Characterization, Testing of Nanotechnology Structures and Materials

E SC 216

Unit 1
Basic Techniques

Lecture 1
Characterizing with Light or Physical Properties

Outline

- Introduction
- Optical Microscopes
- Profilometry
- Ellipsometry
- Reflective Spectroscopy
- Contact Angle
- Film Stress
- Sheet Resistance

Metrology

A group of processes used to determine physical and chemical properties of the nanoproduct during and / or after the fabrication process

Metrology tools are essential to determine the quality of fabrication processes and their resultant materials and devices.

Yield

The percentage of quality goods produced out of the total group of goods started.

Yield is enhanced by the ability to <u>measure specific characteristics</u> of a substrate in production. It is used to <u>improve upon manufacturing processes</u> if necessary.

In-situ Characterization

- In-situ characterization is a key part of manufacturing today. It provides information about the quality of a device before it is completed.
- Improves yield, minimizes waste, monitors processes, provides data for statistical process control.
- This process is often non-invasive

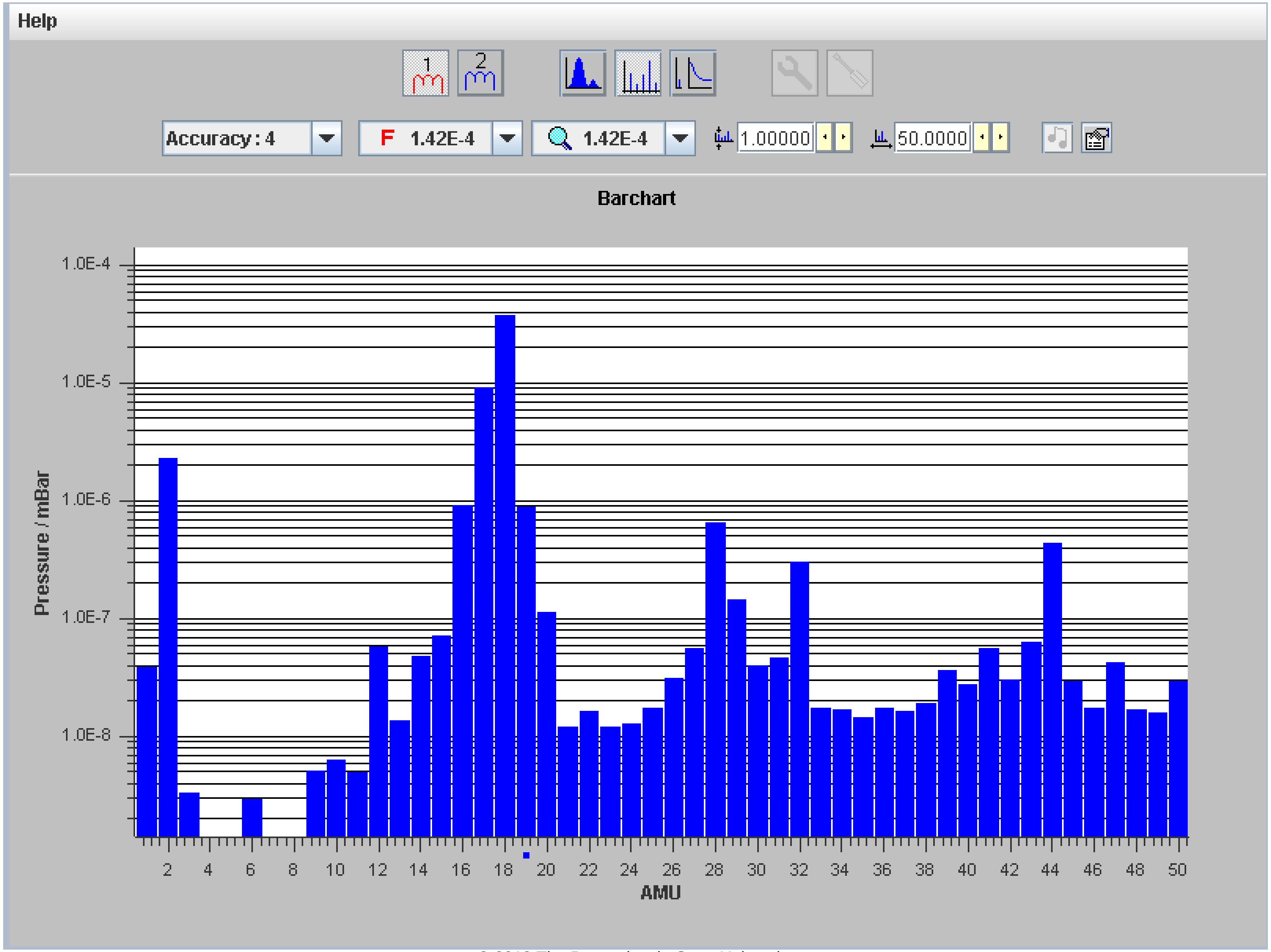
In-situ Characterization

The use of characterization tools in-line to gauge the progress of a manufacturing process.

