''ज्ञान, विज्ञान आणि सुसंस्कार यांसाठी शिक्षण प्रसार'' – शिक्षणमहर्षी डॉ. बापूजी साळुंखे

Shri Swami Vivekanand Shikshan Sanstha, Kolhapur.

VIVEKANAND COLLEGE, KOLHAPUR

(Autonomous)

Affiliated to Shivaji University

NAAC Reaccrediated "A" | College with Potential for excellence | ISO 9001 : 2015

Characterization of Thin Film

By

Mr. A. V. Shinde (M.Sc. SET, GATE)

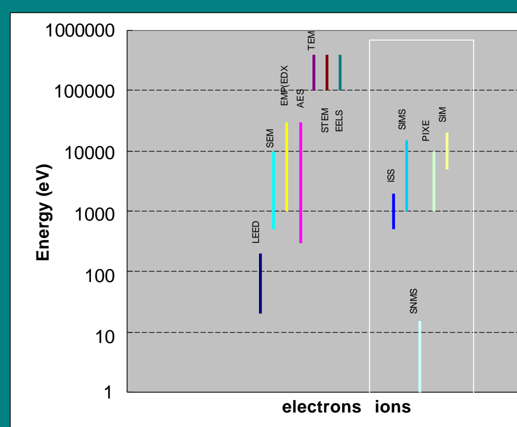
Date: 12/01/2019

Characterization of

Primary beam	Energy range	Secondary signal	Acronym	Technique	Application
Electron	20 - 200 eV 0.3 - 30 keV 1 - 30keV 500eV-10keV 100 -400 keV 100 -400 keV 100 -400 keV	Electron Electron X-ray Electron Electron Electron, X-ray Electron	LEED SEM EMP (EDX) AES TEM STEM EELS	Low-energy electron diffraction Scanning electron microscopy Electron microprobe Auger electron spectroscopy Transmission electron microscopy Scanning TEM Electron energy loss spectroscopy	Surface structure Surface morphology Surface region composition Surface layer composition High-resolution structure Imaging, X-ray analysis Local small-area composition
Ion	0.5 -2.0 keV 1 - 15 keV 1-15 eV 1 keV and up 5-20 keV >1 MeV	Ion Ion Atom X-ray Electron Ion	ISS SIMS SNMS PIXE SIM RBS	Ion-scattering spectroscopy Secondary ion mass spectroscopy Secondary neutral mass spectrometry Particle-induced X-ray emission Scanning ion microscopy Rutherford backscattering	Surface composition Trace composition vs depth Trace composition vs depth Trace composition Surface characterization Composition vs depth
Photon	>1 keV >1 keV >1 keV Laser Laser	X-ray X-ray Electron Ion Light	XRF XRD ESCA, XPS - LEM	X-ray fluorescence X-ray diffraction X-ray photoelectron spectroscopy Laser microprobe Laser emission microprobe	Composition (1 µm depth) Crystal structure Surface composition Composition of irradiated area Trace element analysis

J.B. Bindel, in *VLSI Technol*, McGraw-Hill, NY88

Characterization of Thin Films



Characterization of Thin Films

FILM THICKNESS

Optical methods

Interferometry
- reflection
- spectral

Ellipsometry
- reflection
- spectral

Mechanical methods
- **Profilometry**
- **Quartz crystal microbalace**
- **Ultrasound**

- elegant
- non-destructive
- high-resolution
- fast
- easy to implement

Characterization of Thin Films

FILM THICKNESS

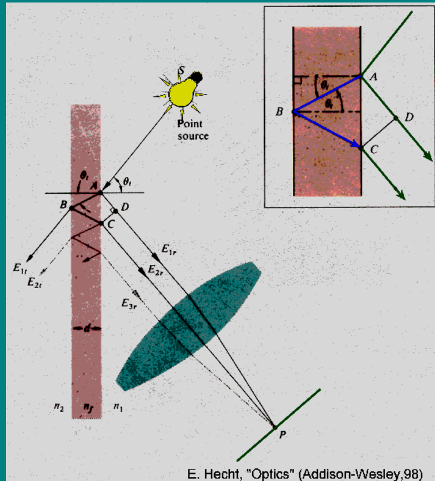
Optical methods

Method	**Range** (nm)	**Characteristics** (M = multiple)
Multiple beam interferometry (**FET**)	3 - 2000 ± 1-3	A step and reflective coating required (1λ, θ = 90°)
Multiple beam interferometry (**FECO**)	1 - 2000 ± 0.5	A step, reflective coating, and spectrometer required; time consuming *(M λ)*
VAMFO (variable-angle monochromatic fringe observation)	80 - 1000 ± 0.02-0.05%	Transparent films on reflective substrate (1 λ, M θ)
CARIS (constant-angle reflection interference spectroscopy)	40 - 2000 ± 1 nm	Transparent films on reflective substrate (M λ, θ = 90°)
Ellipsometry	<0.1-	Transparent films and multilayers, uses polarized light, measures *d,* n, and *k* (1 λ, fixed θ)
Spectral reflectometry (unpolarized)	~ 30 - 2000± 1 nm	Transparent films and multilayers, fast, measures *d, n,* and *k* (λ = ~ 200-1000 nm, θ = 90°) (polarized reflectometry is also performed at l λ, *M* θ)
Spectroscopic ellipsometry	<0.1-	Transparent films and multilayers, uses polarized light (M λ, fixed θ) (multiple-angle ellipsometry is also performed at l λ)

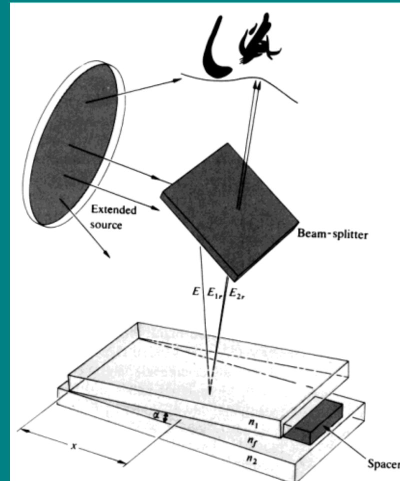
Characterization of Thin Films

FILM THICKNESS

Multiple-beam reflection



Fringes of equal inclination

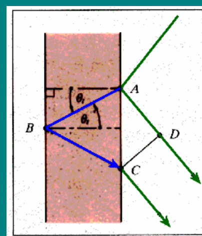


Fringes of equal thickness

Characterization of Thin Films

Interferometry: fringes basics

E. Hecht, Optics (Addison-Wesley, 98)



optical path difference $\Lambda = n_f(\overline{AB}+\overline{BC}) - n_1\overline{AD}$

