

# **Optical spectroscopy in materials science 7.**

## **Practical KK transformation**

### **Ellipsometry**

### **Infrared spectroscopy 1.**

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Emission part by Hajnalka Tóháti, Wigner RCP

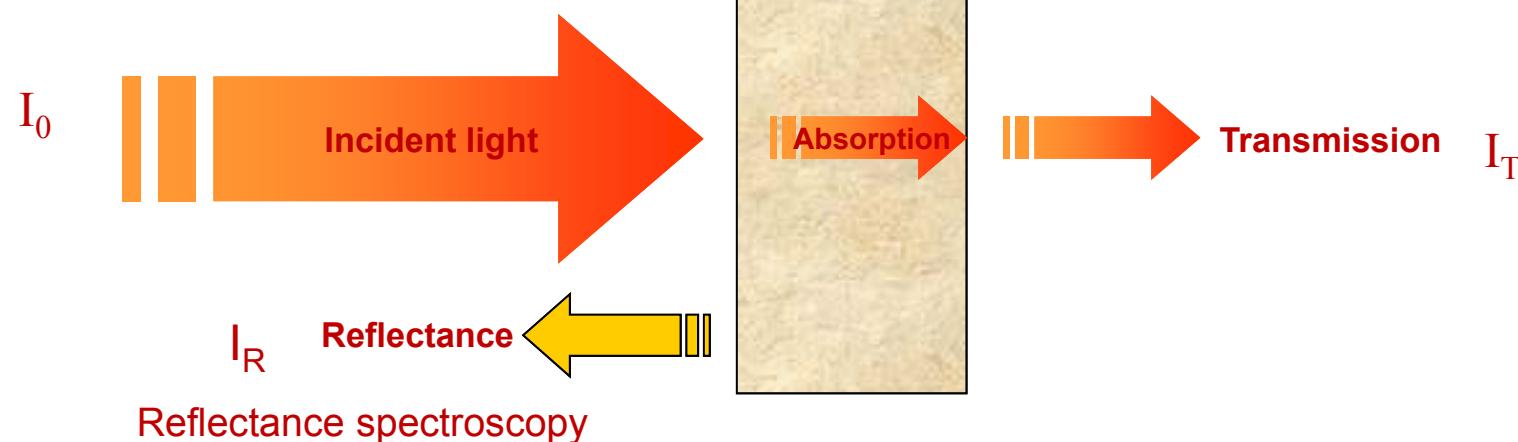


Budapesti Műszaki és Gazdaságtudományi Egyetem

# Reflectance spectroscopy

$$I_0 = RI_0 + (1 - R)I_0 e^{-\alpha d} + I_A$$

$$I_T \approx 0$$



Fresnel equations for normal incidence:

$$r = \frac{E_r}{E_i} = \frac{n' + i n'' - 1}{n' + i n'' + 1} = \sqrt{R} e^{i\theta}$$

$$R = \frac{I_R}{I_0} = \frac{(n' - 1)^2 + n''^2}{(n' + 1)^2 + n''^2}$$



# Dispersion relations - reflectance

$$r = \frac{E_r}{E_i} = \frac{n' + in'' - 1}{n' + in'' + 1} = \sqrt{R} e^{i\theta} \quad \ln r = \frac{\ln R}{2} + i\theta$$

In  $r$  is also a good transfer function, dispersion relations hold  
*measured quantity:*  $R(\omega)$

**Kramers-Kronig (KK) transformation:**

$$\theta(\omega) = -\frac{\omega}{\pi} \text{P} \int_0^\infty \frac{\ln R(\xi)}{\xi^2 - \omega^2} d\xi = \frac{-1}{2\pi} \text{P} \int_0^\infty \ln \left| \frac{\xi + \omega}{\xi - \omega} \right| \frac{d \ln R(\xi)}{d\xi} d\xi$$

**Contribution to  $\theta$ :**  $\xi - \omega$  small or  $d \ln R / d\xi$  large

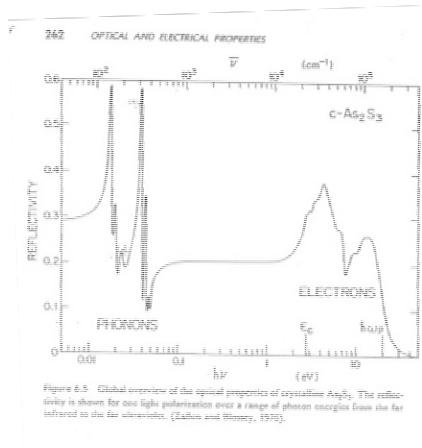


Figure 6.5 Global overview of the optical properties of crystalline  $\text{As}_2\text{S}_3$ . The reflectivity is shown for one light polarization over a range of photon energies from the far infrared to the far ultraviolet. (Zallou and Werner, 1999).



# KK analysis: extrapolations

$\omega \rightarrow 0$ :

- known  $\sigma(\text{dc})$ : fitting, smoothing
- semiconductors, bound electrons: constant R  
 $\epsilon''=0$ ,  $\epsilon'$  can be calculated from R

$\omega \rightarrow \infty$ :

asymptotic limit (free electrons):

$$R \sim \left( \frac{\omega}{\omega_p} \right)^{-4} \quad \text{reflectance of plasma } (\omega > 10^6 \text{ cm}^{-1}, \text{ all electrons excited})$$

intermediate region:  $R \sim \left( \frac{\omega}{\omega_p} \right)^{-S}$  S (and even  $\omega_p$ ) arbitrary, because we only want to approximate the integral,  $R \sim \omega^{-S}$

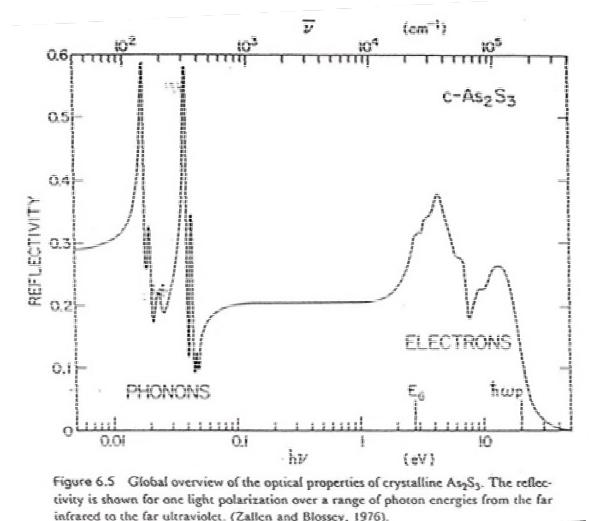


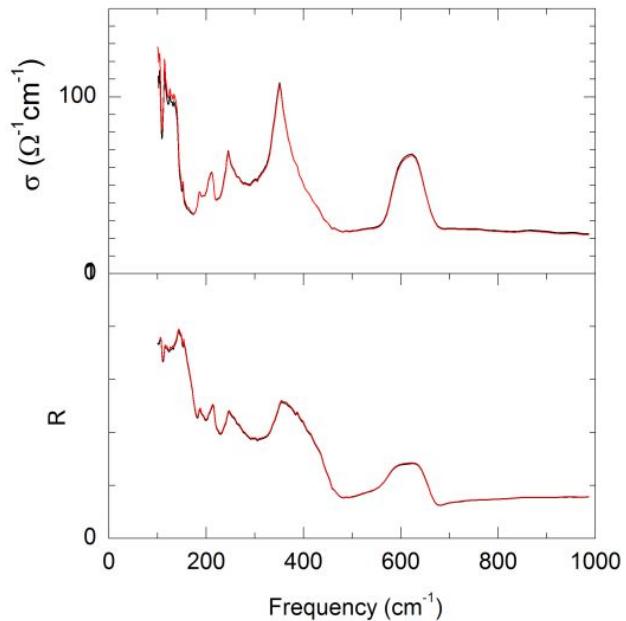
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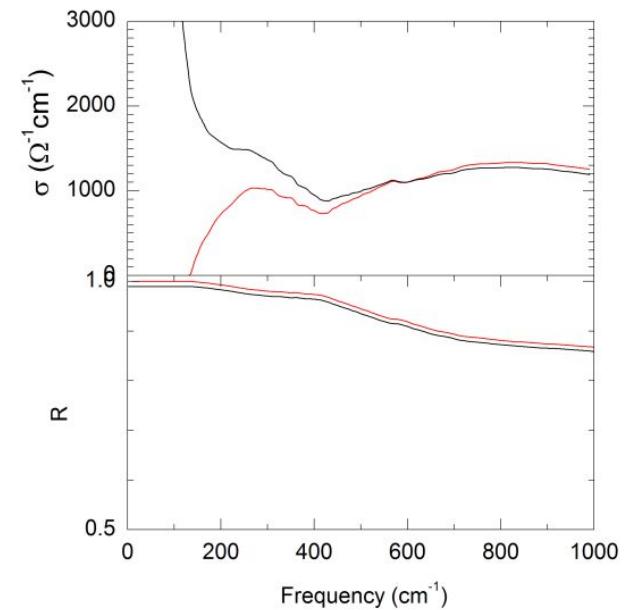
## KK analysis: errors

$$R \cong 0, R \cong 1$$

$$n = \frac{1+r}{1-r}$$



Semiconductor

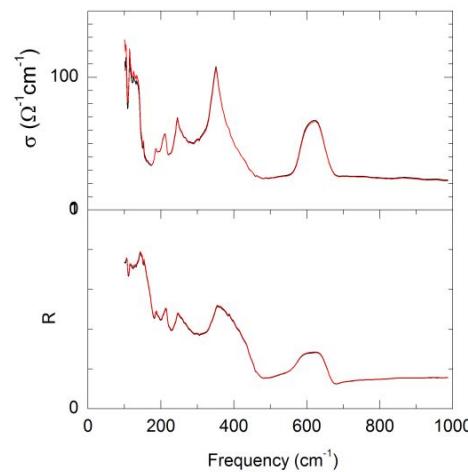


Superconductor



# KK analysis: procedure

1.  $\varepsilon''$  or  $\sigma$  (any extrapolation)
2. starting parameters (Drude-Lorentz  $\omega_p$ ,  $\omega_0$ ,  $\gamma$ )
3. fit to **measured** quantity (reflectance)
4. KK transformation with extrapolation from model frequency range: only **measured** range



# Ellipsometry

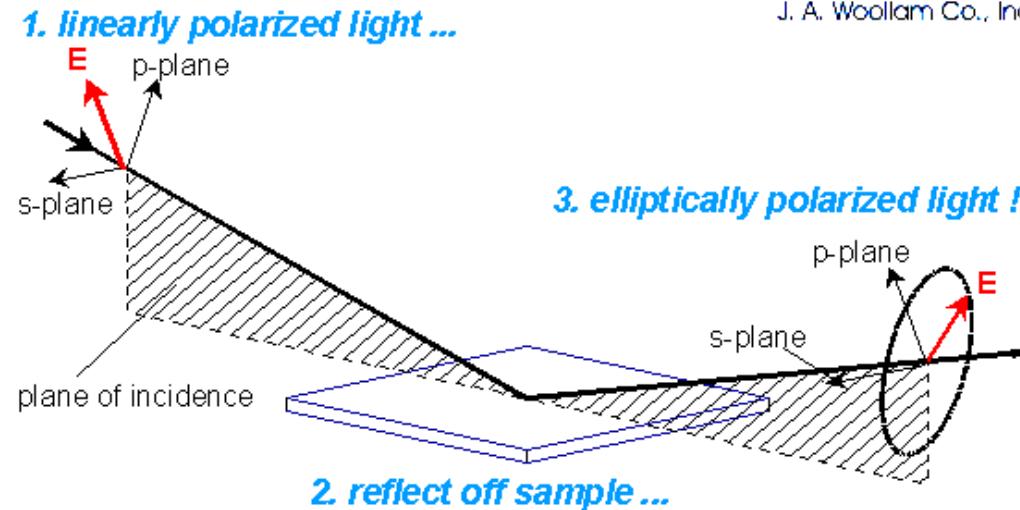
Fried Miklós  
Lohner Tivadar    MTA EK MFA  
Petrik Péter  
SOPRA →Semilab