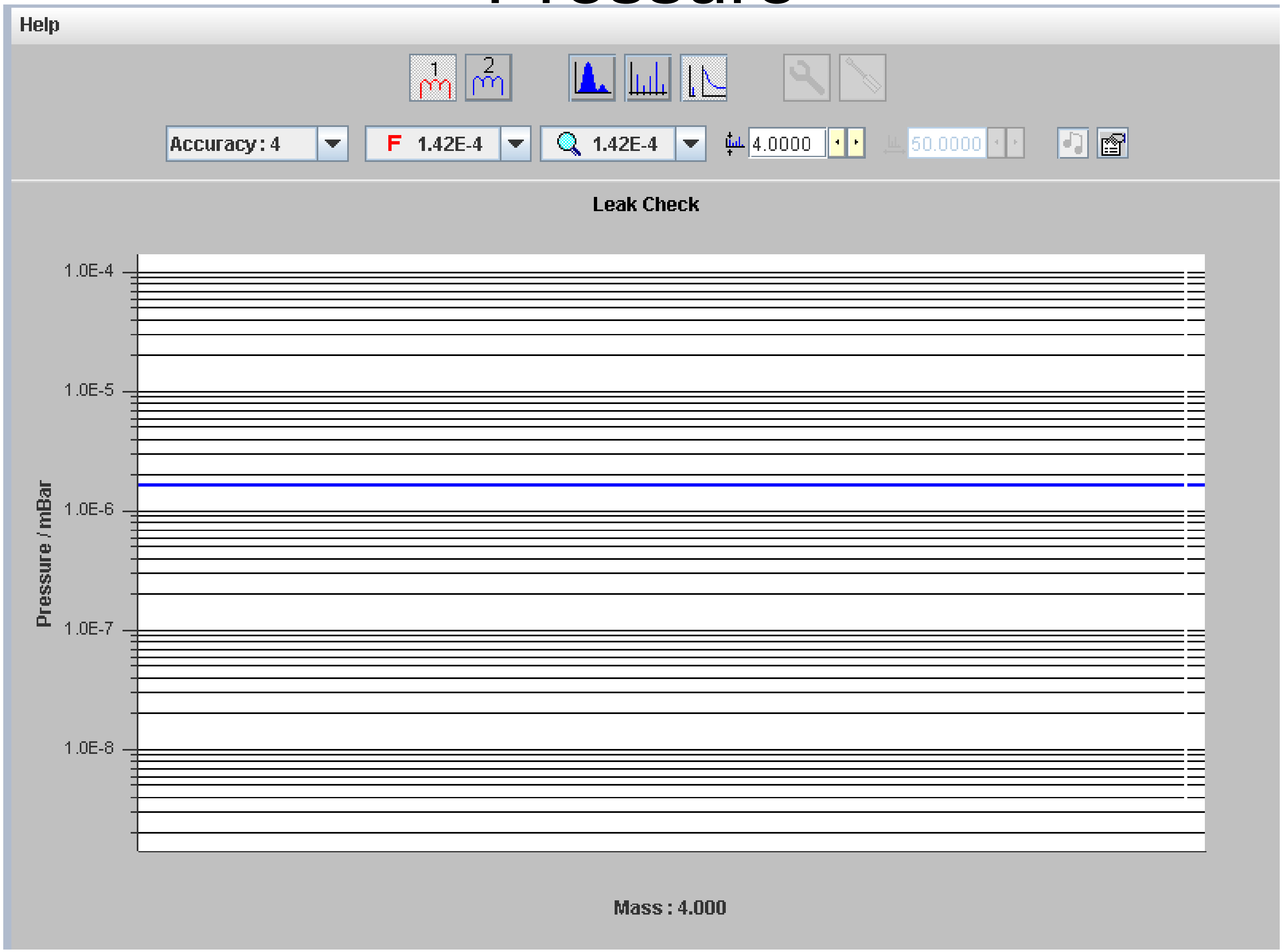
What can be measured in-situ?

substrate temperature, substrate surface quality, film thickness, growth/etch rates, optical constants, residual gas, etc.

Residual Gas Analyzer Graph



Residual Helium at $1.5x10^{-4}$ T System Pressure



Ex-situ Characterization

- This is characterization that necessitates taking a product out of the production line for testing and characterization.
- Reasons for performing certain characterization techniques ex-situ:
  - May be invasive
  - May require special sample preparation
  - May be low throughput
- Whether or not a characterization method is performed in-situ or ex-situ depends on the production process and may vary from process to process.

Ex-situ Characterization

Some Common Examples:

- Structural Characterization Techniques:
  - X-ray Diffraction (XRD)
  - X-ray Photoelectron Spectroscopy (XPS)
  - Auger Electron Spectroscopy (AES)
  - Secondary Ion Mass Spectroscopy (SIMS)

Ex-situ Characterization

Some Common Examples:

- Microscopy:
  - Transmission Electron Microscopy (TEM)
  - Scanning Probe Microscopy (SPM) – AFM and STM
  - Scanning Electron Microscopy (SEM)