$\overline{AB} = \overline{BC} = d/\cos\theta_t \rightarrow \Lambda = n_f \dfrac{2d}{\cos\theta_t} - n_1\overline{AD}$

$\overline{AD} = \overline{AC}\sin\theta_i;\ \overline{AD} = \overline{AC}\dfrac{n_f}{n_1}\sin\theta_t;\ \overline{AC} = 2d\tan\theta_t$

$\Lambda = n_f \dfrac{2d}{\cos\theta_t}(1-\sin^2\theta_t) = 2dn_f\cos\theta_t$

relative phase shift $\delta = k_0\Lambda \pm \pi = \dfrac{4\pi n_f}{\lambda_0}\cos\theta_t \pm \pi =$

$= \dfrac{4\pi d}{\lambda_0}\sqrt{n_f^2 - n^2\sin^2\theta_i} \pm \pi$

due to reflections

$\delta = 2m\pi \rightarrow$

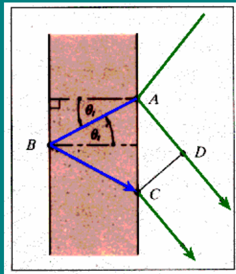
$d\cos\theta_t = (2m+1)\dfrac{\lambda_0}{4n_f}$ (maxima)

$d\cos\theta_t = (2m)\dfrac{\lambda_0}{4n_f}$ (minima)

Fringes of equal inclination

Characterization of Thin Films

Interferometry: fringes basics

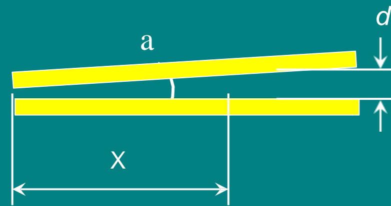


E. Hecht, Optics (Addison-Wesley, 98)

$$\delta = 2m\pi \rightarrow$$

$$d\cos\theta_t = (2m+1)\frac{\lambda_0}{4n_f} \quad \text{(maxima)}$$

$$d\cos\theta_t = (2m)\frac{\lambda_0}{4n_f} \quad \text{(minima)}$$



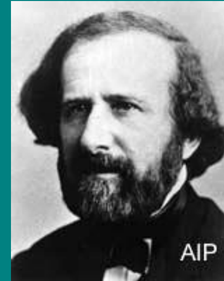
$d = x\alpha$, and for small θ_i:

$$(m+1/2)\lambda_0 = 2n_f d_m \quad \text{or}$$

$$(m+1/2)\lambda_0 = 2\alpha\, x_m n_f$$

$$x_{m+1} - x_m = \frac{\lambda_0}{2\alpha\, n_f} = \text{fringe spacing}$$

$n_f \approx 1$ for air



Fizeau, Armand (1819-1896)

(speed of light)

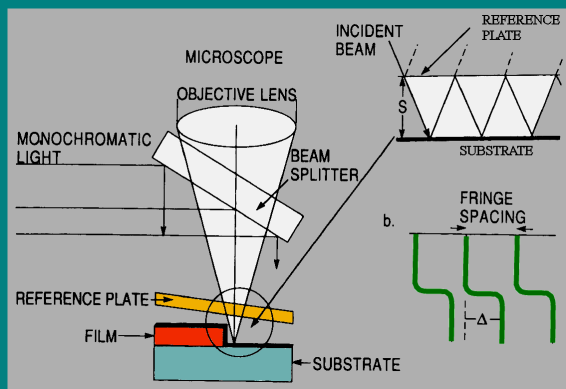
Fringes of equal thickness (Fizeau fringes)

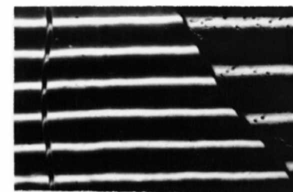
FET

Characterization of Thin Films

FILM THICKNESS- Optics

Multiple-beam reflectometry: non-transparent films




S Y El-Zaiat and H A El-Hennawi

Meas. Sci. Technol. 7 (1996) 1119–1123.

Figure 3. Microinterferogram showing multiple-beam Fizeau fringes crossing a Cashmeline fibre immersed in a liquid with refractive index n_L = 1.5240.

$$d = \frac{\Delta}{\text{Fringe spacings}} \frac{\lambda}{2}$$

For highly reflective surfaces, the fringe width is ~1/40 of Δ, and ~1/5 of that can be detected → the resolution is about 1/400 of λ, i.e. ~15 Å

Characterization of Thin Films

FILM THICKNESS- Optics

Fizeau Fringes

http://www.optics.arizona.edu/jcwyant/Short_Courses/Rochester/Part1.pdf

Hole

Top View

Reference

Test

1 2 3 4 5 6 7

S Δ

Interferogram

For a given fringe the separation between the two surfaces is a constant.

Height error = (λ/2)(Δ/S)

Bump

Top View

Reference

Test

1 2 3 4 5 6 7

S Δ

Interferogram

2002 - James C. Wyant

Part 1 Page 7 of 40

Characterization of Thin Films

FILM THICKNESS- Optics

Transparent films

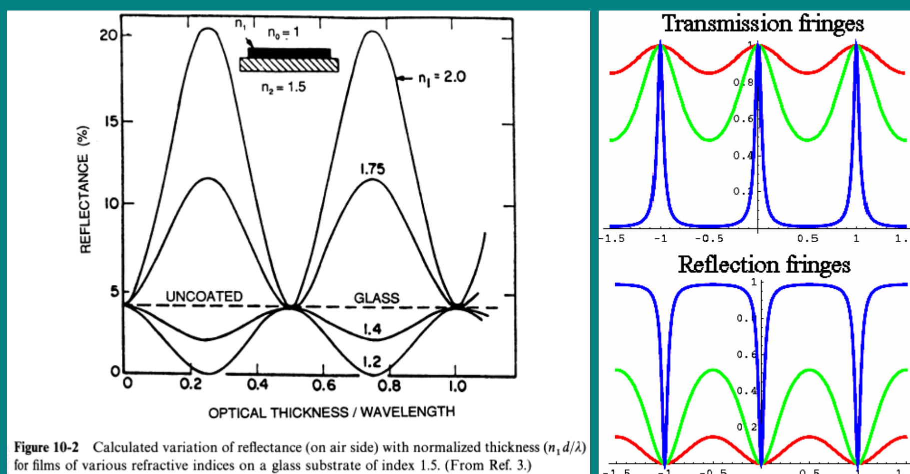
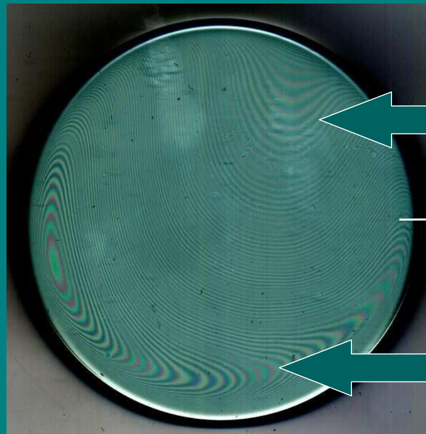


