# Optical Characterization Methods

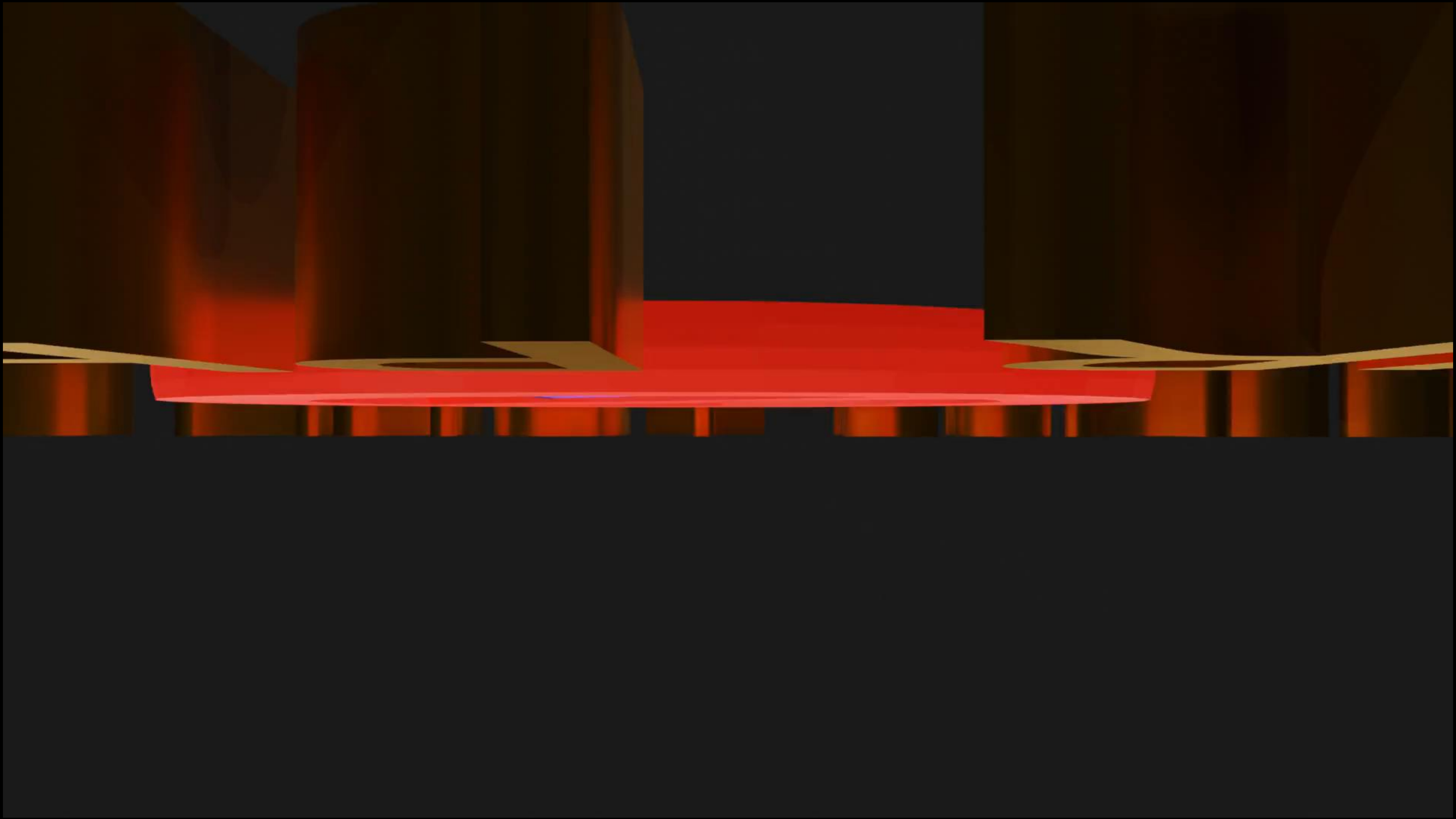
## Part I

Julio A. N. T. Soares

Materials Research Laboratory
University of Illinois at Urbana-Champaign

*go.Illinois.edu/AMC2025*

The Grainger College
of Engineering
UNIVERSITY OF ILLINOIS URBANA-CHAMPAIGN



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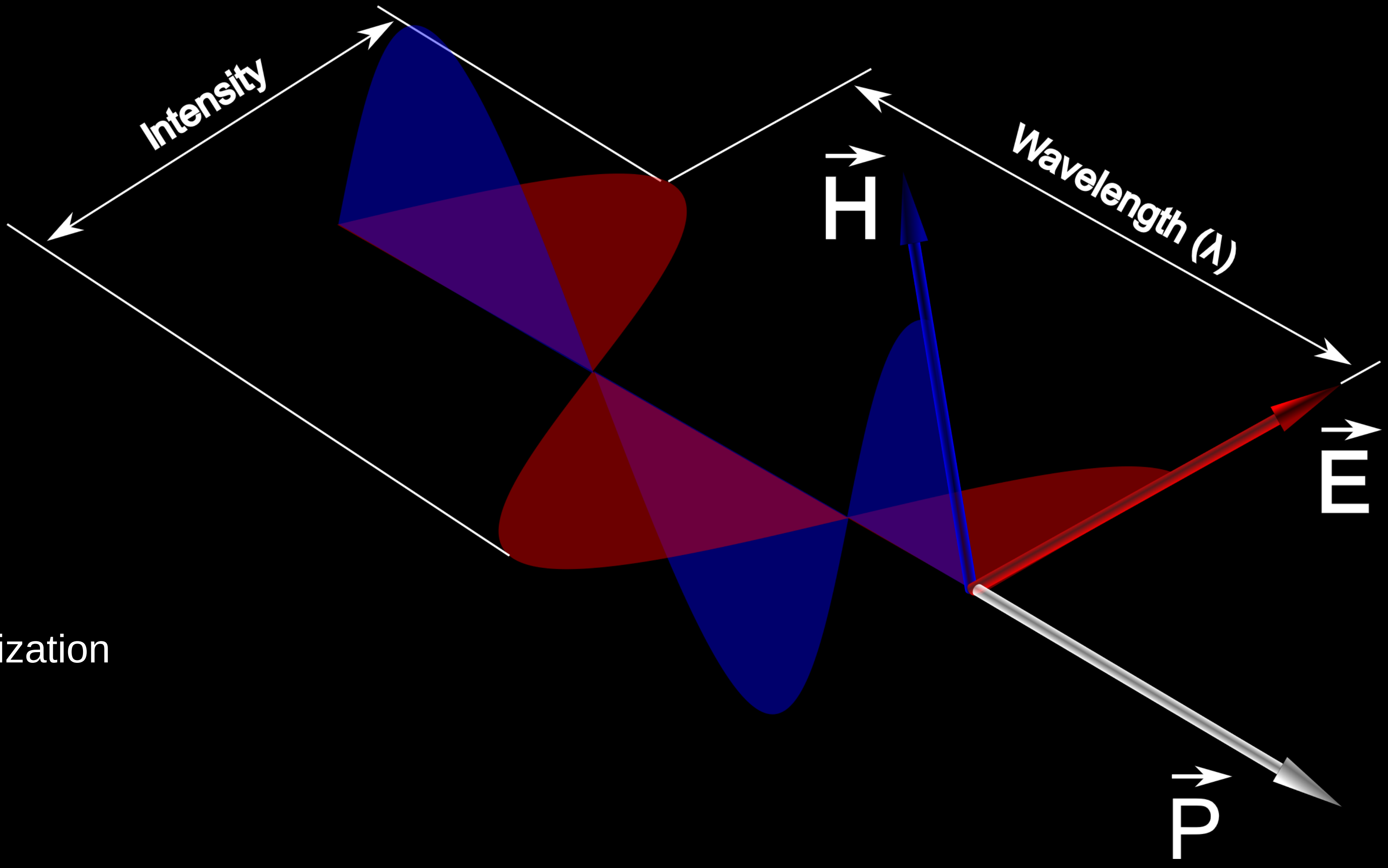
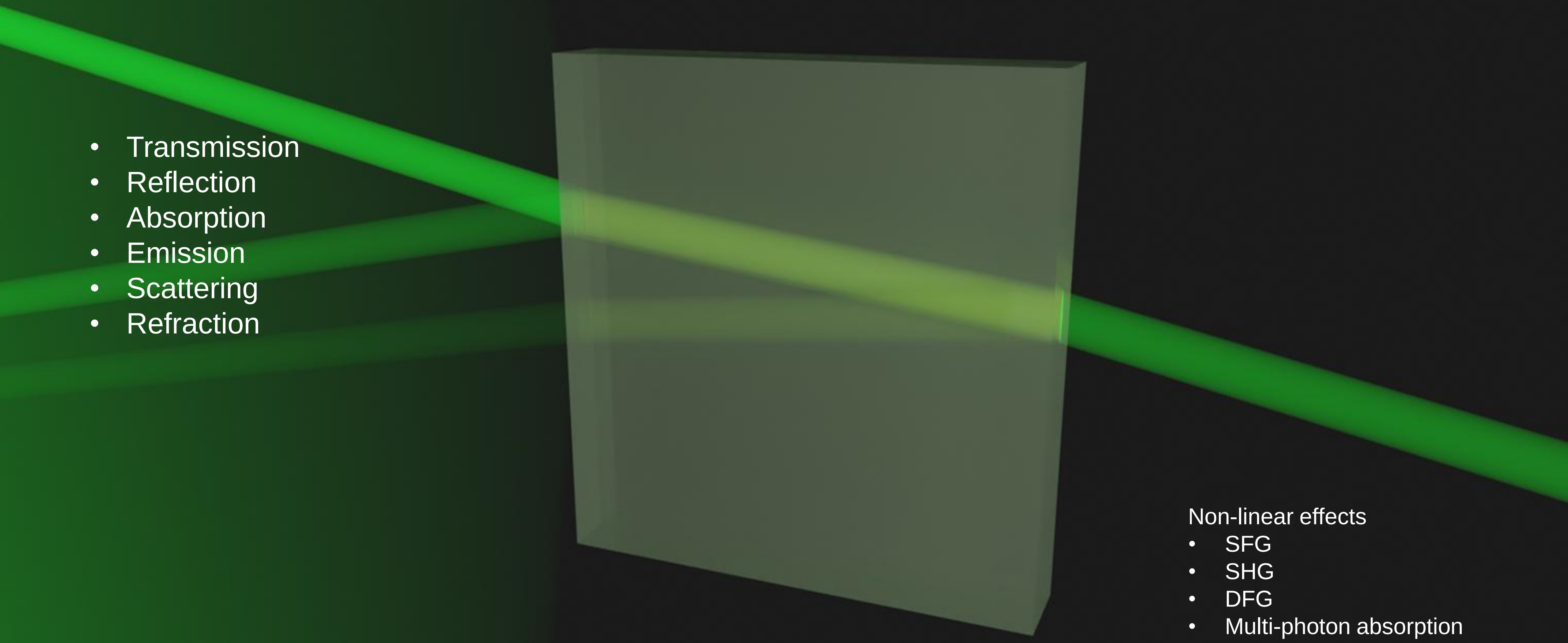
# Optical Characterization

# Light properties



- Direction of propagation
- Electric field direction or polarization
- Photon energy or wavelength
- Intensity
- Propagation speed

# Light interactions

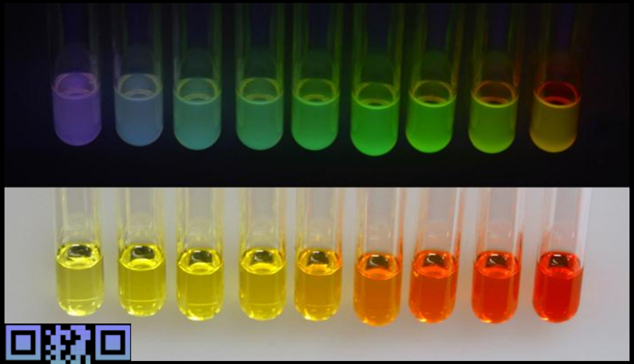


- Transmission
- Reflection
- Absorption
- Emission
- Scattering
- Refraction

Non-linear effects
- SFG
- SHG
- DFG
- Multi-photon absorption

# Light interactions with matter

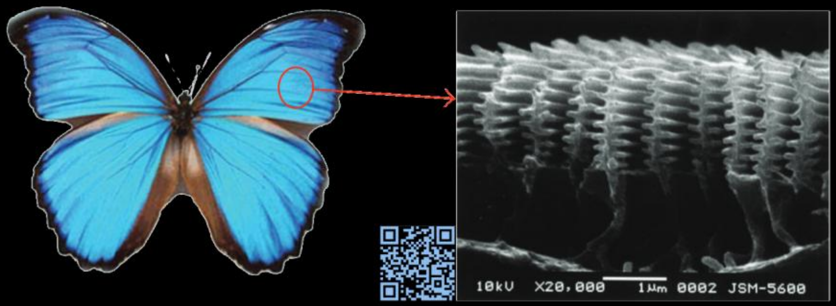
## Size



## Thickness

## Lattice structure, dopants

## Microstructure



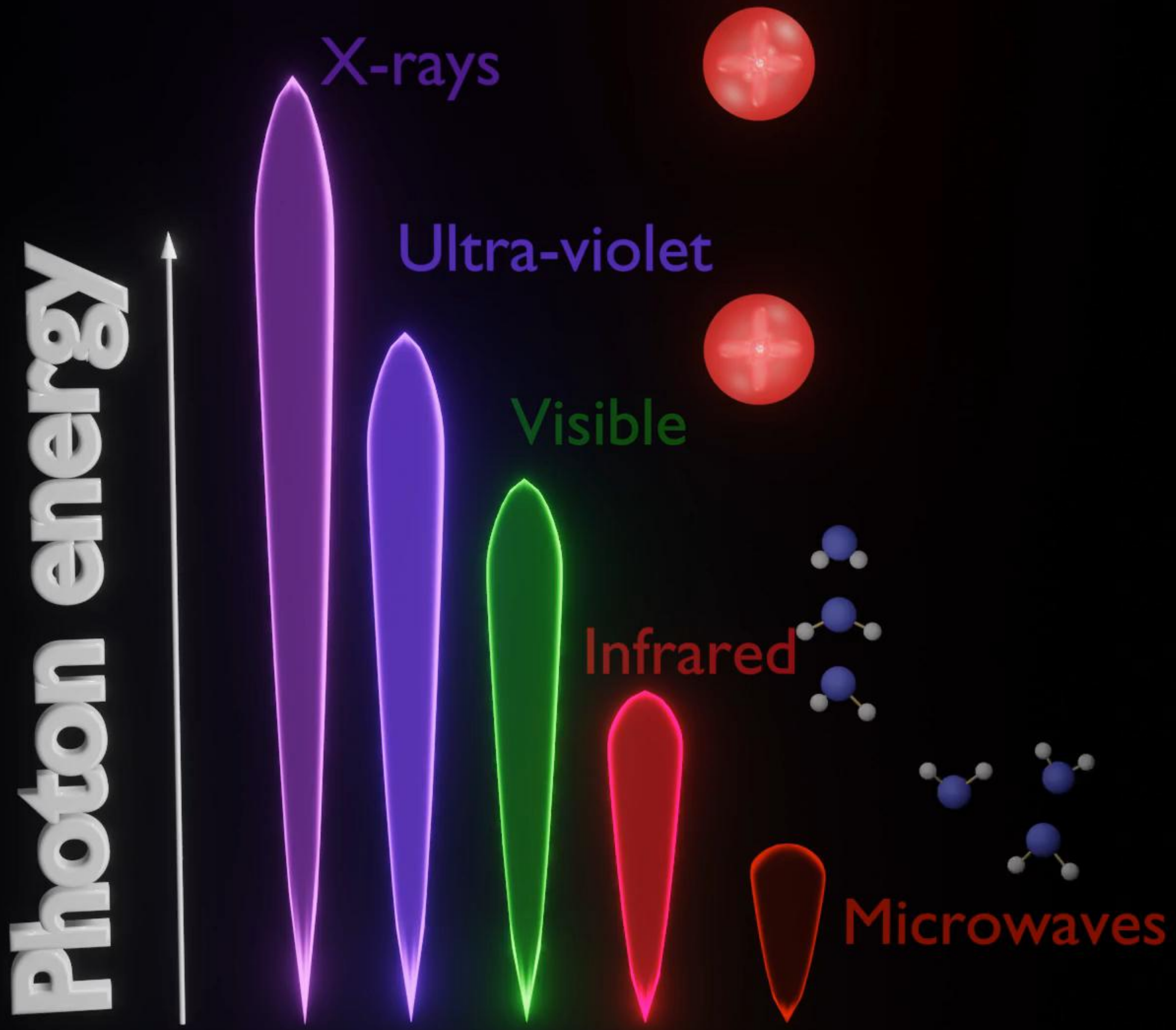
## Temperature

## Concentration

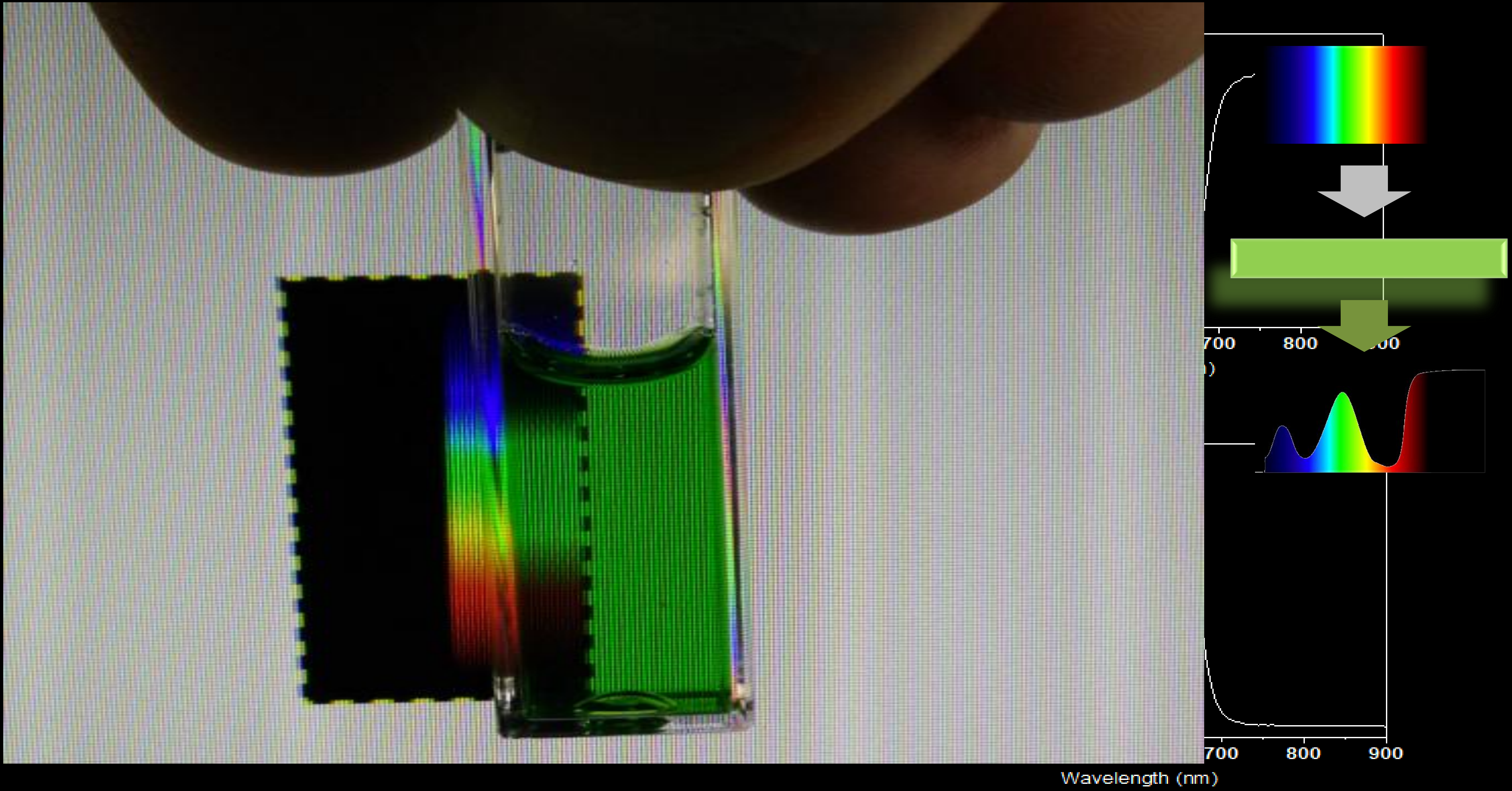
## Composition

## Stress

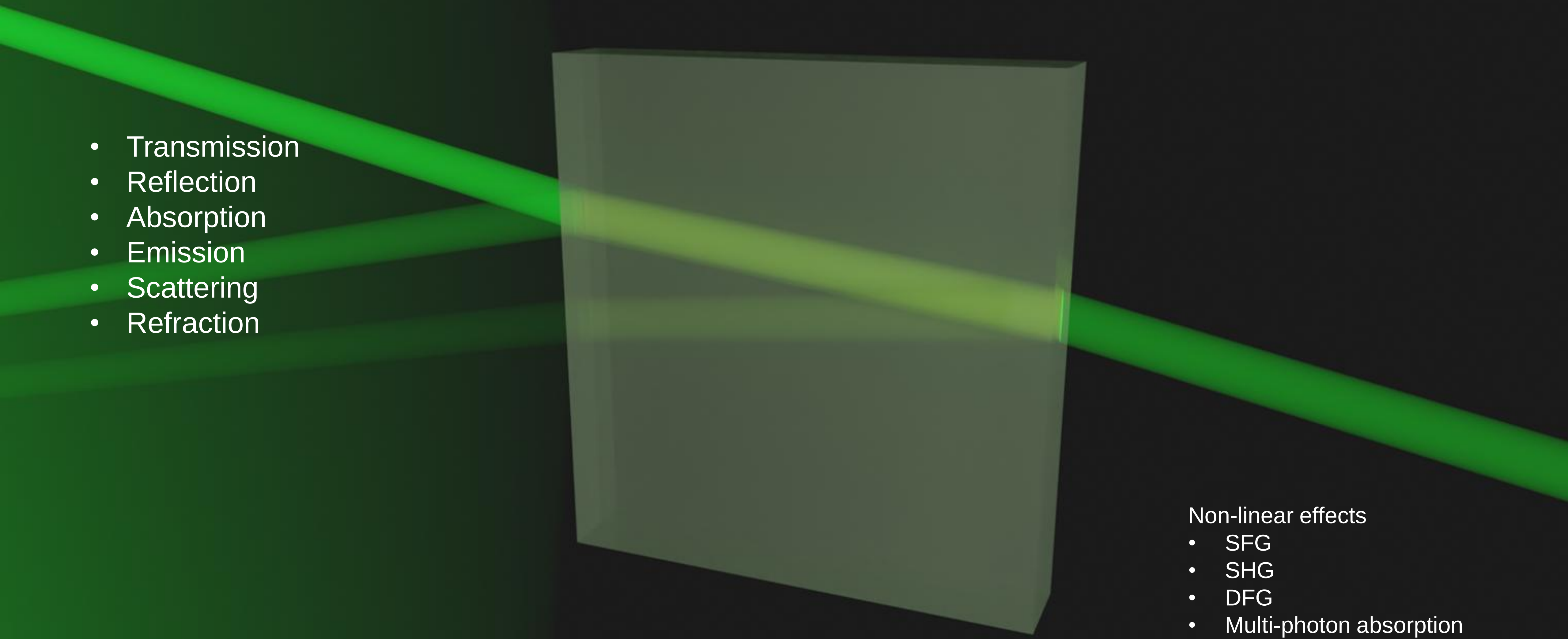
# Light interactions with matter



# Spectroscopy



# Light interactions



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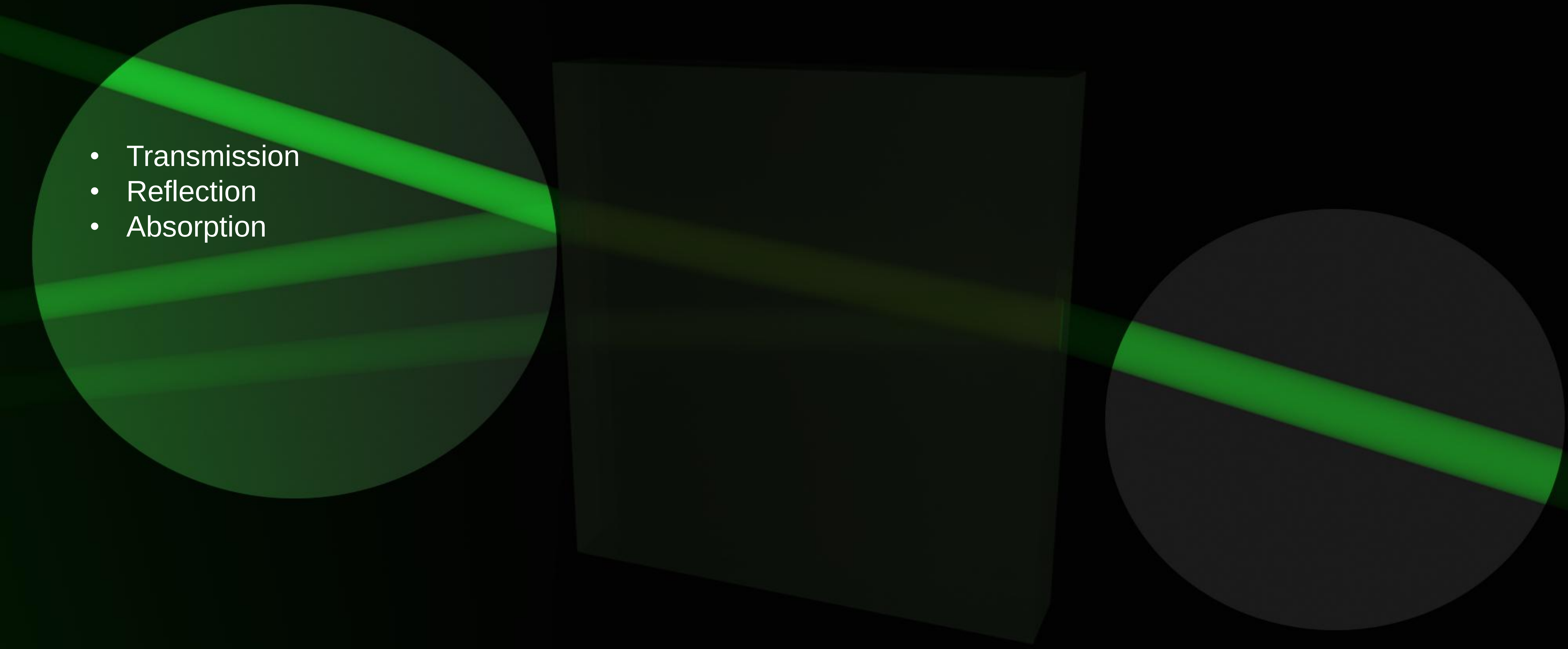

- Transmission
- Reflection
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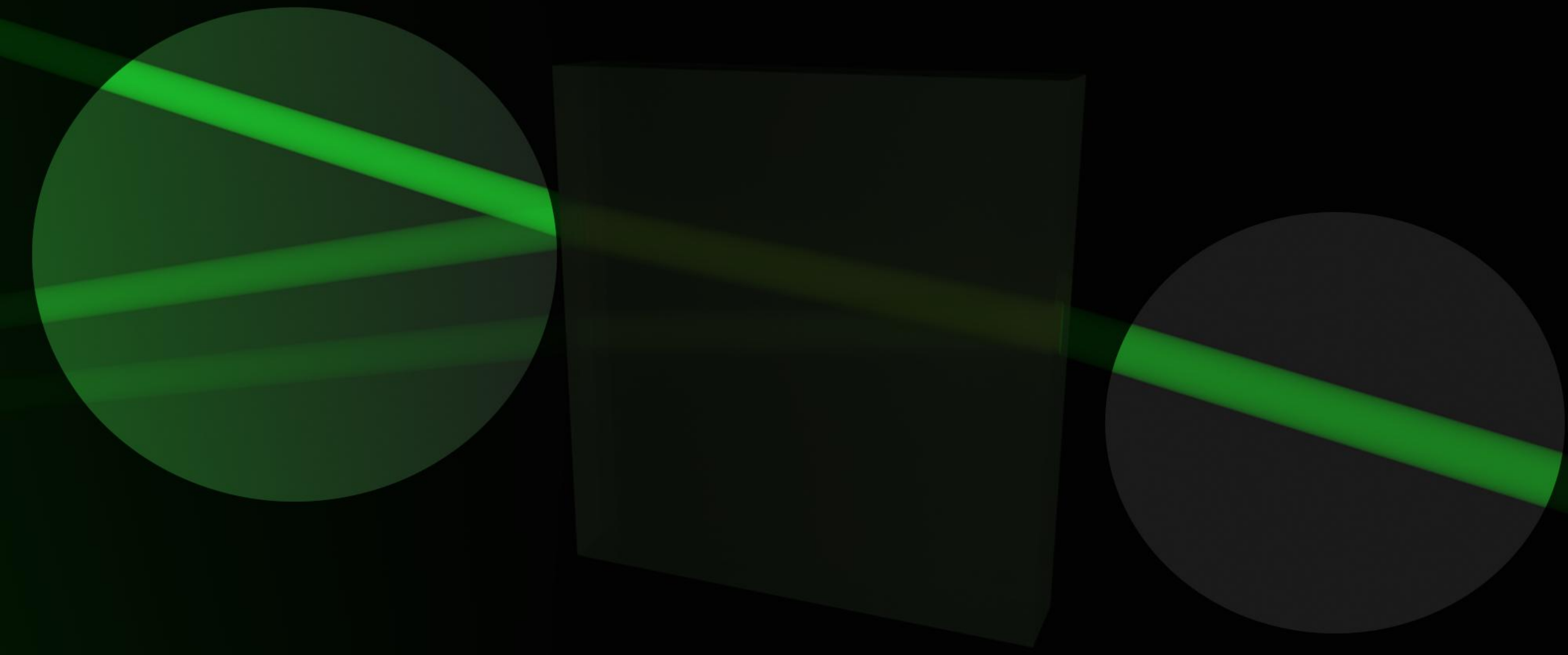
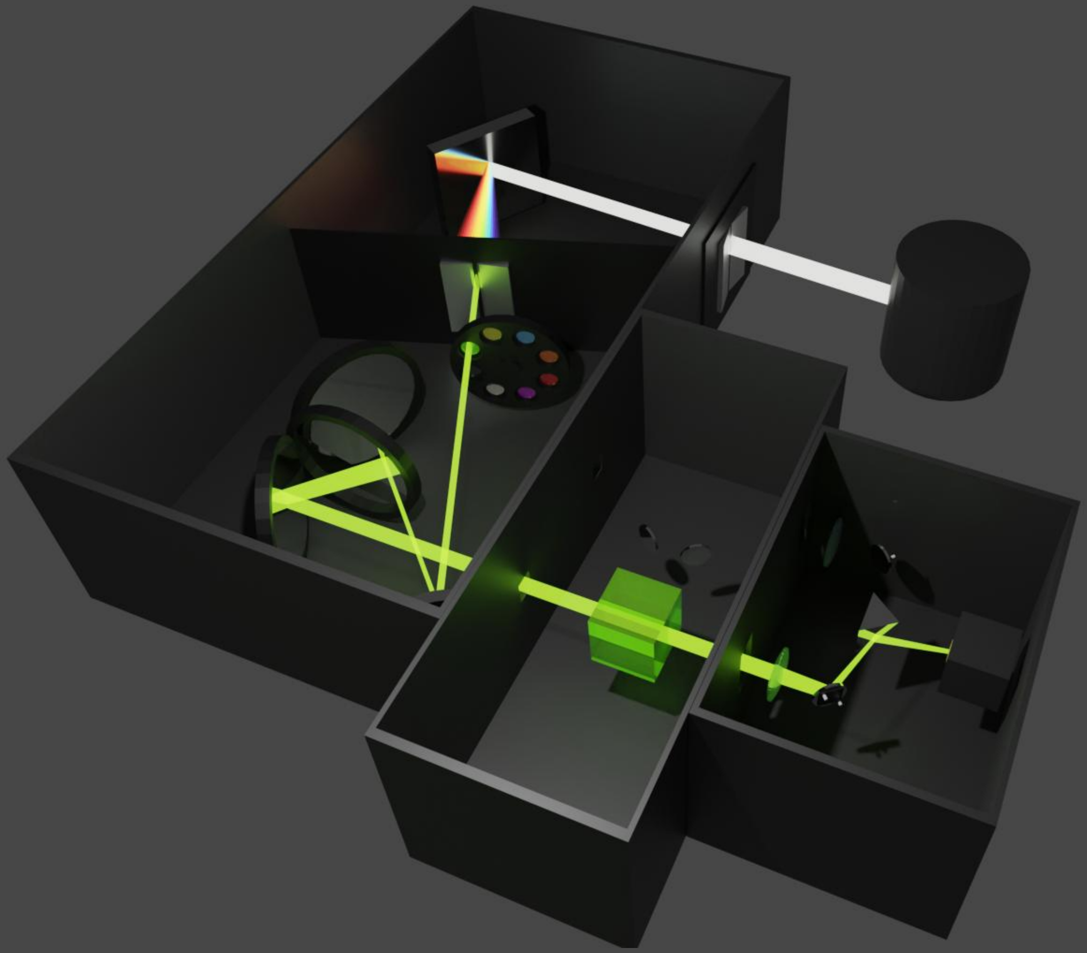


# Spectrophotometry (UV-VIS-NIR)

## Instrumentation:



# Spectrophotometry (UV-VIS-NIR)

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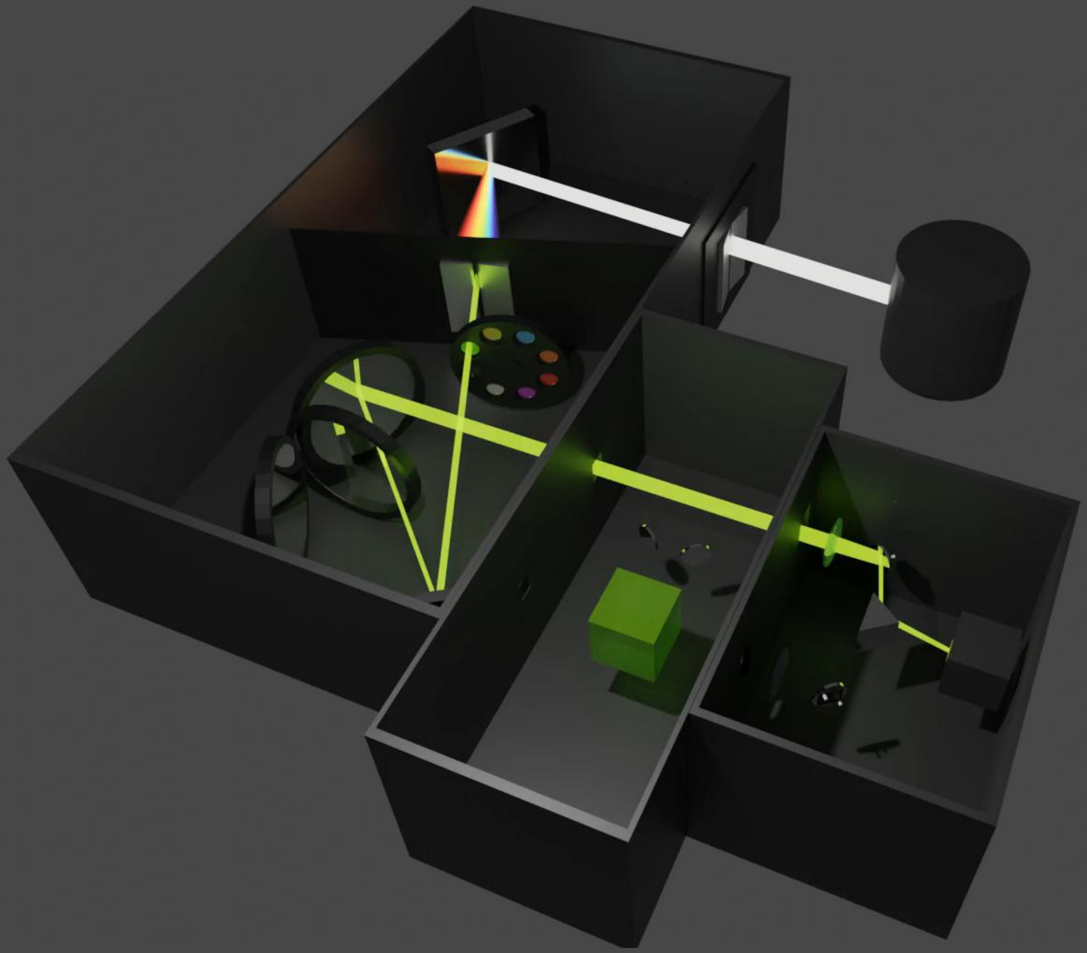
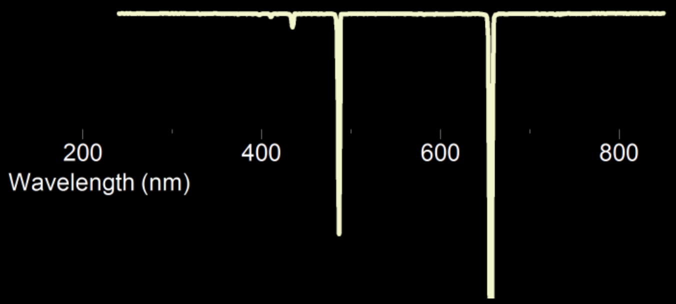
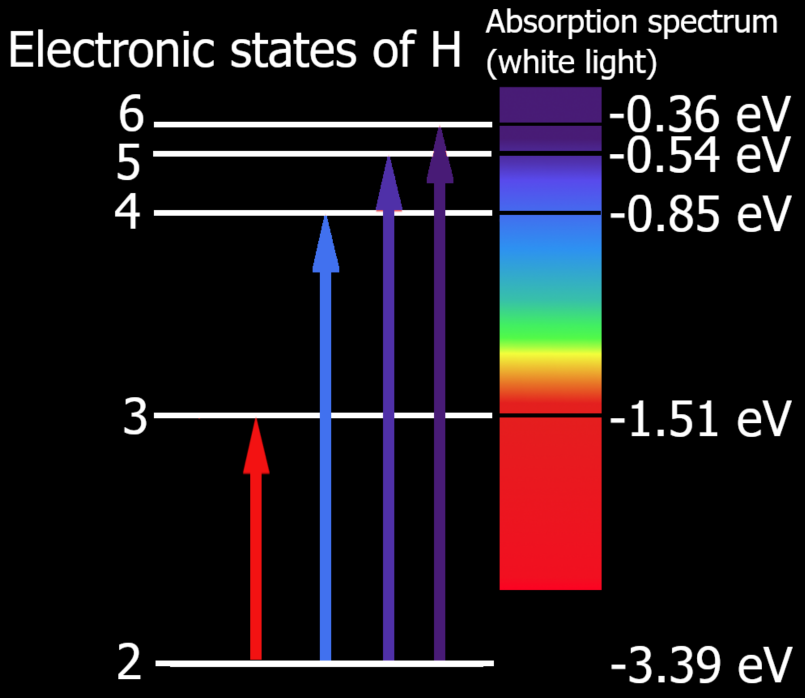


# Spectrophotometry (UV-VIS-NIR)

## Monoatomic gas ($H_2$)

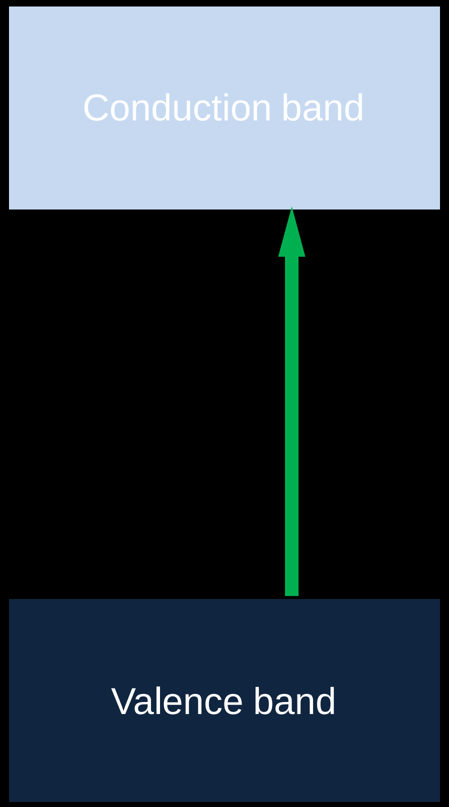
### Electronic levels





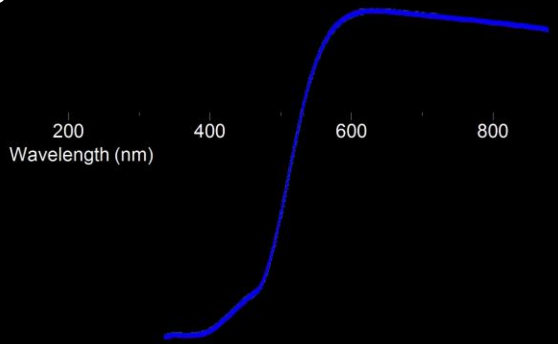
## Solid material ($CdS$)

### Electronic bands

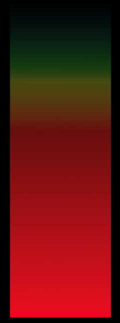


Conduction band

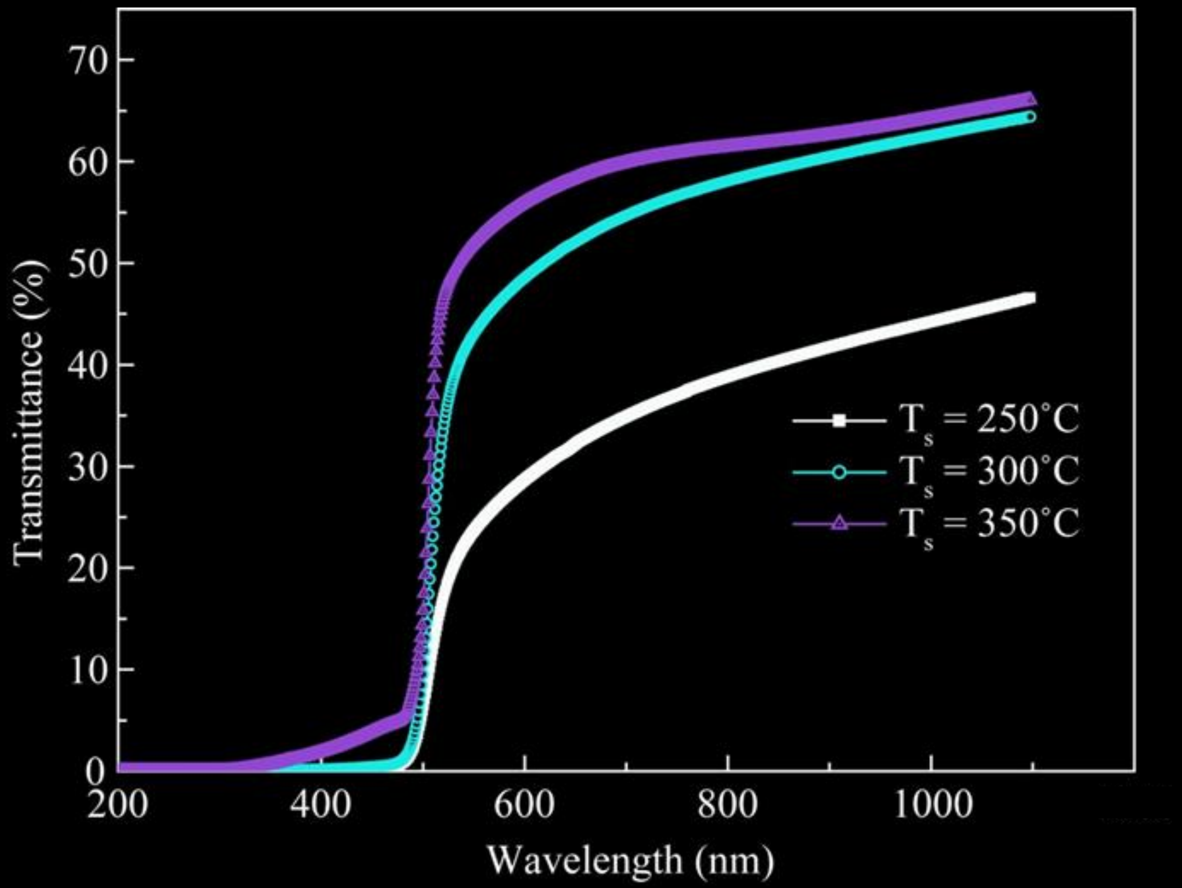
Valence band

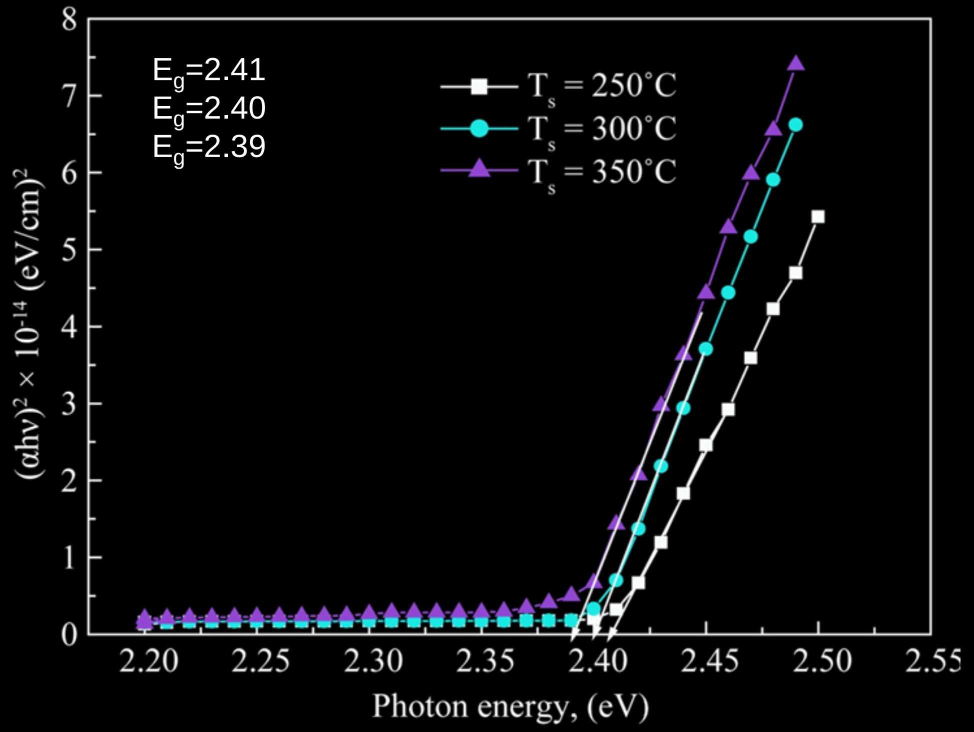


Absorption spectrum
(white light)



# Spectrophotometry (UV-VIS-NIR)





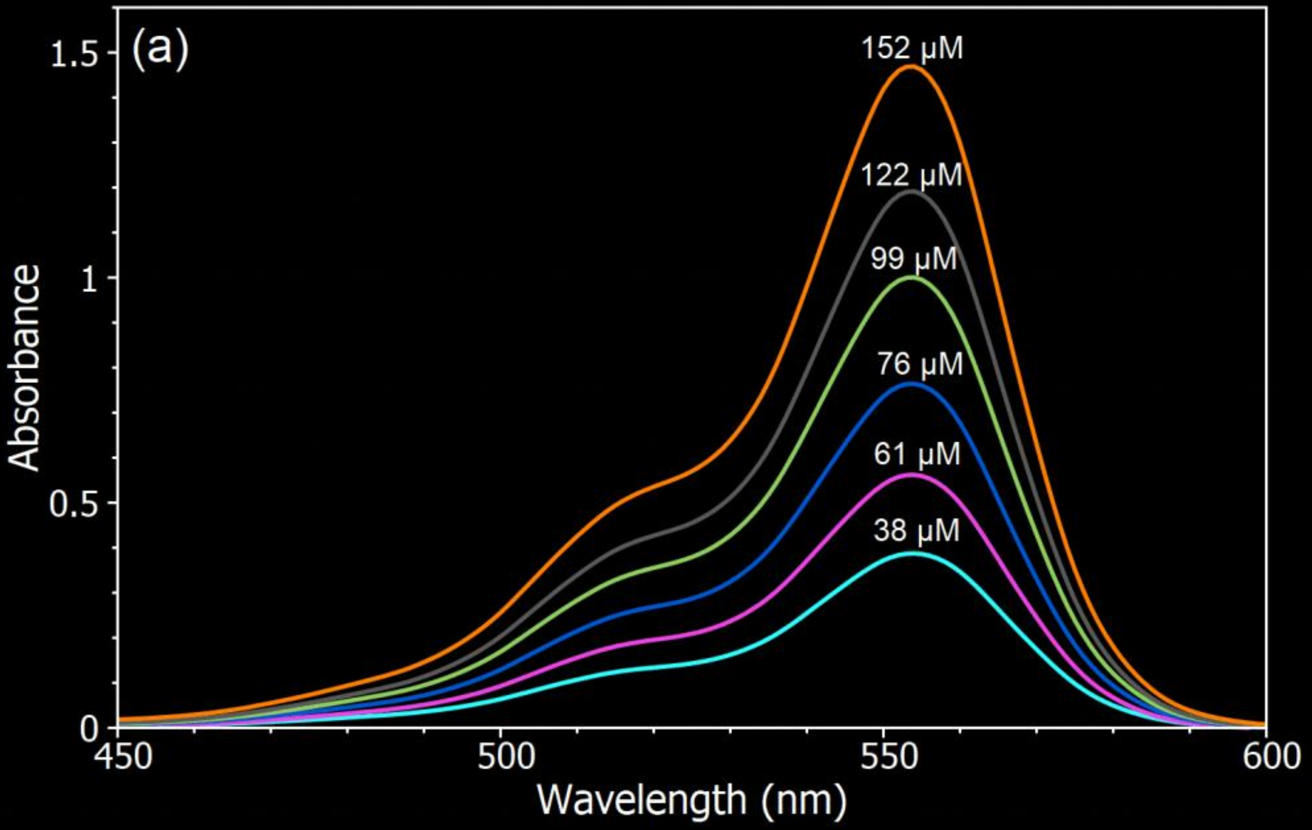
**Optical band gap determination of CdS thin films as a function of growth substrate temperatures**

Tauc's relation: $\alpha h\nu = A\left(h\nu - E_g\right)^m$

m = 0.5 for direct and 2 for indirect allowed transitions.

