Characterization of Thin Films

- Film thickness
- Surface morphology
- Film composition
- Film properties

Characterization methods are very diverse

- Size of equipment. Compare desktop interferometer with 2 m interferometer of RBS.
- Cost. $10^3 - $10^6
- Environment. Ambient = 10^-10 Torr
- Complexity. Scotch tape = SEM, TEM etc.

FILM THICKNESS

Optical methods

- Interferometry
  - reflection
  - spectral
- Ellipsometry
  - reflection
  - spectral

Mechanical methods

- Profilometry
- Quartz crystal microbalance
- Ultrasound

FILM THICKNESS

<table>
<thead>
<tr>
<th>Method</th>
<th>Range (nm)</th>
<th>Characterization (as example)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Interferometry (FET)</td>
<td>1-2000 nm</td>
<td>Young's reflection, coherence (M, F; 0°)</td>
</tr>
<tr>
<td>Interferometry (FECO)</td>
<td>1-2000 nm</td>
<td>Young's reflection, coherence (M, F; 0°)</td>
</tr>
<tr>
<td>VASED (variable angle spectrometry)</td>
<td>0.01 - 100 nm</td>
<td>Thin film on reflective substrate (M, F; 0°)</td>
</tr>
<tr>
<td>CARD (constant angle reflectance)</td>
<td>0 - 200 nm</td>
<td>Thin film on reflective substrate (M, F; 0°)</td>
</tr>
<tr>
<td>Ellipsometry</td>
<td>&lt;10</td>
<td>Thin film and multilayer, non-polarized light, measurement a, b, and c, 2F, 3F</td>
</tr>
<tr>
<td>Spectroscopic ellipsometry (spectroscopic)</td>
<td>1 - 1000 nm</td>
<td>Thin film and multilayer, non-polarized light, measurement a, b, and c, 2F, 3F</td>
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<td>1 - 1000 nm</td>
<td>Thin film and multilayer, non-polarized light, measurement a, b, and c, 2F, 3F</td>
</tr>
</tbody>
</table>
Interferometry: fringes basics

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

\[ \Delta = \frac{2n \lambda}{d} \]

Fringes of equal inclination

\[ \Delta = \frac{2n \lambda}{d} \]

Fringes of equal thickness

Fizeau fringes

Fringes of equal thickness (Fizeau fringes)

\[ n = 1 \text{ for air} \]

For highly reflective surfaces, the fringe width is \( \approx 1/40 \) of \( \lambda \), and \( \approx 1/5 \) of that can be detected — the resolution is about \( 1/400 \) of \( \lambda \), i.e., \( \approx 15 \) Å.

Multiple-beam reflectometry: non-transparent films

FILM THICKNESS- Optics

For transparent films

FILM THICKNESS- Optics

Multiple-beam reflectometry...with a scanner

FILM THICKNESS- Optics

Multiple-beam reflectometry

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

\[ \text{Height error} = (0.7)(\frac{\lambda}{\Delta}) \]

\[ n = 1 \text{ for air} \]

For highly reflective surfaces, the fringe width is \( \approx 1/40 \) of \( \lambda \), and \( \approx 1/5 \) of that can be detected — the resolution is about \( 1/400 \) of \( \lambda \), i.e., \( \approx 15 \) Å.

Multiple-beam reflectometry: non-transparent films

FILM THICKNESS- Optics

Fringe spacing

\[ d = \Delta \]

Fringes

\[ n = 1 \text{ for air} \]

For highly reflective surfaces, the fringe width is \( \approx 1/40 \) of \( \lambda \), and \( \approx 1/5 \) of that can be detected — the resolution is about \( 1/400 \) of \( \lambda \), i.e., \( \approx 15 \) Å.
FILM THICKNESS - Optics

Spectral Reflectometry

Transparent films and multilayers, fast, measures d, n, and k (λ = 200-1000 nm, θ = 30°)

http://www.filmetrics.com/pdf/TMO.pdf

Patterned-Water 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.