Figure 10-2 Calculated variation of reflectance (on air side) with normalized thickness ($n_1 d/\lambda$) for films of various refractive indices on a glass substrate of index 1.5. (From Ref. 3.)

Characterization of Thin Films

FILM THICKNESS- Optics

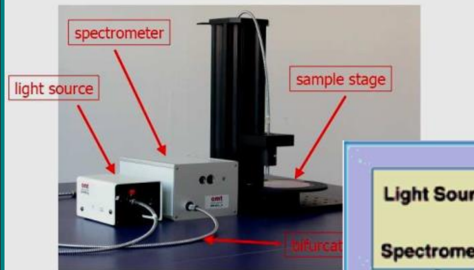
Multiple-beam reflectometry
...with a scanner

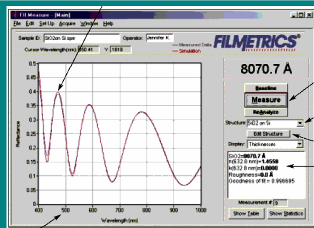


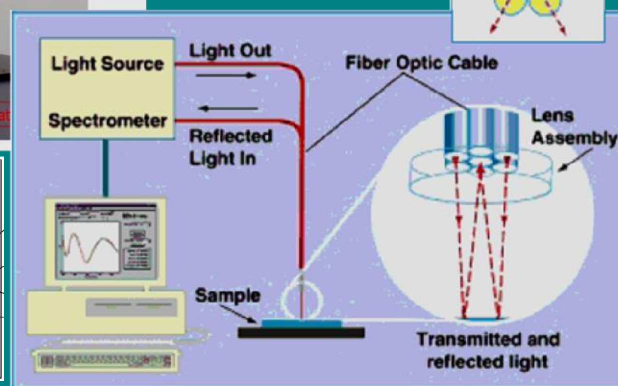
Characterization of Thin Films

FILM THICKNESS- Optics

Spectral Reflectometry
Transparent films and multilayers, fast, measures *d, n,* and *k* (λ = ~ 200-1000 nm, θ = 90°)
http://www.filmetrics.com/pdf/TMO.pdf







Characterization of Thin Films

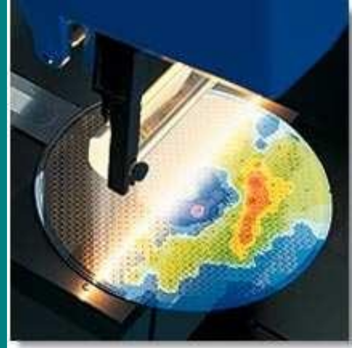
FILM THICKNESS- Optics

Spectral Reflectometry

http://www.filmetrics.com/

Patterned-Wafer Thickness and Defect Mapper

The STMapper uses a new scanning technique to acquire millions of spectral reflectance data points on a 200mm wafer in less than five seconds. An entire cassette of wafers can be mapped in less than five minutes. It can be used to monitor multiple process parameters on patterned wafers, such as ILD thickness, metal residual, and scratches and defects. Available in integrated or stand-alone configurations.

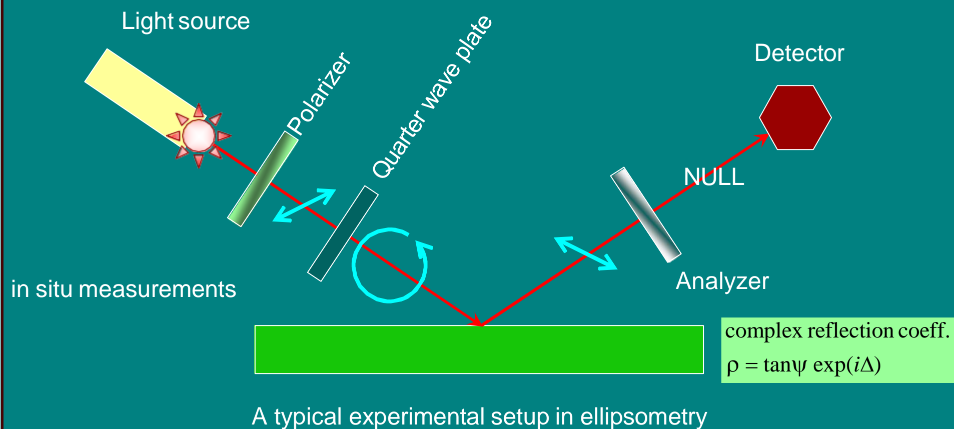


Characterization of Thin Films

FILM THICKNESS- Optics

Ellipsometry:

- reflection ellipsometry
- multiple angle of incidence
- spectroscopic ellipsometry



A typical experimental setup in ellipsometry

Characterization of Thin Films

FILM THICKNESS

Spectroscopic ellipsometry

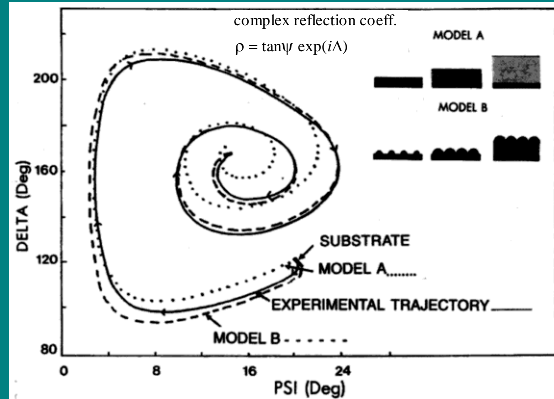


Figure 10-8 Experimental trajectory of Δ and ψ for the growth of an amorphous $Si_{0.65}Ge_{0.35}$:H film deposited by PECVD methods. Predicted trajectories for layer (Model A) and island growth (Model B) mechanisms are also indicated. (Courtesy of Instruments SA, Inc. Reprinted with permission.)

