



Spectroscopic Ellipsometry for Thin Film Characterization

Theory & Fundamentals

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PRESENTATION OVERVIEW

- Ellipsometry technique : capabilities and advantages
- Ellipsometry definition
- Light and polarization
- Optical constants
- Reflection with films
- EMA theory
- Analysis of ellipsometric data
- Phase Modulation Ellipsometer: How does it work ?
- Basic ellipsometer operations: choice of spectral range, angle of incidence

HORIBA JOBIN YVON A Global Company

- **Creation of Jobin Yvon in 1819**

- **Leader company in spectroscopy and analytical instrumentation**

- Headquarters: France (20 km from Paris)
- 300 employees - 3 manufacturing plants in France
- 5 divisions: Molecular & Micro analysis (Raman, fluorescence), elemental analysis, optical components (gratings, spectrometers, monochromators), particle size, emerging business (ellipsometry, process control, forensics)

- **1997: Jobin Yvon merged into HORIBA Group**

- More than 4400 employees
- Annual sales: \$935,000,000
- 5 segments: ATS, Semiconductor, P&E, Medical, Scientific

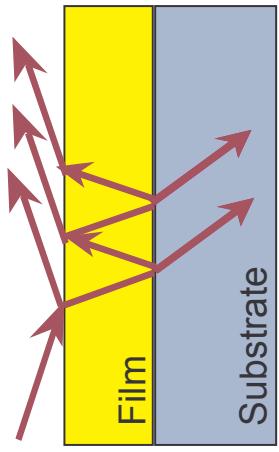
- **2004: Jobin Yvon becomes HORIBA Jobin Yvon**

General Overview of the Thin Film Division

- **Development and manufacture of optical metrology systems for**

⇒ Characterization of thin films, surfaces and interfaces

- Laser and Spectroscopic Ellipsometers from FUV to NIR
- Combined Ellipsometry and Reflectometry

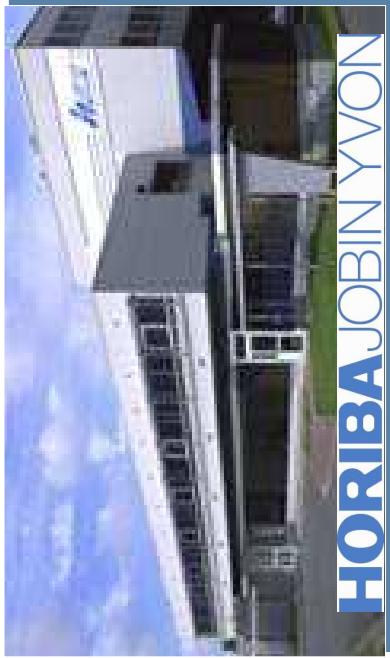


⇒ In-situ Process Control of thin film etching and deposition

- Laser and Polarized Interferometry
- Optical Emission Spectroscopy

- **Welcome to**
- **Thin Film Division**

- 55 persons (sales, manufacturing, R&D)
- Headquarters: France (20 km from Paris)
- 4 application laboratories worldwide (USA, France, Germany, Japan)



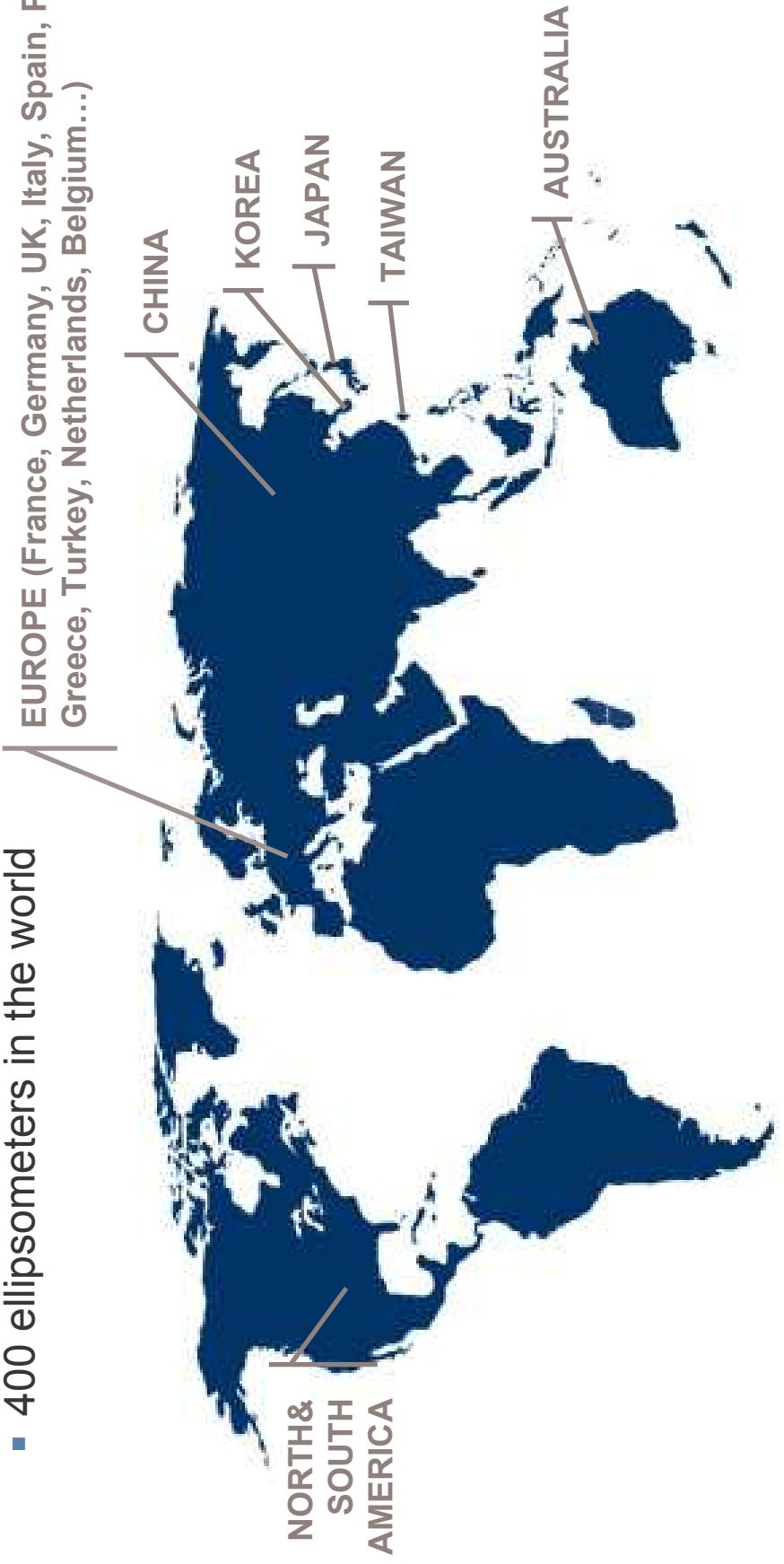
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User Profile of HORIBA Jobin Yvon Ellipsometer

- Universities – R&D: 65 %
- Industry: 35 %
- 400 ellipsometers in the world
- EUROPE (France, Germany, UK, Italy, Spain, Portugal
Greece, Turkey, Netherlands, Belgium...)



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ELLIIPSOMETRY CAPABILITIES

Characterization of thin films, surface and interface

■ Thickness

- Accurate thin film measurement from a few angstroms to several tens of microns
- Characterization of single layer and complex multilayer stacks
- Characterization of surface and interface

■ Optical properties

- Refractive index (n) and extinction coefficient (k) from the far-UV to near-IR for complex materials, graded and anisotropic layers

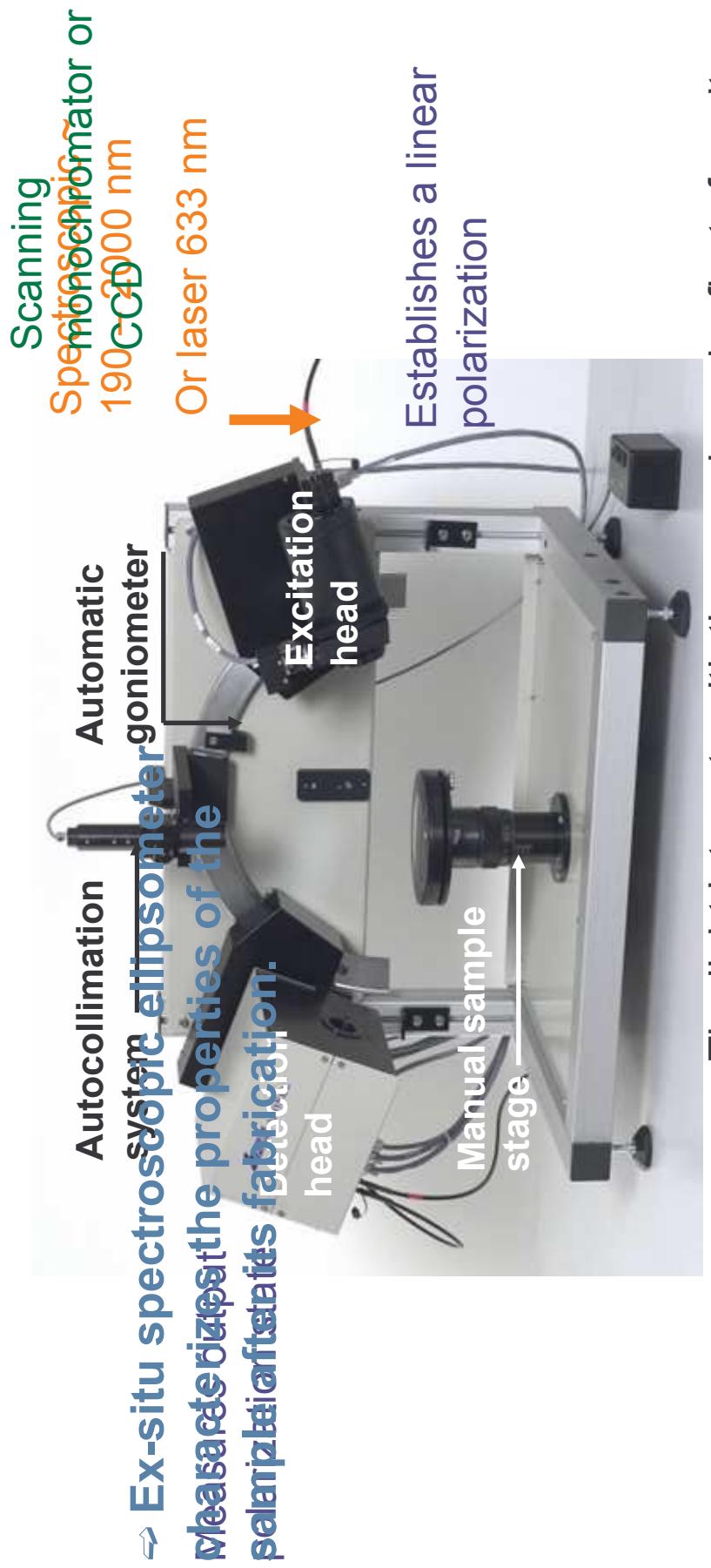
■ Material properties

- Alloy composition
- Microstructure and crystallinity
- Optical bandgap (Eg)
- Film uniformity by area and depth

Advantages of the Spectroscopic Ellipsometry Technique

- Non-destructive & contactless technique
- Highly accurate & reproducible
- Can be used in any transparent and semi-transparent medium
- Very sensitive, especially to ultra-thin films (< 10 nm)
- Spectroscopic measurement: wide spectral range from FUV to NIR
- Simultaneous multiple parameter determination
- Mapping – 2D and 3D cartography
- Pattern measurements

Ellipsometer Optical Set up



The light interacts with the sample and reflects from it. This interaction causes a polarization change of the light.

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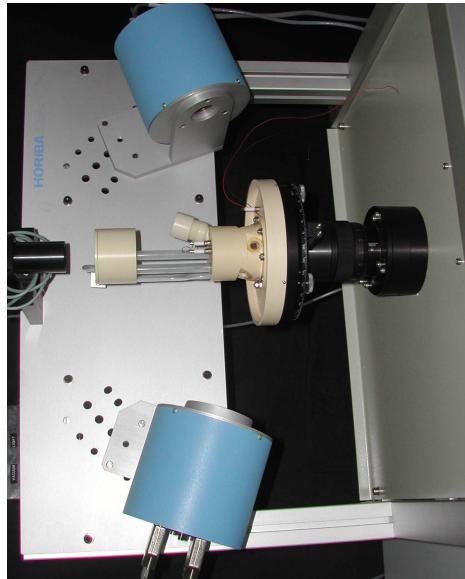
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In Situ Ellipsometry

Real-time monitoring of thin film deposition with submonolayer resolution

- May be mounted on any process chamber with entrance & exit window
- Real-time calculation of thickness & refractive index of deposited/etched layers
- Accessories: liquid cell, electrochemical cell, thermostated sample stage
- Kinetic measurement of surface modifications during the process



⇒ **In-situ ellipsometry provides the advantage to probe the fabricated structures, to investigate the surface modifications during the process.**

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Ellipsometry Definition

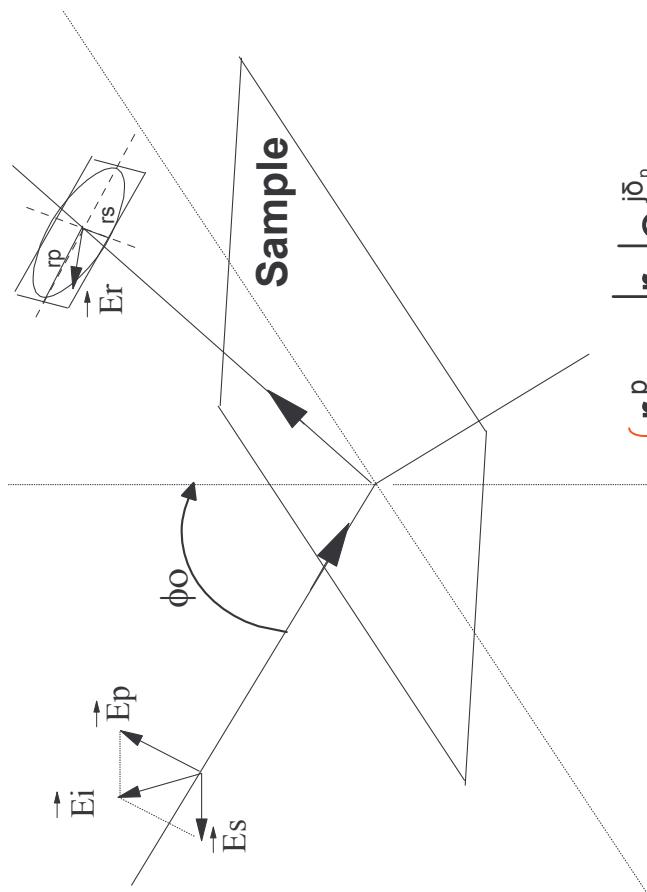
- Ellipsometry is an **optical technique** that measures the change in the polarization state of light reflected from the surface of a sample.

$$\frac{r_p}{r_s} = \rho = \tan\psi \exp(i\Delta)$$

> Ψ and Δ : Ellipsometric angles
Measured data

$$> \tan\psi = \left| \frac{\mathbf{r}_p}{\mathbf{r}_s} \right| : \text{Amplitude ratio}$$

> $\Delta = \delta_p - \delta_s$: Phase shift introduced by reflection from sample



$$\left. \begin{array}{l} \mathbf{r}_{01}^p = |\mathbf{r}_p| e^{j\delta_p} \\ \mathbf{r}_{01}^s = |\mathbf{r}_s| e^{j\delta_s} \end{array} \right\}$$

Fresnel
reflection
coefficients

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Ellipsometry Measurements

Advantages

- **Measure ratio of two parameters**

- Repeatable & accurate
- No reference necessary
- Reduced problems of source fluctuation

↳ Ellipsometry vs
Reflectivity

- **Measure a phase Δ**

- Very sensitive to film thickness (<10 nm)

- **Spectroscopic Ellipsometry**

- Allows multiple film parameter characterization
- Eliminates period problem for thick films
- Measures data at wavelength of interest