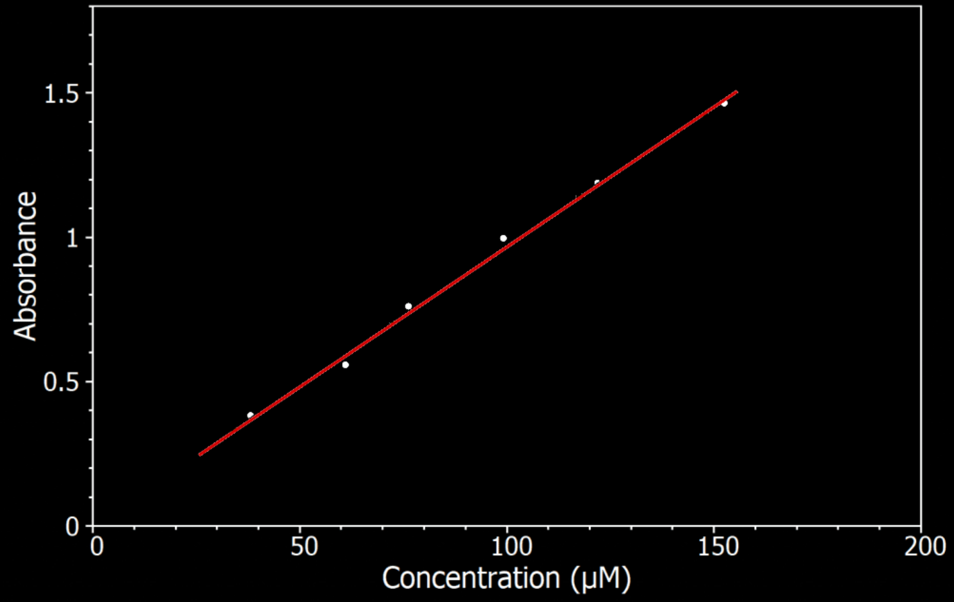
*J. Surf. Eng. Mat. and Adv. Tech*. **3**, 43 (2013)

# Spectrophotometry (UV-VIS-NIR)



Beer-Lambert Law

$Abs = K \ell c = a \ell$
$Abs = \log (1/T)$



**Using absorbance to determine Rhodamine B concentration in water solutions**

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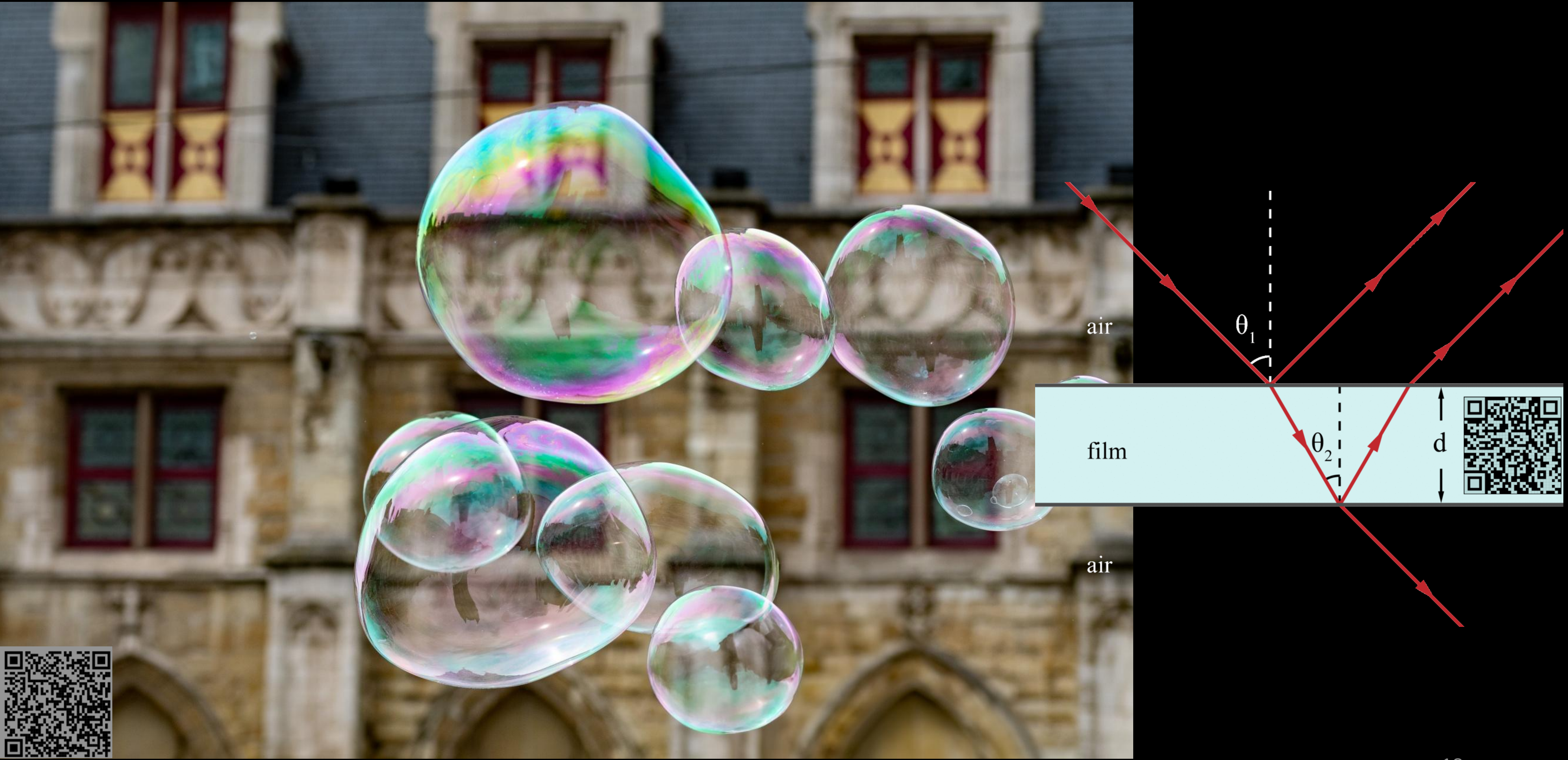
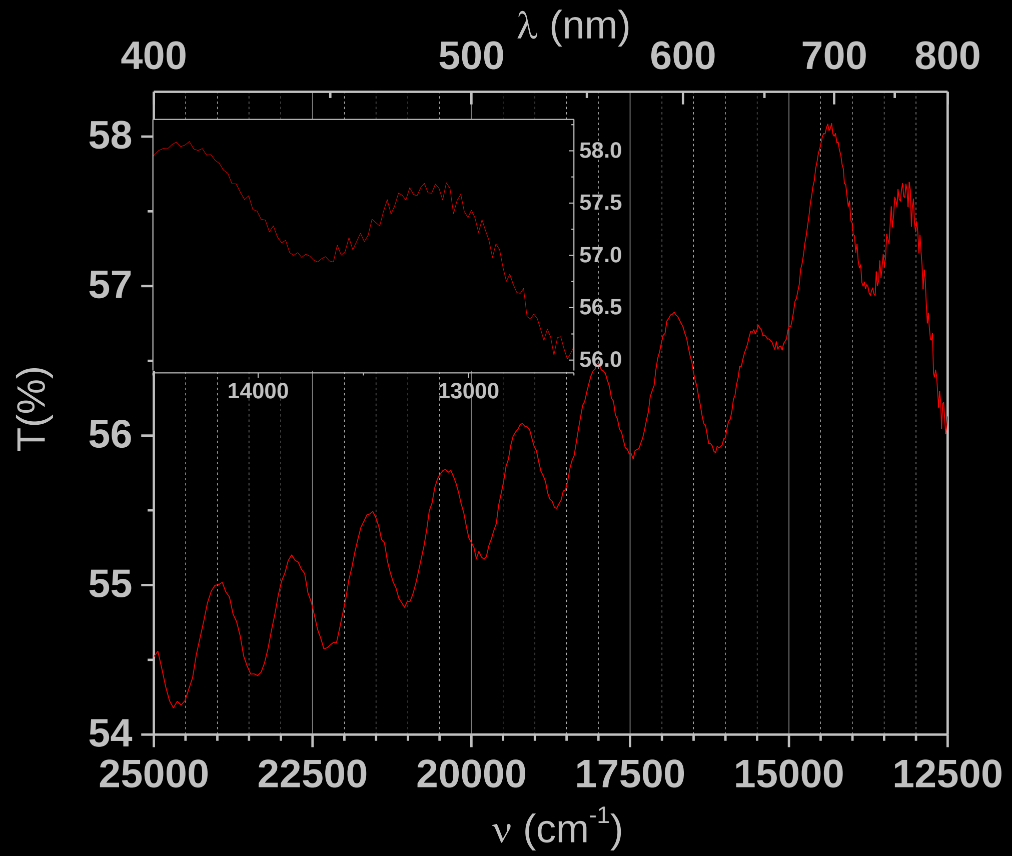
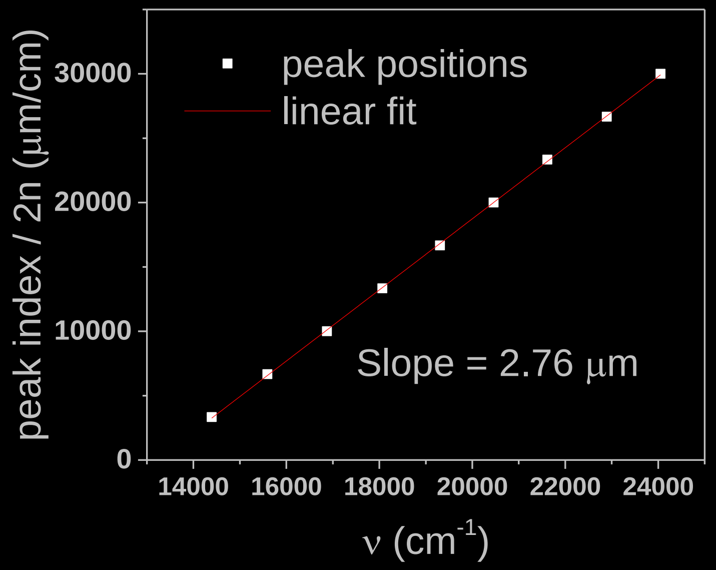
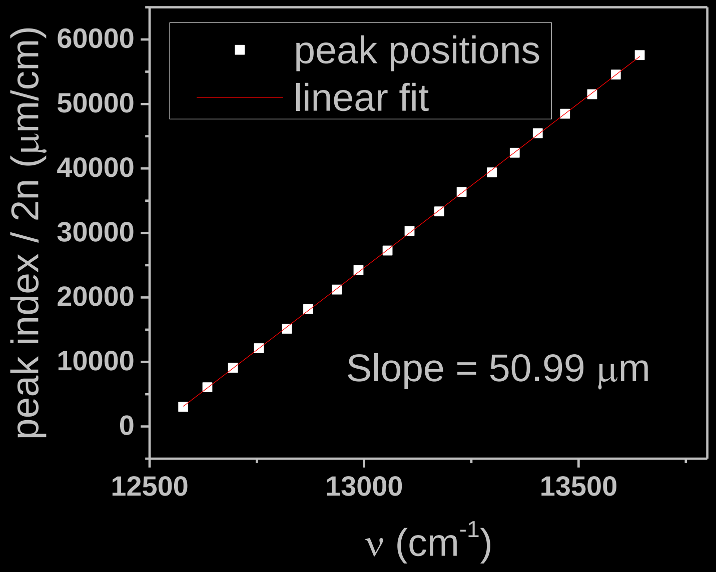
# Spectrophotometry (UV-VIS-NIR)



# Spectrophotometry (UV-VIS-NIR)





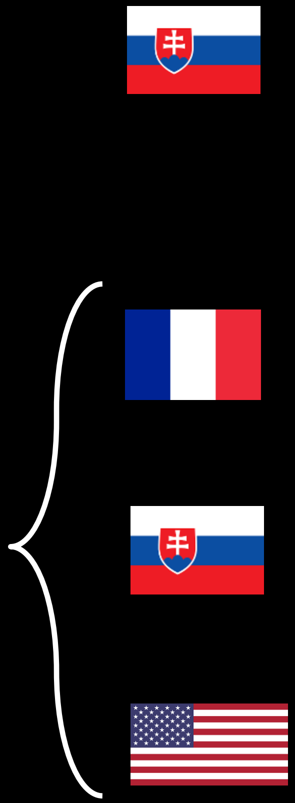


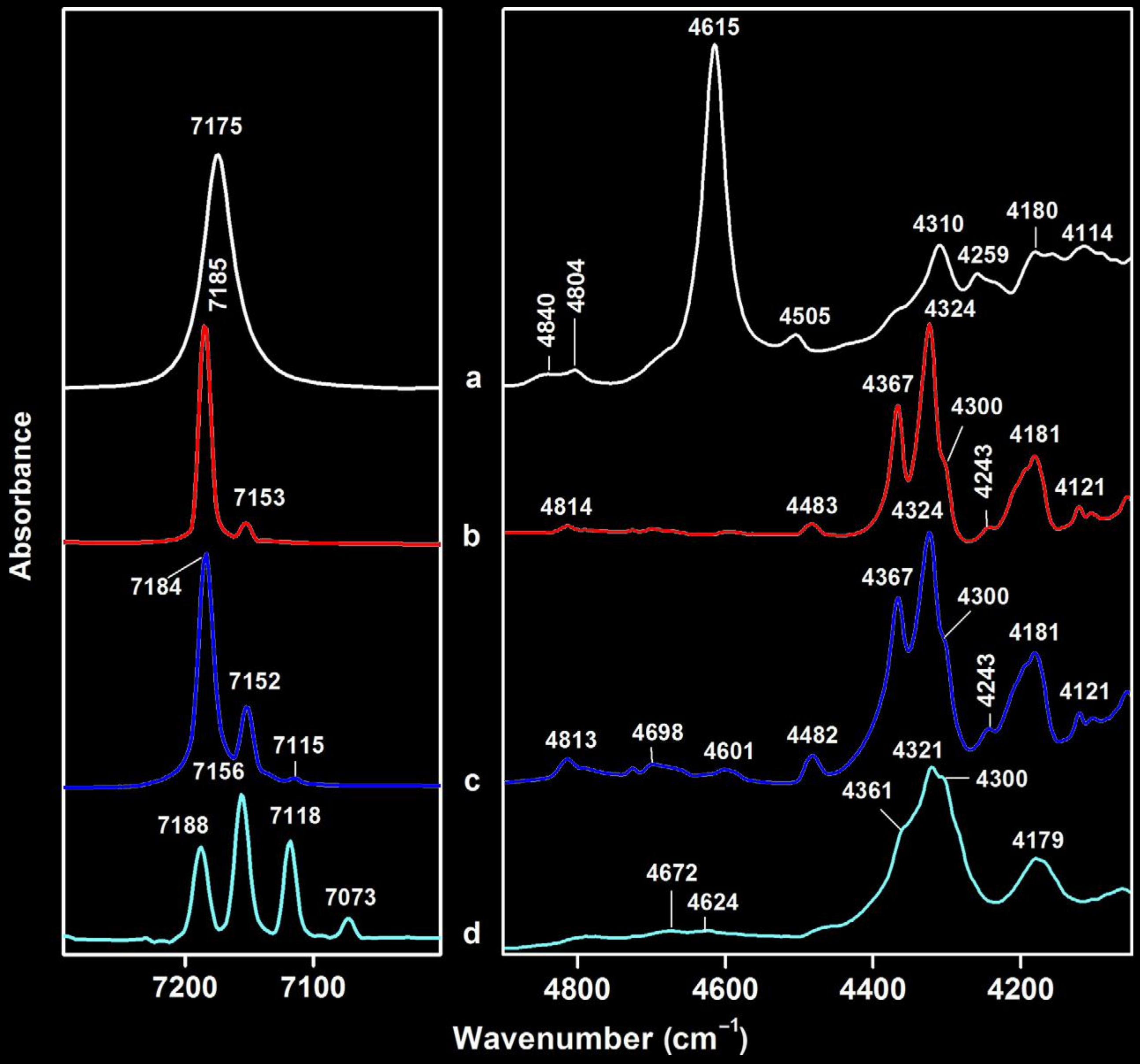
**Using transmission interference fringes to determine thickness**

# Spectrophotometry (UV-VIS-NIR)

$Al_2Si_4O_{10}(OH)_2$

$Mg_3Si_4O_{10}(OH)_2$



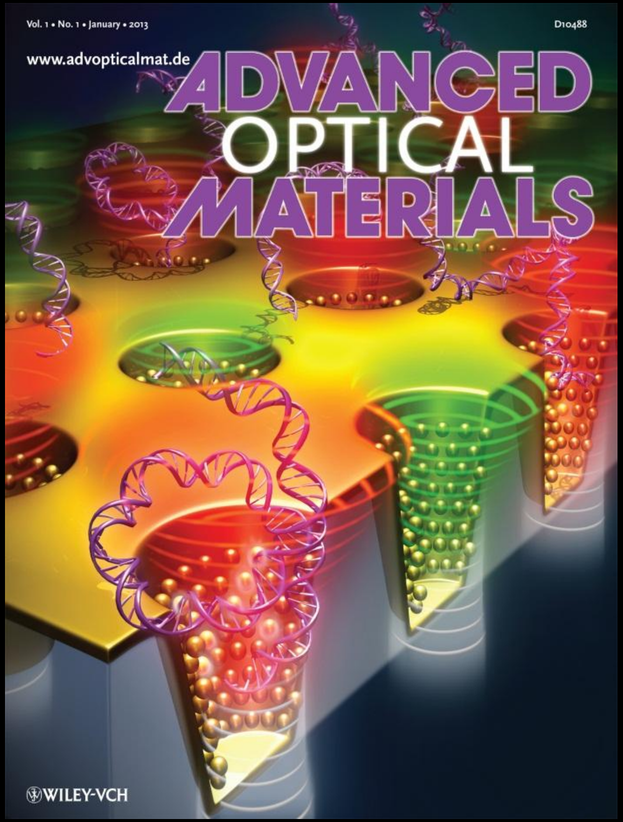


Images from Wikipedia and eurotalc.com
Spectra from *Developments in Clay Science*, Vol. 8. Ch. 5

# Spectrophotometry (UV-VIS-NIR)

**Excitations in materials**

- Plasmons



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D10488

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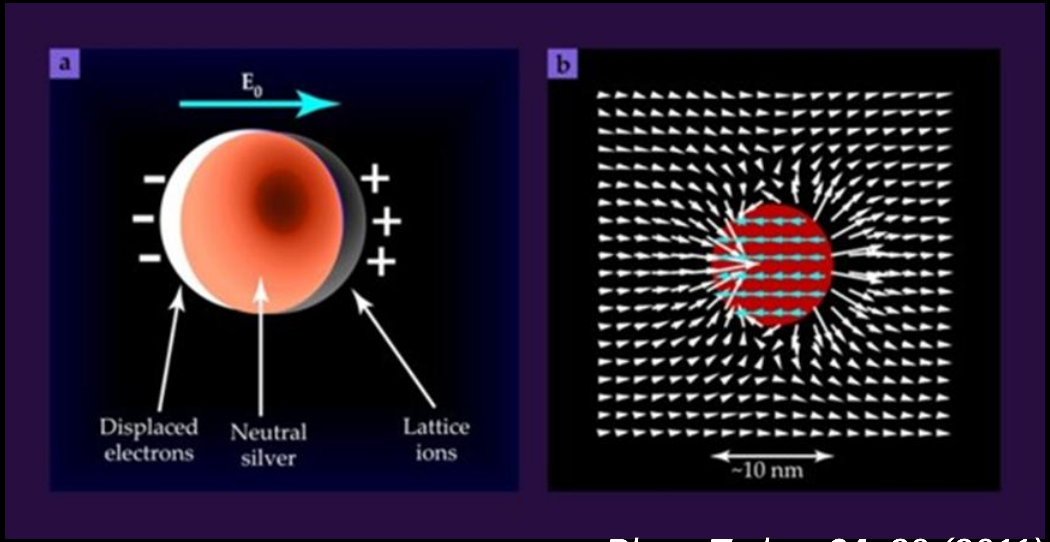
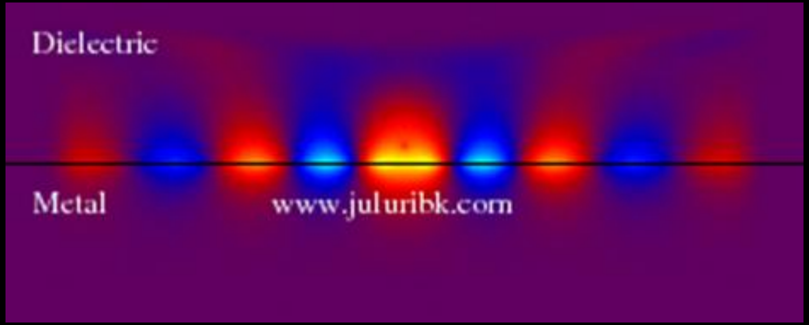
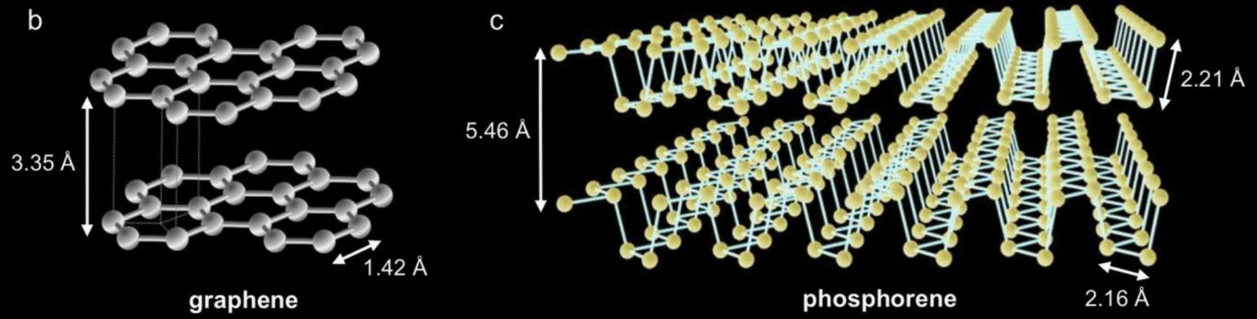


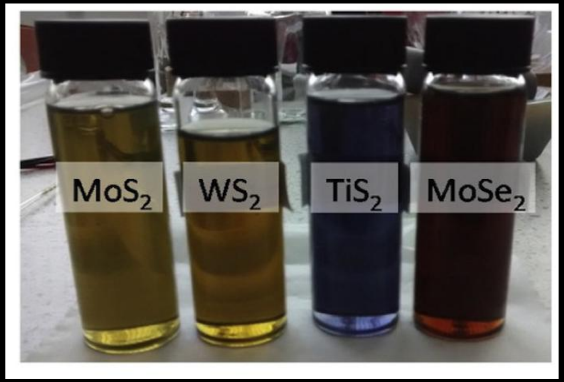
*Phys. Today*, ***64***, *39 (2011)*



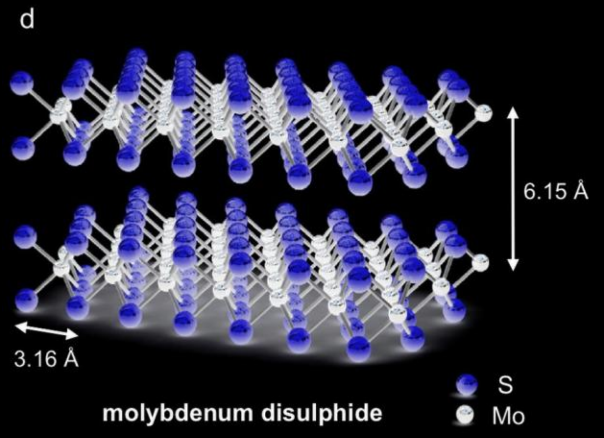
Plasmons are quanta of collective motion of charge-carriers in a gas with respect of an oppositely charged background. They play a significant role on transmission and reflection of light.

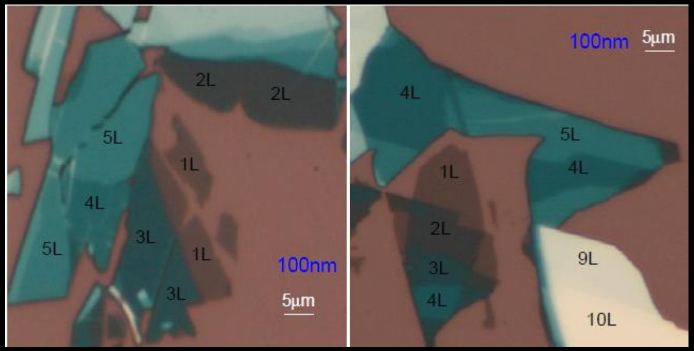
# Spectrophotometry (UV-VIS-NIR)

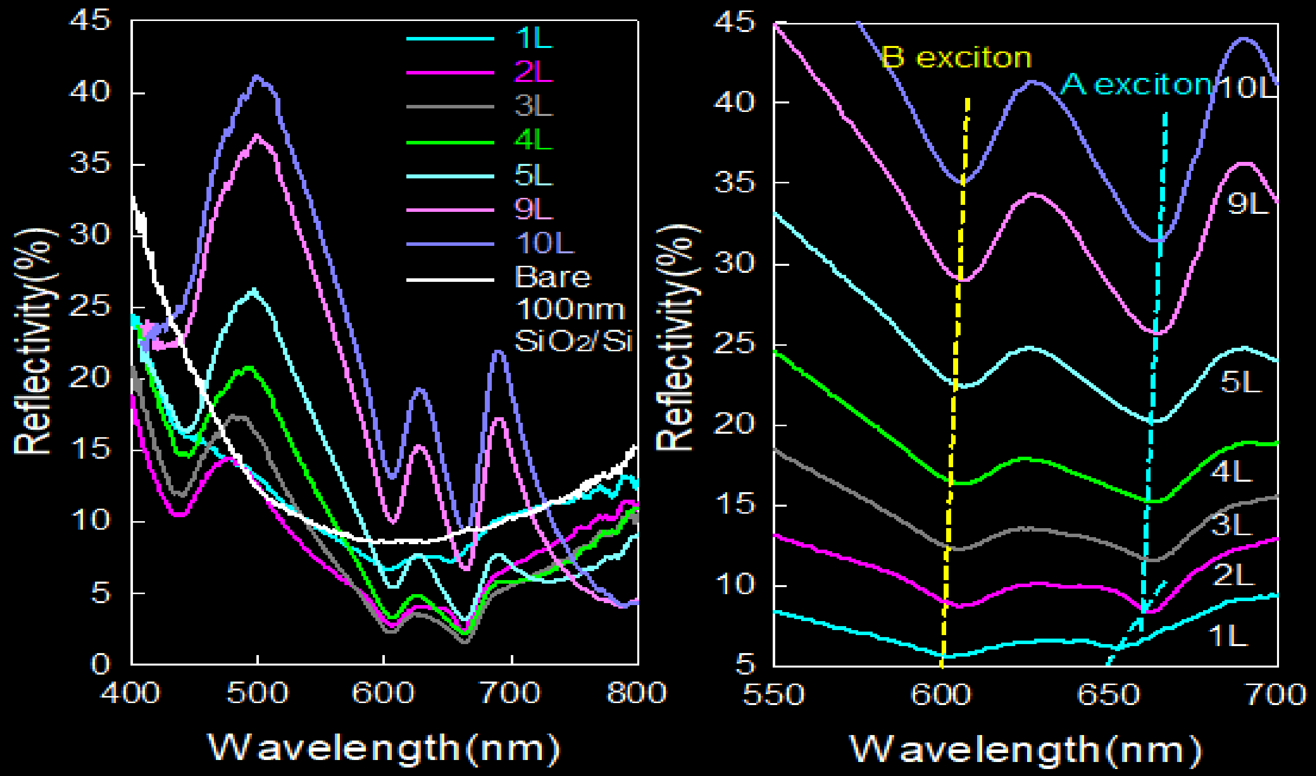




*Applied Materials Today* **8**, 68 (2017)





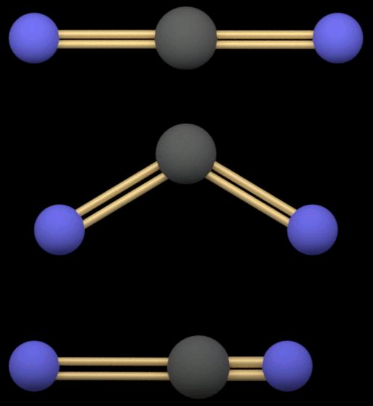


*Optical Materials Express*, 332858 (2018)

# Vibrational spectroscopy

## Normal vibrational modes in molecules:

### $CO_2$ (4 modes)

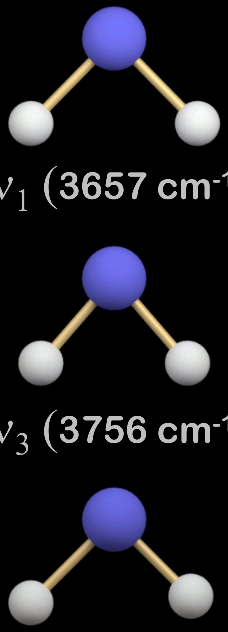


$\nu_1 = 1388$ cm$^{-1}$

$\nu_2 = 667$ cm$^{-1}$

$\nu_3 = 2349$ cm$^{-1}$

### $H_2O$ (3 modes)

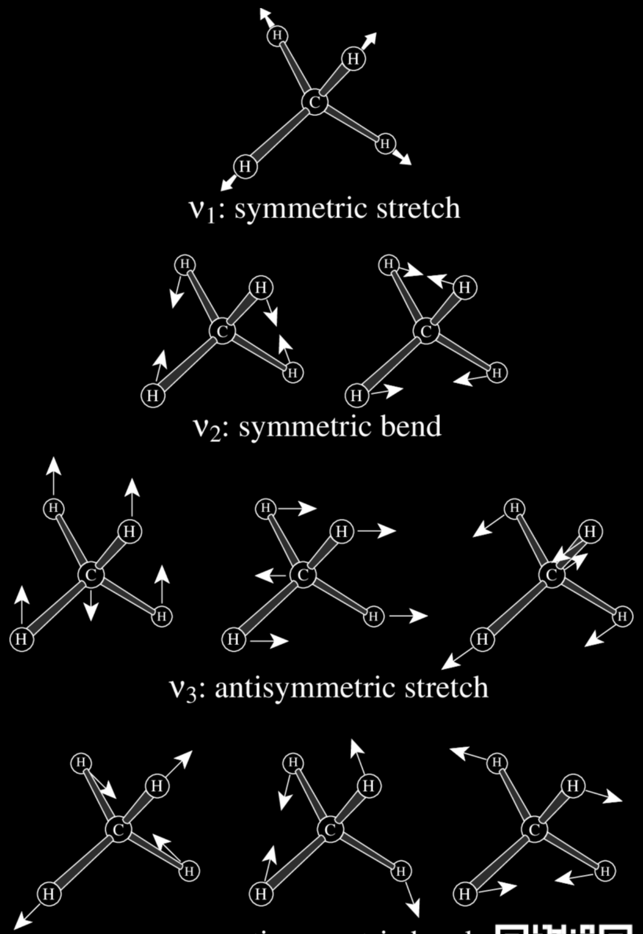


$\nu_1$ (3657 cm$^{-1}$)

$\nu_3$ (3756 cm$^{-1}$)

$\nu_2$ (1595 cm$^{-1}$)

### $CH_4$ (9 modes)



$\nu_1$: symmetric stretch

$\nu_2$: symmetric bend

$\nu_3$: antisymmetric stretch

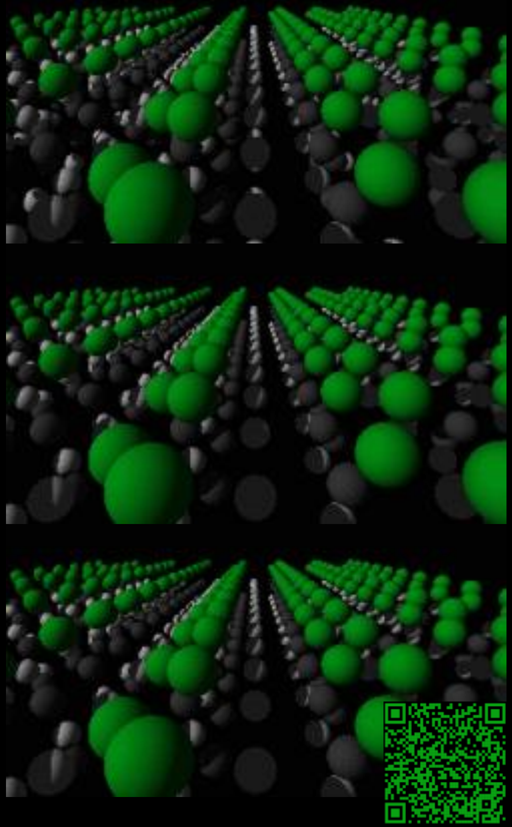
$\nu_4$: antisymmetric bend

**Number of modes:**
**3N-6 for non-linear molecules**
**3N-5 for linear molecules**

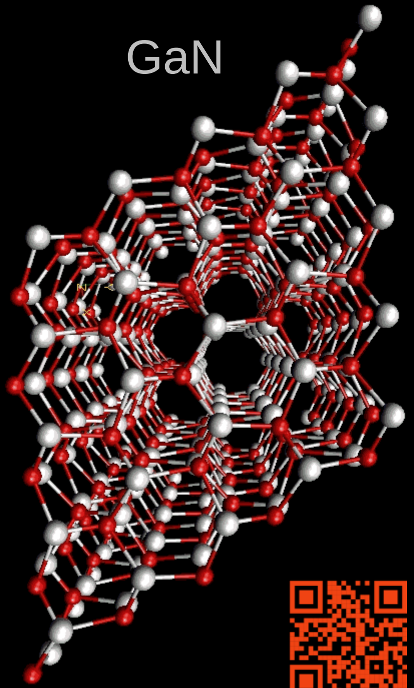
# Vibrational spectroscopy

Normal vibrational modes in solids:

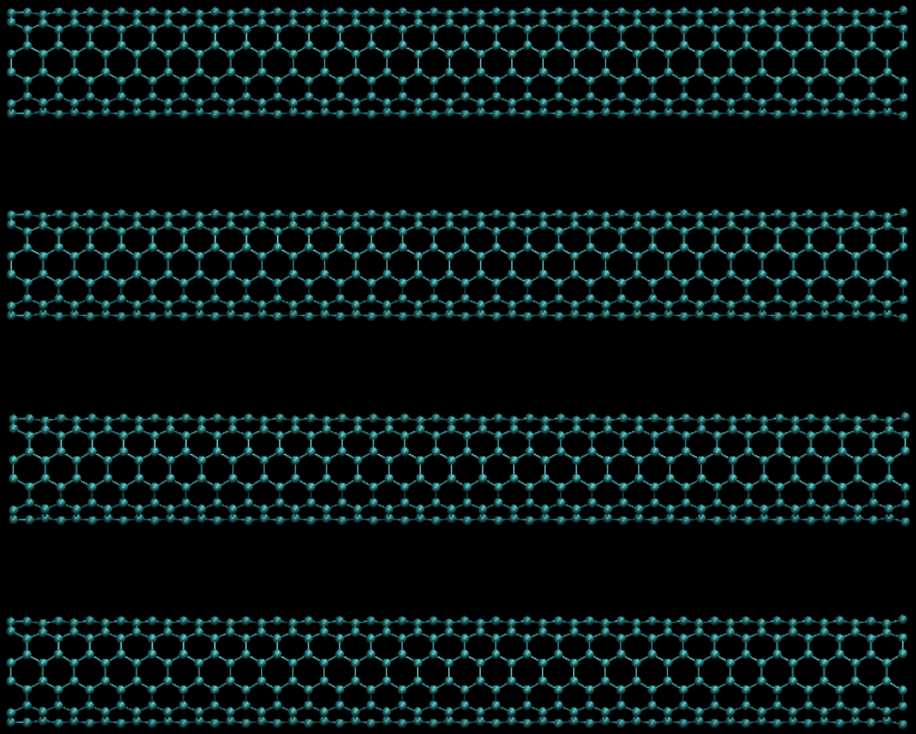
Sb/GaAs(110)



GaN



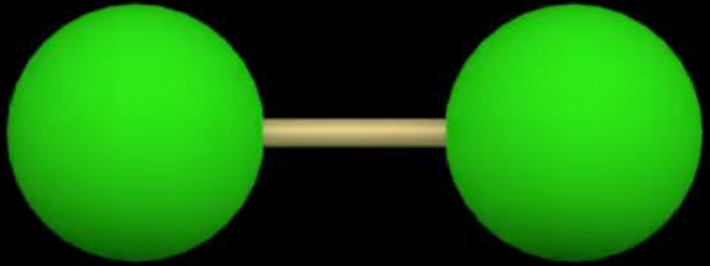
SWCNT



# Fourier Transform IR spectroscopy (FTIR)

## IR active vibrations

The intensity of a vibrational absorption depends on the change of the transition’s dipole momentum caused by that vibration, so a vibration mode $\nu_i$ will be “IR active” only when the vibration causes a change in the dipole momentum of the molecule, i.e. $\Delta\mu \neq 0$



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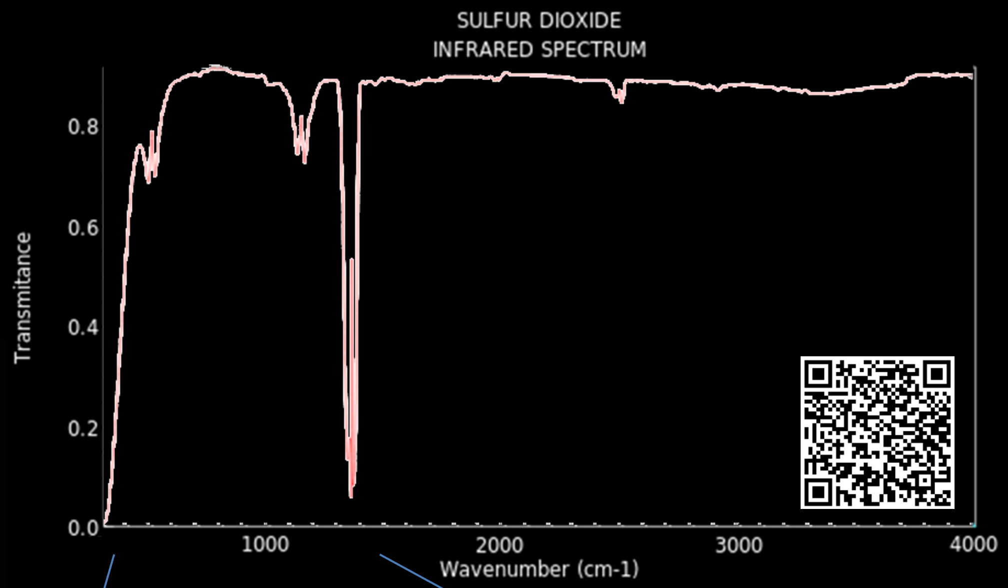
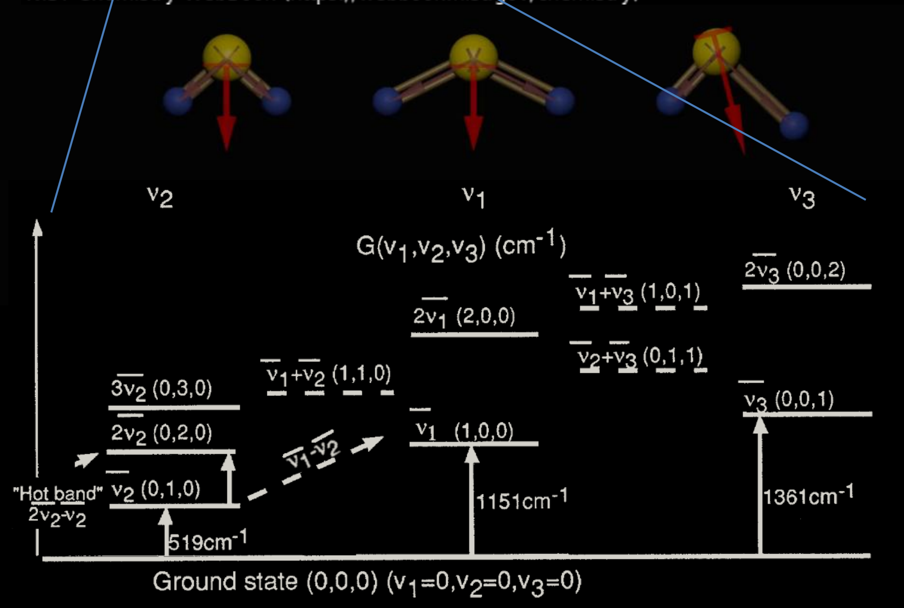
These vibrations appear as "dark bands" in the transmitted light spectrum



NIST Chemistry WebBook (https://webbook.nist.gov/chemistry)



Journal of Near Infrared Spectroscopy, **6** (1998)

# Fourier Transform IR spectroscopy (FTIR)

**The Nobel Prize in Physics 1907**
**Albert A. Michelson**

*"for his optical precision instruments and the spectroscopic and metrological investigations carried out with their aid"*

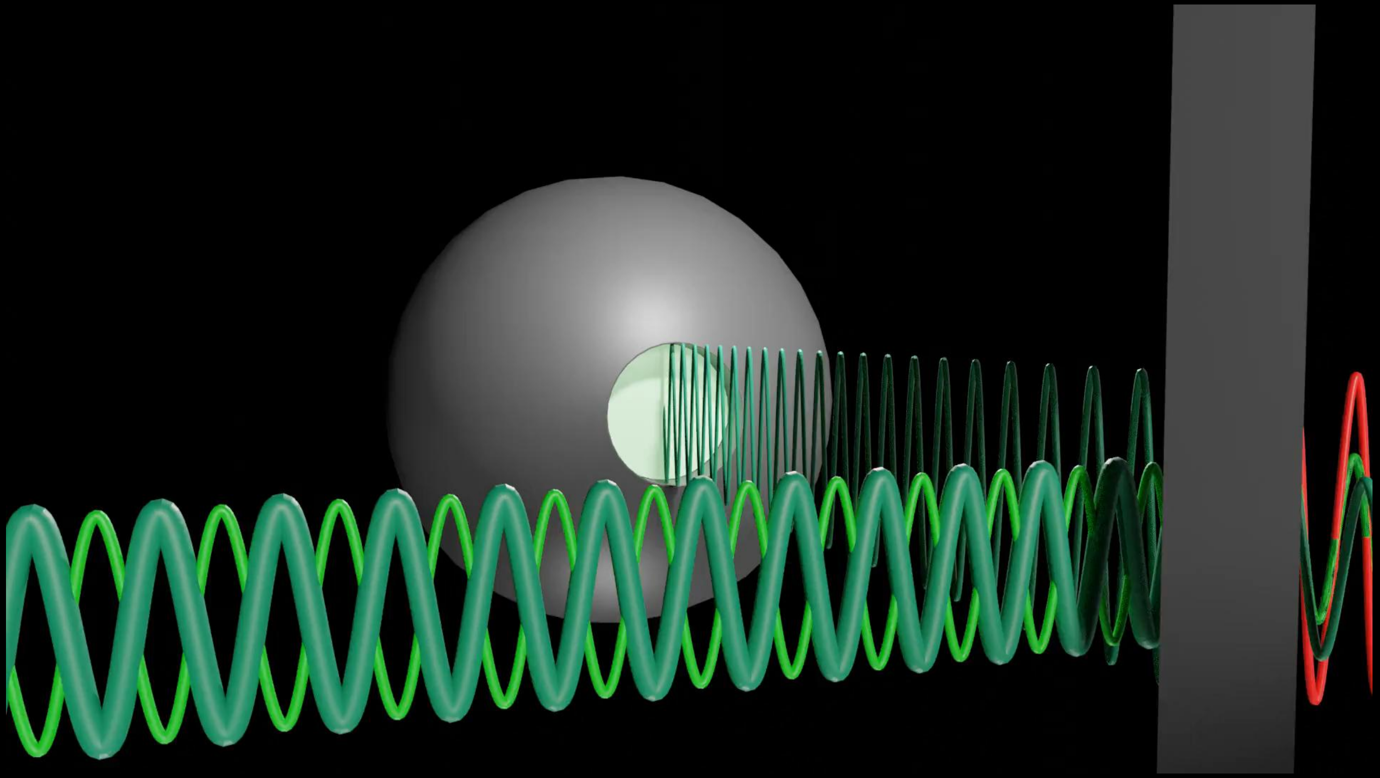
The Nobel Foundation

## Instrumentation:

The FTIR uses a Michelson interferometer with a moving mirror, in place of a diffraction grating or prism.

