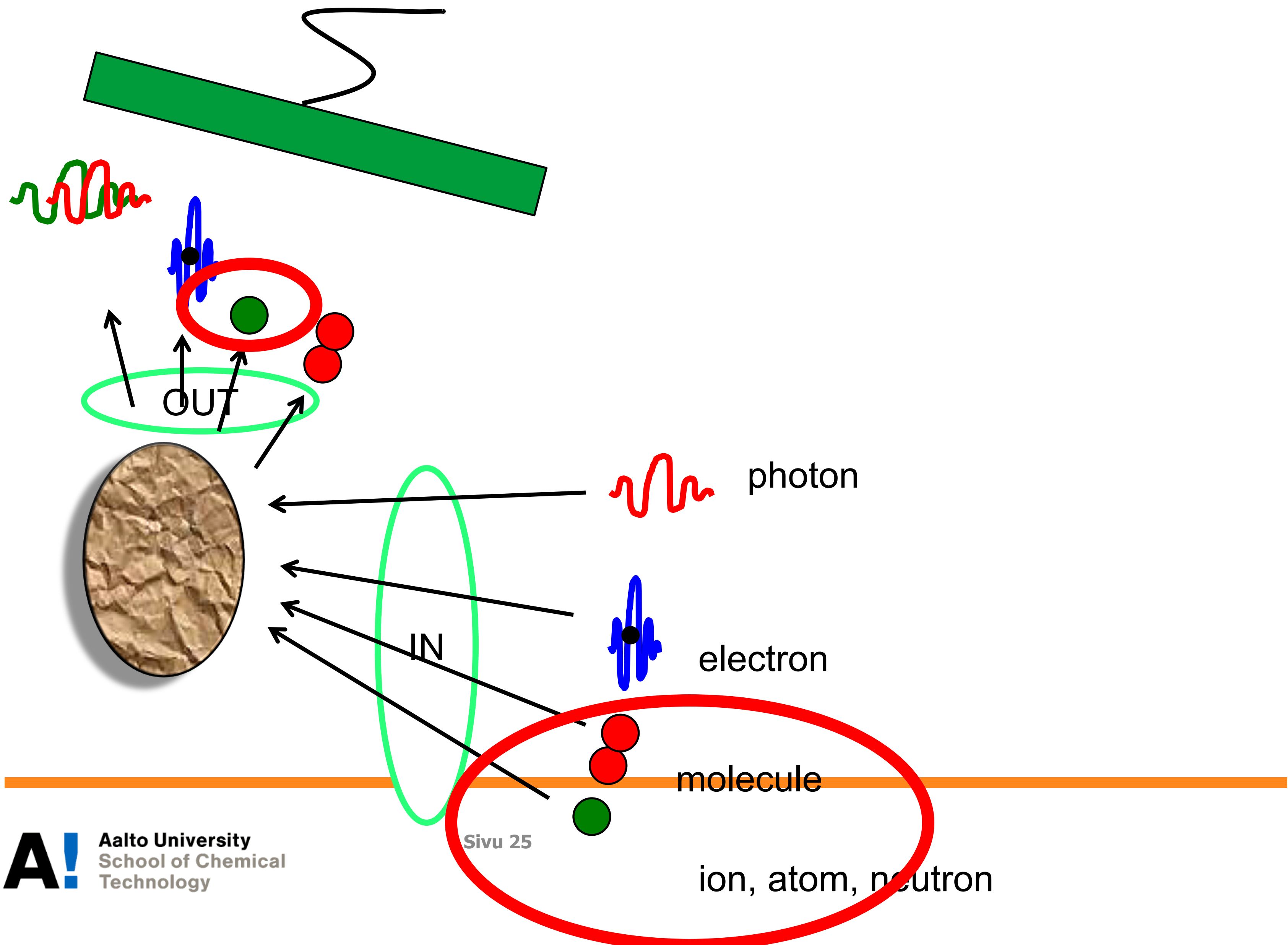
Comparing surface sensitive microanalysis methods

Method	Depth resol.	Lateral resol.	Elements	DL (ppm)	Depth profiling	Quant. accuracy
XRF	> 10 µm	> 5 mm	Z = 9+	> 10	___	1...3 %
EPMA	0.1...2 µm	> 0.5 µm	Z = 4+	> 10	nondestr.	2...5 %
SEM+EDS	0.5...2 µm	> 0.5 µm	Z = 5+	> 1000	___	3...10 %
AES	1 nm	> 0.1 µm	Z = 3+	> 1000	sputter.	10...20 %
XPS	2 nm	> 100 µm	Z = 2+	> 1000	sputter.	10...20 %
RBS	2...20 nm	> 100 µm	Z = 6+	> 1000	nondestr.	< 5 %
SIMS	2 nm	> 0.1 µm	all	< 1	sputter.	difficult

Methods complete each other, all have their strengths.

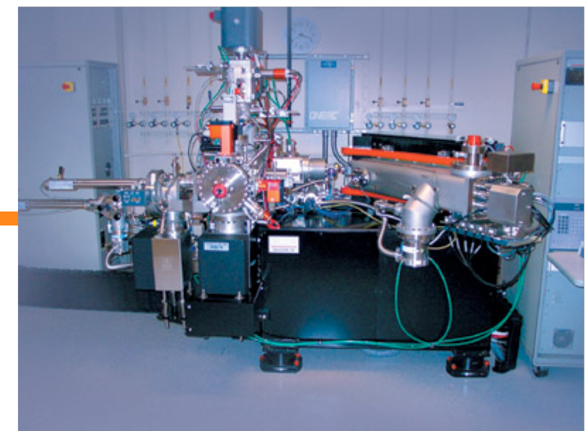
EPMA: combination of accuracy in quantification, detection limit and lateral resolution. In addition possibility to study “sandwich structure” without destroying the sample.

Scattering experiment – Ion in Ion out



Secondary Ion Mass Spectrometry - SIMS

1. Cesium ion source
2. Duoplasmatron
3. Electrostatic lens
4. Sample
5. Electrostatic sector - ion energy analyser
6. Electromagnet - mass analyser
7. Electron multiplier / Faraday cup
8. Channel-plate / Fluorescent screen - ion image detector

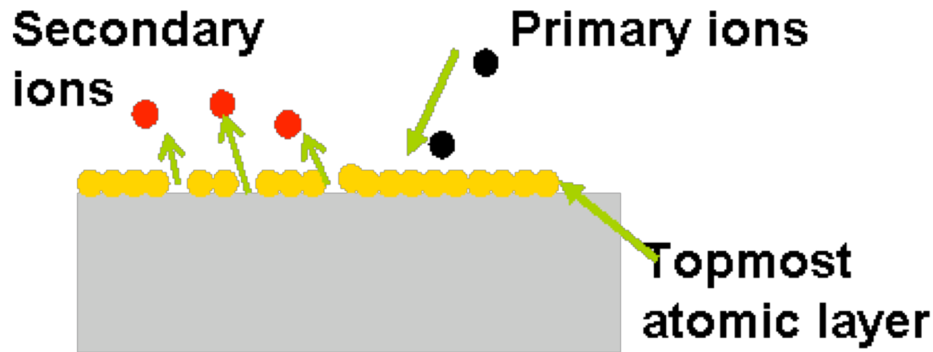


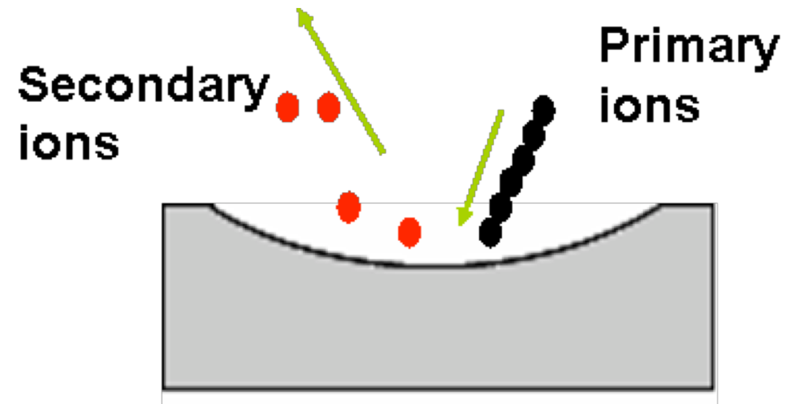
SIMS by Leena-Sisko Johansson

Secondary Ion Mass Spectrometry - SIMS

Static SIMS

Dynamic SIMS

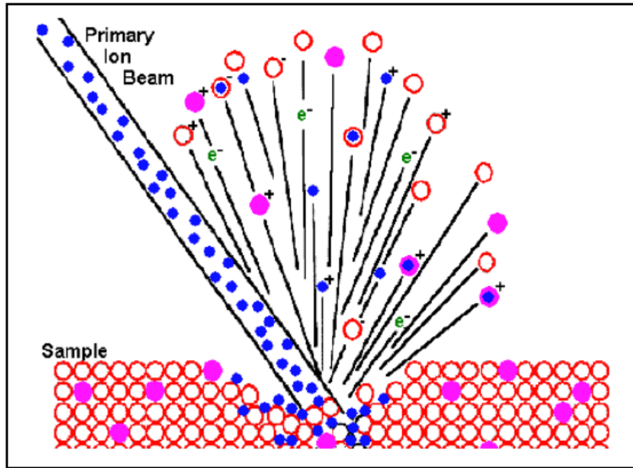


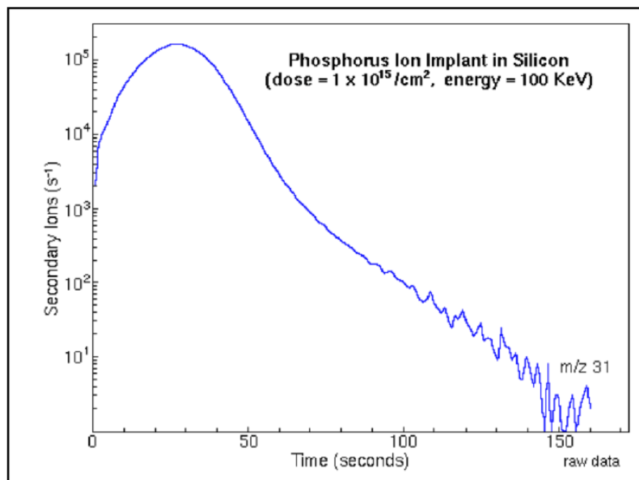


Static SIMS: Secondary ions are ejected only from the topmost atomic layer

Dynamic SIMS: Top few monolayers are removed because of sputtering caused by the high does of primary ions

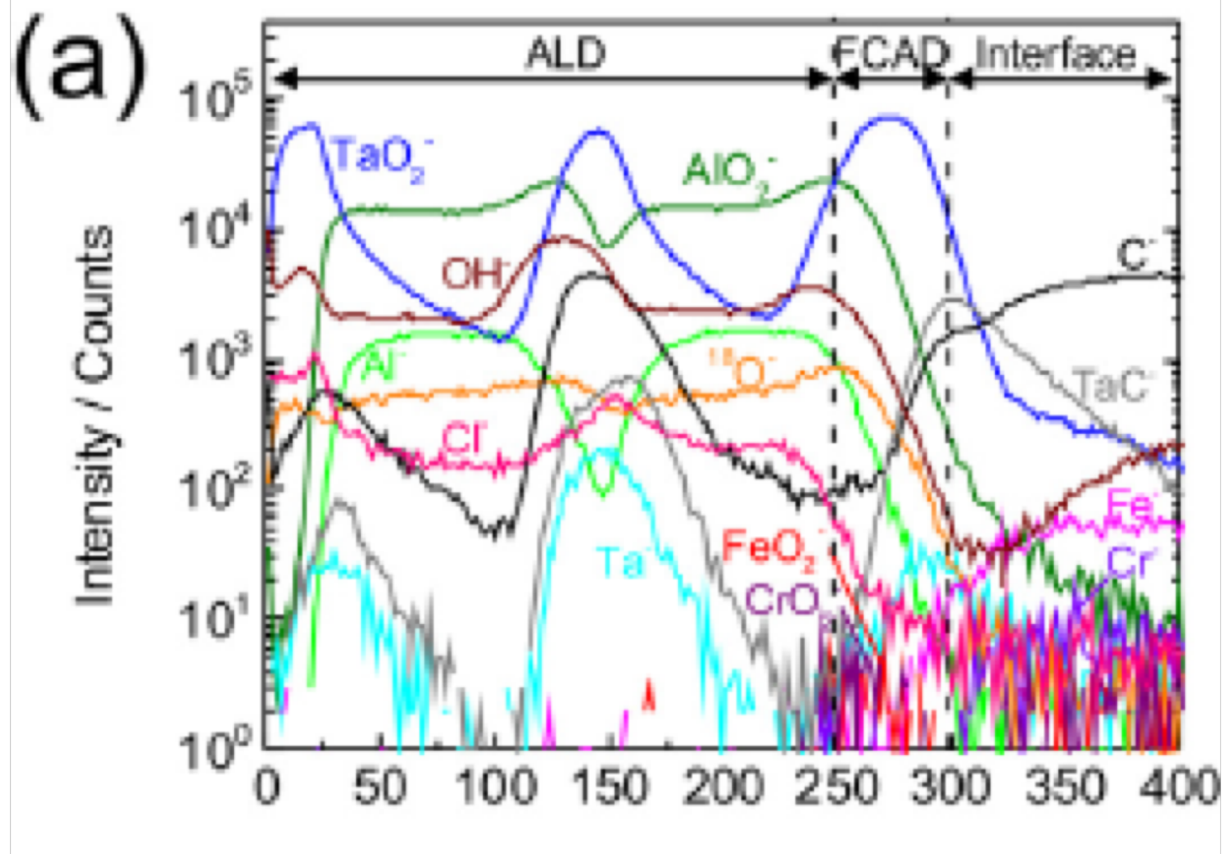
Secondary Ion Mass Spectrometry - SIMS



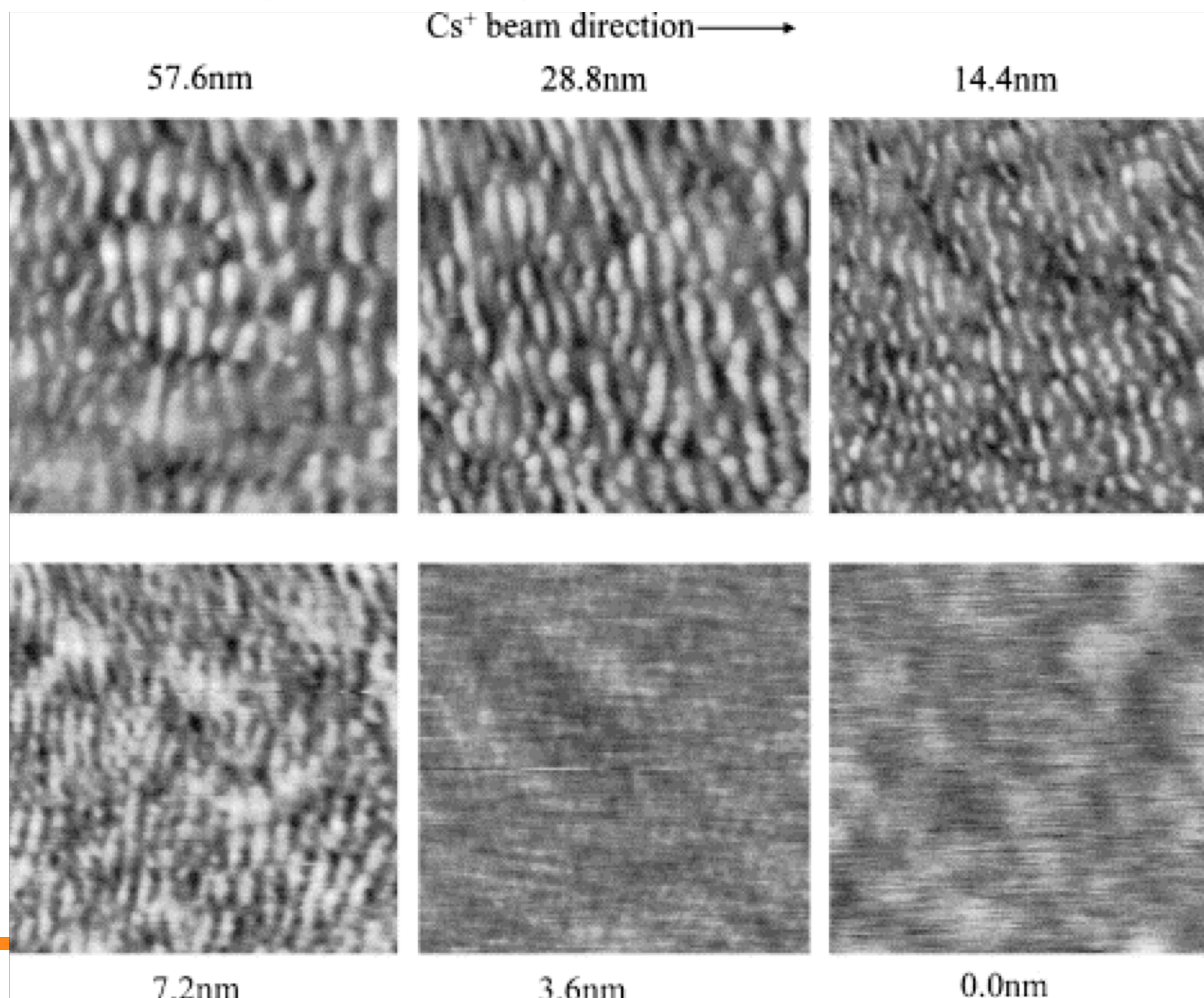


- Vacuum roughly 10^{-6} mbar
- Ions: Ar^+, O_2^+, Cs^+ (M 133) 1 – 30 keV
- sensitivity 10^{12} – 10^{16} atoms/cm^3
- beam focus down to 1 µm
- mapping of elements
- Depth profiling by sputter etching
- seconday ion yield depends on chemical composition of sample
  - reference samples with known composition necessary for quantitative analysis
- Sputtering - > mixing of composition
  - depth resolution decreases when sputtering deeper

  - http://www.youtube.com/watch?v=-7gSbasIRCU&feature=related


Before immersion
(a)
ALD
ECAD
Interface
Intensity / Counts
TaO2-
AlO2-
OH-
C-
Al-
18O-
Cl-
TaC-
Fe-
Ta-
FeO2-
Cr-
CrO2-
0
50
100
150
200
250
300
350
400

Surface roughening

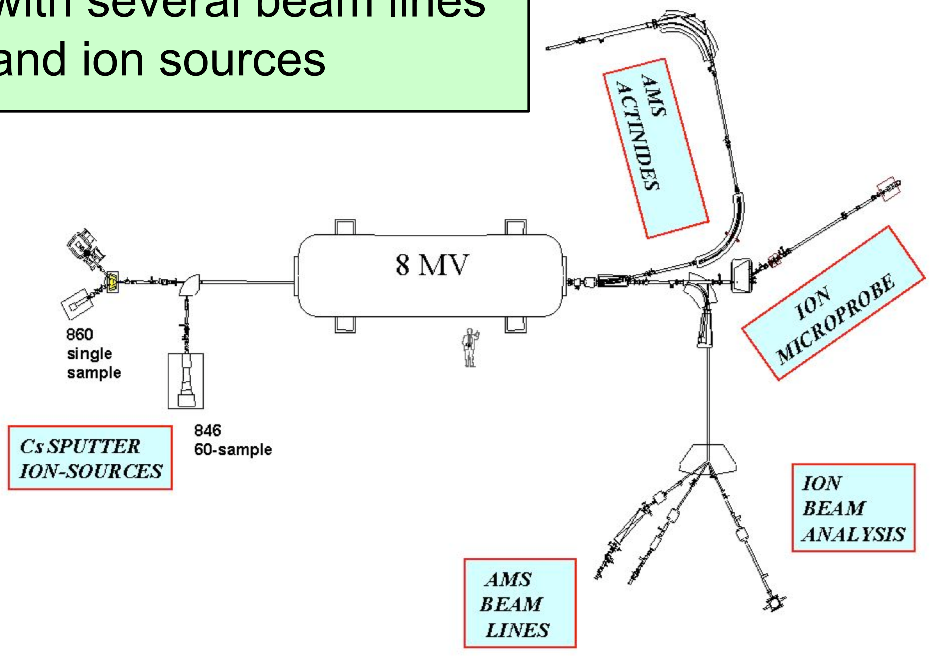


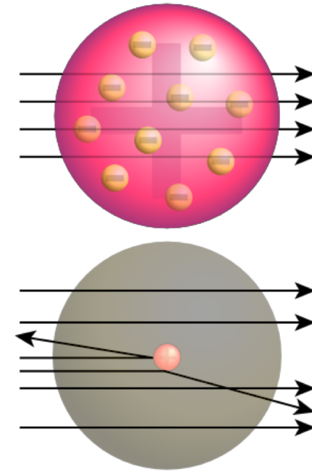
Ion beam analysis

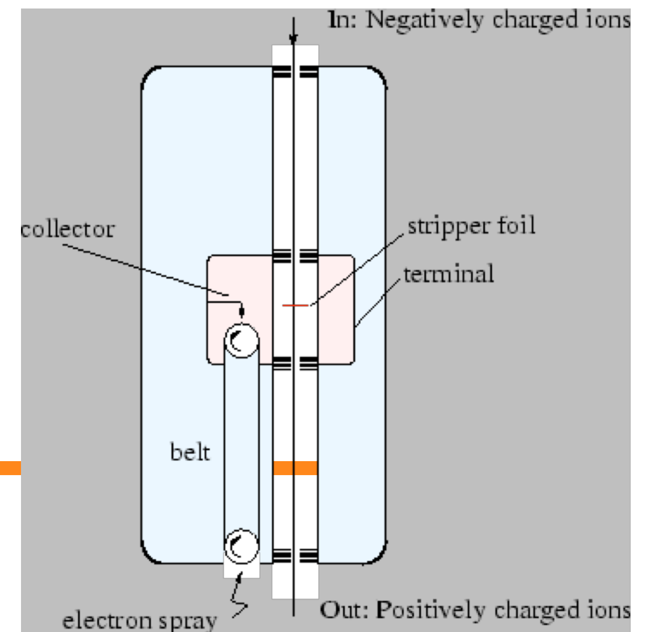
- Backscattering spectroscopy

http://www.youtube.com/watch?v=Vu-JBGP_Xzk

Ion beam accelerator with several beam lines and ion sources







Ion beam analysis

- Backscattering spectroscopy

recoil energy -> what element

atomic mass of element

Probability of recoil -> amount of element

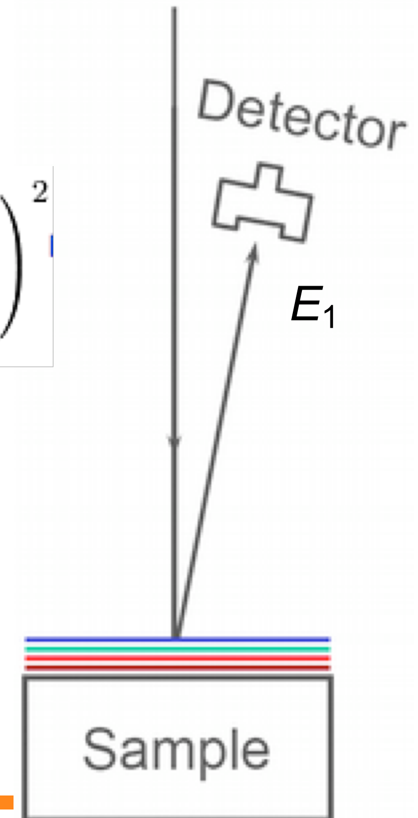
Kinetic energy loss inside material – stopping -> depth scale