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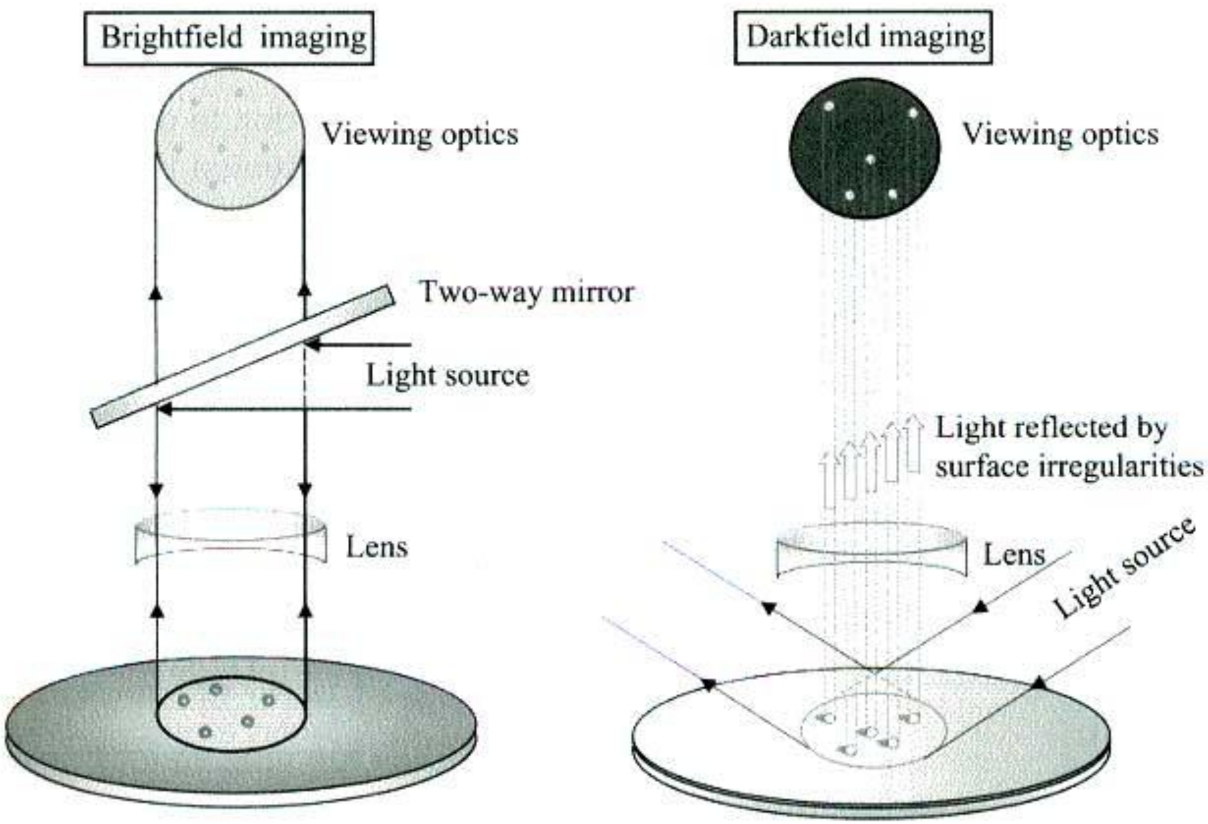
Optical Microscopy

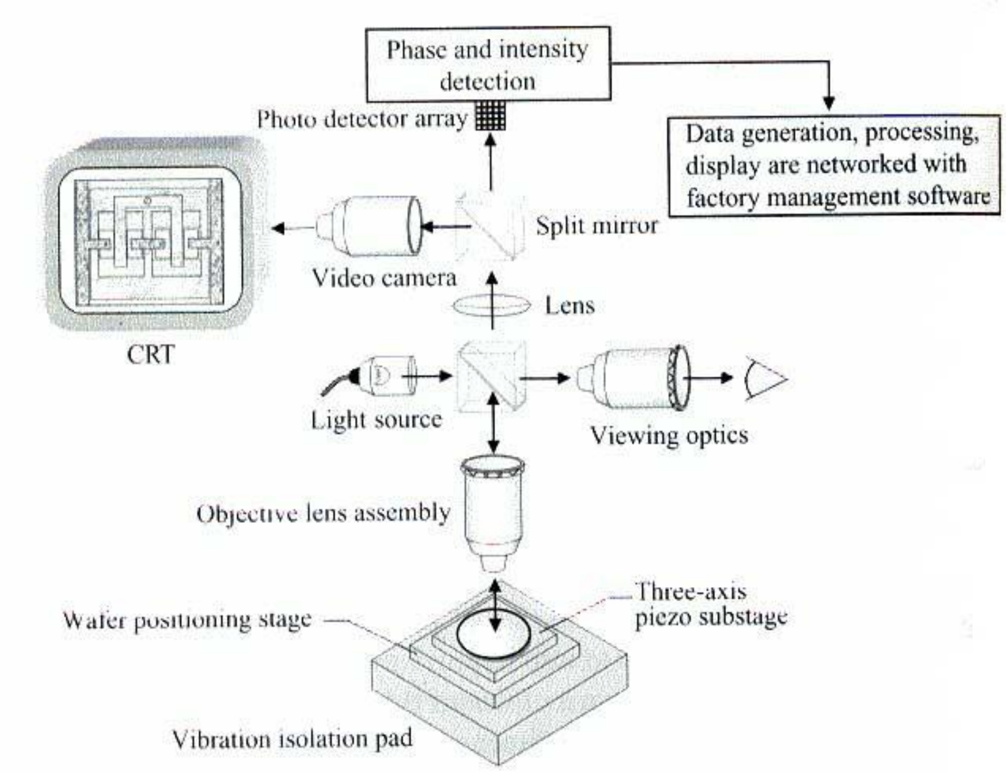
- This microscope is the oldest type of microscope and uses visible light and a system of lenses to magnify an image
- Due to the size of the wavelength, only low-magnification views and pictures (~1000x) are possible
- Some light microscopes can be connected to cameras and computers for simple and cost effective defect detection

Optical Microscopy



Quirk, M., Serda, J. *Semiconductor Manufacturing Technology*. Prentice Hall, Upper Saddle River. 2001

Optical Microscopy



The microscopes in our lab are Leitz Ergolux
Optical Microscopes with video and still camera CCD attachments

Quirk, M., Serda, J. *Semiconductor Manufacturing Technlogy*. Prentice Hall, Upper Saddle River. 2001

Optical Microscopy

Specifications	**(CNEU) Leitz Ergolux**
Stand with coarse/fine adjustment	Bright field / dark field vertical illuminator
12v / 100w lamp housing with socket & bulb	Ergonomic tilting trinocular head
Pair of 10x oculars	
Motorized 5-Place nosepiece	5x, 10x, 20x, 50x, 100x BF/DF objectives
6" x 6" X-Y mechanical stage with glass plate	Variable 12v/20w power supply
CCD camera	Photoshop imaging software
Advantages	
Able to view a variety of samples	Ease of use
Bright field/dark field modes	Ability to capture images with camera
Disadvantages	
Limited magnification range (5 – 1000x)	Cannot measure feature depth on patterned substrates
Imaging software requires manual adjustment	