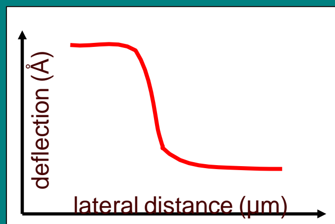
in situ measurements during thin film growth

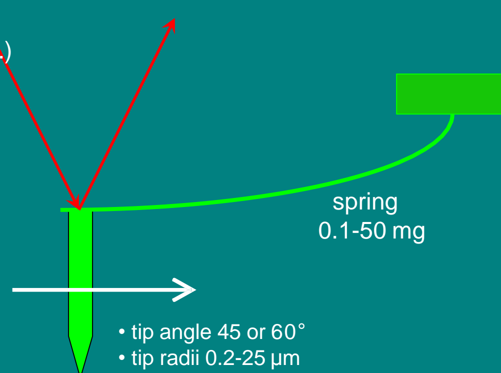
Characterization of Thin Films

FILM THICKNESS- Mechanics

Profilometry (one-shot AFM)

deflection measurements
(interferometry, capacitance, etc...)
resolution: ~1Å





- tip angle 45 or 60°
- tip radii 0.2-25 μm

thin film

substrate

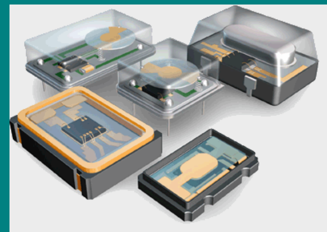
- scratching of thin film
- substrate roughness
- vibrations

Characterization of Thin Films

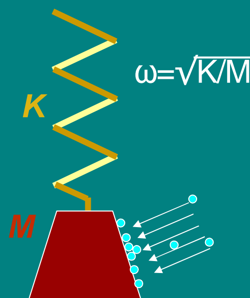
FILM THICKNESS- Mechanics

Quartz Crystal Microbalances

Ametist 13.9 x 10.6 x 8.3 cm^3, ~1 kg



clock crystals



$\omega = \sqrt{K/M}$

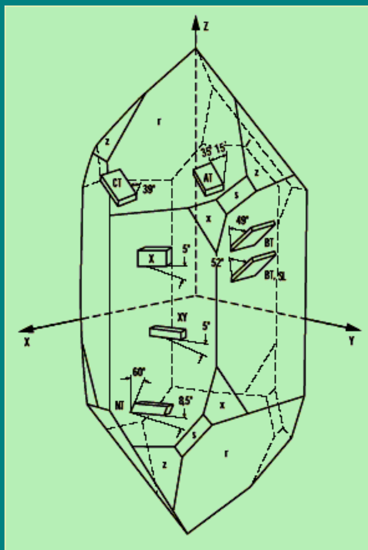
$$\delta f = -\frac{f^2 \delta m}{C \rho_f A} = -\frac{f^2 d}{C}$$

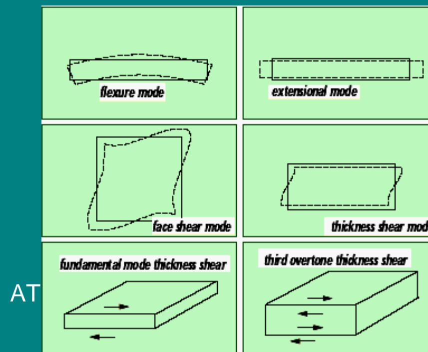
C=1656 kHz mm (AT cut)

Characterization of Thin Films

FILM THICKNESS- Mechanics

Quartz Crystal Microbalances

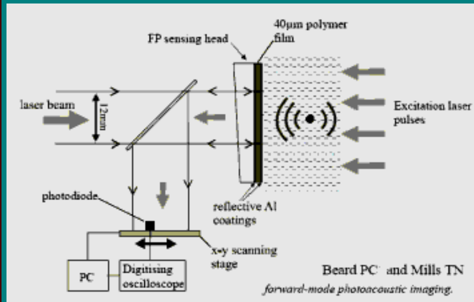


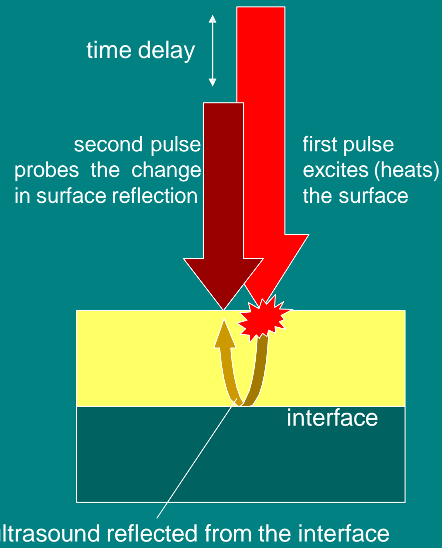


- f = 6 MHz typical (AT cut, thickn. shear)
- temperature sensitive (-)

Characterization of Thin Films

FILM THICKNESS- Mechanics Ultrasonic methods





local heating → local sudden expansion → acoustic wave pulse → change of reflection at the surface

Characterization of Thin Films

FILM THICKNESS- Mechanics Ultrasonic methods

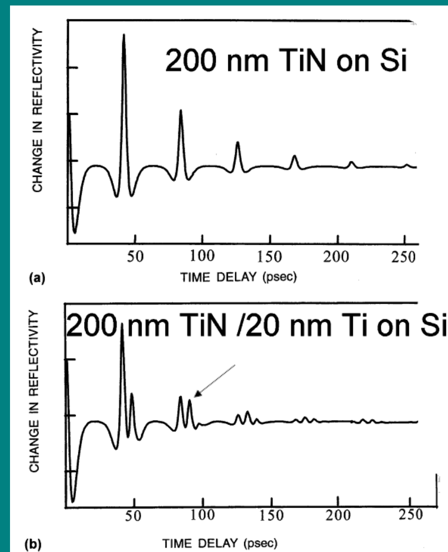
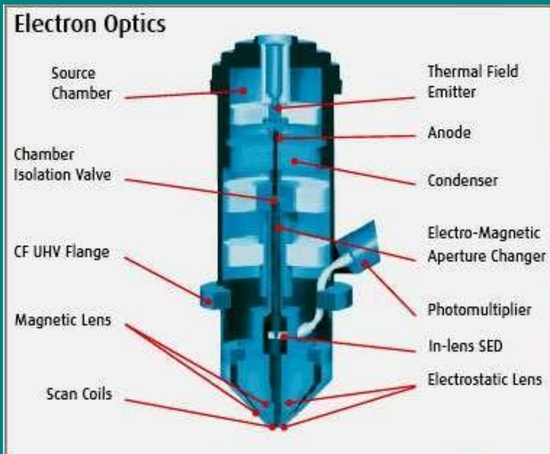


Figure 10-10 Time-dependent change in reflectivity for (a) 200 nm TiN/Si and (b) 200 nm TiN/20 nm Ti/Si. (Courtesy of G. J. Collins, Rudolph Technologies Inc. From Ref. 17.)