$E_1 = k * E_0$

$$k = \left(\frac{m_1 \cos\theta_1 \pm \sqrt{m_2^2 - m_1^2 (\sin\theta_1)^2}}{m_1 + m_2} \right)^2$$

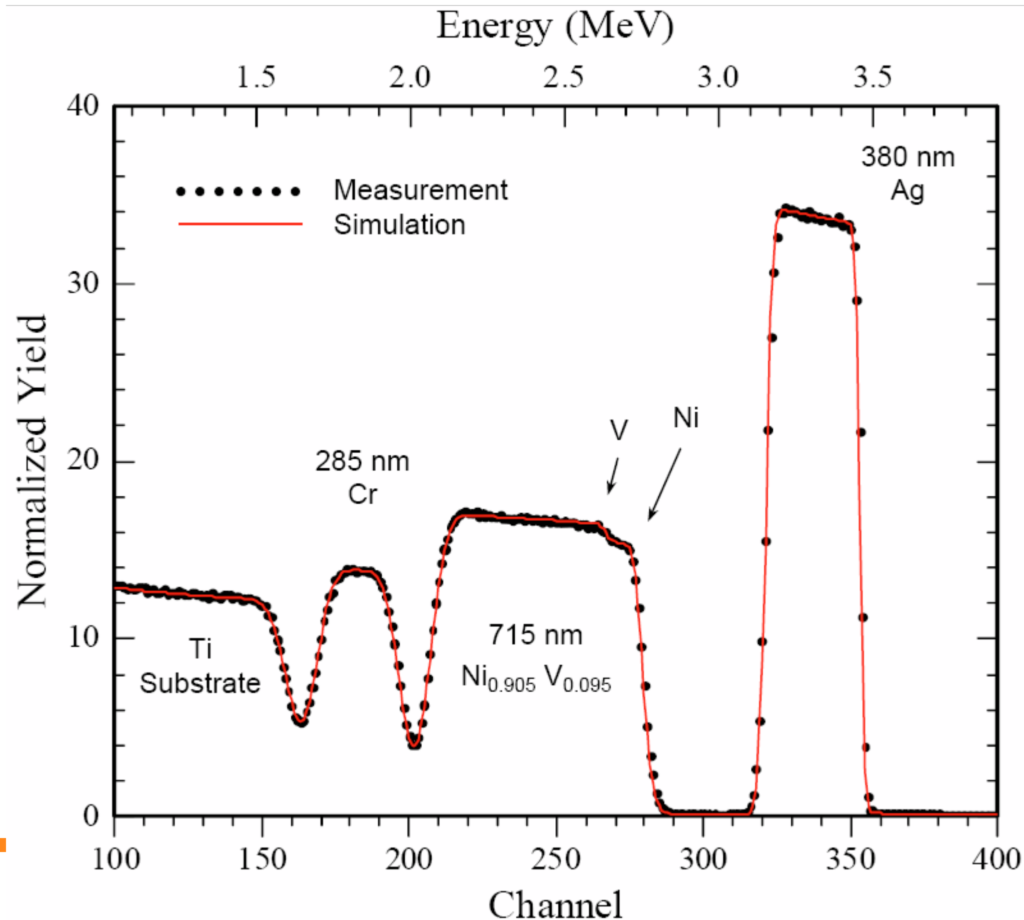
$$\frac{d\omega}{d\Omega} = \left(\frac{Z_1 Z_2 e^2}{4E_0} \right)^2 \frac{1}{(\sin\theta/2)^4}$$

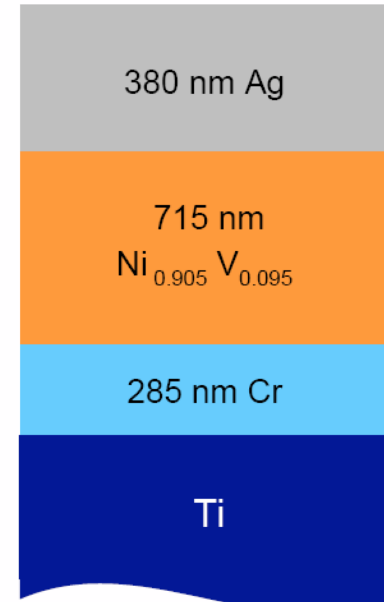
$$S(E) = -\frac{dE}{dx}$$



Ion beam analysis

- Backscattering spectroscopy

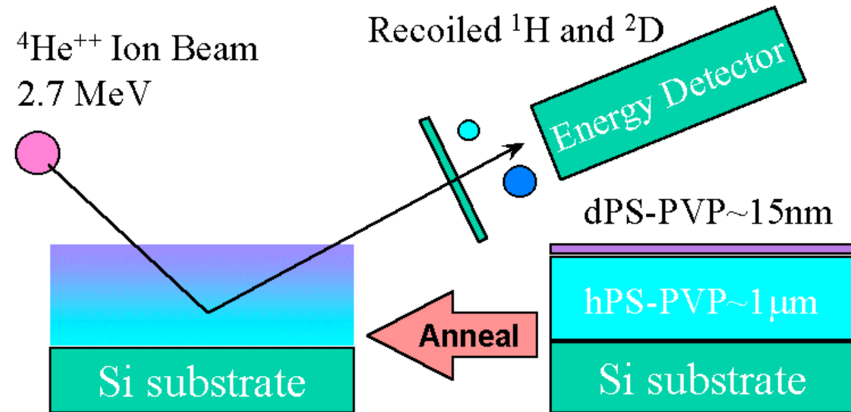




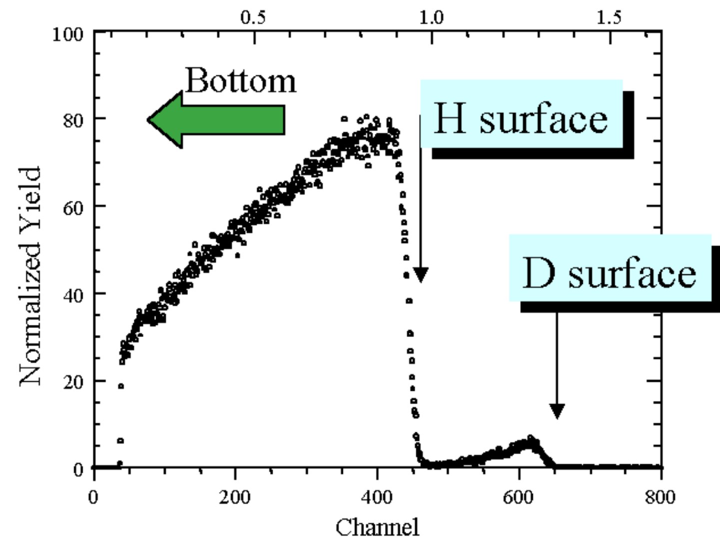
Ion beam analysis

http://www.youtube.com/watch?v=K7O3FLpXL7A&feature=related

Forward Recoil Spectrometry (FRES)



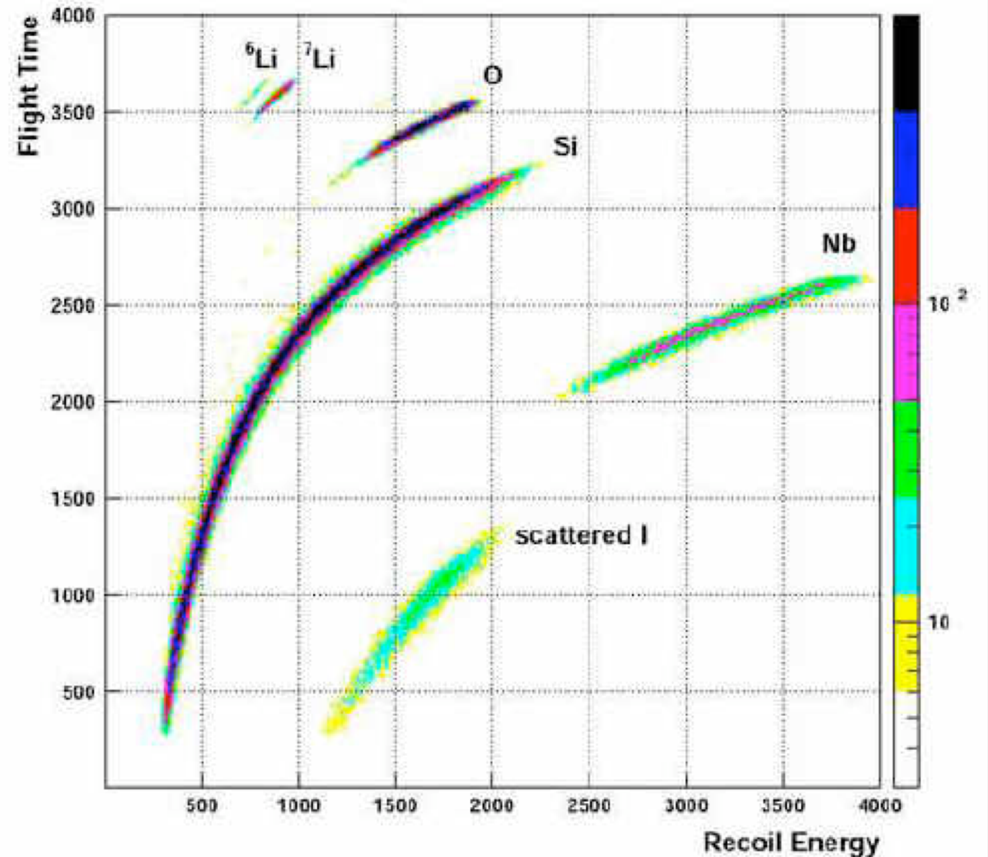
FRES measures Depth-Concentration Profile of ^2D



Ion beam analysis

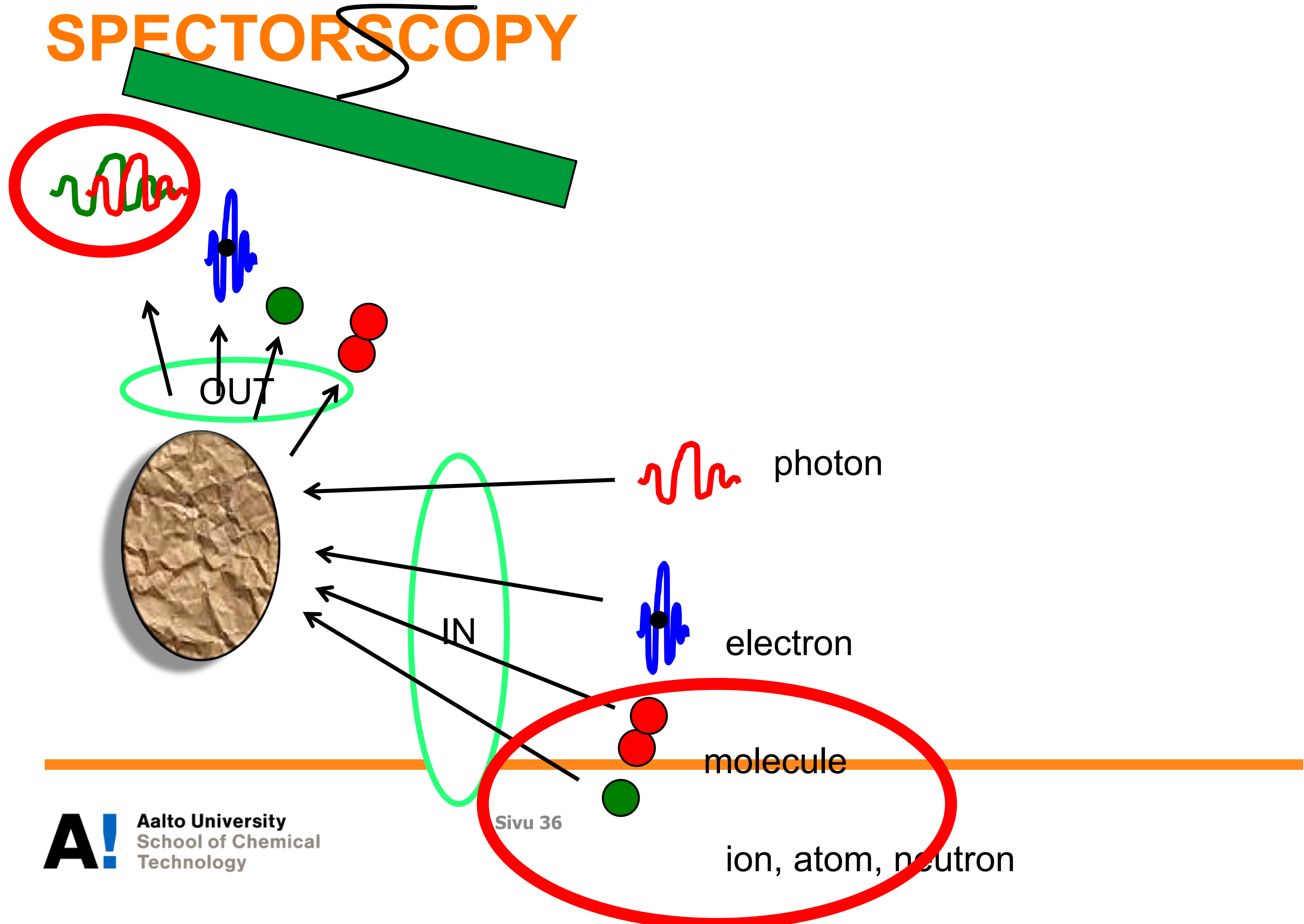
Forward recoil with heavy ions with time-of-flight detector (energy and velocity measured)

-> depth distribution of many elements in one run



(~ 100nm) lithium niobate film LiNbO3 deposited on a silicon wafer

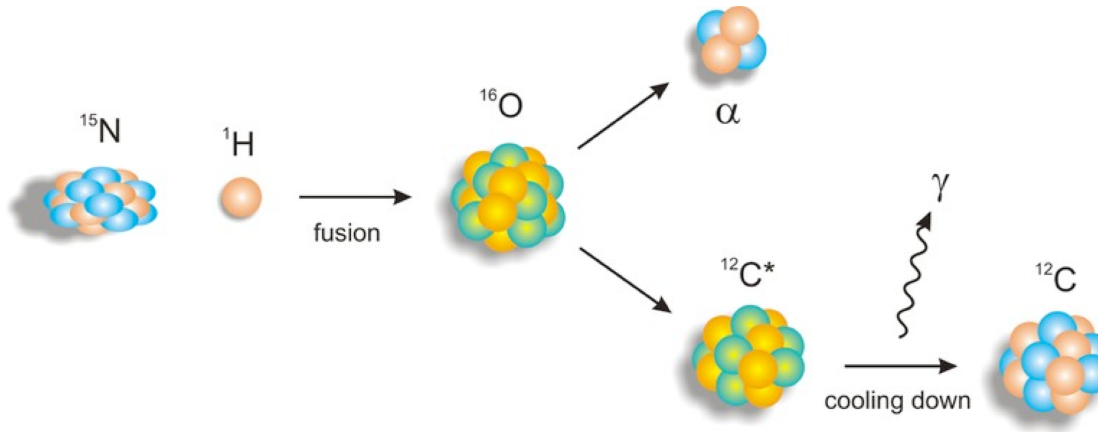
Scattering experiment – ION SPECTORSCOPY



Ion beam analysis

- Nuclear Reaction Analysis NRA

  Detection of **hydrogen** – depth distribution in surface



E_{res} = 6.385 MeV

GDOES - Glow Discharge Optical Emission Spectroscopy

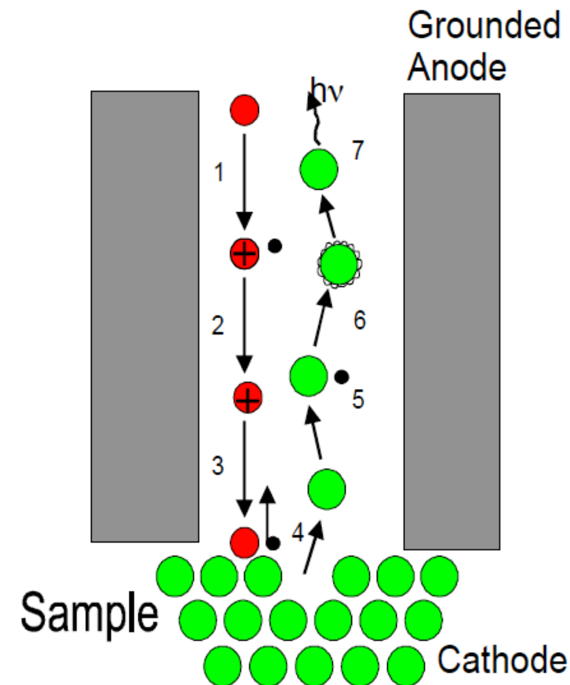
Principle of GD-OES (= GDOS)

- Sample atoms are sputtered by fast Argon ion bombardment, forming a plasma
- The plasma emission contains the typical spectral wavelength's of the elements
- Emission is registred by a simultaneous optical spectrometer with fast acquisition electronics and Windows software

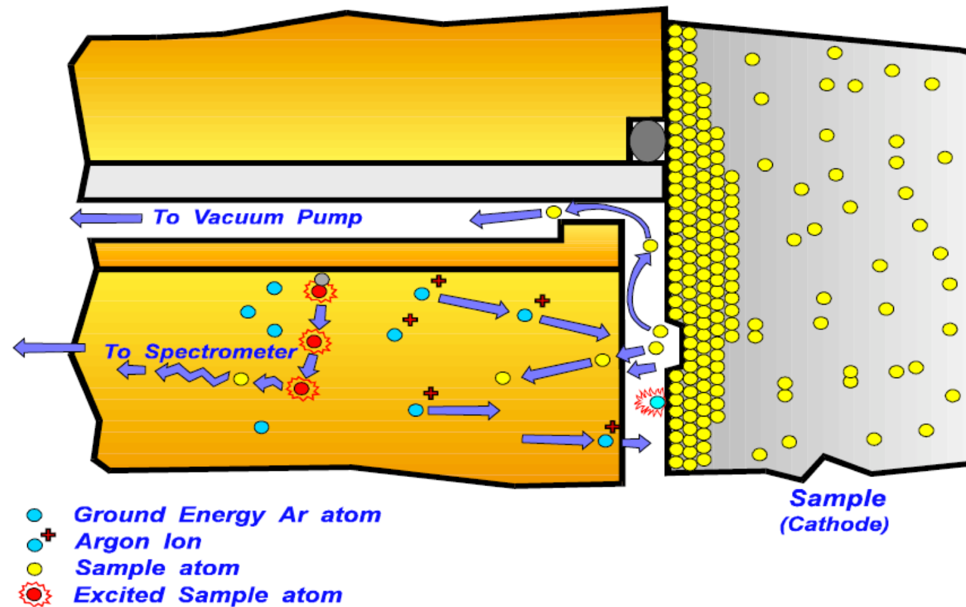
GDOES

Principle of GD-OES

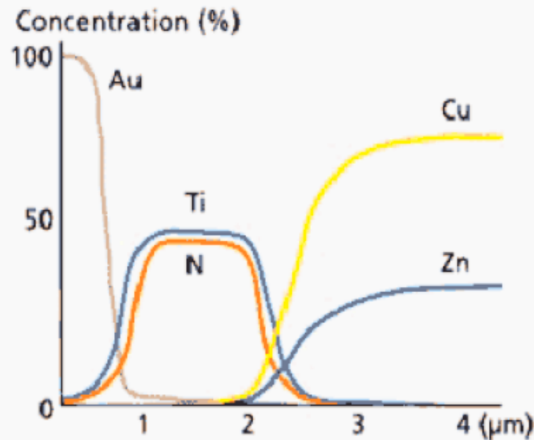
- The light intensity emitted by the plasma is a function of the element concentration in the plasma
- The element concentration in the plasma is a proportional to the product of the element concentration in the sample and the sputtering rate or transfer rate



GDOES



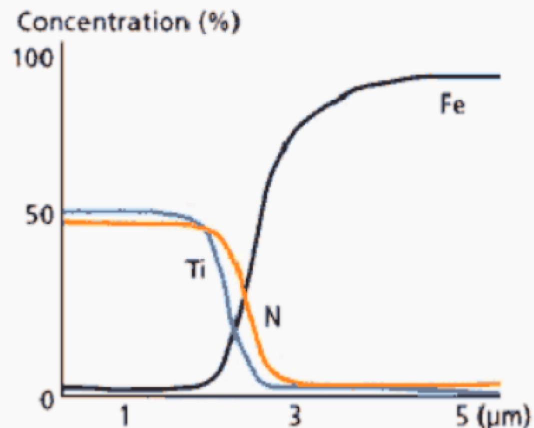
GDOES



Coated sheets

Complete characterization of the coating layer with respect to chemical composition, thickness and element distribution.