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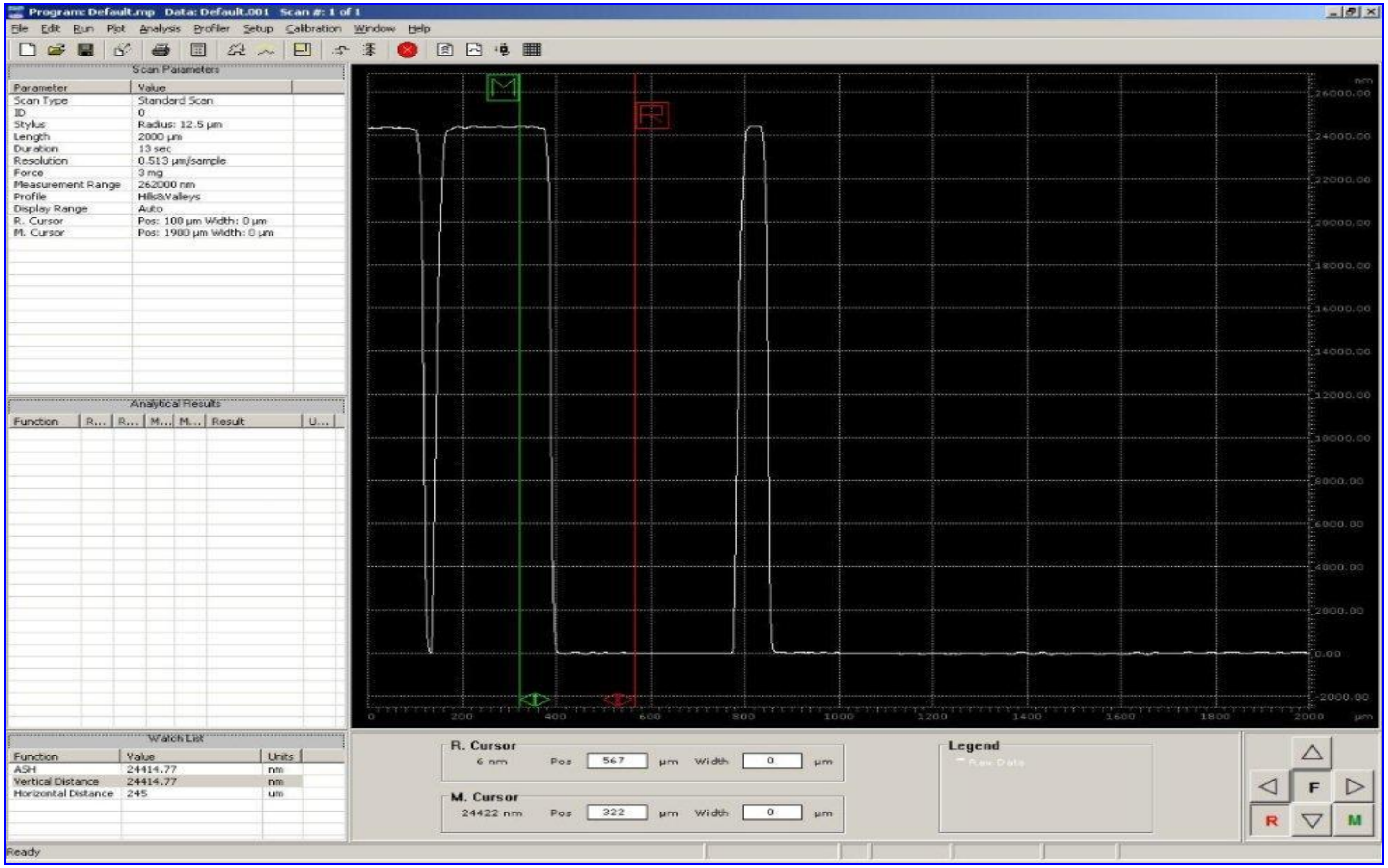
Profilometry

- Uses a sharp probe and physically contacts the surface to determine the topography of the substrate's surface.
- Used to measure:
  - Film thickness
  - Surface topography
  - Step heights

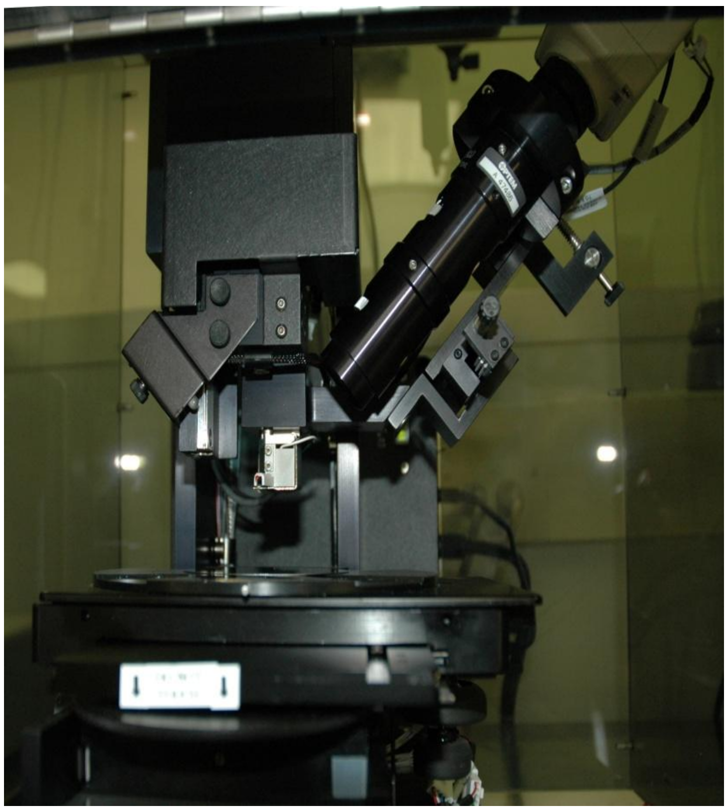
Typical Profilometry Scan



Profilometry

A Tencor 500 Alpha Step Profilometer.