↳ Spectroscopic vs
Laser Ellipsometry

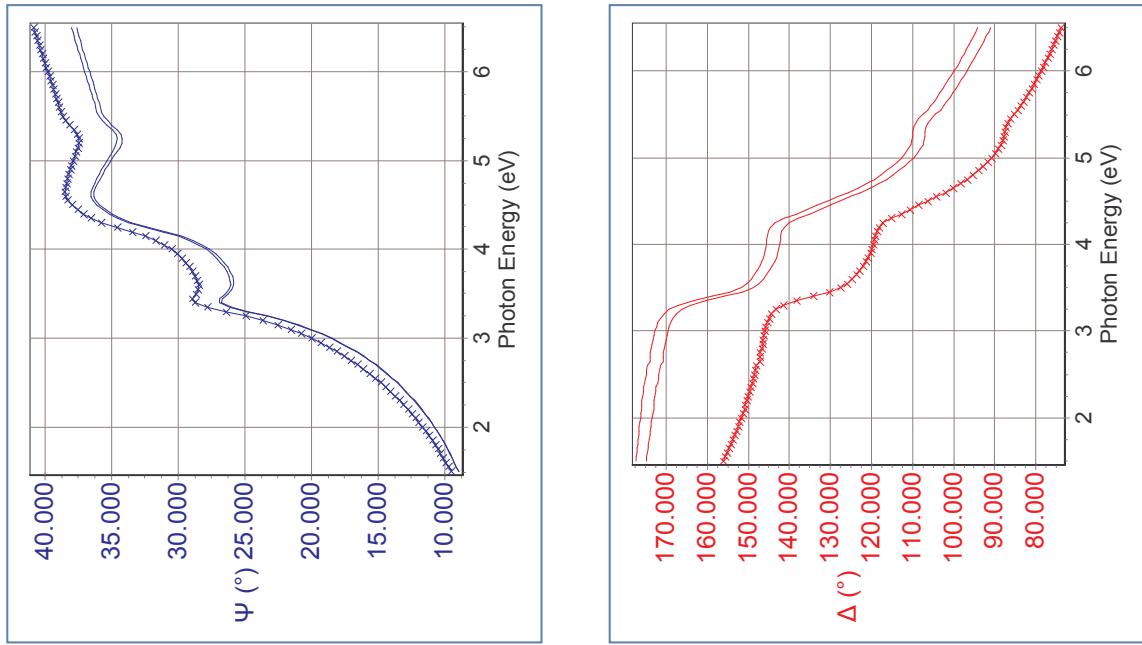
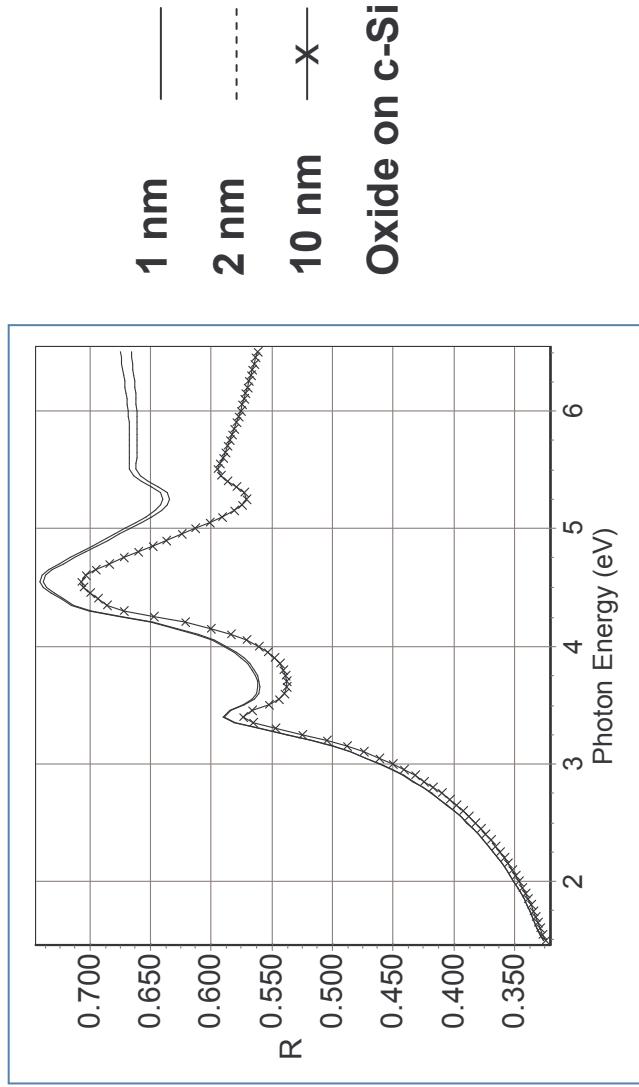
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Ellipsometry vs Reflectivity

⇒ Phase (Δ parameter) information measured by ellipsometer gives much higher sensitivity to ultra-thin films



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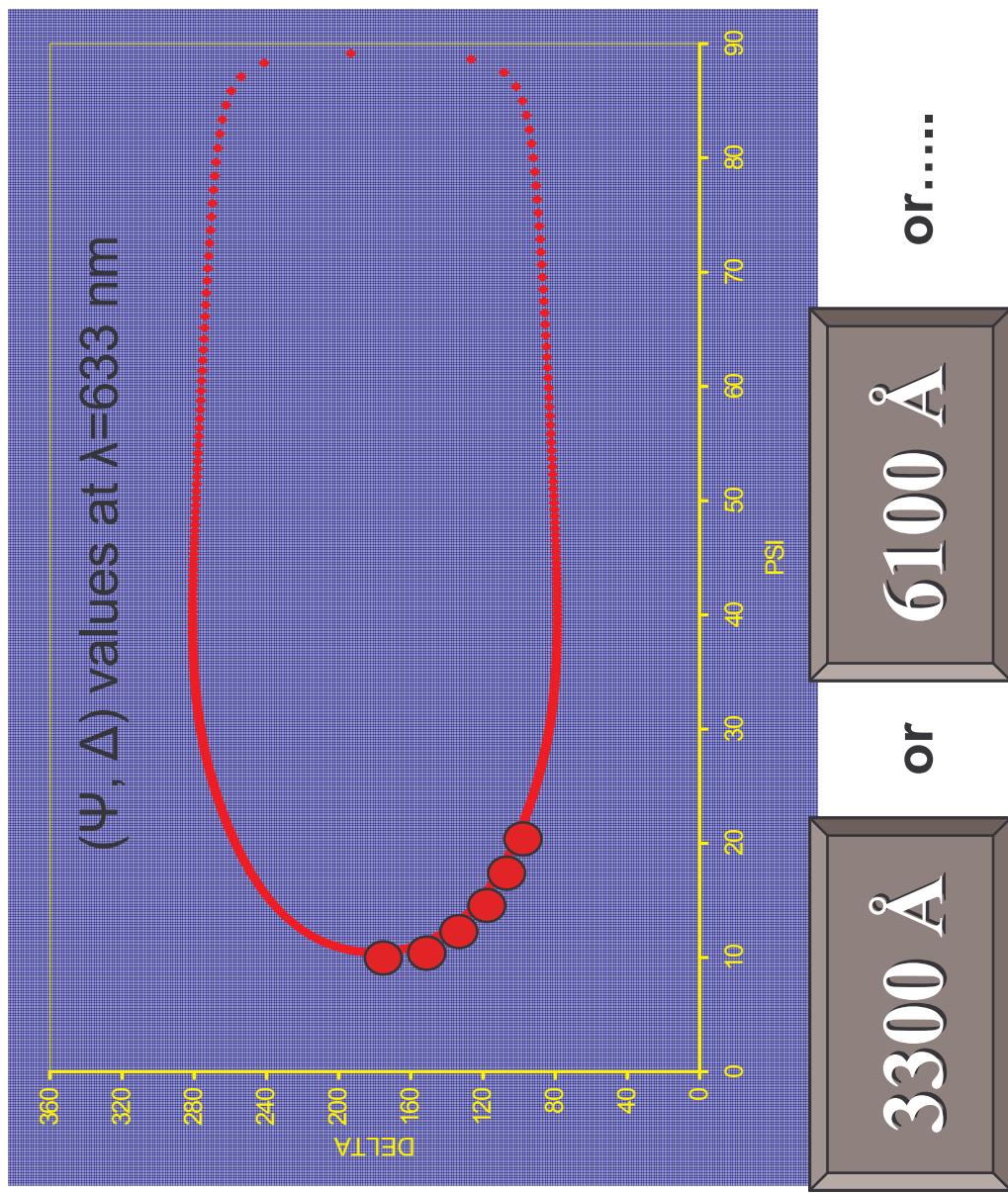
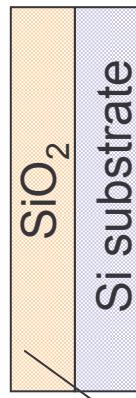
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Spectroscopic Ellipsometry vs Laser Ellipsometry

⇒ Data cycle as film thickness increases

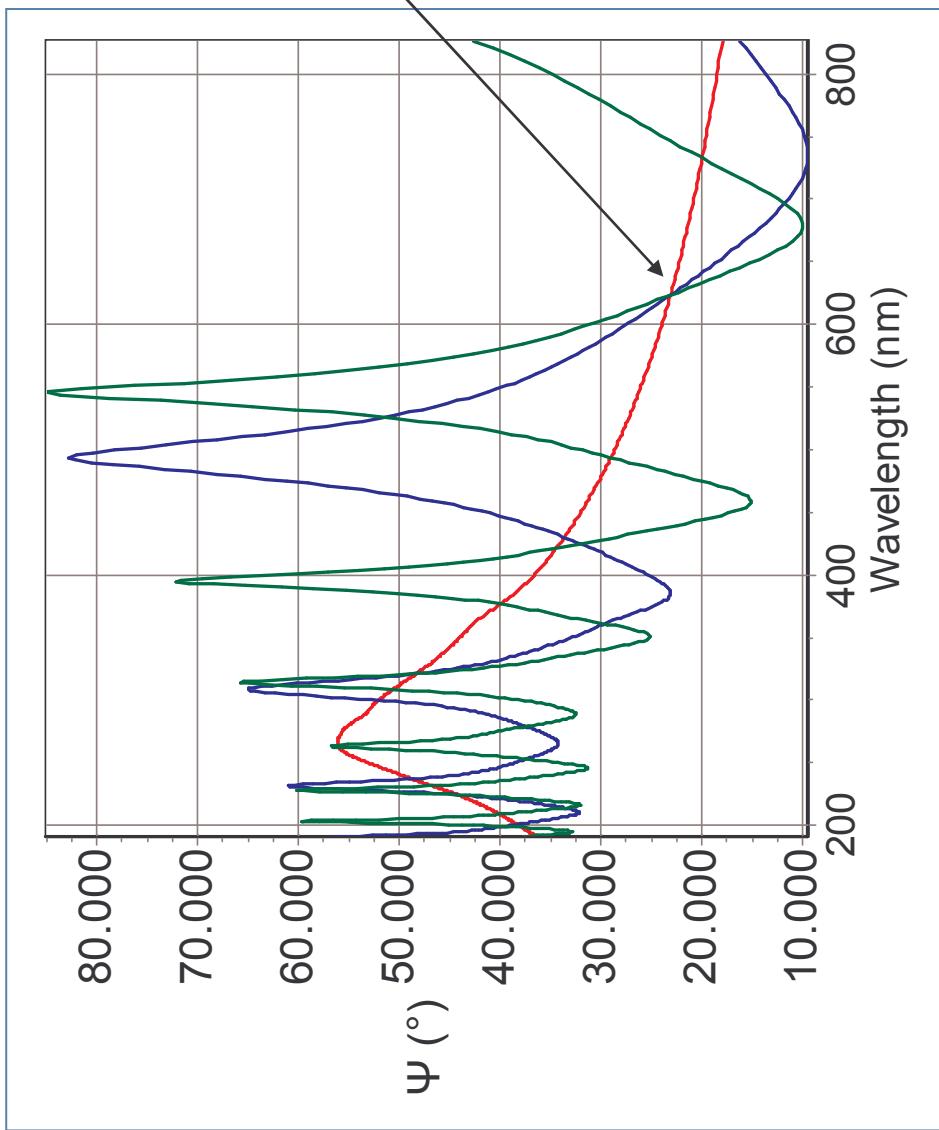
$$D_\phi = \frac{\lambda}{2\sqrt{\tilde{n}_1^2 - \tilde{n}_0^2 \sin^2 \phi}}$$



Spectroscopic Ellipsometry vs Laser Ellipsometry

- **Vary wavelength**
 - Probe different (n, k)
 - Thickness constant

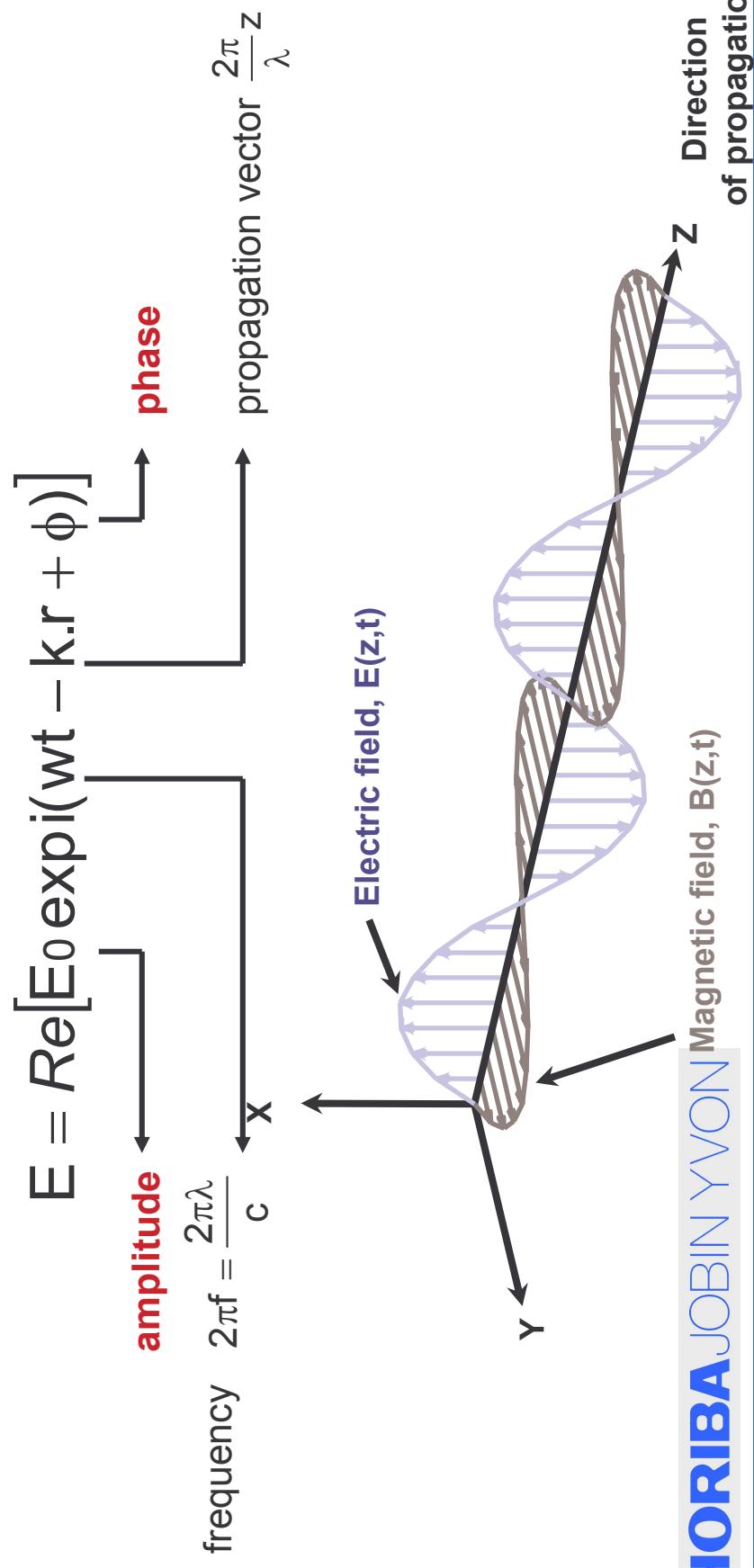
**Data match at
633 nm**



Interaction of Light with Matter

■ Description of light as an electromagnetic plane wave

- Derives from Maxwell's equations
- Consists of an electric and magnetic field vector oscillating perpendicularly to the direction of propagation



HORIBA JOBIN YVON Magnetic field, $B(z,t)$

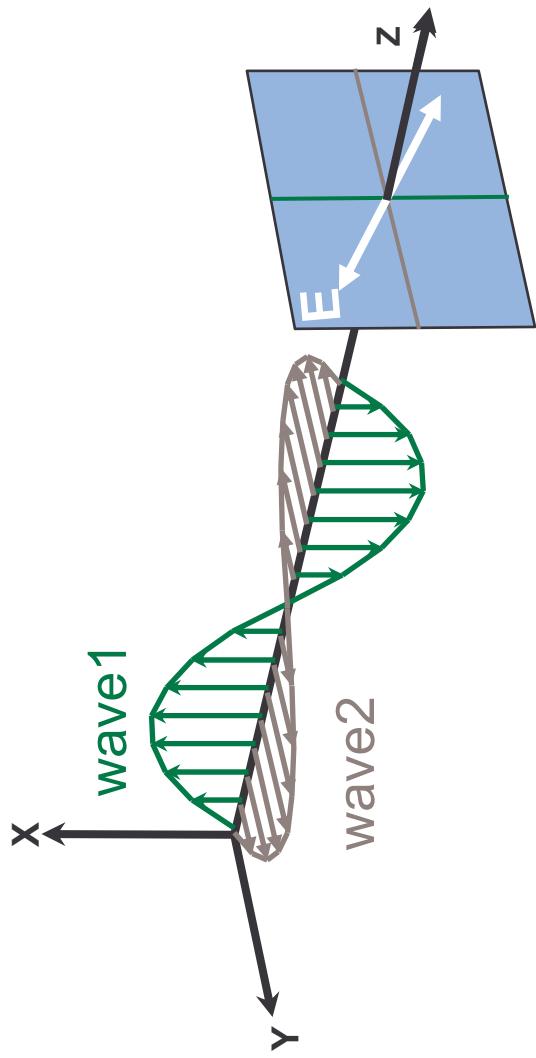
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Light Polarization

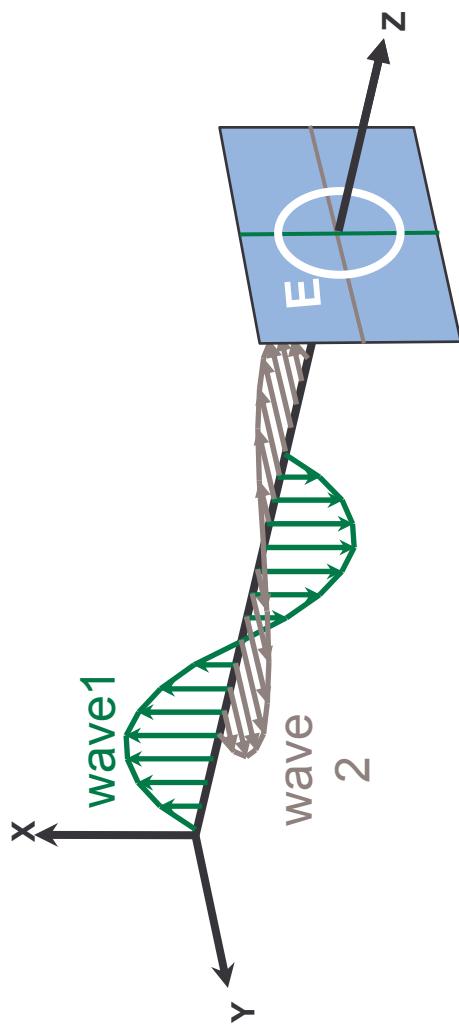
- **Polarization State**
 - Defined by 2 parameters: phase & amplitude of the electric field
 - Describes the direction of the electric field