Analyze non-conductive coatings such as varnishes and paints with the optional RF source.



Hardphase coatings

Compound layer development can be determined by rapid analysis of the chemical composition. Other important material aspects such as depth penetration of the treatment process are possible.

Ceramics

Precise and accurate determination of the chemical composition is possible with the optional Radio Frequency source.

A

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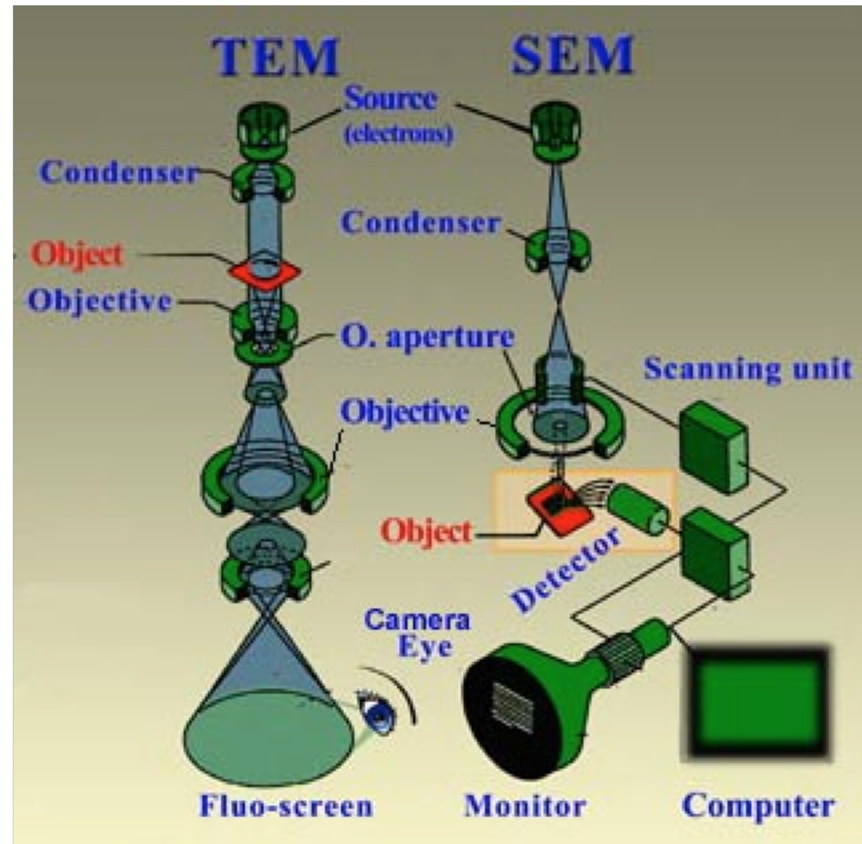
Microstructure

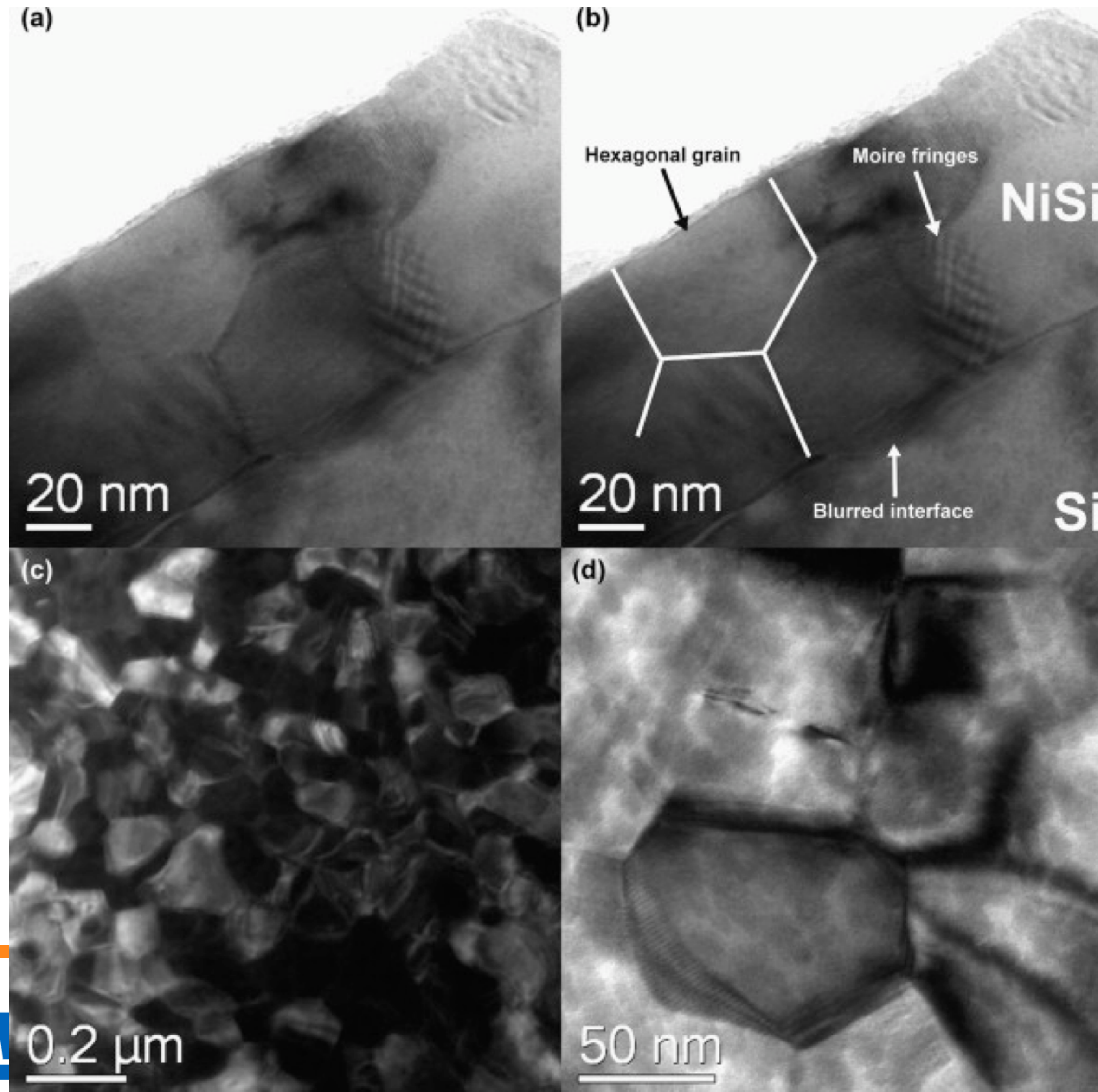
- Crystallinity
- crystal size
- orientation – texture
- Defects

Transmission electron microscopy TEM

Atomic level resolution 0.7 Å

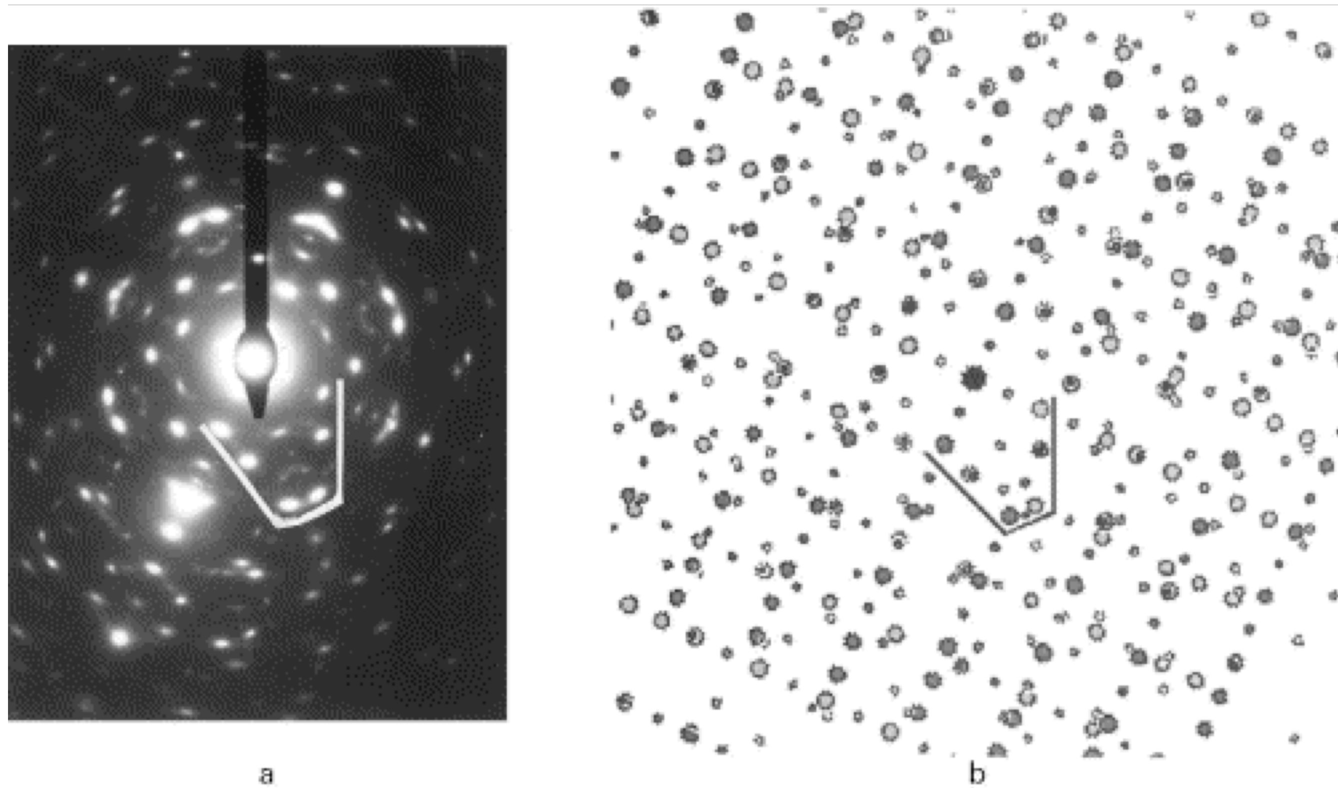


NiSi thin film



Results from TEM analysis of NiSi thin films: (a) XTEM highlighting equiaxed grains in the NiSi film in which Moiré (interference) fringes due to orientation differences between grains can be observed; (b) notable features in the as-obtained image (a) are indicated; (c) plan view, elastic hollow cone dark field image of the film, highlighting individual grains with diameters of 60–200 nm; and (d) plan view TEM image showing polygon al NiSi grains.

Electron diffraction

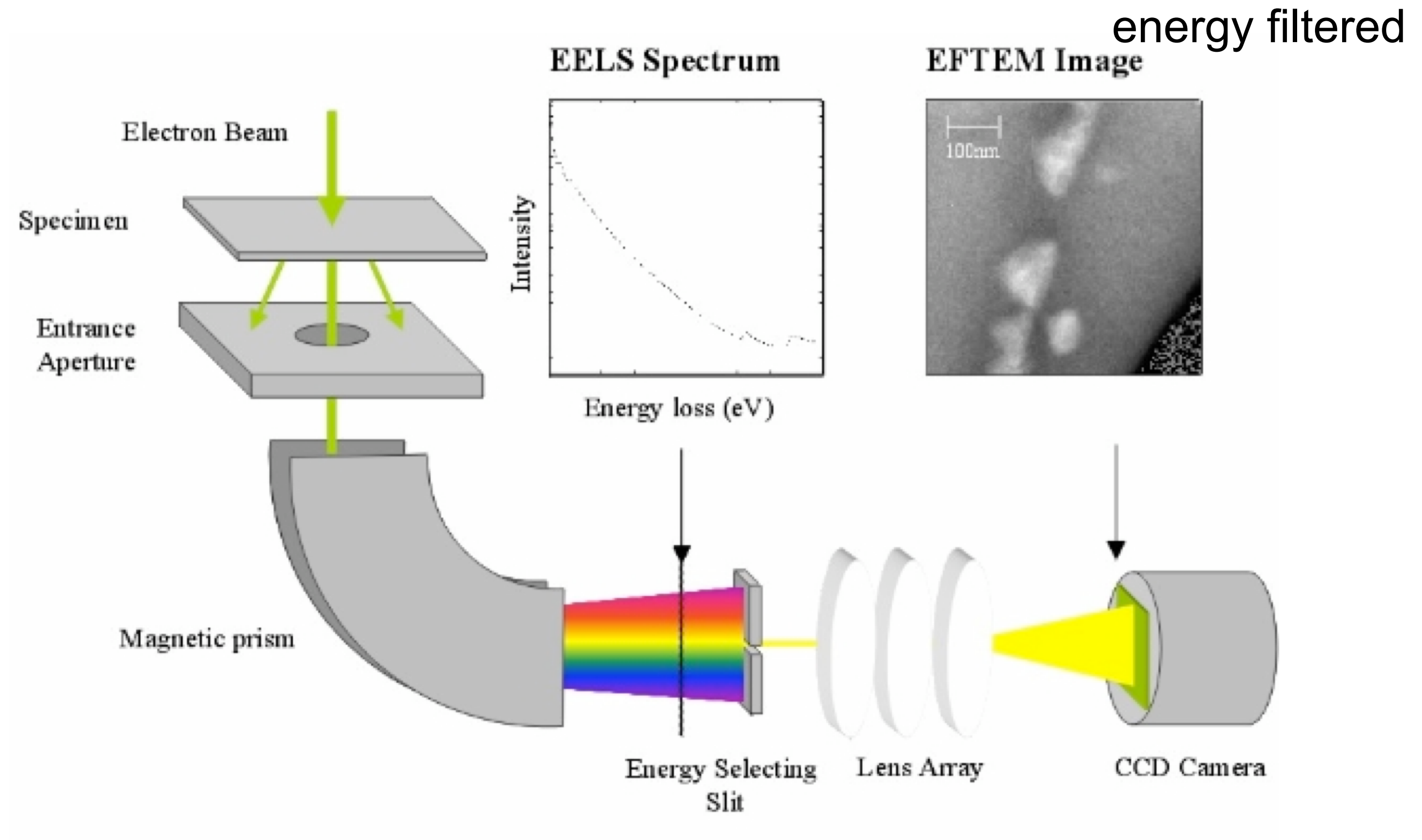


Measured diffraction pattern of a plan-view prepared ReSi1.75 film on Si (100) (—— guiding line for orientation); (b) theoretical diffraction diagram of ReSi1.75 with zone axis [0 1 0] and four superposed patterns, each turned around 45°.

D. Hofman et al. / Ultramicroscopy 81 (2000) 271–277

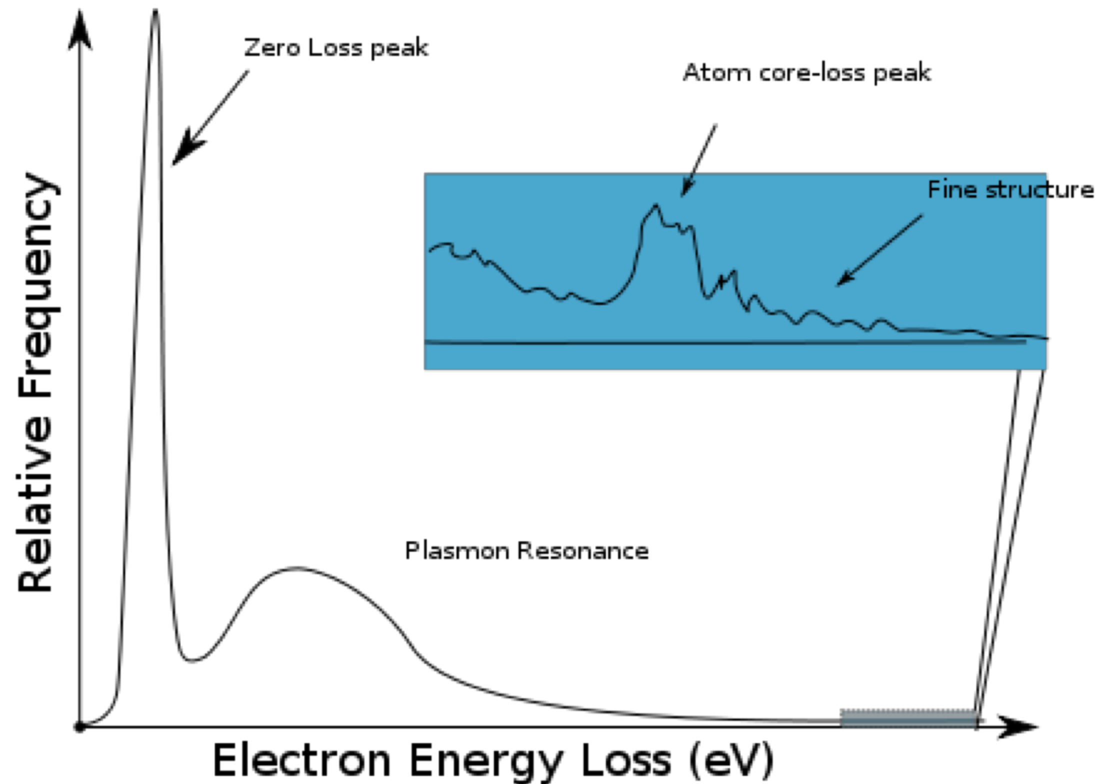
Electron energy loss spectroscopy EELS

energy filtered



Electron energy loss spectroscopy EELS

- Elemental analysis
  - light elements C
    ➔ 3d transition metals Sc, Zn
- chemical bonding e.g carbon sp^2/sp^3



Electron energy loss spectroscopy EELS

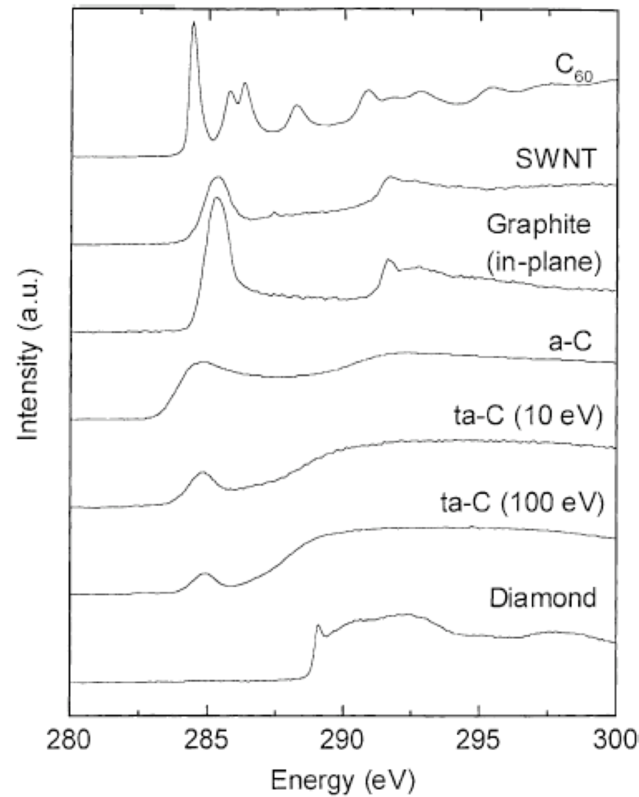


Fig. 29. Carbon K edge electron energy loss spectra of various carbon phases, after Waidmann et al. [196].