**Snell's law:**

**(Snellius – Descartes-törvény:**

**Snellius - Gesetz: )**

$$n_a \sin \varphi_a = n_b \sin \varphi_b$$



J. A. Woollam Co., Inc

**From the boundary conditions of**

**Maxwell's equations:**

the tangential components of E and H are continuous at the interface

making use of

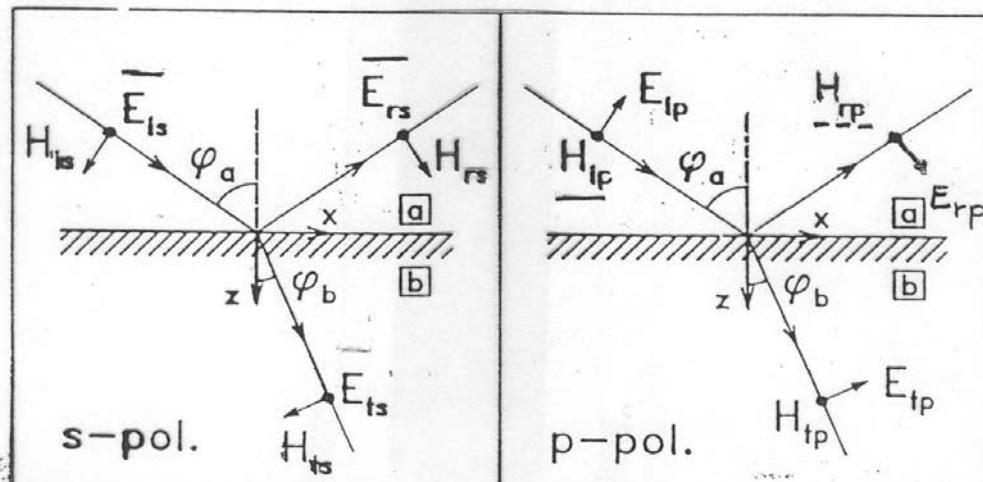
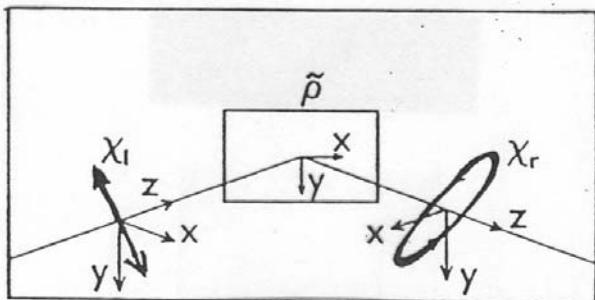
$$\mathbf{k} \times \mathbf{E} = \frac{\omega}{c} \mathbf{H}$$

$$|\mathbf{k}| |\mathbf{E}| = \frac{\omega}{c} |\mathbf{H}|$$

$$H = \frac{kc}{\omega} E = nE$$



# Fresnel's equations



$$E_{rs} + E_{is} = E_{ts}$$

$$n_a \cos \varphi_a (E_{rs} - E_{is}) = -n_b \cos \varphi_b E_{ts}$$

$$n_a (E_{ip} - E_{rp}) = n_b E_{tp}$$

$$\cos \varphi_a (E_{rp} + E_{ip}) = \cos \varphi_b E_{tp}$$

**Fresnel coefficients:**

$$r_p = \frac{E_{rp}}{E_{ip}} \quad r_s = \frac{E_{rs}}{E_{is}} \quad t_p = \frac{E_{tp}}{E_{ip}} \quad t_s = \frac{E_{ts}}{E_{is}}$$

$$r_p = |r_p| e^{i\theta_{rp}} \quad r_s = |r_s| e^{i\theta_{rs}}$$

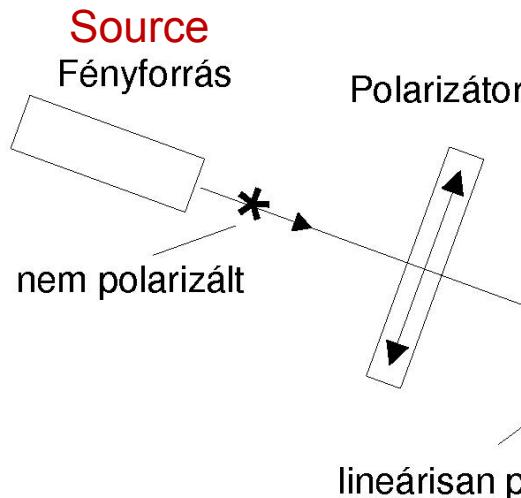
$$R = r^* r = |r|^2$$

$$r_p, r_s, t_p, t_s = f(n_a, n_b, \varphi_a, \varphi_b)$$

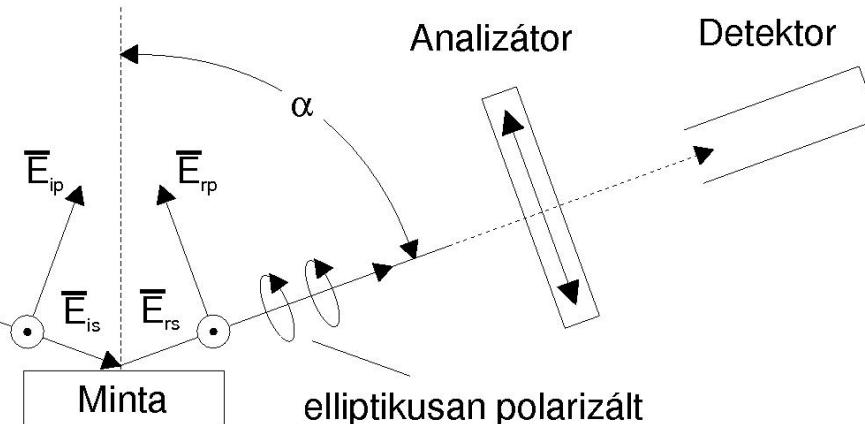


# Measured quantities

Incident light: linear polarization



Reflected light: elliptical polarization



$$\rho = \frac{r_p}{r_s} = \tan \Psi e^{i\Delta}$$

Ellipsometric angles:

Sample

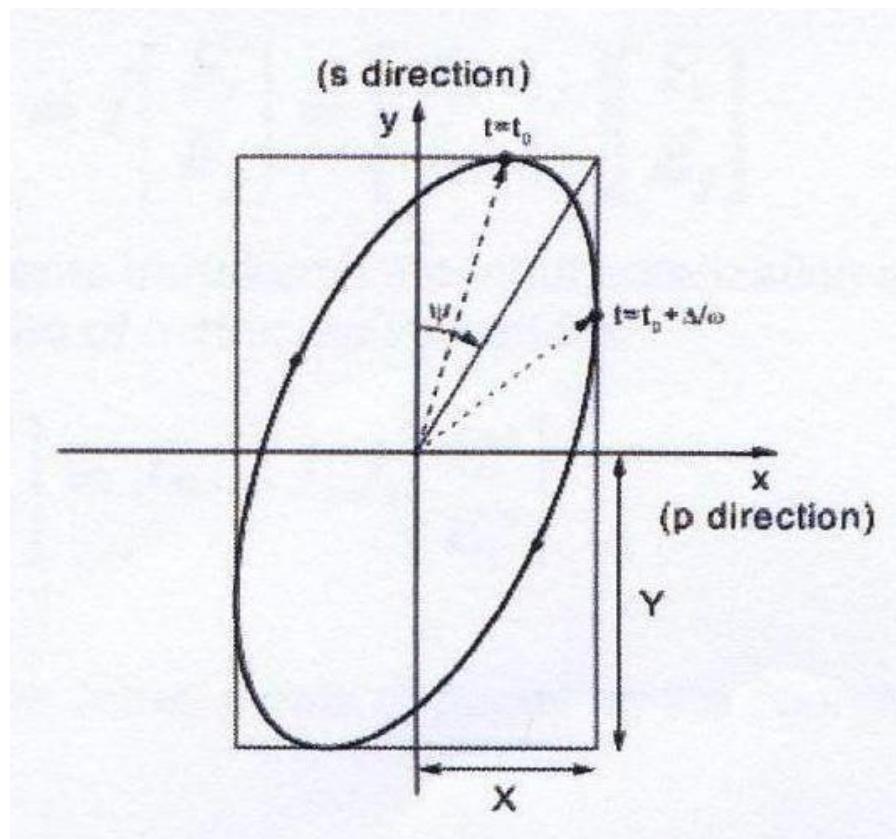
$$\tan \Psi = \left| \frac{r_p}{r_s} \right| \quad \Delta = \theta_{rp} - \theta_{rs}$$

Spectroscopic ellipsometry:  $\Psi(\omega), \Delta(\omega)$

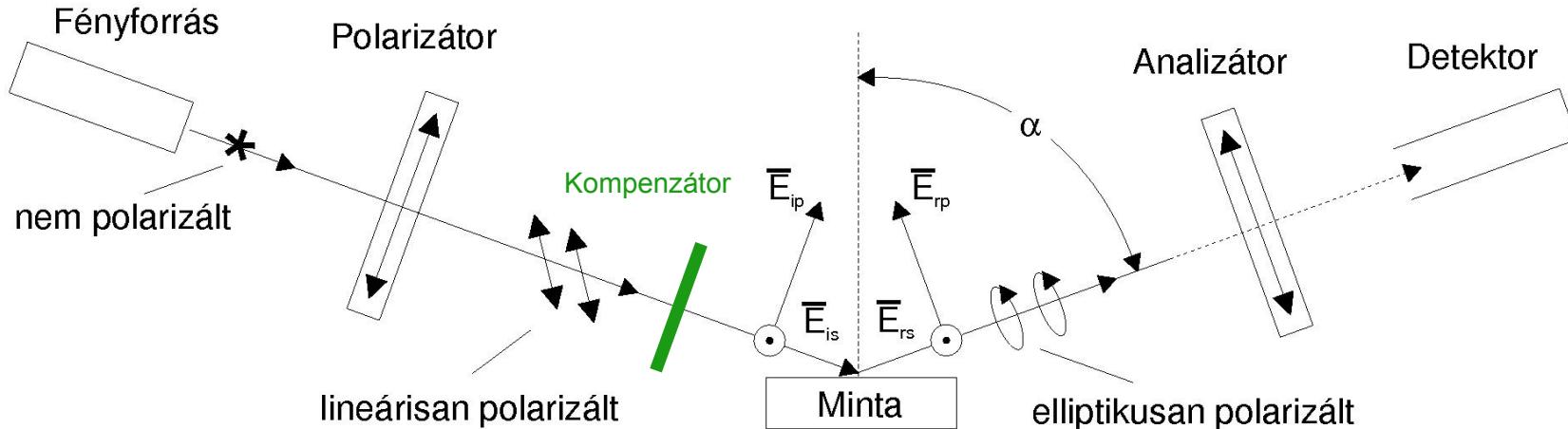


# Ellipsometric angles

Forrás: Tamáska István, 2009



# Measurement: rotating analyzer setup



Polarizer: fixed angle (P)  
 Analyzer rotating: A(t)

$$I_{\text{det}}(A) = 1 + \frac{|\rho|^2 \cos^2 P - \sin^2 P}{|\rho|^2 \cos^2 P + \sin^2 P} \cos 2A + \frac{\text{Re}(\rho) \sin 2P}{|\rho|^2 \cos^2 P + \sin^2 P} \sin 2A$$

$$I_{\text{det}}(A_i) \sim 1 + \alpha(\rho, P) \cos 2A_i + \beta(\rho, P) \sin 2A_i$$

$$\tan \Psi = \sqrt{\frac{1+\alpha}{1-\alpha}} \tan P \quad \cos \Delta = \frac{\beta}{\sqrt{1-\alpha^2}}$$