- **3 Polarization States**
 - Linear polarization
 - > Orthogonal E_x & E_y propagating in same direction
 - > Waves are in phase with each others



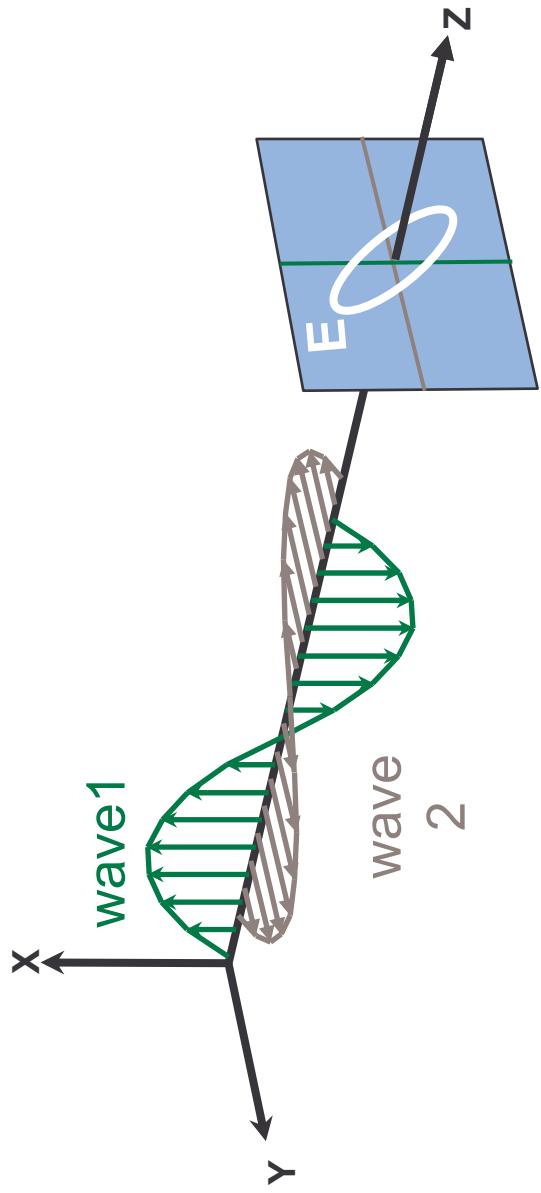
Light Polarization

- **3 Polarization States**
 - Circular polarization
 - > Orthogonal E_x & E_y are 90° out of phase
 - > Equal amplitude



Light Polarization

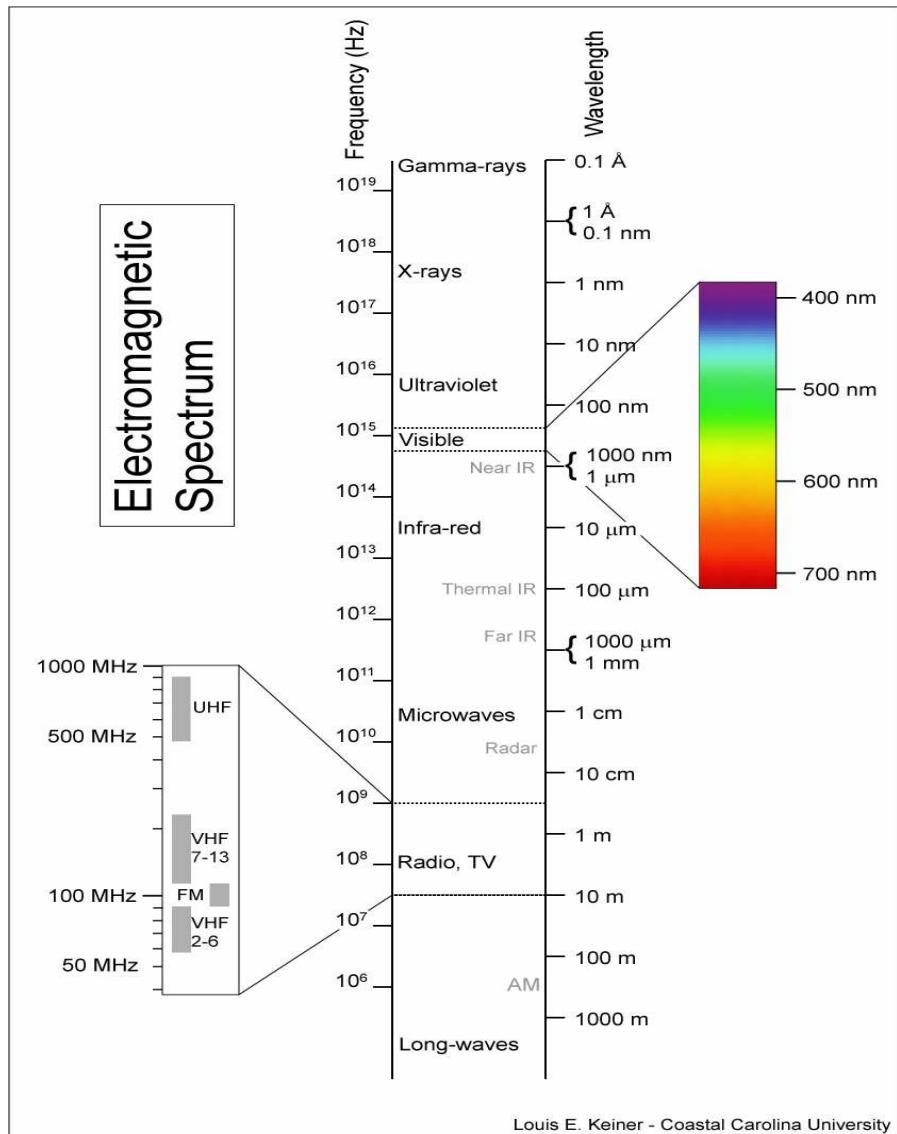
- **3 Polarization States**
 - Elliptical polarization
 - > Orthogonal E_x and E_y have different phase and amplitude



Electromagnetic Spectrum

■ Wavelengths units

- Wavelength (λ)
- Photon energy (eV)
- Wavenumber (K)



■ Relationships

$$E(\text{eV}) = h\nu = \frac{1240}{\lambda(\text{nm})}$$

$$E(\text{nm}) = E(\text{microns}) \times 1000$$

$$\lambda(\mu\text{m}) = \frac{10000}{K(\text{cm}^{-1})}$$

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What are optical constants ?

- 2 parameters used to describe the interaction of light with matter

- Complex Index of refraction :

$$\left. \begin{aligned} \tilde{N} &= n - ik \\ \tilde{\epsilon} &= \epsilon_1 - i\epsilon_2 \end{aligned} \right\} \quad \tilde{\epsilon} = \tilde{N}^2$$

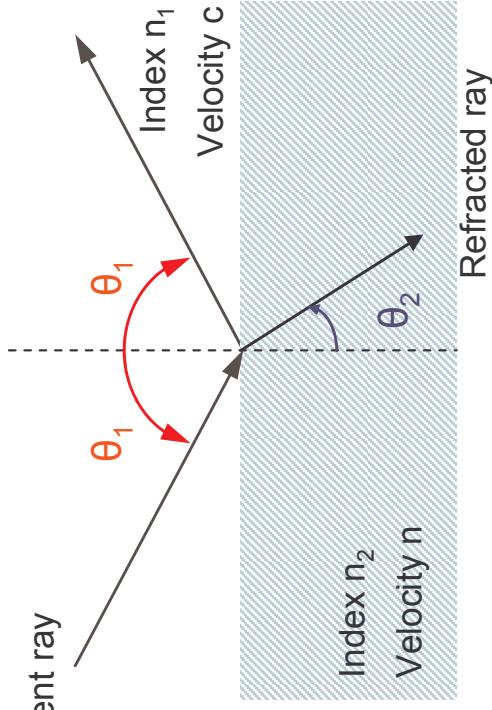
- Complex dielectric constant :

- Ratio of the velocity of light in vacuum (c) to its velocity (v) in the medium

- **Refractive index** (or **index of refraction**) : $n = c/v$

- Amount of absorption loss when light propagates through the material

- **Extinction coefficient** : k

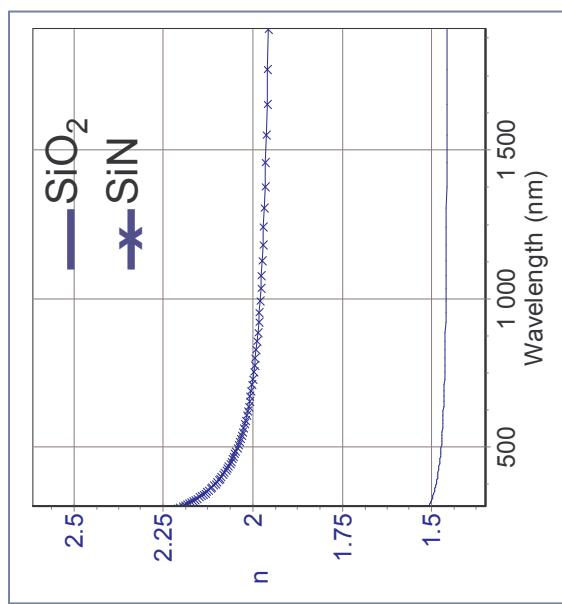


■ Absorption coefficient $\alpha = \frac{4\pi k}{\lambda}$

Optical Constants

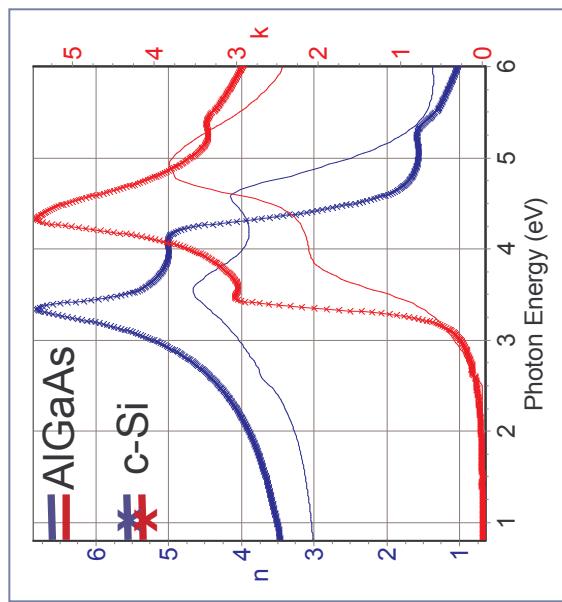
- Depend on properties of the material
- Depend on wavelength range measured

Dielectric or insulator



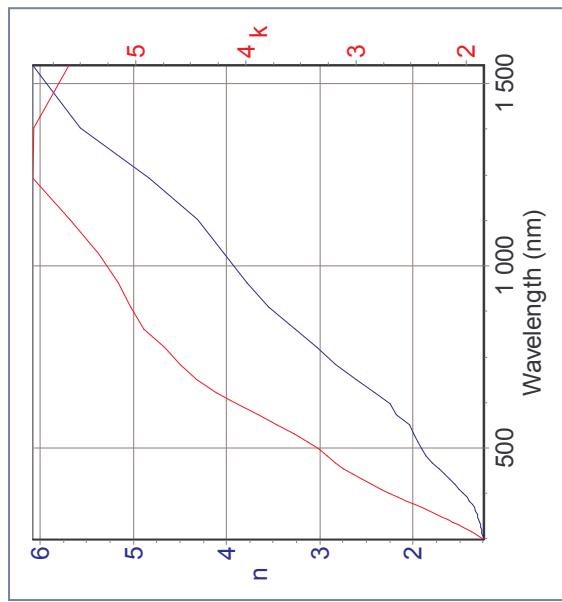
Transparent in the NIR-VIS

Semiconductor



Opaque in the VIS

Metal



Highly absorbing in the NIR

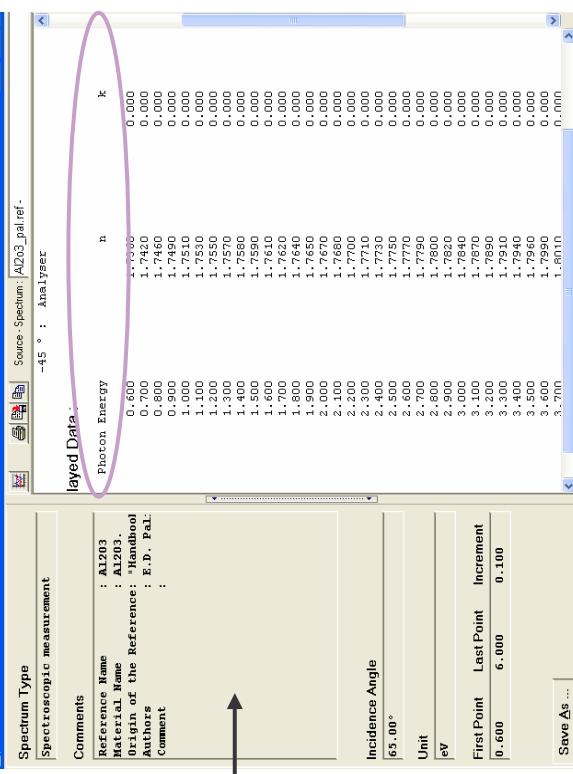
Optical Constants

- Representing the (n,k) of the material in the ellipsometric model

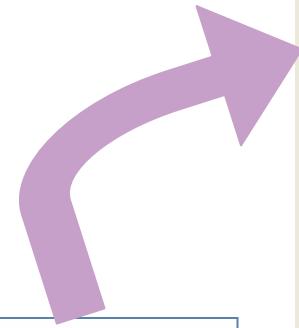
⇒ Tabulated references

- No variation, fixed (n,k) values, often used as a starting values

Library of >100 materials



List of (n,k) values with
wavelengths



| | | |
|---|---|---------------|
| 1 | F | 20.0 |
| S | | Al2o3.pal.ref |

| | | |
|---|--|------------|
| X | | Al.asp.ref |
| S | | Al.asp.ref |

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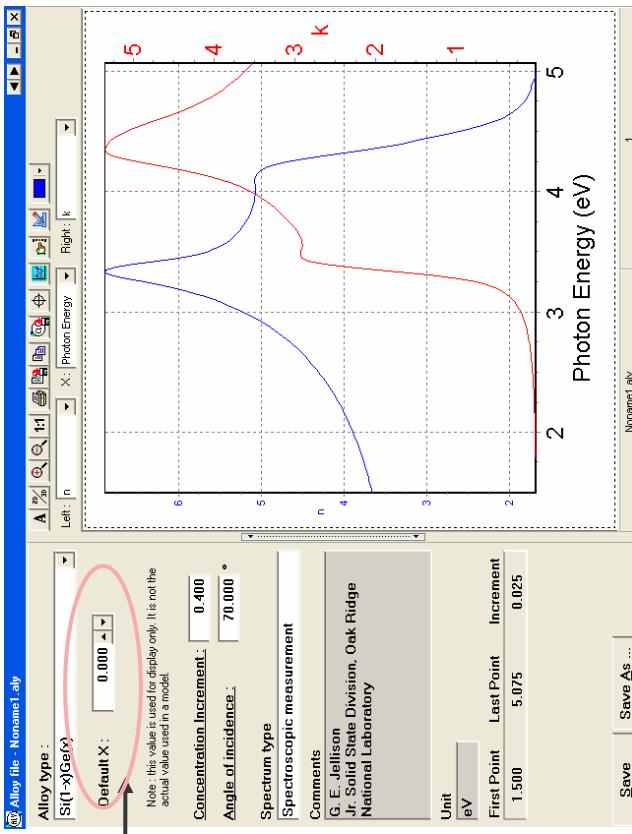
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Optical Constants

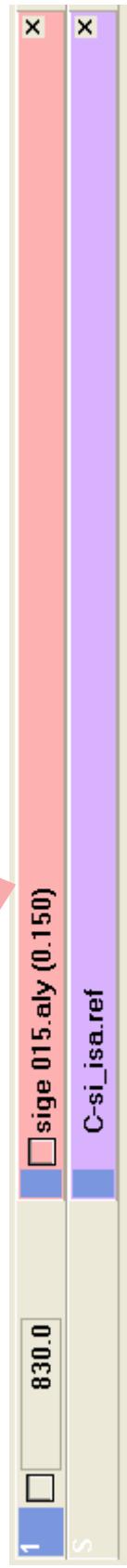
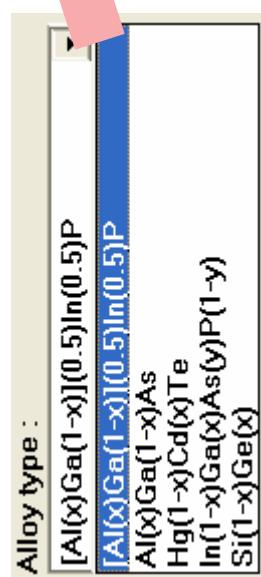
- Representing the (n,k) of the material in the ellipsometric model
- ⇒ Compound alloy semiconductor references

– Known variation

Adjust the composition
and generate the (n,k)