S. Waidmann, M. Knupfer, J. Fink, B. Kleinsorge, J. Robertson, J. Appl. Phys. 89 (2001) 3783.

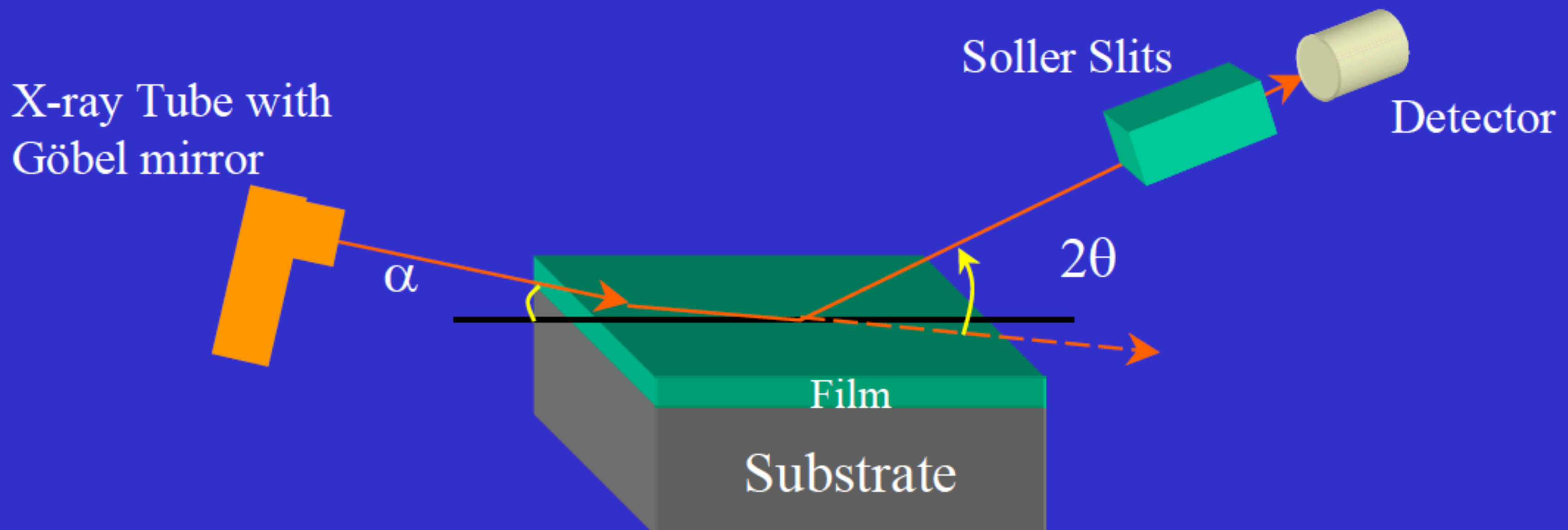
Glancing Angle X-ray Diffraction (GAXRD)

- In the x-ray diffraction pattern of thin films deposited on a substrate, contribution from substrate to the diffraction can sometimes overshadow the contributions from thin film.
- *GAXRD is used to record the diffraction pattern of thin films, with minimum contribution from substrate.*
- Non-destructive surface sensitive technique

*www1.chm.colostate.edu/Files/GA**XRD**.pdf*

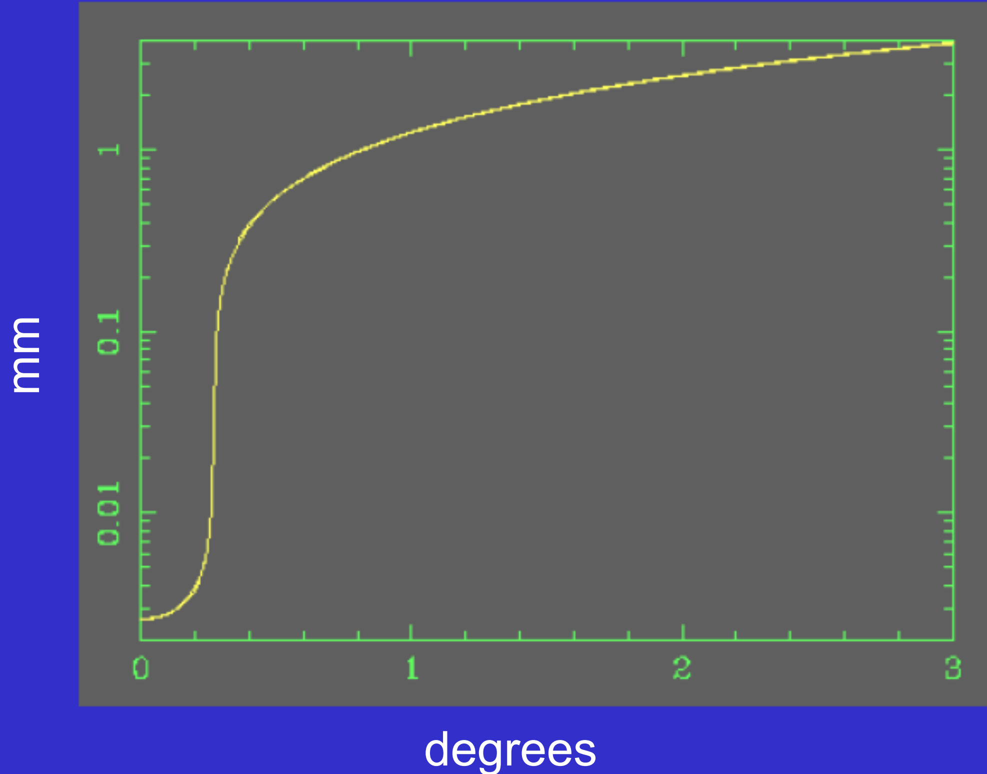
Technique

- Parallel, monochromatic X-ray beam falls on a sample surface at a fixed angle of incidence (α_I) and diffraction profile is recorded by detector only scan.



www1.chm.colostate.edu/Files/GA**XRD**.pdf

Penetration Depth Vs Angle of Incidence

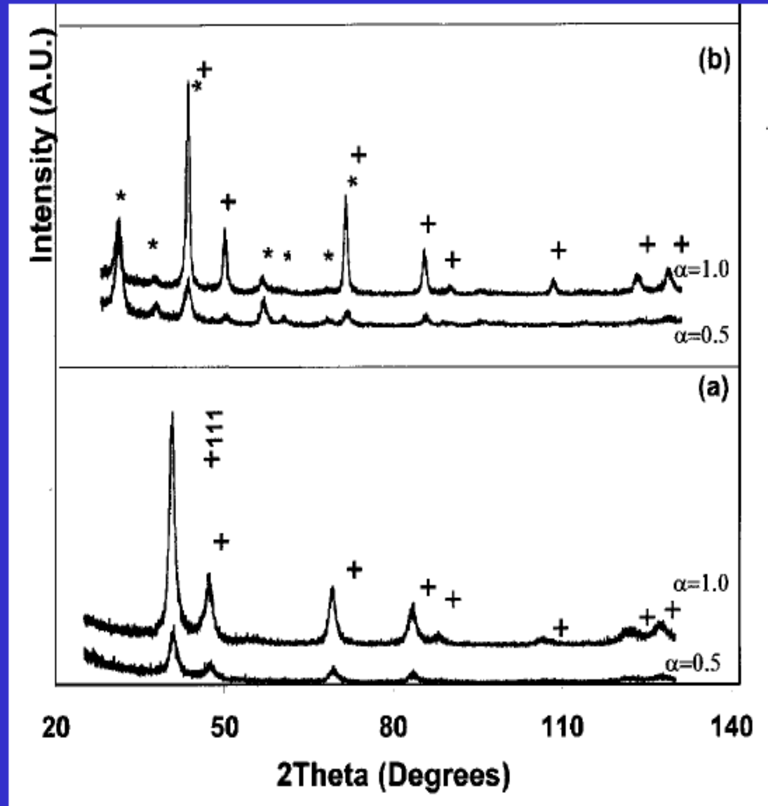


This figure shows penetration depth as a function of incident angle for Si_3N_4 for CuK_α (λ=0.154 nm) radiation

*www1.chm.colostate.edu/Files/GA**XRD**.pdf*

GAXRD: Example

- (a) As deposited 20 nm Ir metal film deposited on Si wafer. XRD curve for ***α=0.5º and 1.0º*** shows the peaks for cubic iridium metal phase represented by (+)
- (b) Ir film annealed at 873K for 1hr. XRD curve for ***α=0.5º*** shows the presence of the dominating IrO_2 phase (*). As ***α*** was increased to ***1.0°,*** the contribution from the underlying layer of Ir metal increased and the Ir peaks dominated the XRD curve. The results indicate the presence of an overlying oxidized layer of Ir metal



www1.chm.colostate.edu/Files/GA**XRD**.pdf

X-Ray Reflectivity XRR

- Thin Film
  - thickness
  - density
  - roughness
  - rougness of interface

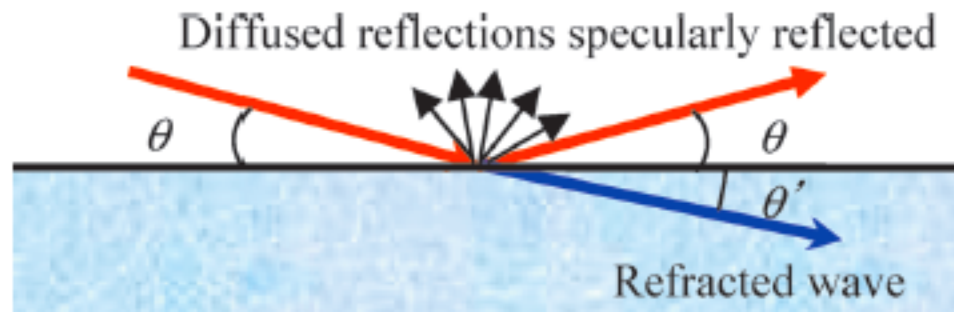
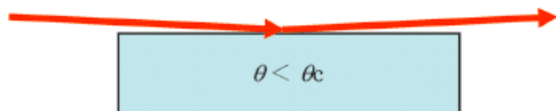


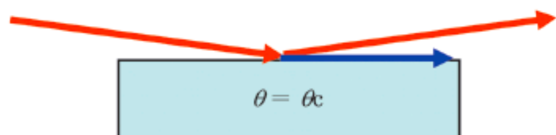
Fig. 1. Reflection and refraction of X-rays on material surface.

Miho Yasaka, The Rigaku Journal, 26(2), 2010

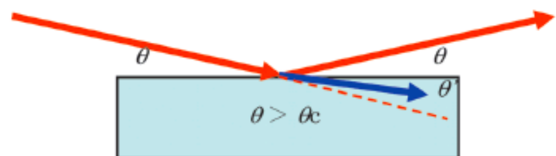
X-Ray Reflectivity XRR



A) Incident angle < Total reflection critical angle
All incident X-rays are reflected.



B) Incident angle = Total reflection critical angle
Incident X-rays propagate along the sample surface.



C) Incident angle > Total reflection critical angle
Incident X-rays penetrate into the material by refraction

Fig. 3. Reflection and refraction of X-rays at material surface with the changes in the grazing angle.

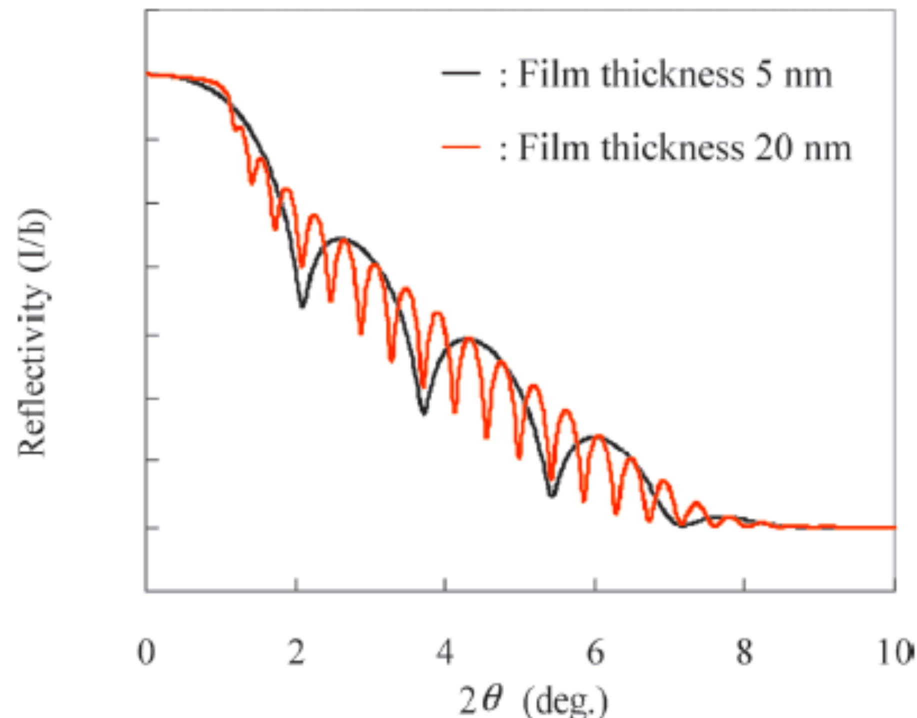


Fig. 4. Reflectivity of Au film on Si substrate.

Miho Yasaka, The Rigaku Journal, 26(2), 2010

X-Ray Reflectivity XRR

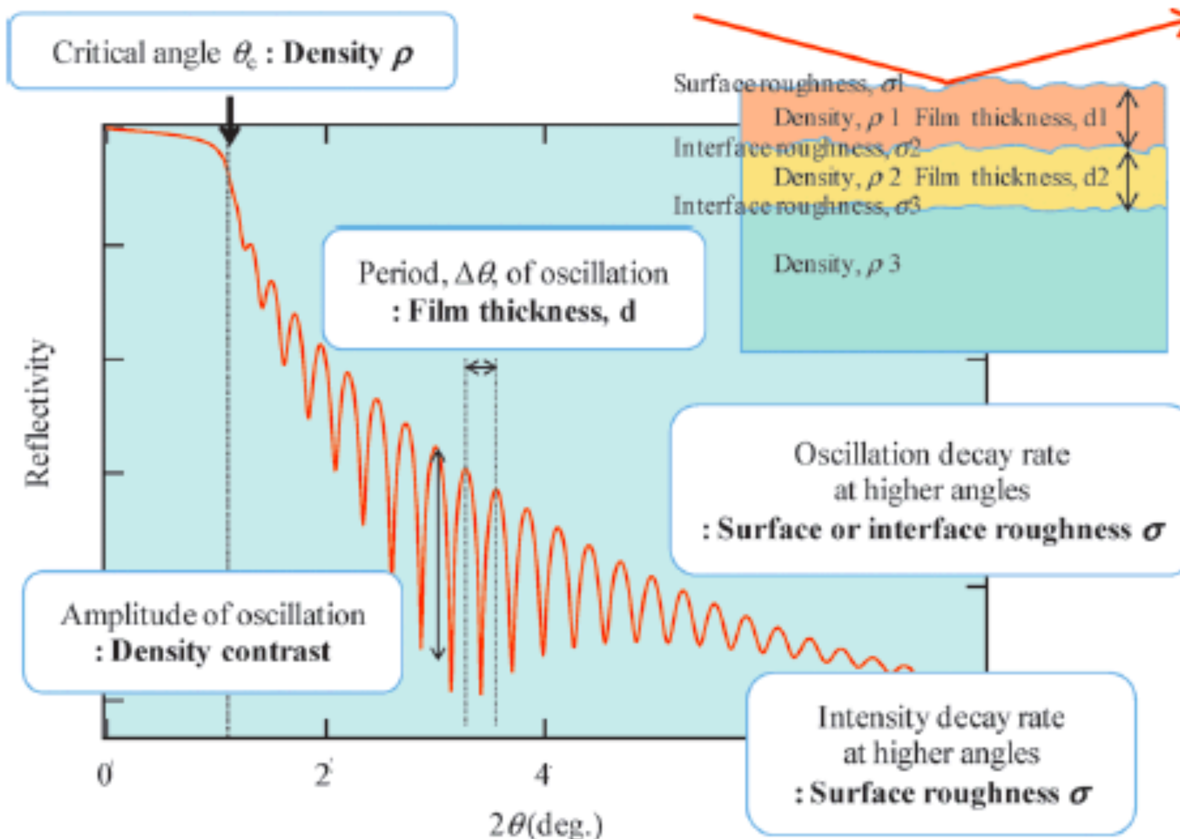


Fig. 8. Information provided by X-ray reflectivity profile.

Miho Yasaka, The Rigaku Journal, 26(2), 2010

X-Ray Reflectivity XRR

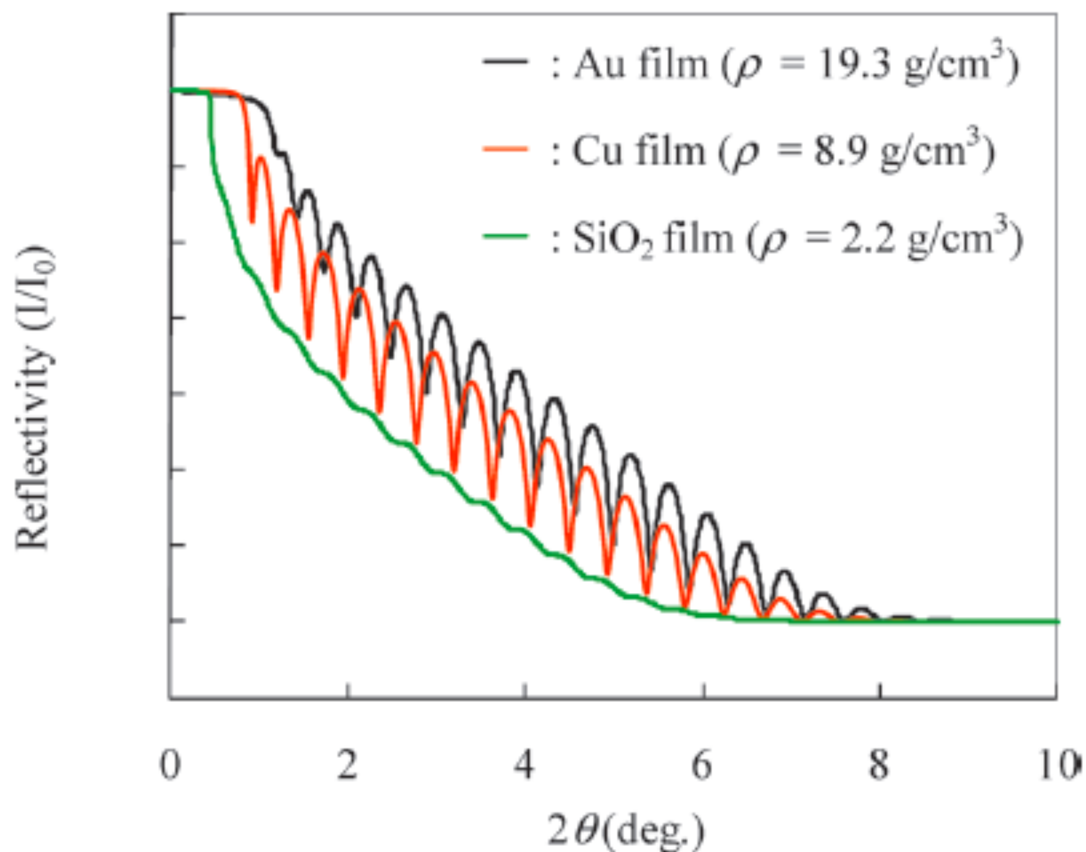


Fig. 5. X-ray reflectivity curves of Au, Cu and SiO_2 film on Si substrates (film thickness is 20 nm).

Mino Yasaka, "The Rigaku Journal", 26(2), 2010

Contents

- Thin film properties
- Wealth of methods – MATRIX
- Scattering
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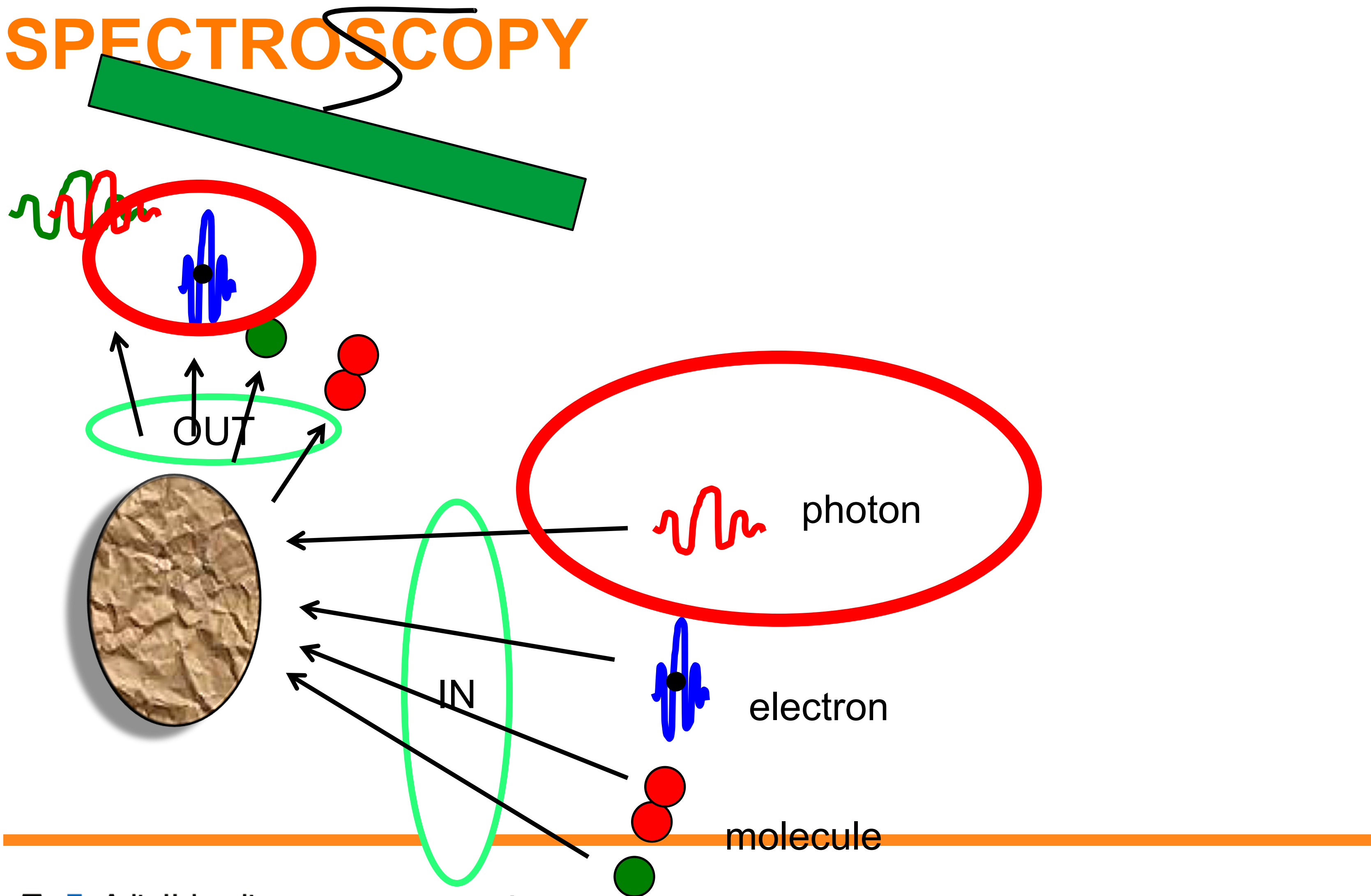
- Bonding – ESCA, RAMAN
- Topography - ADM
- Electrical conductivity – four point probe
- Mechanical properties - indentation
- Optical transmittance- FTIR (???)