Characterization of Thin Films

STRUCTURAL CHARACTERIZATION

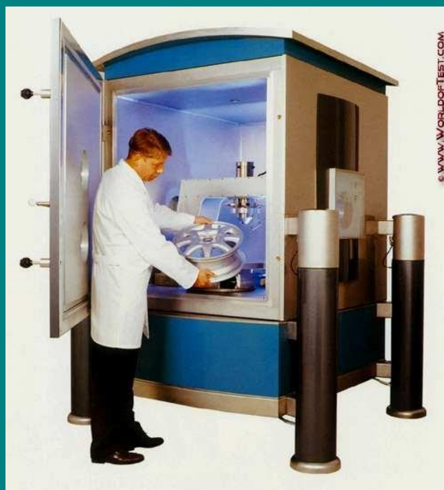
SEM

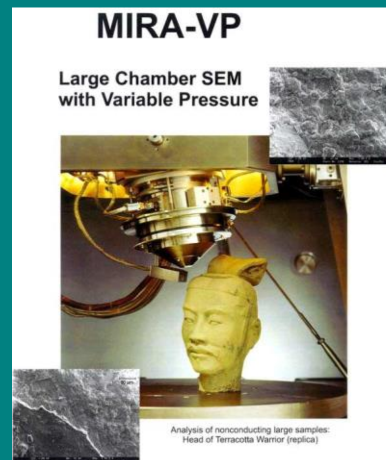


Characterization of Thin Films

STRUCTURAL CHARACTERIZATION

Large SEM

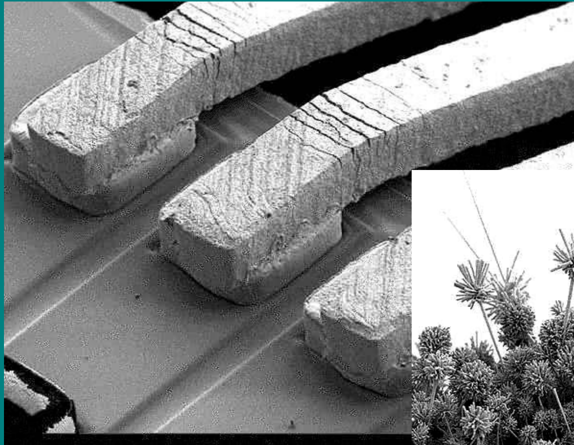




Characterization of Thin Films

STRUCTURAL CHARACTERIZATION

Jeol SEM


Integrated circuit bond pads ---2.5kV--- 500X -

Chemical vapor deposited copper oxide---5kV---100X

courtesy of Jim Stets, Air Products Co.

http://www.jeol.com/sem/gallery

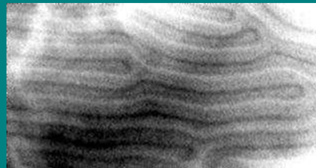
Characterization of Thin Films

STRUCTURAL CHARACTERIZATION

125 – 300 keV (up to 1 MeV) → short wavelength
100 keV → 0.035 Å

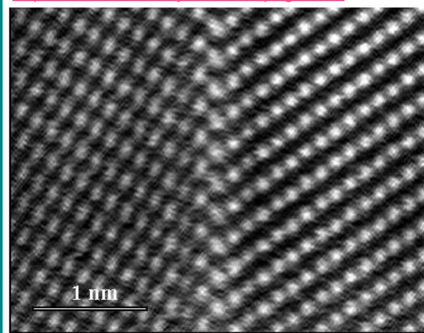
TEM

http://www.fzu.cz/~vystav/TEMpage.html


magnetic domains in Co

TEM cross sections

HRTEM: of coherent twin in molybdenum
Microscope: JEOL 4000EX
(CEA/DRFMC Grenoble)
http://www.fzu.cz/~vystav/TEMpage.html

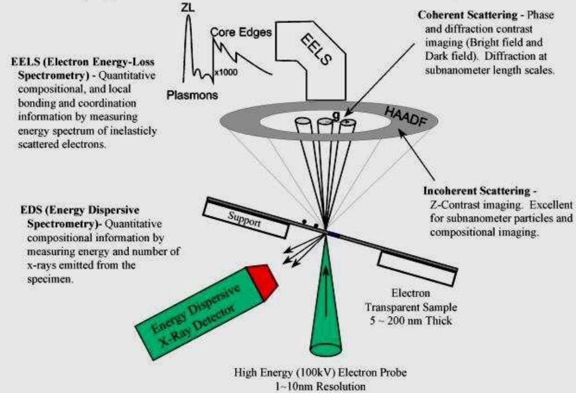


TEM modes:
- Bright-field imaging
- Dark-field imaging
- Lattice imaging
- Diffraction
- X-ray spectroscopy
- Electron energy loss spectroscopy
- Lorentz microscopy

Characterization of Thin Films

STRUCTURAL CHARACTERIZATION

TEM



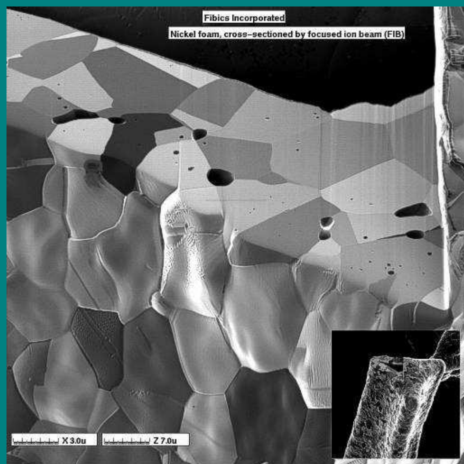
TEM modes:

- Bright-field imaging
- Dark-field imaging
- Lattice imaging
- Diffraction
- X-ray spectroscopy
- Electron energy loss spectroscopy
- Lorentz microscopy

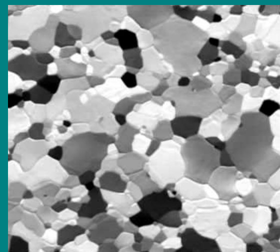
Characterization of Thin Films

STRUCTURAL CHARACTERIZATION

FIB microscopy



- Voltage contrast: insulators look dark while conductors are bright
- Materials contrast: differences in yield of secondary particles
- Crystallographic orientation contrast (channeling contrast)



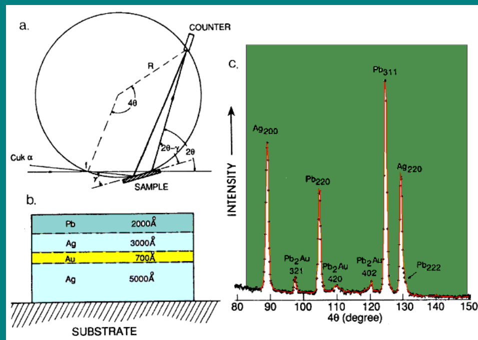
A grain-size distribution can be deduced

Characterization of Thin Films

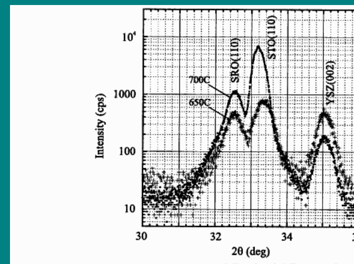
STRUCTURAL CHARACTERIZATION

X-ray diffraction

Monitoring of interdiffusion in thin films



Epitaxial thin films



Khaled Khamchane, Lic 2003

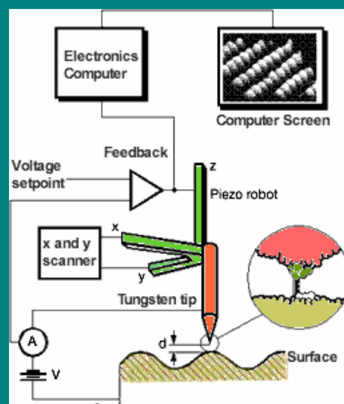
Figure 6.11: *The θ − 2θ scans of SRO/STO/CeO₂/YSZ films grown at 690°C and 700°C on Si substrate.*

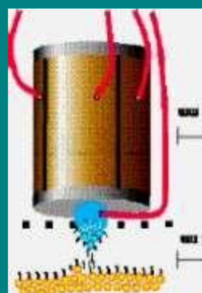
Characterization of Thin Films

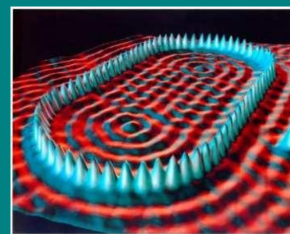
STRUCTURAL CHARACTERIZATION

STM

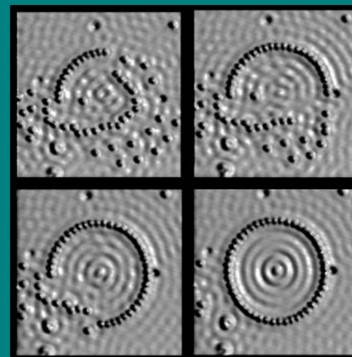
http://www.fysik.dtu.dk/stm/instrument.htm







STM allows manipulation of individual atoms



Fe on Cu

Crommie, Lutz & Eigler in http://www.almaden.ibm.com/

Characterization of Thin Films

CHEMICAL CHARACTERIZATION

- SEM/EDX (energy dispersive X-ray
- AES (Auger electron spectroscopy)
- XPS (X-ray photoelectron spectroscopy)
- RBS (Rutherford backscattering)
- SIMS (Secondary-ion mass spectroscopy)

capable of detecting almost all elements of the periodic table

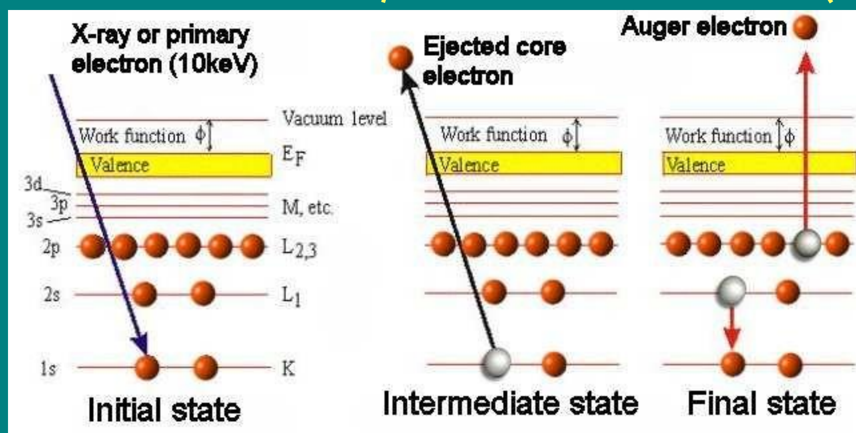
Summary of Major Chemical Characterization Techniques

Method	Elemental sensitivity	Detection limit (at.%)	Lateral resolution	Effective probe depth
Scanning electron microscope/energy dispersive X-ray (SEM/EDX)	Na–U	~0.1	~1 μm	~1 μm
Auger electron spectroscopy (AES)	Li–U	~0.1–1	500 Å	15 Å
X-ray photoelectron spectroscopy (XPS)	Li–U	~0.1–1	~100 μm	15 Å
Rutherford backscattering (RBS)	He–U	~1	1 mm	~200 Å
Secondary-ion mass spectrometry (SIMS)	H–U	10^{-4}	~1 μm	15 Å