Library of 5 semiconductor alloys



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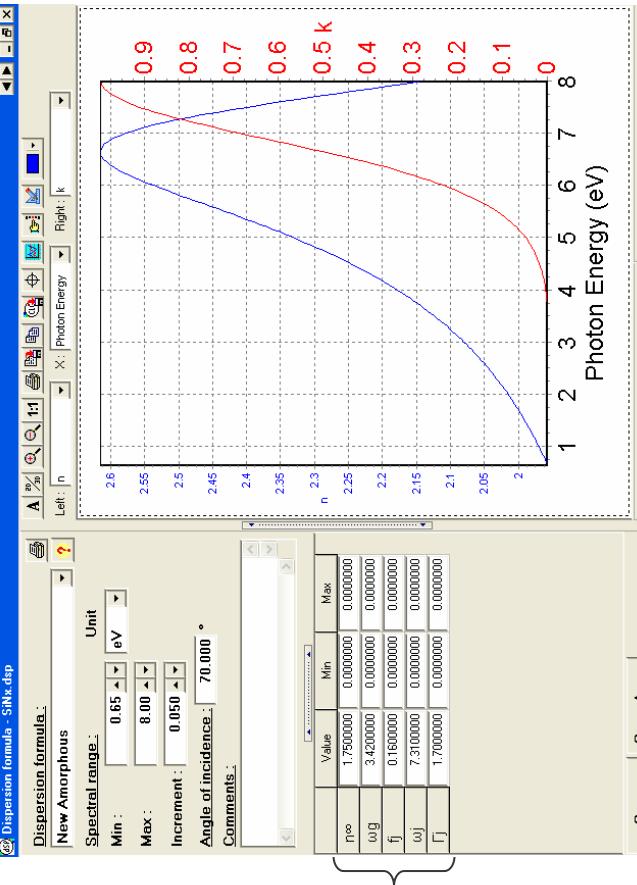
Optical Constants

- Representing the (n,k) of the material in the ellipsometric model

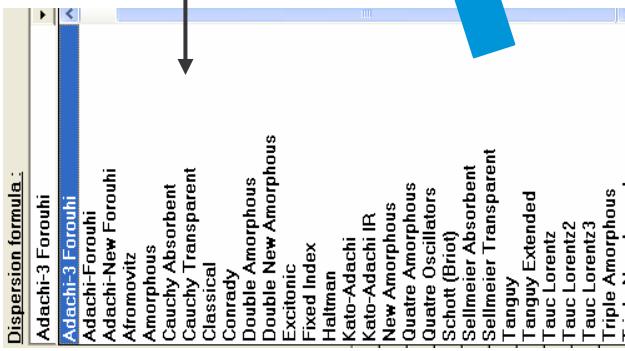
⇒ Dispersion relationships

- Build « by yourself » the (n,k) of your material

Library of > 30 dispersion formula



Setting values of parameters describing the dispersion formula



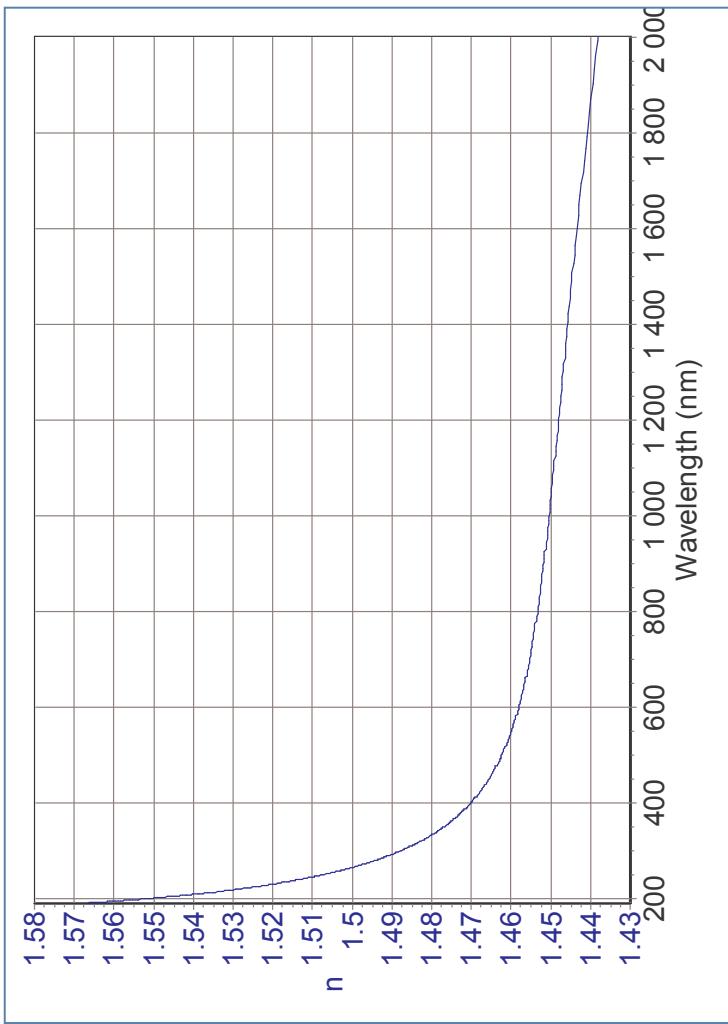
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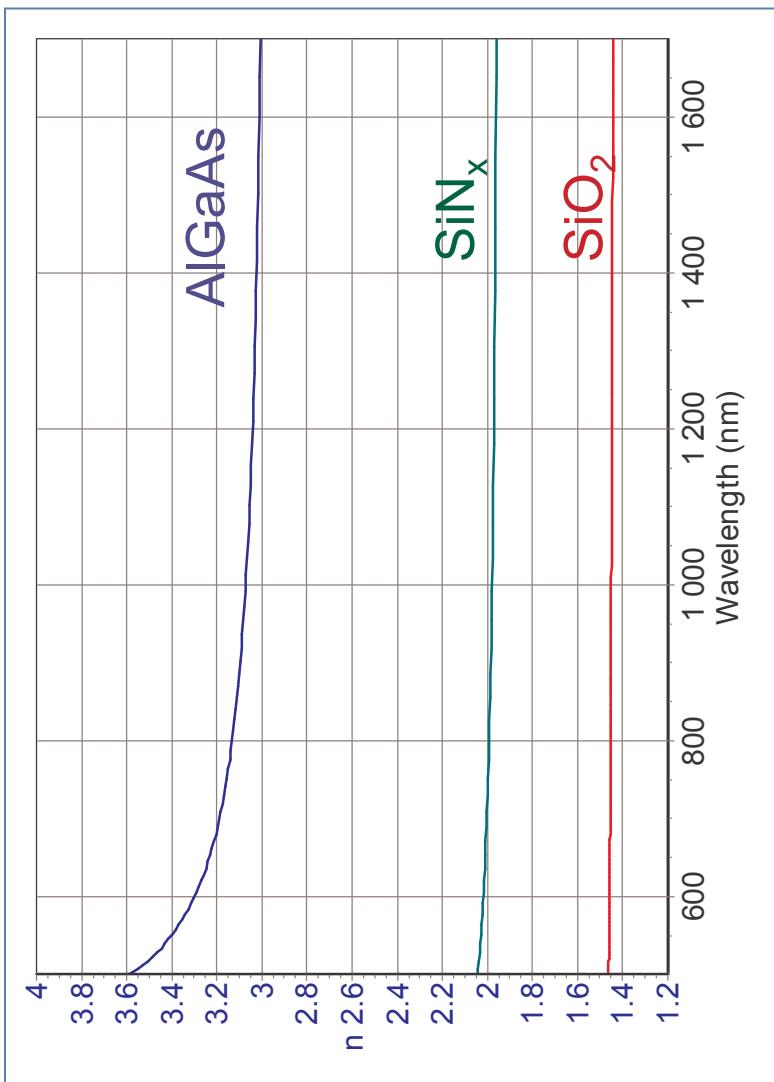
Material Dispersion in Optics

- Materials dispersion comes from a wavelength dependant response of a material to waves.
⇒ Optical constants vary with wavelength, it is called a dispersion.



Normal Dispersion: Transparent Material

- Refractive index decreases with increasing wavelength λ
- No absorption ($k=0$): transparent material
- Higher index for stronger UV absorption



⇒ Normal dispersion

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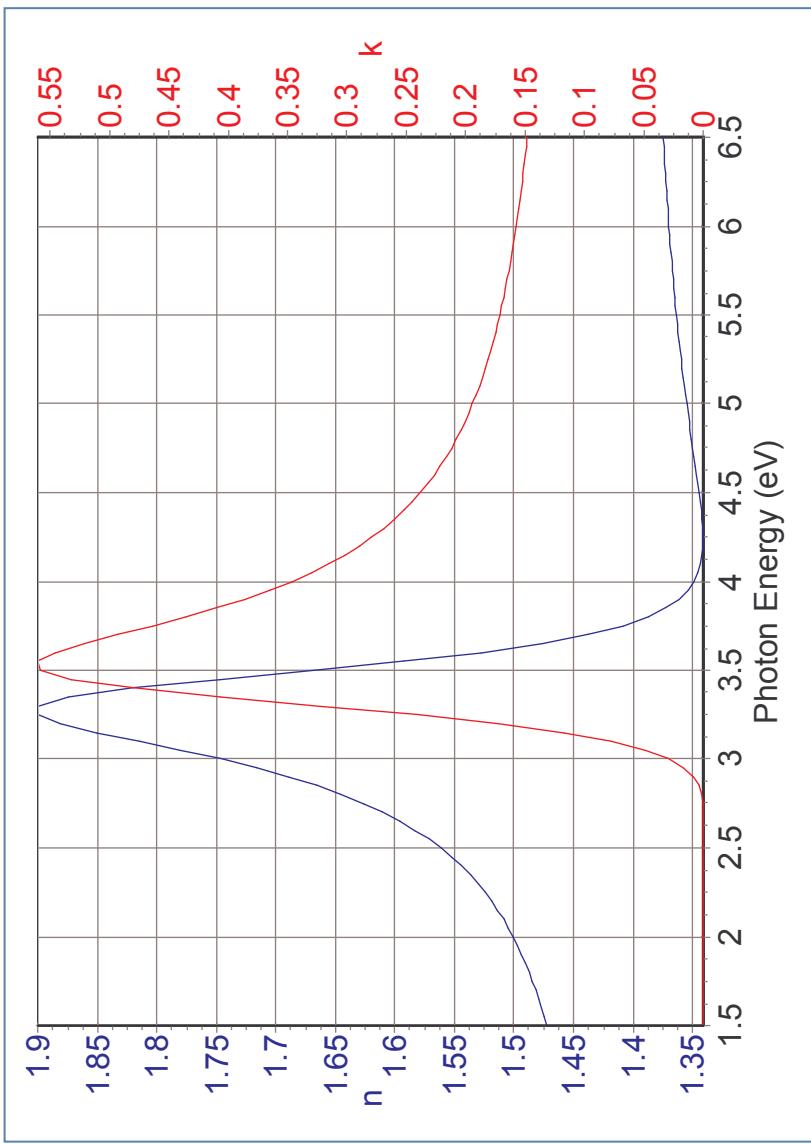
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Anomalous Dispersion: Absorbing Material

- Refractive index increases with increasing wavelength λ
- Absorption ($k \neq 0$): absorbing material
- Higher index for stronger UV absorption

⇒ Anomalous dispersion



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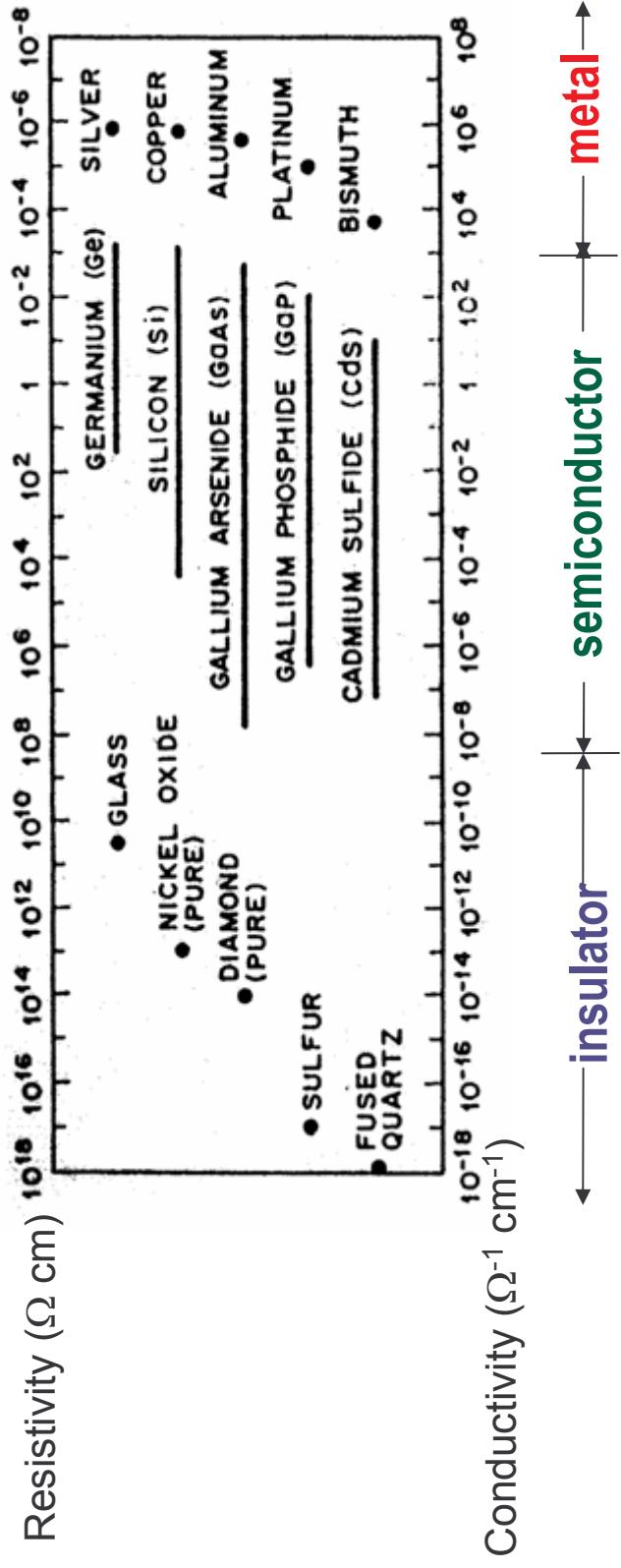
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Classes of Materials

■ Classified versus electrical conductivity

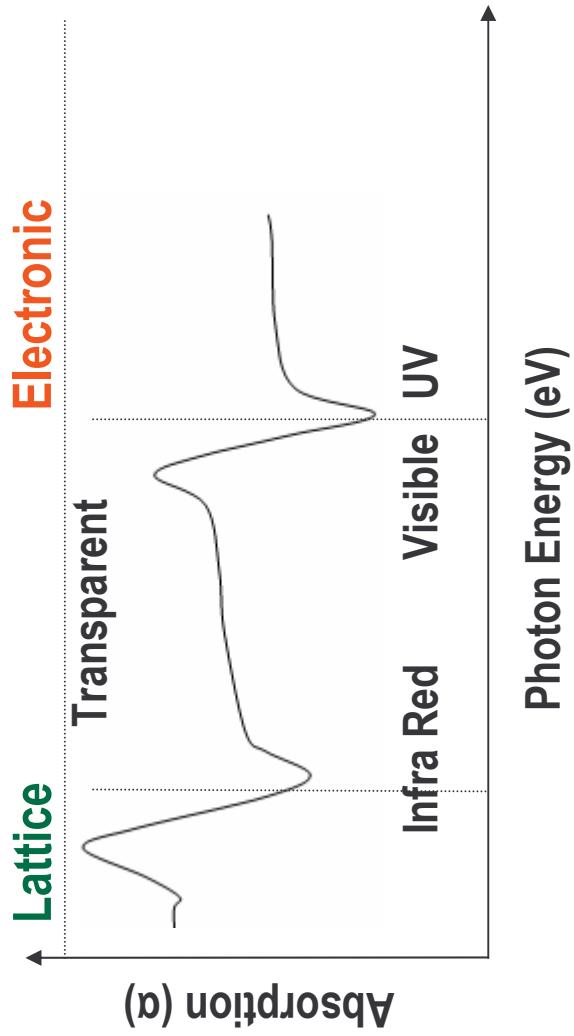
- Metal: no gap $E_g = 0$
- Insulator (dielectric): large gap $E_g > 4 \text{ eV}$
- Semiconductor: $0 < E_g < 4 \text{ eV}$



Material Absorption Theory

- **3 fundamental processes involving interaction between material and EM wave**

- Electronic absorption
- Lattice or phonon absorption
- Free carrier absorption



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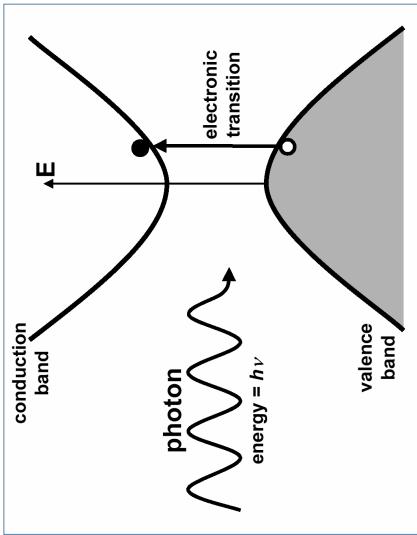
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Absorbing Regions

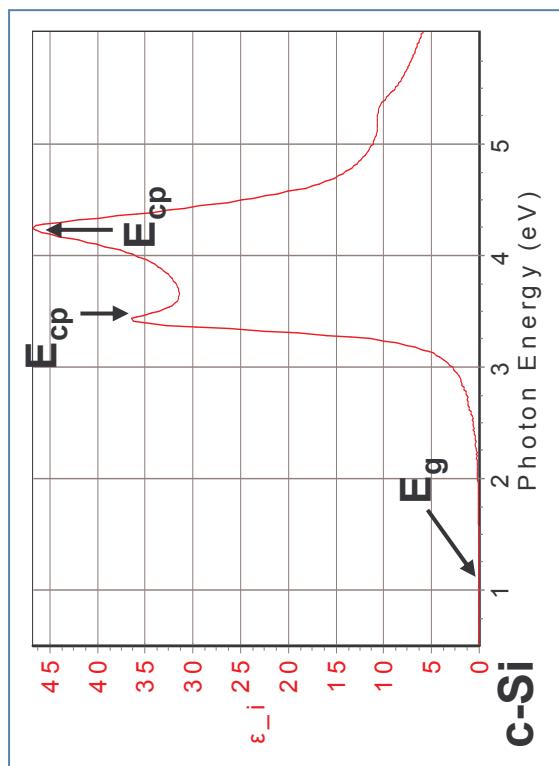
Electronic transitions

- Result of interaction between the incident light and the motions of e^- or holes within the material