$\Delta L = n\lambda$ => constructive interference

$\Delta L = (n+1/2)\,\lambda$ => destructive interference



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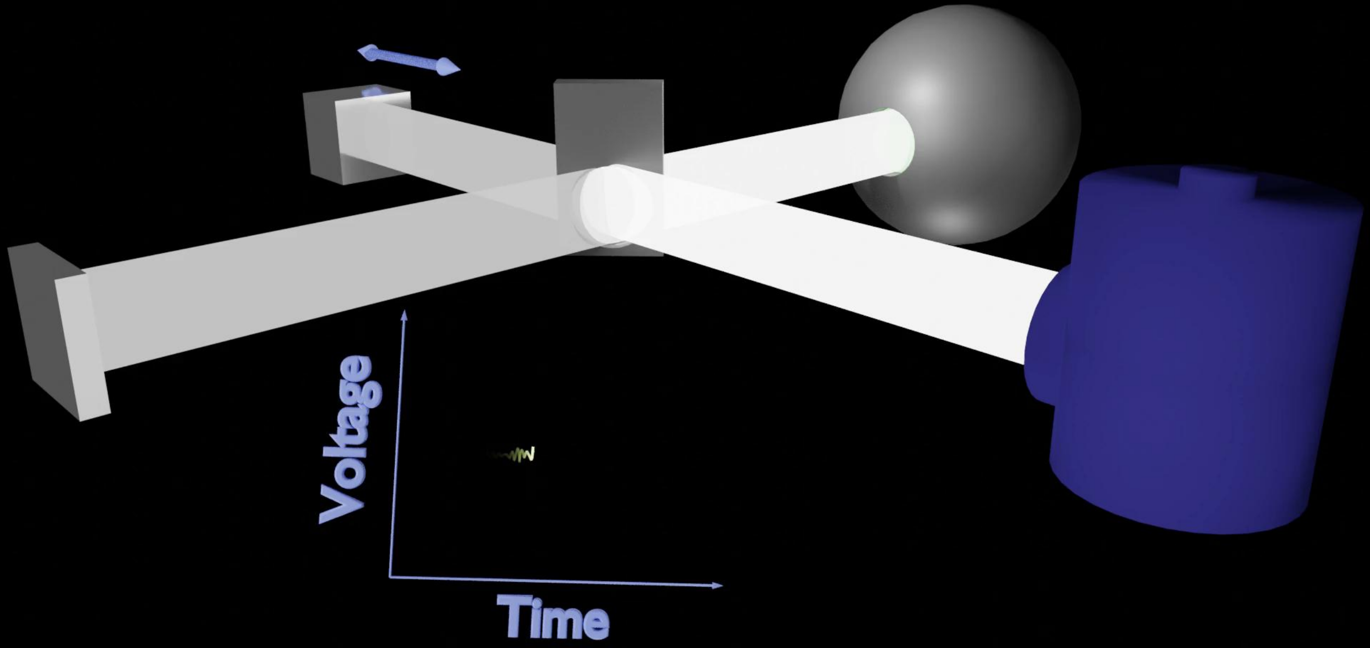
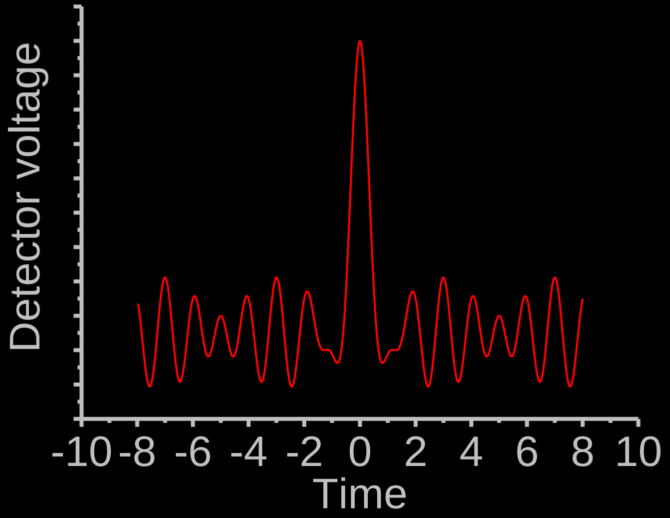
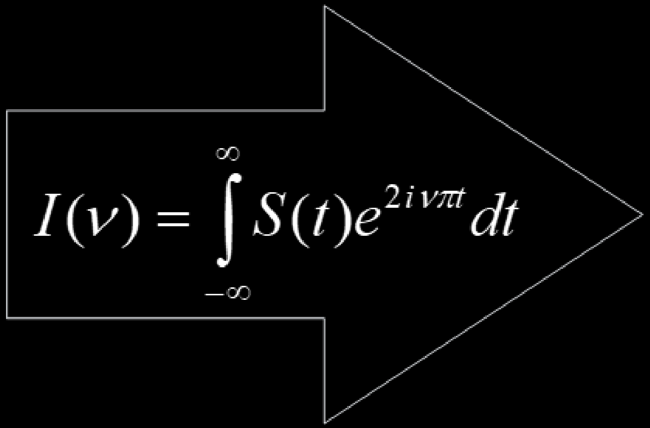
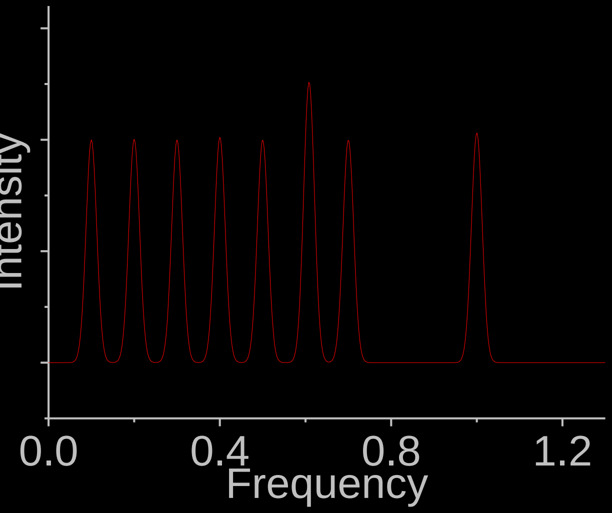


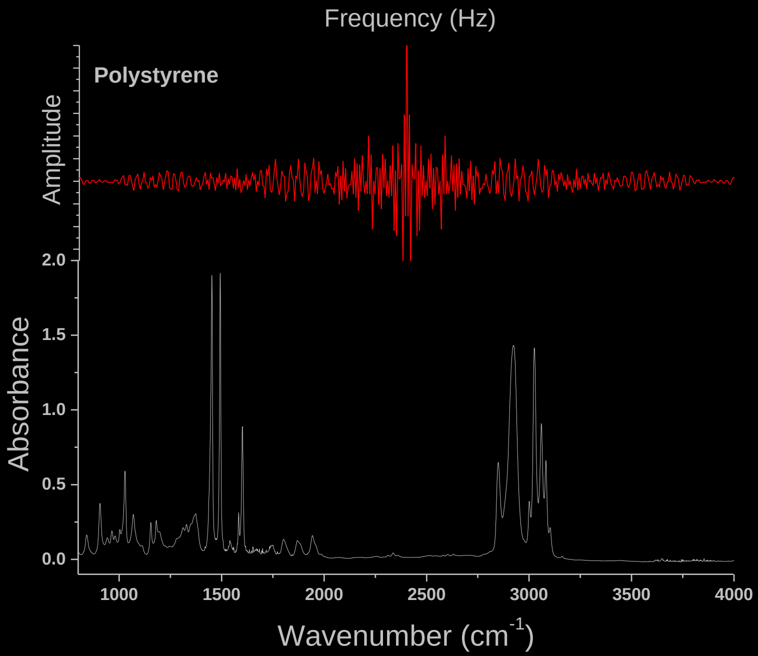
# Fourier Transform IR spectroscopy (FTIR)





$$I(\nu) = \int_{-\infty}^{\infty} S(t)e^{2i\nu\pi t}dt$$



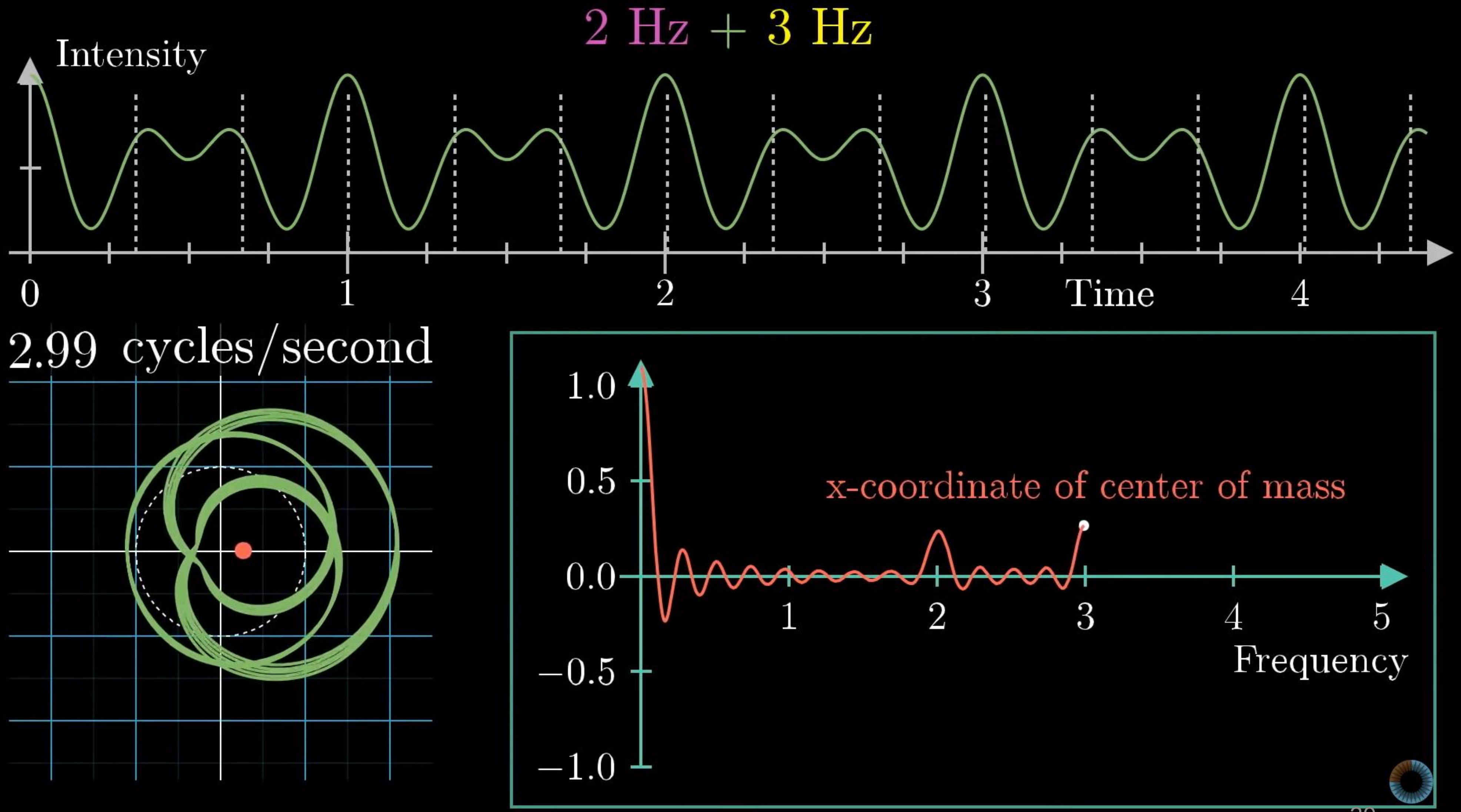


# Fourier Transform IR spectroscopy (FTIR)

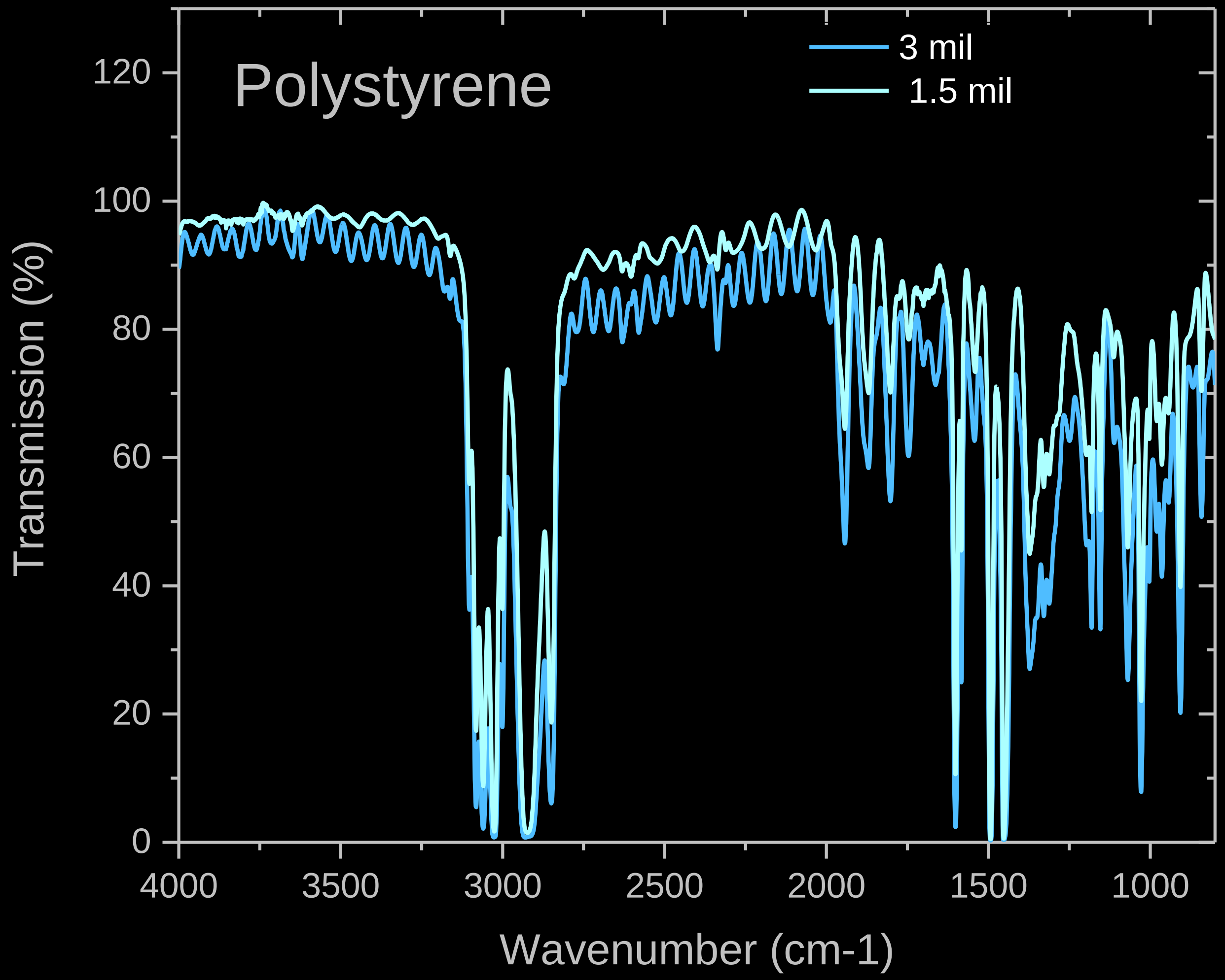
**But what is the Fourier Transform? A visual introduction.**

**3Blue1Brown**

Grant Sanderson



# Fourier Transform IR spectroscopy (FTIR)

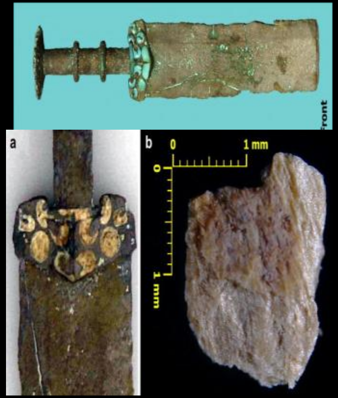


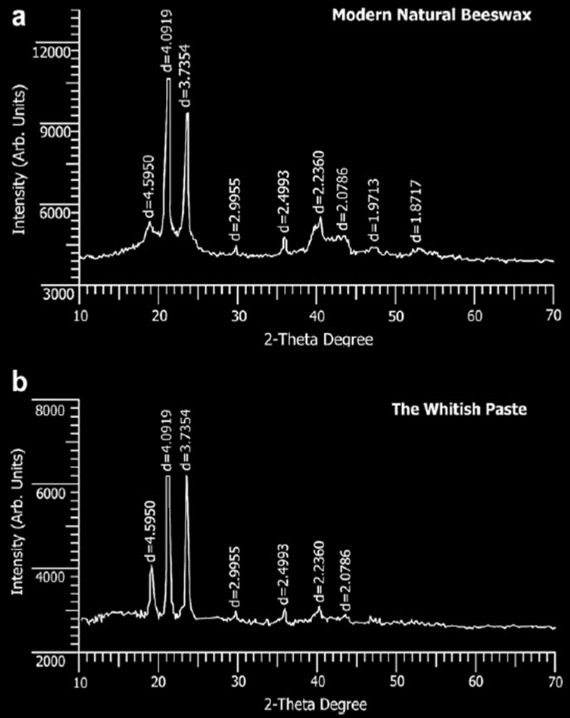
# Fourier Transform IR spectroscopy (FTIR)

FTIR can be used to identify components in a mixture by comparison with reference spectra.

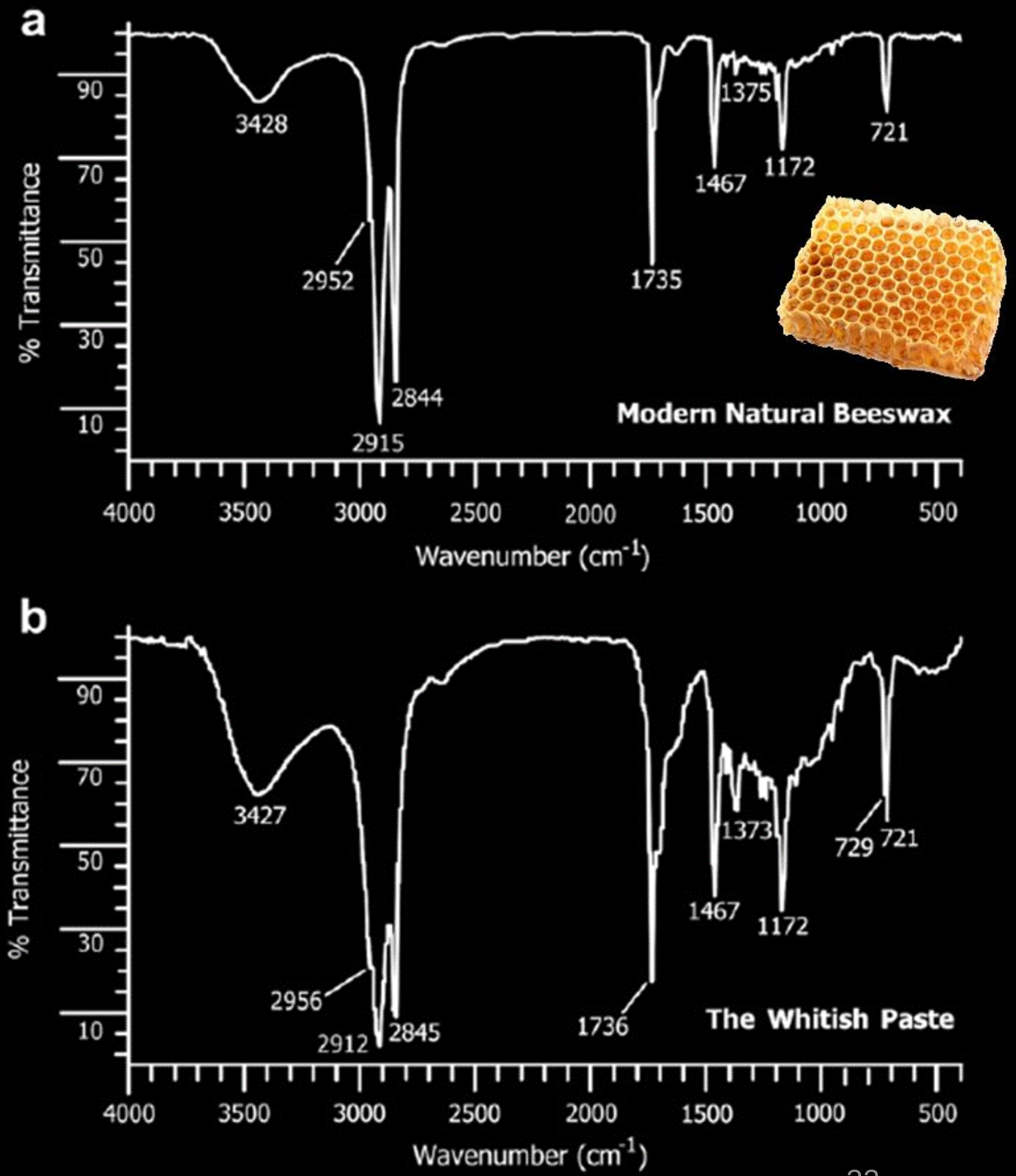
***Discovery of beeswax as binding agent on a 6th-century BC Chinese turquoise-inlaid bronze sword***

Wugan Luo, Tao Li, Changsui Wang, Fengchun Huang





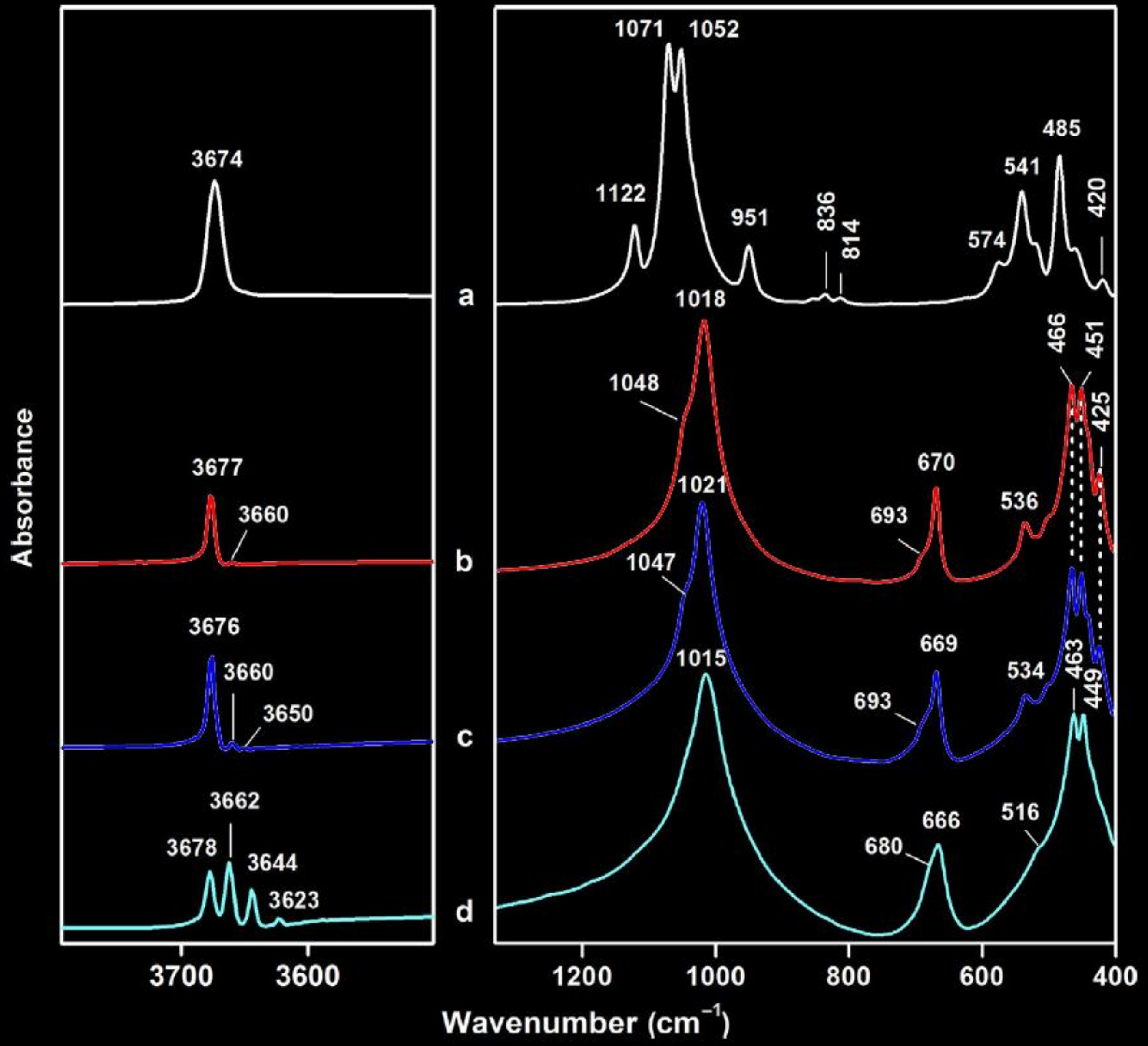
*J. of Archaeological Sci*. **39** (2012), 1227



# Fourier Transform IR spectroscopy (FTIR)

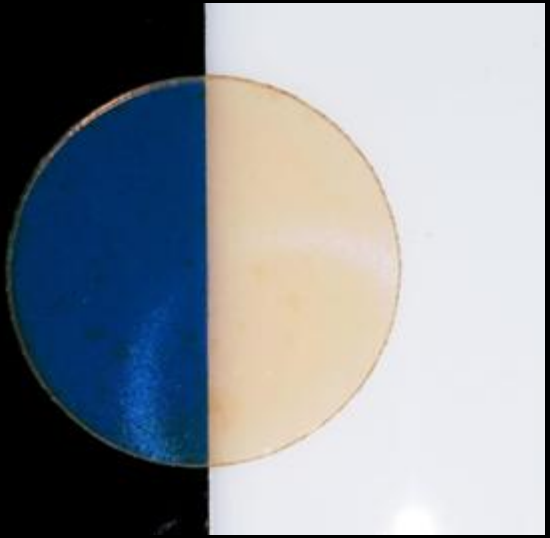
$Al_2Si_4O_{10}(OH)_2$

$Mg_3Si_4O_{10}(OH)_2$



Images from Wikipedia and eurotalc.com
Spectra from *Developments in Clay Science*, Vol. 8. Ch. 5

# Spectrophotometry (UV-VIS-NIR) and FTIR



**Limitations:**

- Reference sample is often needed for quantitative analysis.
- Many contributions to the spectrum are small and can be buried in the background.
- Usually, unambiguous chemical identification requires the use of complementary techniques.
- Limited spatial resolution.

**Strengths:**

- Very little or simple sample preparation.
- Simplicity of use and data interpretation.
- Short acquisition time, for most cases.
- Non destructive.
- Broad range of photon energies.
- High sensitivity (~ 0.1 wt% typical for FTIR).

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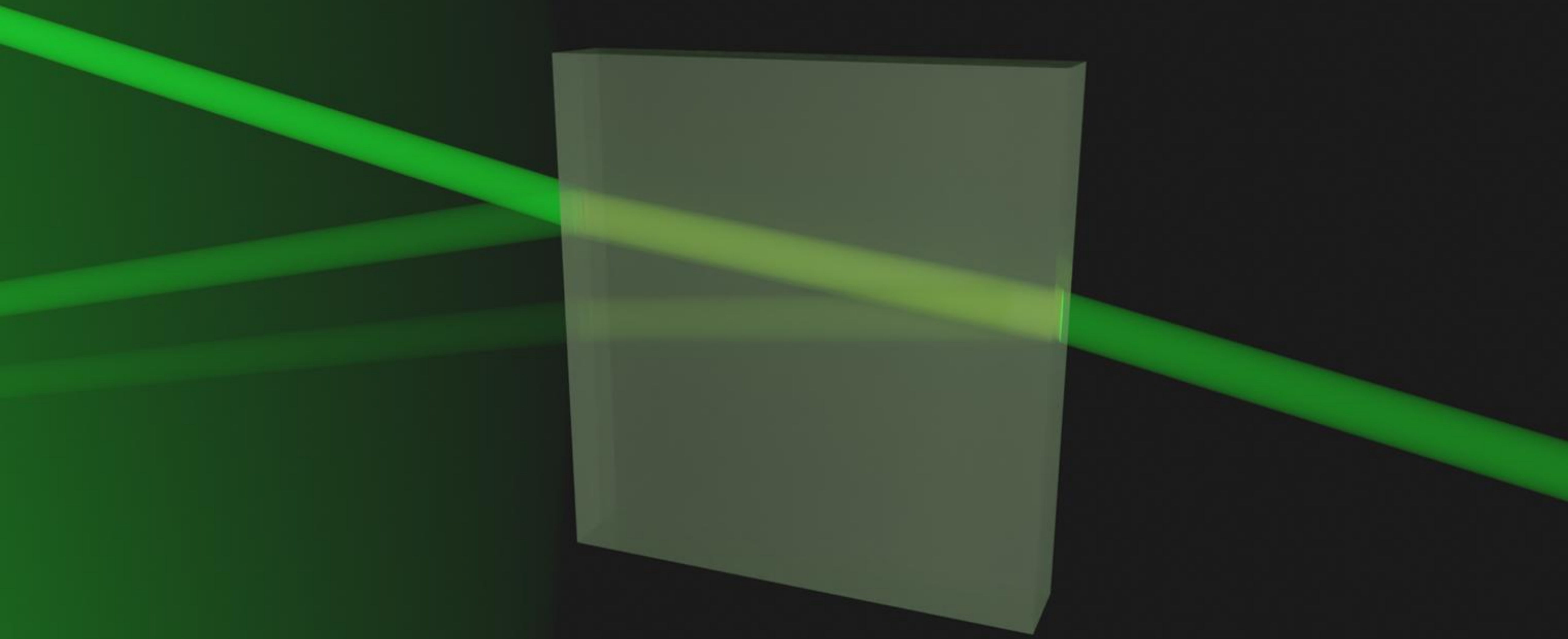
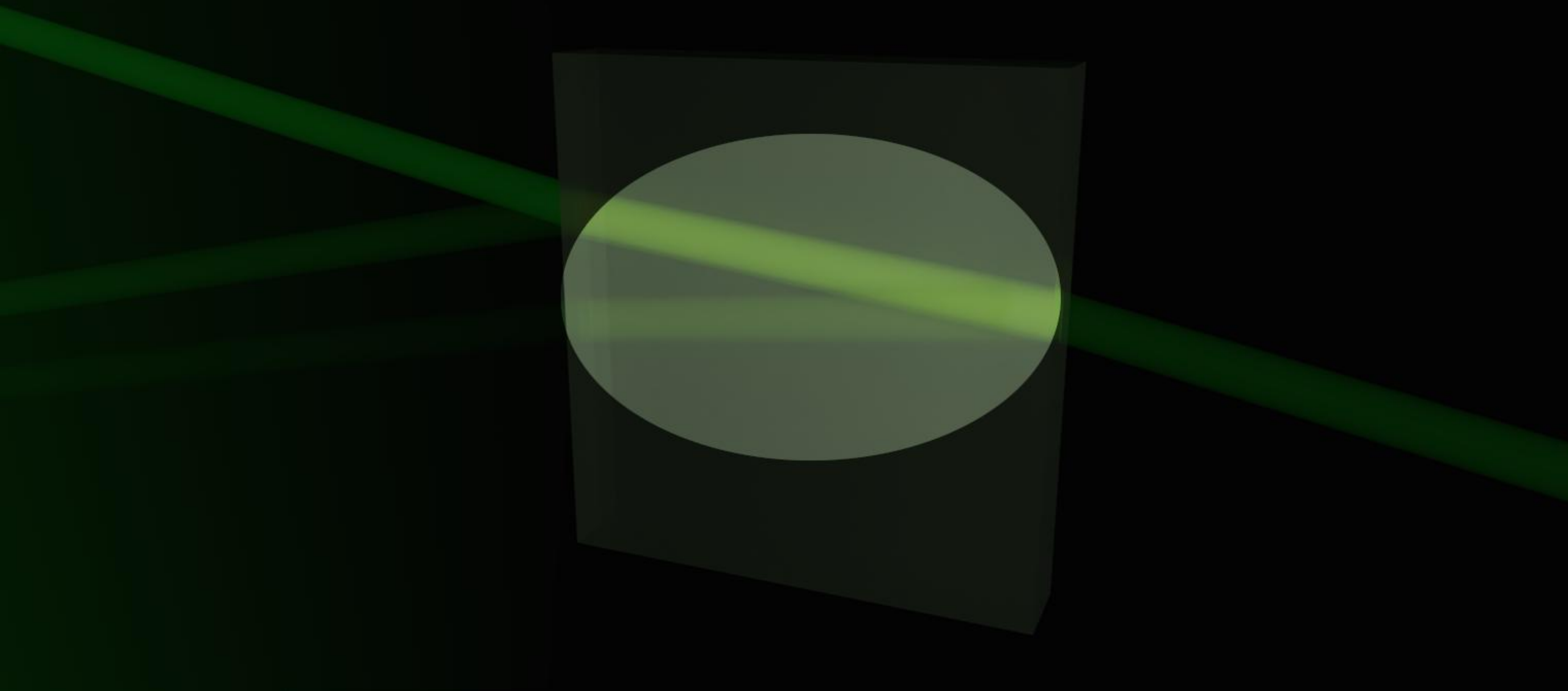
**Complementary techniques:**

Raman, Electron Energy Loss Spectroscopy (EELS), Extended X-ray Absorption Fine Structure (EXAFS), XPS, Auger, SIMS, XRD, SFG.

# Light scattering



# Light scattering



# Light scattering

## Sir Chandrasekhara Venkata Raman

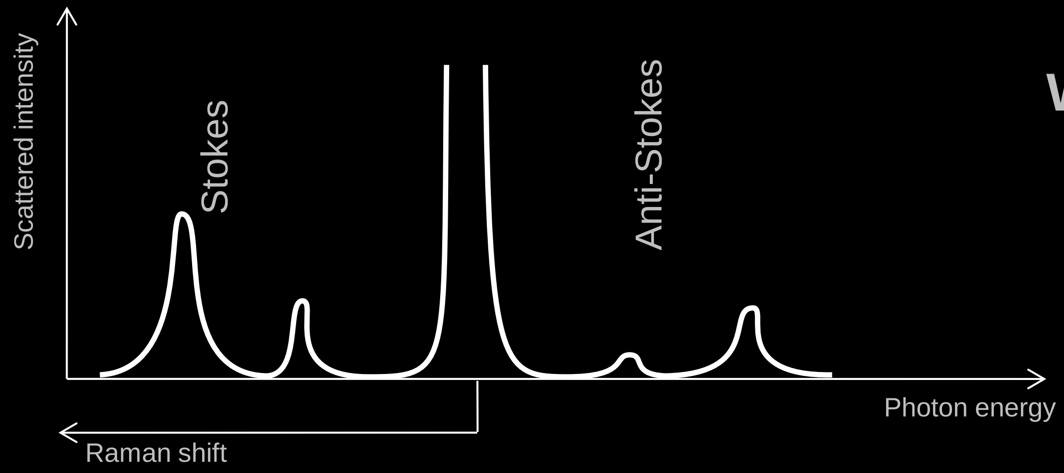
The Nobel Prize in Physics 1930 was awarded to Sir Venkata Raman *"for his work on the scattering of light and for the discovery of the effect named after him"*.

The Nobel Foundation

## Sir Kariamanikkam Srinivasa Krishnan

Co-discoverer of Raman scattering, for which his mentor C. V. Raman was awarded the 1930 Nobel Prize in Physics

# Raman spectroscopy

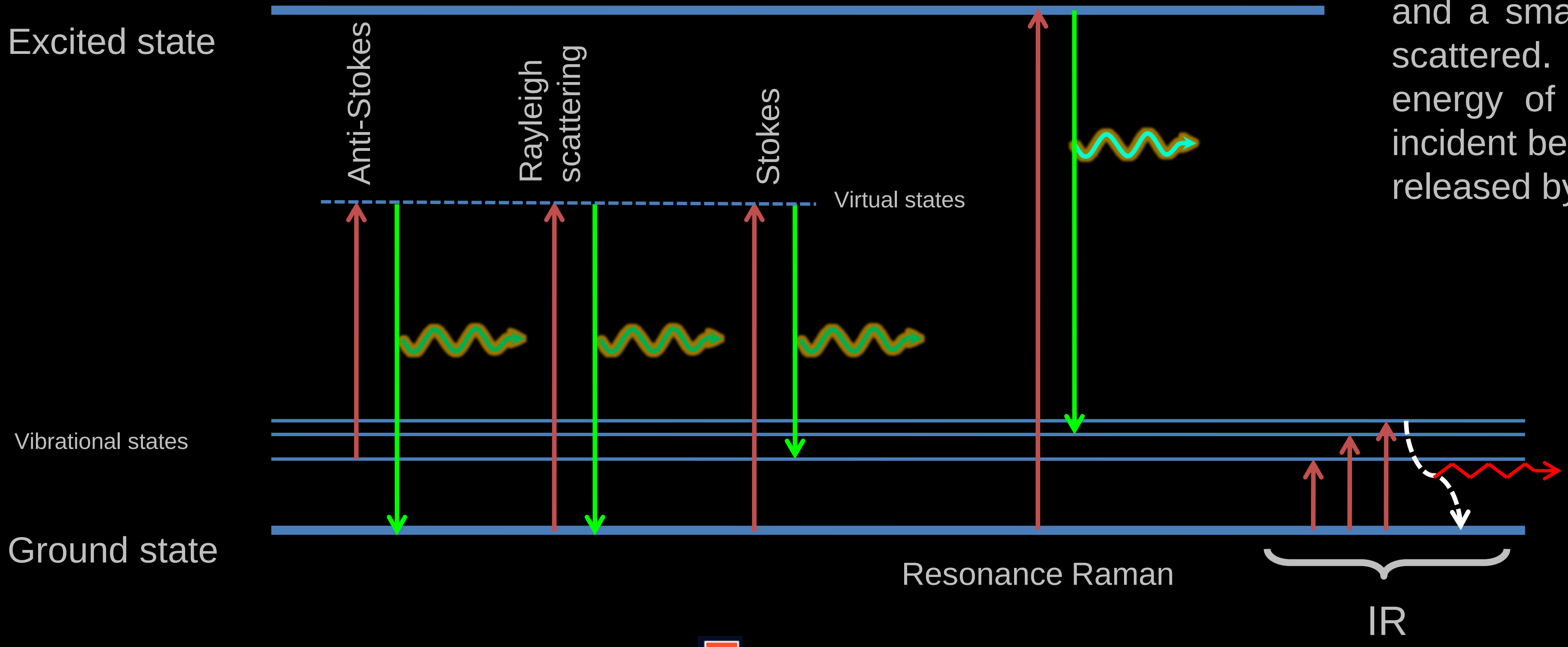


**What is measured:**

The light inelastically scattered by the material.

**Basic principle:**

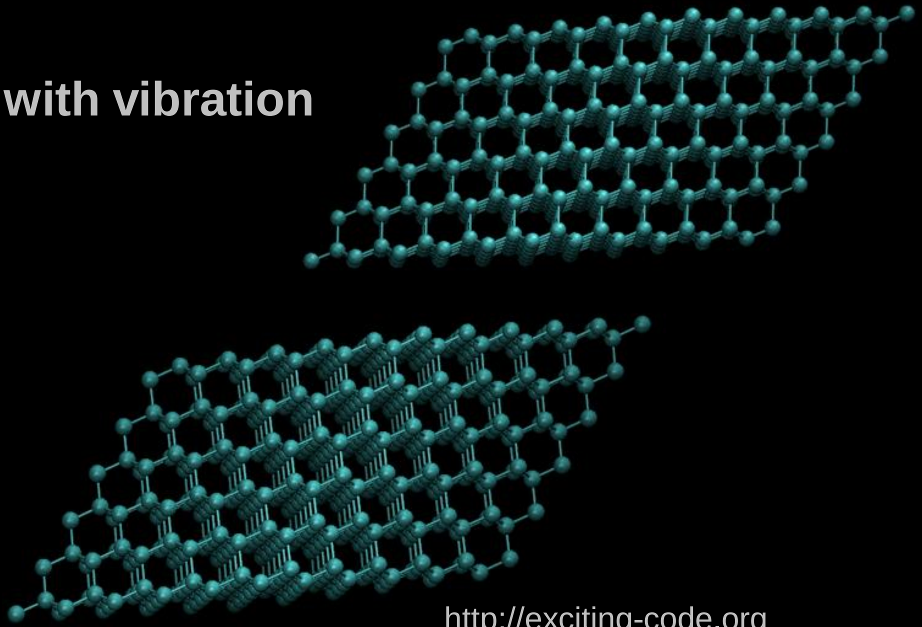
The impinging light couples with the lattice vibrations (phonons) of the material, and a small portion of it is inelastically scattered. The difference between the energy of the scattered light and the incident beam is the energy absorbed or released by the phonons.