## Evaluation: Isotropic, infinite two-phase model

$$n_b^2 = \varepsilon_r(\text{sample}) = n_a^2 \left( \sin^2 \varphi_a + \frac{(1-\rho)^2}{(1+\rho)^2} \sin^2 \varphi_a \tan^2 \varphi_a \right)$$

$$\rho = \rho' + i\rho''$$

If  $n_a = 1$ :

$$\varepsilon_r' = \sin^2 \varphi_a + \frac{(1-\rho^2)^2 - 4(\rho'')^2}{1+\rho^2 + 2(\rho')^2} \sin^2 \varphi_a \tan^2 \varphi_a$$

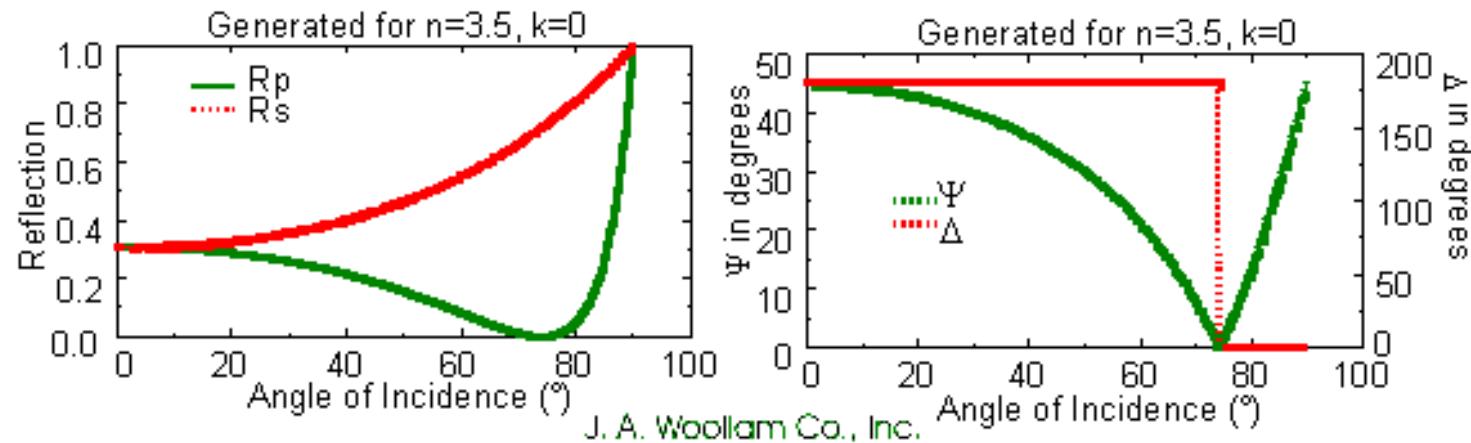
$$\varepsilon_r'' = -\frac{4\rho''(1-\rho^2)}{1+\rho^2 + 2(\rho')^2} \sin^2 \varphi_a \tan^2 \varphi_a$$

$$\varepsilon_r'' > 0 \Rightarrow \rho'' = \tan \Psi \sin \Delta < 0 \Rightarrow -\pi < \Delta < 0$$

**convention! (measured quantity:  $\cos \Delta$ )**



# Accuracy of measurement

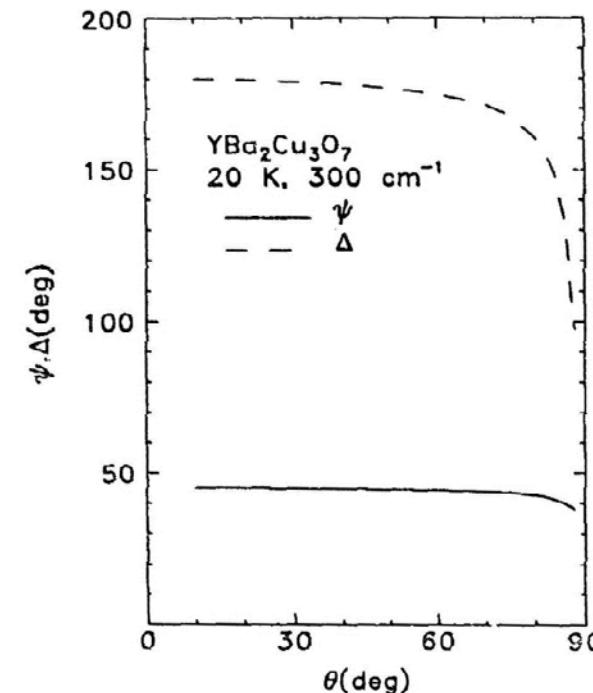
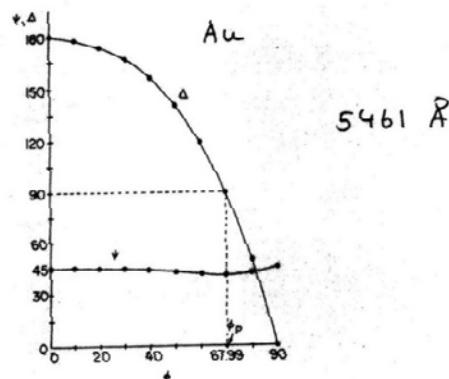
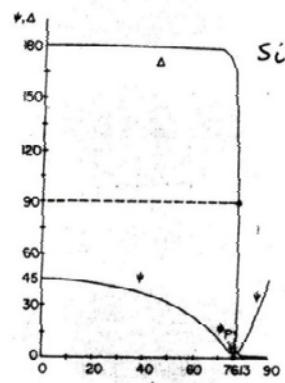
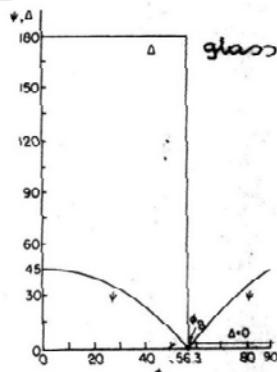


**Brewster angle:**  $r_p=0$        $\Psi = 0, \Delta = \frac{\pi}{2}$   
 $\tan \varphi_a(B) = n_b/n_a$

**$\cos \Delta$**  is the measured quantity – optimal range is where it is the most sensitive to  $\Delta$   
however, sensitivity to angle of incidence is also large there  
sensitivity of  $\Psi$  to angle is small around minimum



## Dependence on angle of incidence



R.M.A. Azzam, N.M. Bashara: Ellipsometry and Polarized Light.  
North-Holland, Amsterdam, 1977

$\text{YBa}_2\text{Cu}_3\text{O}_7$  20 K

K.Kamarás, D.van der Marel, C.C.Homes, T.Timusk:  
*Physica C* **235**, 1085 (1994)



# **Advantages - disadvantages**

## **Advantages:**

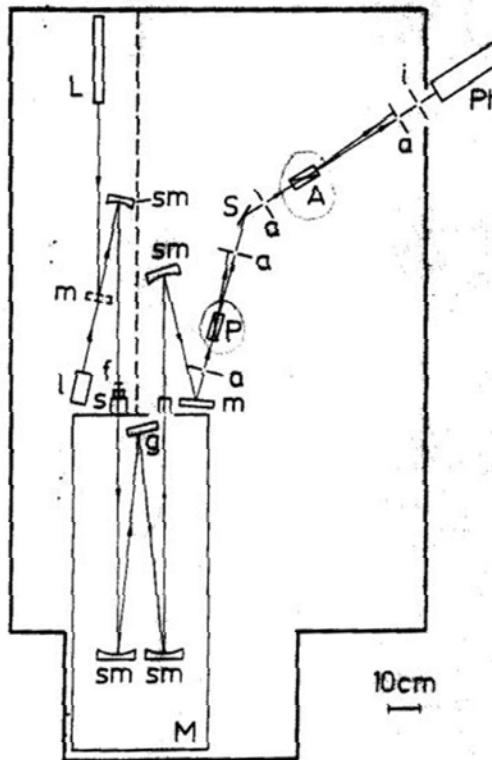
- direct determination of complex dielectric function (with appropriate model)
- no reference needed
- scattered light, small surface discontinuities cause small errors
- non-destructive
- remote sensing possible (visible range)

## **Disadvantages:**

- large angle of incidence – large light spot, large sample area required
- evaluation complicated
- many parameters of the sample have to be known beforehand.



# Experimental setup



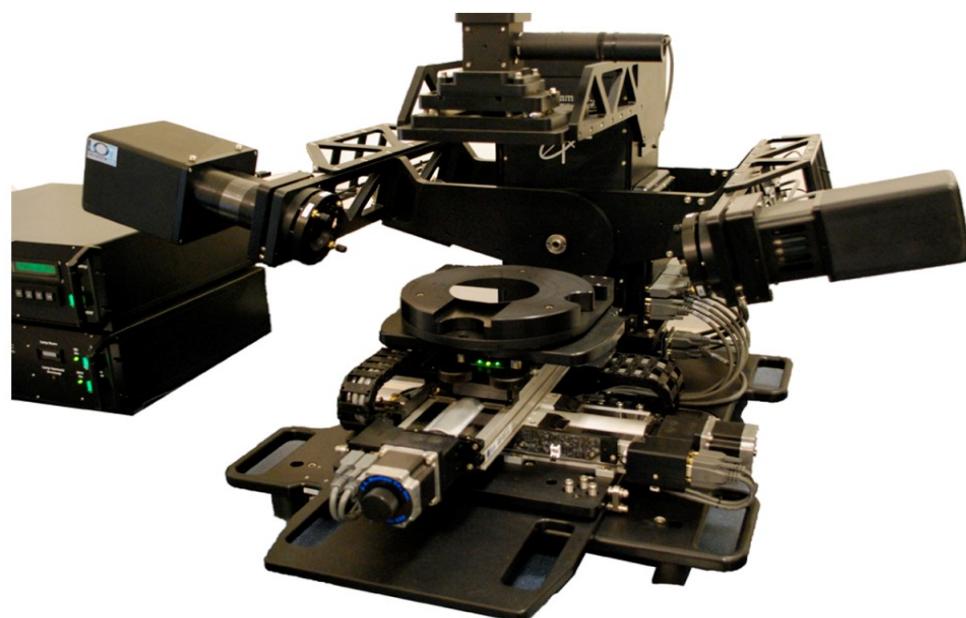
**UV-VIS ellipsometer**  
(MPI Stuttgart)

$$I(A, \omega) \Rightarrow \Psi, \Delta(\omega) \Rightarrow \langle \epsilon \rangle(\omega)$$