- Electrons in valence band
- Gap between valence & conduction band
- If photon has energy larger than gap, electron may excite to higher state

- Absorption associated with each transition
- Band-gap energy (E_g): minimum energy where transitions can occur
- Critical point energies (E_{cp}) = photon energies where there is a high probability of band-to-band transitions



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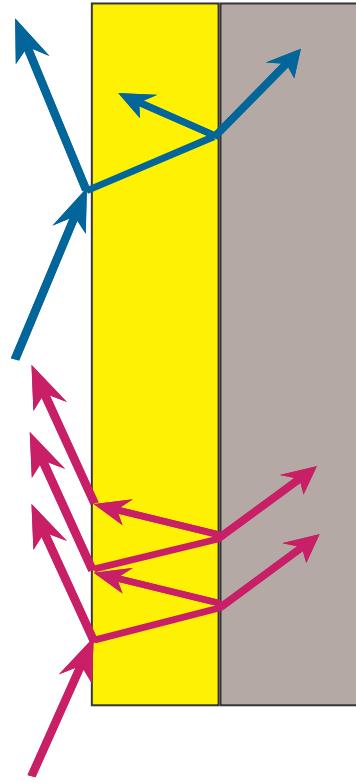
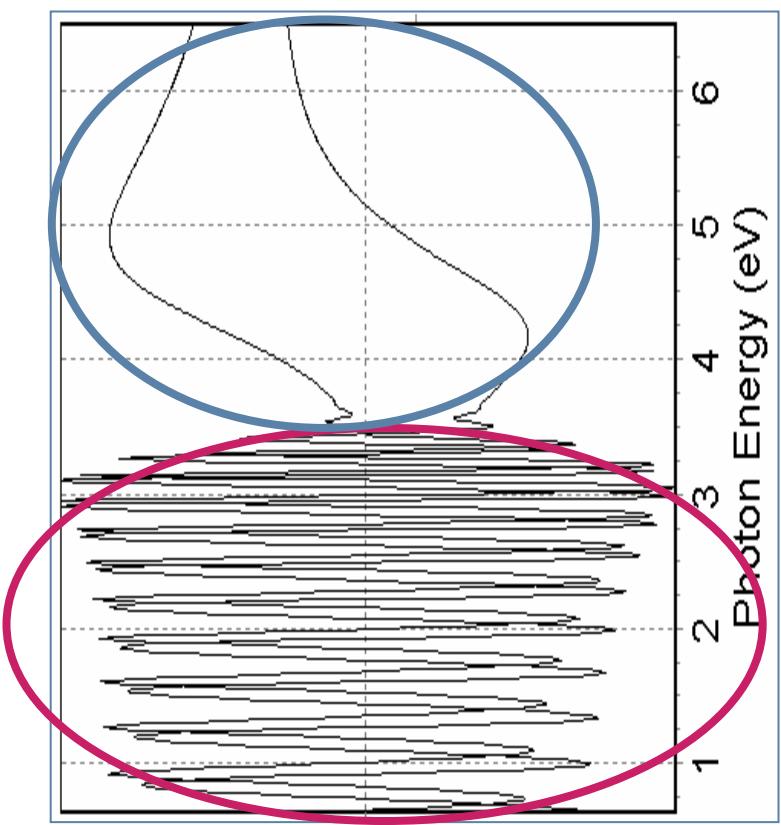
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Reflections with Films

- **Transparent film:** see interference fringes ($>1000 \text{ \AA}$)
⇒ Thicker films produces more interference fringes (in the example
 TiO_2 layer thickness: 8000 \AA)

- **Absorbing film:** no interference fringes, behaves as a substrate



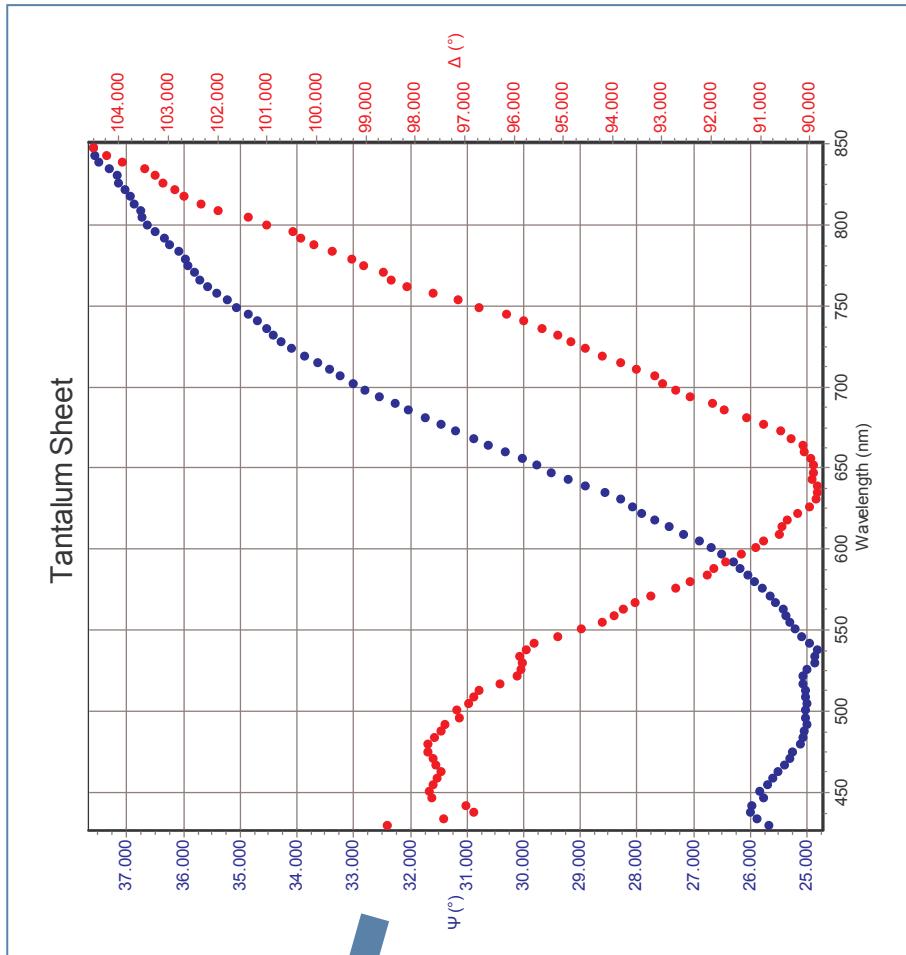
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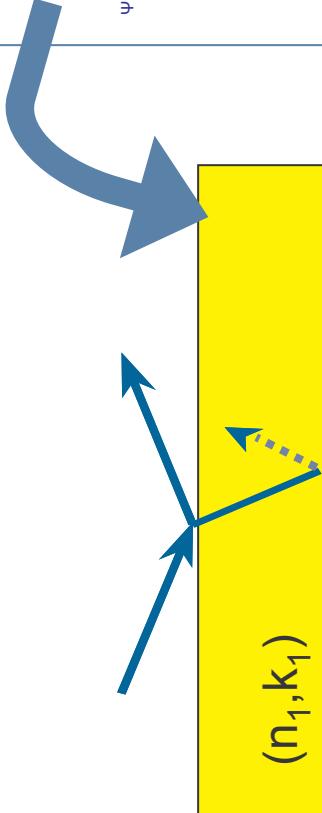
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Bulk Sample or Substrate

- Measurement of a bulk sample gives directly the (n, k) of the material



- Ratio $\rho \Rightarrow (\Psi, \Delta) = f(\varepsilon_0, \varepsilon_1, \theta_0)$
- 2 measured parameters : (Ψ, Δ)
- 2 unknowns : n_1, k_1



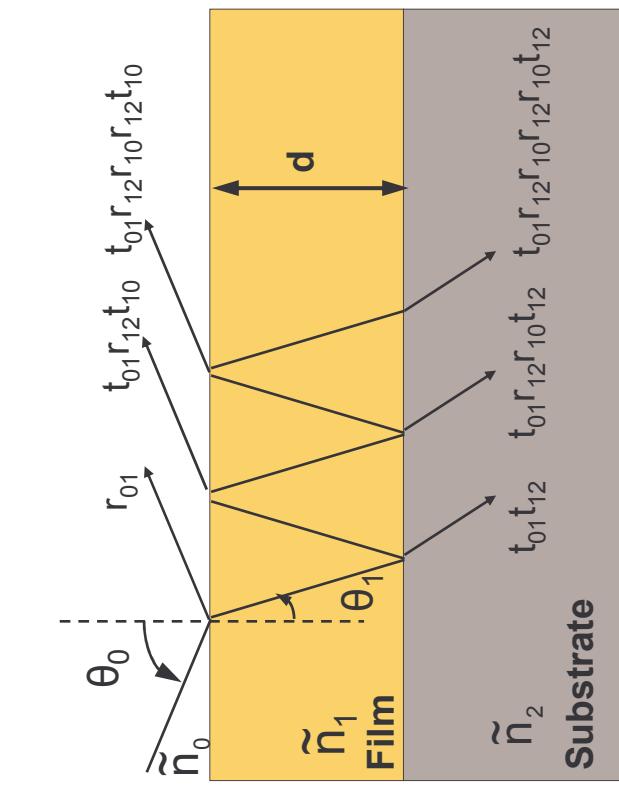
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Thin Film on Substrate

- Thin film characterization: thickness and (n, k)



$R = \Sigma r$ related to the 1&2 interfaces

$$R = \frac{r_{01} + r_{12}e^{-2j\beta}}{1 + r_{01}r_{12}e^{-2j\beta}}$$

β : Film phase thickness

$$\beta = 2\pi \left(\frac{d}{\lambda}\right) n_1 \cos \phi_1$$

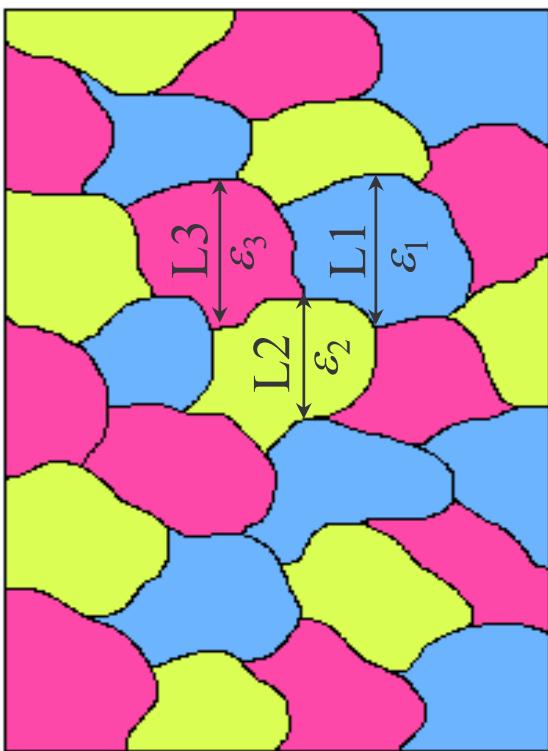
$$\text{Ratio } \rho \Rightarrow (\Psi, \Delta) = f(\varepsilon_0, \varepsilon_1, \varepsilon_2, \theta_0, d, \lambda_0)$$

2 measured parameters : (Ψ, Δ)

3 unknowns : n_1 , k_1 and d

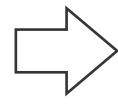
Effective Medium Approximation Theory

- All materials are inhomogeneous on atomic scale
- Part of macroscopically homogeneous materials is inhomogeneous on microscopic scale



If each separate region is large enough to possess their own dielectric identities, but small compared to wavelength of light

$$(L_1, L_2, L_3 < \lambda)$$



Effective Medium Approximation

can be used

- EMA is used to calculate the dielectric functions of mixed phases based on their microstructure and component volume fractions

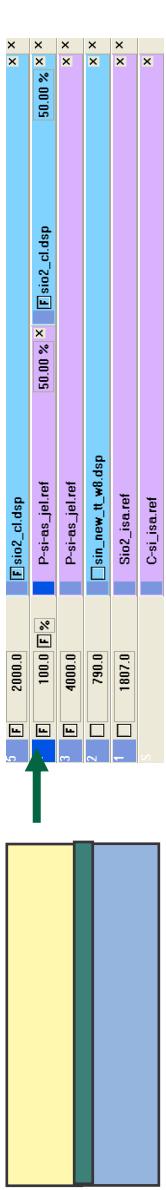
Effective Medium Approximation (EMA)

⇒ Calculate optical constants of mixed materials (handled by the software !)

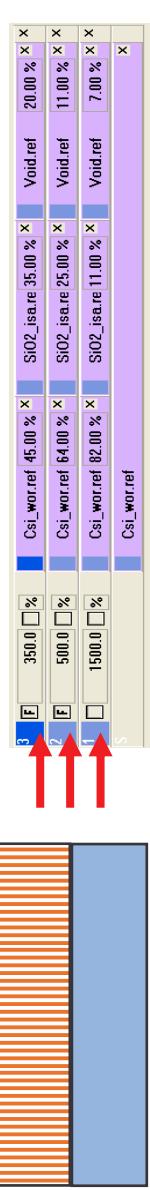
Surface roughness



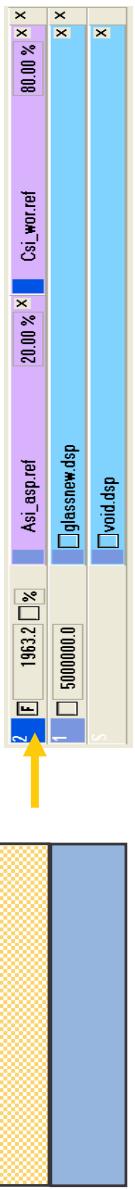
Interface



Porosity



Polycrystalline materials



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Effective Medium Theory

Maxwell Garnett and Bruggeman models

■ Maxwell Garnett model

- The Maxwell-Garnett is derived assuming spherical inclusions (denoted ϵ_i) with a volume fractions f exist in a host matrix of the second material (denoted ϵ_h)

$$\epsilon = \epsilon_h \frac{\epsilon_i(1+2f) + 2\epsilon_h(1-f)}{\epsilon_i(1-f) + \epsilon_h(2+f)}$$

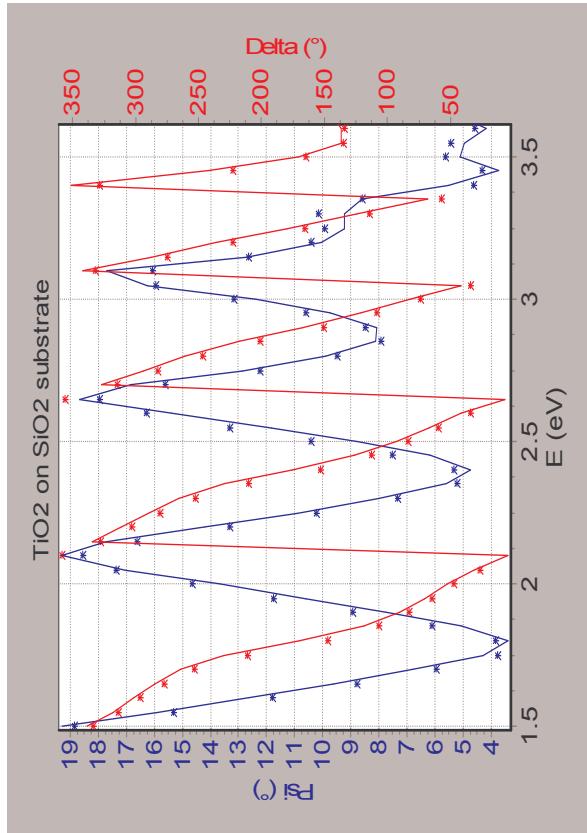
■ Bruggeman model

- Non restrictive of the number of components of the mixture which are treated in an equivalent way by holding account only of their proportion
- Self-consistent theory: Host material complex dielectric function equalling the final effective complex dielectric function of the multi-constituent material