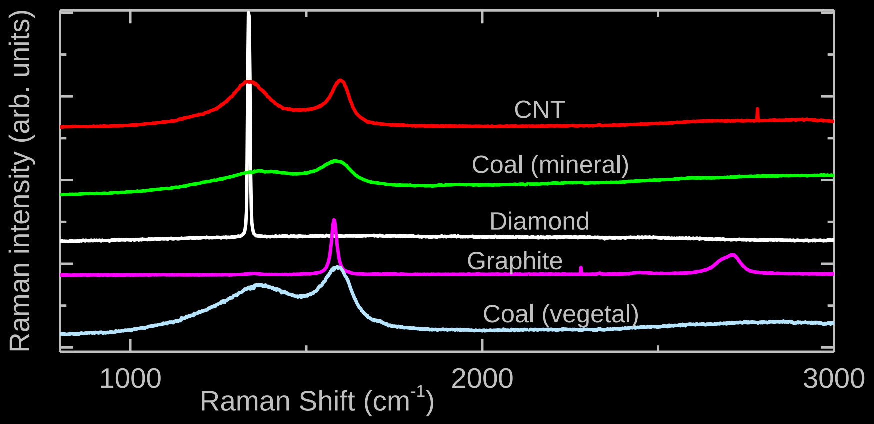
# Raman spectroscopy

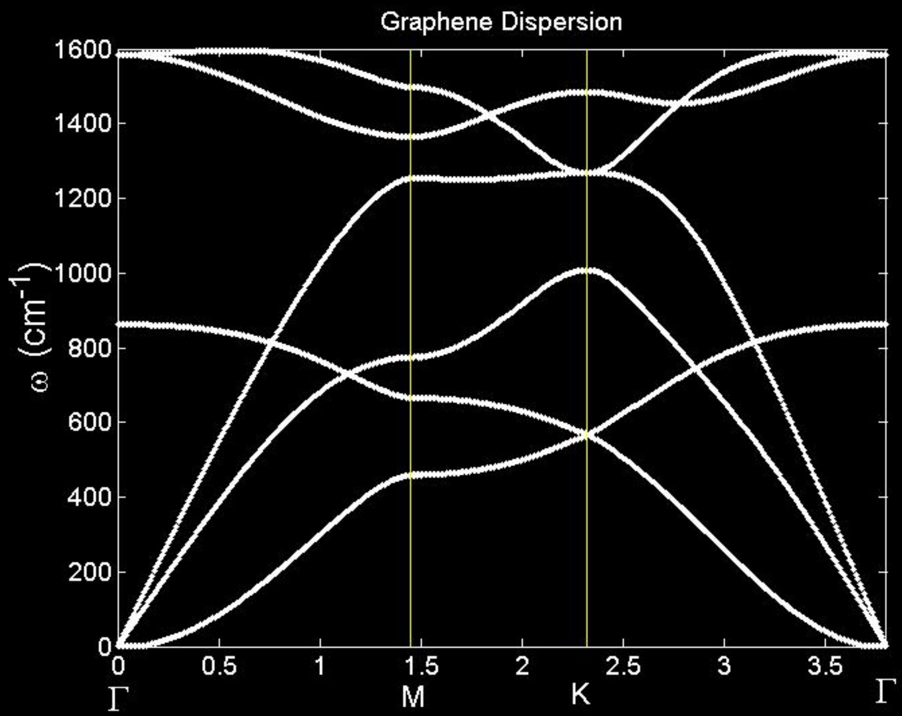
**Impinging light couples with vibration modes of the material:**

- Phonons
- Molecular vibrations



http://exciting-code.org

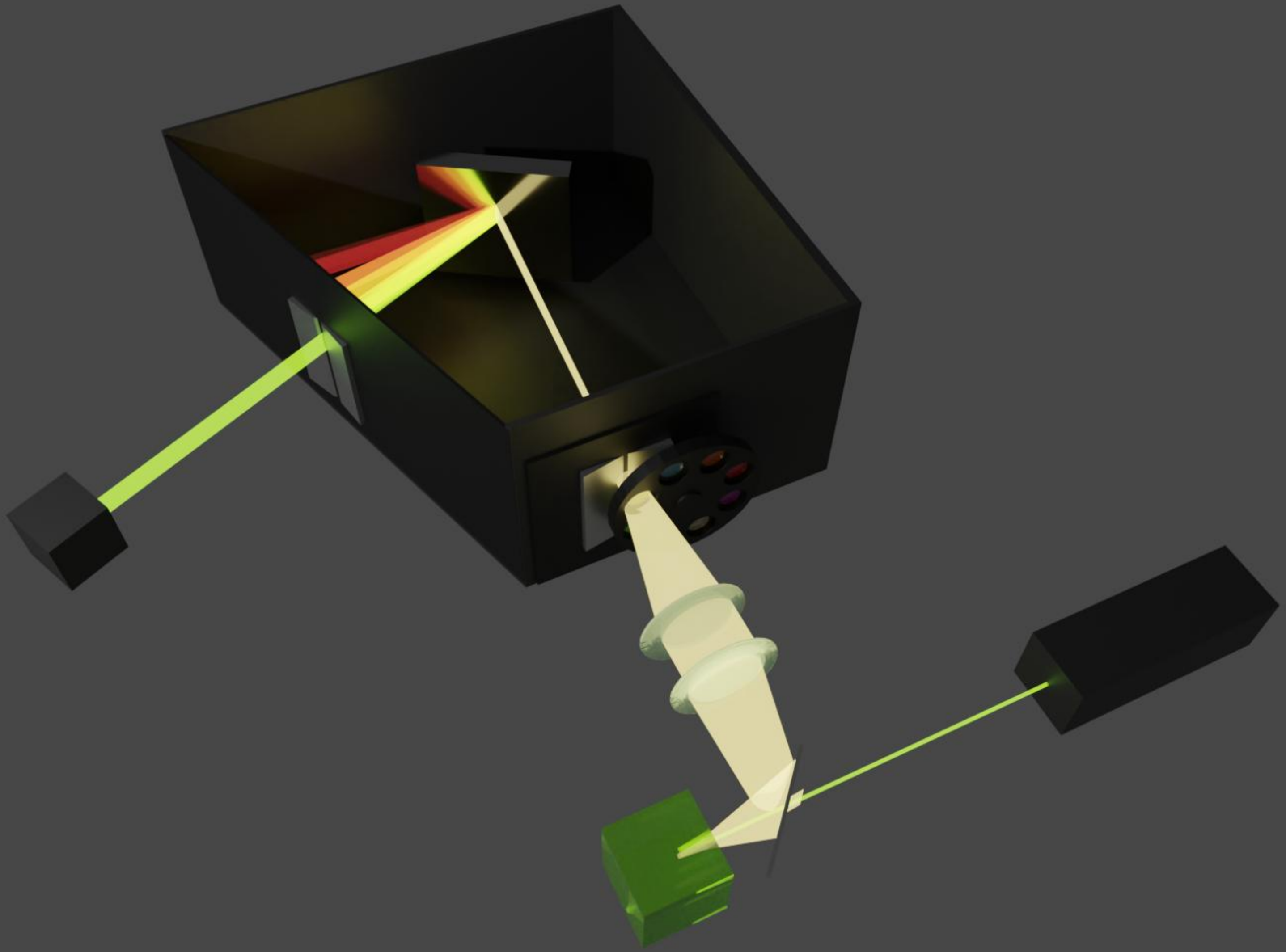




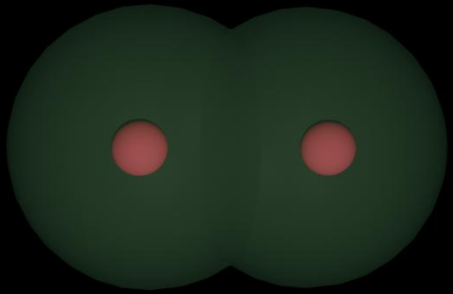
http://flex.phys.tohoku.ac.jp/~pourya

# Raman spectroscopy

**Instrumentation:**



# Raman spectroscopy



$$\mathbf{E} = \mathbf{E}_0 \cdot \cos\left(2\pi \cdot \nu_0 \cdot \mathrm{t}\right)$$

$$\mathbf{p} = \mathbf{0}$$

$$\mathbf{p} = \alpha \cdot \mathbf{E_0} \cdot \cos\left(2\pi \cdot \nu_0 \cdot \mathrm{t}\right)$$

# Raman spectroscopy

The α tensor is dependent on the shape, strength, and dimensions of the chemical bond. Since chemical bonds change during vibrations, α is dependent on the vibrations of the molecule:

$$\alpha_k = \alpha_0 + \sum_k \alpha'_k \left(\frac{\partial\alpha}{\partial Q_k}\right)_0 \cdot Q_k$$

$$Q_k = Q_{k0} \cdot \cos\left(2\pi \cdot \nu_k \cdot t + \varphi_k\right)$$

$$\alpha_k = \alpha_0 + \alpha'_k \cdot Q_{k0} \cdot \cos\left(2\pi \cdot \nu_k \cdot t + \varphi_k\right)$$

$$\mathbf{p} = \alpha_0 \cdot \mathbf{E}_0 \cdot \cos\left(2\pi \cdot \nu_0 \cdot \mathrm{t}\right) +$$

$$+ \frac{1}{2} \cdot \alpha'_k \cdot Q_{k0} \cdot \mathbf{E}_0 \cdot \left[\cos\left(2\pi \cdot t \cdot (\nu_0 + \nu_k) + \varphi_k\right) +\right.$$

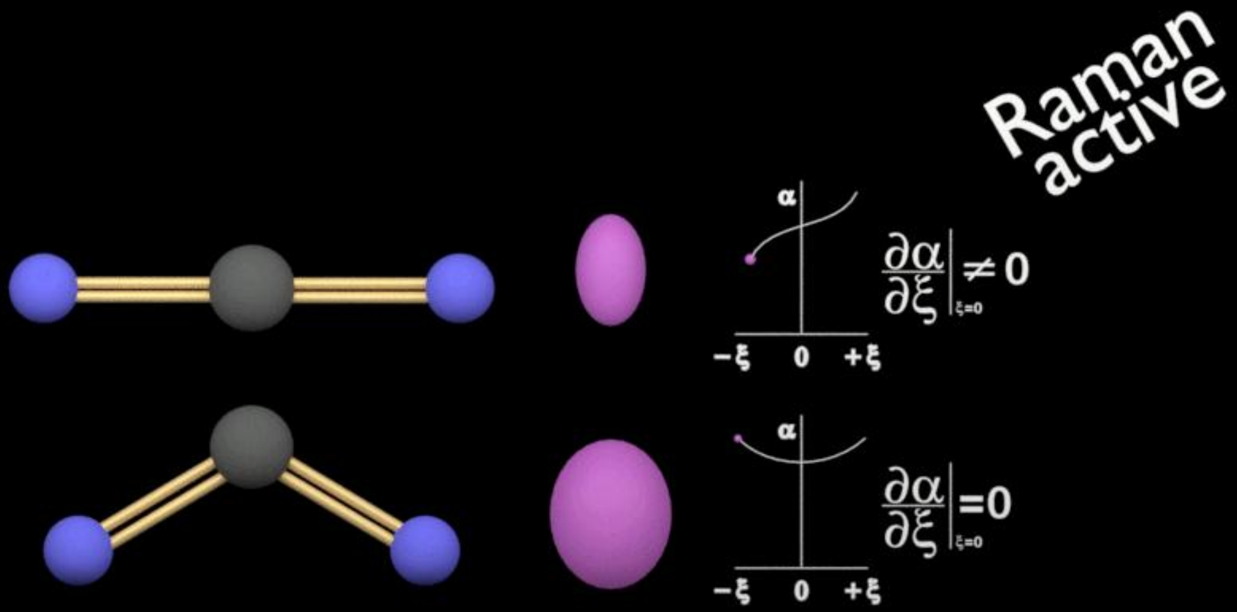
$$\left. + \cos\left(2\pi \cdot t \cdot (\nu_0 - \nu_k) - \varphi_k\right)\right]$$

$$\alpha'_k = \left(\frac{\partial\alpha}{\partial Q_k}\right)_0 \neq 0$$

the dipole oscillates with three frequencies simultaneously, corresponding to the three possible scattering modes (Rayleigh, Stokes Raman and anti-Stokes Raman)

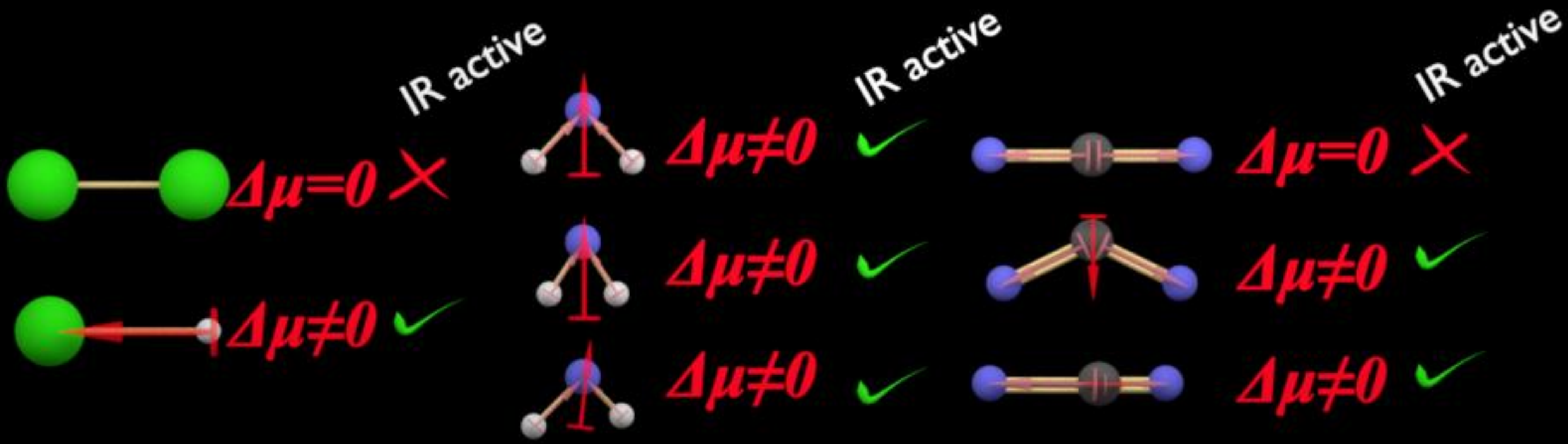
# Raman spectroscopy

$$\alpha'_k = \left(\frac{\partial \alpha}{\partial Q_k}\right)_0 \neq 0$$



# Fourier Transform IR spectroscopy (FTIR)

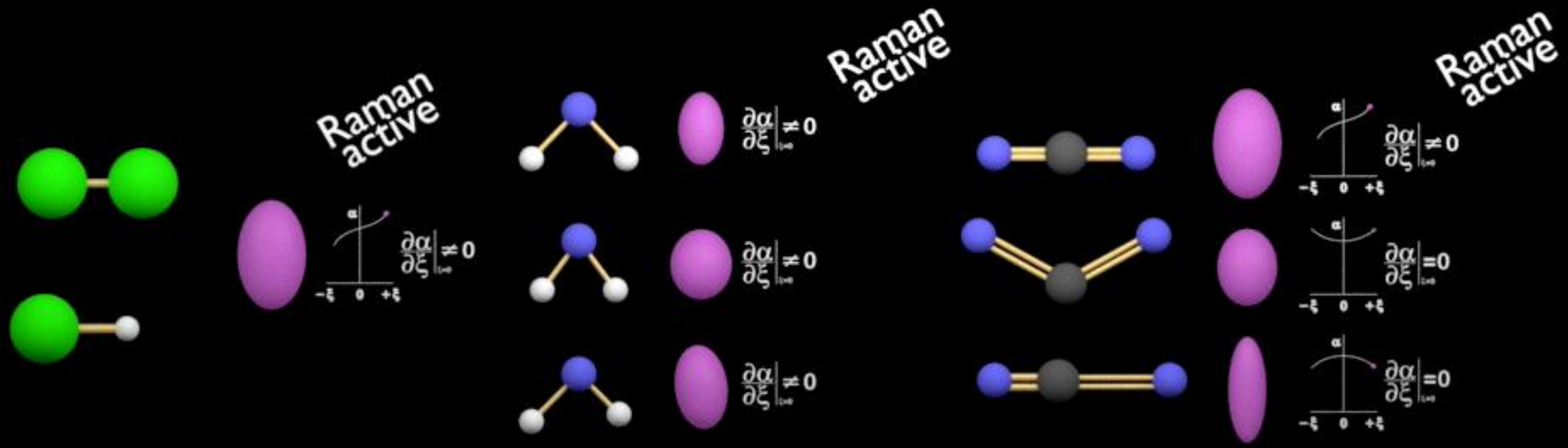
## IR active vibrations



# Raman spectroscopy

## Raman active vibrations

The intensity of the Raman scattering linked to a vibrational state depends on the change in the polarizability tensor



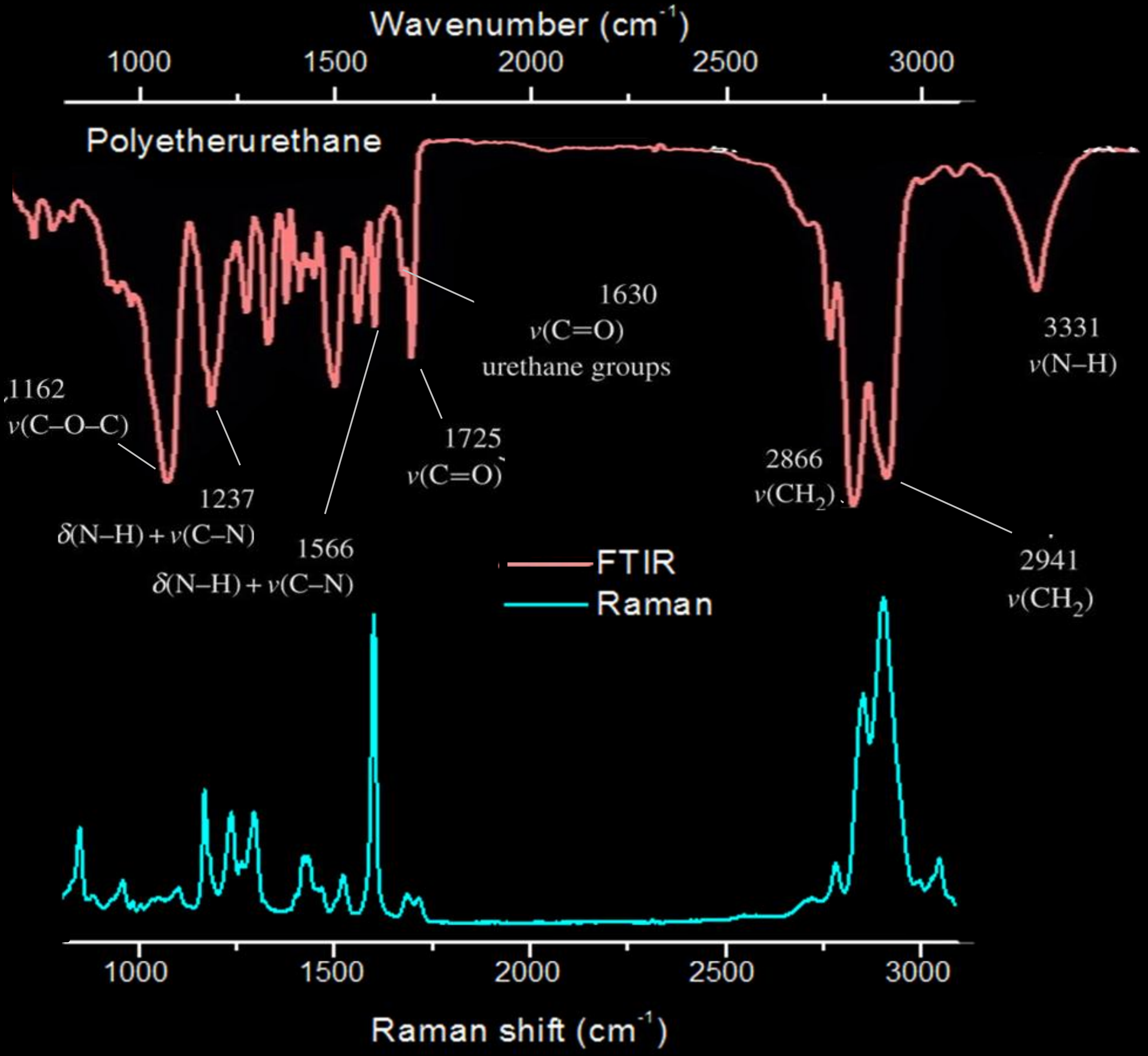
# Raman spectroscopy compared to FTIR

FTIR and Raman:

**The two techniques are complementary** (different selection rules).

$$\Delta\mu \neq 0$$

$$\alpha'_k = \left(\frac{\partial \alpha}{\partial Q_k}\right)_0 \neq 0$$



# Raman spectroscopy compared to FTIR

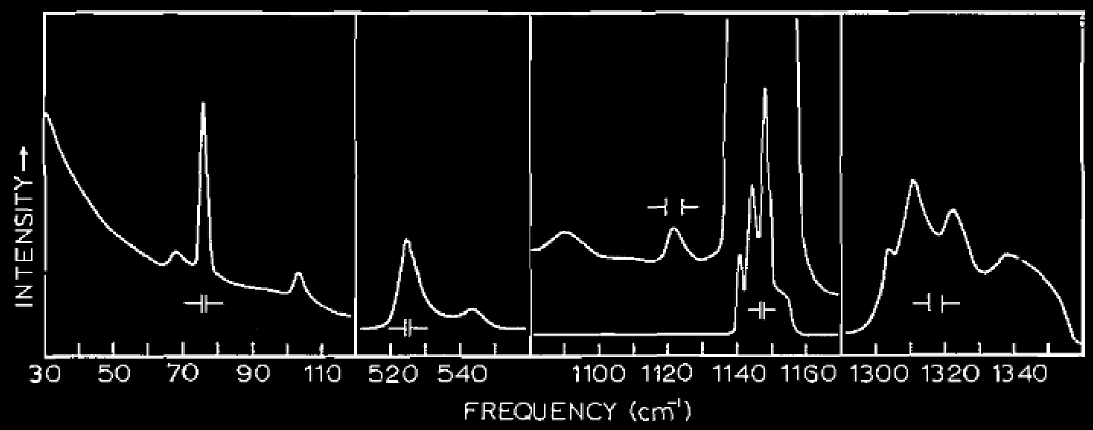


Fig. 1. Raman spectrum of crystalline $SO_2$ at 9 °K.

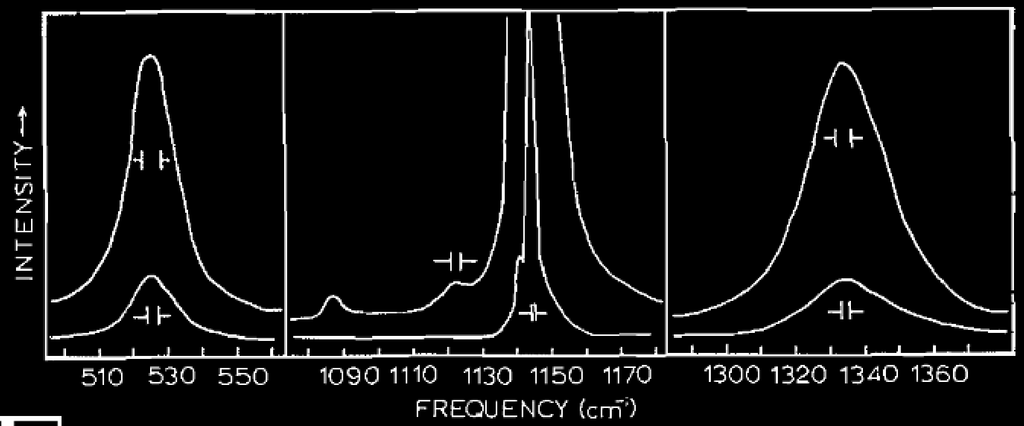
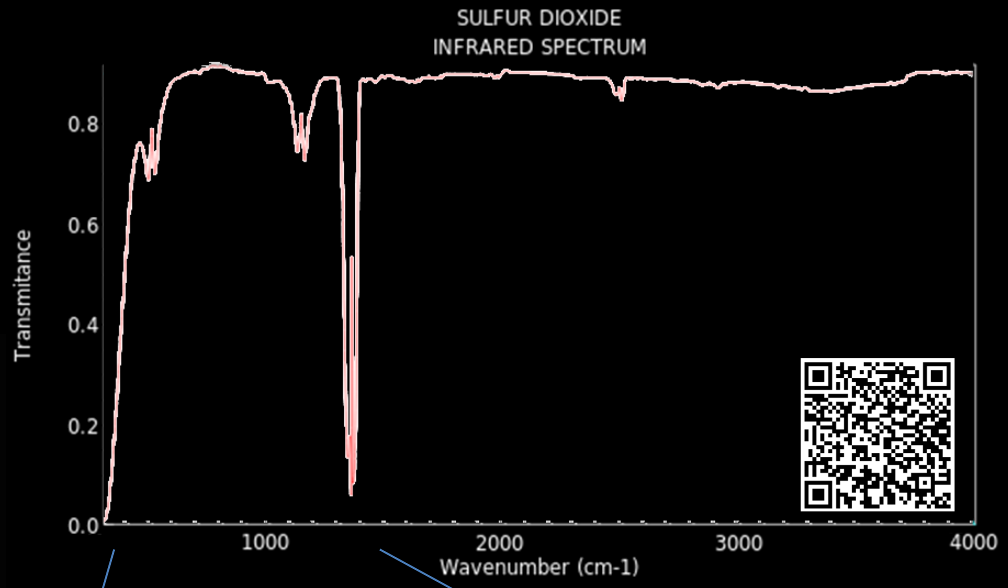
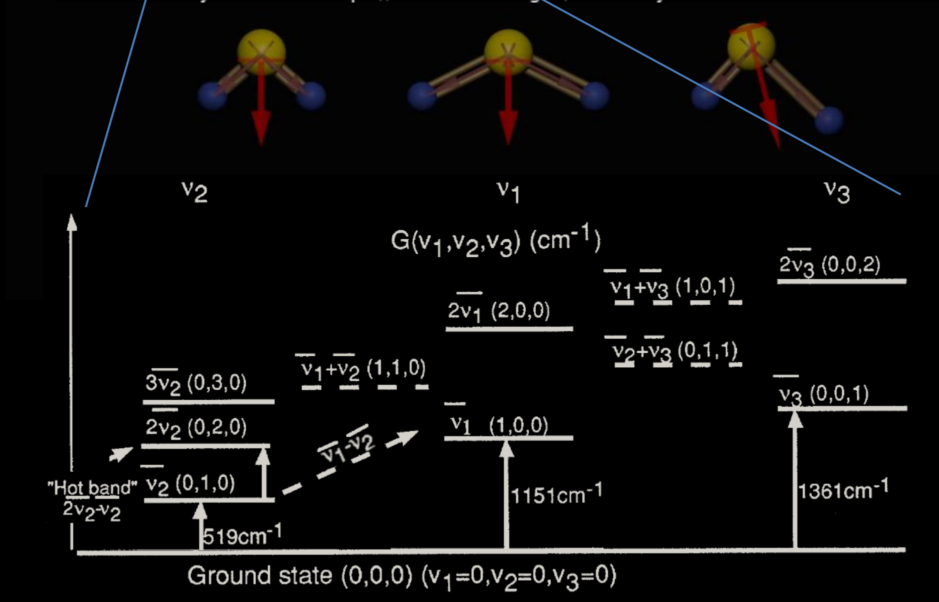


Fig. 2. Raman spectrum of liquid $SO_2$ at 225 °K.

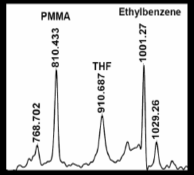
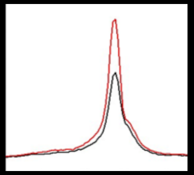
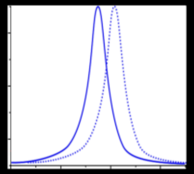
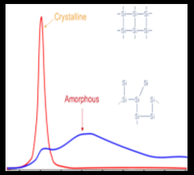
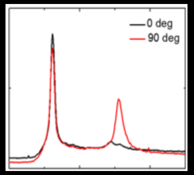
Canadian Journal of Chemistry, **43** (1965)



NIST Chemistry WebBook (https://webbook.nist.gov/chemistry)



Journal of Near Infrared Spectroscopy, **6** (1998)

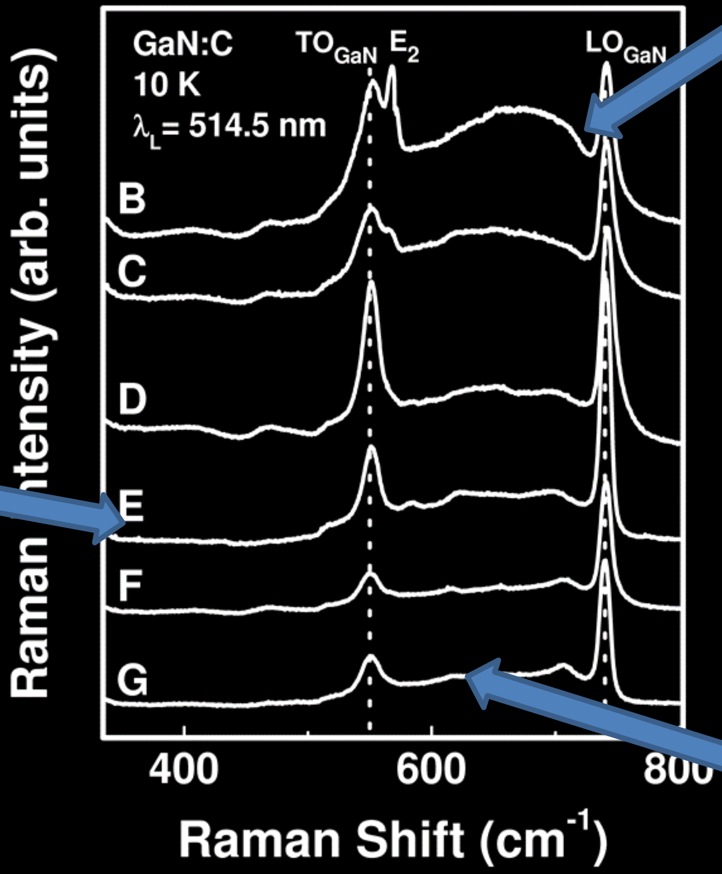
| | Studying the … | … we can estimate … |
|---|---|---|
|  | Characteristic Raman frequencies | Identity and composition of materials |
|  | Raman peak intensity | Volume of material probed |
|  | Raman peak frequency shift | Strain, stress, crystal lattice distortion |
|  | Raman peak width | Crystallinity of material |
|  | Raman peak polarization dependency | Crystal orientation and symmetry |

# Raman spectroscopy

**Molecular and crystalline structure characterization**

Presence of N vacancies yields poor crystallinity

Substitutional C fills N vacancies improving the crystallinity



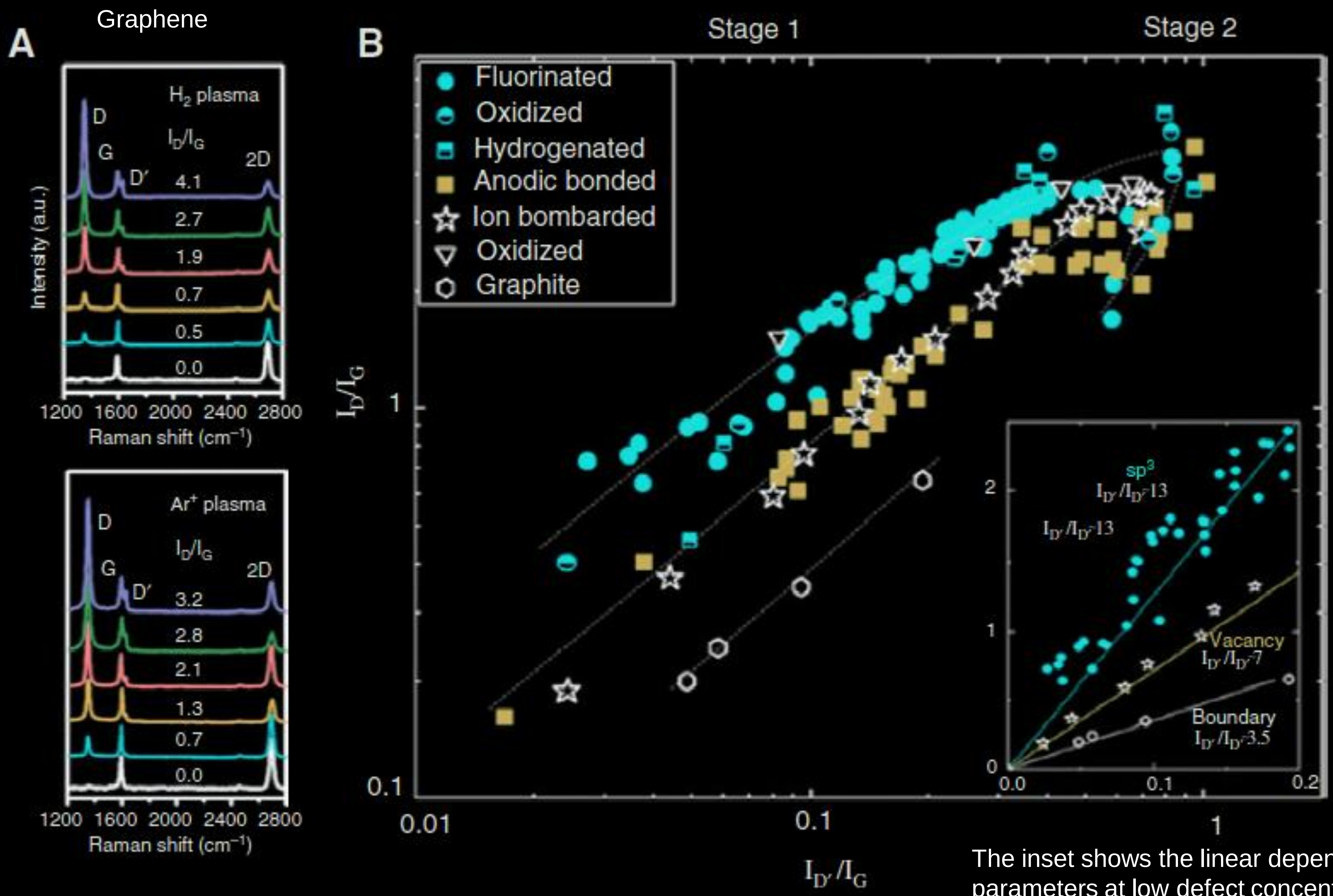
**C content**

C incorporates interstitially causing a degradation of the crystal lattice

PHYSICAL REVIEW B **68**, 155204 (2003)

# Raman spectroscopy

## Crystalline structure and defect characterization

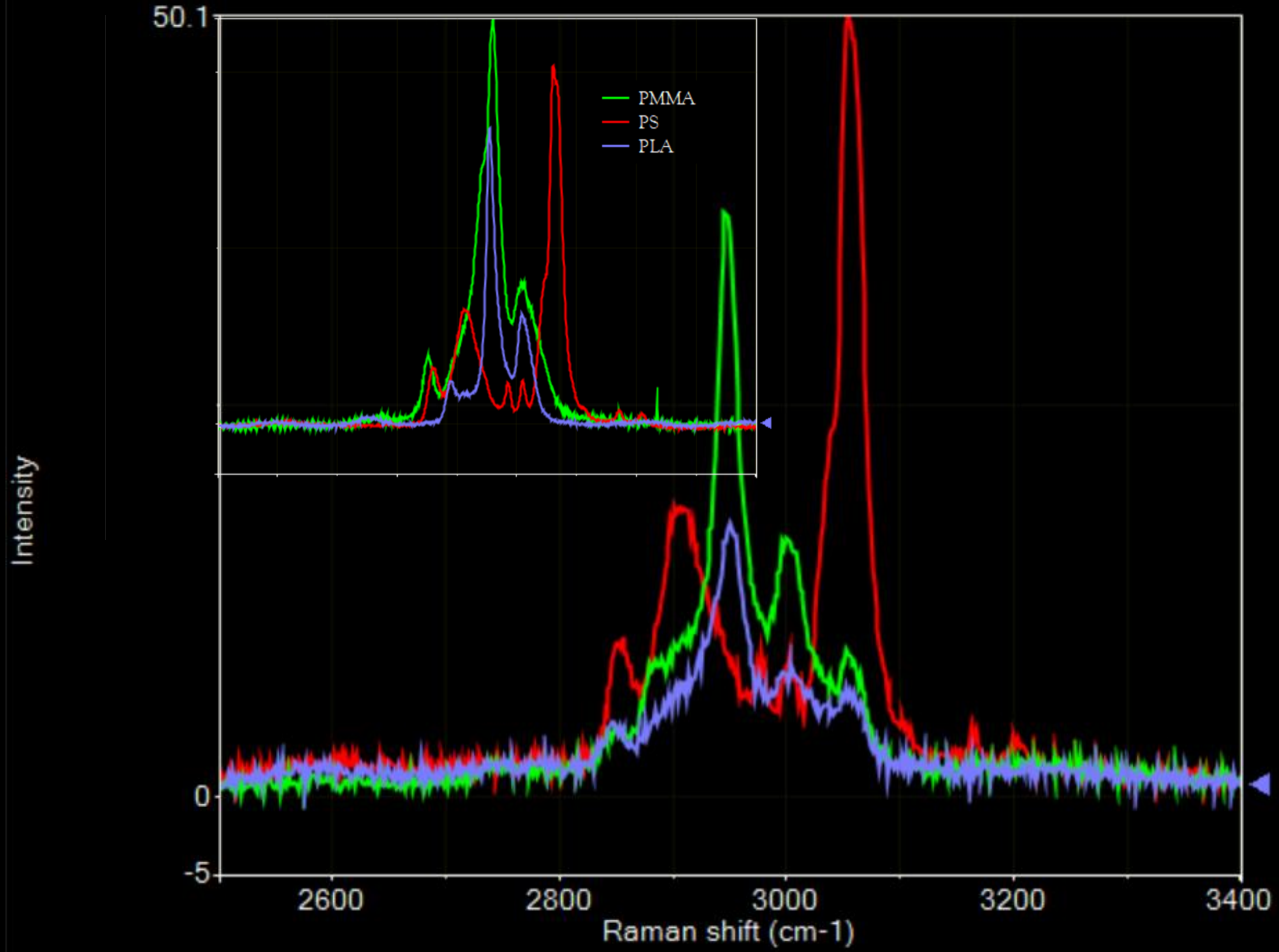


The inset shows the linear dependence between the two parameters at low defect concentration.

*Nanophotonics* **6,** 1219 (2017)

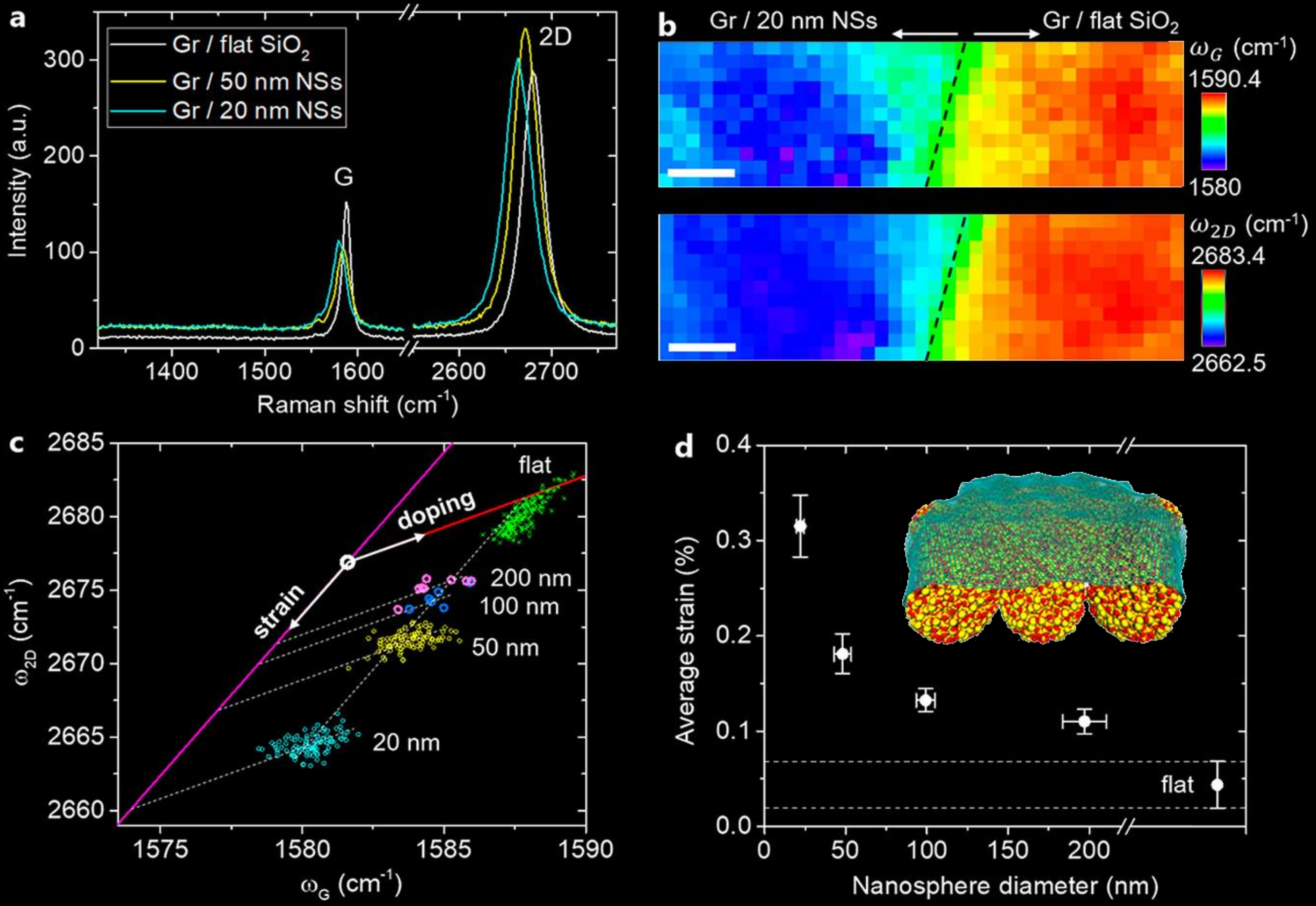
# Raman spectroscopy

## Composition and distribution of compound polymer components



# Raman spectroscopy

**Strain/stress**

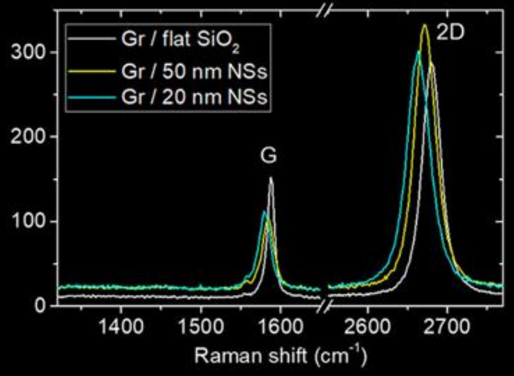


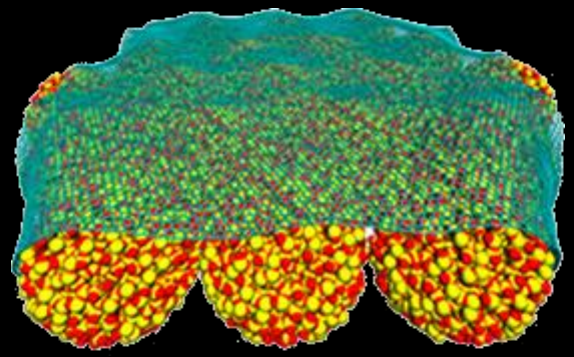
*Nano Lett.* **18**, 2098 (2018)

# Raman spectroscopy

## Primary Strengths:

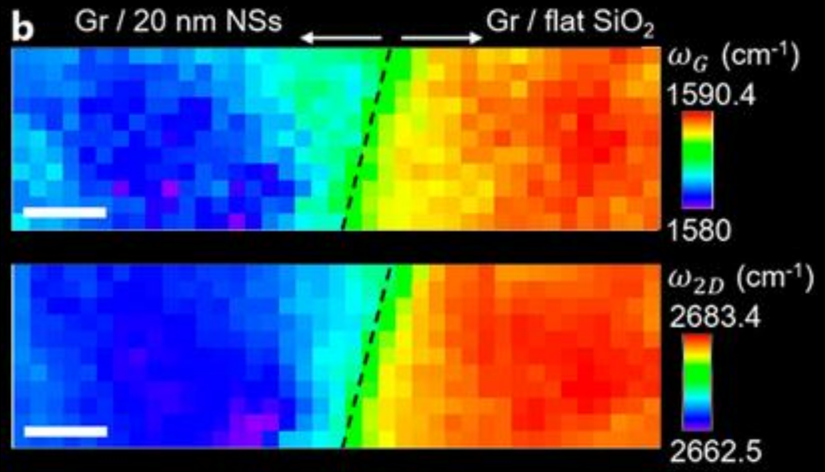
- Very little sample preparation.
- Structural characterization.
- Non-destructive technique.
- Chemical information.
- Complementary to FTIR.





## Primary Limitations:

- Expensive apparatus (for high spectral/spatial resolution and sensitivity).
- Weak signal, compared to fluorescence.
- Limited spatial resolution (diffraction limited).



## Complementary techniques:

FTIR, EELS, Mass spectroscopy, EXAFS, XPS, AES, SIMS, XRD, SFG.

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# Optical Characterization Methods

## Part II

Julio A. N. T. Soares

Materials Research Laboratory
University of Illinois at Urbana-Champaign

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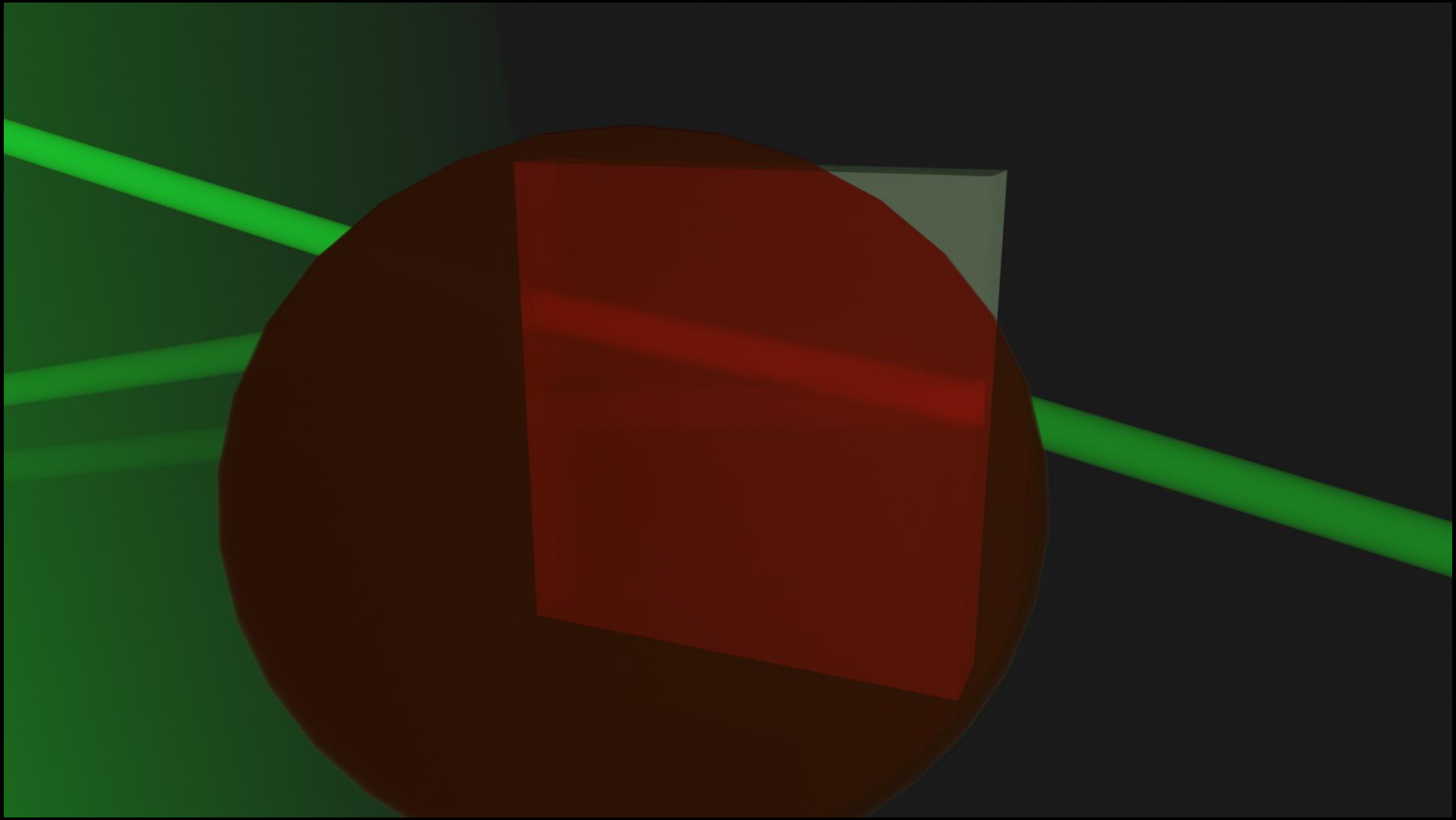
go.Illinois.edu/AMC2025

The Grainger College of Engineering
UNIVERSITY OF ILLINOIS URBANA-CHAMPAIGN

# Luminescence



# Luminescence

**Lifetime: Phosphorescence, fluorescence**

**Mechanism: Photoluminescence, bioluminescence, chemoluminescence, thermoluminescence, piezoluminescence, etc.**

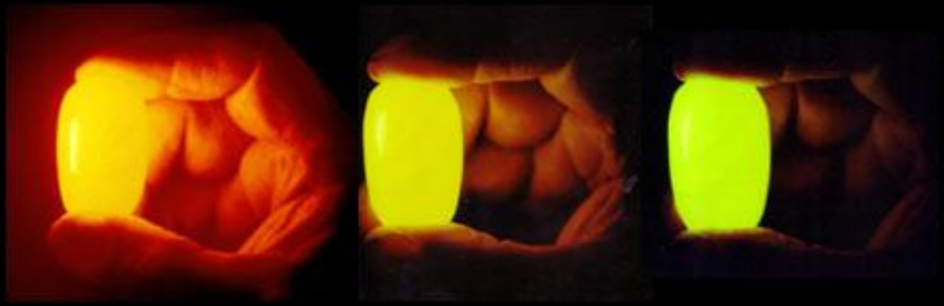
Disney Pixar

Charles Hedgcock ©

Profilephotocovers.com

Radim Schreiber

Profilephotocovers.com



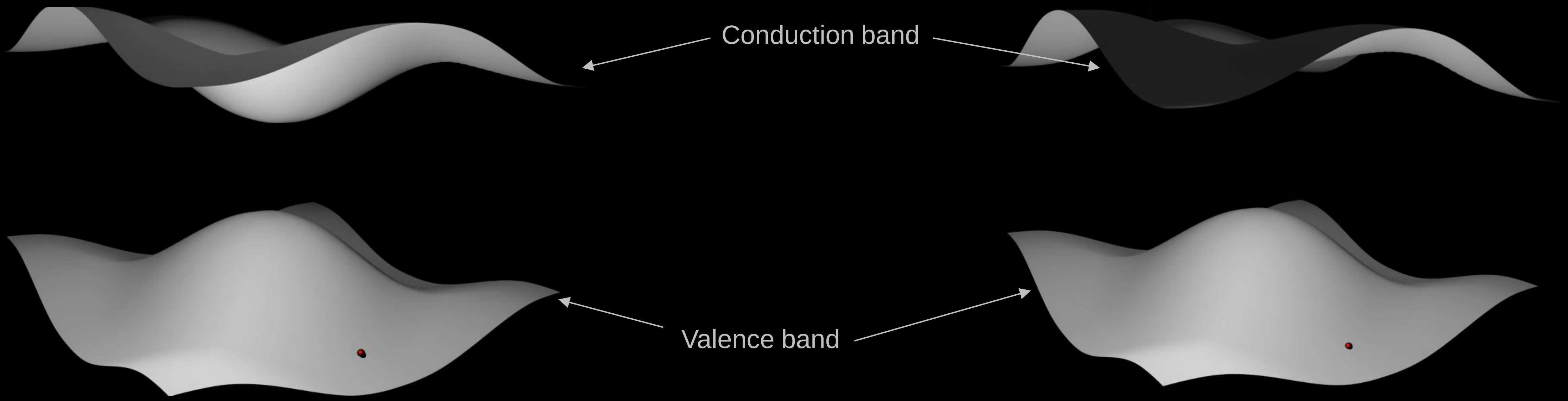
Trevor Morris

# Photoluminescence

**What is measured:**

The emission spectra of materials due to radiative recombination following photo-excitation.