Characterization of Thin Films

CHEMICAL CHARACTERIZATION

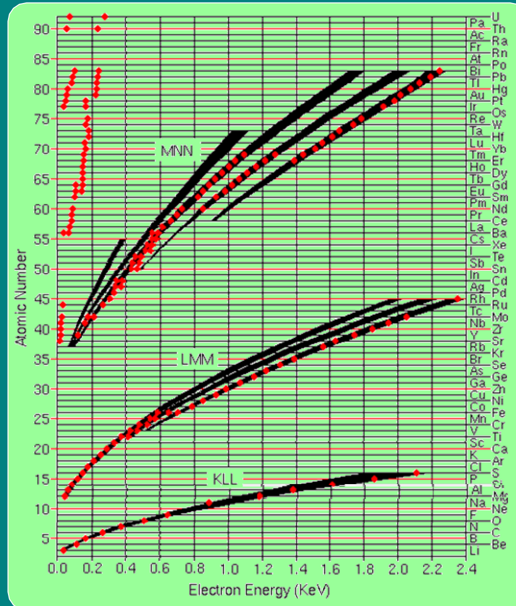
these can be collected and analyzed (XPS or AES)



Characterization of Thin Films

CHEMICAL CHARACTERIZATION

AES energies



Characterization of Thin Films

CHEMICAL CHARACTERIZATION

AES

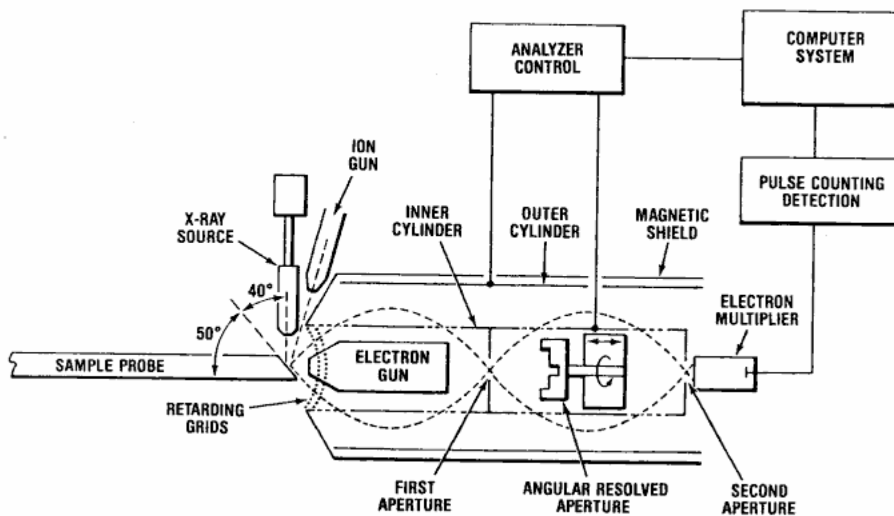
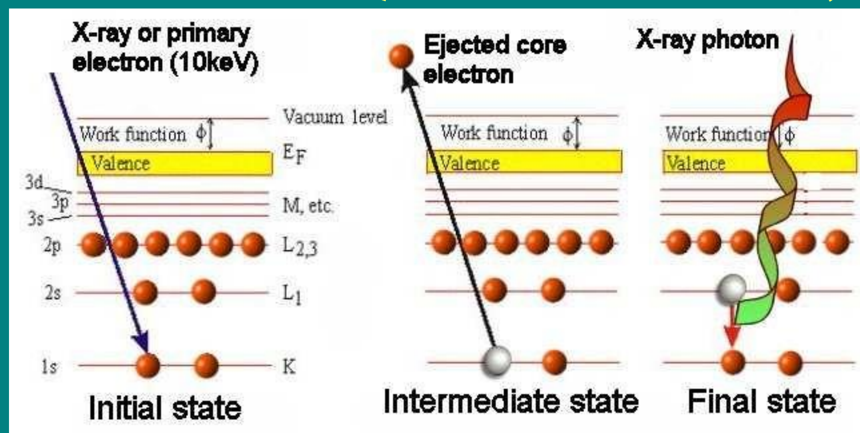


Figure 10-28 Schematic of spectrometer with combined AES and XPS capabilities. (Courtesy of Physical Electronics Industries, Inc.)

Characterization of Thin Films

CHEMICAL CHARACTERIZATION

these can be collected and analyzed



Characterization of Thin Films

CHEMICAL CHARACTERIZATION

Auger Electron Spectroscopy (AES)

• The sample is irradiated with a high energy primary electron beam (2 - 10 keV).

•Backscattered, secondary, and Auger electrons can be detected and analyzed. These can also be used for imaging purposes similar to that in SEM.

•The Auger electrons are emitted at discrete energies, that are characteristic of the elements present on the sample surface (the peak positions are used to identify the elements and their chemical states) All elements in the periodic table, except hydrogen and helium, can be detected, and

• The depth of analysis is 3 - 5 nm

•In the scanning mode, the secondary electrons yield information on the surface topography. Excellent spatial resolution (0.5 μm).

• Top layers can be spattered with ions and depth profiles can be measured.

Characterization of Thin Films

CHEMICAL CHARACTERIZATION

X-RAY PHOTOELECTRON SPECTROSCOPY (XPS)
also Electron Spectroscopy for Chemical Analysis (ESCA)

- The sample is irradiated with soft X-rays photons (1-2 keV) which induces direct emission of photoelectrons.
- The energy of photoelectrons is characteristic of the material.
- Depth 2-20 atomic layers.
- Peak position and area are used to study the composition. The peak shape give information about the chemical bonds of the elements.

Modes of Operation

1. Energy spectrum. Survey spectra (0-1000 eV) - to estimate the composition, while high-resolution spectra (10-20 eV) - information about the chemical bonds.
2. Mapping. Choosing a single peak and scanning the focal point across the sample gives information on the lateral distribution of species on the surface.
3. Imaging with high spatial resolution (<10-15 μm) and high sensitivity.

Characterization of Thin Films

CHEMICAL CHARACTERIZATION

XPS vs AES (are complimentary to each othe)

- Give similar information.
- The Auger spot size is smaller than the XPS.
- The XPS spectra are well-documented -> study of surface chemical bonding through the use of tabulated chemical shifts. The Auger chemical shifts are weaker.
- X-rays produce less damage to the surface compared to the primary electrons of AES.

Characterization of Thin Films

CHEMICAL CHARACTERIZATION

Secondary Ion Mass SpectroscopyDescription **(SIMS)**
The most sensitive method for detection of elements.

- masses up to 10^4 mass units can be detected;
- separation of isotopes can be made;
- chemical information can be obtained by identifying sputtered ions;
- detection limits of 1 ppm of a monolayer;
- surface sensitivity < 1nm; depth resolution < 1nm; lateral resolution 100 nm.