$$\sum_i f_i \frac{\epsilon_i - \epsilon}{\epsilon_i + 2\epsilon}$$

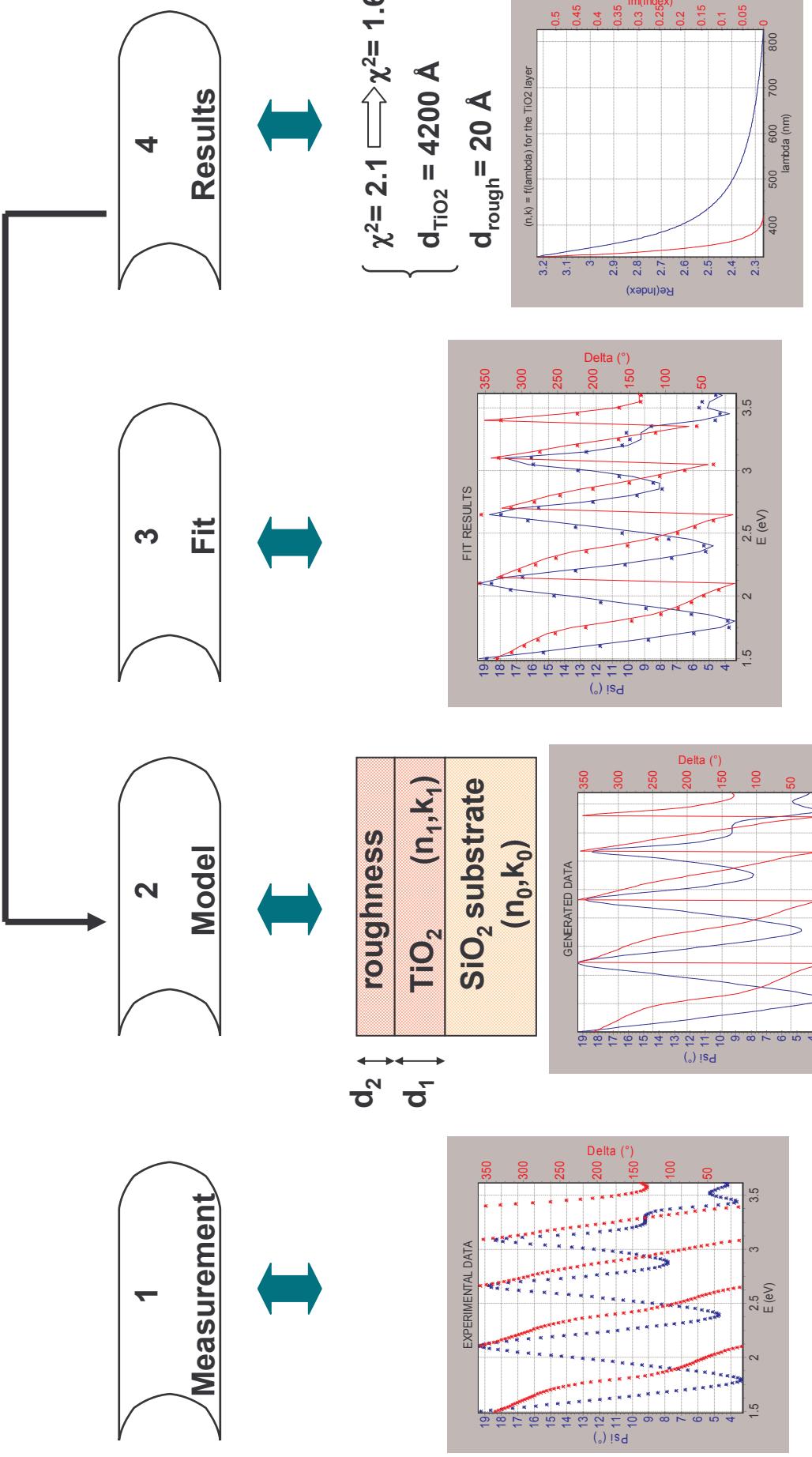
ANALYSIS OF ELLIPSOMETRIC DATA

- Ellipsometry does not measure film thicknesses or optical constants, it measures Ψ and Δ



- To extract these informations from a sample, it is necessary to perform a **model dependant analysis** of the ellipsometric angles
- A model is an idealized mathematical representation of the sample

SPECTROSCOPIC ELLIPSOMETRY DATA ANALYSIS FLOWCHART



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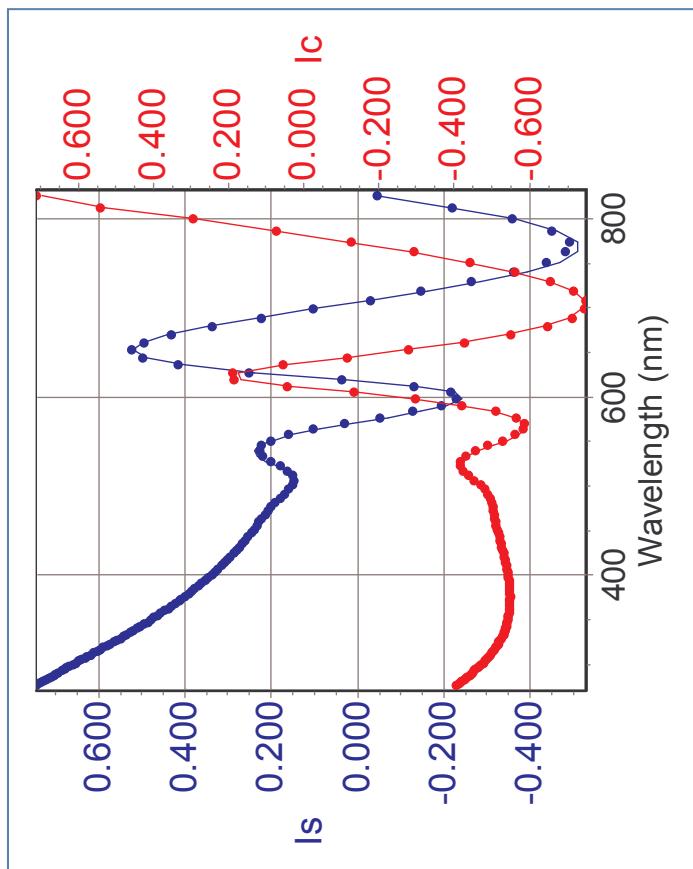
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Data Fitting

$$\chi^2 = \min \sum_{i=1}^n \left[\frac{(\psi_{th} - \psi_{exp})_i^2}{\Gamma_{\Psi,i}} + \frac{(\Delta_{th} - \Delta_{exp})_i^2}{\Gamma_{\Delta,i}} \right]$$

- χ^2 parameter quantifies the difference between experimental and model data
- Software adjusts fit parameters to find best match between model and experimental curves



Roughness
23 Å
a-Si
Glass substrate

Results $\chi^2 = 7.8 \rightarrow \chi^2 = 0.5$

- A smaller χ^2 implies a better fit

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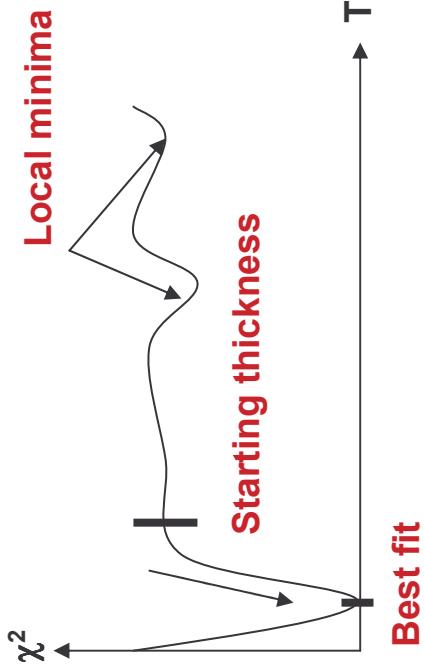
Data Fitting

■ Minimization methods

- Levenberg-Marquardt algorithm : commonly used
- Others: Simplex, ...

■ Difficulties

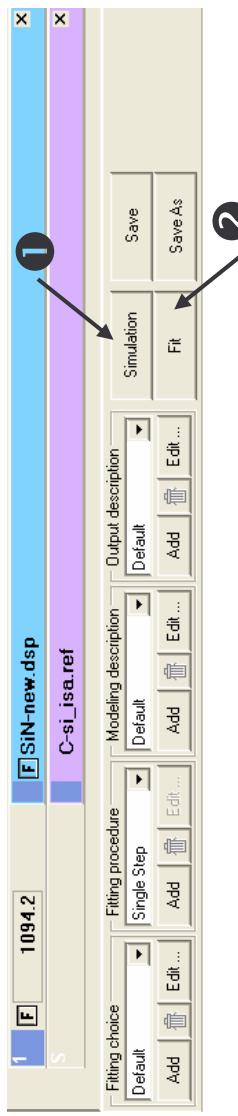
- Local minimum
- Many variables
- Setting starting parameters: have to be close to the final solution



■ Getting good starting values

- ① Use the simulation function

- Generate from model and compare with experimental data



- ② Fit the selected parameters

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Evaluate the Quality of Results

- Compare experimental data with generated data
- How low is the χ^2 ? Can it be reduced further by increasing model complexity ?
- Are fit parameters physical ?
- Check the correlation matrix. Values close to 1 mean correlated parameters, and a non-unique solution.

Correlation matrix \Rightarrow 6 fit parameters

| =1= | =2= | =3= | =4= | =5= | =6= |
|-------|--------|--------|--------|--------|-------|
| 1.000 | -0.431 | -0.148 | -0.012 | -0.225 | 0.115 |
| 1.000 | 0.475 | 0.127 | -0.746 | -0.014 | |
| 1.000 | 0.890 | 0.474 | 0.765 | | |
| 1.000 | 0.205 | 0.970 | | | |
| 1.000 | -0.123 | | | | |
| 1.000 | | | | | |

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General Rules for Ellipsometric Data Analysis

3 essential steps

1 - Experimental measurement

→ Check the good working of the ellipsometer (NIST)

→ Sample preparation

→ Acquisition parameter choices

- Spectral range (NIR-FUV) / sample properties
- Monochromator step / layer thickness
- Angle of incidence (brewster)
- Beam diameter

General Rules for Ellipsometric Data Analysis

3 essential steps

2 - Modeling

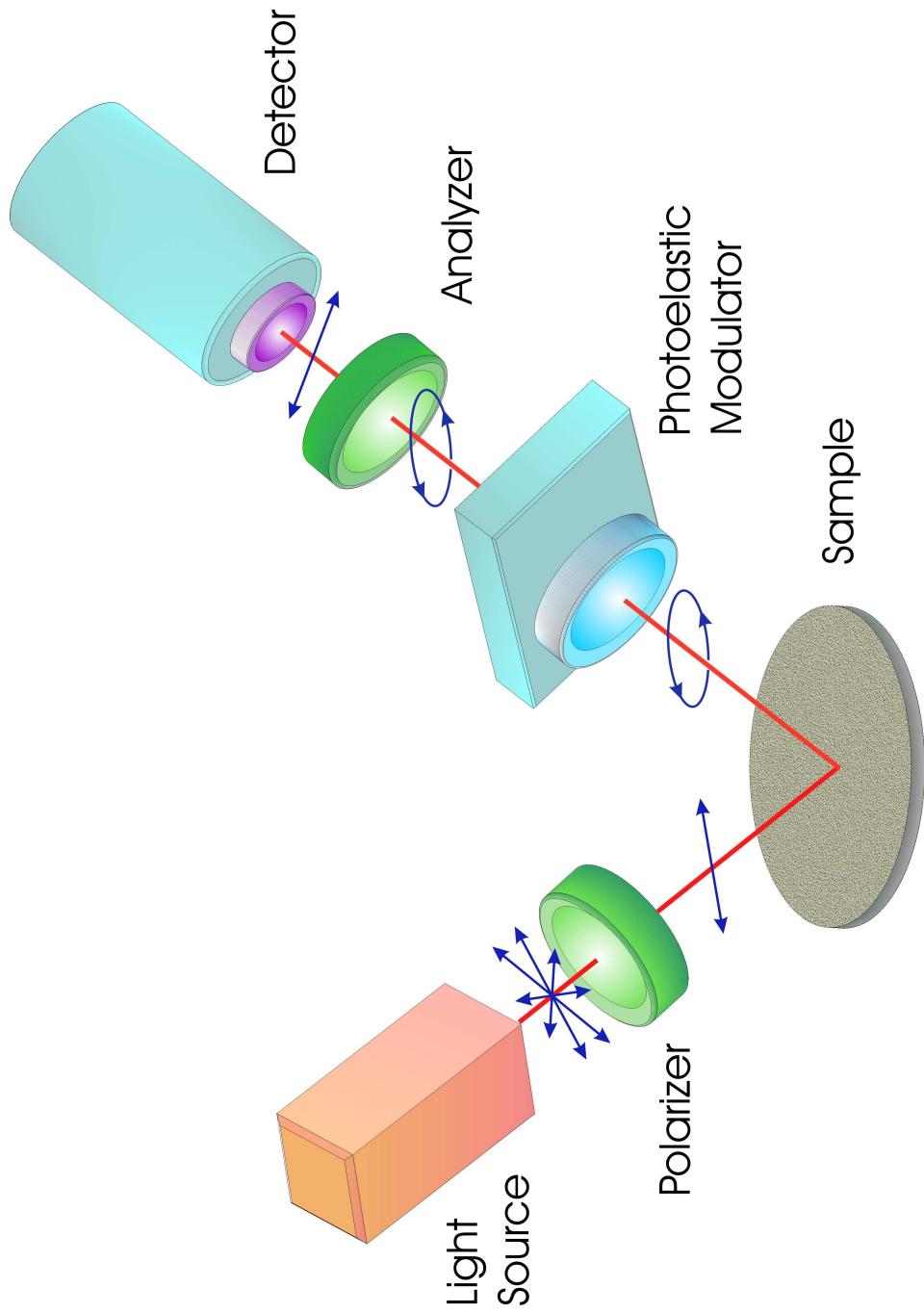
→ Build the most realistic optical model

3 - Reliable model choice

- Criteria and quality of a good fit
- Physical result (model and parameter values)
 - Slight correlation between parameters : uniqueness of the solution
 - Final result independent of initial parameters
 - Quick convergence

Phase Modulation Spectroscopic Ellipsometer

Optical Set-up



UVISEL

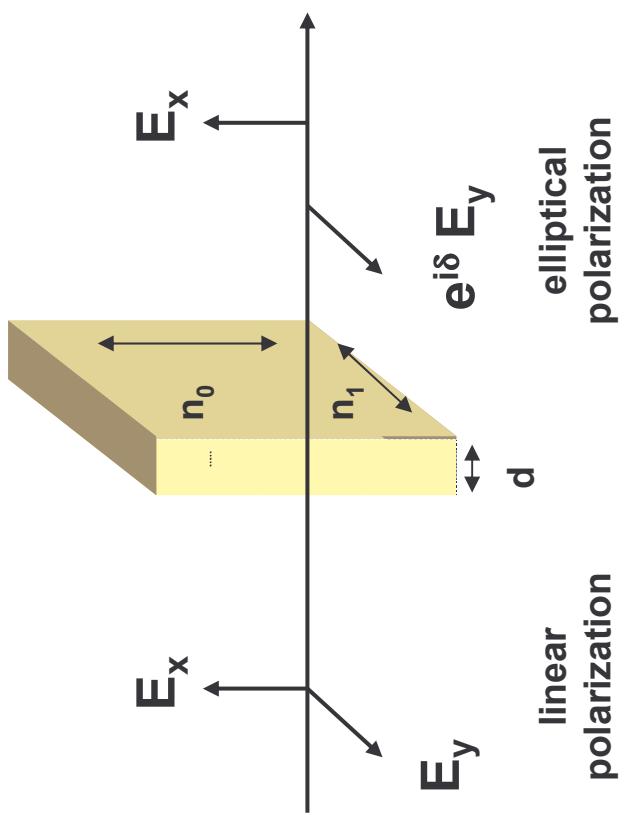
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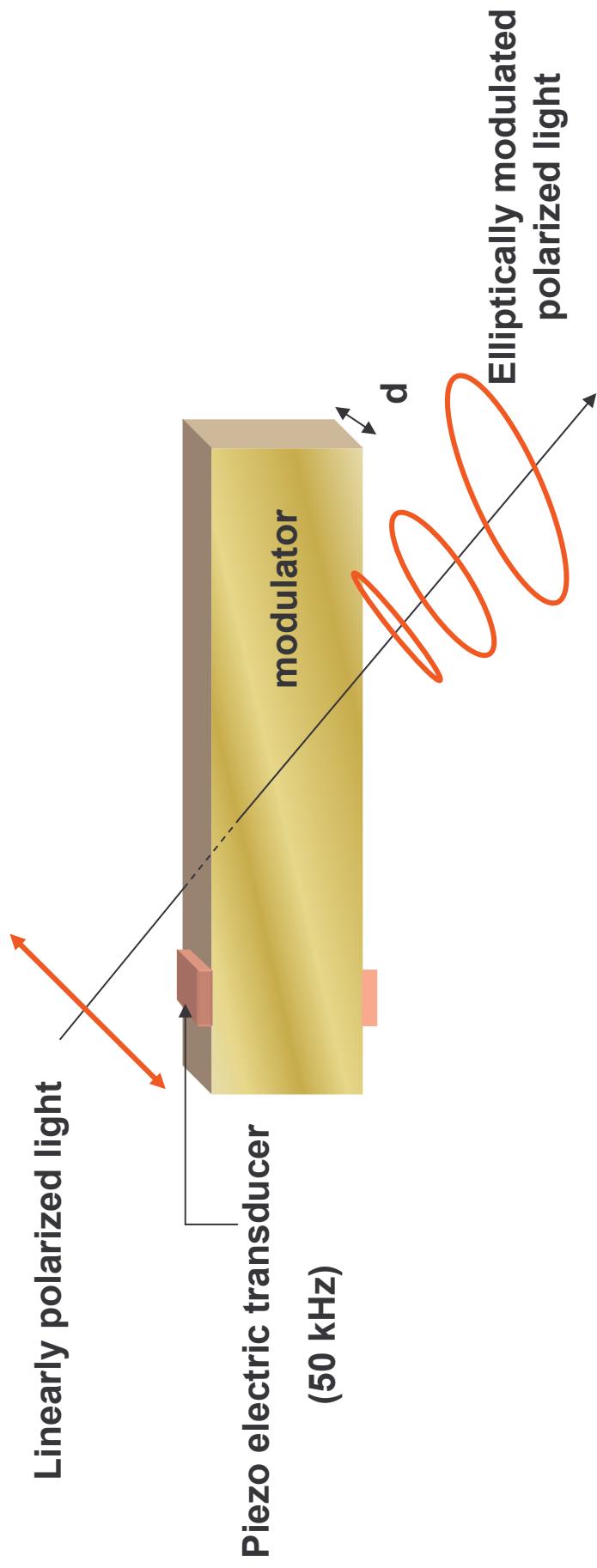
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What is a photoelastic modulator and How does it work ?