**Pseudodielectric function  $\langle \epsilon \rangle$ :**  
*approximation calculated with isotropic two-phase model  
independent of angle of incidence!!!!  
can be used for routine tasks with appropriate calibration*



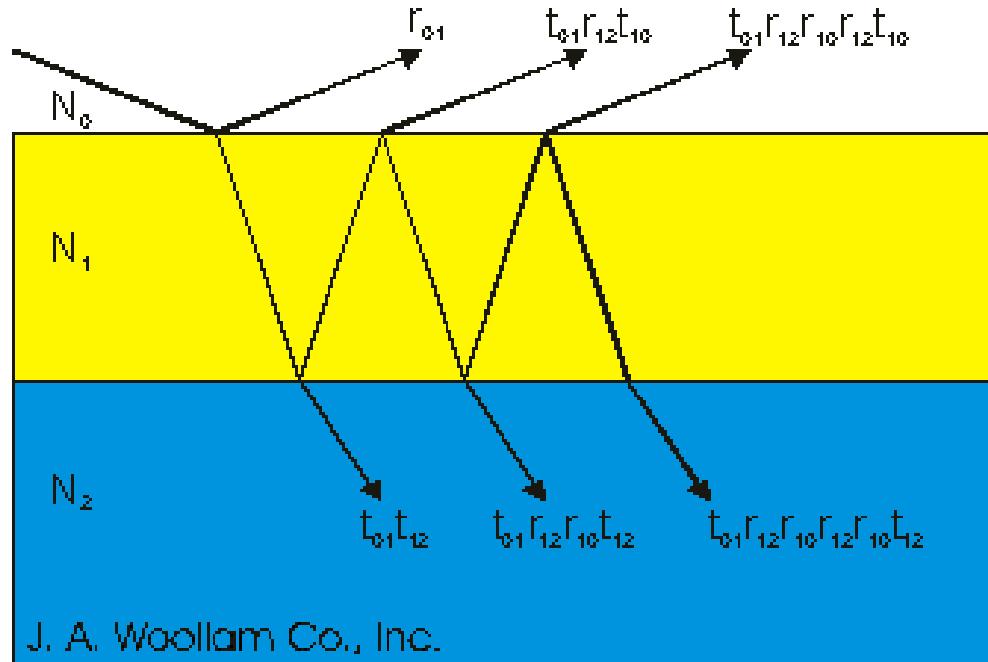
## Modern ellipsometer



**Woollam M2000DI – (MFA)**  
Rotating compensator  
spectroscopic ellipsometer  
range: **190-1700 nm**  
minimum focus spot **0.15 mm**



## Evaluation: multilayer systems



$$\rho(\varphi_a, \varepsilon_b, d_b, \varepsilon_c, d_c \dots)$$

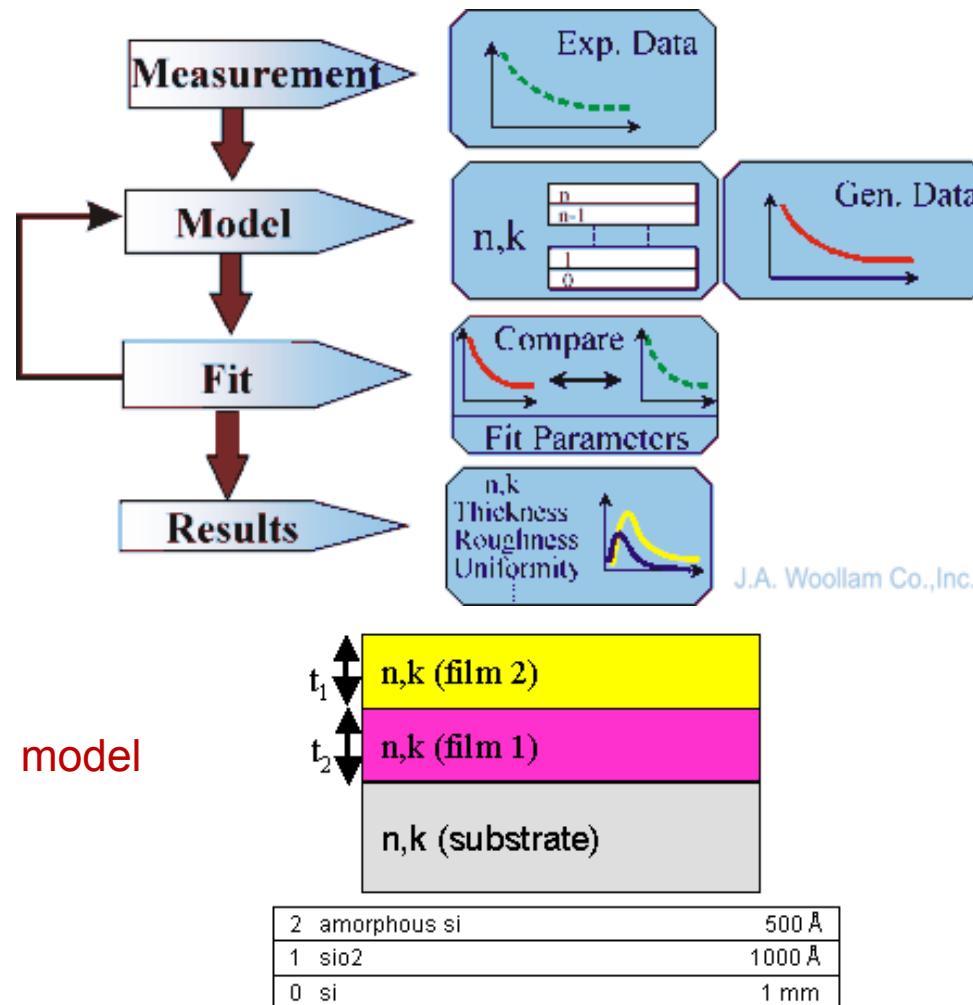
knowing  $(n-2)$  parameters,  
any two unknown quantities  
can be determined  
(e.g. thickness)

[www.jawoollam.com](http://www.jawoollam.com)



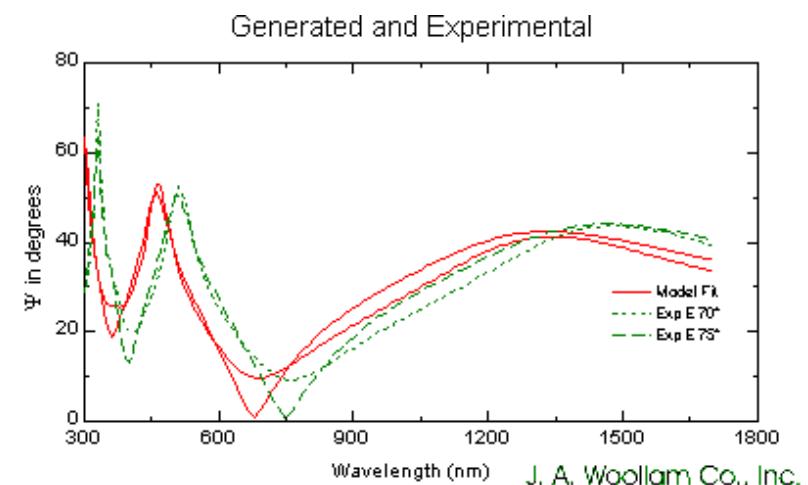
# Fitting procedures

www.jawoollam.com



If  $n(\omega)$  is known, more than two parameters can be fitted

more angles of incidence – more information



# Sensitivity of ellipsometry

$\Psi, \Delta = 0.01 - 0.02^\circ$        $\rightarrow 0.01\text{nm}$  sensitivity on layer thickness

d (nm)	$\Delta$	$\Psi$
0	179.257	10.448
0.1	178.957	10.448
0.2	178.657	10.449
0.3	178.356	10.450
0.4	178.056	10.451
0.5	177.756	10.453
1	176.257	10.462

Precise calibration (e.g. angle of incidence) is crucial!

**Technology:** process monitoring  
process control

Source: Tamáska István, 2009



# Application

- quick determination of dielectric function
- thickness measurement, technology control
- investigation of distribution in layered systems  
(comparison with model calculations)
- ideal for semiconductors, multilayers
- small sensitivity in case of transparent and strongly absorbing samples

*“Kramers-Kronig transformation is arbitrary –  
ellipsometry gives directly the dielectric function”*

**What's wrong with this sentence?**



# Combination of ellipsometry and Kramers-Kronig analysis

K. Kamarás, K.-L.Barth, F.Keilmann, R.Henn, M.Reedyk, C.Thomsen,  
M.Cardona, J.Kircher, P.L.Richards, J.-L.Stehlé:  
J. Appl. Phys. **78**, 1235 (1995)

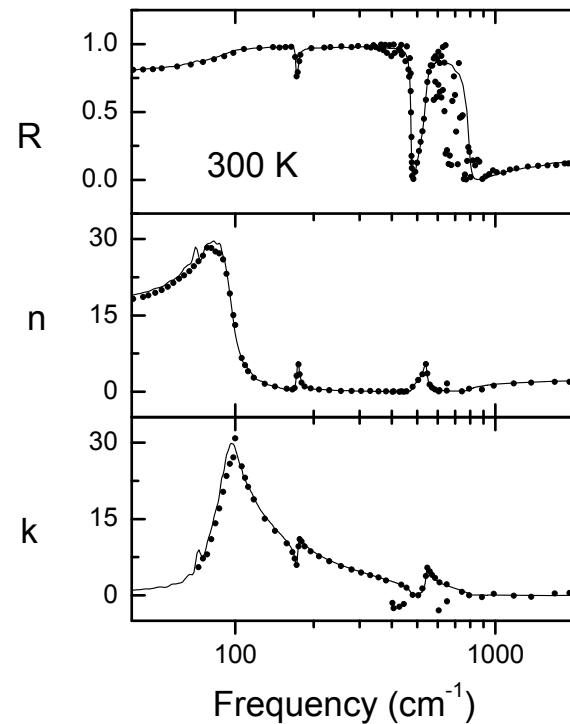


Fig. 1. Kamaras et al.