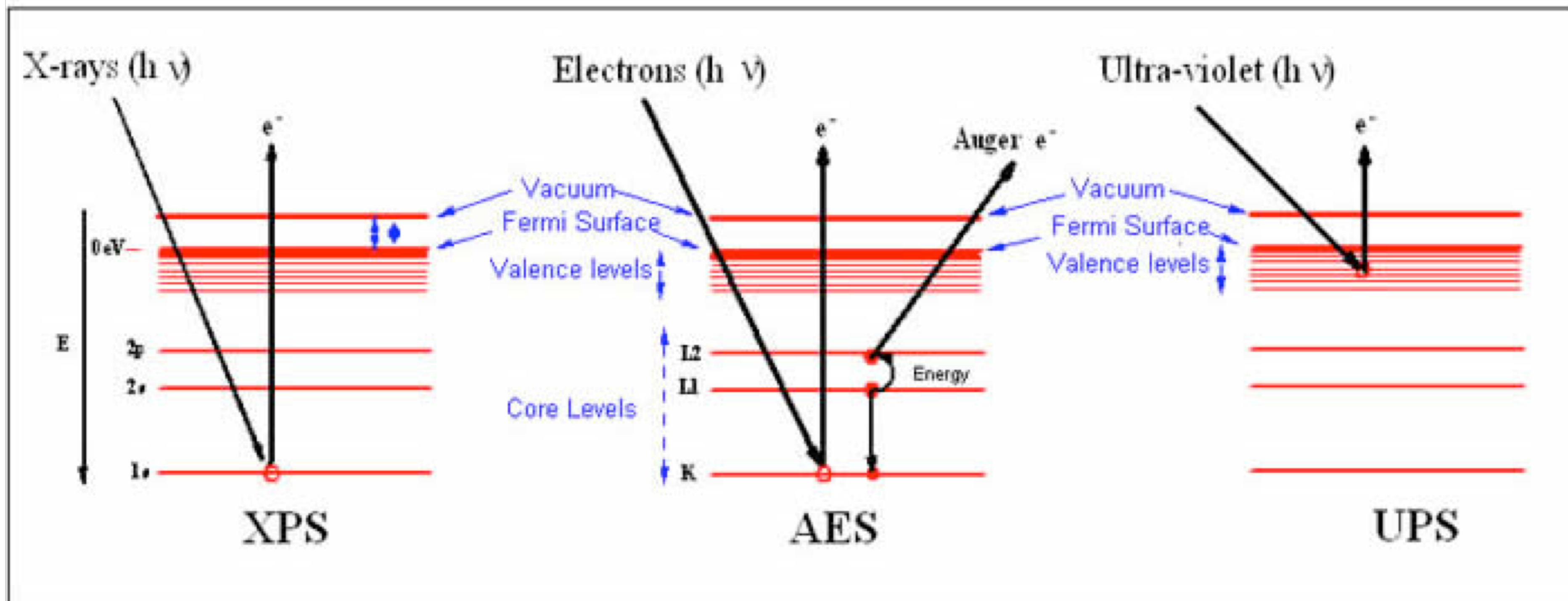
Bonding

- Chemical reactions particularly on surface
- Cemical bonding in amorphous thin films (oxides, carbon, some metals)
- Contaminations
- Methods used also to determine composition

Scattering experiment ELECTRON SPECTROSCOPY



Photoelectron spectroscopy techniques



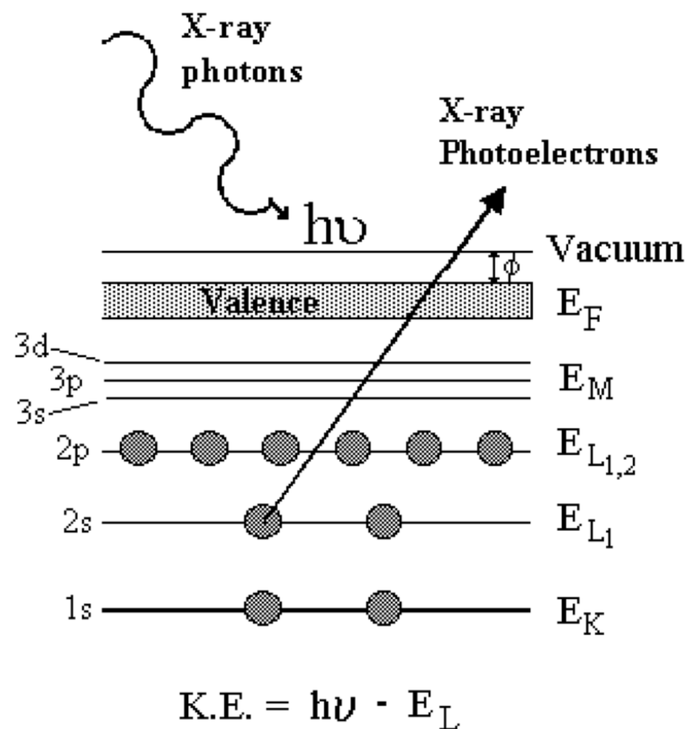
X-ray photoelectron spectroscopy -
Electron Spectroscopy for Chemical Analysis ESCA

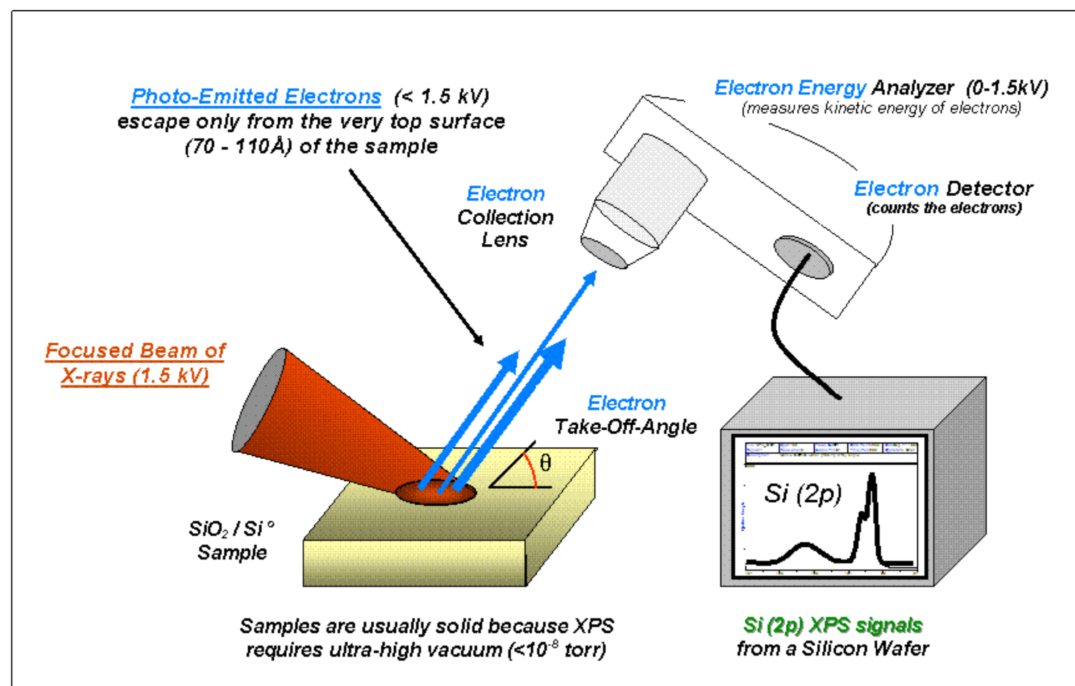
Auger electron spectroscopy

Ultraviolet phoelectron spectroscopy

XPS by Leena-Sisko Johansson

XPS





XPS

(4)

(5)

(3)

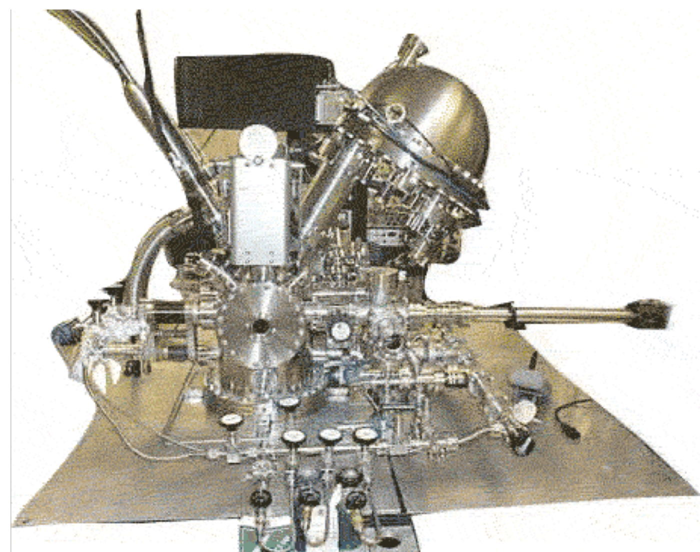
(1)

é

rX

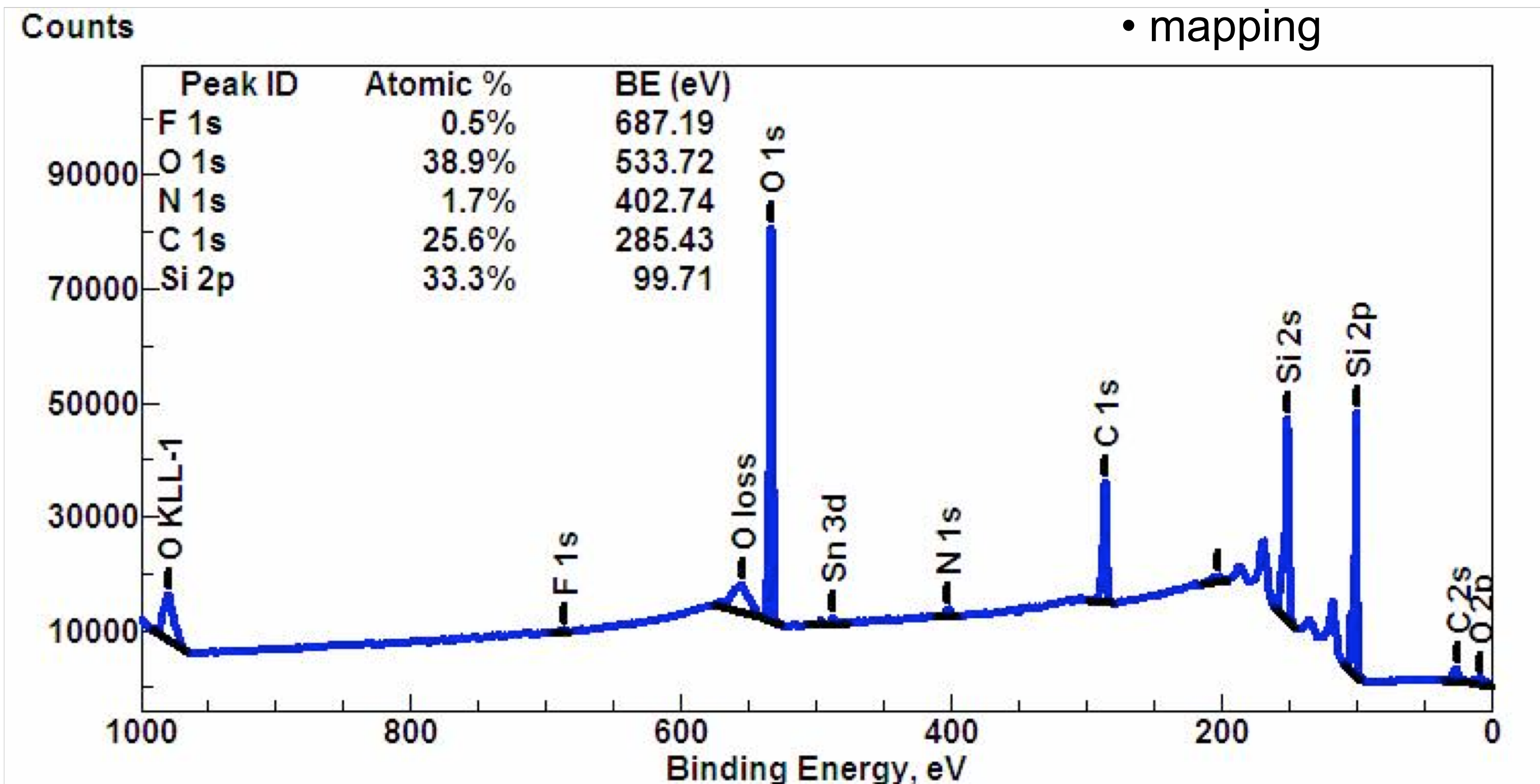
(2)

(6)



XPS

- used for elemental analysis
- all elements Z > 2
- detection limit 1/1000
- 1 – 10 nm depth
- x-ray beam 20 – 200 μm
- mapping



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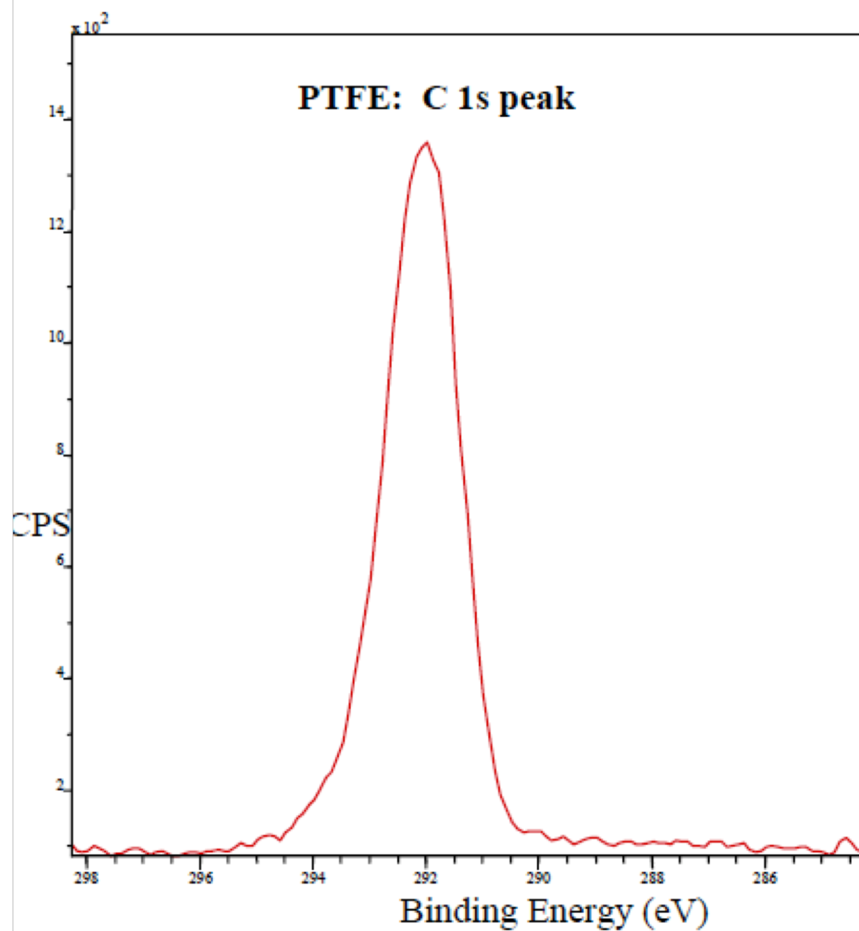
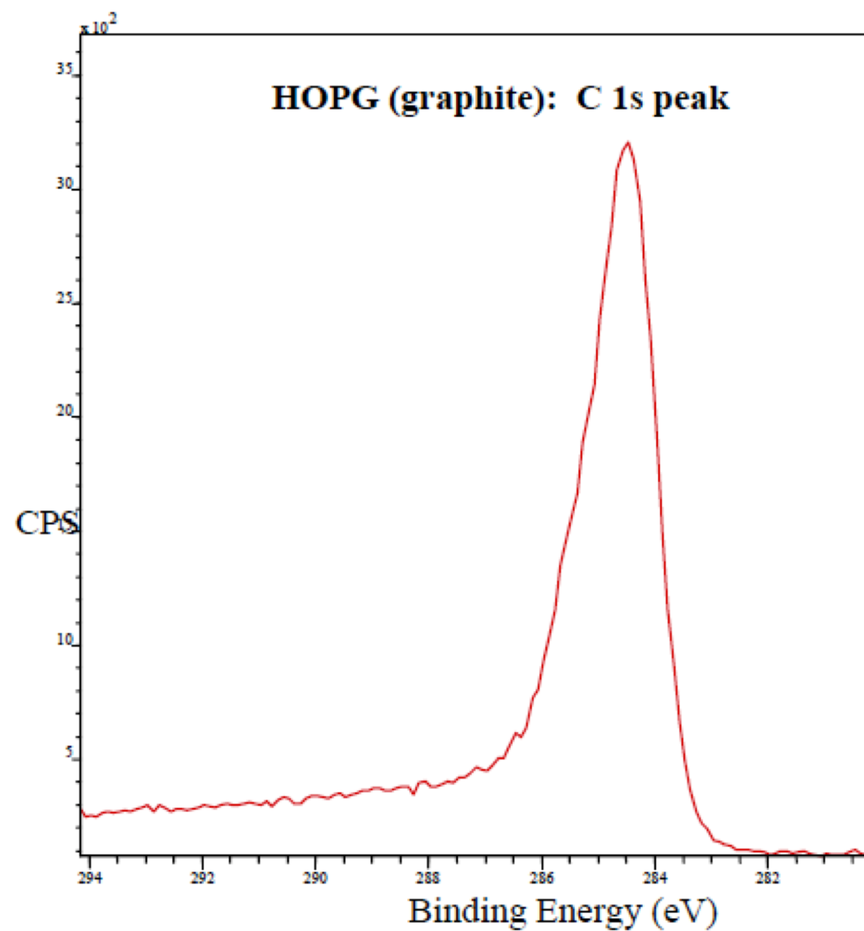
XPS

sensitive to chemical bonding, e.g. type of bonding of carbon

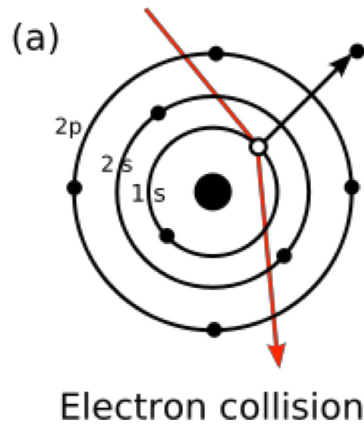




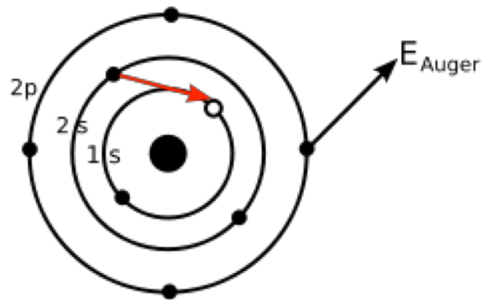
UPS Ultraviolet phoelectron spectroscopy

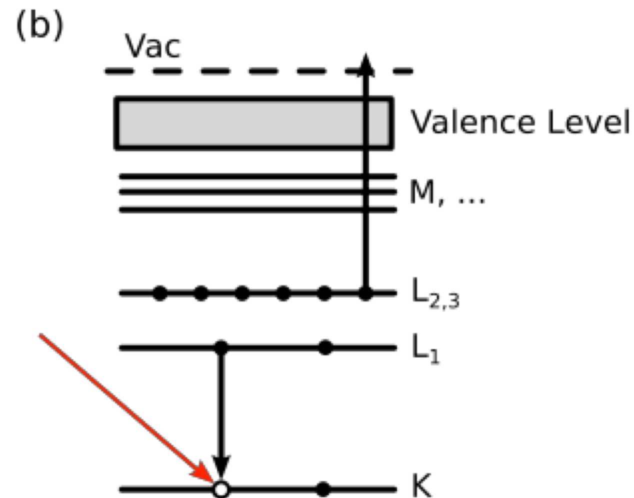
- typical energy 20 eV
- more surface sensitive than XPS

AES Auger electron emission spectroscopy



Electron collision



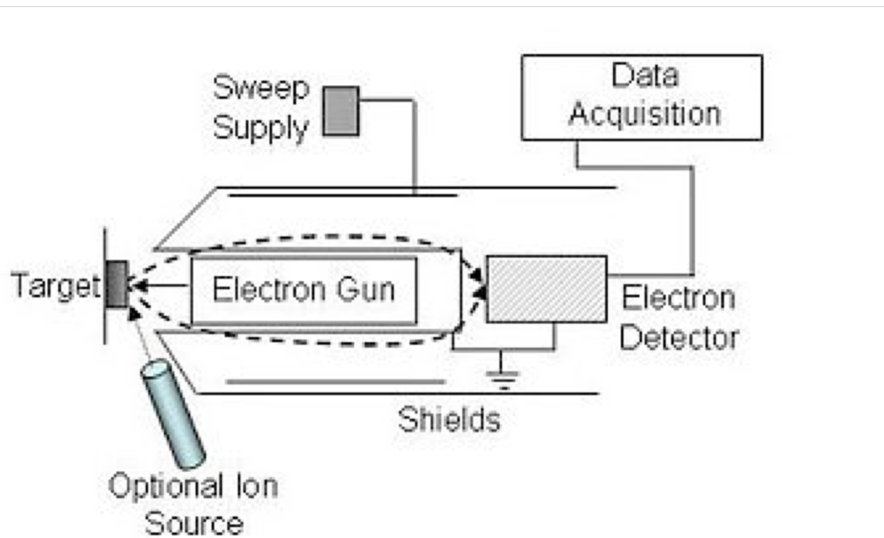


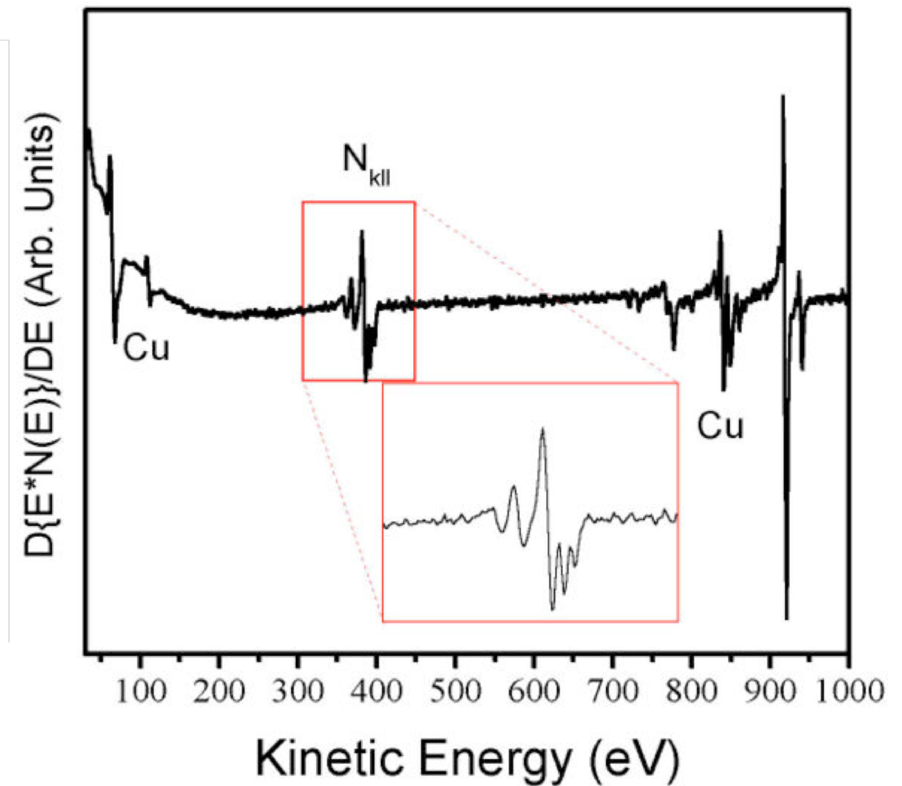
$$E_{kin} = E_{Core\ State} - E_B - E_C'$$