Veeco Dektak 6



Specification	Standard	Option
Vertical Range	50 Å to 2,620 kÅ	1 mm maximum
Vertical Resolution (at various ranges)	1 Å/65 kÅ, 10 Å/655 kÅ, 40 Å/2620 kÅ	160 Å 1 mm
Scan Length Range	50 µm to 30 µm (2 mils to 1.18 in)	
Scan Speed Ranges	3 seconds to 100 seconds	
Software Leveling	Two-point programmable or cursor leveling	
Stage Leveling	Manual	
Stylus (standard)	Diamond, 12.5 µm radius	0.2 µm, 0.7 µm, 2.5 µm, 5 µm
Stylus Tracking Force	Programmable, 1-15 mg	
Maximum Sample Thickness	31.75 mm (1.25")	
Sample Stage Diameter	6" for 150 mm and smaller samples	
Manual Stage Position Translation	X Axis, 20 mm Y Axis, 77mm	
Sample Stage Rotation	Manual Theta, 360°	
Power Requirements Current Phase	120 V, 60 Hz,5A@ 120 (+/-10%)Single Phase	
Camera Field of View	2.6 mm horizontal field of view.	1.1- 4.6 mm zoom
Color Camera	45° side view	

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Ellipsometry

- Ellipsometry is a measurement technique that uses the polarization of laser light to determine film thickness.
- Non-destructive and non-contact technique
- Very useful method of measuring thickness of materials that are transparent

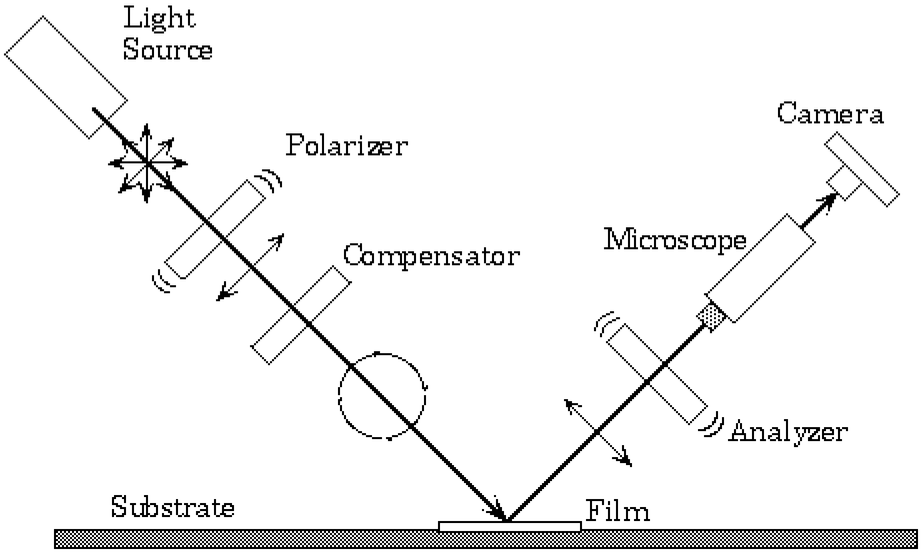
Ellipsometry

- Use a linearly polarized laser light source that when reflected from the sample becomes elliptically polarized
- Polarized light, is light that consists of only light rays traveling in one plane
- The shape of the reflected ellipse is measured and film thickness is determined based on given information: angle of reflection, index of refraction, etc.

Ellipsometry

The ellipsometer in the Nanofab is a variable Angle Spectroscopic Ellipsometer (Gaertner Scientific L116C Ellipsometer).

This refers to the ability of the machine to vary the angle of the incident light, optimizing measurements.



Rensselaer Polytechnic Institute. Joel L. Plawsky
Professor of Chemical Engineering