**Basic principle:**

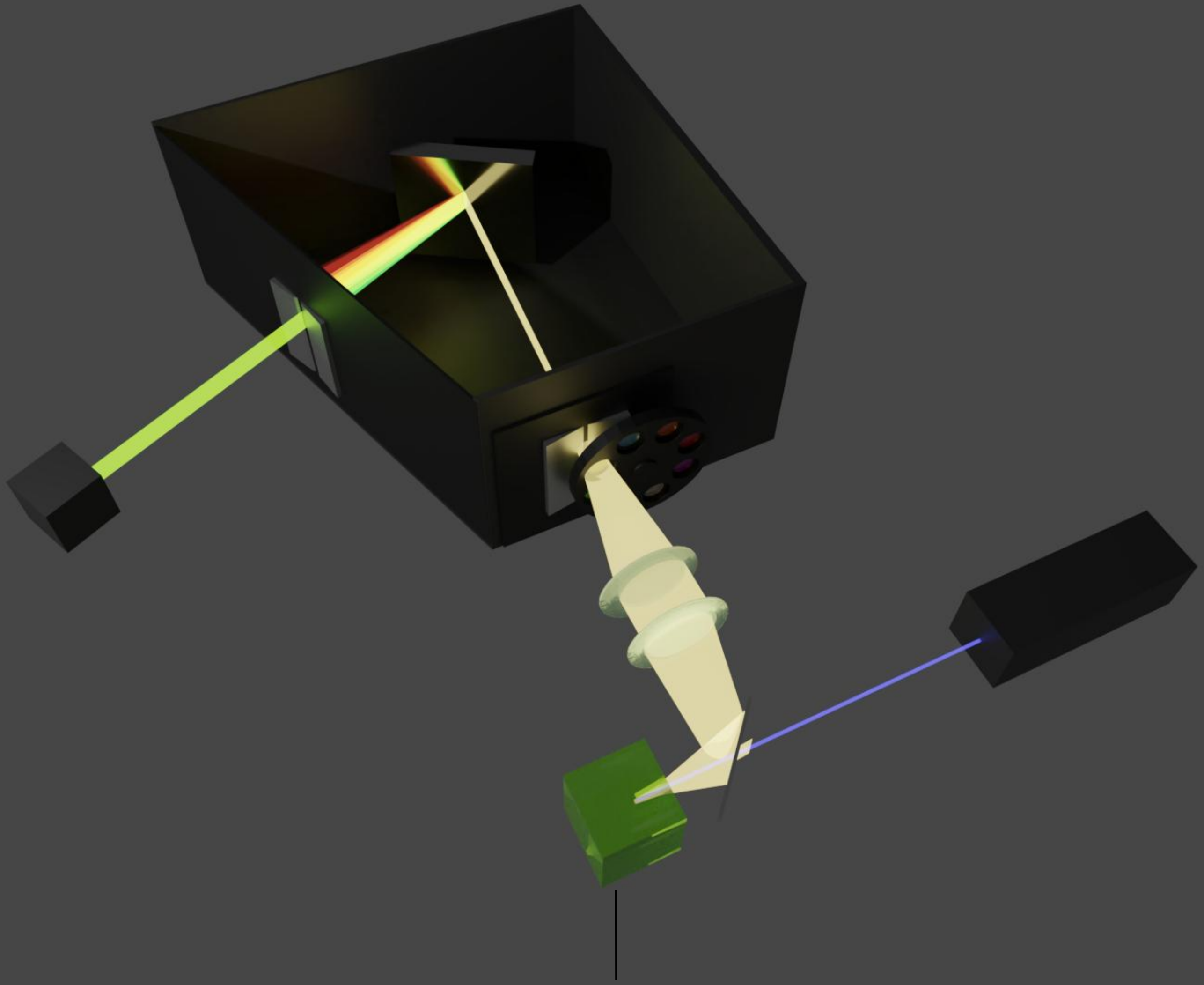


direct band gap

indirect band gap

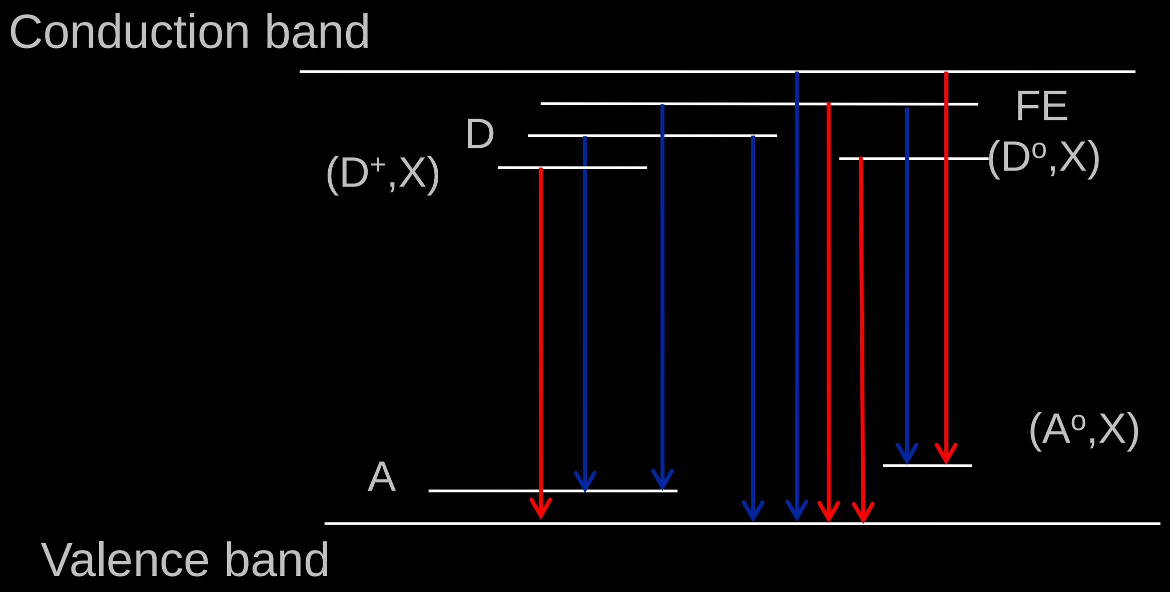
# Photoluminescence

## Instrumentation:



# Photoluminescence

## Defect luminescence in GaAs



Conduction band

FE

D

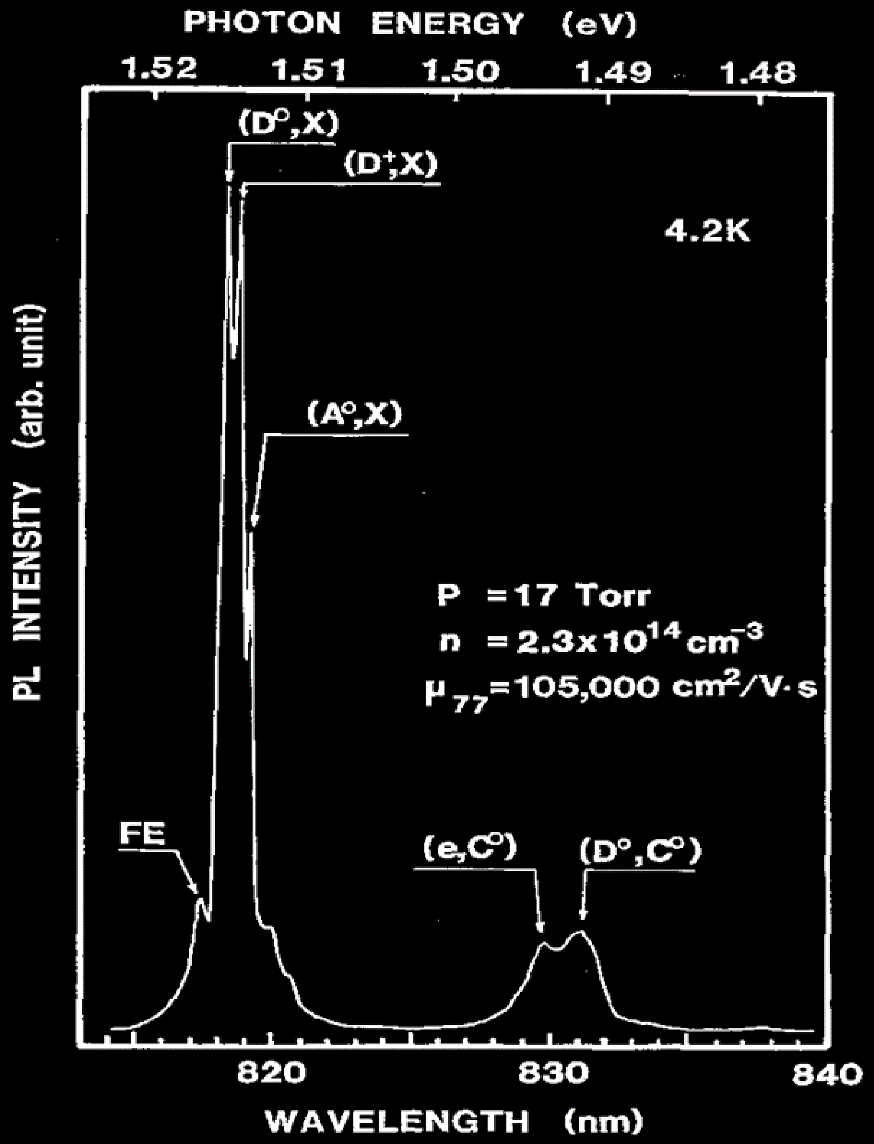
$(D^+,X)$

$(D^o,X)$

$(A^o,X)$

A

Valence band



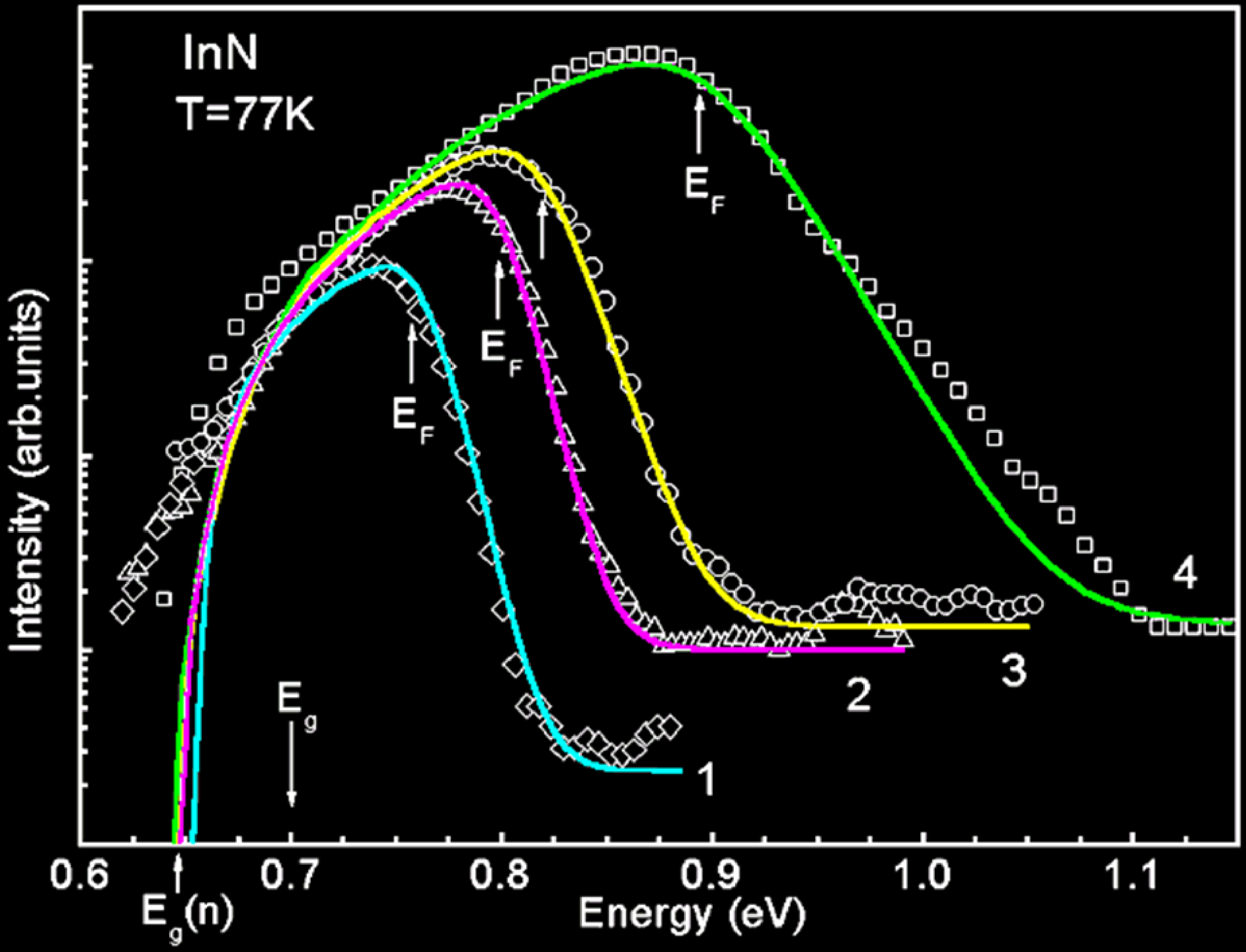
Jap. J. App. Phys. **23**, L100 (1984)

# Photoluminescence

## Carrier concentration

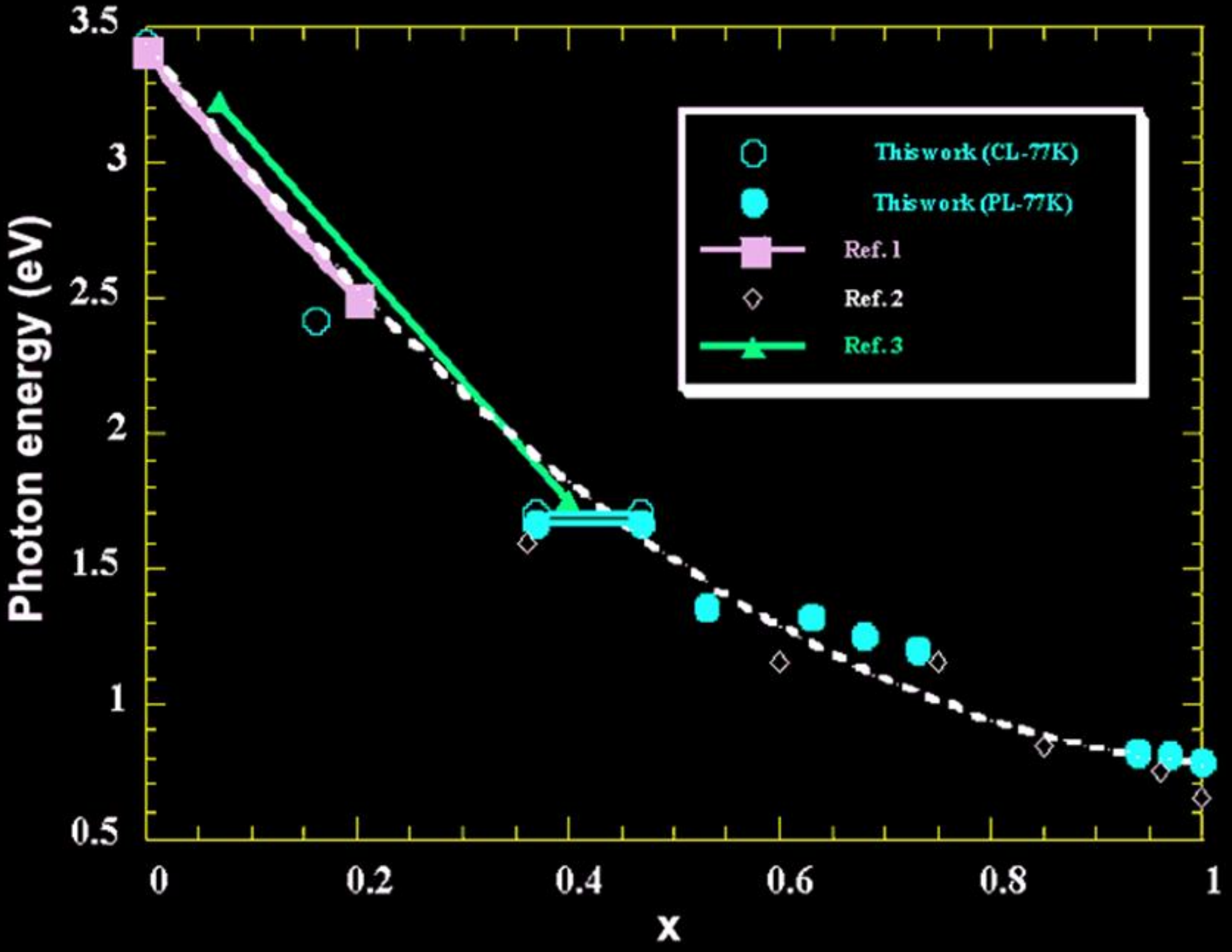
**Photoluminescence spectra of InN layers with different carrier concentrations.**

1 - n = $6 \times 10^{18}$ cm$^{-3}$ (MOCVD);
2 - n = $9 \times 10^{18}$ cm$^{-3}$ (MOMBE);
3 - n = $1.1 \times 10^{19}$ cm$^{-3}$ (MOMBE);
4 - n = $4.2 \times 10^{19}$ cm$^{-3}$ (PAMBE).



*Phys. Stat. Solidi* **(b) 230** (2002**b**), R4

# Photoluminescence



## Alloy composition

**In$_x$Ga$_{1-x}$N alloys. Luminescence peak positions of catodoluminescence and photoluminescence spectra vs. concentration *x*.**

The plots of luminescence peak positions can be fitted to the curve

$$E_g(x)=3.48 - 2.70x - bx(1-x)$$

with a bowing parameter of **b=2.3 eV**

Ref.1 - Wetzel., *Appl. Phys. Lett.* **73,** 73 (1998).
Ref.2 - V. Yu. Davydov., *Phys. Stat. Sol.* **(b) 230,** R4 (2002).
Ref.3 - O'Donnel., *J. Phys .Condens. Matt.* **13,** 1994 (1998).

Extracted from *Phys. Stat. Sol.* **(b) 234** (2002) 750

# Photoluminescence

Width and quality of semiconductor quantum wells.

3-QWs

100 nm GaAs cap

3nm InGaAsN QW

35 nm GaAs barrier

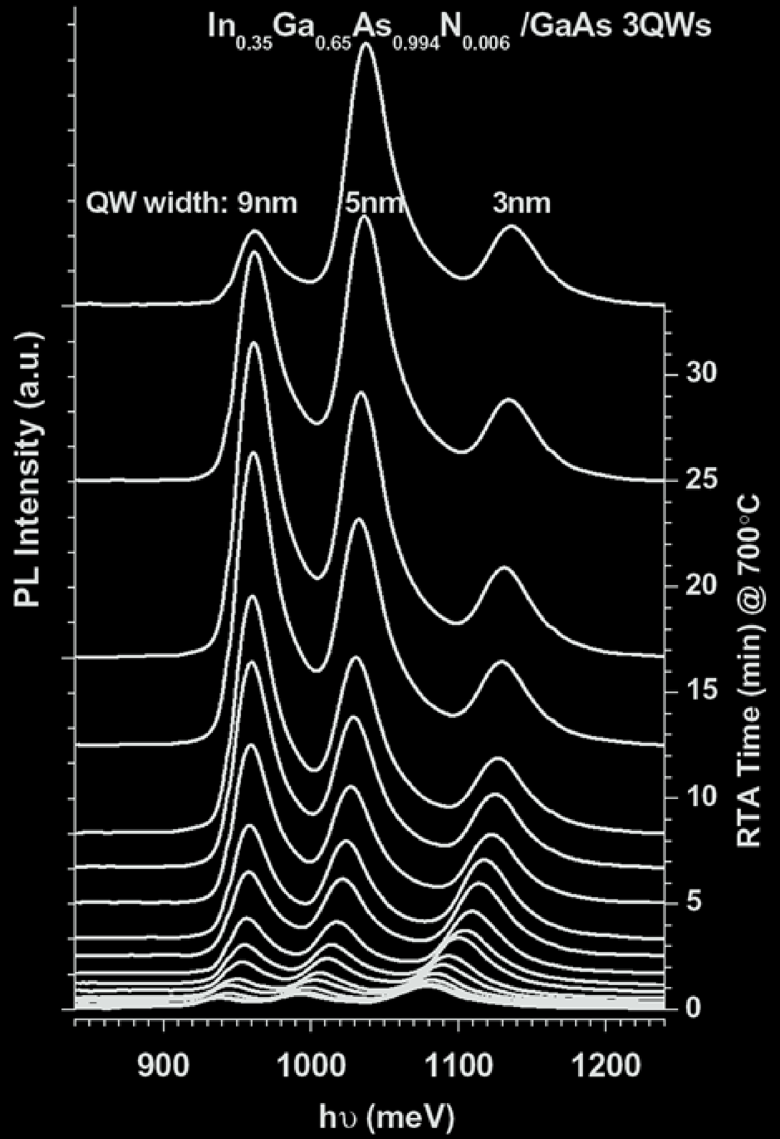
5nm InGaAsN QW

35 nm GaAs barrier

9nm InGaAsN QW

100 nm GaAs buffer

GaAs (001) SUB

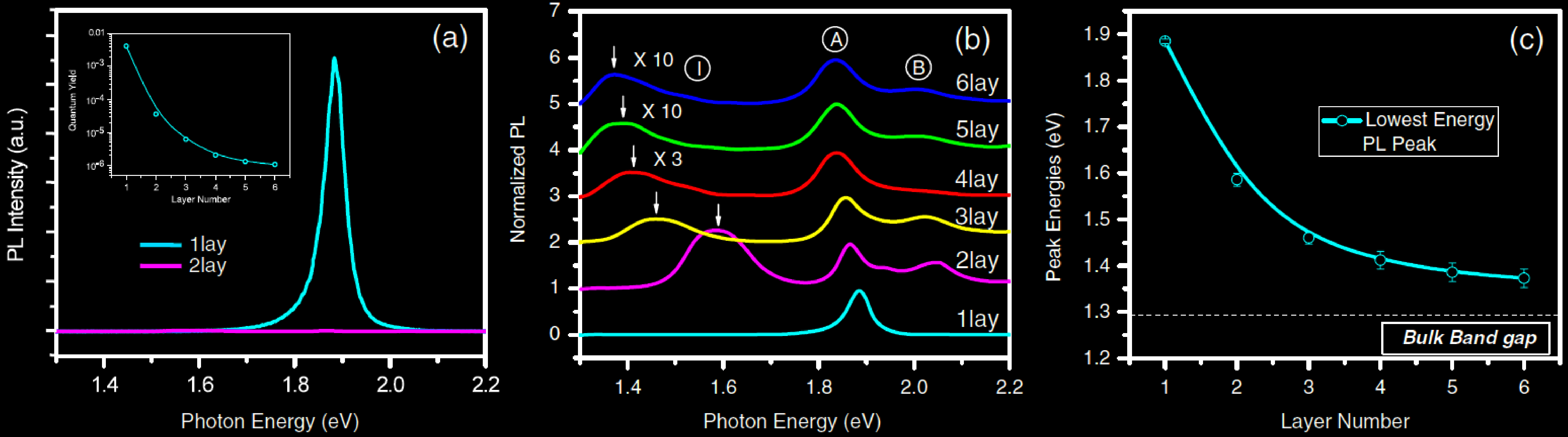


Journal of Crystal Growth 278 (2005) 259–263

# Photoluminescence

## Number of layers in 2D materials

a) PL spectra for mono- and bilayer $MoS_2$.
Inset: PL QY of thin layers for N = 1–6.
b) Normalized PL spectra by the intensity of peak A of thin layers of $MoS_2$ for N = 1–6. Feature I for N = 4–6 is magnified for clarity.
c) Band-gap energy of thin layers of $MoS_2$, inferred from the energy of the PL feature I for N = 2–6 and from the energy of the PL peak A for N = 1. The dashed line represents the (indirect) band-gap energy of bulk $MoS_2$.



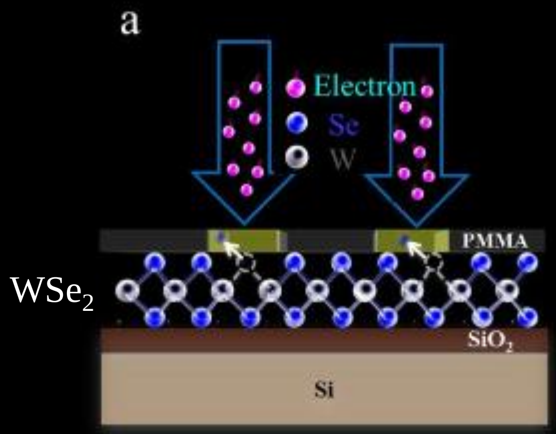
*Phys. Rev. Lett.* **105**, 136805 (2010)

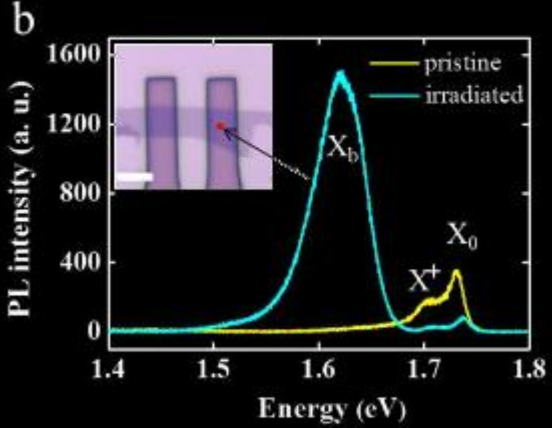
# Photoluminescence

## Defects in 2D materials

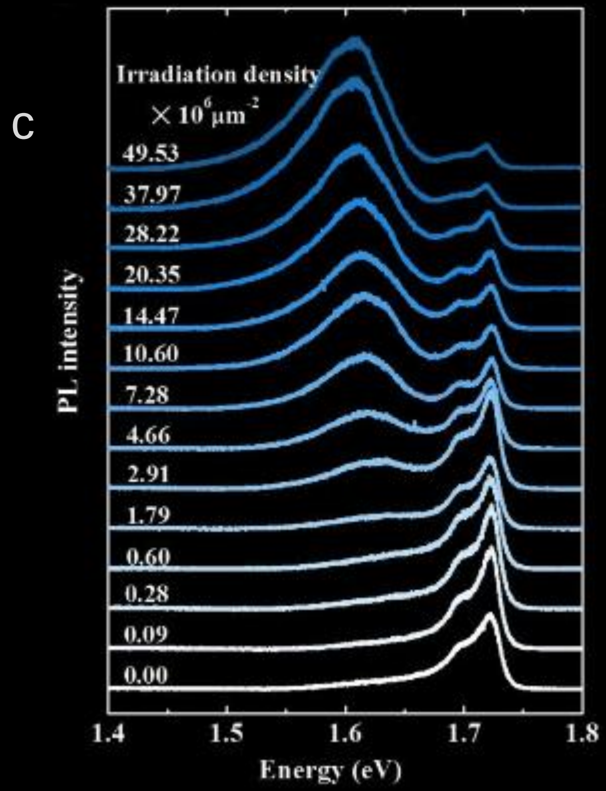
Defect induced PL emission.

a) Schematic diagram of electron beam irradiation on monolayer $WSe_2$ sample during the EBL process.
b) PL spectrum of pristine monolayer $WSe_2$ and monolayer $WSe_2$ after EBL.
The inset shows optical image of $WSe_2$ with PMMA patterned by EBL, scale bar is 5 μm
c) PL spectra of a pristine $WSe_2$ under different $e^-$ beam irradiation density.

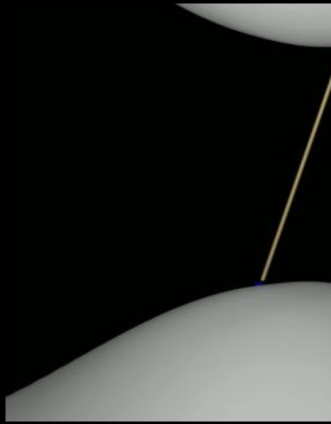




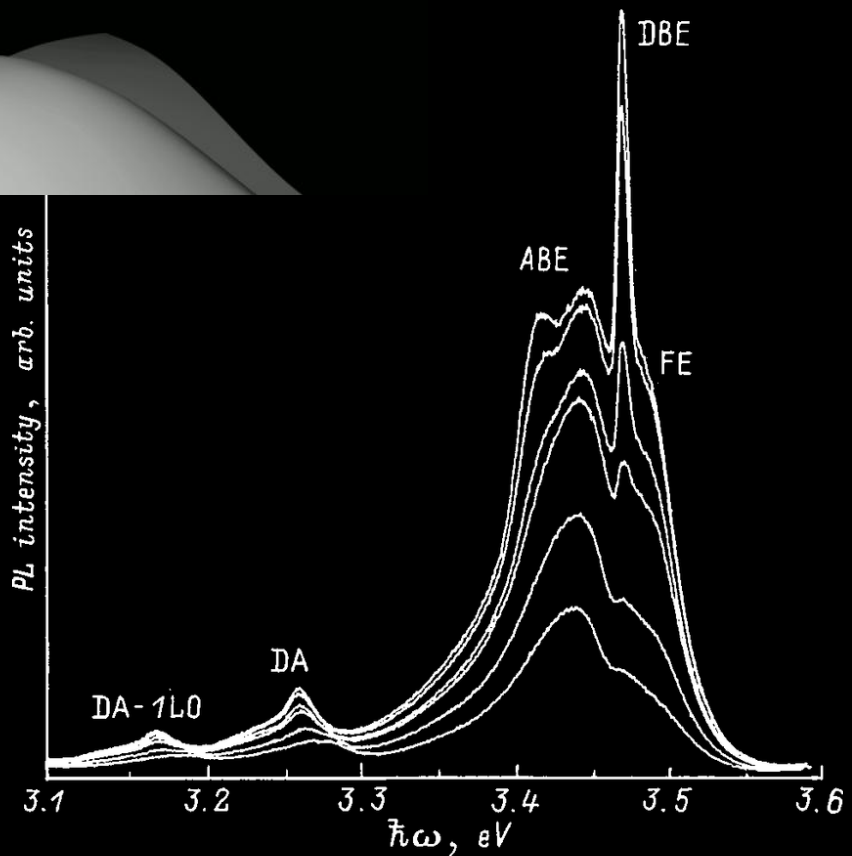
arXiv:1608.02043



# Photoluminescence



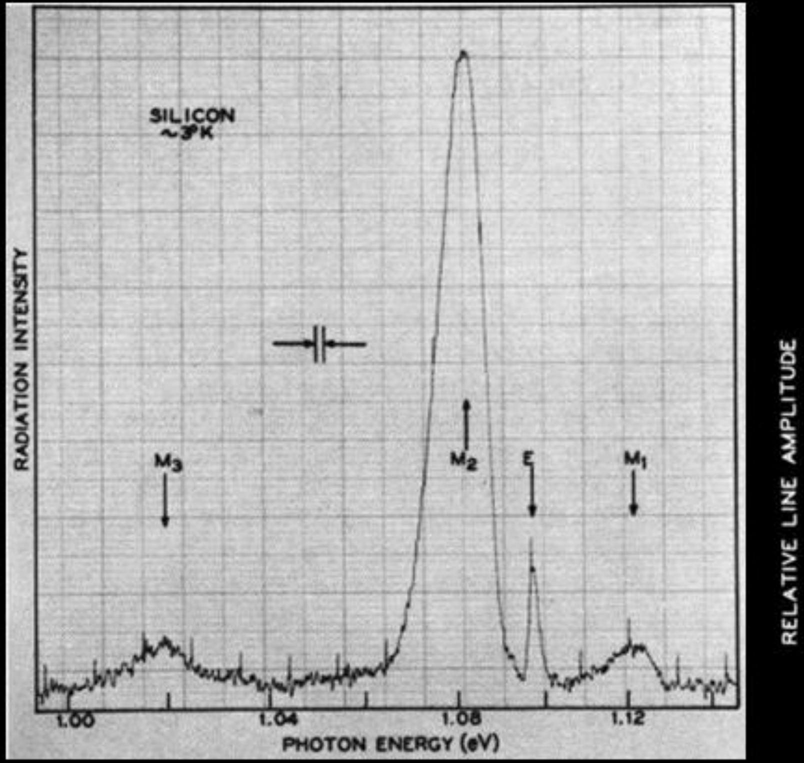
## Free-exciton and bound-exciton luminescence in GaN



Low-temperature photoluminescence spectra of a sample of bulk GaN crystal at temperatures (from top to bottom) of 6, 10, 15, 20, 30, and 45 K. Excitation light comes from a DRSh- 250 lamp.

Semiconductors 33 (10), October 1999

## Excitonic molecule luminescence in Si

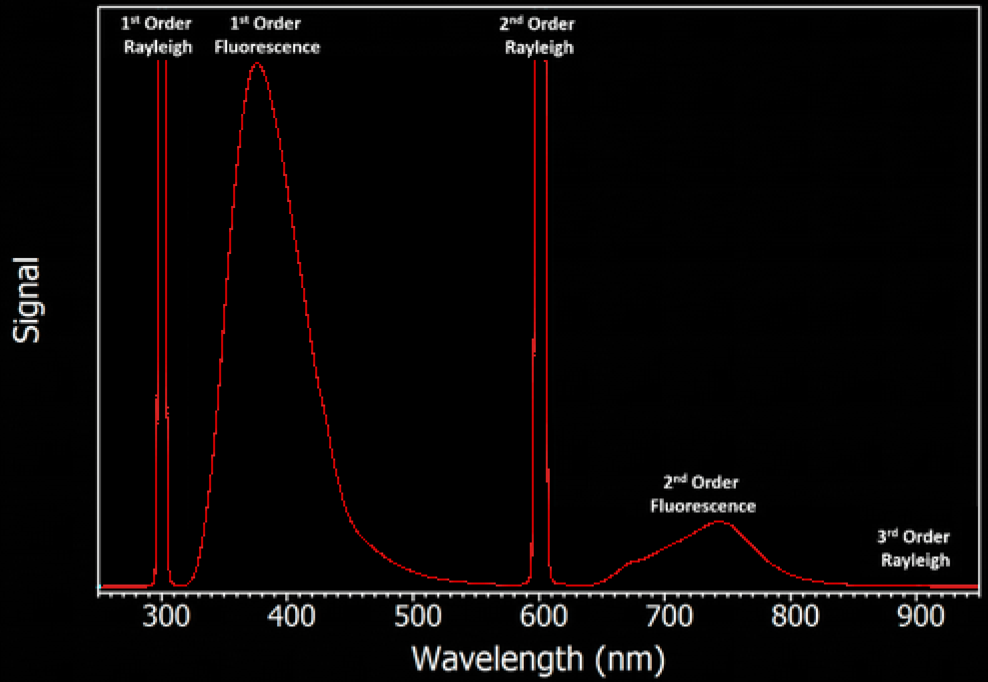


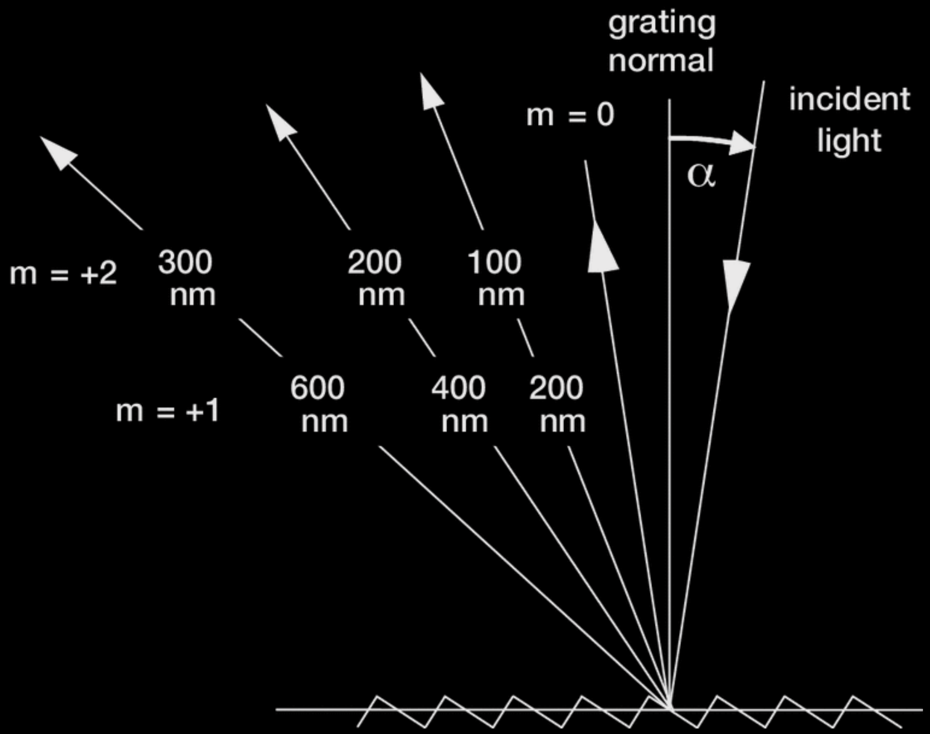
Spectrogram of a Si specimen at ~3 K. The horizontal axis is the energy of the emitted photons in eV. The vertical response is nearly proportional to the number of photons per unit energy interval. The specimen resistivity at room temperature was $9\times10^3$ Ω cm.

Phys. Rev. Lett. **17**, 860 (1966)

# Photoluminescence

Pitfalls, artifacts, corrections …





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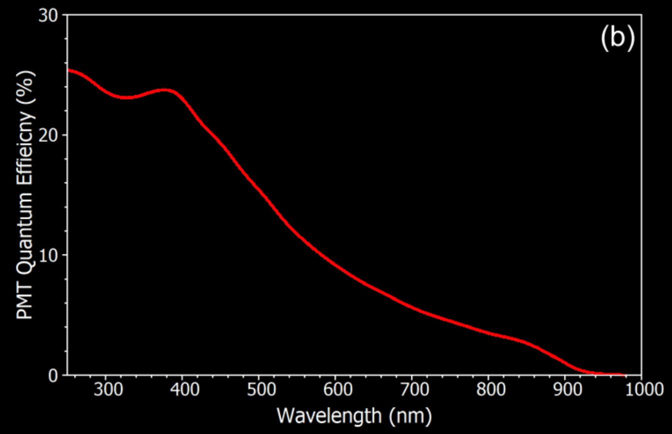
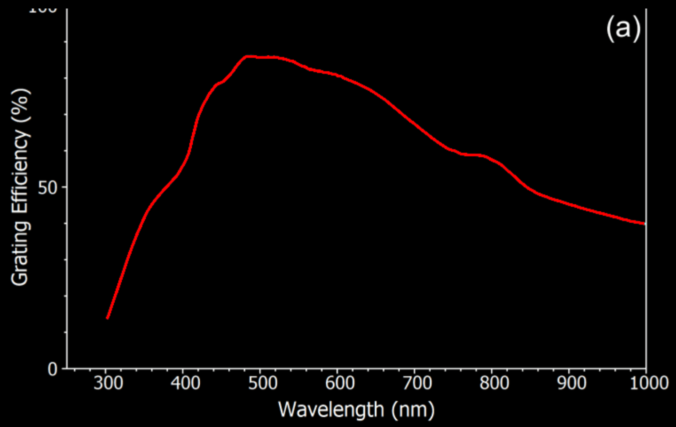
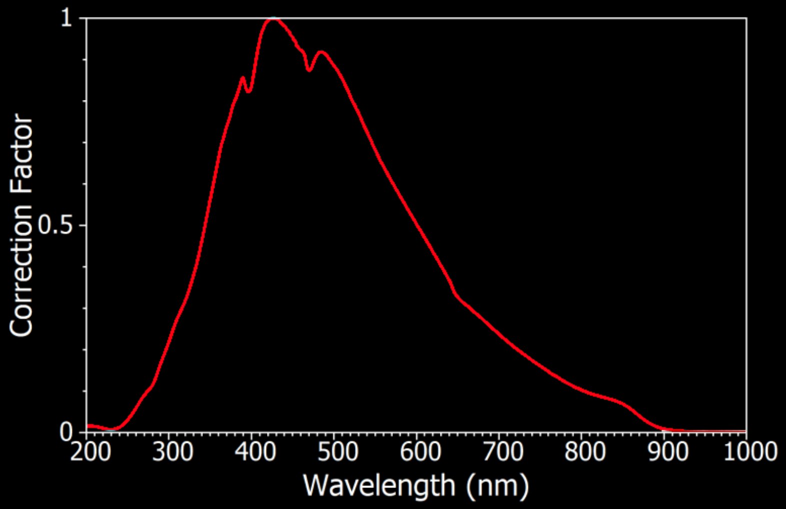
# Photoluminescence

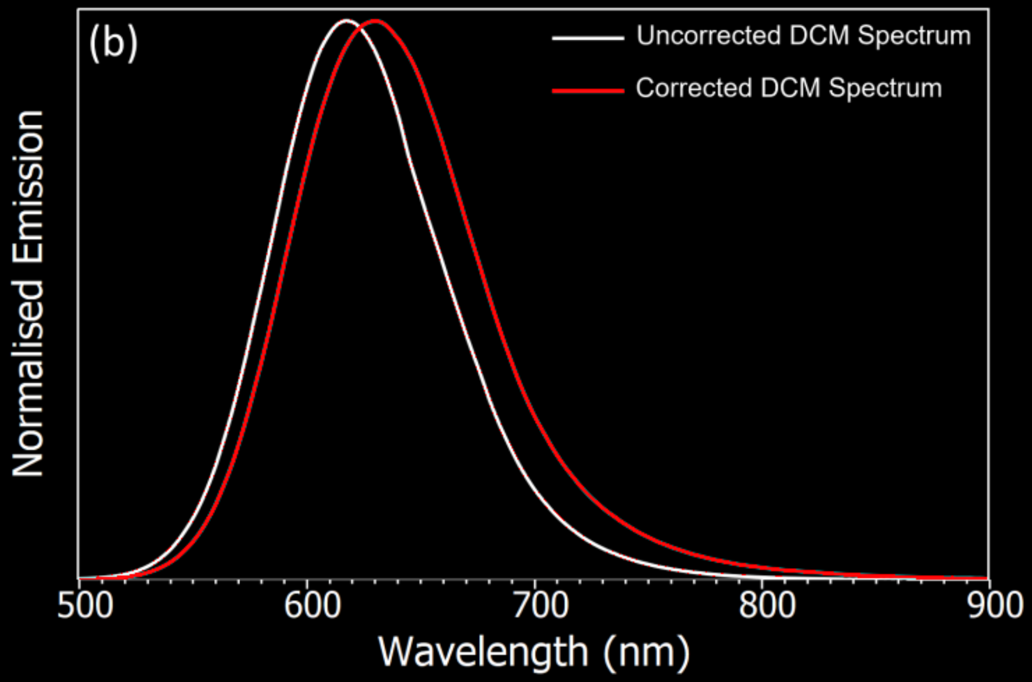
Pitfalls, artifacts, corrections …







Non-ideal components introduce spectral distortions



EDINBURGH INSTRUMENTS

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# Photoluminescence

**Strengths:**

- Very little to none sample preparation.
- Non destructive technique.
- Very informative spectrum.