Auger electron emission

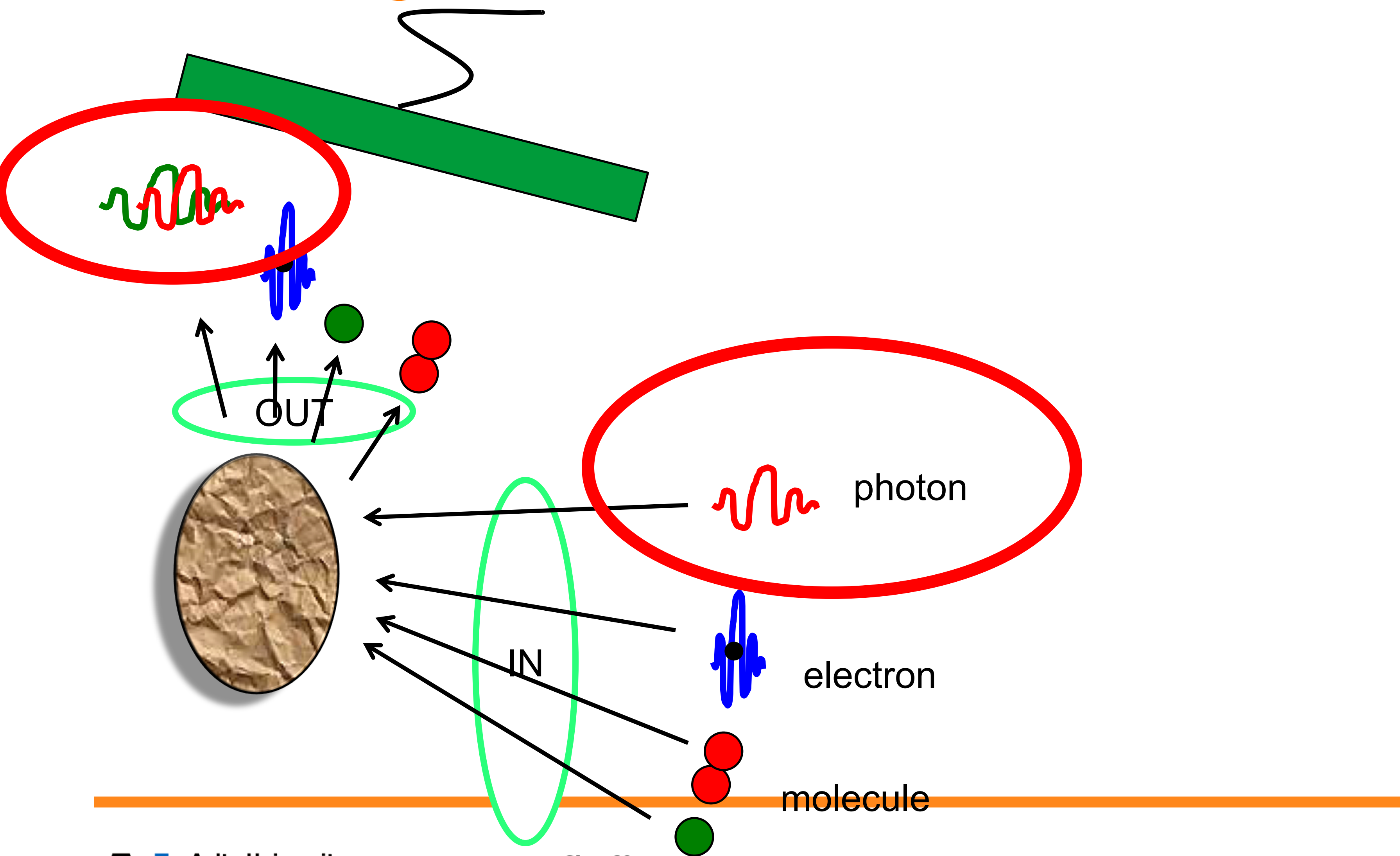
AES

used to analyze chemical composition



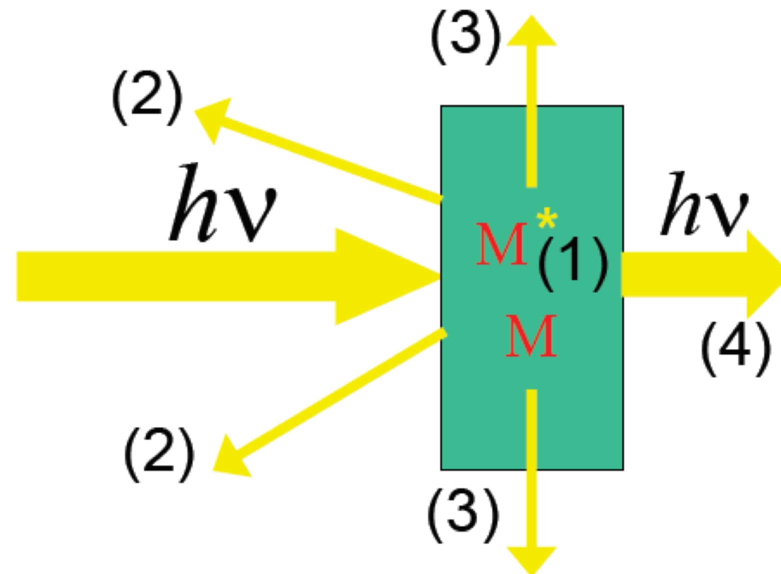


Scattering experiment - OPTICAL



When electromagnetic radiation passes through matter, it interacts with the matter and can be:

- absorbed (1)
- reflected (2)
- scattered (3)
- transmitted (4)

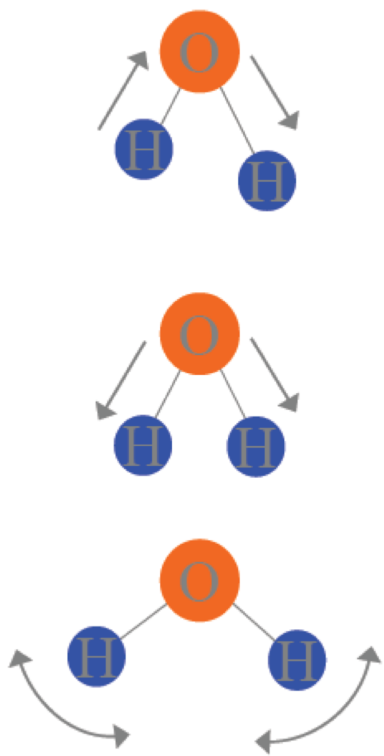


- depending upon:
  - its **frequency**
  - the **structure of molecules** of the matter it encounters.

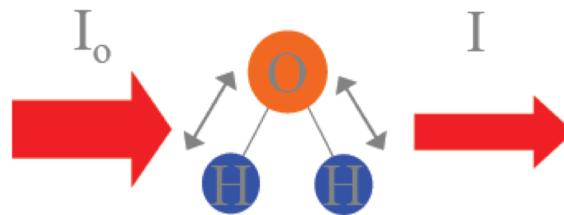
- **Vibrational Spectroscopy**

  Vibrational spectroscopy is a method of chemical analysis where the sample is illuminated with incident radiation in order to excite molecular vibrations. Vibrational excitation is caused by the molecule absorbing, reflecting or scattering a particular discrete amount of energy. There are two major types of vibrational spectroscopy: Infrared (IR) and Raman.

IR Light *Absorption* Measurement.



- Light energy absorbed by increasing vibrations between atoms in a molecule.
- Energy (wavelength) absorbed related to strength of bond.
- Strength of bond related to molecular structure and environment.
- Amount of light absorbed related to concentration and *absorptivity* constant.



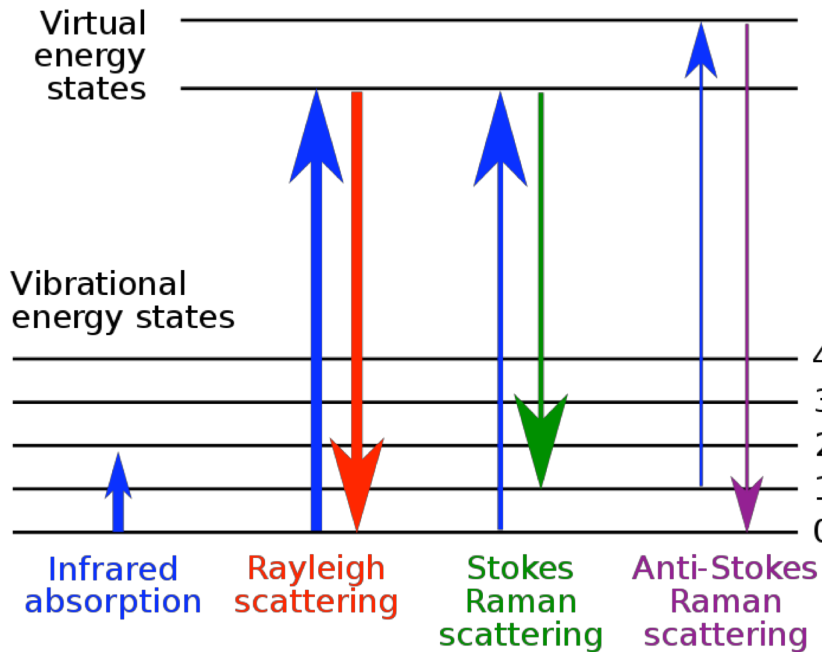
Infrared

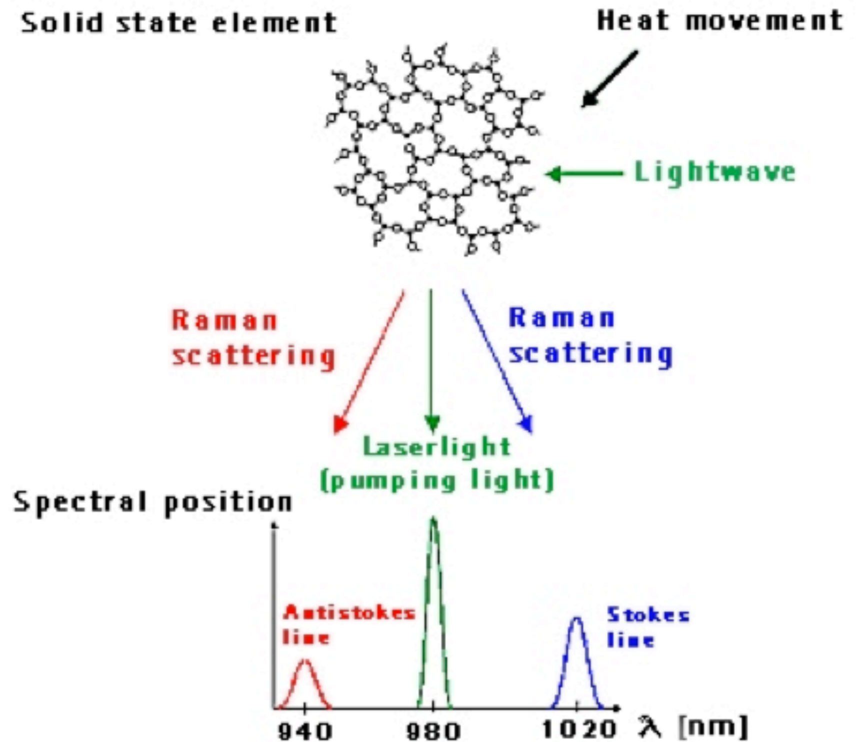
- Absorption
- Requires a dipole moment change (O-H, N-H, C=O)
- Sample preparation or accessory usually necessary
- Short optical pathlength required
- Non-aqueous samples

Raman

- Emission of scattered laser light
- Requires polarizability change (C=C, aromatics)
- Little or no sample preparation necessary
- Measure through transparent packaging
- Aqueous samples

RAMAN





RAMAN
example carbon

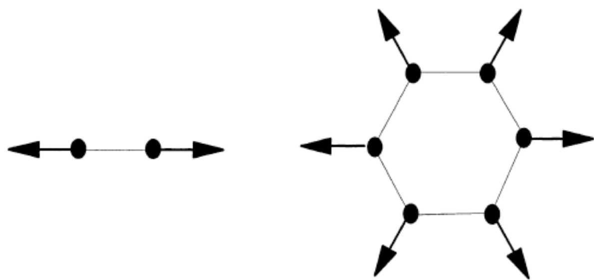


Fig. 34. Eigenvectors of the Raman G and D modes in graphite and amorphous carbons.

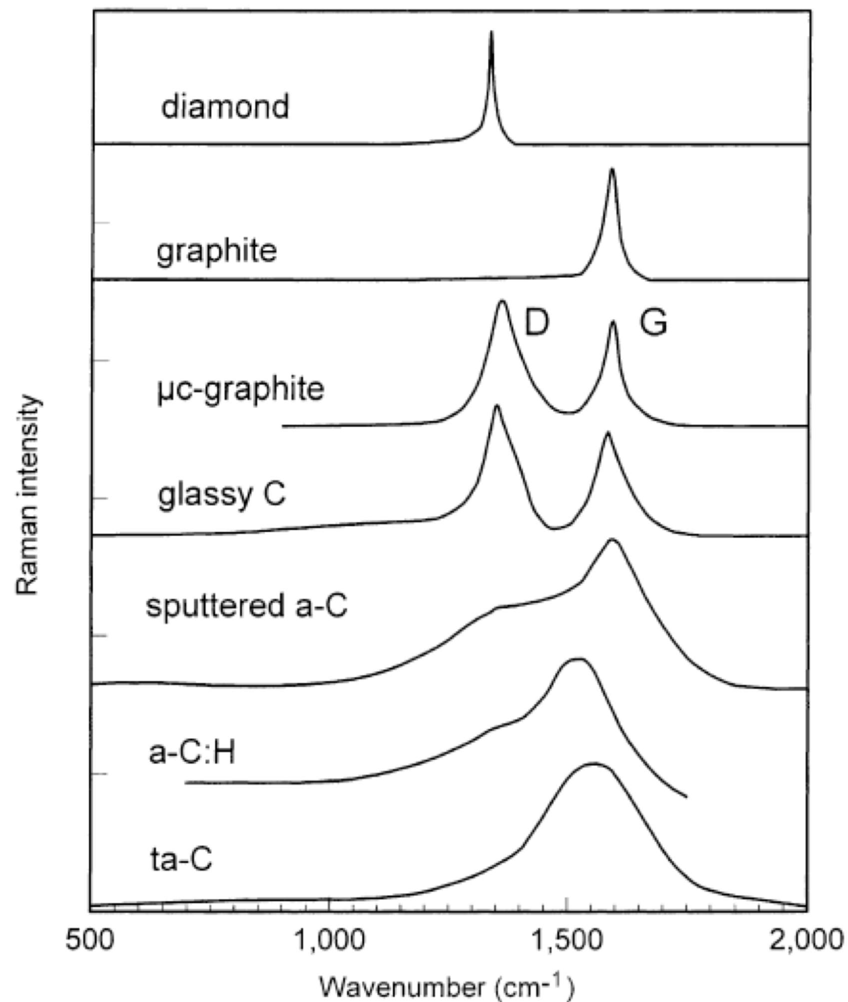
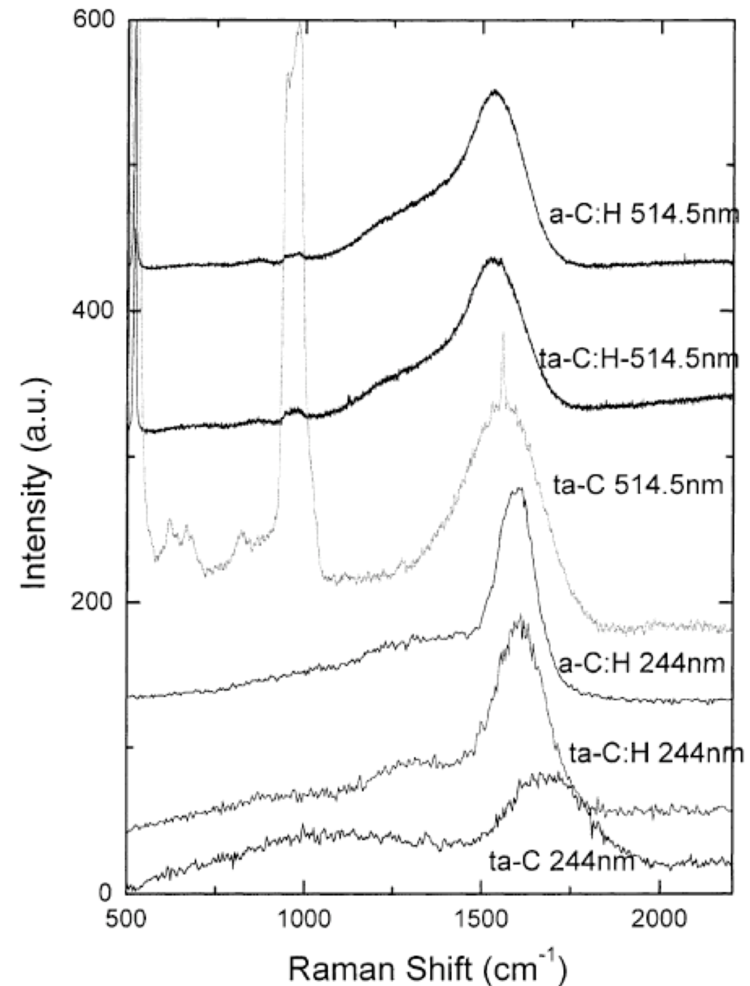


Fig. 33. Comparison of typical Raman spectra of carbons.

J. Robertson / Materials Science and Engineering R 37 (2002) 129–281

RAMAN
example carbon

- amorphous materials
- finger print of different bonds (materials)
- mapping



J. Robertson/Materials Science and Engineering R 37 (2002) 129–281

Mapping

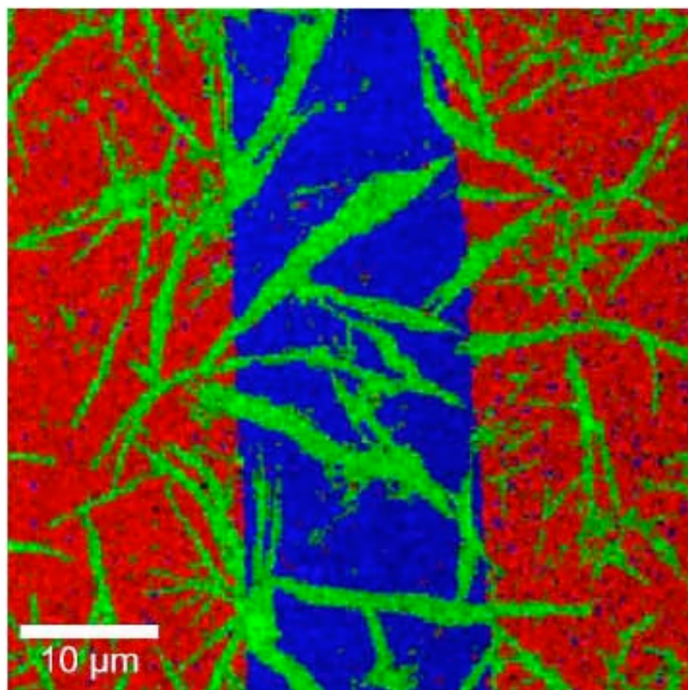
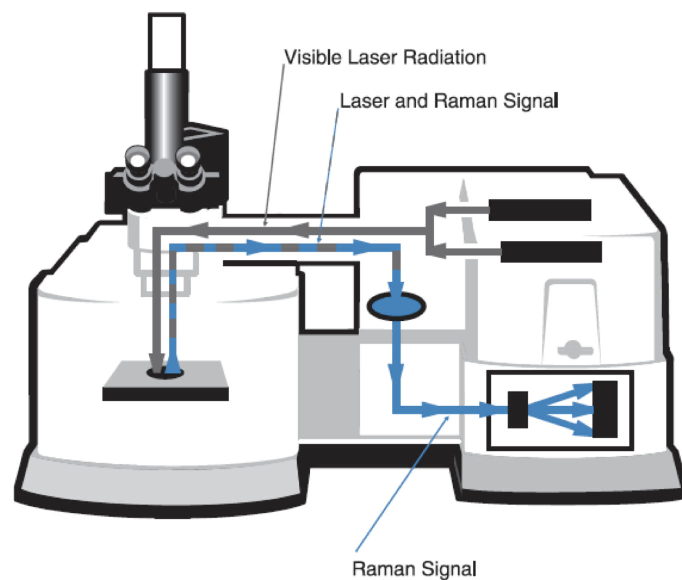


Figure 4. Color-coded confocal Raman image of a 7.1 nm PMMA layer (red) and a 4.2 nm contamination layer (green) on glass (blue). 200 x 200 spectra, 7 ms integration time/spectrum. Total acquisition time 5.4 minutes.