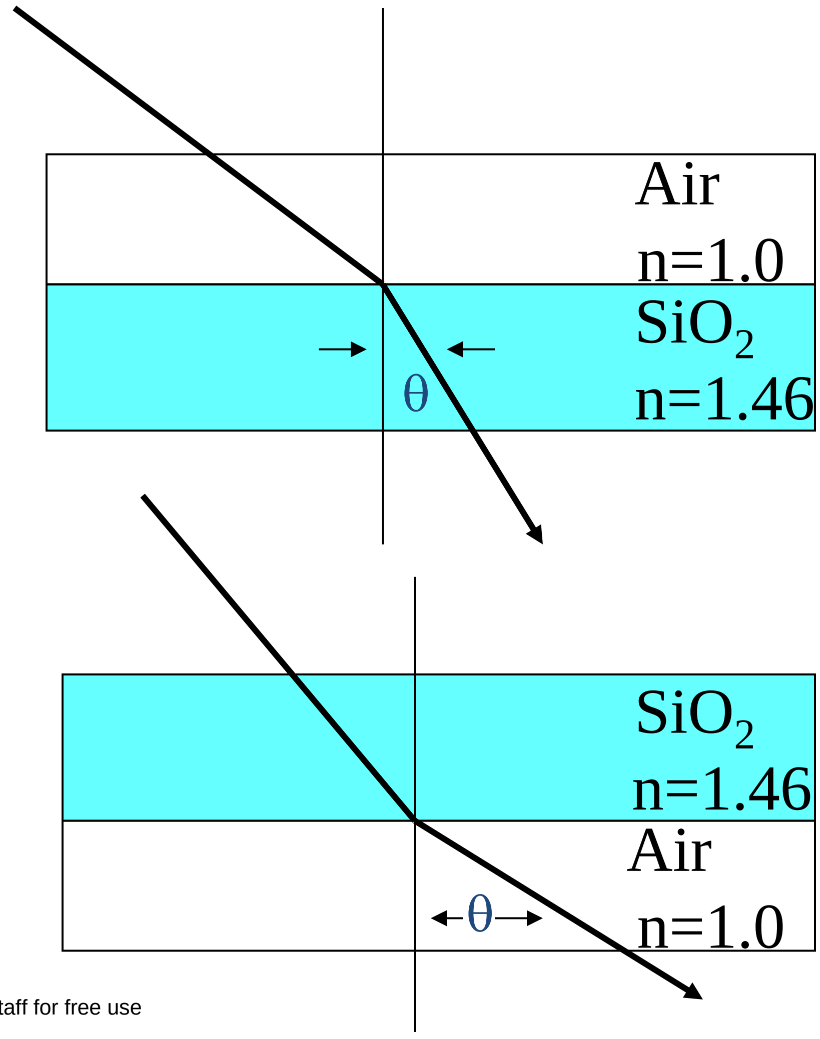
Ellipsometry

Specifications	**Gaertner L115S-8**
Laser Light Source	**632.8 nm Helium-Neon (Red)**
Incidence Angles	**50º and 70º are used mostly**
Beam Diameter	**1.0 x 1.6 mm at 50º and 1.0 x 3.0mm at 70º**
Method of Measurement	**Four detector-voltages are used to determine state-of-polarization of light of reflected beam. The surface parameters Psi and Delta, and hence film thickness and index of refraction, are calculated.**
Film Thickness Range	**0 to 6000 nm**
Accuracy	**± 3 Angstroms**
Repeatability	**± 1 Angstrom**
Refractive Index	**± 0.005**
Scanning Modes	**Operator selectable; 5 point, 9 point, XY grid or contour map**
Scanning Stages	**Rotation with translation stages with built in stepping motors**
Stepping Motor Drive Source	**Two axis, programmable controller; manually or computer controlled**
Scanning Increments	**0.01º steps rotation ; 0.01 mm steps to translation**

Refractive Index

- A property of transparent substance that addresses how much a light beam bends as it travels through the test media
- By measuring two unique angles of incidence, the ellipsometer can determine index of refraction and thickness.
- Changes in the index of refraction can represent changes in stoichiometry

Refractive Index



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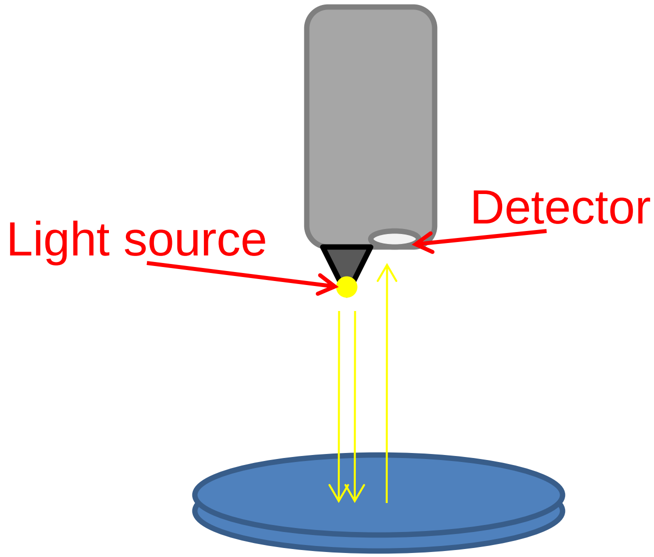
Reflection Spectroscopy

- Reflection spectroscopy is the analysis of light that has been reflected or scattered from a solid, liquid or gaseous medium.
- Commonly used to determine film thickness and index of refraction.
- Non-destructive and non-contact.
- Simple and relative low cost

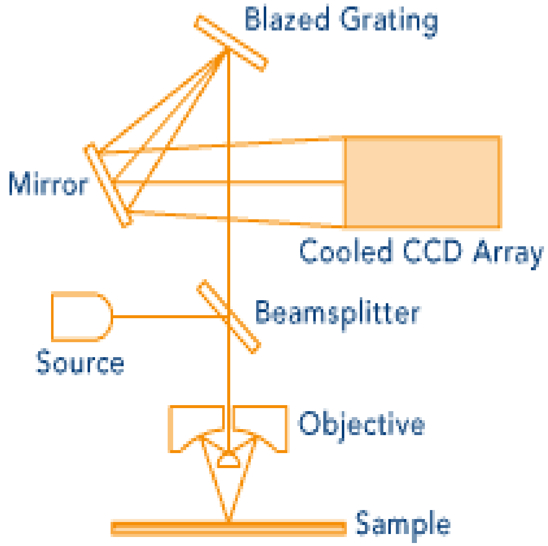
Reflection Spectroscopy



- A broadband light source is reflected off the sample at normal incidence.
- The intensity of the reflected light is measured over the range of wavelengths.
- Computer software utilizes the property of dispersion (the wavelength dependence of a medium's index of refraction) to determine the film thickness

Reflection Spectroscopy

The reflection spectroscopy equipment in the Lab is a Nanometrics Nanospec AFT model 010-180.