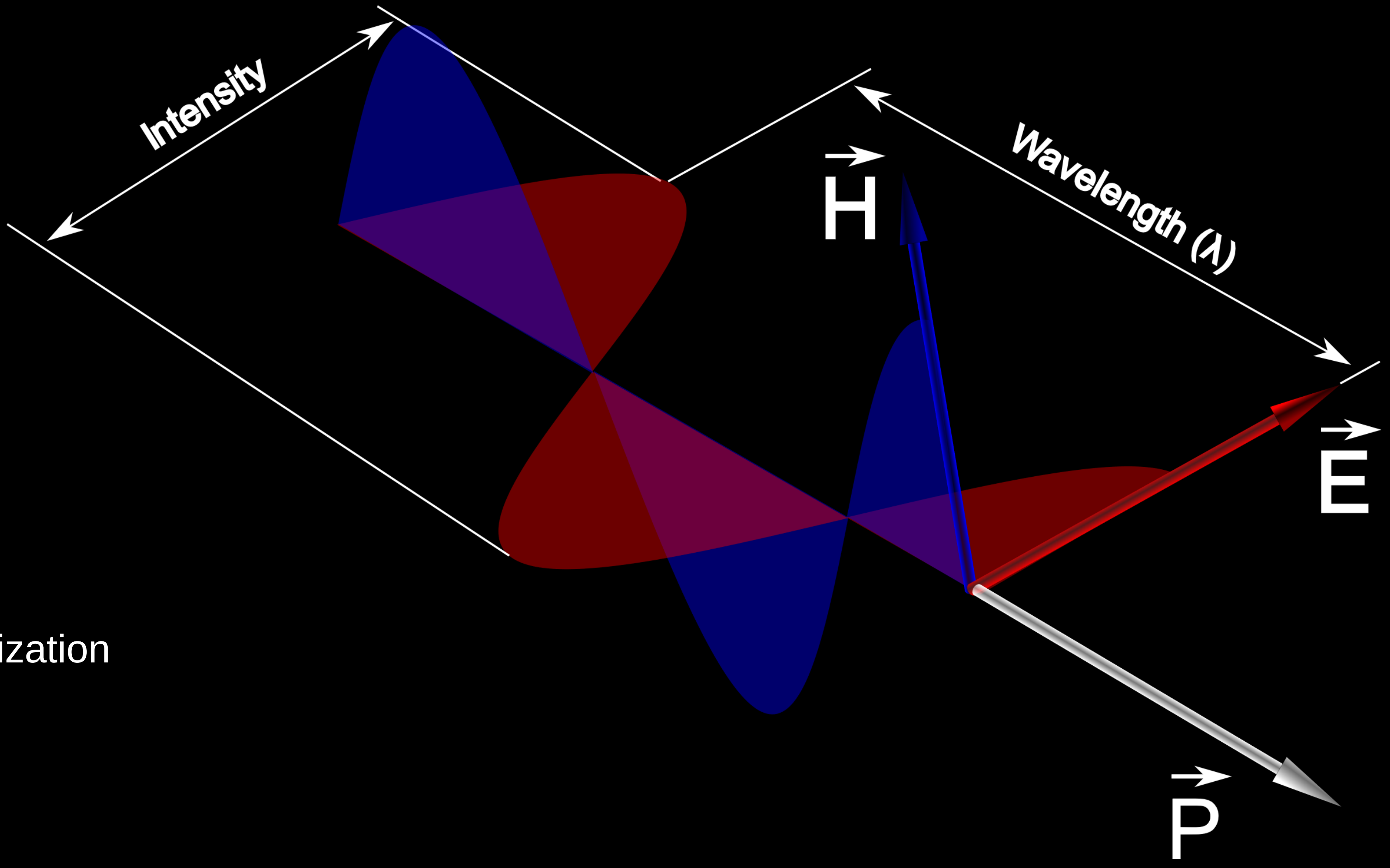
**Limitations:**

- Often requires low temperature.
- Data analysis may be complex.
- Many materials luminescence weakly.

**Complementary techniques:**

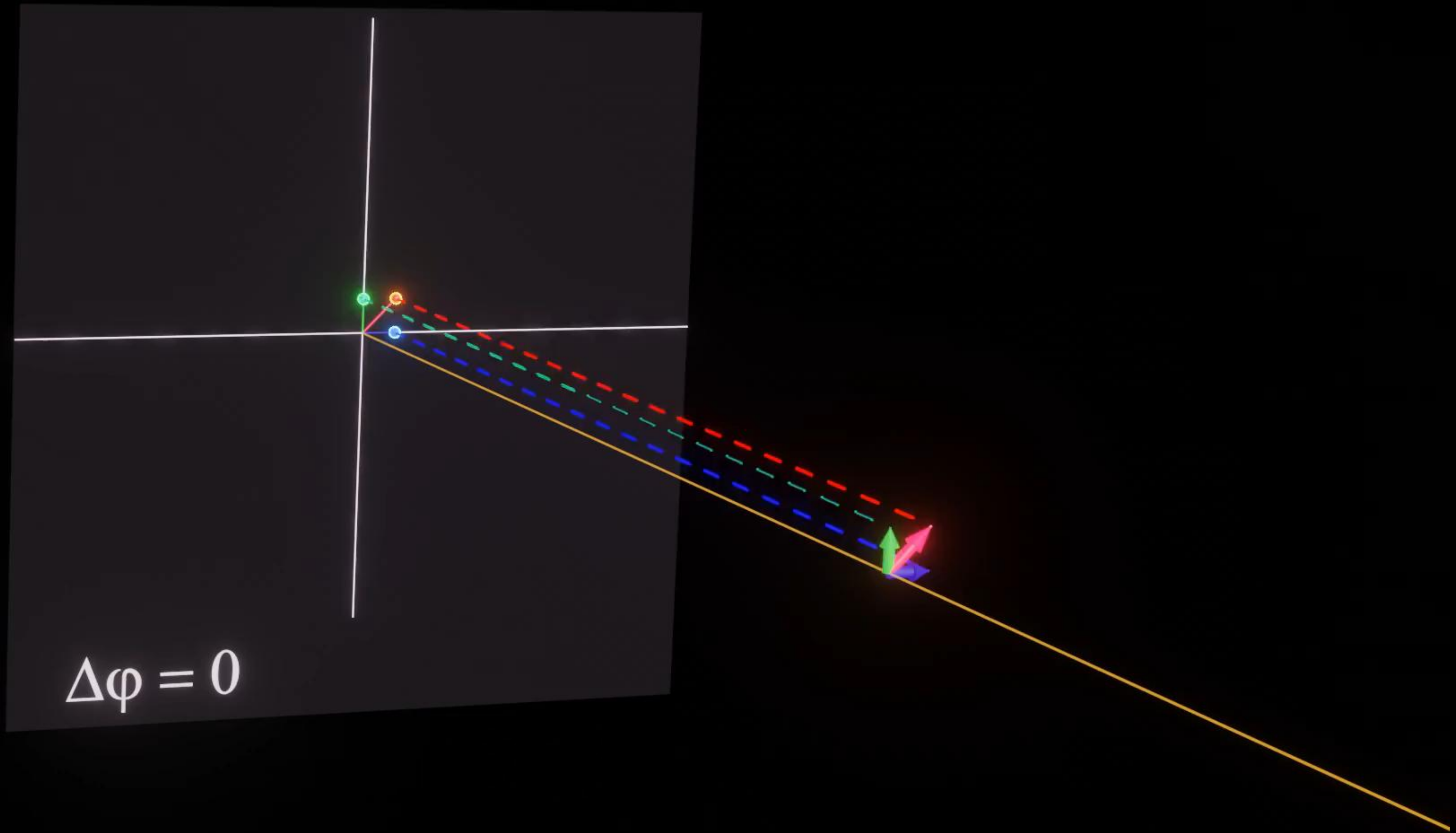
Ellipsometry, Modulation spectroscopies, Spectrophotometry, Raman.

# Light properties

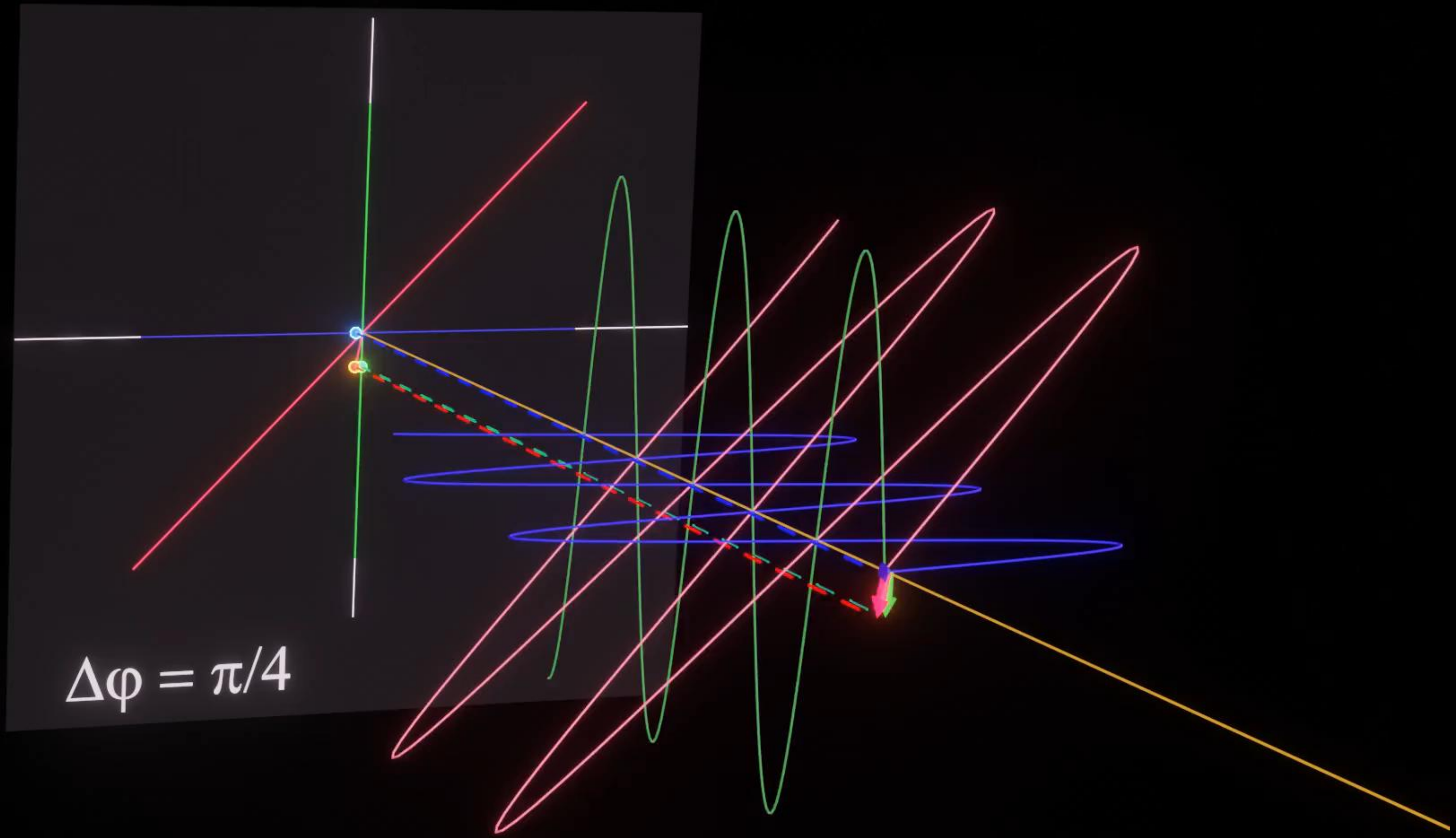


- Direction of propagation
- Electric field direction or polarization
- Photon energy or wavelength
- Intensity

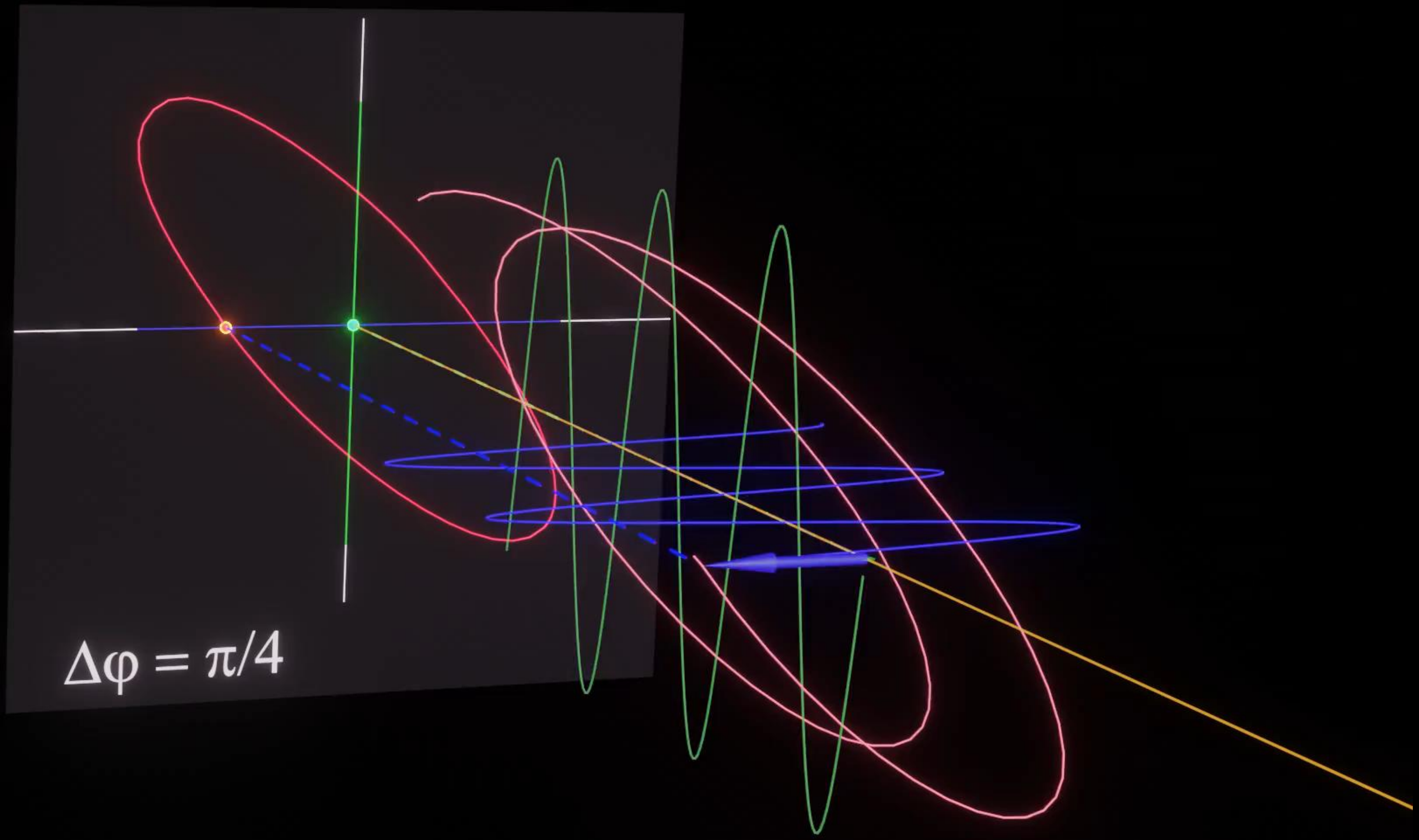
# Polarization



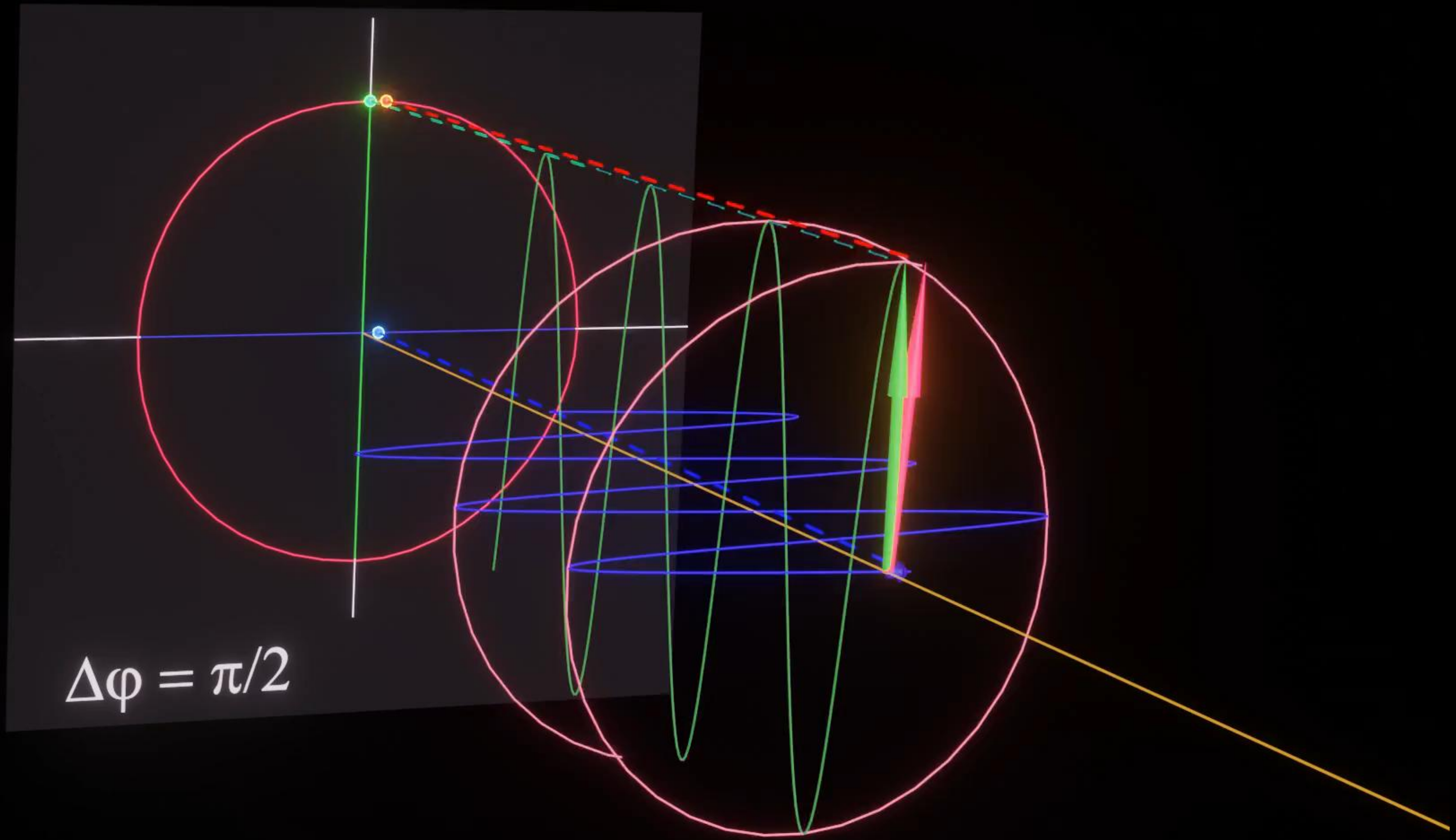
# Polarization



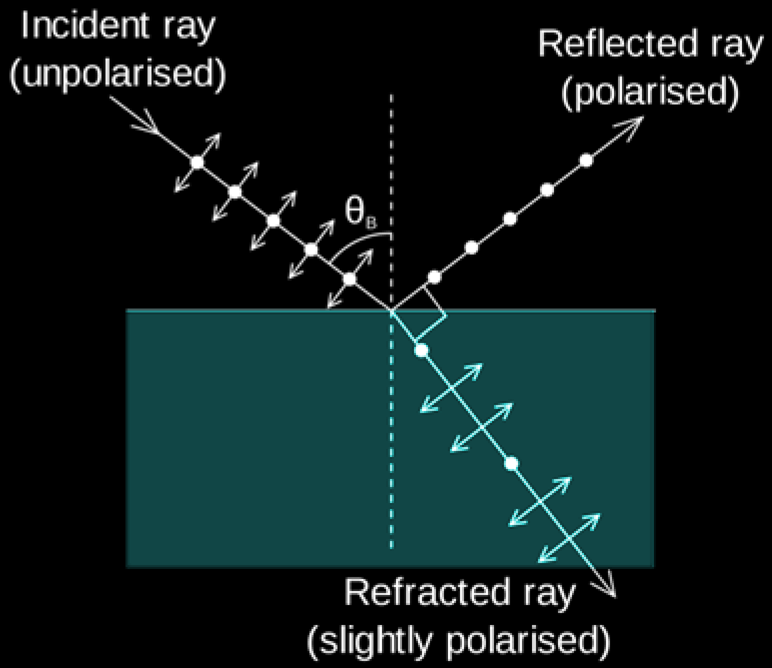
# Polarization



# Polarization



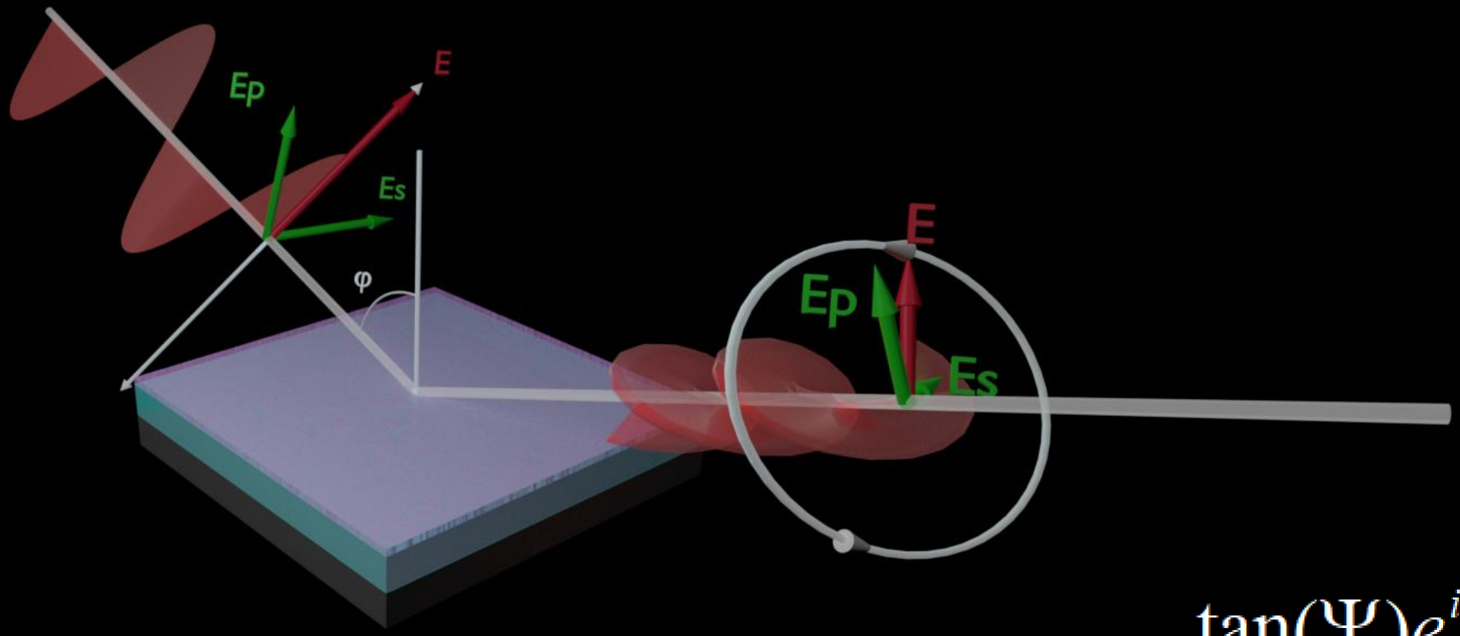
# Polarization



# Ellipsometry

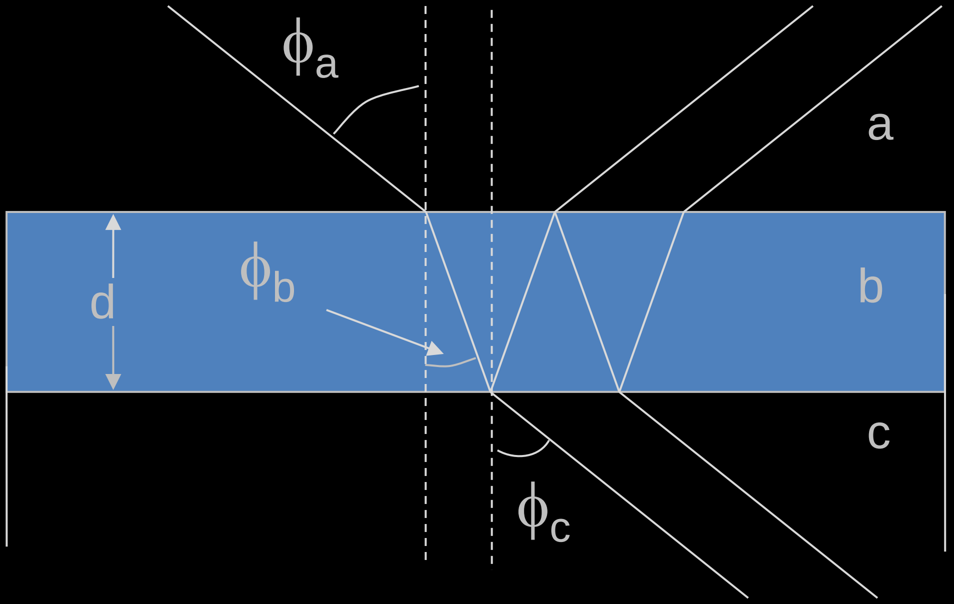
**Basic principle:**

The reflected light emerges from the surface elliptically polarized, i.e. its p and s polarization components are generally different in phase and amplitude.



$$\tan(\Psi)e^{i\Delta} = \frac{\tilde{R}_p}{\tilde{R}_s}$$

# Ellipsometry



$$\tilde{n} = n + ik \qquad \tilde{n}_1 \sin\phi_1 = \tilde{n}_2 \sin\phi_2$$

$$\tilde{r}_{12}^{p,s} = \frac{\tilde{n}_{2,1}\cos\phi_1 - \tilde{n}_{1,2}\cos\phi_2}{\tilde{n}_{2,1}\cos\phi_1 + \tilde{n}_{1,2}\cos\phi_2}$$

$$\tilde{R}_{p,s} = \frac{\tilde{r}_{ab}^{p,s} + \tilde{r}_{bc}^{p,s} e^{-2i\beta}}{1 + \tilde{r}_{ab}^{p,s}\tilde{r}_{bc}^{p,s} e^{-2i\beta}}$$

$$\beta = \frac{2\pi d}{\lambda}\tilde{n}_b \cos\phi_b$$

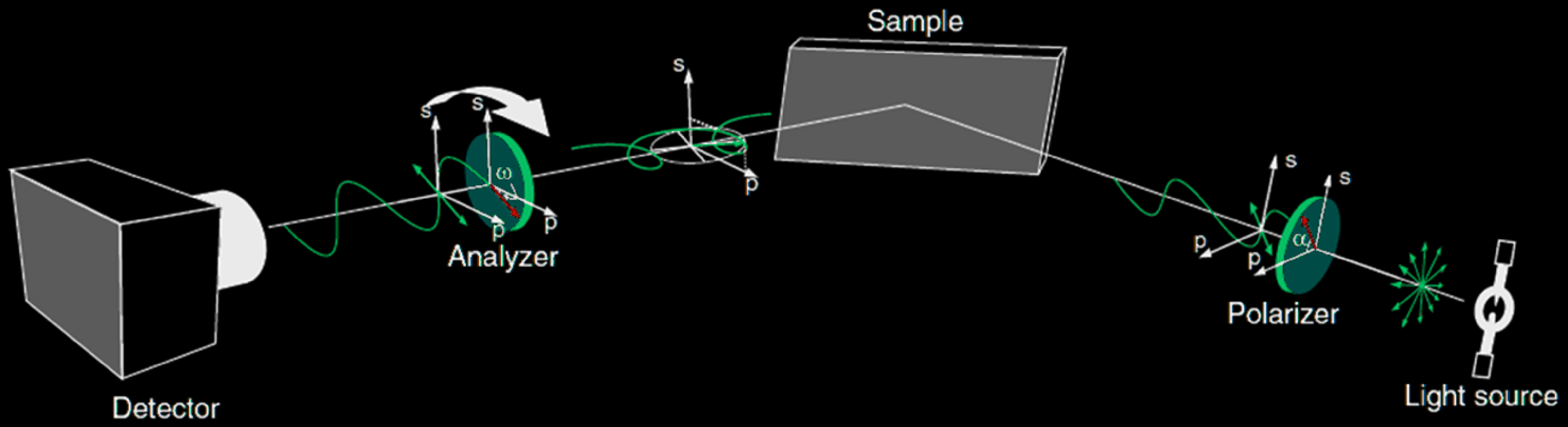
$$\tilde{R}_{p,s} = \frac{E_{p,s}^{r}}{E_{p,s}^{i}} e^{i(\delta_{p,s}^{r} - \delta_{p,s}^{i})}$$

$$\tan(\Psi)e^{i\Delta} = \frac{\tilde{R}_p}{\tilde{R}_s} \Rightarrow \begin{cases} \tan(\Psi) = \dfrac{|\tilde{R}_p|}{|\tilde{R}_s|} \\ \Delta = \delta^r - \delta^i \end{cases}$$

# Ellipsometry

**What is measured:**

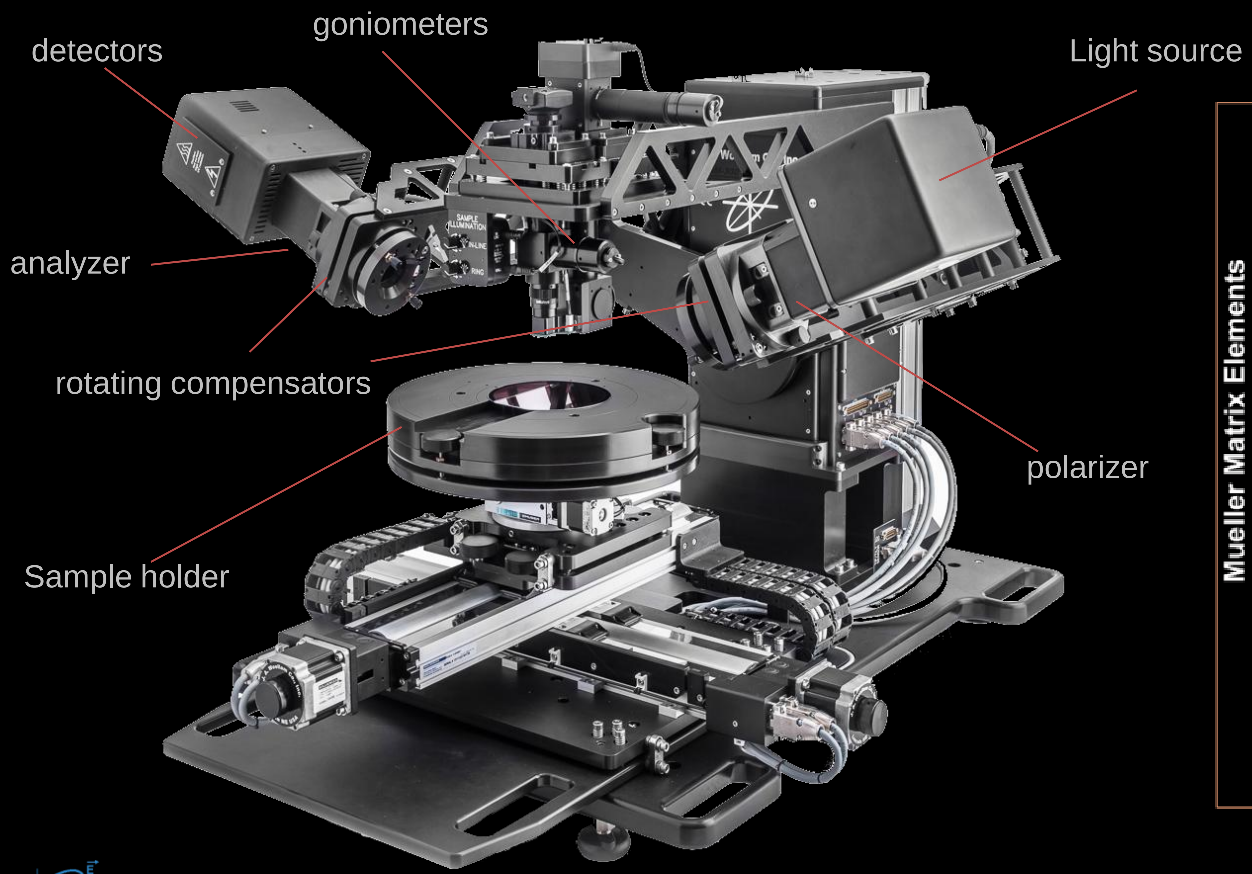
The changes in the polarization state of light upon reflection from a mirror like surface.

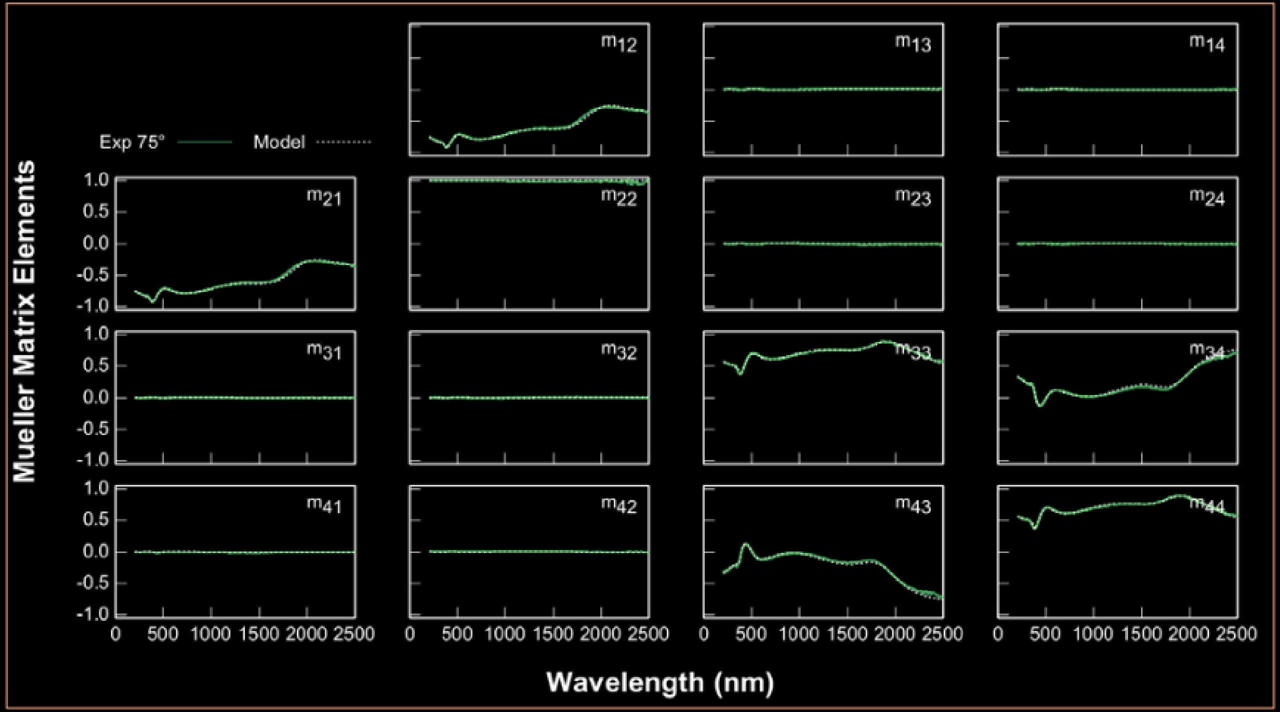


# Ellipsometry

## What is measured:

The changes in the polarization state of light upon reflection from a mirror like surface.





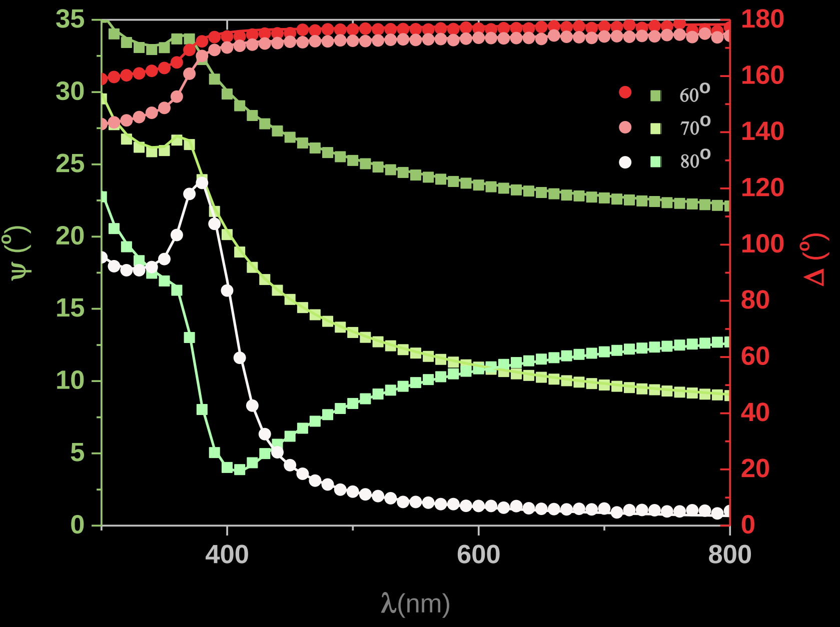
J.A. Woollam Co., Inc.

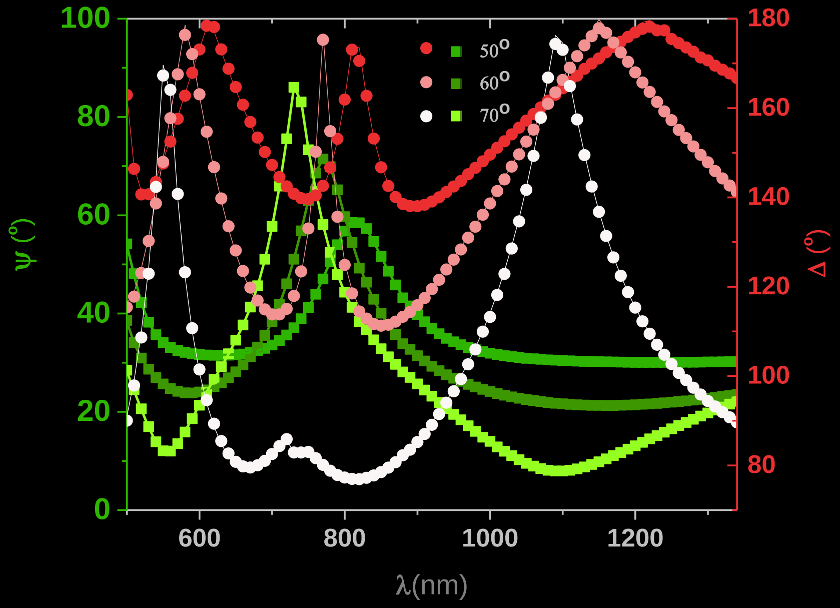
# Ellipsometry

## Applications

| | |
|---|---|
| $SiO_2$ | 18.7 ± 0.2 Å |
| Si | |

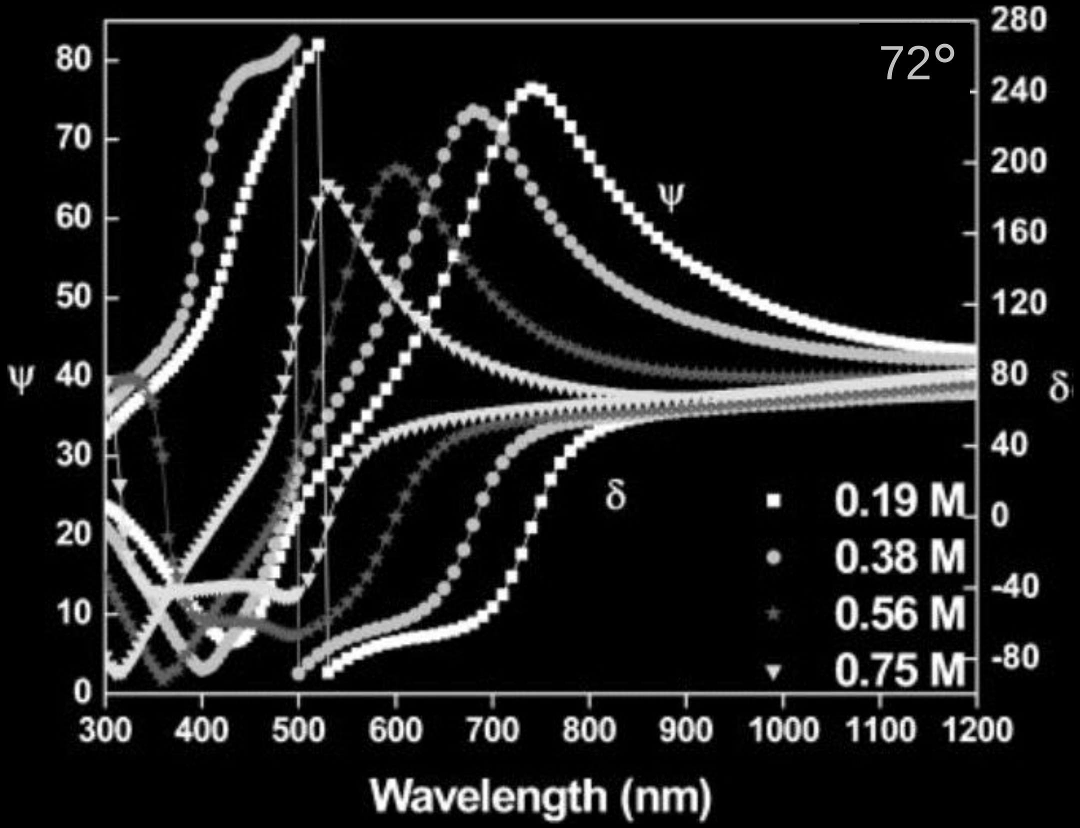
| | |
|---|---|
| $SiO_2$ | 4923.1 ± 0.2 Å |
| Si | |





# Ellipsometry

## Applications

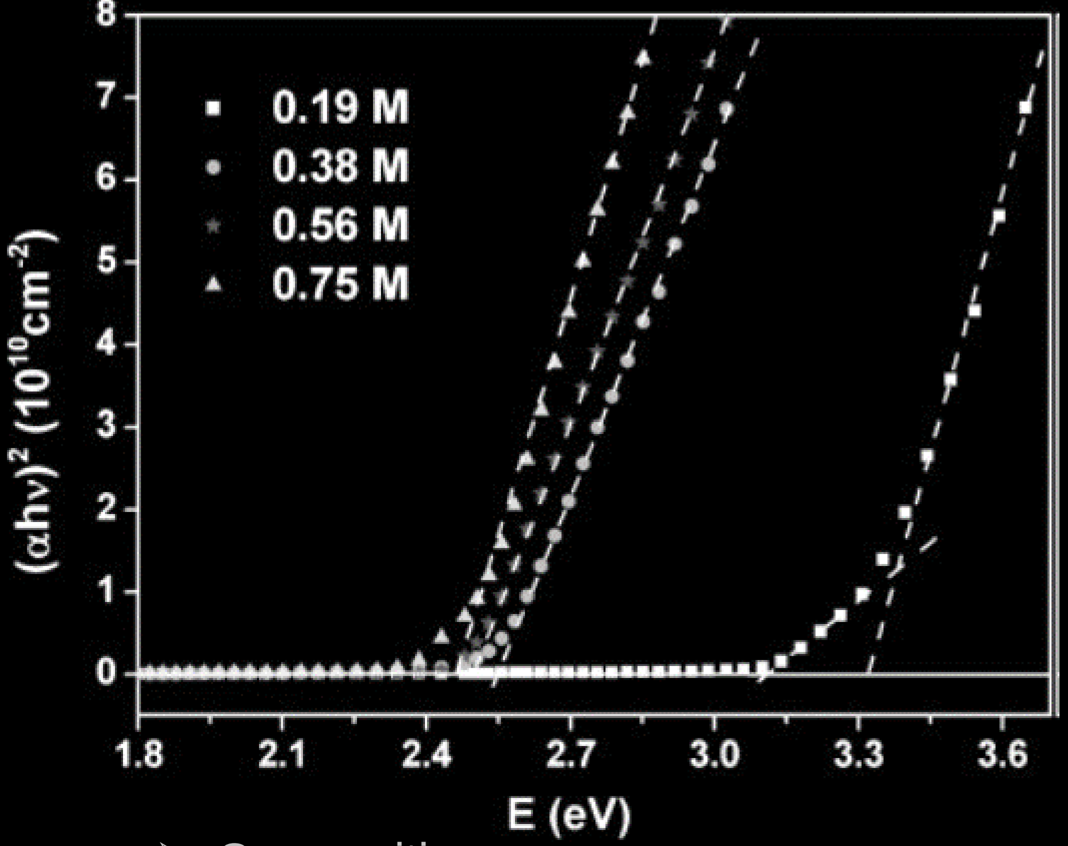


| [NH4OH] (M) | Thickness (nm) | Roughness (nm) | ZnS (%) | Band-gap (eV) |
|---|---|---|---|---|
| 0.19 | 42.12 | 23.77 | 99.7 | 3.49 |
| 0.38 | 73.79 | 7.15 | 45.5 | 2.52 |
| 0.56 | 50.89 | 5.94 | 32.3 | 2.45 |
| 0.75 | 18.59 | 4.54 | 5.2 | 2.43 |

Ellipsometric $\Psi(\lambda)$ and $\Delta(\lambda)$ spectra of $Cd_{1-x}Zn_xS$ thin films deposited under the different concentration of ammonia: 0.19, 0.38, 0.56, and 0.75 M

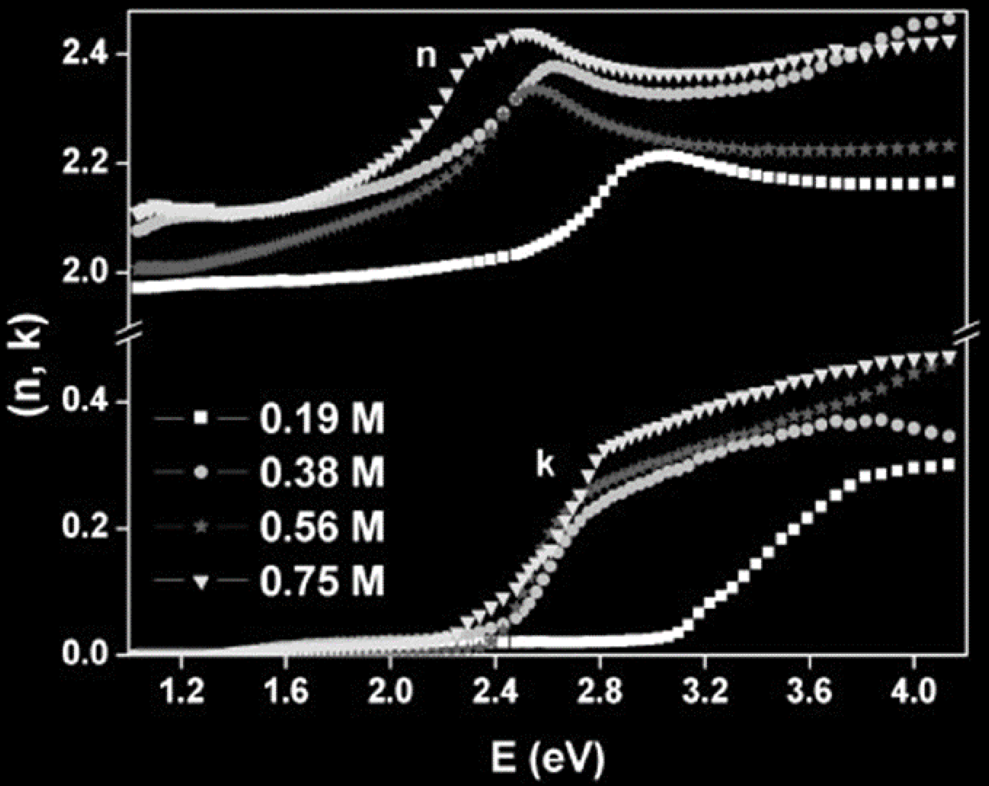
Jpn. J. Appl. Phys. 49 (2010) 081202

# Ellipsometry



## Applications

- Composition
- Surface roughness
- Film thickness
- Band gap energy
- Optical constants (dielectric function)



Jpn. J. Appl. Phys. 49 (2010) 081202

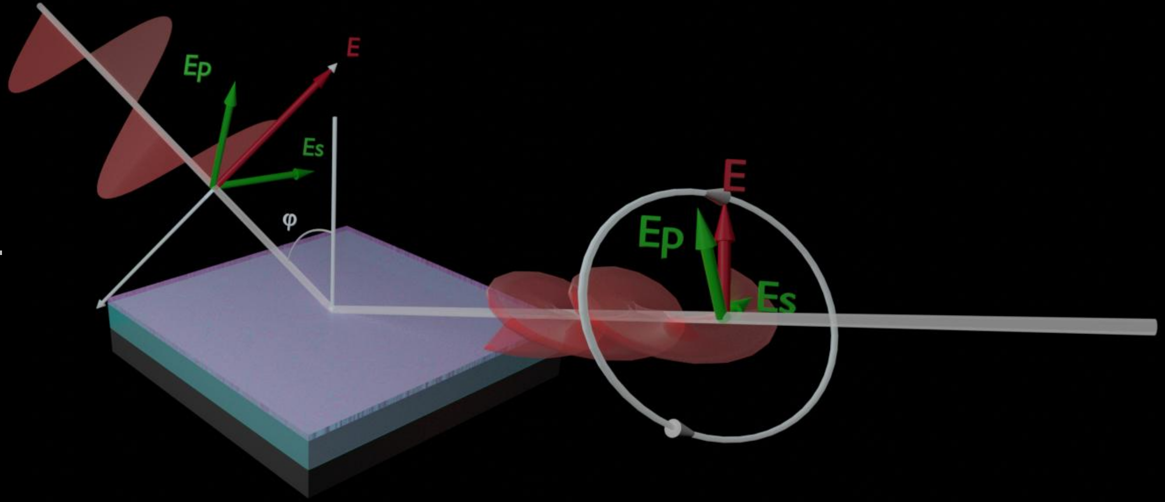
# Ellipsometry

## Strengths:

- Fast.
- Measures a ratio of two intensity values and a phase.
  - Highly accurate (even in low light levels).
  - No reference sample necessary.
  - Not susceptible to scatter, lamp or purge fluctuations.
  - Increased sensitivity, especially to ultrathin films (<10nm).
- Can be used in-situ.