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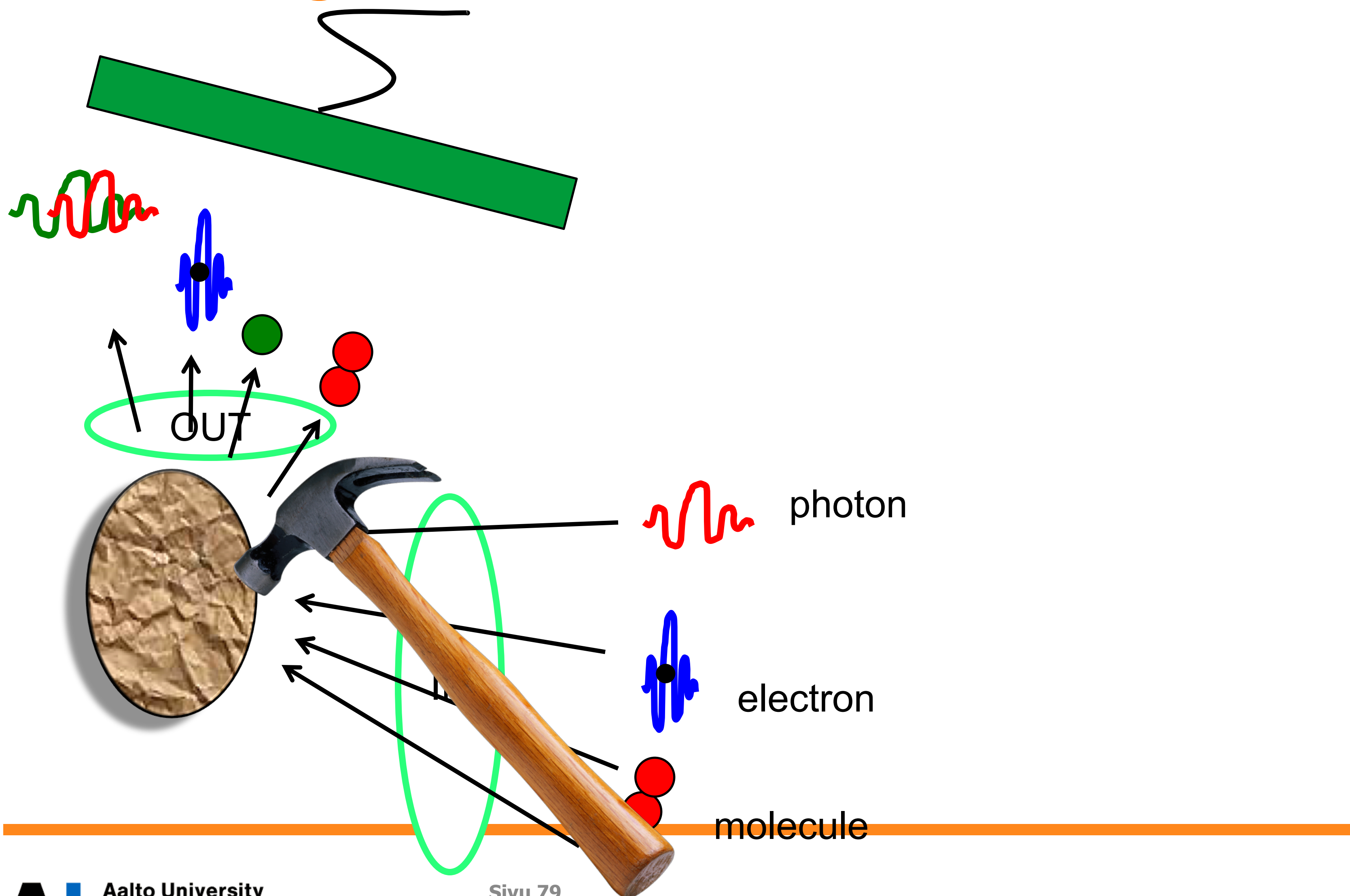
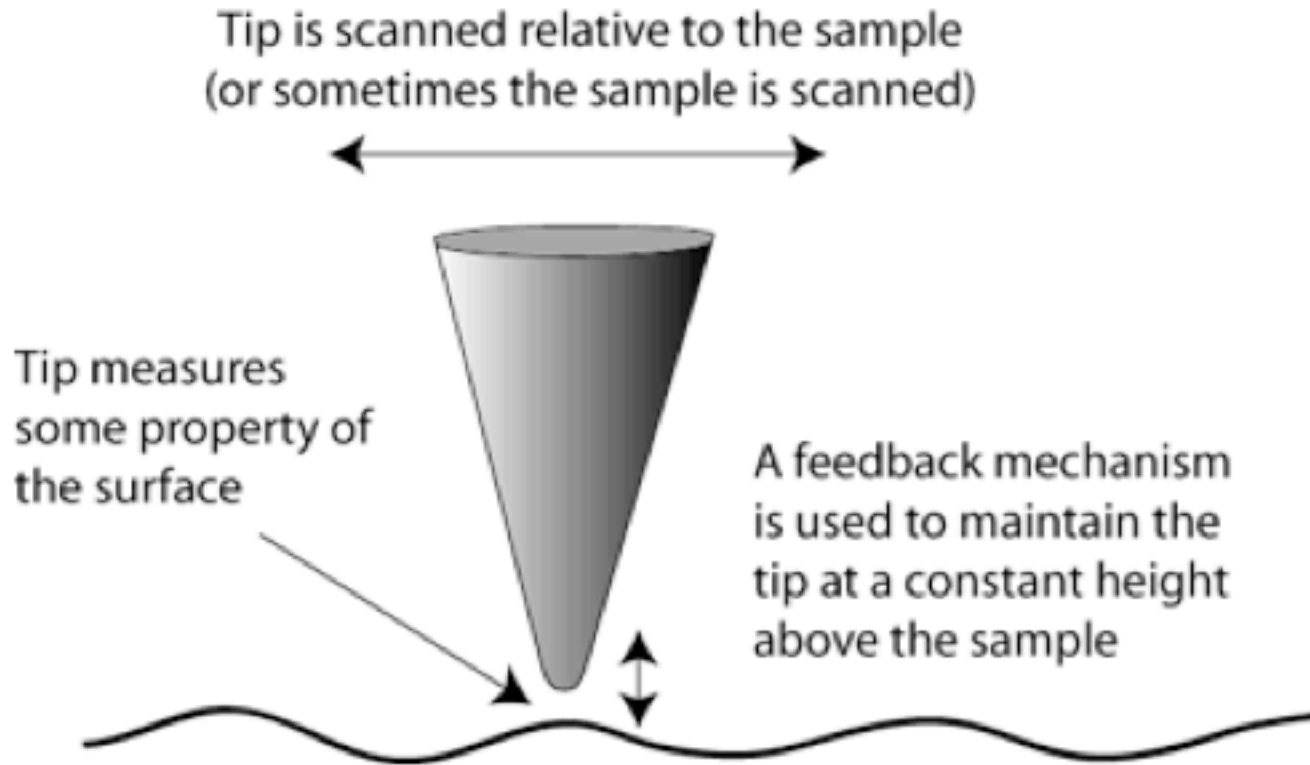
- Electrical conductivity – four point probe
- Mechanical properties - indentation
- Optical transmittance- FTIR (???)

Scattering experiment- Mechanical



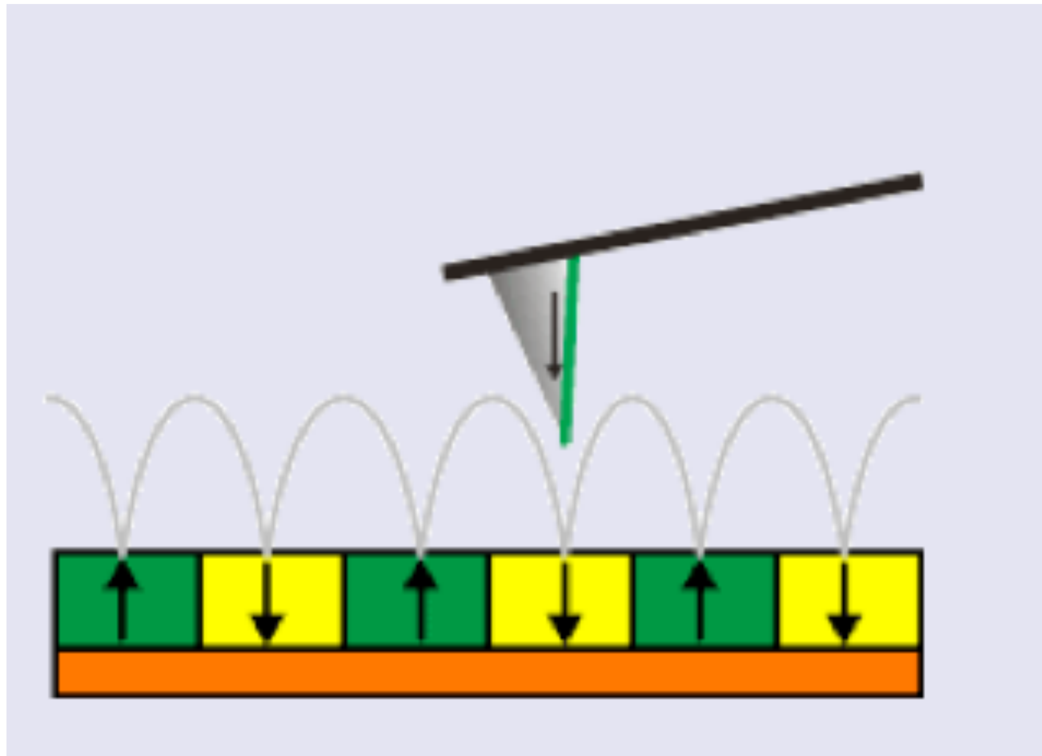
ion, atom, neutron

Scanning Probe Microscopy



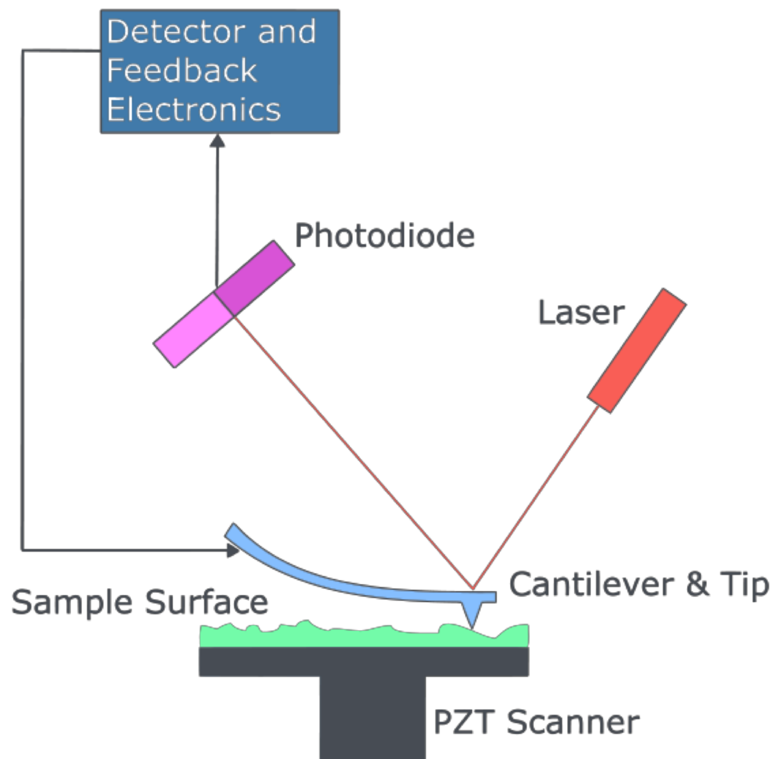
nanoScience Inc.

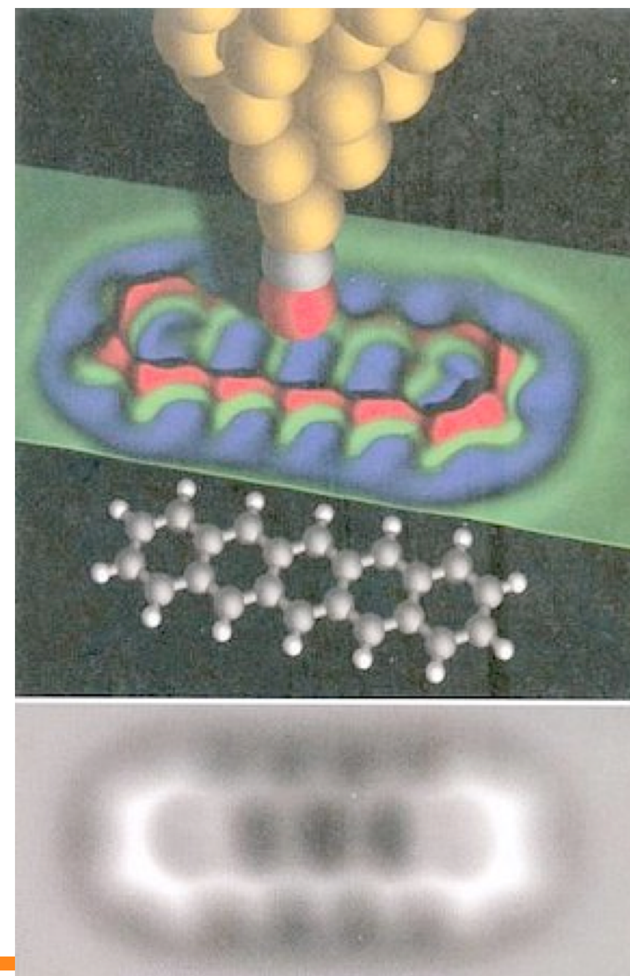
Magnetic Force Microscopy



nanoScience Inc.

Atomic force microscope AFM atomic resolution in UHV





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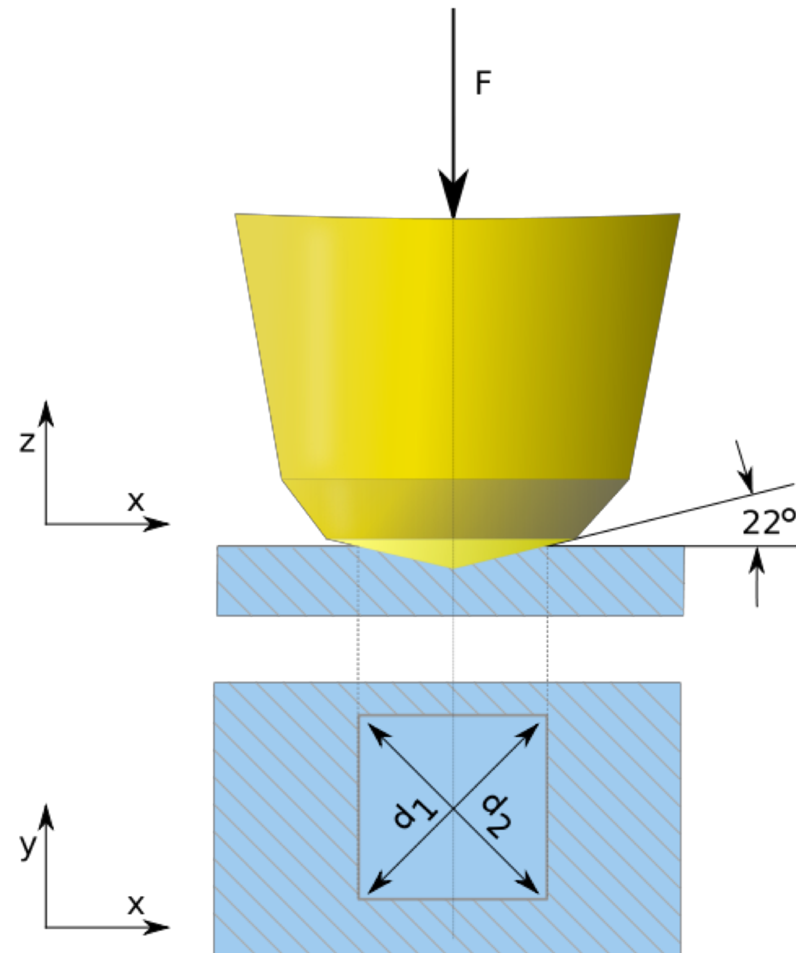
Indentation test



Indentation

- H = constant*load/(indentation area)
- Thin film/substrate: composite hardness
- Coating hardness: h< film thickness
- Very thin coatings: hardness by modelling (FEM, MD)

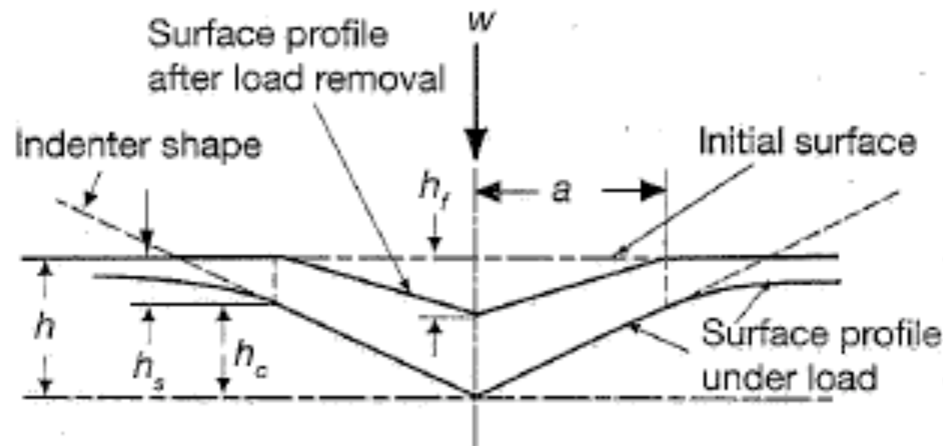


Fig. 5.9. Schematic representation of a section through an indentation using a conical indenter. h_c = contact depth, h_s = sink-in depth and h_f = final depth.

E from loading - unloading curve

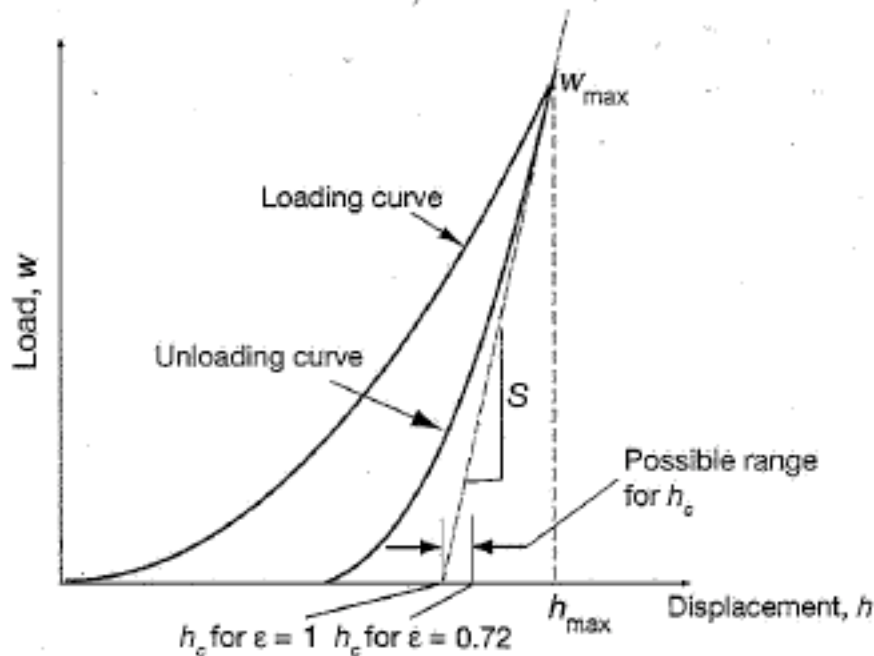


Fig. 5.10. A typical load–displacement indentation curve.