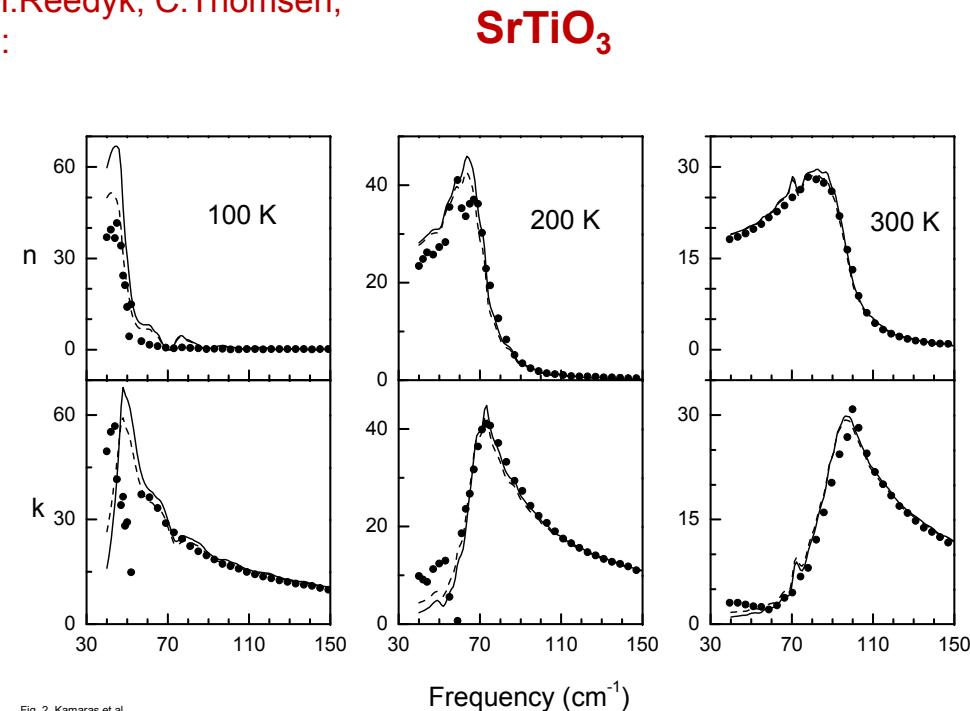


Fig. 2. Kamaras et al.

scaling of normal-incidence reflectance to ellipsometry  
low-frequency extrapolations depend on slope of curve



## Take-home message

- Basics of ellipsometry: illumination of sample with linearly polarized light under finite angle; analyzing polarization state of reflected (elliptically polarized) light

- Measured quantity: ratio of Fresnel coefficients  $\rho = \frac{r_p}{r_s} = \tan \Psi e^{i\Delta}$
- Ellipsometric angles  $\psi, \Delta$  depend on sample dielectric function and angle of incidence
- Pseudodielectric function (isotropic, infinite, two-phase model)
- Multilayer systems: any 2 parameters can be determined when the others are known (mostly thickness of known materials)
- Modeling, process control, remote sensing



# Összefoglalás

- Ellipszometria alapjai: minta megvilágítása lineárisan polarizált fénnel véges beesési szöggel ; a visszavert (elliptikusan polarizált) fény analizálása

- Mért mennyiség: Fresnel-együtthatók aránya 
$$\rho = \frac{r_p}{r_s} = \tan \Psi e^{i\Delta}$$
- A  $\Psi$ ,  $\Delta$  ellipszometrikus szögek a minta dielektromos függvényétől és a beesési szögtől függenek
- Pszeudodielektronos függvény (izotrop, végtelen, kétfázisú modell)
- Többrétegű rendszerek: bármely 2 paraméter meghatározható, ha a többi ismert (legtöbbször ismert anyagokból álló rétegek vastagsága)
- Modellezés, folyamatirányítás, távoli érzékelés



# Infrared spectroscopy



# Damped harmonic oscillator

electron of charge  $-e$ , mass  $m$ , in the field of a nucleus of infinite mass

$$\frac{md^2\mathbf{r}}{dt^2} + m\gamma \frac{d\mathbf{r}}{dt} + m\omega_0^2 \mathbf{r} = -e\mathbf{E}$$

Look for  $\mathbf{r}$  in the form  $\mathbf{r}_0 e^{-i\omega t}$ ;

using

$$\frac{d\mathbf{r}}{dt} = -i\omega \mathbf{r}, \frac{d^2\mathbf{r}}{dt^2} = -\omega^2 \mathbf{r}$$

$$\mathbf{r} = \frac{-e\mathbf{E}/m}{\omega_0^2 - \omega^2 - i\gamma\omega}$$

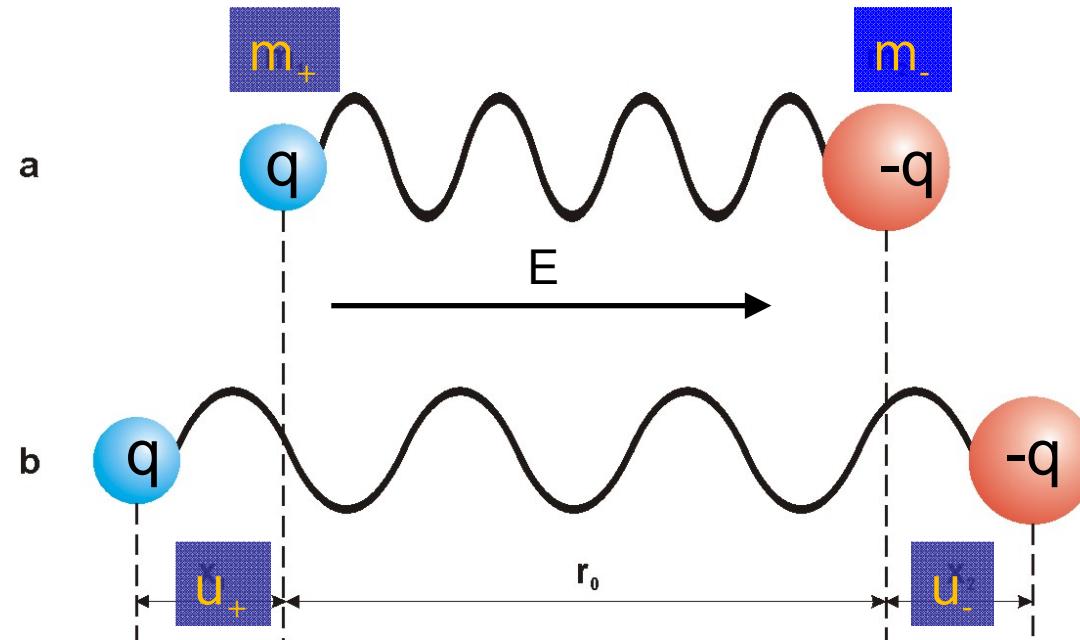
$$\mathbf{P} = \frac{1}{V} Ne\mathbf{r} = \frac{e^2 N}{m V} \frac{\mathbf{E}}{\omega_0^2 - \omega^2 - i\gamma\omega} = \chi \epsilon_0 \mathbf{E}$$

$$\chi = \frac{e^2 N}{\epsilon_0 m V} \frac{1}{\omega_0^2 - \omega^2 - i\gamma\omega}$$

$$\boxed{\epsilon_{rel} = \frac{\epsilon}{\epsilon_0} = 1 + \chi = 1 + \frac{Ne^2}{\epsilon_0 m V} \frac{1}{\omega_0^2 - \omega^2 - i\gamma\omega}}$$



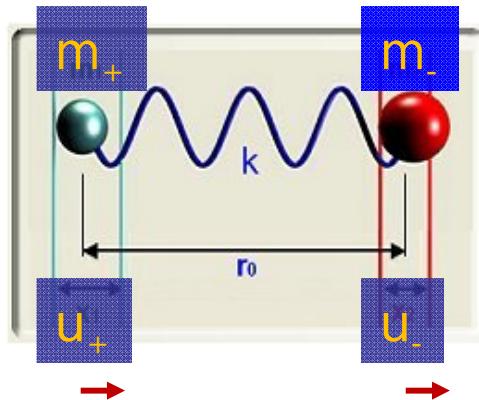
# Molecular vibrations



*Mechanical model of a vibrating diatomic molecule*



# Vibrational spectra



$$\mu = qu_+ - qu_-$$

$$^*m_- \quad m_+ \frac{d^2u}{dt^2} = -\kappa(u_+ - u_-) + qE$$

$$^*m_+ \quad m_- \frac{d^2u}{dt^2} = -\kappa(u_- - u_+) - qE$$

$$m_r = \frac{m_+ m_-}{m_+ + m_-}$$

$$r = (u_+ - u_-)$$

Introducing damping:

$$m_r \frac{d^2r}{dt^2} + m_r \gamma \frac{dr}{dt} + m_r \omega_0^2 r = qE$$

$$\omega_0 = \sqrt{\frac{\kappa}{m_r}} \quad \text{eigenfrequency}$$

$$E = E_0 e^{-i\alpha t} \longrightarrow r = r_0 e^{-i\alpha t}$$

For a system of volume V, containing N molecules:

$$\mu = \alpha E \rightarrow \chi = \frac{\alpha N}{V} \rightarrow \epsilon_{rel} = 1 + \chi$$

$$\epsilon_{rel} = 1 + \frac{Nq^2}{\epsilon_0 m_r V} \frac{1}{\omega_0^2 - \omega^2 - i\gamma\omega}$$

---


$$\frac{m_+ m_-}{m_+ + m_-} \frac{d^2(u_+ - u_-)}{dt^2} = qE + \kappa(u_- - u_+)$$



# Vibrational spectra – dielectric formalism

For extended charge distribution, an *effective charge* can be defined:

$$\mu = \left( \frac{\partial \mu}{\partial u_+} \right) u_+ + \left( \frac{\partial \mu}{\partial u_-} \right) u_- = Qr \quad Q = \left( \frac{\partial \mu}{\partial u_+} \right) = - \left( \frac{\partial \mu}{\partial u_-} \right)$$

$$\epsilon_{rel} = \epsilon_\infty + \frac{NQ^2}{\epsilon_0 m_r V} \frac{1}{\omega_0^2 - \omega^2 - i\gamma\omega} = \epsilon_\infty + \frac{\Omega^2}{\omega_0^2 - \omega^2 - i\gamma\omega}$$

$$\Omega^2 = \frac{NQ^2}{\epsilon_0 m_r V} \quad \text{oscillator strength (if } Q=0, \text{ no response)}$$

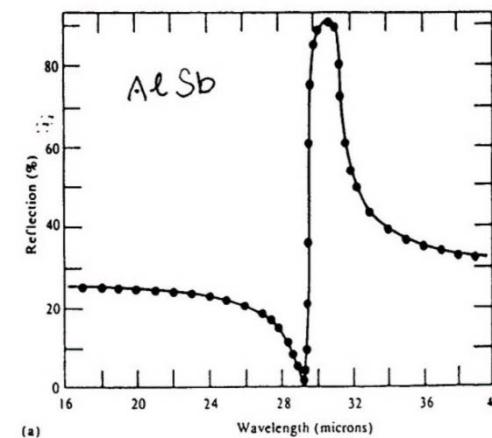
$m_r >> m_e$  and  $Q < e \rightarrow$  usually weaker transitions

Longitudinal excitation if  $\epsilon_{rel}(\omega_l) = 0$ :  $\epsilon_{rel}' = \epsilon_\infty + \frac{\Omega^2(\omega_0^2 - \omega_l^2)}{(\omega_0^2 - \omega_l^2)^2 + \gamma^2 \omega_l^2} = 0$

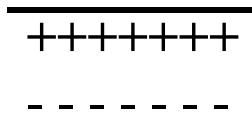
$$\text{since } \gamma \ll \omega_0, \omega_l \quad \omega_l^2 = \omega_0^2 + \frac{\Omega^2}{\epsilon_\infty}$$

$\omega_l - \omega_0$ : LO-TO splitting (intensity!)

$\omega_l = \omega_{LO}$  longitudinal optical frequency  
 $\omega_0 = \omega_{TO}$  transverse optical frequency



# Plasma oscillations



The oscillations persist in zero external field:

$$\mathbf{E}_{\text{ext}} = \mathbf{D} = \epsilon \mathbf{E} = \mathbf{0}$$

since  $\mathbf{E} \neq \mathbf{0}$  (charge separation),  $\epsilon = 0$  ( $\epsilon' = \epsilon'' = 0$ )

Plasma oscillations occur at the frequency where the condition is met, but we do **not** excite the plasma oscillations with light at normal incidence!