## Limitations:

- Flat and parallel surface and interfaces with measurable reflectivity.
- A realistic physical model of the sample is required to obtain most useful information.



## Complementary techniques:

PL, Modulation spectroscopies, X-Ray Photoelectron Spectroscopy, Secondary Ion Mass Spectroscopy, XRD, Hall effect.

# Optical microscopy

Von Leeuwenhoek microscope
ca. late 1600's

Hooke microscope
ca. 1670

Hand-held microscope ca. early 1700's

British microscope
ca. 1850

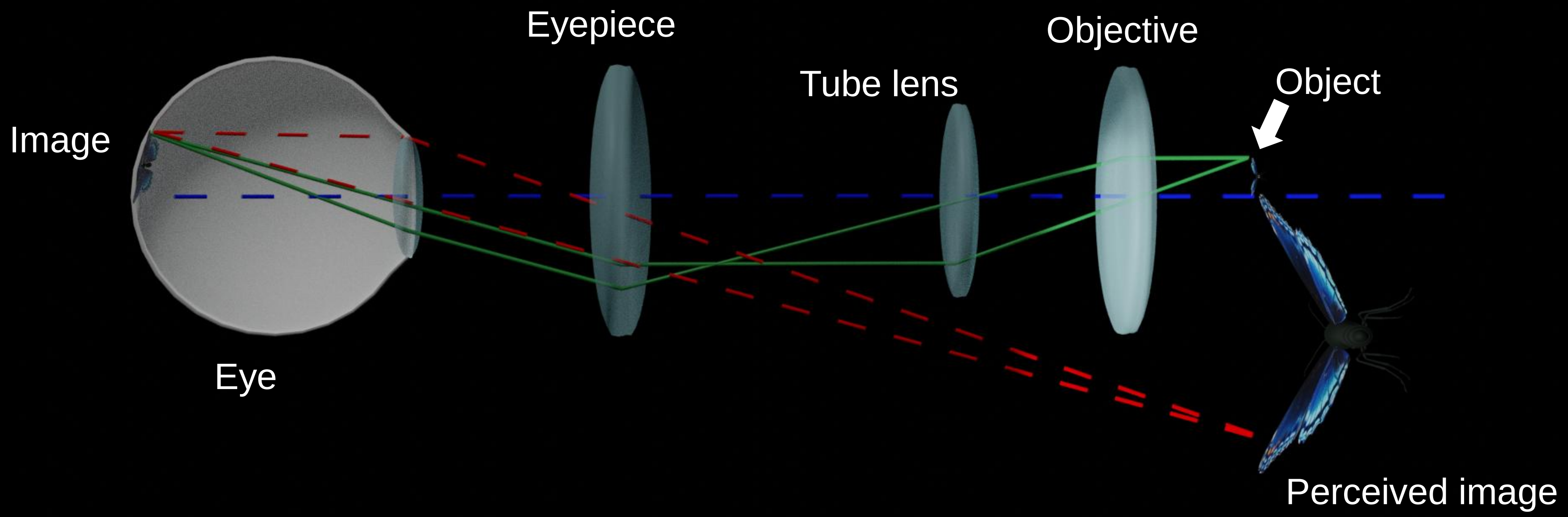
Zeiss microscope
ca. 1930

Modern scientific microscope

ZEISS
We make it visible.

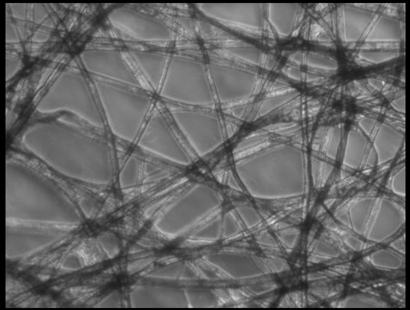
# Optical microscopy

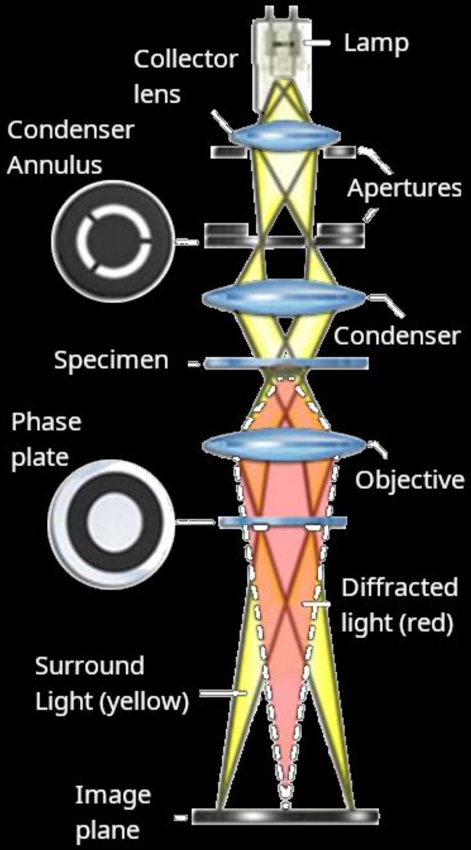
## “Conventional" Optical Microscopy



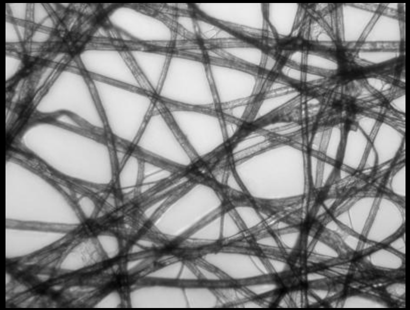
# Optical microscopy

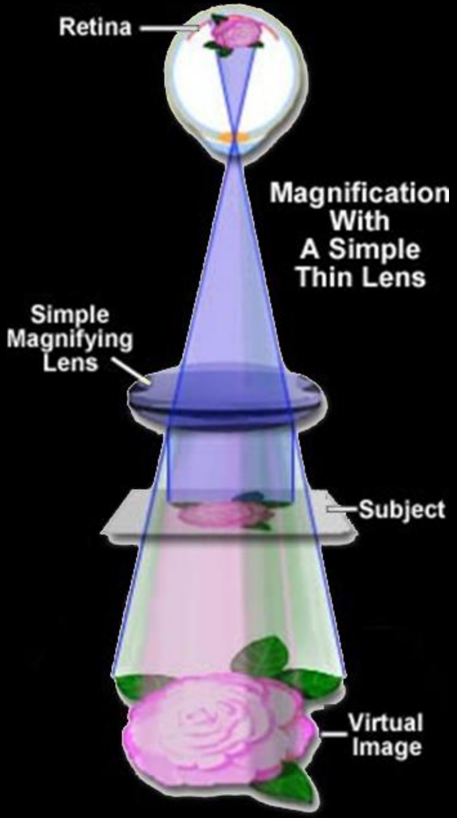
## Phase contrast



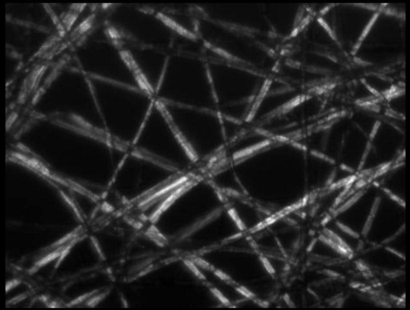


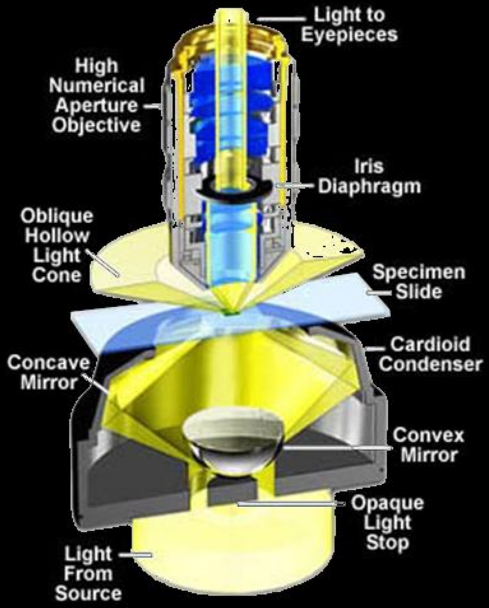
## Bright field



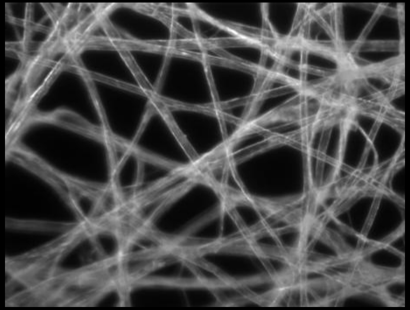


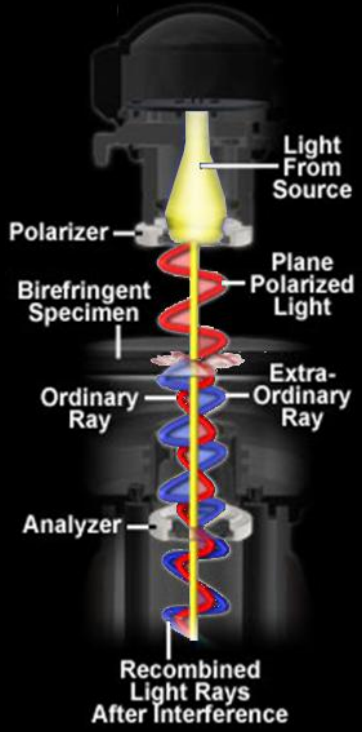
## Dark field





## Polarizing





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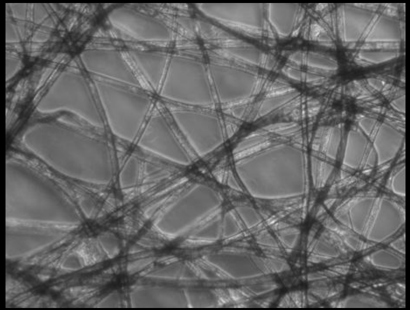
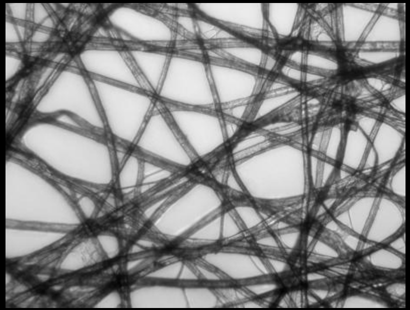
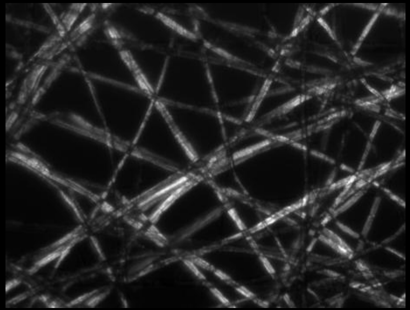
# Optical microscopy



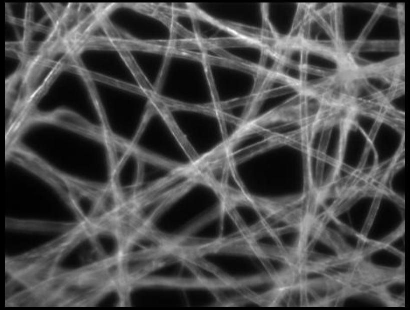
Phase contrast



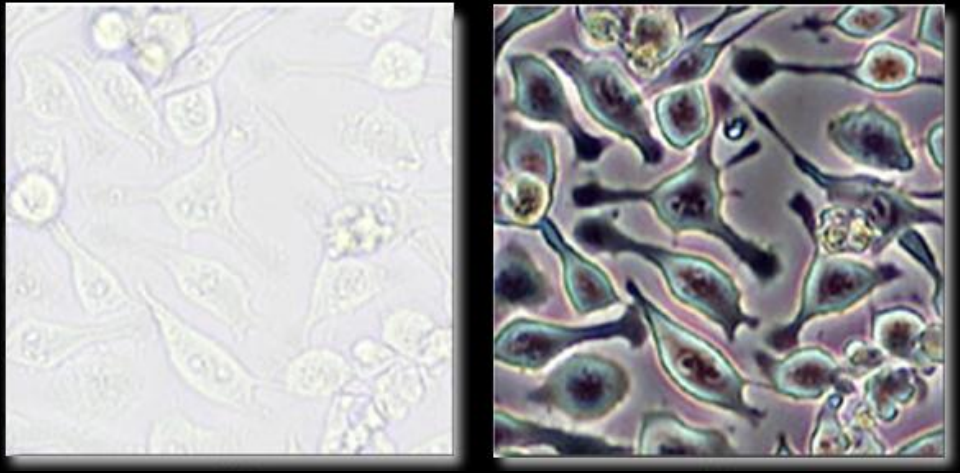
Bright field



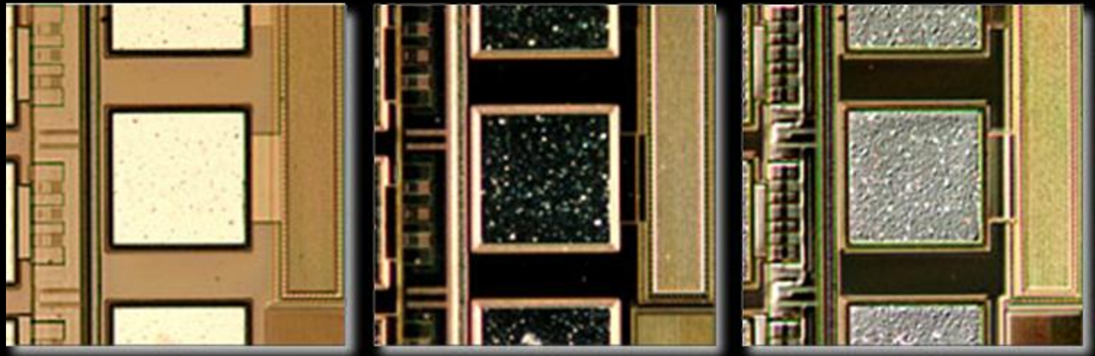
Dark field



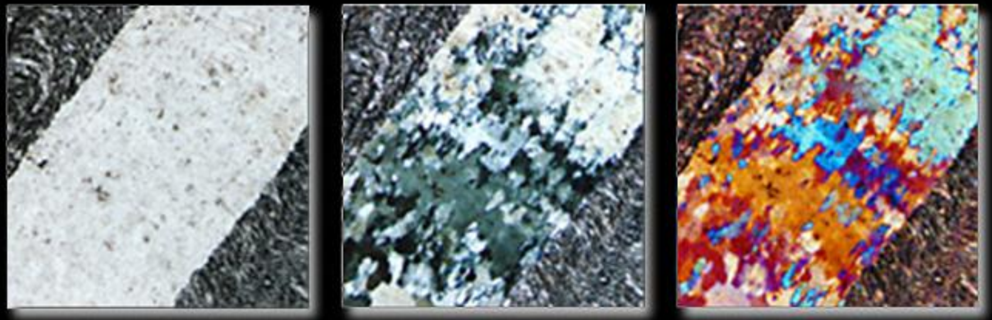
Polarizing



Living Cells in Brightfield and Phase Contrast



Integrated Circuit in Brightfield, Darkfield, and DIC with Reflected Light



Phyllite Thin Section in Polarized Light

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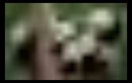
# Resolution

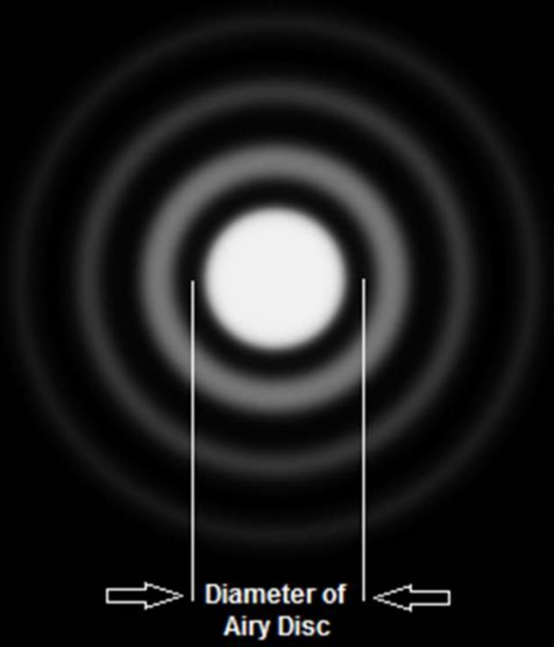
- But how small a thing can we see?

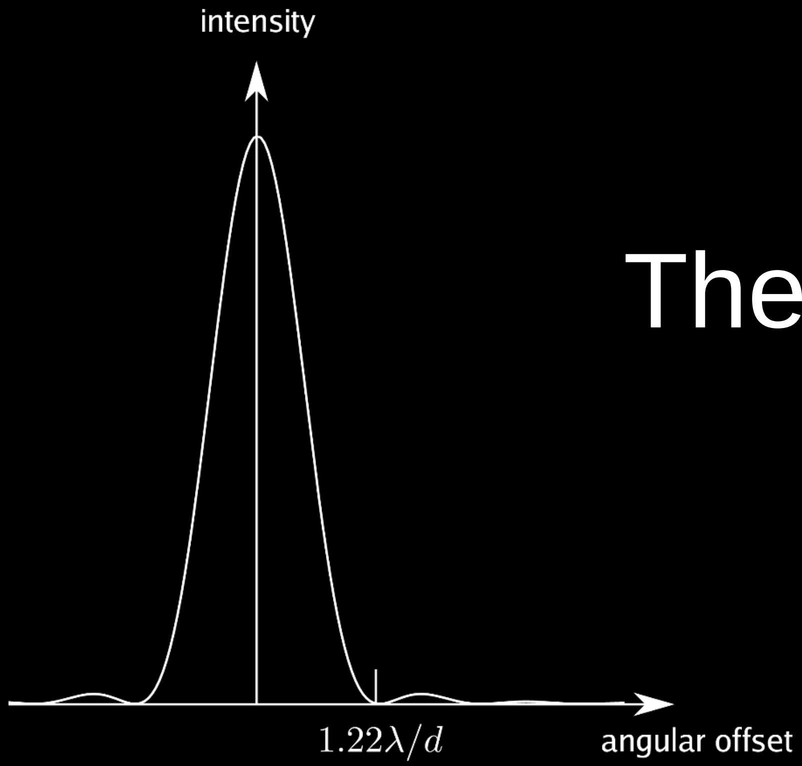


# Resolution

- But how small a thing can we see?

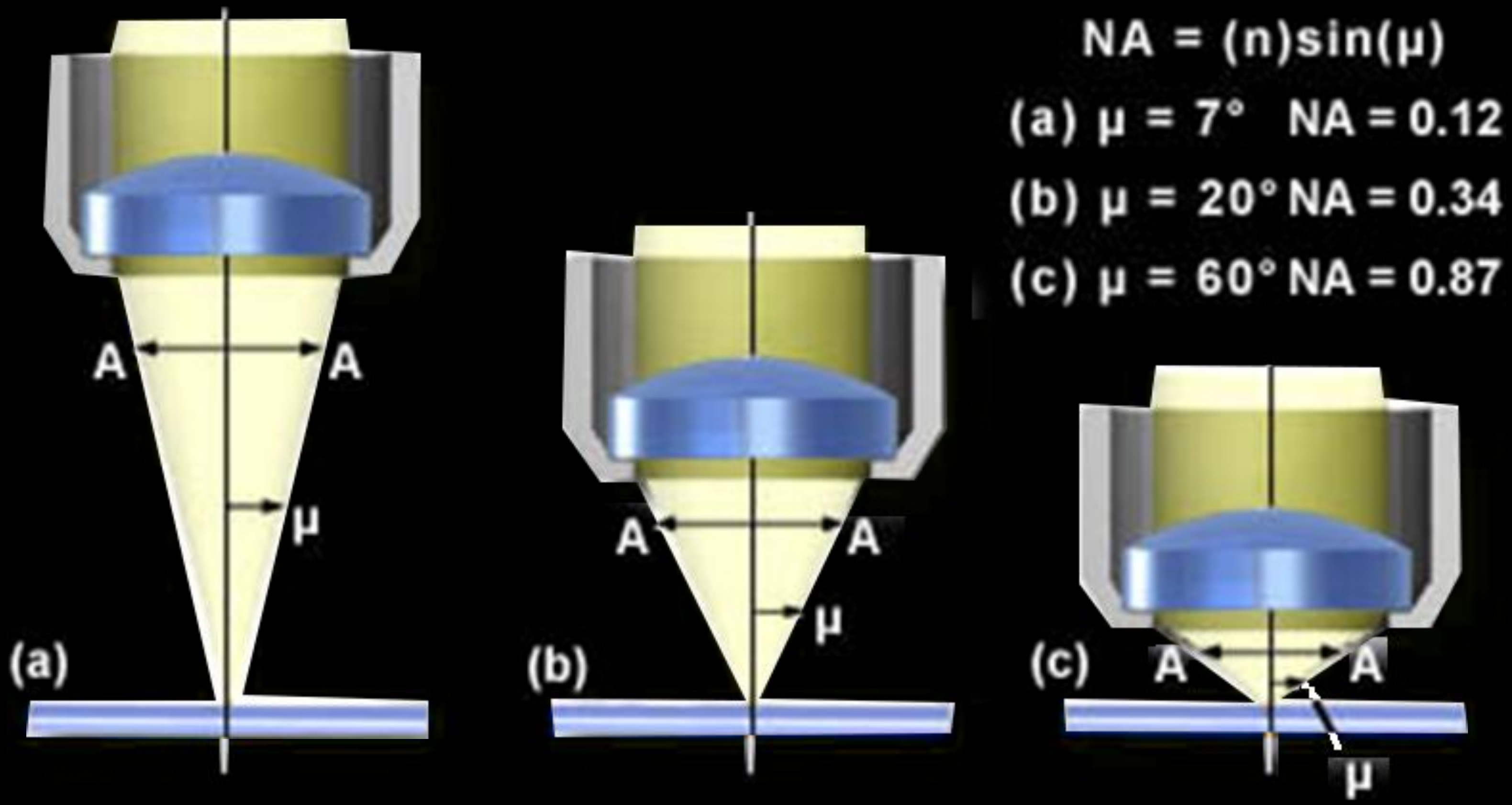
# Lateral resolution





The Airy pattern

# Lateral resolution



$\mathrm{NA} = (n)\sin(\mu)$

(a) $\mu = 7°$ NA = 0.12

(b) $\mu = 20°$ NA = 0.34

(c) $\mu = 60°$ NA = 0.87

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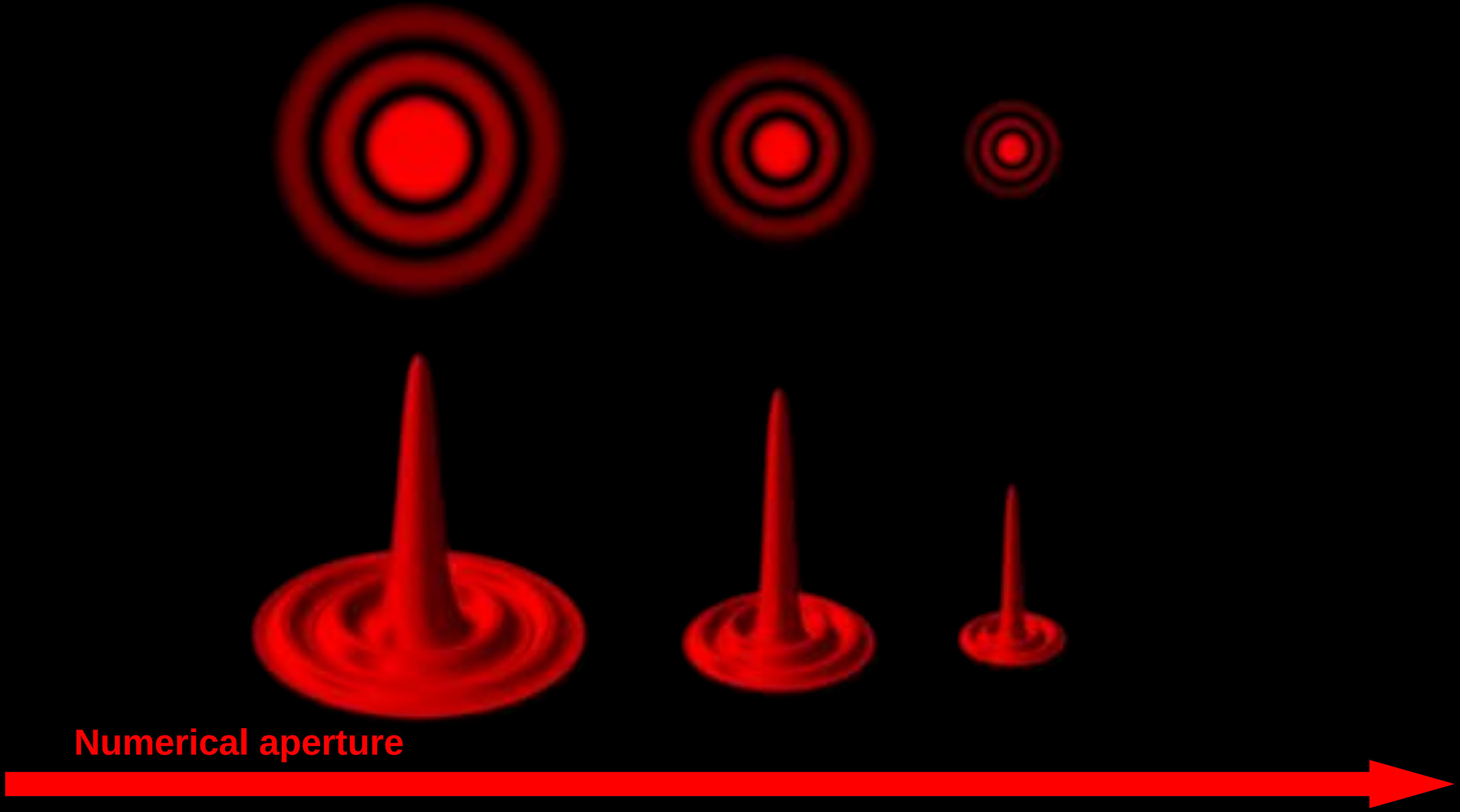
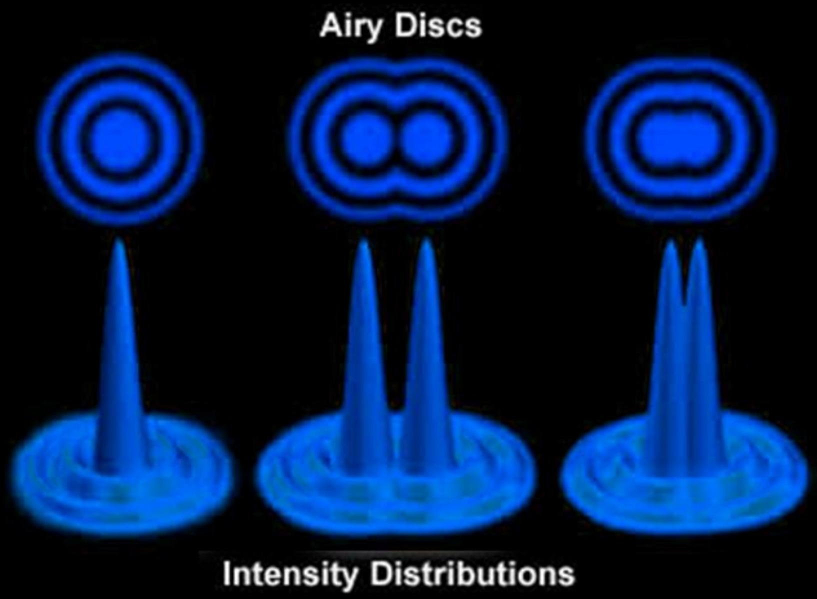
# Lateral resolution



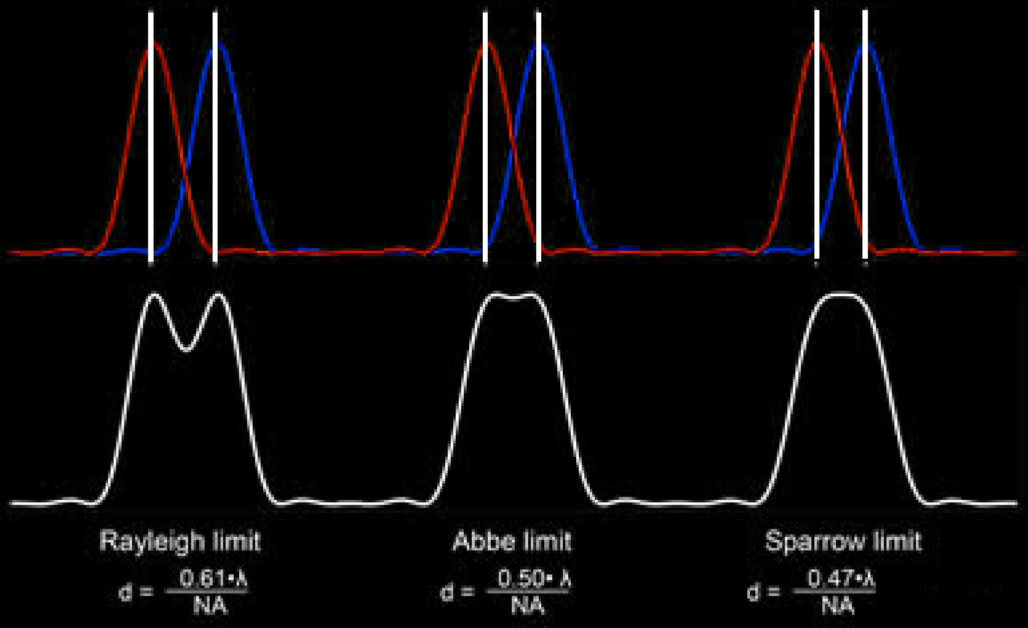
# Lateral resolution

**Resolution**



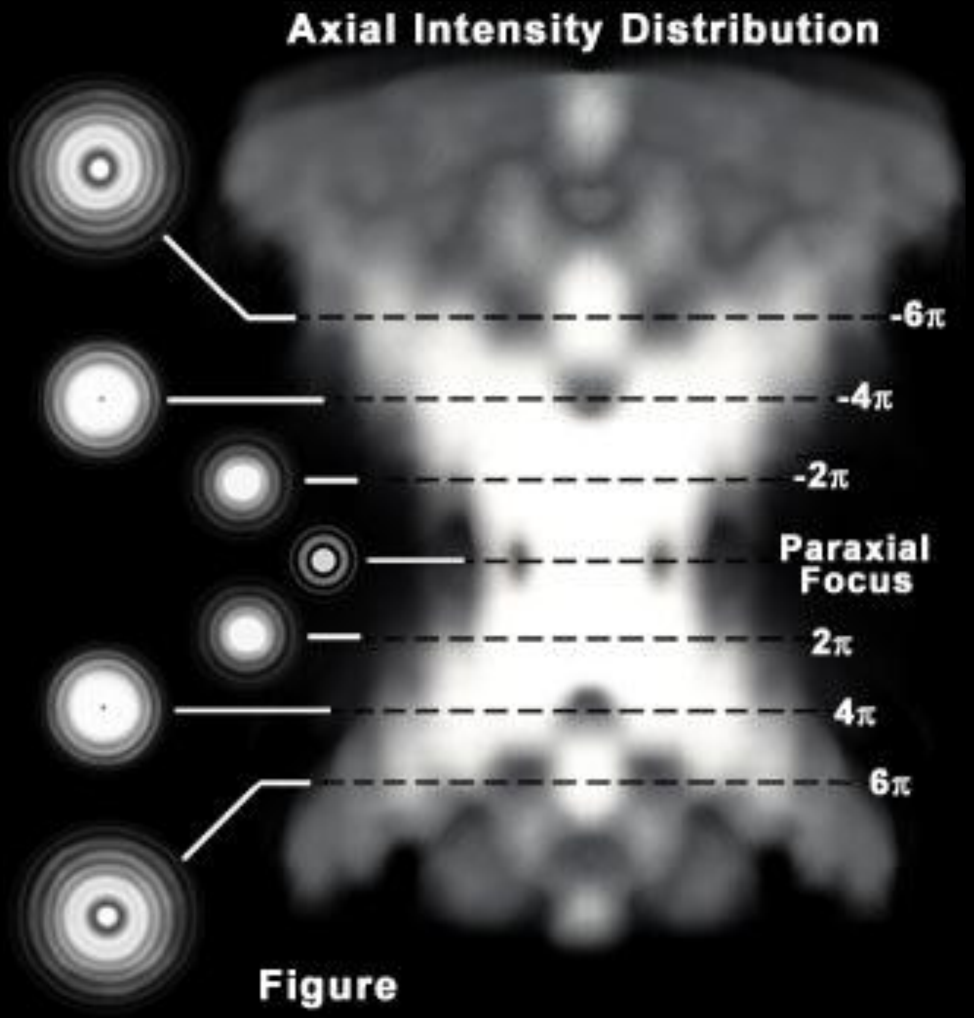
$$d \approx \frac{0.61\lambda}{NA}$$

Rayleigh criterion

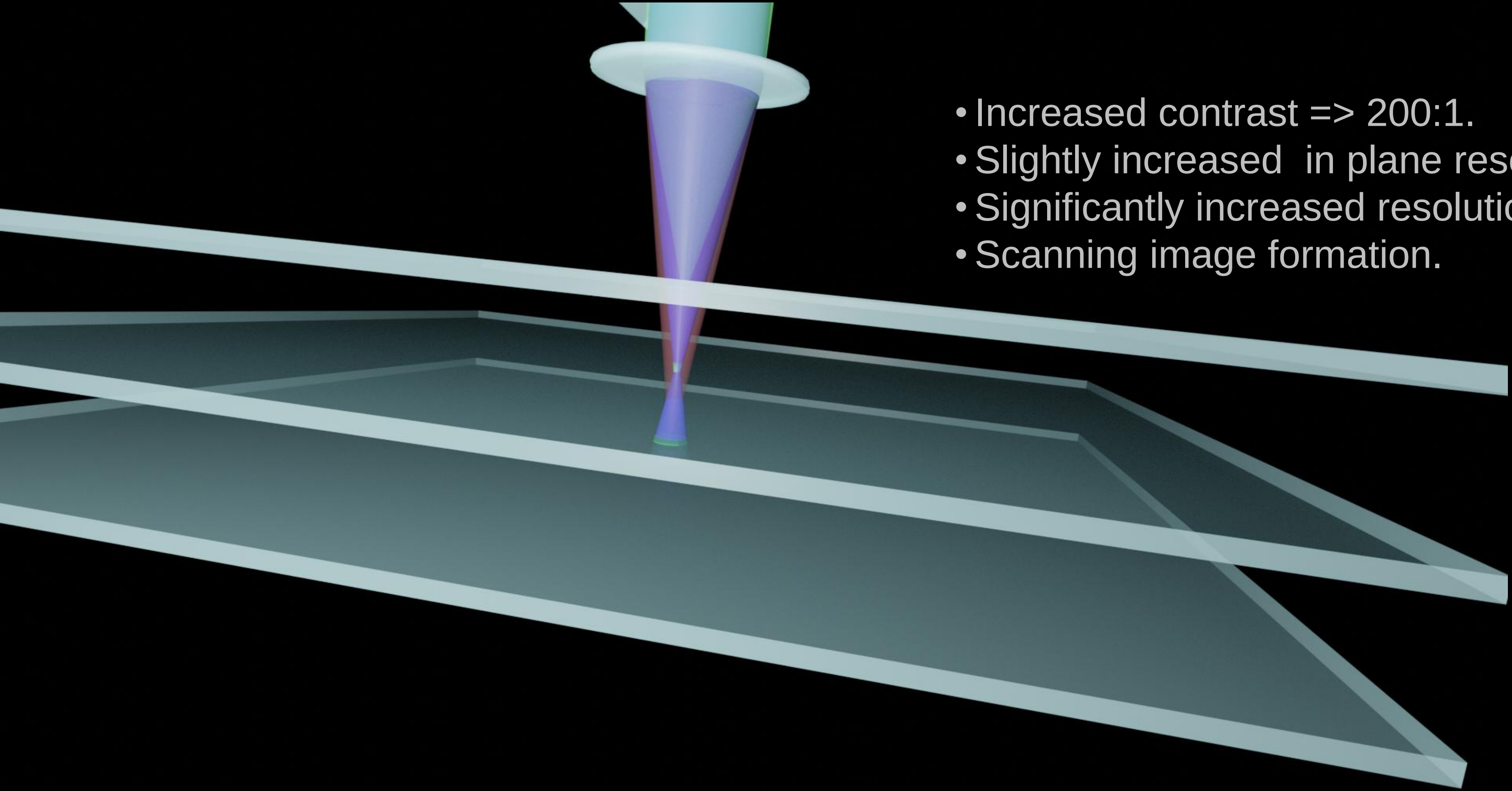


Abbé criterion

# Depth resolution

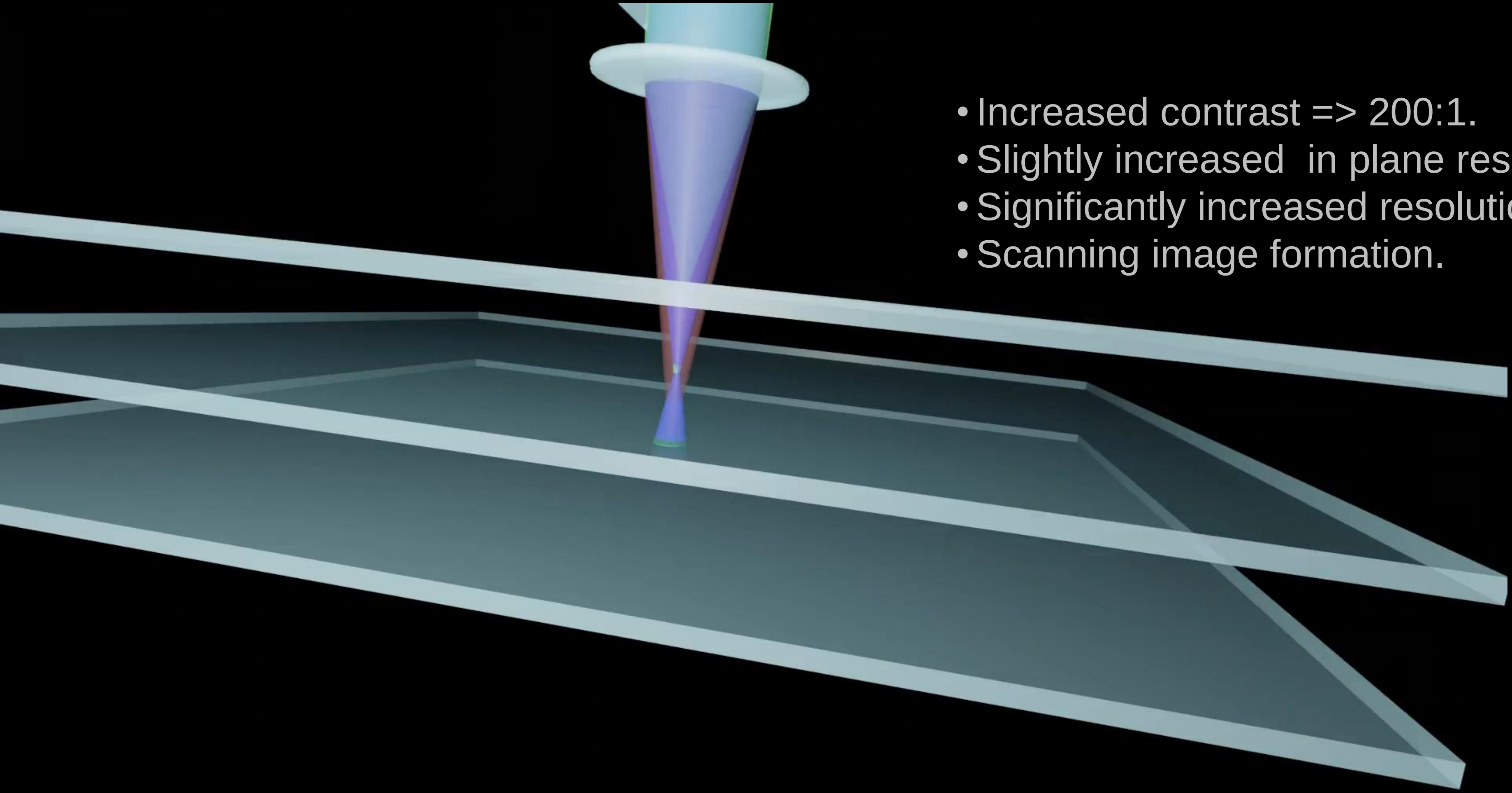


# Confocal microscopy



- Increased contrast => 200:1.
- Slightly increased  in plane resolution (1.5 x)
- Significantly increased resolution along the optical axis.
- Scanning image formation.