FILM THICKNESS - Optics

Ellipsometry:
- reflection ellipsometry
- multiple angle of incidence
- spectroscopic ellipsometry

Spectroscopic ellipsometry

A typical experimental setup in ellipsometry

FILM THICKNESS - Mechanics

Profilometry (one-shot AFM)

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

Spring 0.1-50 mg

- tip angle 45 or 60°
- tip radius 0.3-25 µm

in situ measurements during thin film growth
FILM THICKNESS - Mechanics

Quartz Crystal Microbalances

\[ \omega = \sqrt{K/M} \]

Amethyst 13.9 x 10.6 x 8.3 cm\(^3\), ~1 kg

\[ C = 1656 \text{ kHz mm (AT cut)} \]

FILM THICKNESS - Mechanics

Quartz Crystal Microbalances

\[ f = 6 \text{ MHz typical (AT cut, thickn. shear)} \]

\[ \text{temperature sensitive (-)} \]

FILM THICKNESS - Acoustics

Ultrasonic methods

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

ultrasound reflected from the interface

FILM THICKNESS - Acoustics

Ultrasonic methods

time delay

second pulse probes the change in surface reflection

first pulse excites (heats) the surface

200 nm TiN on Si

200 nm TiN/20 nm Ti on Si

STRUCTURAL CHARACTERIZATION

SEM

JEOL JSM-6351F SEM

Installed: 1994

Funded by: Wallenberg

Max sample size: 100 mm resolution 1.2 nm specified

Acc. voltages 0.5kV - 30kV

Source: Cold Field Emission

MCZ's SEM
**STRUCTURAL CHARACTERIZATION**

**Large SEM**

**Jeol SEM**

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

**TEM**

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

TEM cross sections

**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!

**X-ray diffraction**

Monitoring of interdiffusion in thin films.
STRUCTURAL CHARACTERIZATION

STM

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

STM allows manipulation of individual atoms

Fe on Cu


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)

capability of detecting almost all elements of the periodic table

Summary of Major Chemical Characterization Techniques

<table>
<thead>
<tr>
<th>Method</th>
<th>Elemental sensitivity</th>
<th>Detection limit (at %)</th>
<th>Lateral resolution</th>
<th>Depth of depth</th>
</tr>
</thead>
<tbody>
<tr>
<td>Scanning electron microscopy/energy dispersive X-ray (SEM/EDX)</td>
<td>Na – U</td>
<td>~0.1</td>
<td>~1 μm</td>
<td>~1 μm</td>
</tr>
<tr>
<td>Auger electron spectroscopy (AES)</td>
<td>Li – U</td>
<td>~0.1 – 1</td>
<td>100 Å</td>
<td>15 Å</td>
</tr>
<tr>
<td>X-ray photoelectron spectroscopy (XPS)</td>
<td>Li – U</td>
<td>~0.1 – 1</td>
<td>100 μm</td>
<td>15 Å</td>
</tr>
<tr>
<td>Rutherford backscattering (RBS)</td>
<td>He – U</td>
<td>~1</td>
<td>1 mm</td>
<td>~200 Å</td>
</tr>
<tr>
<td>Secondary-ion mass spectroscopy (SIMS)</td>
<td>He – U</td>
<td>~10^4</td>
<td>~1 Å</td>
<td>~10 Å</td>
</tr>
</tbody>
</table>

CHEMICAL CHARACTERIZATION

AES

these can be collected and analyzed

X-ray or primary electron (10keV)

Initial state

Vacuum level

Final state

Intermediate state

Ejected core electron

Auger electron

Work function φ

Spin

Energy

CHEMICAL CHARACTERIZATION

AES

these can be collected and analyzed

X-ray or primary electron (10keV)

Initial state

Vacuum level

Final state

Intermediate state

Ejected core electron

X-ray photon

Work function φ

Spin

Energy

August Yurgens
### 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.

#### X-RAY PHOTON ELECTRON 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.

#### Secondary Ion Mass Spectroscopy (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

#### FILMS

#### SIMS

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.

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

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.

---

**Mechanical Properties**

**Methods**

*Indentation*

Nano indentation test can be used in the analysis of organic and inorganic soft and hard coatings. Examples are thin and multilayer PVD, CVD, PECVD, photoresist, and many other types of films. Substrates can be hard or soft, including metals, semiconductors, glass, and organic materials.