# (Bulk) plasmons

## Plasmons:

longitudinal oscillations of the electron gas  
cannot be excited by normal incidence  
light

they appear if  $\epsilon' = \epsilon'' = 0$

Detection of plasmons: electron energy loss spectroscopy (EELS)

EELS measures the **loss function**:

$$\text{Im}\left(-\frac{1}{\tilde{\epsilon}}\right)$$

T. Pichler, M. Knupfer, M.S. Golden, J. Fink,  
A.G. Rinzler, R.E. Smalley:  
Phys. Rev. Lett. **80**, 4729 (1998)

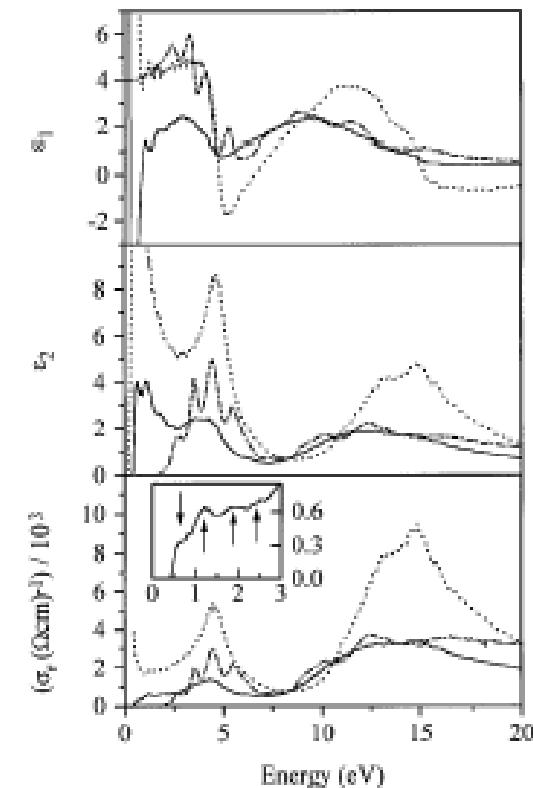


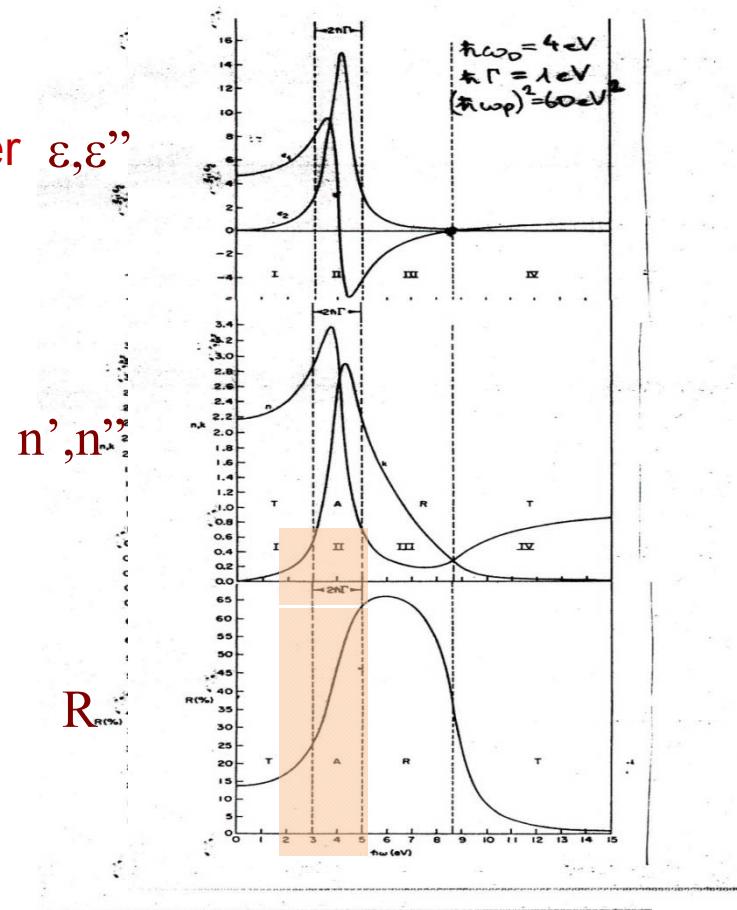
FIG. 3. The real and imaginary parts of the dielectric function (upper panels) and the real part of the optical conductivity ( $\sigma_r$ ) at low momentum transfer: SWNTs (—) at  $q = 0.1 \text{ \AA}^{-1}$ ,  $C_{60}$  (— · —) and graphite [polarized in plane (---)] at  $q = 0.15 \text{ \AA}^{-1}$ , respectively. The inset shows  $\sigma_r$  for the four lowest-lying interband transitions of SWNTs in an expanded range.



# Reststrahlen

“Reststrahlen” (residual rays) – frequency filter  $\varepsilon, \varepsilon'$

“Reststrahlen” region  $n' < 1$



# Static dielectric constant

$$\varepsilon_{rel}'(0) = \varepsilon_\infty + \frac{\Omega^2}{\omega_0^2} \quad (\text{one vibrational transition + electronic excitations})$$

$$\frac{\varepsilon_{rel}'(0)}{\varepsilon_\infty} = \frac{\omega_l^2}{\omega_0^2} = \frac{\omega_{LO}^2}{\omega_{TO}^2} \quad \text{Lyddane-Sachs-Teller relation}$$

For multiple oscillators:

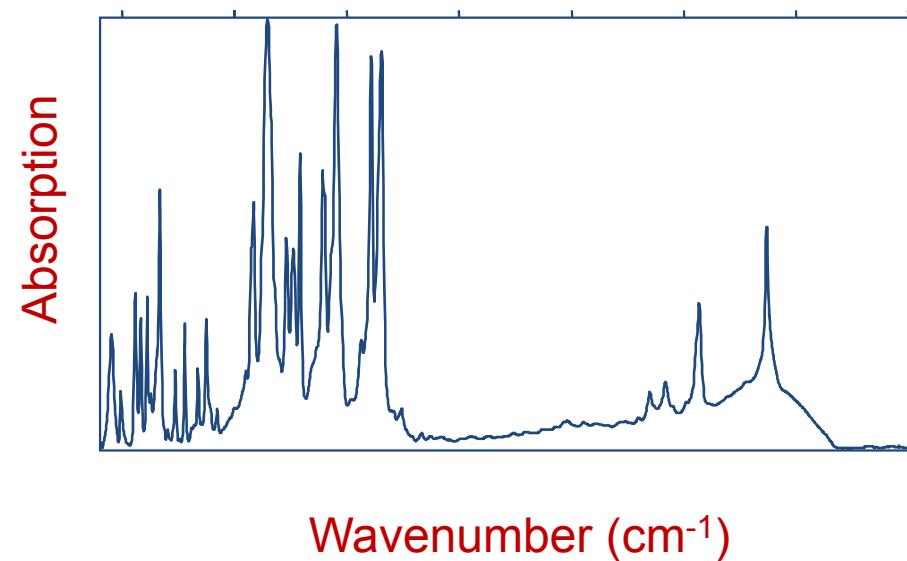
$$\frac{\varepsilon_{rel}'(0)}{\varepsilon_\infty} = \prod_i \frac{\omega_{li}^2}{\omega_{0i}^2}$$

## Application:

- is there a vibration below a given frequency? (comparison with dielectric constant)
- Estimation of high-frequency dielectric constant from vibrational spectra and static dielectric constant



# Molecular spectra



$$\alpha = \frac{2\omega n''}{c} = \frac{\varepsilon''_{rel}\omega}{n'c}$$



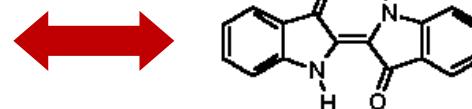
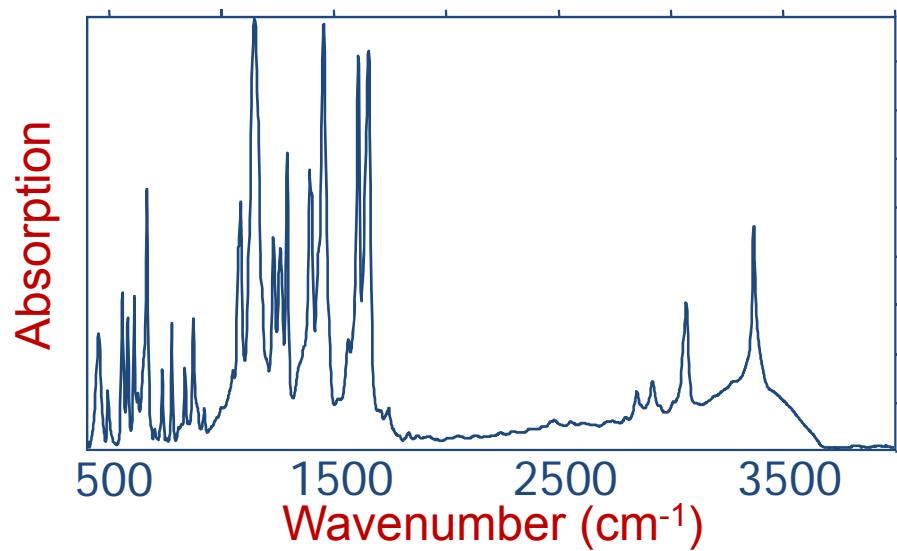
# Vibrations and spectra

Vibrations need a well-defined energy

$$\text{Frequency: } \omega_0 = \sqrt{\frac{\kappa}{m_r}}$$

$\kappa$  bond strength (spring constant)  
 $m_r$  reduced mass

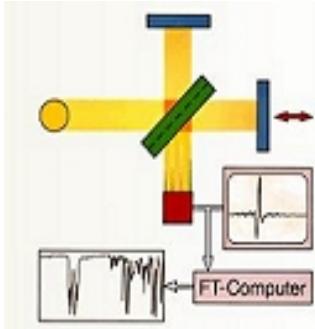
Number of lines: symmetry  
complicated structure