Modes of operation:

1. Surface analysis – (Static SIMS) - low primary ion densities to prevent surface destruction
2. Imaging. Focused ion beam scanning over the surface produces images of the surface (recall FIB !)
3. Depth profiling (Dynamic SIMS) - high primary ion dose densities to remove the surface layer by layer. Spectra taken during the sputtering can give the thickness distribution of elements

Characterization of Thin Films

CHEMICAL CHARACTERIZATION

http://hyperphysics.phy-astr.gsu.edu/hbase/magnetic/maspec.html

Ionization

Accelerating voltage applied

Velocity selector

Magnetic field region

B out toward viewer

Detector

After ionization, acceleration, and selection of single velocity particles, the ions move into a mass spectrometer region where the radius of the path and thus the postion on the detector is a function of the mass.

$$r = \frac{mv}{qB} = \frac{mE_s}{qBB_s}$$

The mass spectrometer is an instrument which can measure the masses and relative concentrations of atoms and molecules.

It makes use of the magnetic force on a moving charged particle.

Characterization of Thin Films

CHEMICAL CHARACTERIZATION

SIMS

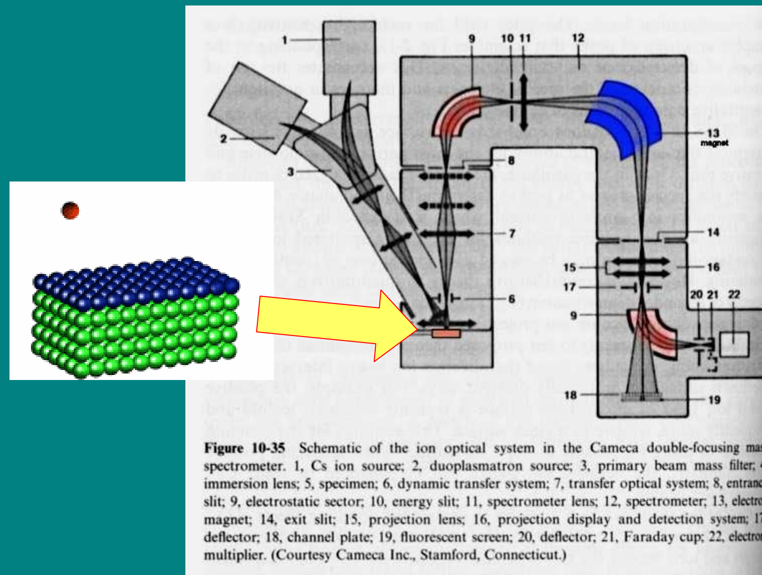


Figure 10-35 Schematic of the ion optical system in the Cameca double-focusing mass spectrometer. 1, Cs ion source; 2, duoplasmatron source; 3, primary beam mass filter; 4, immersion lens; 5, specimen; 6, dynamic transfer system; 7, transfer optical system; 8, entrance slit; 9, electrostatic sector; 10, energy slit; 11, spectrometer lens; 12, spectrometer; 13, electromagnet; 14, exit slit; 15, projection lens; 16, projection display and detection system; 17, deflector; 18, channel plate; 19, fluorescent screen; 20, deflector; 21, Faraday cup; 22, electron multiplier. (Courtesy Cameca Inc., Stamford, Connecticut.)

Characterization of Thin Films

CHEMICAL CHARACTERIZATION

RBS

Easiest to understand: two-body elastic scattering

Rutherford backscattering is an analytical technique in which a high energy beam (2 - 4 MeV) of low mass ions (He ++) is directed at a surface. A detector collects particles which scatter from the sample at close to a 180 degree angle.

•The energy of scattered ions depend on their incident energy and on the mass of the sample atom which they hit. The energy of scattered ions therefore indicates the chemical composition of the sample.

•RBS can be used to perform a depth profile of the composition of a sample. This is especially useful in analysis of thin-film materials.

CHEMICAL CHARACTERIZATION

RBS

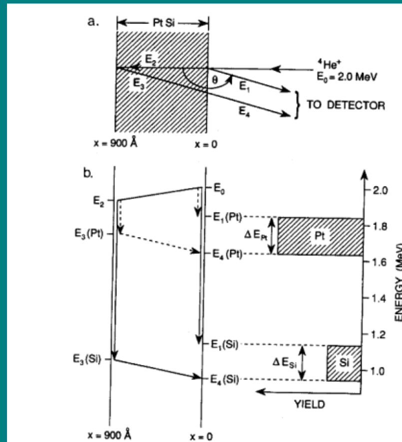


Figure 10-31 (a) Geometry of scattering and notation of energies at the front and back surfaces of a 900 Å thick PtSi film. (b) $^4He^+$ ion energy as a function of film depth due to scattering from Pt and Si. Schematic RBS spectrum shown rotated by 90°. (From W. K. Chu, J. W. Mayer, M. A. Nicolet, T. M. Buck, G. Amsel, and F. Eisen, *Thin Solid Films* **17**, 1 (1963), with permission from Elsevier Sequoia S.A.)

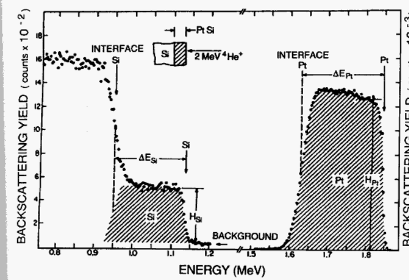


Figure 10-34 Energy spectrum for 2 MeV $^4He^+$ ions backscattered from 900 Å of PtSi. (From W. K. Chu, J. W. Mayer, M. A. Nicolet, T. M. Buck, G. Amsel, and F. Eisen, *Thin Solid Films* **17**, 1 (1963), with permission from Elsevier Sequoia S.A.)

$$E_1 = \left[\frac{\sqrt{M^2 - M_0^2 \sin^2\theta} + M_0 \cos\theta}{M_0 + M}\right]^2 E_0$$

$$E_1 = K_M E_0$$

Home assignment:

- Assume that across a 5MHz AT-cut quartz crystal 0.334 mm thick, with the quality factor Q of 20000, and the piezoelectric strain coefficient of 3.1×10^{-12} m/V, the resonance a.c. voltage of 1V peak-to-peak is applied.

Evaluate the acceleration of a thin film deposited on the surface of this quartz-crystal resonator.

THANK YOU