**Deflection of microbeams**

also resonance frequency of induced oscillations


**Bulge testing**

- $P = \frac{4hP}{r^2}$
- $\sigma_r = \frac{2 E h^3}{3(1-v)r^3}$
- $\sigma_r$ - residual stress
- $h$ - dome height
- $r$ - radius and thickness
Measurements of Internal Stresses

Adhesion Tests

Improving ADHESION

- Low-energy pre-sputtering
- Ion-beam-assisted deposition
- Reactive ion implantation
- Ion-beam stitching (disorder)
- Chemical etching/cleaning

Electrical resistivity measurements

Electrical resistivity: T-dependence
**Properties of Thin Films**

### Electrical Properties

**Electrical resistivity: T-dependence**

**Table 10.1 - Electrical Properties of Thin and Thick Film Resistive Materials**

<table>
<thead>
<tr>
<th>Material</th>
<th>Resistivity (ohm·cm)</th>
<th>TEC (ppm/°C)</th>
<th>YEC (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni-Hg</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
</tr>
<tr>
<td>Ni-NiCu62Sn38</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
</tr>
<tr>
<td>Ni-Fe19Cu60Sn21</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
</tr>
<tr>
<td>Ni-Fe19Cu60Sn21</td>
<td>0.01</td>
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<td>Ni-Fe19Cu60Sn21</td>
<td>0.01</td>
<td>0.01</td>
<td>0.01</td>
</tr>
</tbody>
</table>

**Properties of Thin Films**

**Electrical Properties: size effect**

- $J_s = 5 \text{ Å for Cu, but}$
- $J_s = 400 \text{ Å for Bi}$
- Specular reflection
- The topology of the Fermi surface is important in understanding the electronic properties of materials.
- Fermi sphere for free electrons in a 3D-box ($K, N, R_B$)

**Properties of Thin Films**

**Magneto-Electric Properties: GMR**

**Superconductivity**

- Discovered in 1911 by Kamerlingh Onnes:
  - Superconductivity of Hg

**Table 10.2 - Values of $T_c$ and $R_0$ for Superconducting Materials**

<table>
<thead>
<tr>
<th>Material</th>
<th>$T_c$ (K)</th>
<th>$R_0$ (ohm·cm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ag</td>
<td>1.18</td>
<td>0.66</td>
</tr>
<tr>
<td>Cu</td>
<td>0.35</td>
<td>1.15</td>
</tr>
<tr>
<td>Ni</td>
<td>2.20</td>
<td>0.68</td>
</tr>
<tr>
<td>Pd</td>
<td>2.85</td>
<td>0.65</td>
</tr>
<tr>
<td>Pt</td>
<td>7.05</td>
<td>0.65</td>
</tr>
<tr>
<td>Au</td>
<td>1.15</td>
<td>0.65</td>
</tr>
<tr>
<td>Pb</td>
<td>3.25</td>
<td>0.65</td>
</tr>
<tr>
<td>Sn</td>
<td>4.76</td>
<td>0.65</td>
</tr>
<tr>
<td>Sb</td>
<td>5.97</td>
<td>0.65</td>
</tr>
<tr>
<td>Bi</td>
<td>12.9</td>
<td>0.65</td>
</tr>
<tr>
<td>Hg</td>
<td>13.8</td>
<td>0.65</td>
</tr>
<tr>
<td>Tl</td>
<td>14.2</td>
<td>0.65</td>
</tr>
<tr>
<td>PbSe</td>
<td>13.0</td>
<td>0.65</td>
</tr>
<tr>
<td>PbTe</td>
<td>13.0</td>
<td>0.65</td>
</tr>
<tr>
<td>InSb</td>
<td>13.0</td>
<td>0.65</td>
</tr>
</tbody>
</table>

**Properties of Thin Films**

**Electrical Properties: Superconductivity**

When the mean-free path becomes larger than the thin-film thickness, the film boundaries take over the scattering of electrons. Size effect.
**Superconductivity**

Bulk and room-temperature deposited Bi-films are semi-metallic. Quench-condensed Bi thin films are metallic and superconducting!

---

**High-Tc Superconductivity**

-10 mA/cm²

Superconducting motor


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**Superconductivity and tunneling**

Fig. 4: The I-V characteristics of an Al-Fe junction (Al screen) after Gomes [46] (after: Physica C).