Qualitative analysis

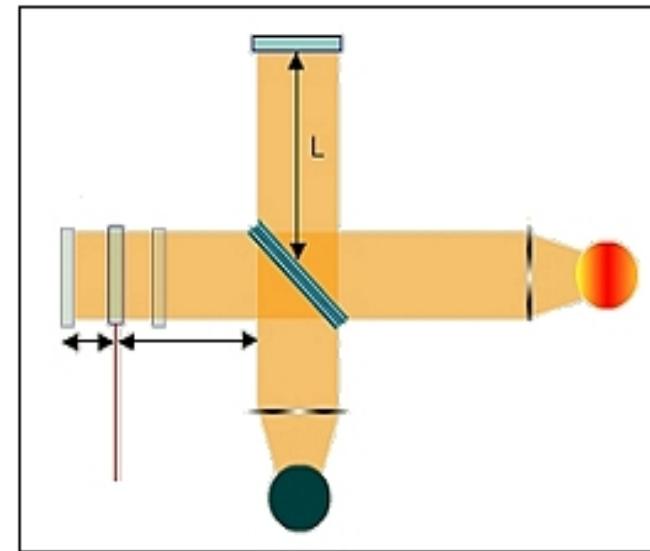


# Fourier-transformation infrared (FTIR) spectroscopy

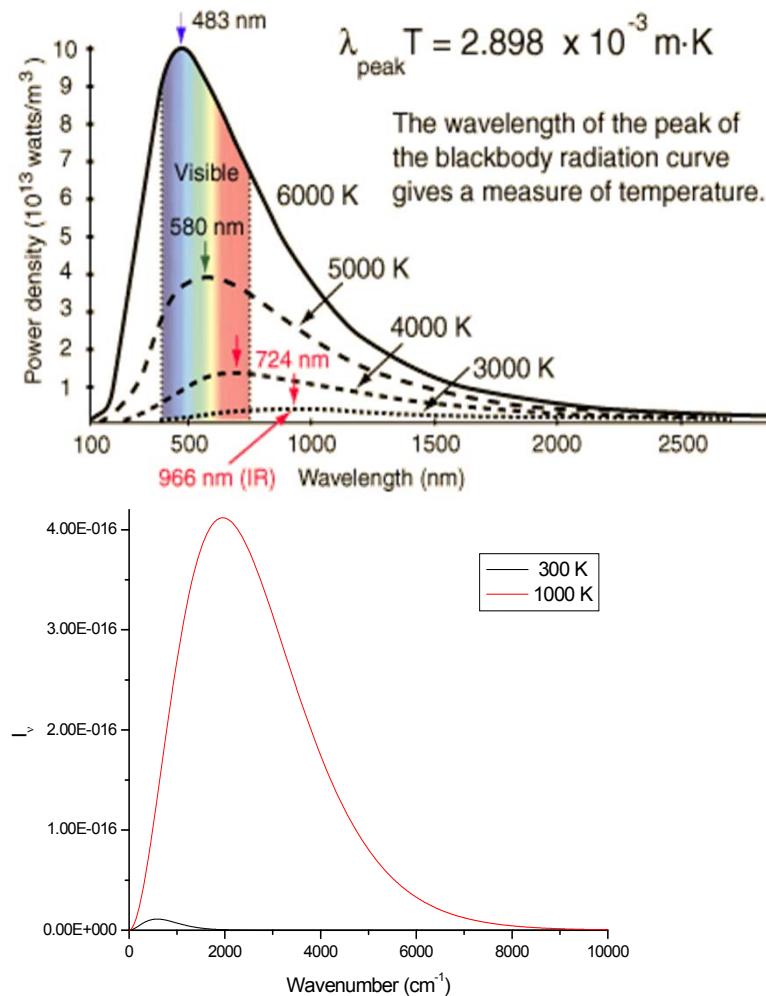


$$I(\nu^*) = \int_{-\infty}^{+\infty} I(x) \cos(2\pi\nu^* x) dx$$

$$i(\nu^*) = \Delta x \sum_{-M}^M I(m\Delta x) \cos(2\pi\nu^* m\Delta x)$$



# Why FTIR?



high temperature: intensity grows also at high frequency (short wavelength)

environmental thermal radiation cannot be filtered out

FTIR: only light getting into the interferometer will be modulated



# FTIR advantages

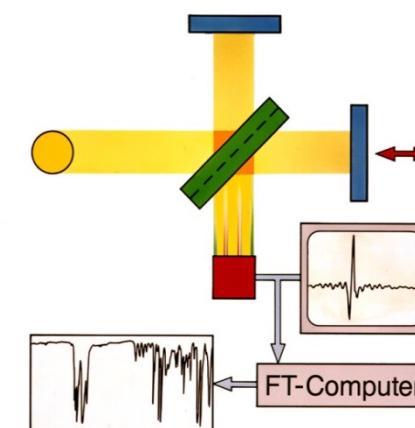
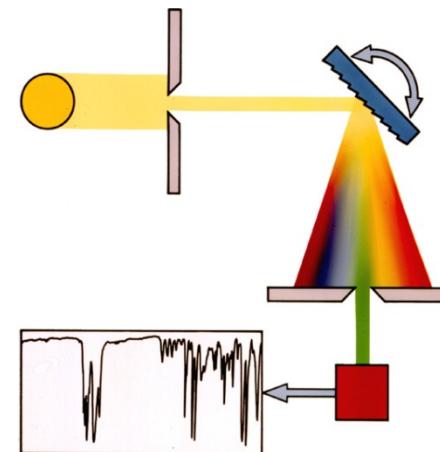
**Jacquinot's advantage:** intensity

no need for narrow slit, as in monochromators  
shape of illuminating light spot not critical  
(detector: large dynamic range!)

**Fellgett's advantage (multiplex):**

multiple frequencies in one scan  
(in dispersive systems, scan time is determined by  
the frequency range with the smallest intensity)  
signal-to-noise ratio improves with more scans  
time resolution possible on the scale of a few seconds

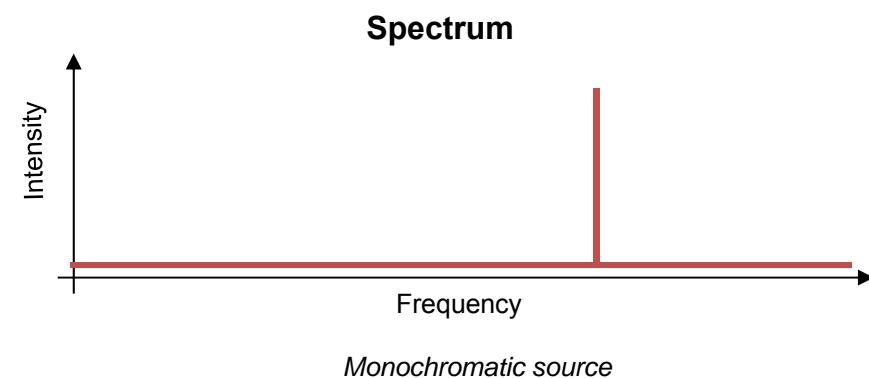
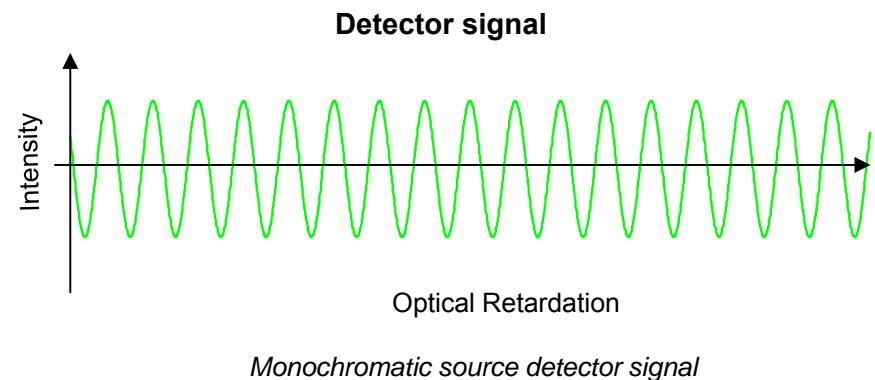
Dispersive IR spectrometer



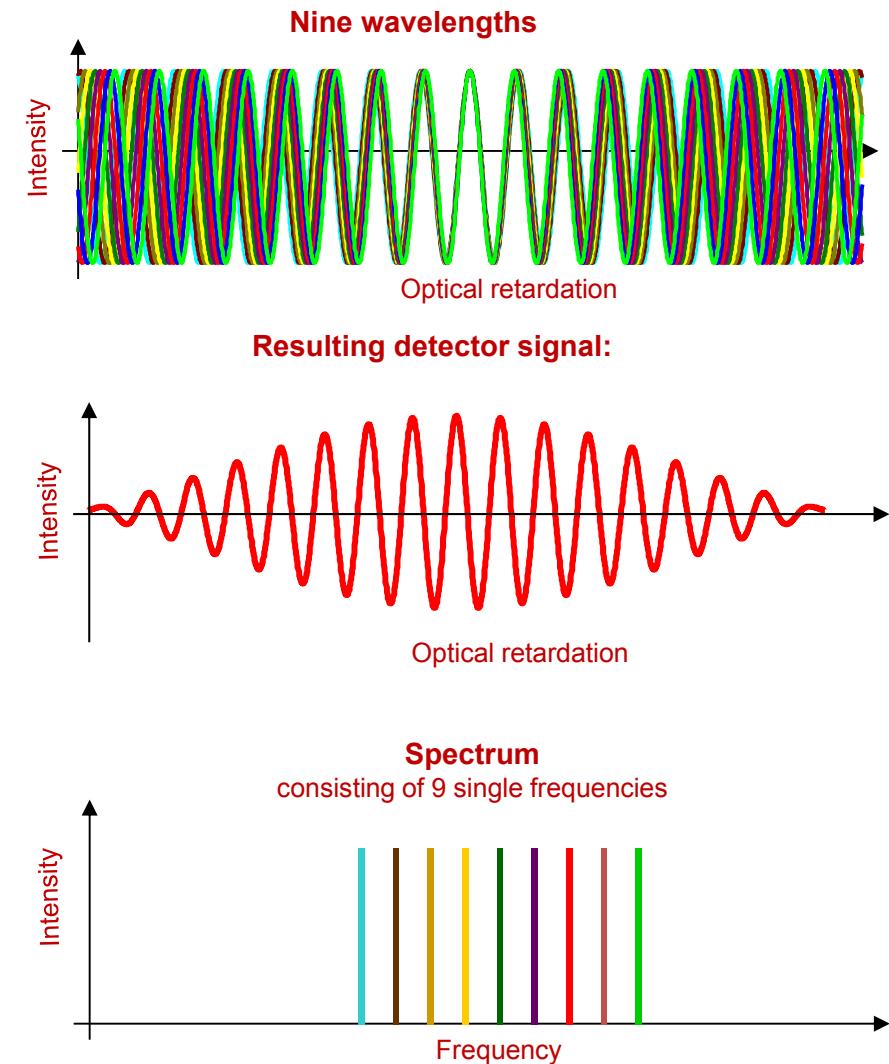
FT-IR spectrometer



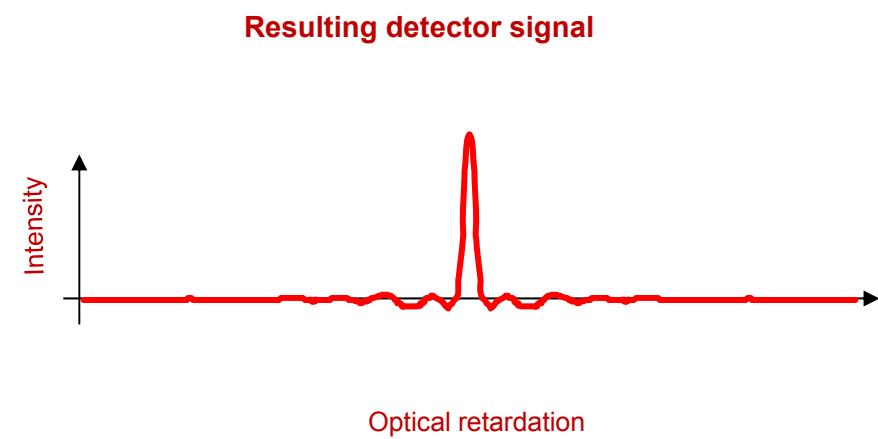
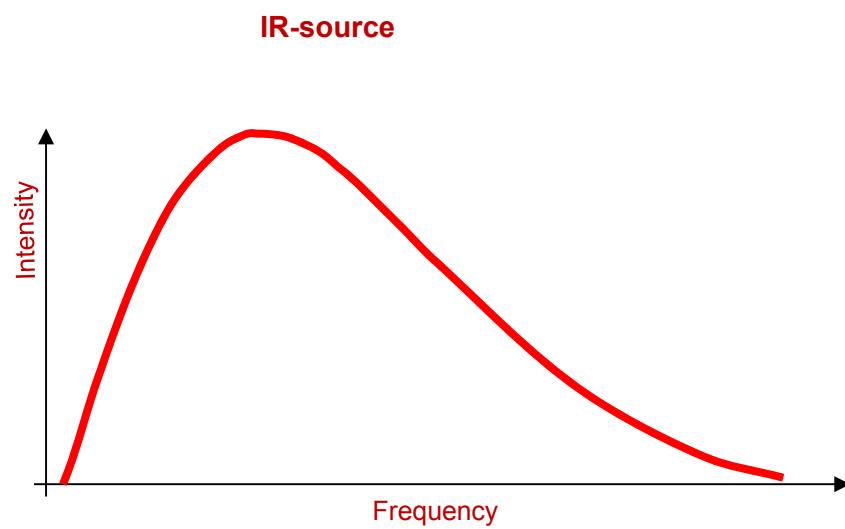
# Interferogram of a monochromatic source