- **PEM definition**
 - fused silica bar sandwiched between piezo oscillating at the frequency w=50 kHz
- **Stress effect**
 - creation of an optical anisotropy in the silica bar
- **Strain modulation**
 - optical anisotropy modulated
 - polarization modulated
- **Modulated phase shift $\delta(t)$**
 - $\delta(t) = A \sin \omega t$
 - with : $A = 2\pi d (N_1 - N_0)/\lambda$



What is a photoelastic modulator and How does it work ?



Phase Modulation Formalism

Based on Jones Formalism

- Reference basis (\vec{p}, \vec{s})

- Each element of the optical chain is associated to a matrix (2×2)

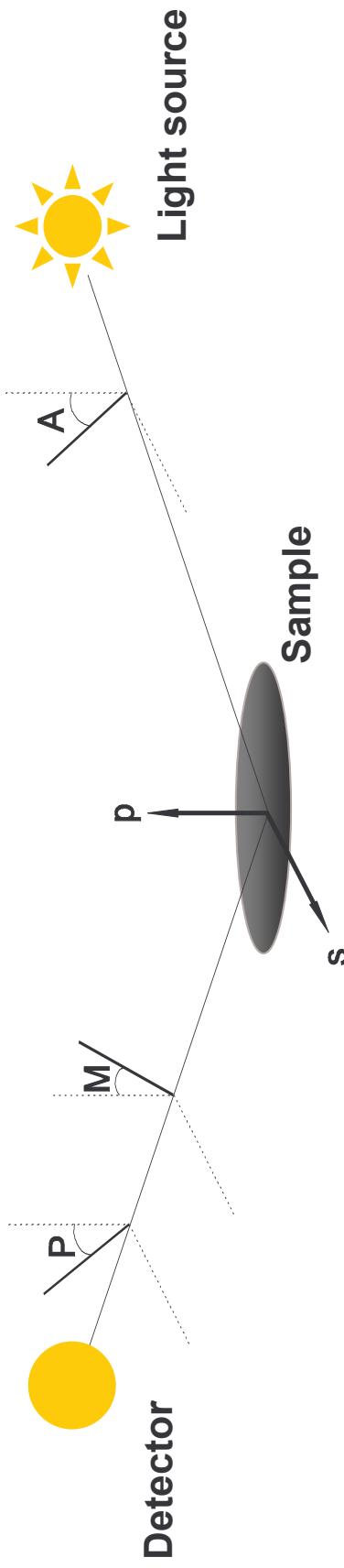
- Sample $E = \begin{pmatrix} r_p & 0 \\ 0 & r_s \end{pmatrix}$

- Polarizer $P = \begin{pmatrix} 1 & 0 \\ 0 & 0 \end{pmatrix}$

- Modulator $M = \begin{pmatrix} e^{i\delta} & 0 \\ 0 & 1 \end{pmatrix}$

- Coordinate system rotation $R(\theta) = \begin{pmatrix} \cos\theta & -\sin\theta \\ \sin\theta & \cos\theta \end{pmatrix}$

Phase Modulation Formalism



■ Jones formalism for transmitted field : $E_t = T(A R_A S R_M M R_{P-M} P) E_i \quad (I)$

■ Leading to detected intensity : $I(t) = E_t E_t^* = I_0 + I_s \sin\delta(t) + I_c \cos\delta(t)$

■ Development of equation (I) leads to :

$$I_0 = 1 - \cos 2\Psi \cos 2A + \cos 2(P-M) \cos 2M (\cos 2A - \cos 2\Psi) + \\ \cos 2(P-M) \sin 2A \sin 2M \sin 2\Psi \cos \Delta$$

$$I_s = \sin 2(P-M) \sin 2A \sin 2\Psi \sin \Delta$$

$$I_c = \sin 2(P-M) [\sin 2M (\cos 2\Psi - \cos 2A) + \sin 2A \cos 2M \sin 2\Psi \cos \Delta]$$

Phase Modulation Formalism

■ 2 Common Measurement Configurations

- Configuration II: P – M=45 [90]; M = 0 [90]; A = 45 [90]

$$\rightarrow |s = \sin 2\Psi \sin \Delta$$

$$|c = \sin 2\Psi \cos \Delta$$

➔ Accurate measurement of Δ over [0-360°]

- Configuration III: P – M=45 [90]; M = 45 [90]; A = 45 [90]

$$\rightarrow |s = \sin 2\Psi \sin \Delta$$

$$|c' = \cos 2\Psi$$

➔ Accurate measurement of Ψ over [0-90°]

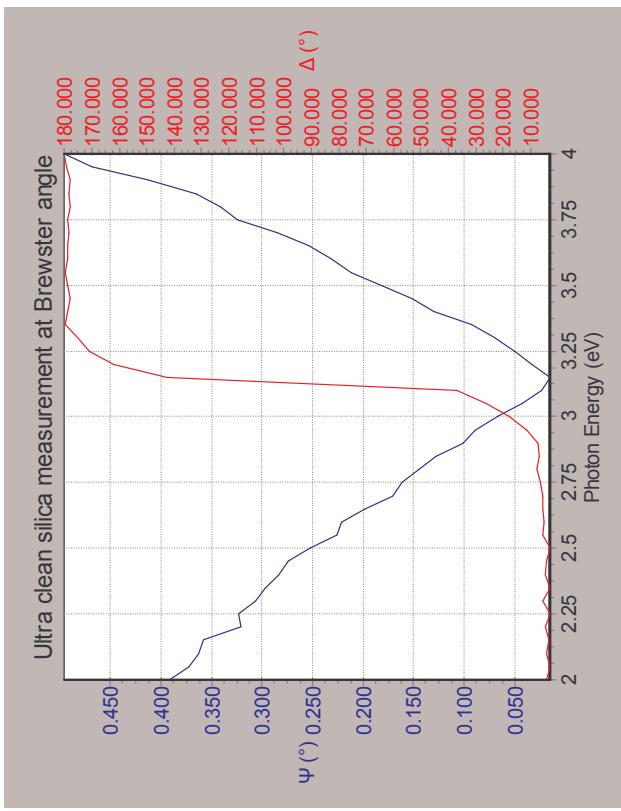
■ Depolarization Measurement

- Given by: $P = (|s|^2 + (|c'|^2 + (|c|^2)^2)$
- $P=1$: the sample is not depolarizing
- $P<1$: the sample is depolarizing

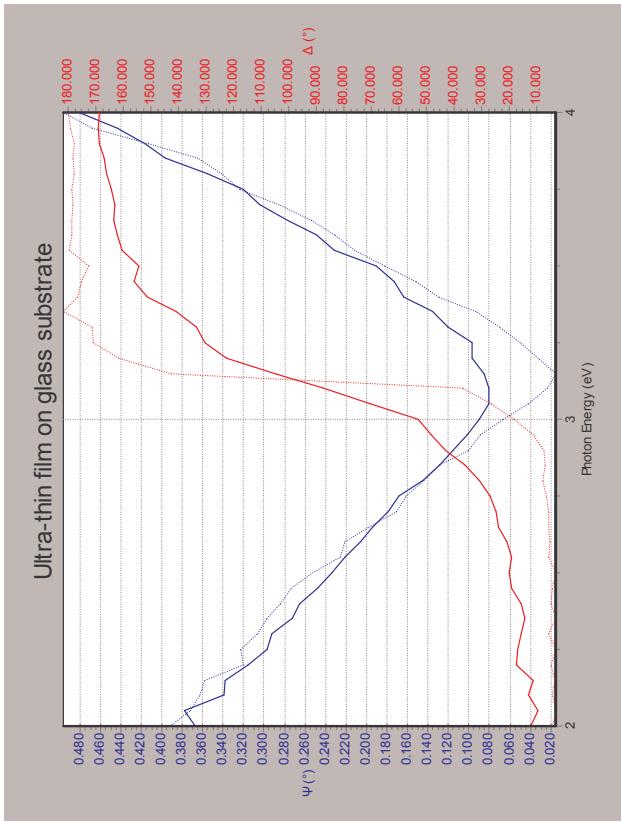
High Accuracy, High Sensitivity Phase Modulation Ellipsometer

➔ Accurate measurement of
 Δ parameter around 0 & 180°

➔ High sensitivity to ultra-thin films



Measurement of silica substrate at
the Brewster angle



10 Å monolayer deposition on silica
substrate

— Ultra-thin monolayer
— Silica substrate

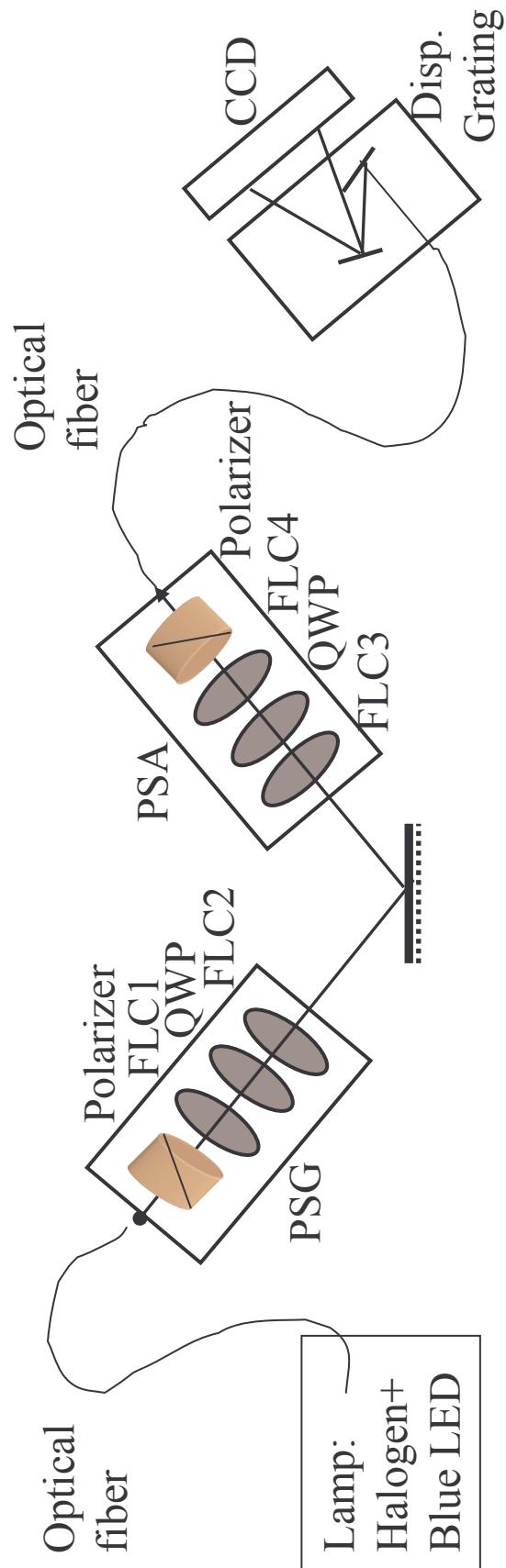
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Liquid Crystal Modulation Spectroscopic Ellipsometer

Optical Set-up



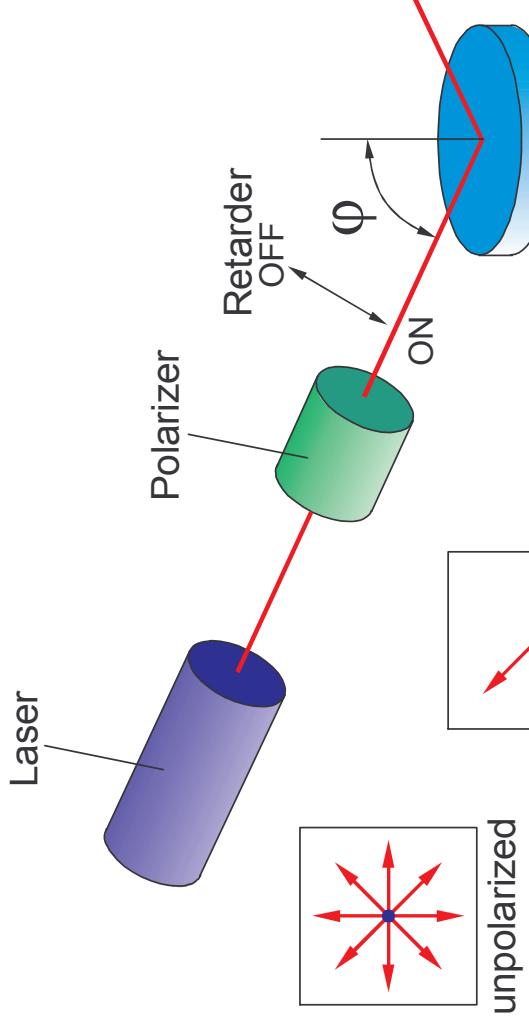
> Input and output heads are symmetrical

- Polarizer
- LC Modulator – states 0° and 45°
- Quarter Waveplate
- LC Modulator – states 0° and 45°

Laser Ellipsometer

Optical Set-up

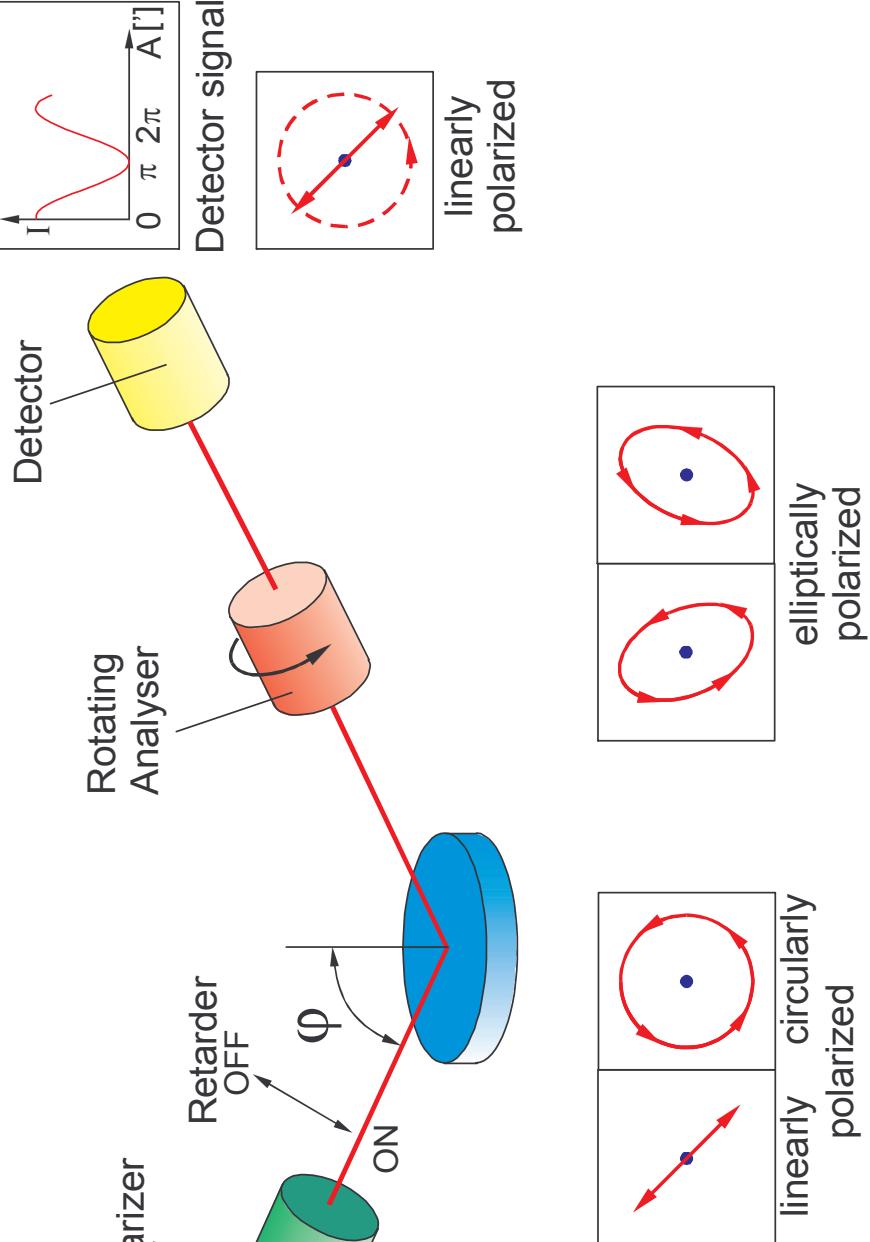
Polarizer Arm:



linearly polarized

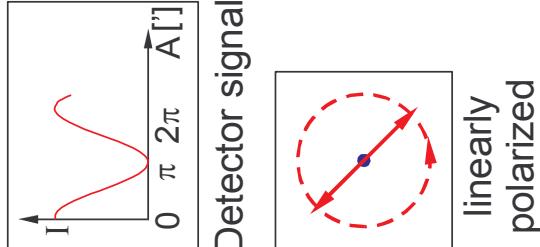
linearly circularly polarized

Analyzer Arm:



linearly polarized

elliptically polarized



PZ2000

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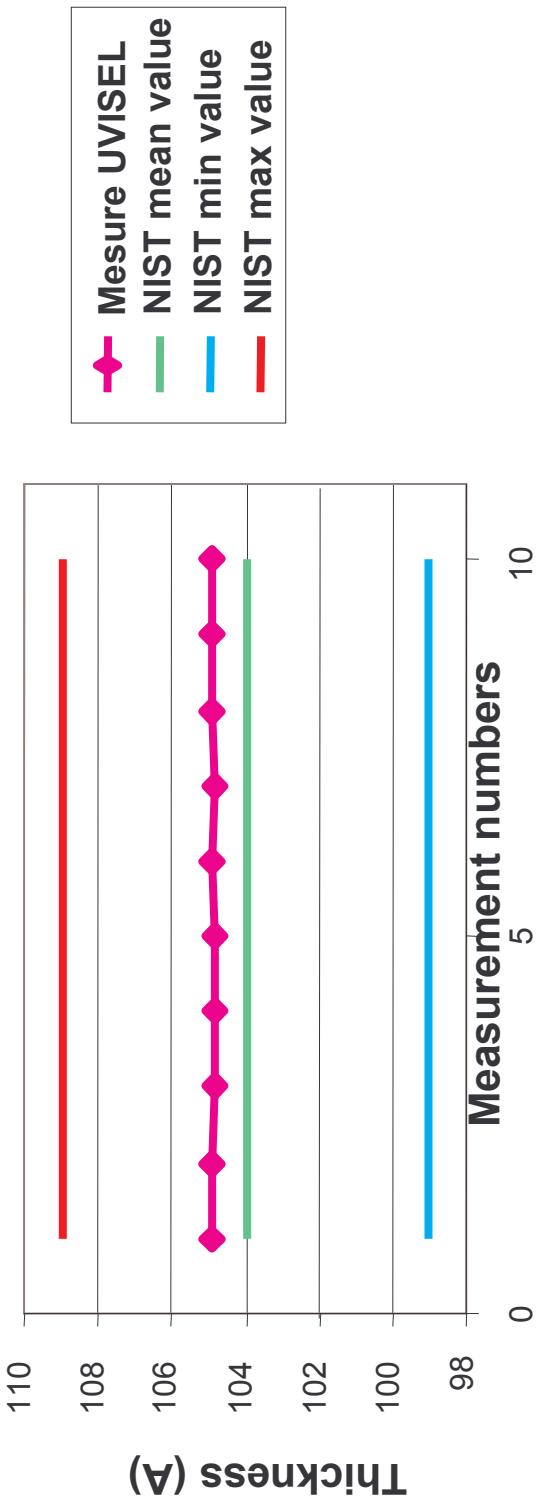
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Ellipsometer Accuracy

■ Reference standards NIST

- Check the good working of the ellipsometer by measuring one time per week your standard sample.