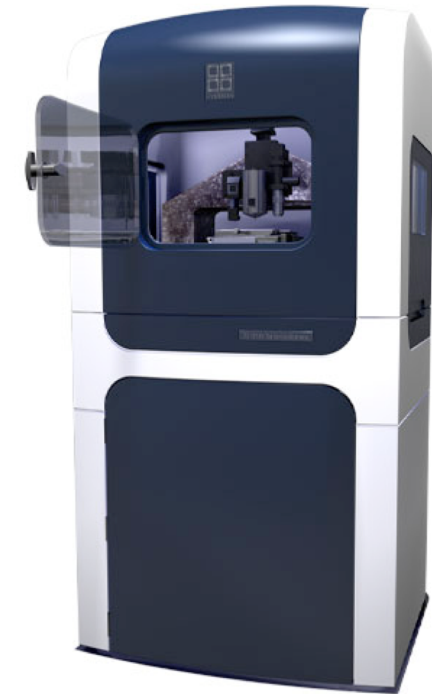
$$E_r = \frac{\sqrt{\pi} \cdot S}{2 \cdot \sqrt{A}}$$

$$h_c = h_{\max} - \varepsilon \cdot w_{\max}/S$$

$$\frac{1}{E_r} = \frac{1 - \nu_i^2}{E_i} + \frac{1 - \nu^2}{E}$$

- h < 1/10 film thickness
- Very thin coatings: E by modelling (FEM, MD)

Hardness and E as a function of indentation depth



Depth profiles of Er and H data from 200 μN partial-unload nanoindentation tests on 50 nm TiN thin film samples.
www.hysitron.com

Er Sample 1 Er Sample 2 Er Sample 3 Er Sample 4
H Sample 1 H Sample 2 H Sample 3 H Sample 4

E_r (GPa)

H (GPa)

h_c (nm)

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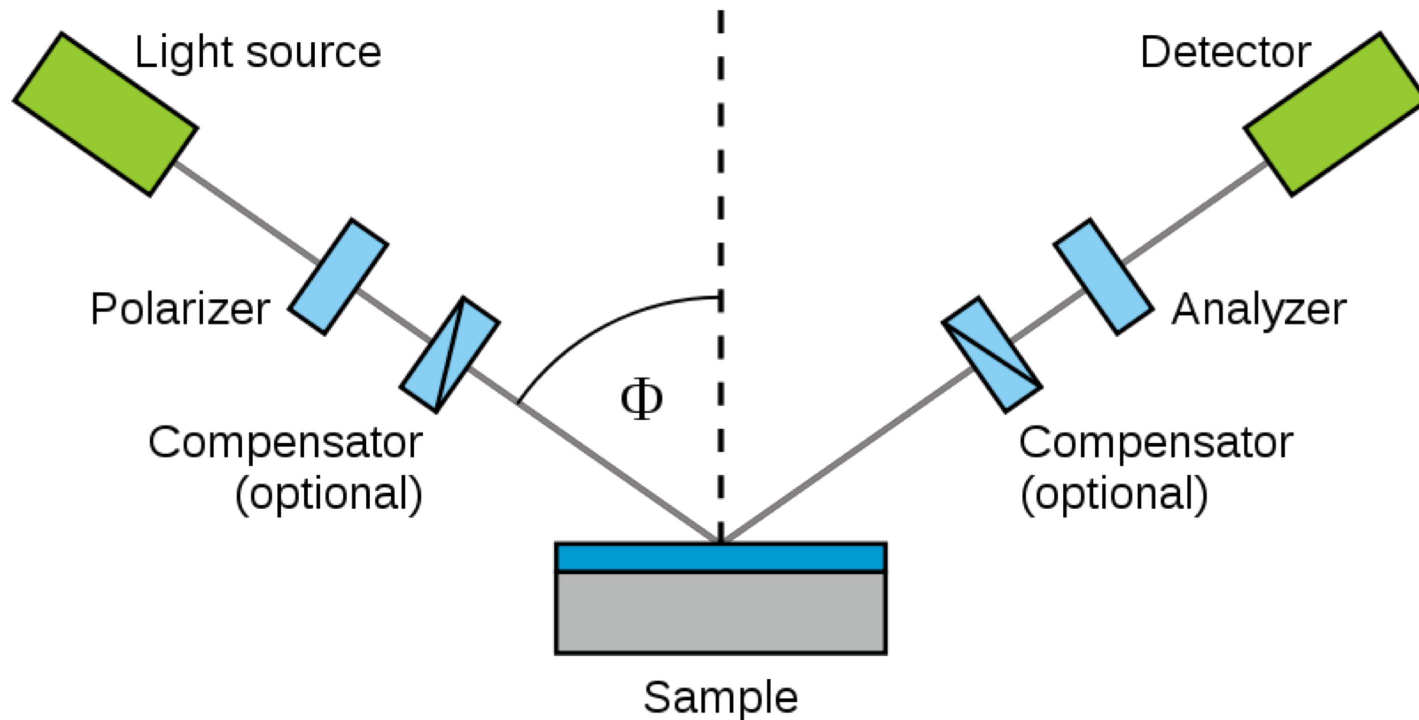
- Optical properties

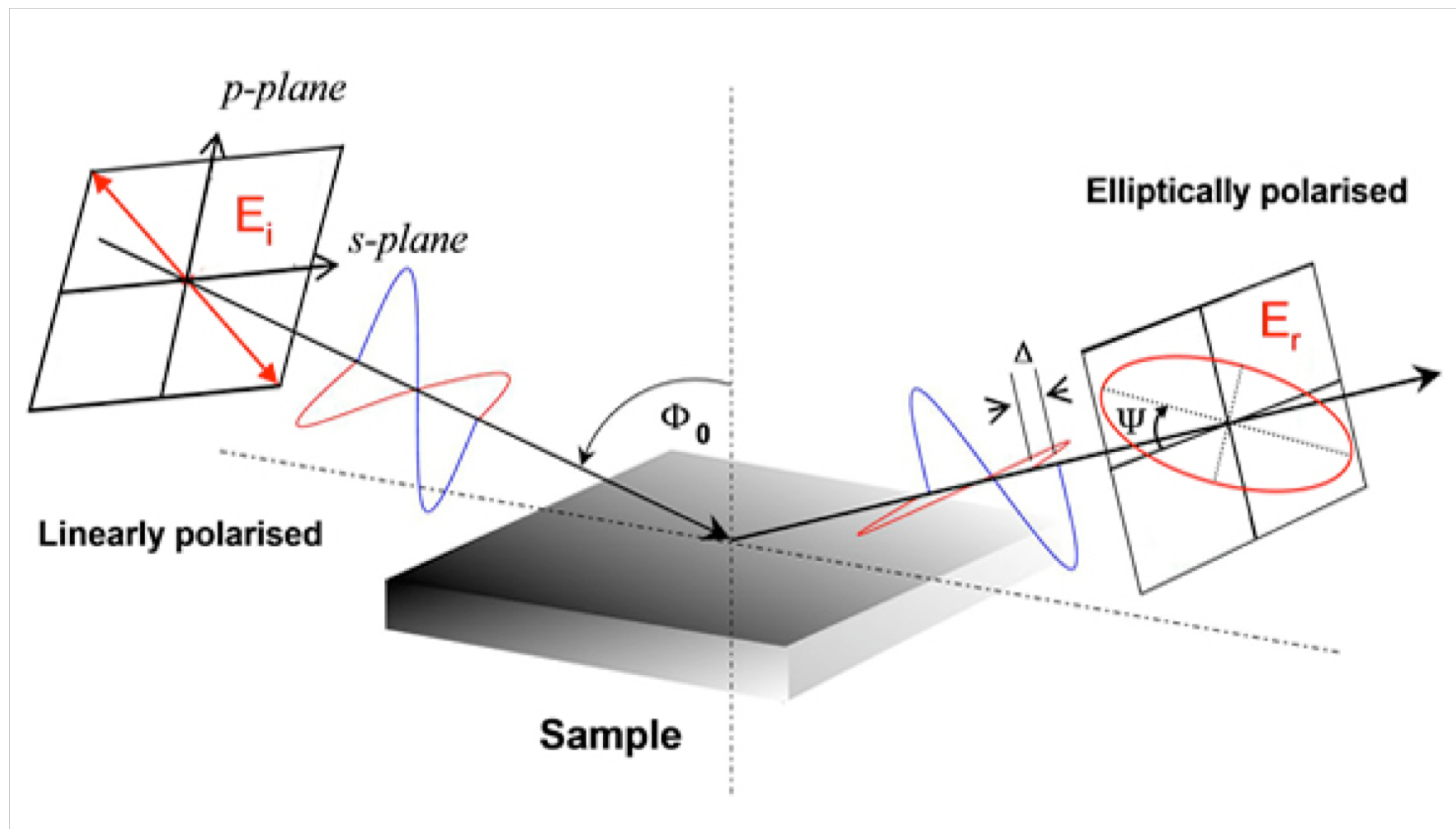
Optical coatings

- Control of reflectance and emission
  - Lenses
  - Photo voltaic
  - Solar thermal
- Protective optical coatings
- Self-cleaning or easy to clean films on optical surfaces
- Measuring thin dielectric film properties

Ellipsometry



Ellipsometry



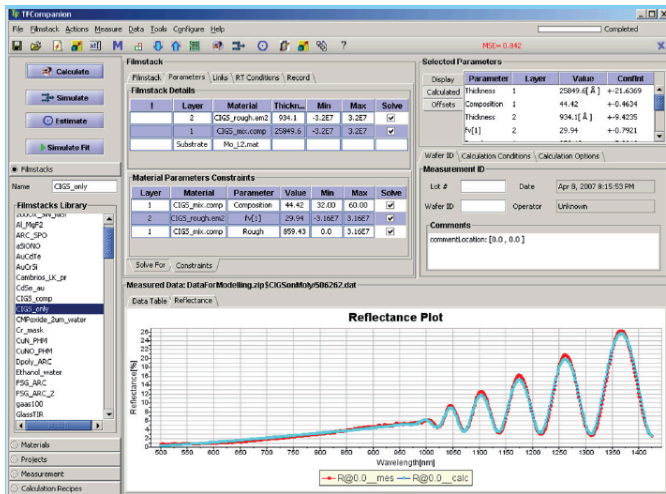
Ellipsometry

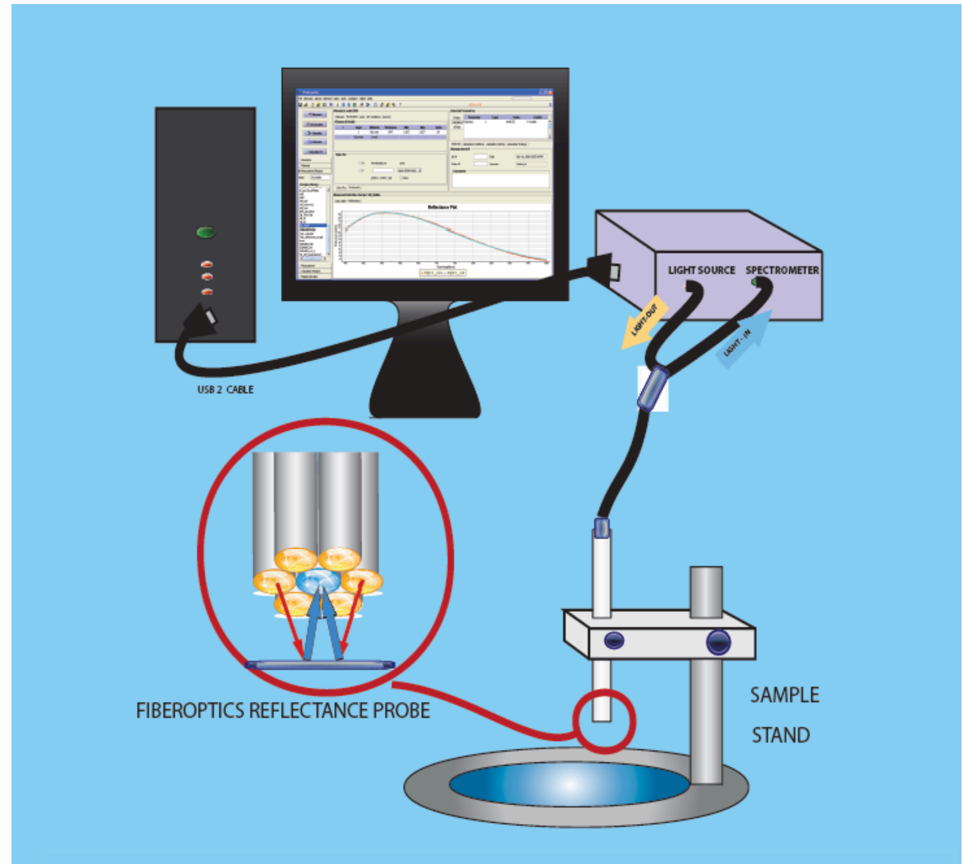
- real part (refractive index), n(λ)
- imaginary part (extinction coefficient), k(λ)
- complex refractive index, of a material, N(λ) = (n(λ)+ik(λ)), where λ

- If N(λ) known film thickness
- measure of phase shift ➔ very thin films can be measured < 1nm – several μm
- multilayer films may be measured when using numerical models

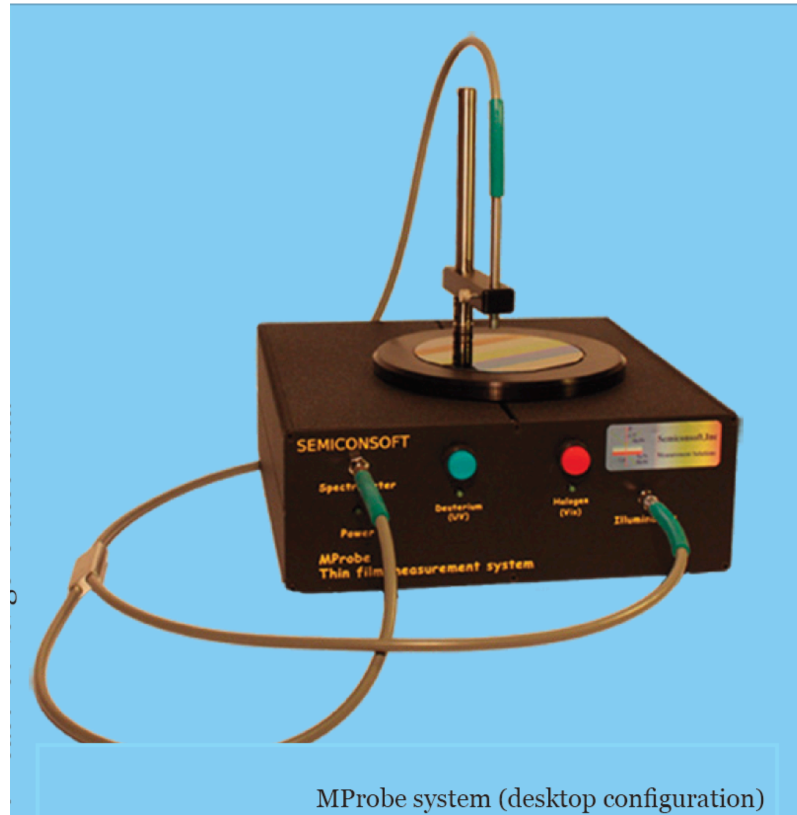
Reflectometry

- Film thickness
  - 3 nm -> 200 µm
- n and k values
- multilayers





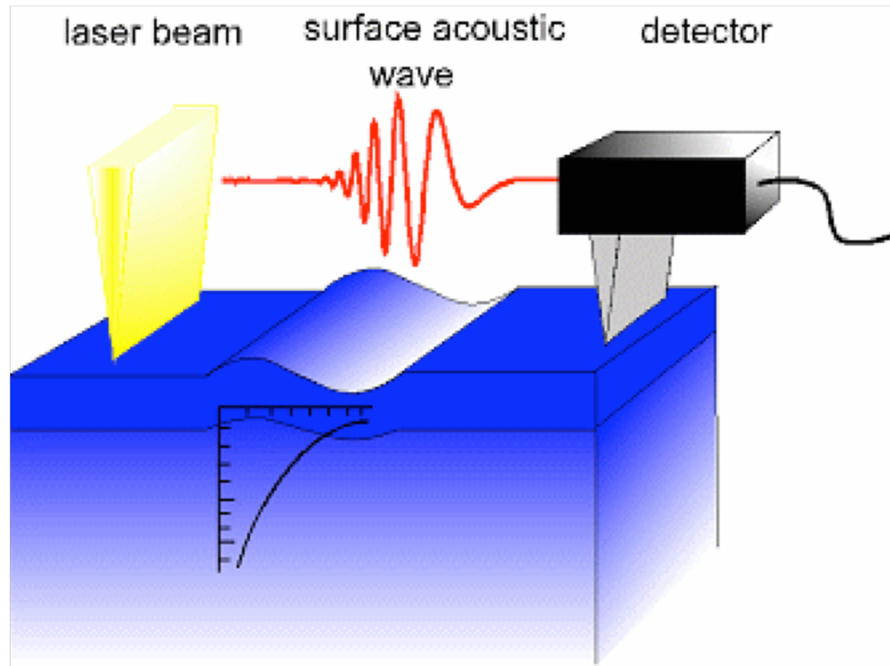
Reflectometry



MProbe system (desktop configuration)

SAW Surface acoustic wave

Pulsed laser generates shock waves



Surface wave dispersion velocity related to elastic modulus of coating substrate system (E_{film} and $E_{substrate}$)

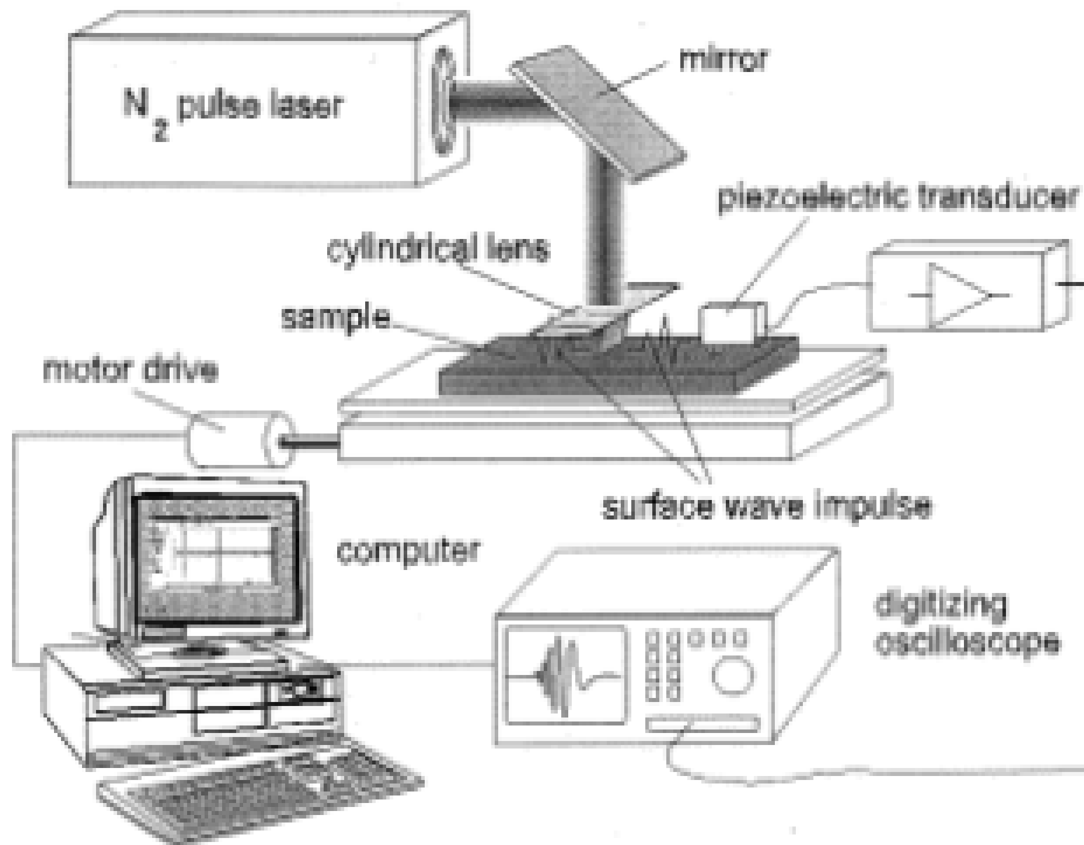


Fig. 1. Schematic representation of the surface acoustic wave (SAW) equipment.

Microstructural changes in DLC films due to tribological contact
J. Koskinen, D. Schneider, H. Ronkainen, T. Muukkonen, S. Varjus, P. Burck, K. Holmberg and H. -J. Scheibe

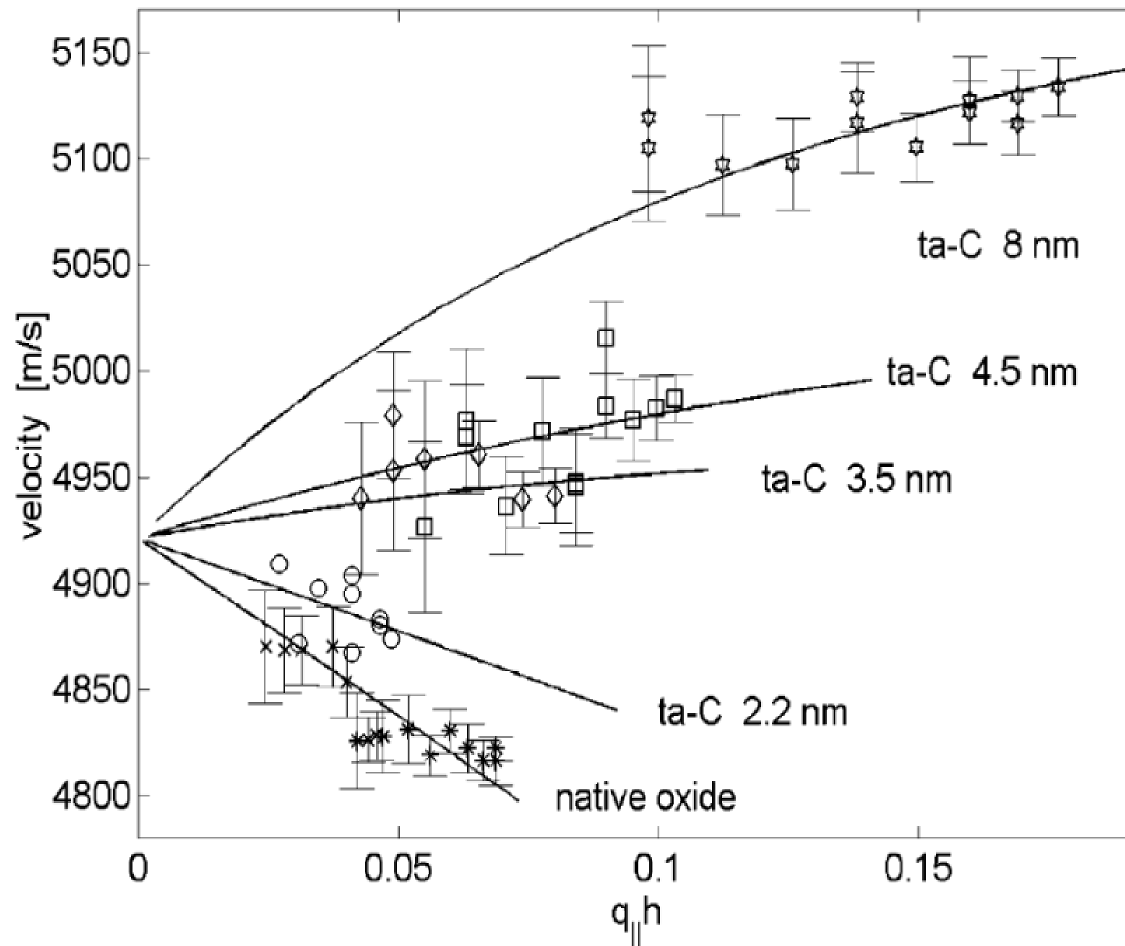


Fig. 2. Measured and computed dispersion relations for substrates A(*) and D(x), and samples of increasing thickness, B(□), C(▶), E(○) and F(◇), see Table 1. The error bars for sample E are not shown for clarity.

And there is whole lot more…But one can get an idea with simple methods:

- interference colors: thickness, absoption
- reflection: metals identification
- Scotts Tape Test: adhesion
- electrical conductivity
- scraching by a tip: hardness, adhesion, friction
- shine light tanget to surface: impurities, particles on film
- breathe moisture (no slime!): surface energy, hydrophilicity, adhesion (try only on your own samples)