# Interferogram of a polychromatic source

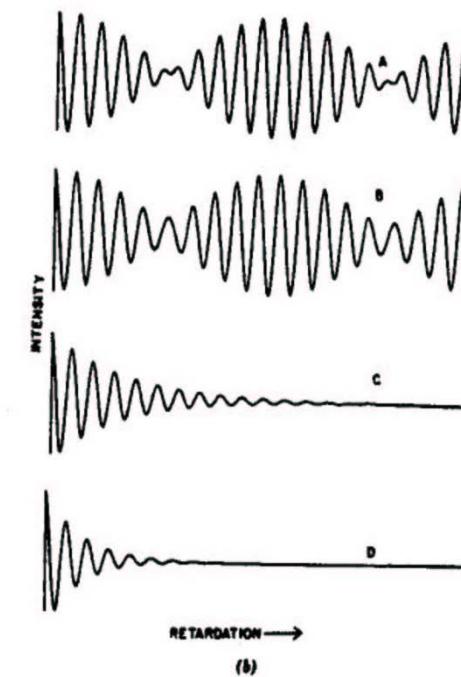
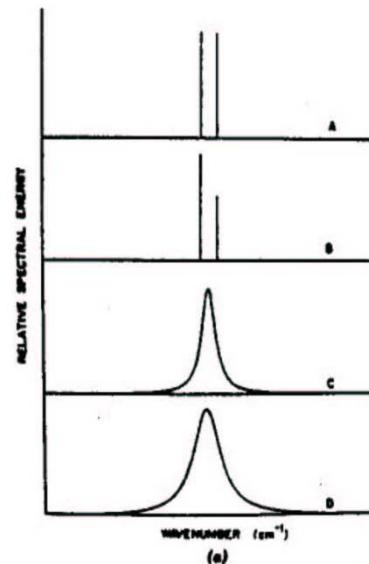


# Interferogram of a continuous source

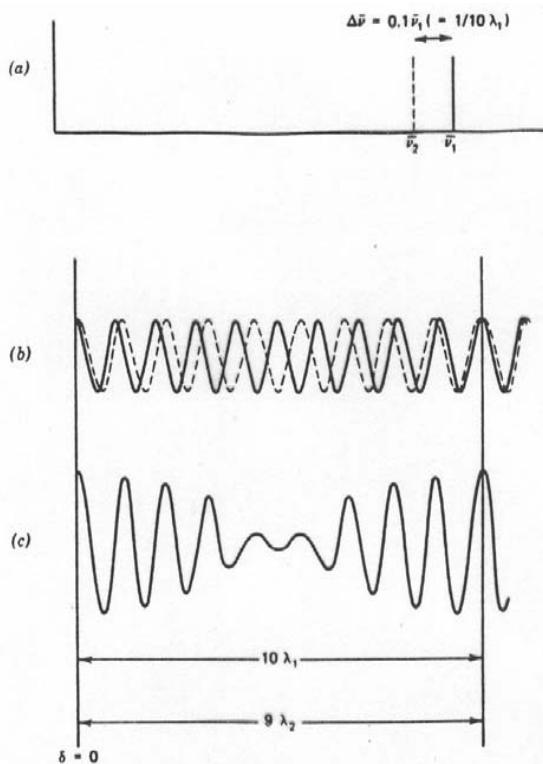


# Converting interferograms to spectra

P.R. Griffiths, J.A. de  
Haseth:  
Fourier Transform  
Infrared Spectrometry  
Wiley, 2007



# Resolution



**Fig. 1.4.** (a) Spectrum of two lines of equal intensity at wavenumbers  $\bar{\nu}_1$  (solid line) and  $\bar{\nu}_2$  (broken line) separated by  $0.1\bar{\nu}_1$ . (b) Interferogram for each spectral line shown individually as solid and broken lines, respectively. (c) Resultant interferogram with the first maximum of the beat signal at  $10/\bar{\nu}_1$ ; to resolve these two spectral lines, it is necessary to generate an optical retardation of at least this value.

$$10\lambda_1 = 9\lambda_2$$

$$\nu_2^* = 0.9\nu_1^*$$

$$\Delta\nu^* = 0.1\nu_1^*$$

$$x_{\min} = 10\lambda_1 = \frac{1}{\Delta\nu^*}$$

P.R. Griffiths, J.A. de Haseth:  
Fourier Transform Infrared Spectrometry  
Wiley, 2007

$$\Delta\nu^* = 1\text{cm}^{-1} \rightarrow x = 1\text{cm}$$

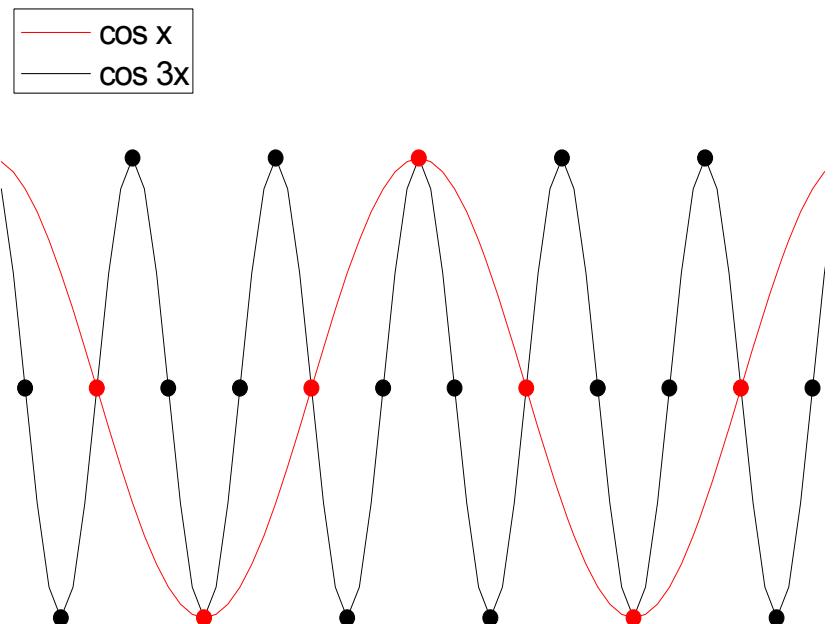
$$\Delta\nu^* = 0.01\text{cm}^{-1} \rightarrow x = 1\text{m}$$



# Frequency range

**Nyquist's theorem:** a specific frequency should be distinguished from the harmonics by using appropriate sampling frequency

**Example:**

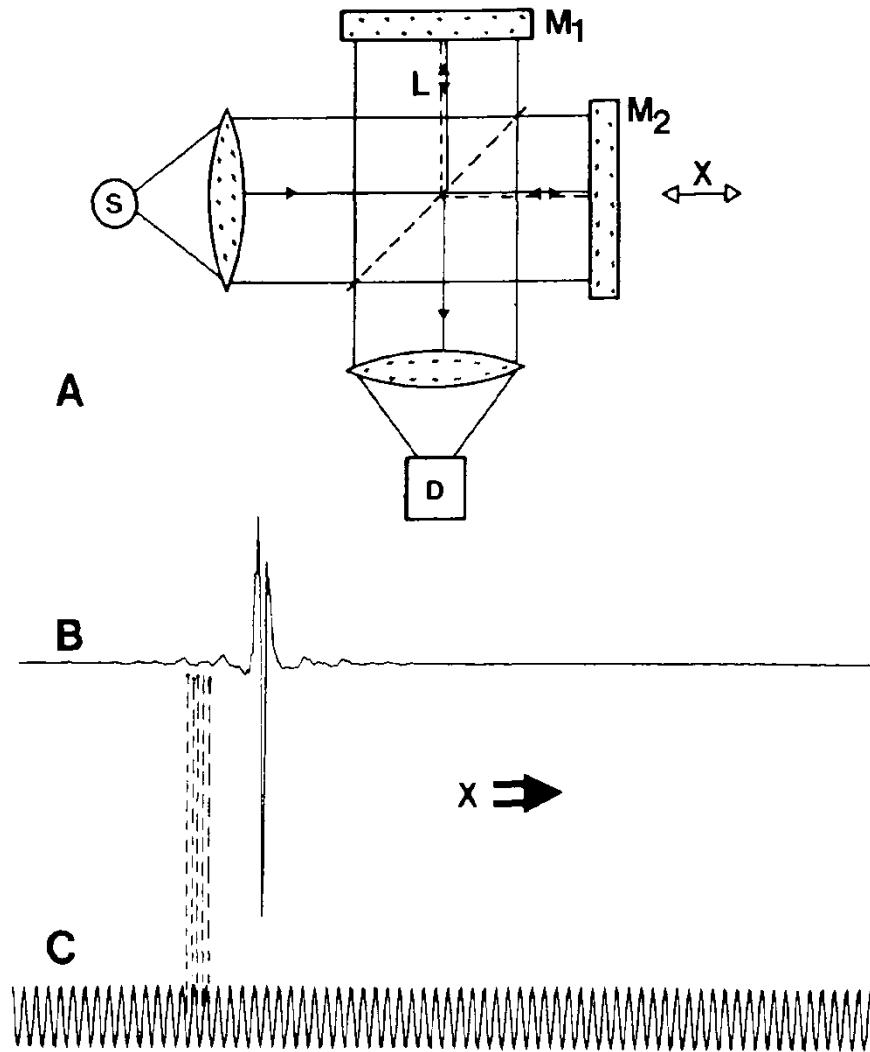


$$\nu_{\max}^* = \frac{1}{2\Delta x}$$

$$\nu_{\max} = 1000 \text{ cm}^{-1} \rightarrow \Delta x = 5 \mu\text{m}$$



# Sampling control: He-Ne laser



# Typical parameters

**Mirror velocity:** 0.5-60 mm/sec

**He-Ne laser wavelength:** 632.8 nm, wavenumber  $15800 \text{ cm}^{-1}$

**minimal distance of zero points:** 316.4 nm  $n_{\max} = 15800 \text{ cm}^{-1}$   
632.8 nm  $n_{\max} = 7900 \text{ cm}^{-1}$

**frequency of signal at detector:**  $f=2vn^*$

for  $f=1.58 \text{ mm/sec}$

$400 \text{ cm}^{-1} f=126 \text{ Hz}$

$4000 \text{ cm}^{-1} f=1260 \text{ Hz}$