Nanometrics Inc

Reflection Spectroscopy

Advantages and Disadvantages:

- Able to analyze multilayer films.
- Can measure organic films like photoresist.
- Usually requires a reference sample of known composition and thickness

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Contact Angle

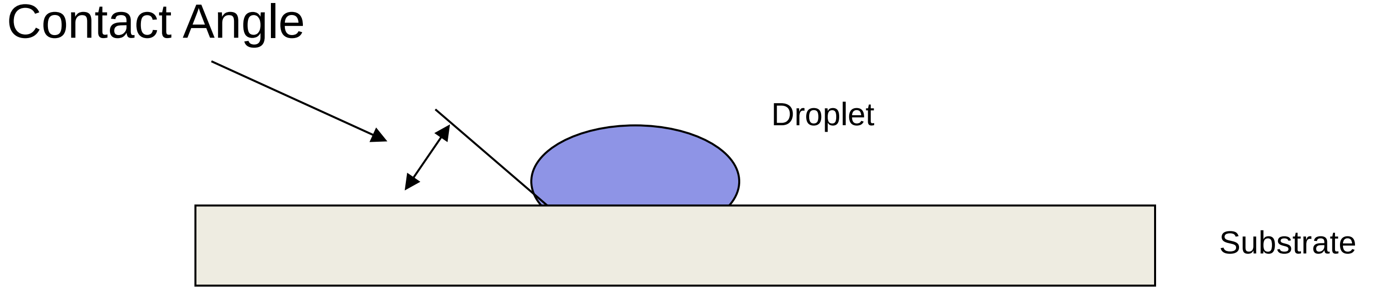
- Measures the adhesion of liquids to a surface can be used to calculate surface energies or adhesion tension
- Characterizes surface parameters:
  - wettability (hydrophobicity), cleanliness, finish, and adhesion
- These parameters can be used predict things such as adhesion, corrosion, or biocompatibility

Contact Angle

- Instruments that analyze contact angles are either referred to as:
  - Tensiometer
  - Contact Angle Analyzer
  - Geniometer

Contact Angle

- The tangent angle formed between a liquid drop and its supporting surface is relative to the forces at the liquid/solid or liquid/liquid interface.
- This angle is representative of surface bonding energy.



Contact Angle Tools

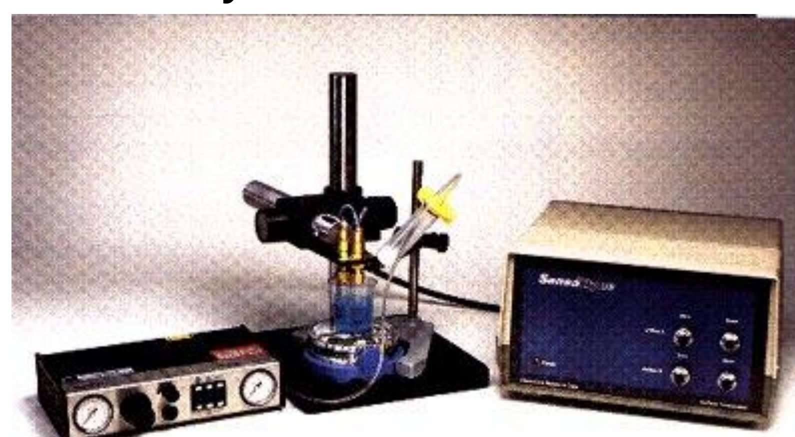
Kruss DSA 10 Contact Angle Analyzer

http://www.kruss.de/en/products/contact-angle/easydrop.html

Cahn Radian DCA Analyzer

http://www.qtech.com.pk/thermo.html

SensaDyne Bubble Tensiometer



http://www.sensadyne.com/sts.html

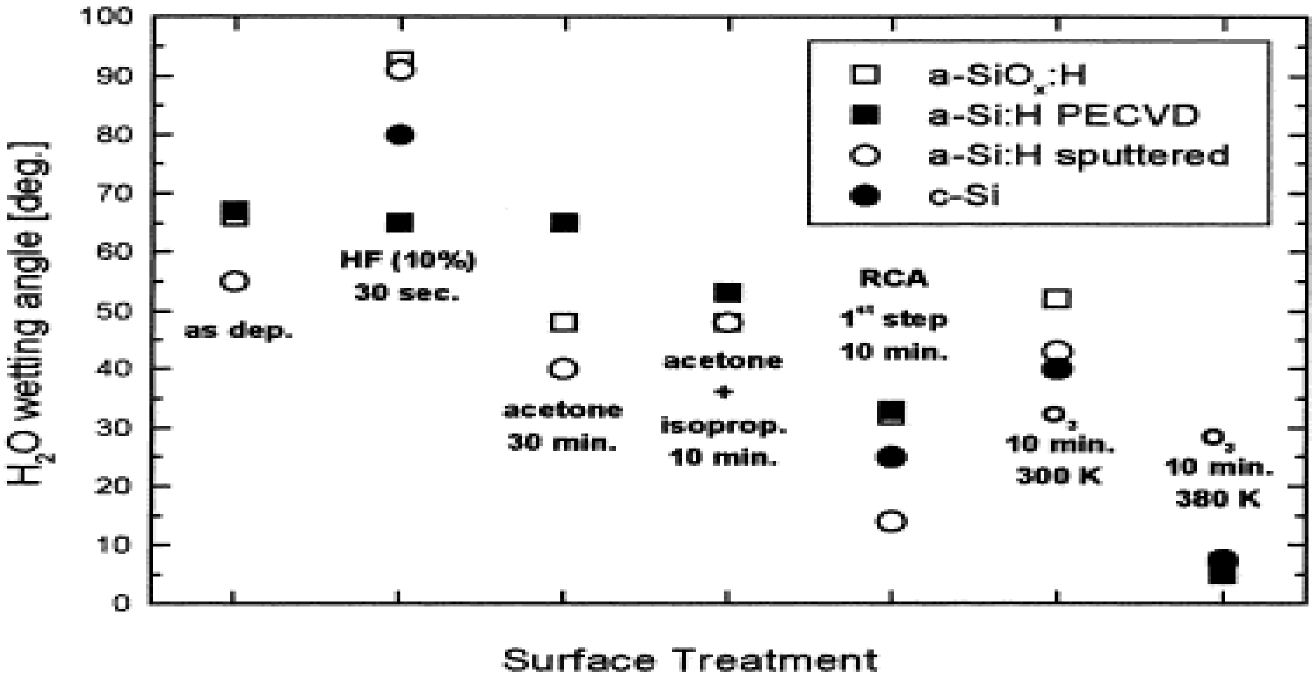
Contact Angle

Case Study: Biological Applications

C. Dahmen, et al. studied *Surface functionalization of amorphous silicon and silicon suboxides for biological applications.* There findings were published in Thin Solid Films Vol 427, Issues 1-2 , 3 March 2003, Pages 201-207