■ Straight-through air measurements

- The only material for which the ellipsometric parameters are absolutely known is “Air”
- An ellipsometric measurement in the straight-through configuration should by definition return $\Psi=45^\circ$ and $\Delta=0^\circ$

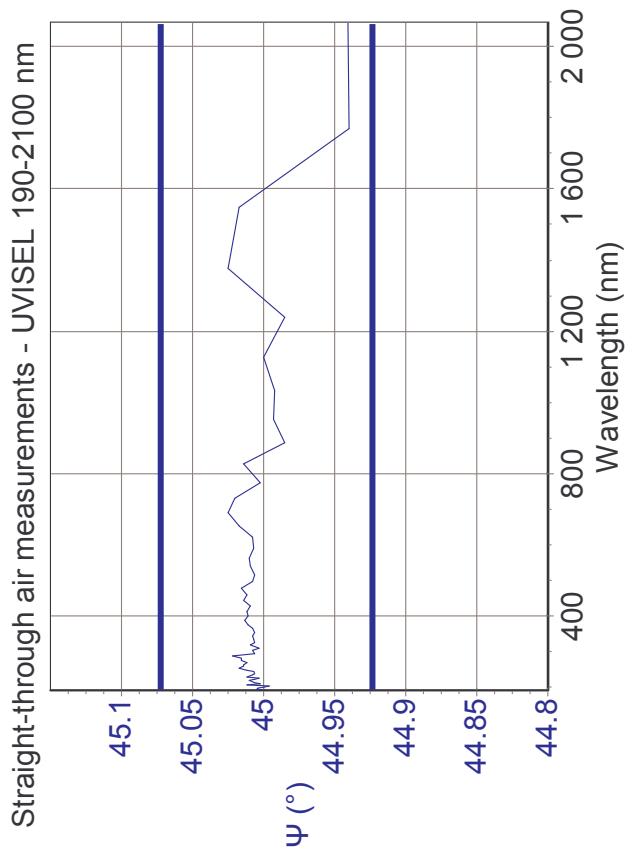
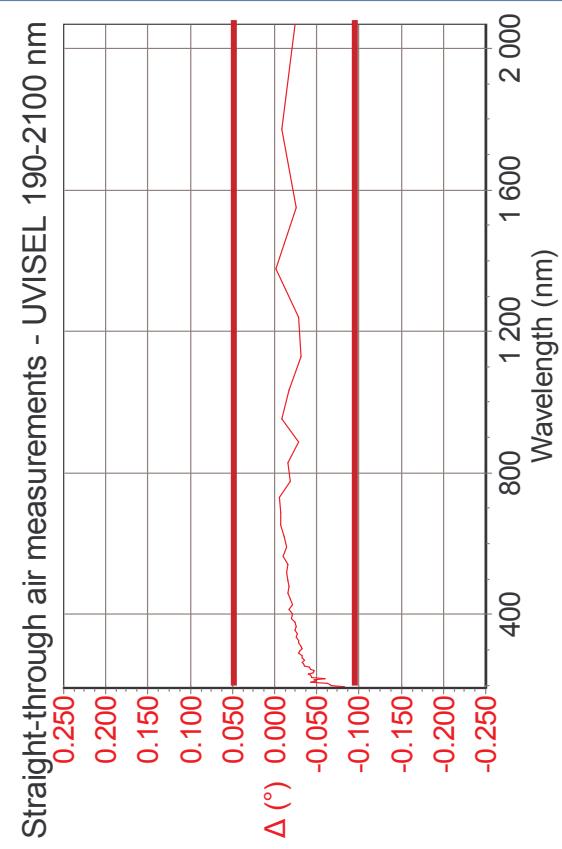
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Ellipsometer Accuracy

Straight-through air measurements



- **HJY UVISEL Specifications**

- $\Delta = 0^{\circ} \pm 0.083^{\circ}$
 - Spectral range: 190 – 2100 nm
- $\Psi = 45^{\circ} \pm 0.06^{\circ}$
 - Spectral range: 190 – 2100 nm

- **UVISEL Specifications**

- $\Delta = 0^{\circ} \pm 0.083^{\circ}$

- $\Psi = 45^{\circ} \pm 0.06^{\circ}$
 - Spectral range: 190 – 2100 nm

Setting up Ellipsometric Measurement

Spectral range

■ What are your materials ?

- Semiconductor: transparent in NIR, strongly absorbing in VIS-FUV
- Dielectric: transparent, absorbing in the FUV
- Metal: strongly absorbing from NIR to FUV, especially in NIR

■ Which properties of interest ?

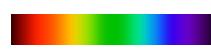
- Thickness
- Optical constants
- Material properties: composition, crystallinity...

■ Final applications

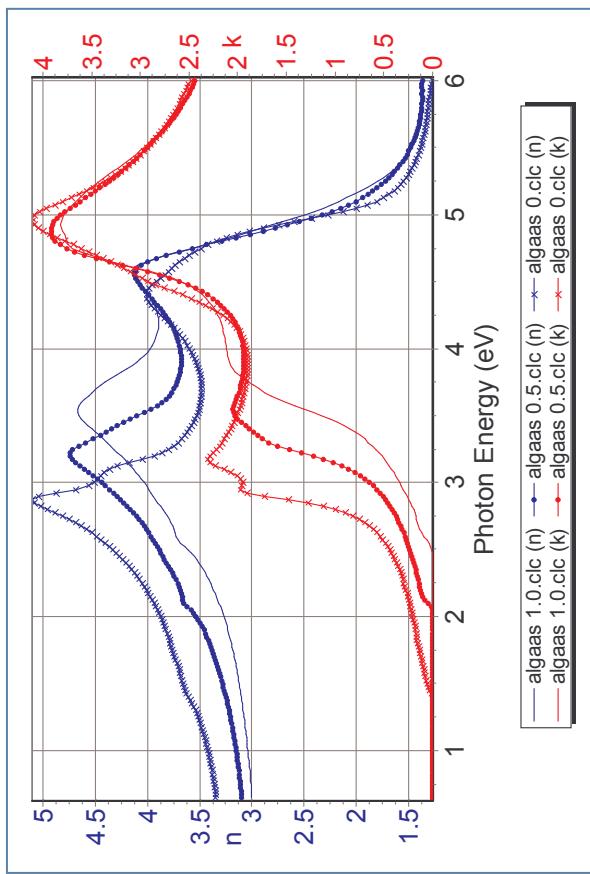
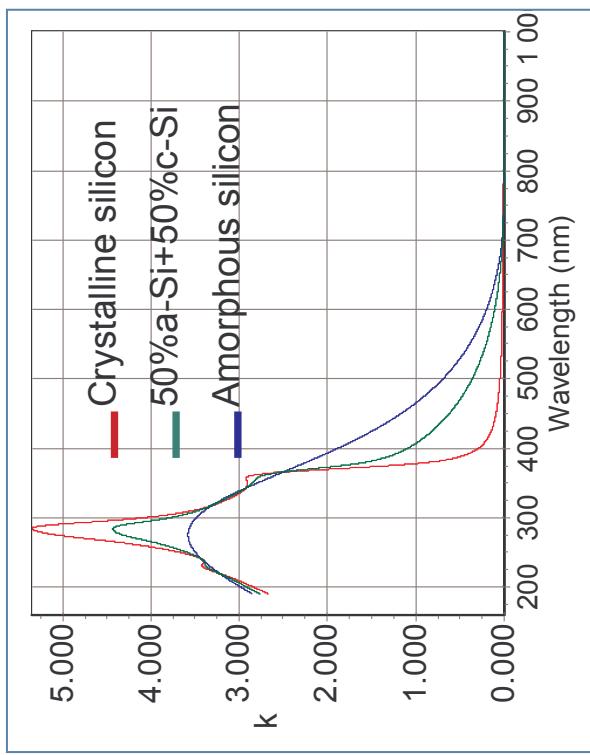
- Telecommunications, data storage applications require NIR wavelengths
- Lithography requires FUV wavelengths
- Advanced applications may require a special spectral range to provide accurate results

Spectral range

⇒ Transparent region: thickness characterization



⇒ Absorptive region: crystallinity, composition characterization



- Increasing Al increases the bandgap, which shifts the absorption edge to shorter λ

- Electronic transitions critical point broaden in amorphous materials

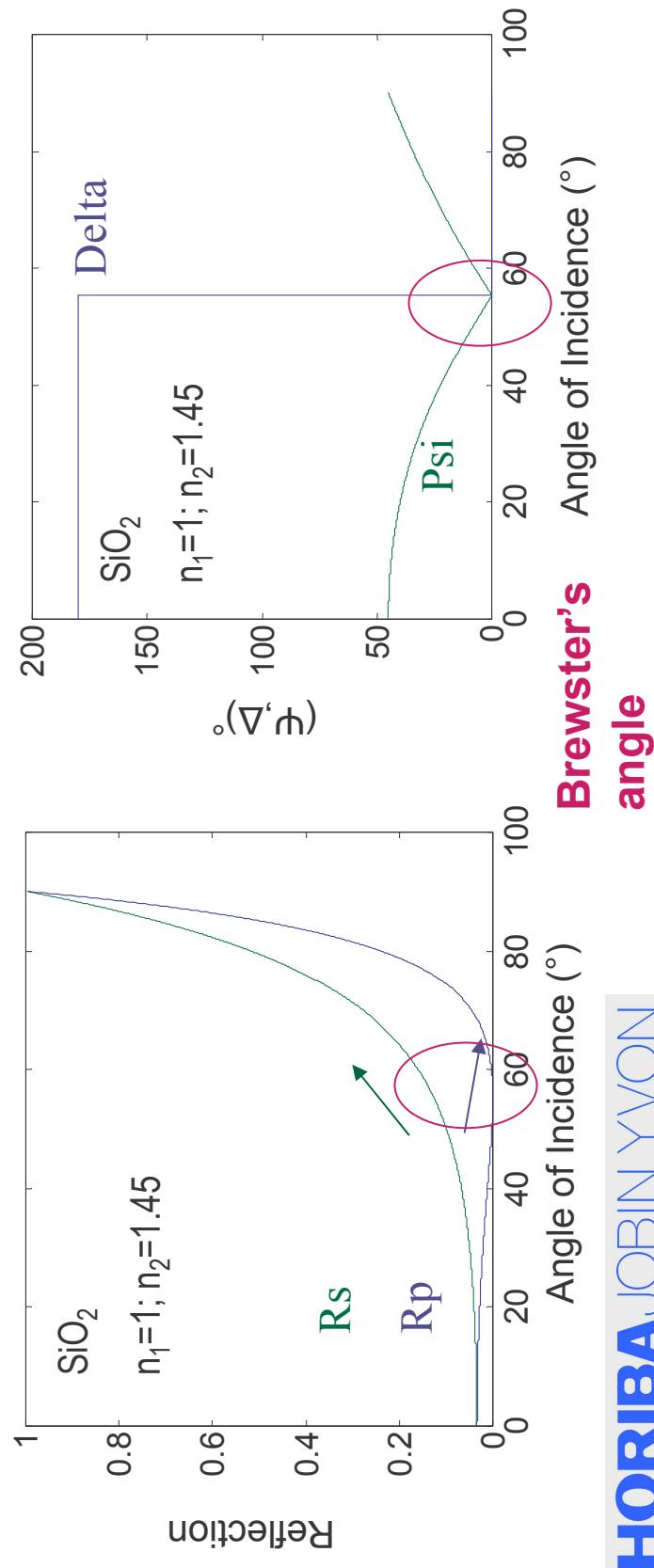
Angles of incidence

- Ellipsometry measurements are acquired at oblique angle of incidence

▪ Why typically between 50° and 80° ?

- To maximize the difference between s and p polarized light

⇒ Brewster Angle



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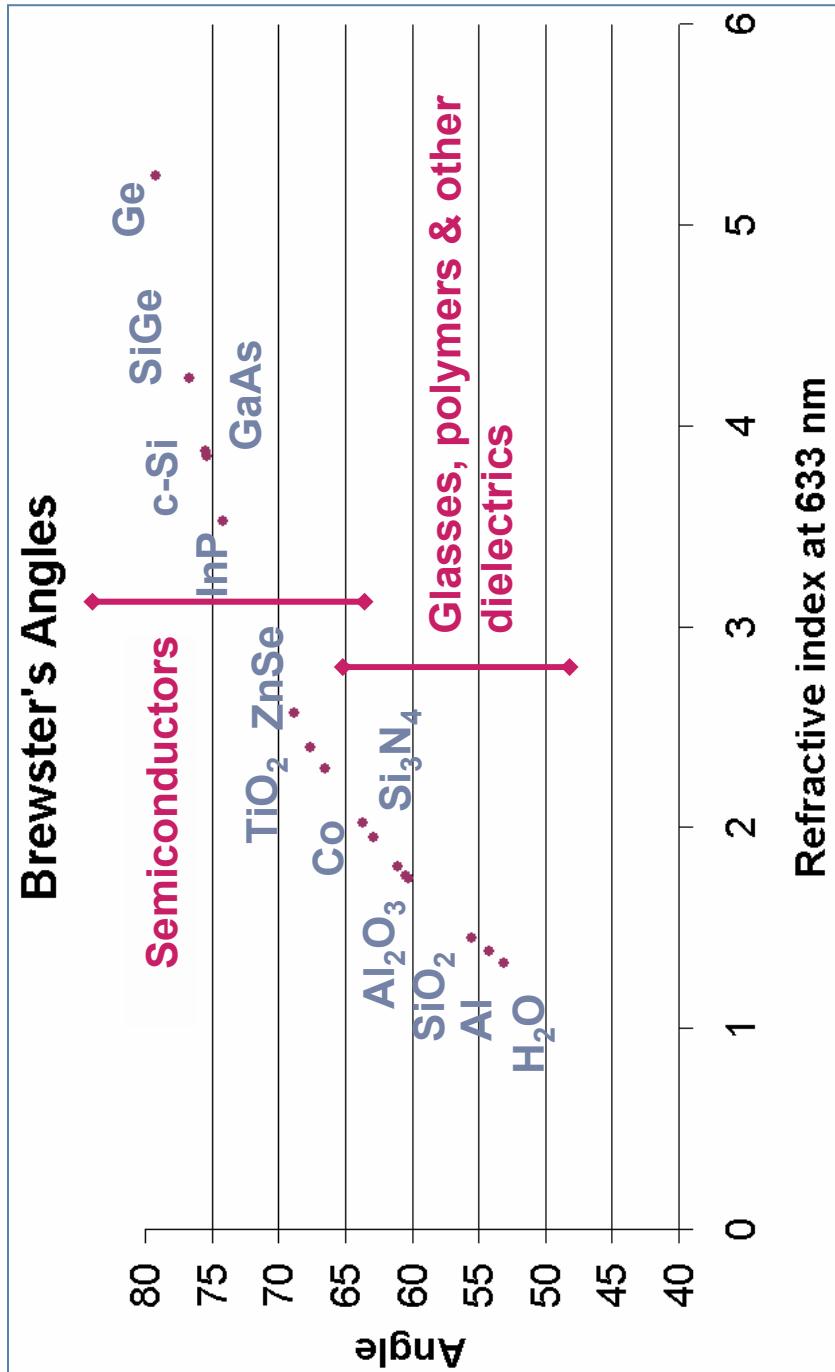
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Brewster's Angles

⇒ Brewster angle defined by

$$\tan \phi_B = \frac{n_2}{n_1}$$



Ellipsometry = Versatile Technology

Overview of Applications Fields

■ Semiconductor

- Transistors, memories, nanostructures...

■ Optoelectronic technologies

- Flat panel display, photovoltaic cells

■ Optical coatings

- ARC, electrochromic coating, auto-cleaning glass, mirror ...

■ Telecommunications

- IR materials, data storage, non linear optical devices

■ Bio-Nano technology

- Protein adsorption, SAM, surface chemistry, carbon nanotubes

■ Packaging

- Barrier coating for food plastic film, bottles

S

OT

OC

T

BNT

P

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CONCLUSION

- Why are Spectroscopic Ellipsometers powerful tools for materials characterization ?
 - Non destructive technique
 - Wide range of materials properties and excellent thickness resolution
 - Interesting correlation with many others techniques
 - Modular design
 - Robust technique for both research and industry