Contact Angle

Wetting angle for four different silicon and suboxide samples as a function of surface treatment with HF.



The longer these four samples were treated with HF the more the surface topography changed and the smaller the contact angle became.

C. Dahmen, et al. *Surface functionalization of amorphous silicon and silicon suboxides for biological applications. Thin Solid Films Vol 427, Issues 1-2 , 3 March 2003, Pages 201-207*

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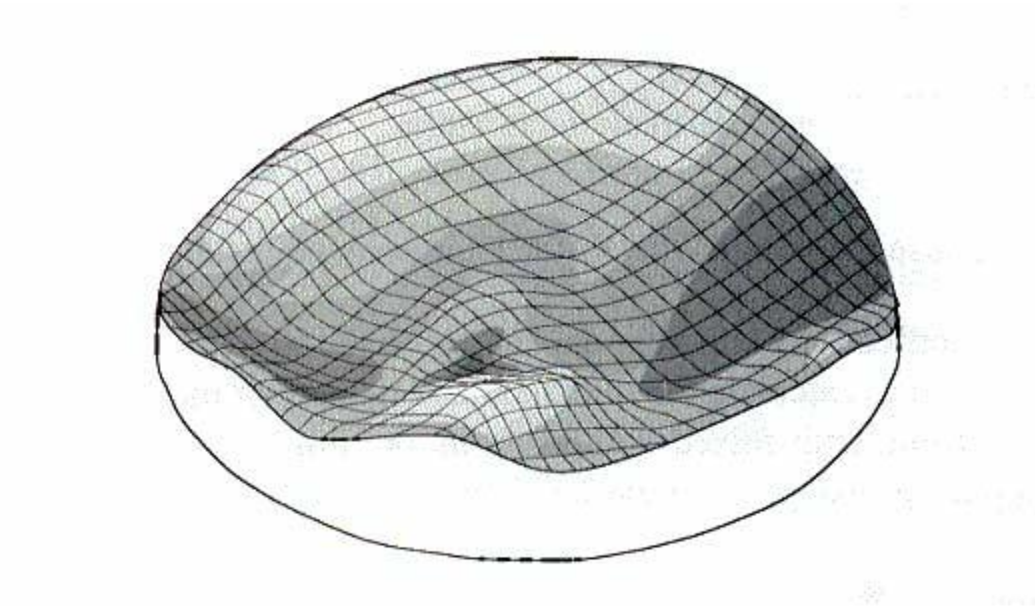
Film Stress

- Stress in a layer that may deform the substrate and create uniformity, reliability, or reproducibility concerns
- Mechanical stress from the deposited film is transmitted to the underlying substrate and to other layers
- Film stress is an important concern of top down manufacturing

Film Stress

- Tools based on a scanning laser-beam or a split-beam laser are commonly used
- Changes in the radius of curvature of the substrate resulting from a film's deposition can be measured and analyzed
- Applies to all types of standard thin films
- Metals, dielectrics, semiconductors, and polymers

Film Stress



Film Stress Map

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Sheet Resistance

- Resistance: a measure of how much a sample opposes the flow of electrical current
- Resistivity: a material property that factors out the dimensions of the sample
- Sheet resistance: a measure of resistance of uniform thin films

Sheet Resistance

- Sheet resistivity can be determined by running a current between two points on a square of the layer to be measured
- Can be interpreted as the end-to-end resistance of a square sample of a thin film on a substrate
- It depends on film resistivity and thickness, but is independent of the size of the square being measured

Sheet Resistance

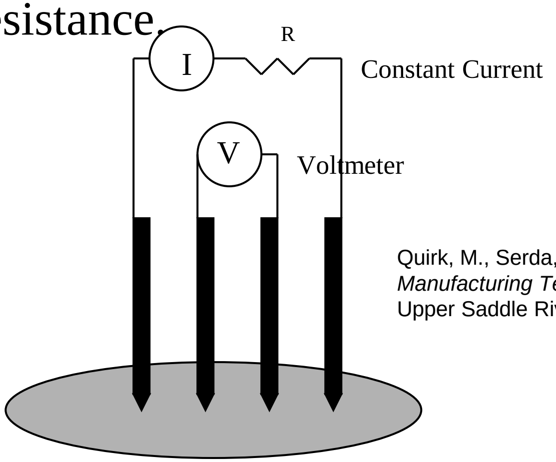
To determine resistivity and therefore resistance, the following equation can be used:

$$\rho_s = \frac{V}{I} \times 2\pi s$$

ρ - Sheet resistivity
V – DC voltage across probes
I – DC current through probes
s – spacing between the probes

This information can be determined using the 4-point probe. It is a tool that is makes contact with the substrate. This contact is required to determine the resistivity, but is destructive to the surface.

The 4-point probe has four equally spaced probes that are in a single line. A known current (I) is passed between the probes and the potential difference (V) between the two inner probes is measured. This method avoids contact resistance.



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