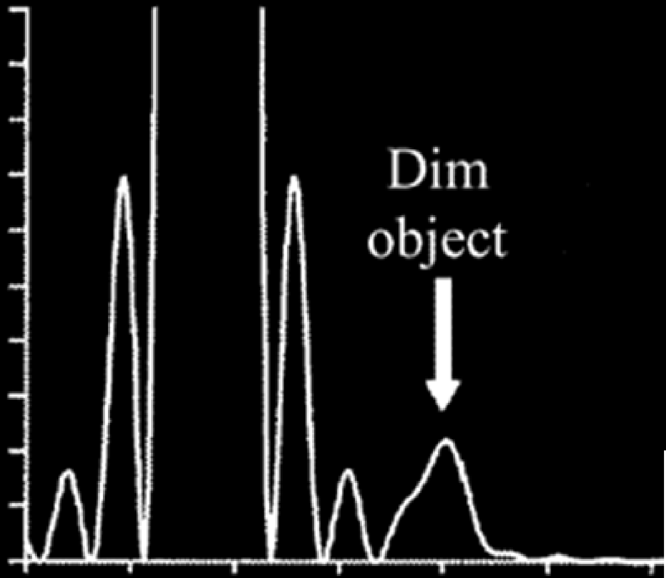
# Confocal microscopy

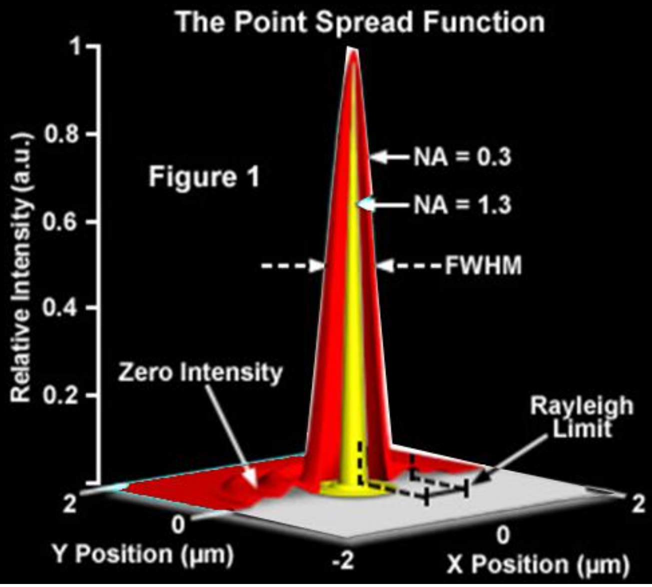


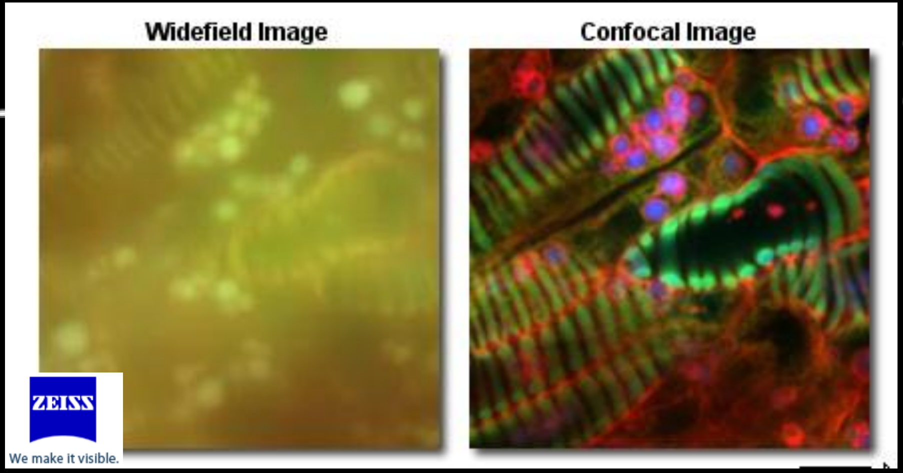
- Increased contrast => 200:1.
- Slightly increased  in plane resolution (1.5 x)
- Significantly increased resolution along the optical axis.
- Scanning image formation.

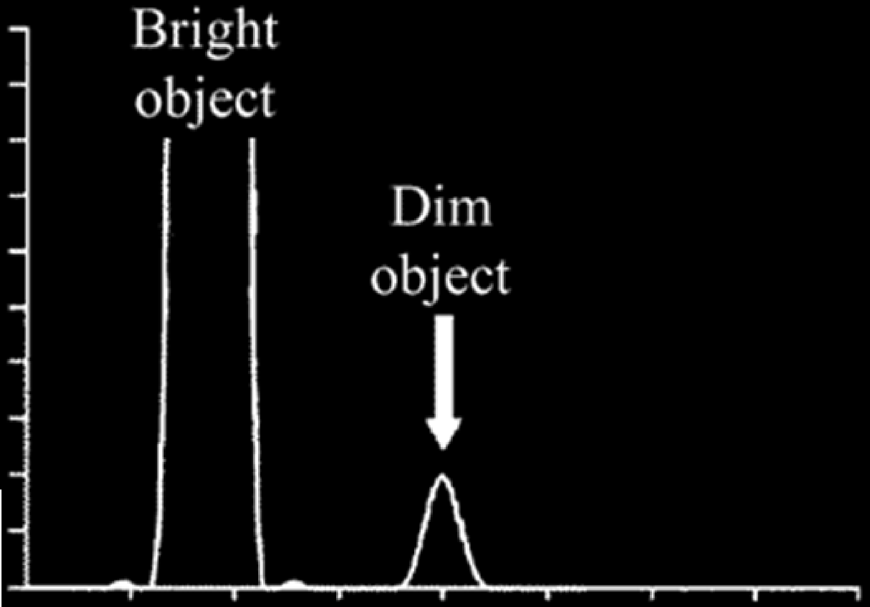
# Confocal microscopy

The relation of the first ring maximum amplitude to the amplitude in the center is 2% in case of conventional point spreading function (PSF) in a focal plane, while in case of a confocal microscope this relation is 0.04%.





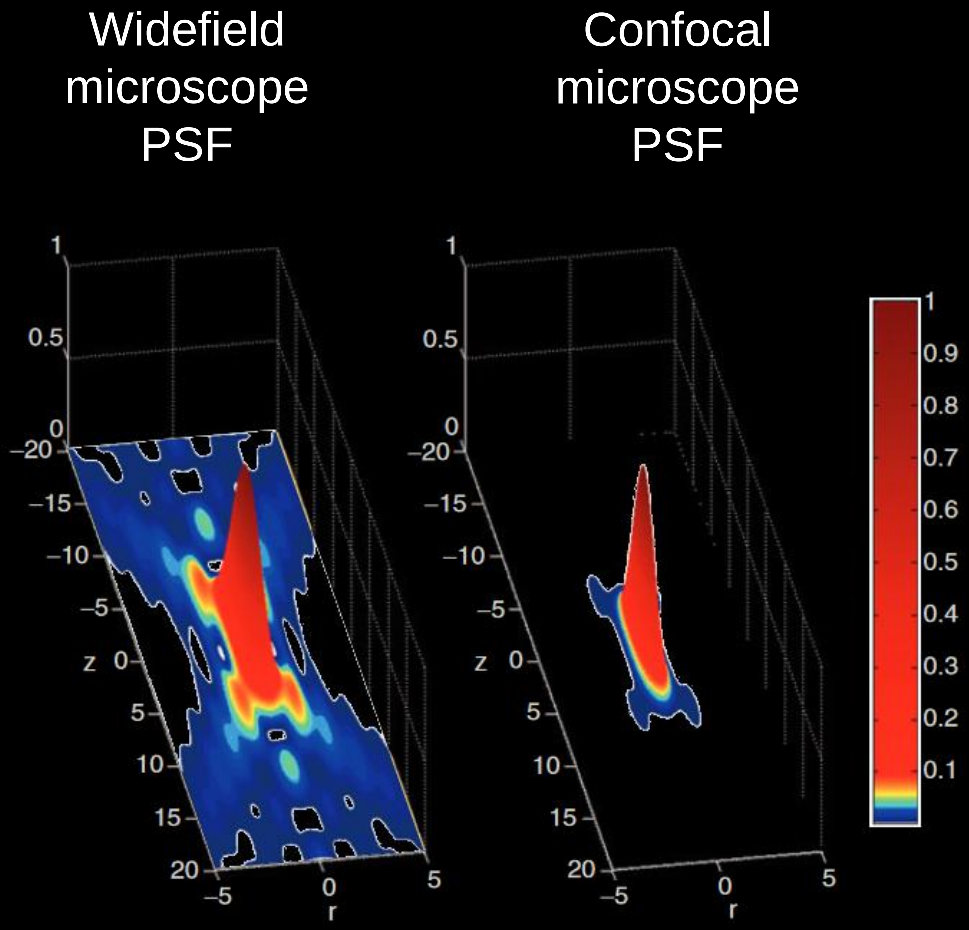




$$r_{\mathrm{resel}} = 0.61\frac{\lambda}{n\sin\theta} = 1.22\frac{\lambda'}{D}F$$

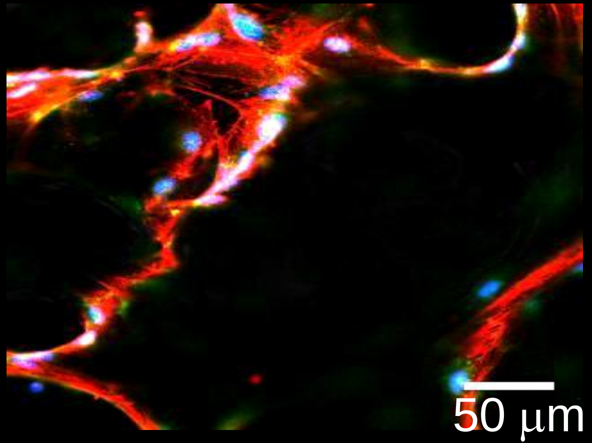
$$r_{\mathrm{conf}} = 0.44\frac{\lambda}{n\sin\theta} = 0.88\frac{\lambda'}{D}F$$

# Confocal microscopy combined with spectroscopy

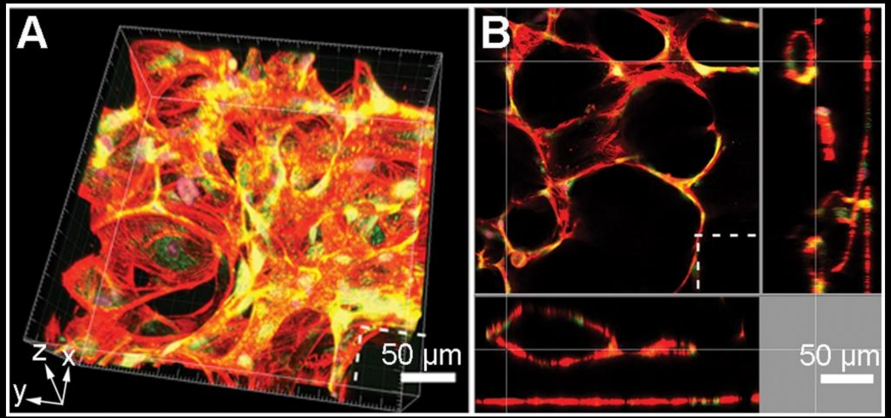


Widefield microscope PSF

Confocal microscope PSF

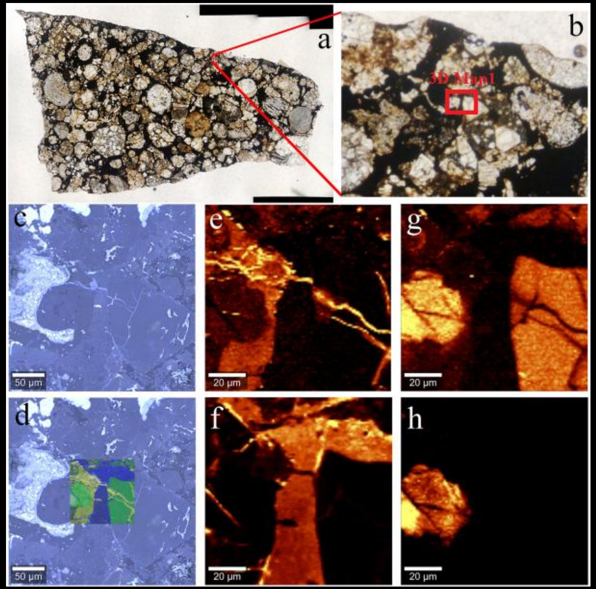


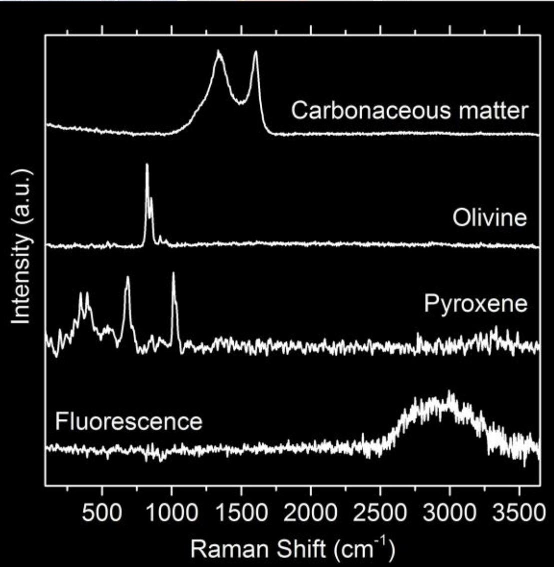
Confocal microscopy reconstruction of a 3D capillary bed



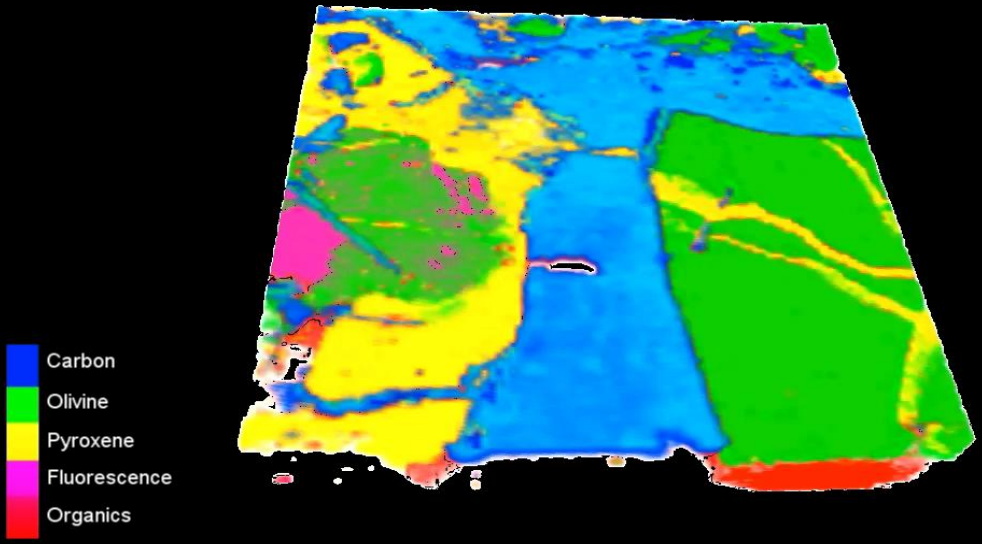
PLOS ONE 7(12): e50582 (2012)

# Confocal microscopy combined with spectroscopy



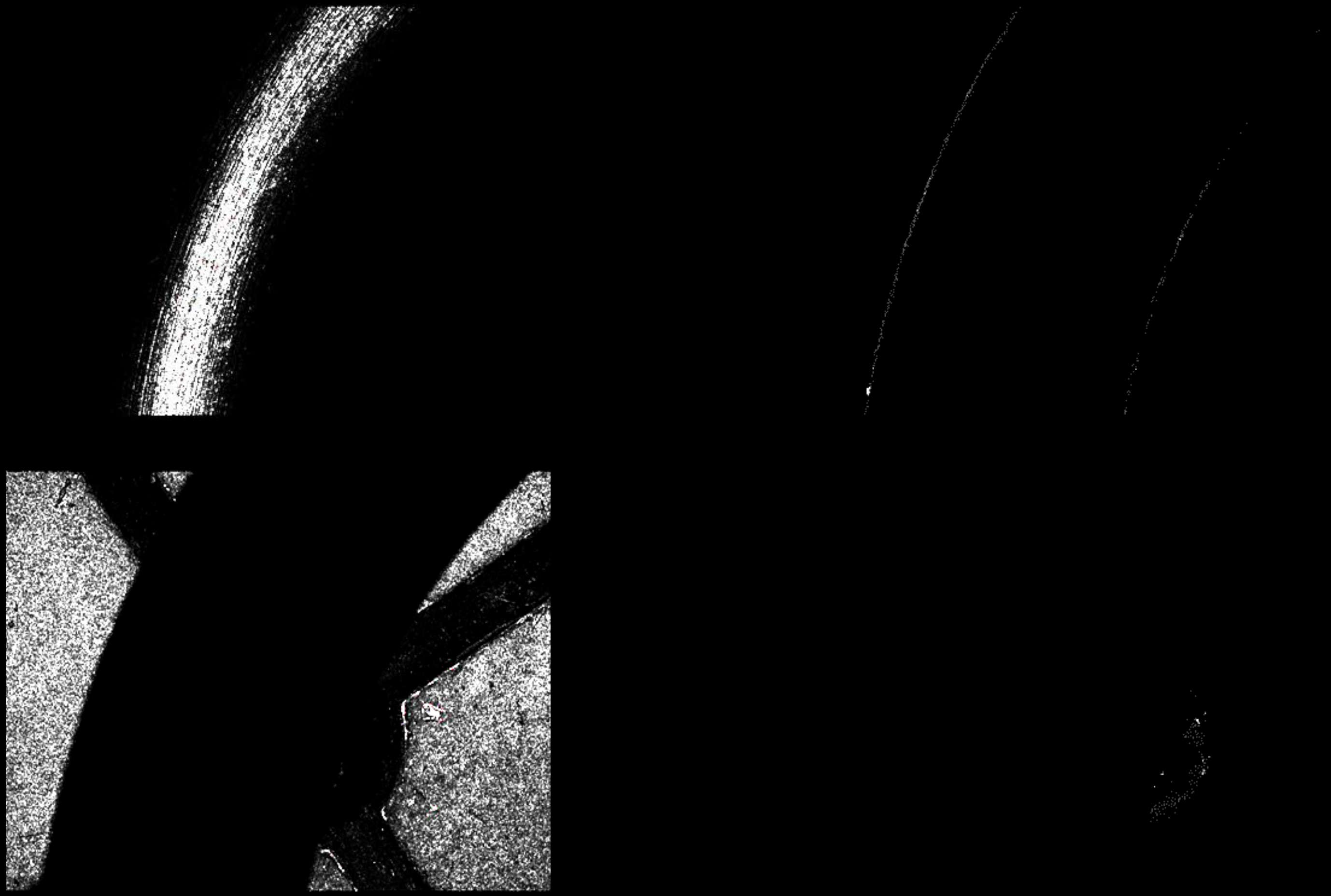


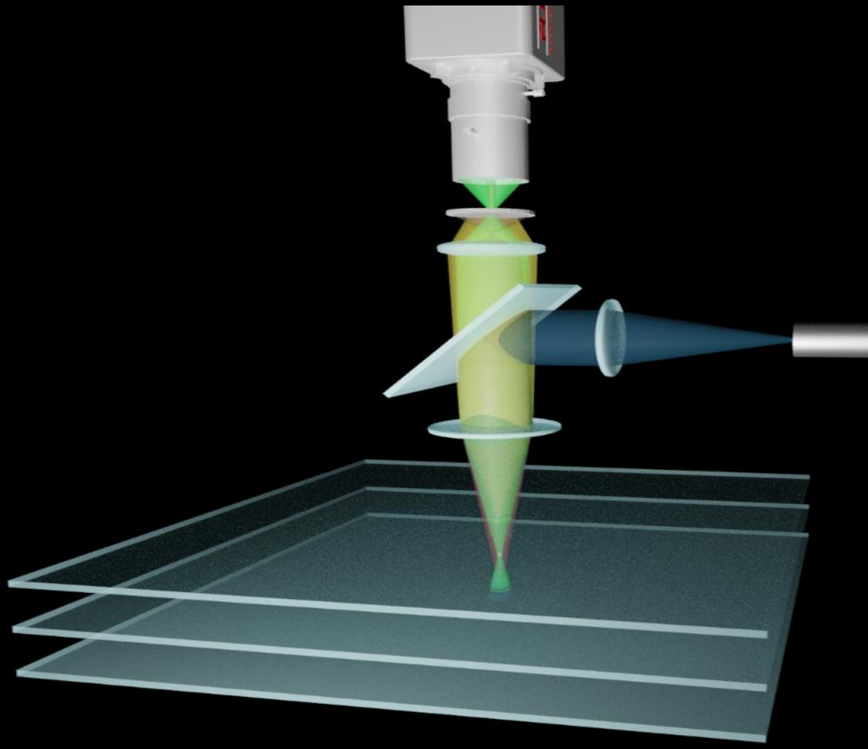
**Chemical composition**
**Component identification**
**Components distribution**



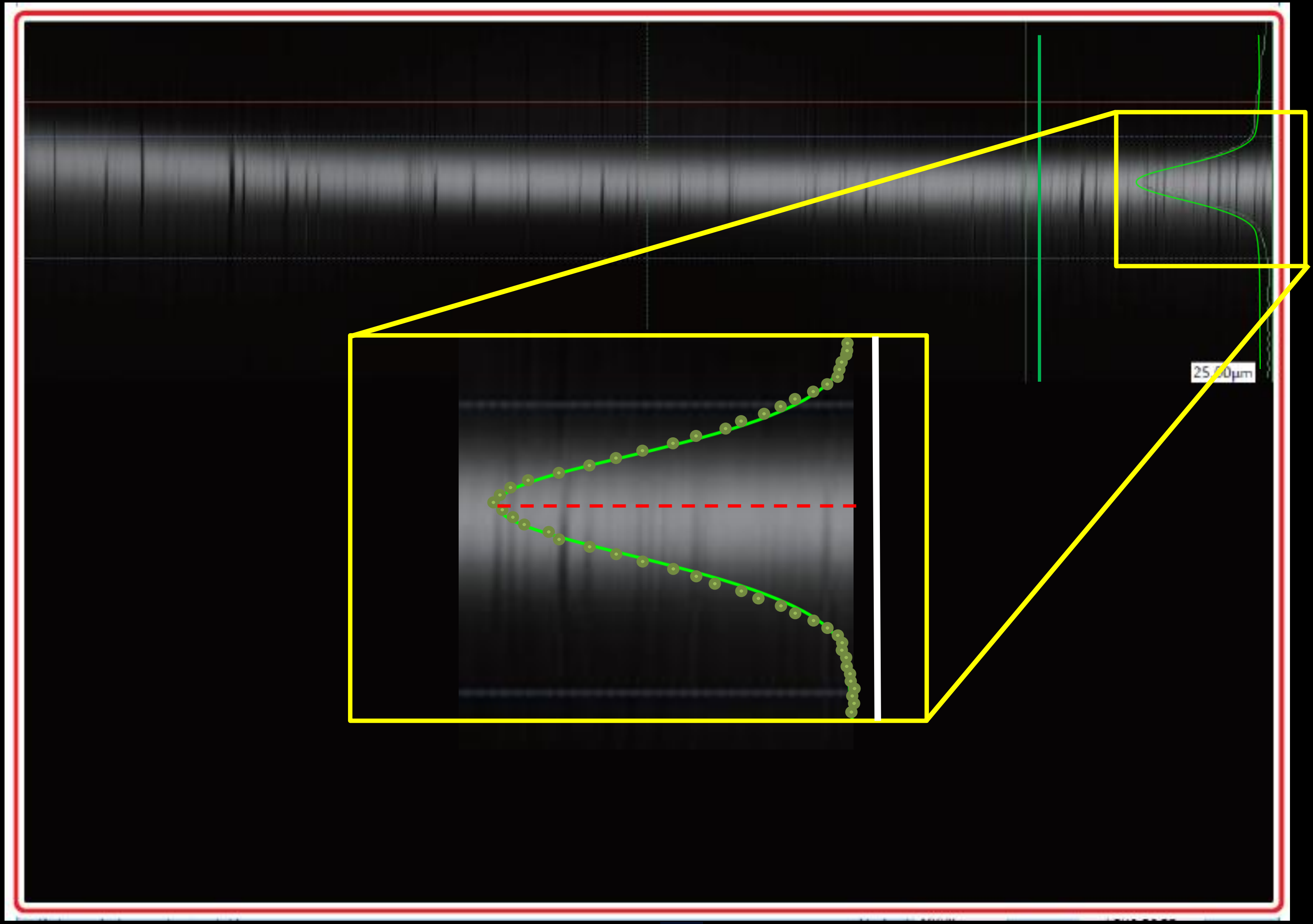
*Earth and Space Science* **5** (8), 380 (2018) DOI: (10.1029/2018EA000369)

# Confocal microscopy z-stack



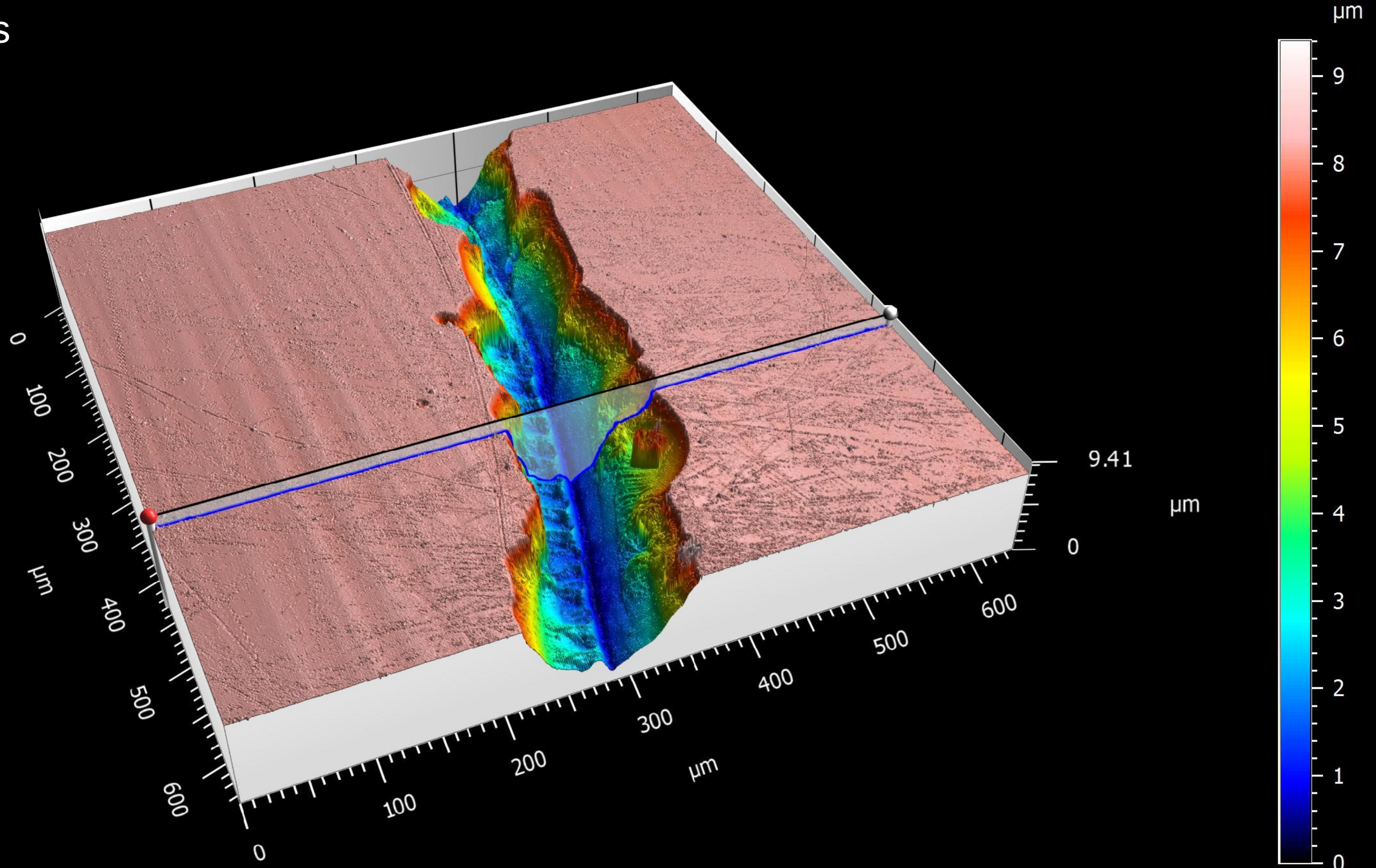


# Confocal microscopy for measuring topography



# Confocal microscopy

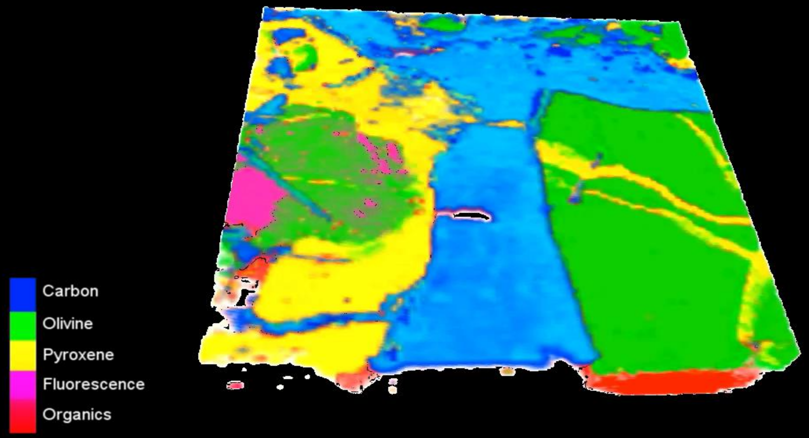
Scratch on glass

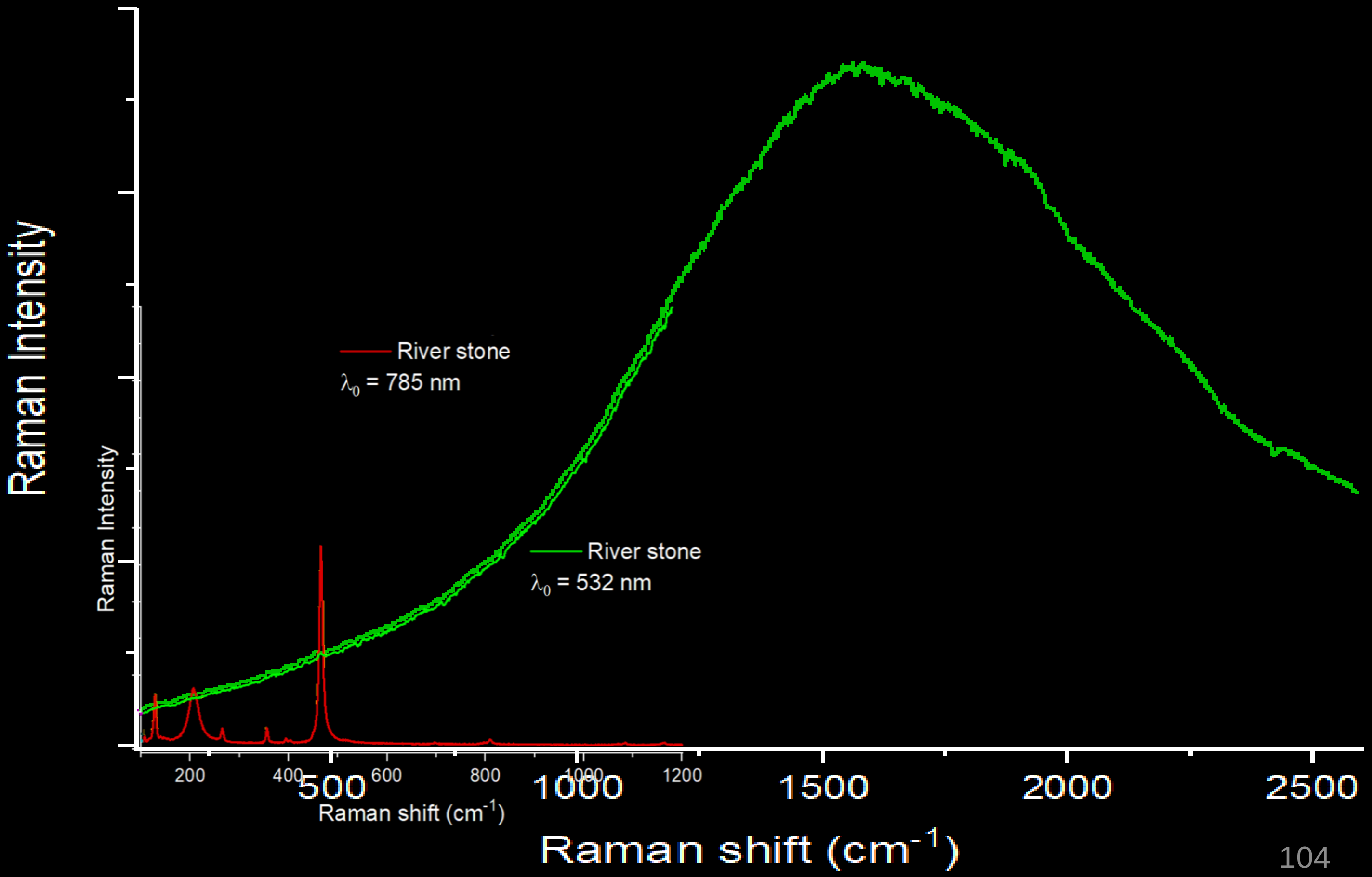


# Raman spectroscopy

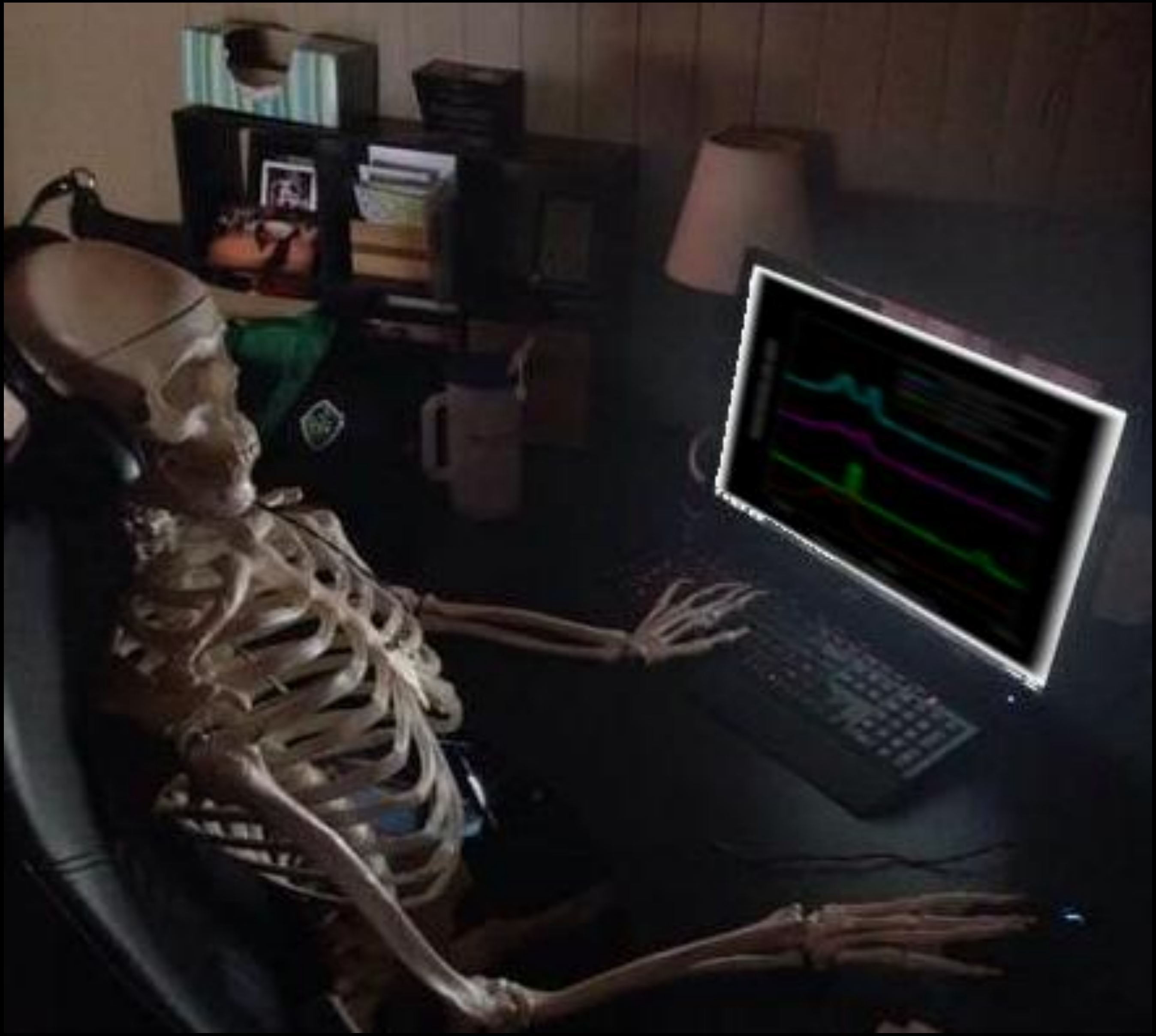
**Primary Limitations:**

- Expensive apparatus (for high spectral/spatial resolution and sensitivity).
- Weak signal, compared to fluorescence.
- Limited spatial resolution (diffraction limited).

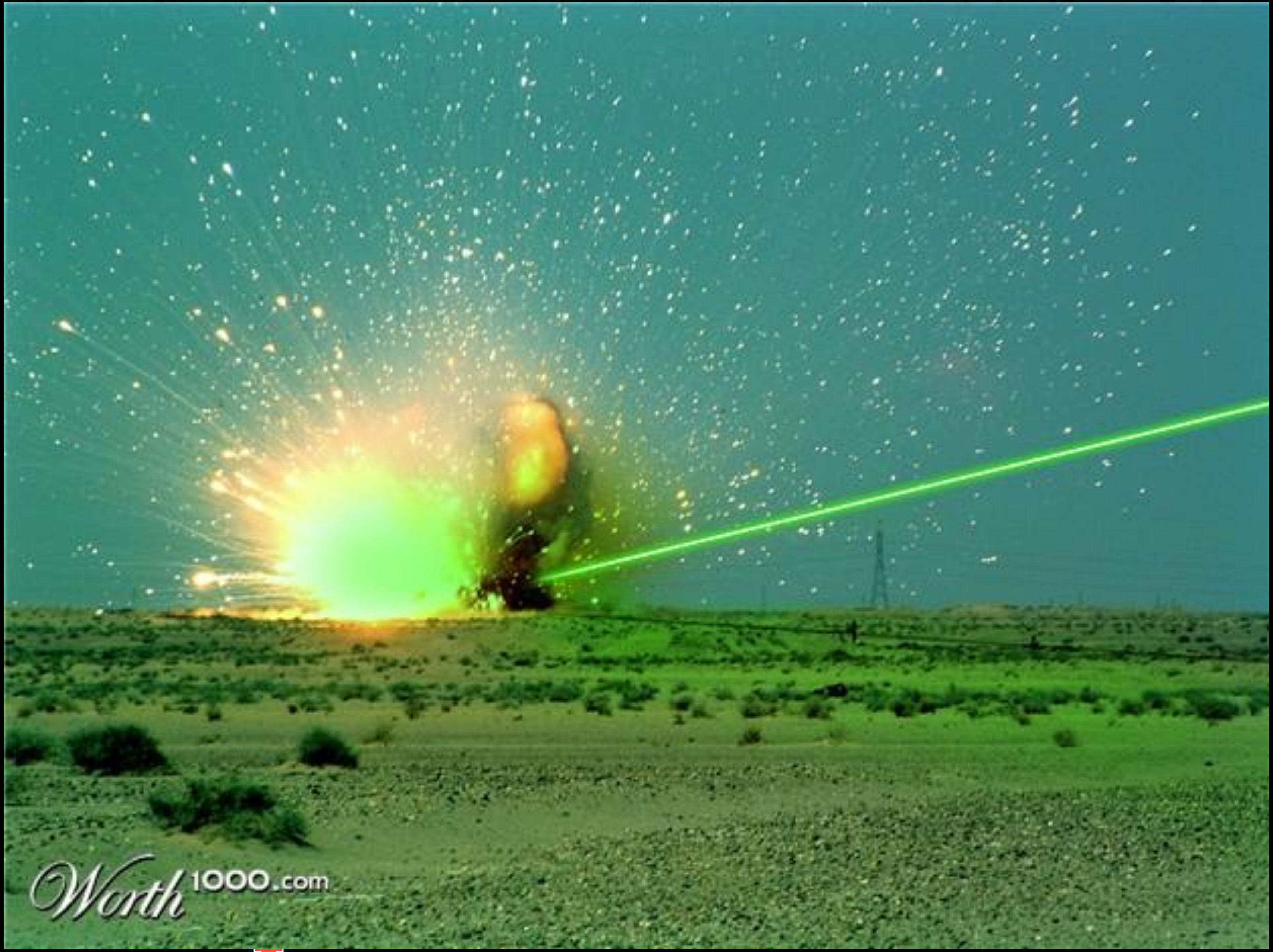




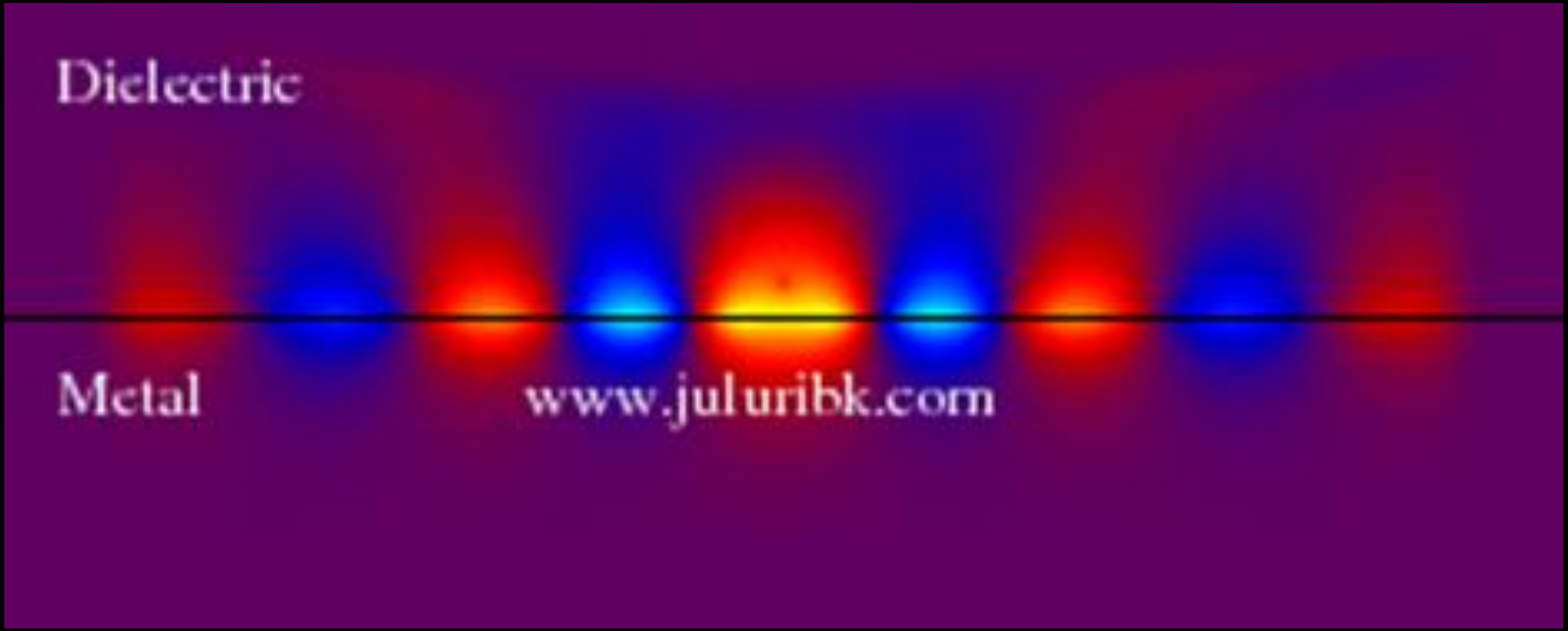
# *The More Time Approach*



# The More Power Approach



# Surface Plasmons



Plasmons can be driven by photons at resonance to build large standing wave electric fields.

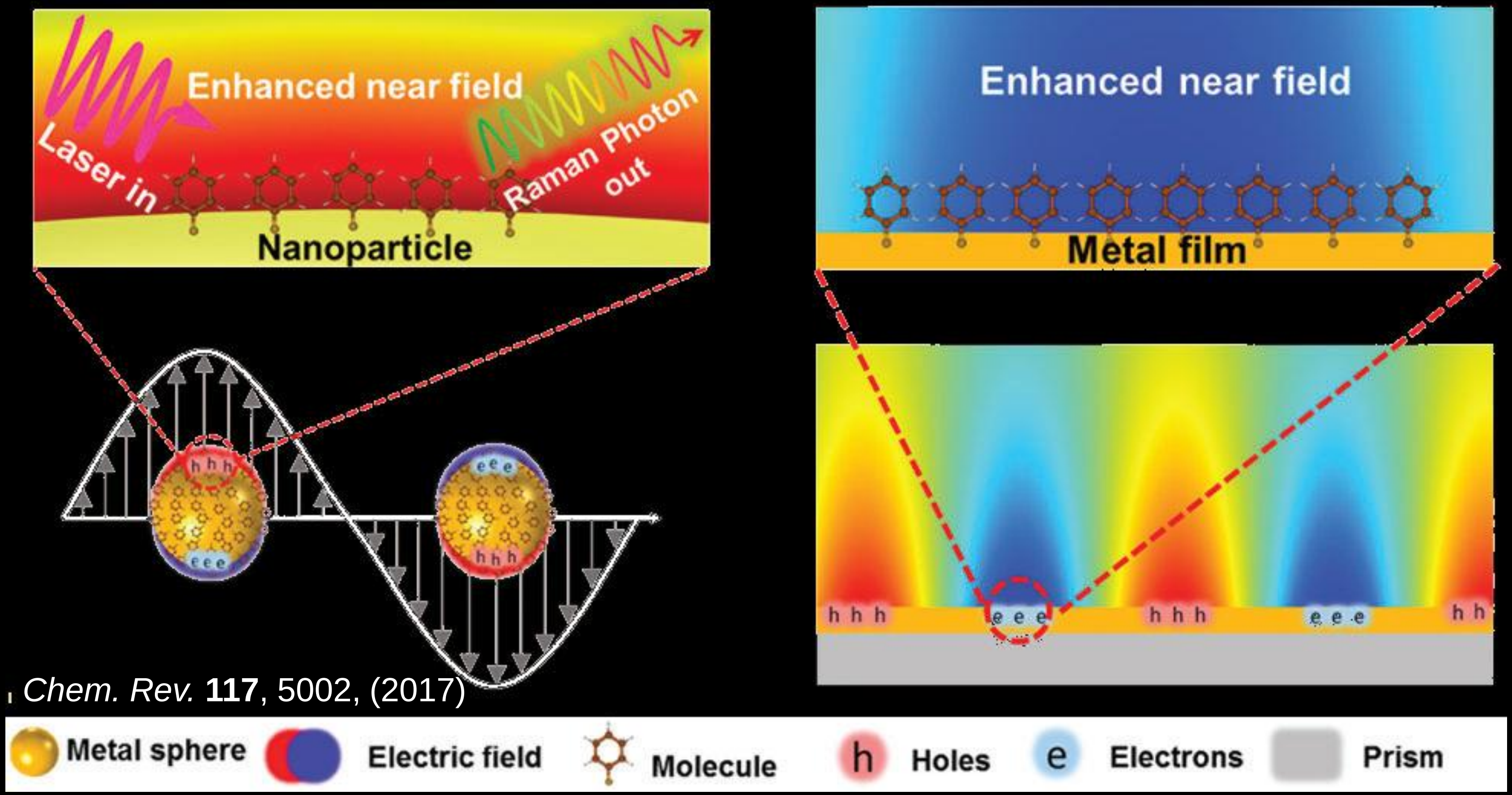
That leads to a strong enhancement of Raman scattering, proportional to the squared E field strength.

$$I = K\nu^4 p_0^2 \sin^2\theta$$

# Surface Enhanced Raman Spectroscopy (SERS)

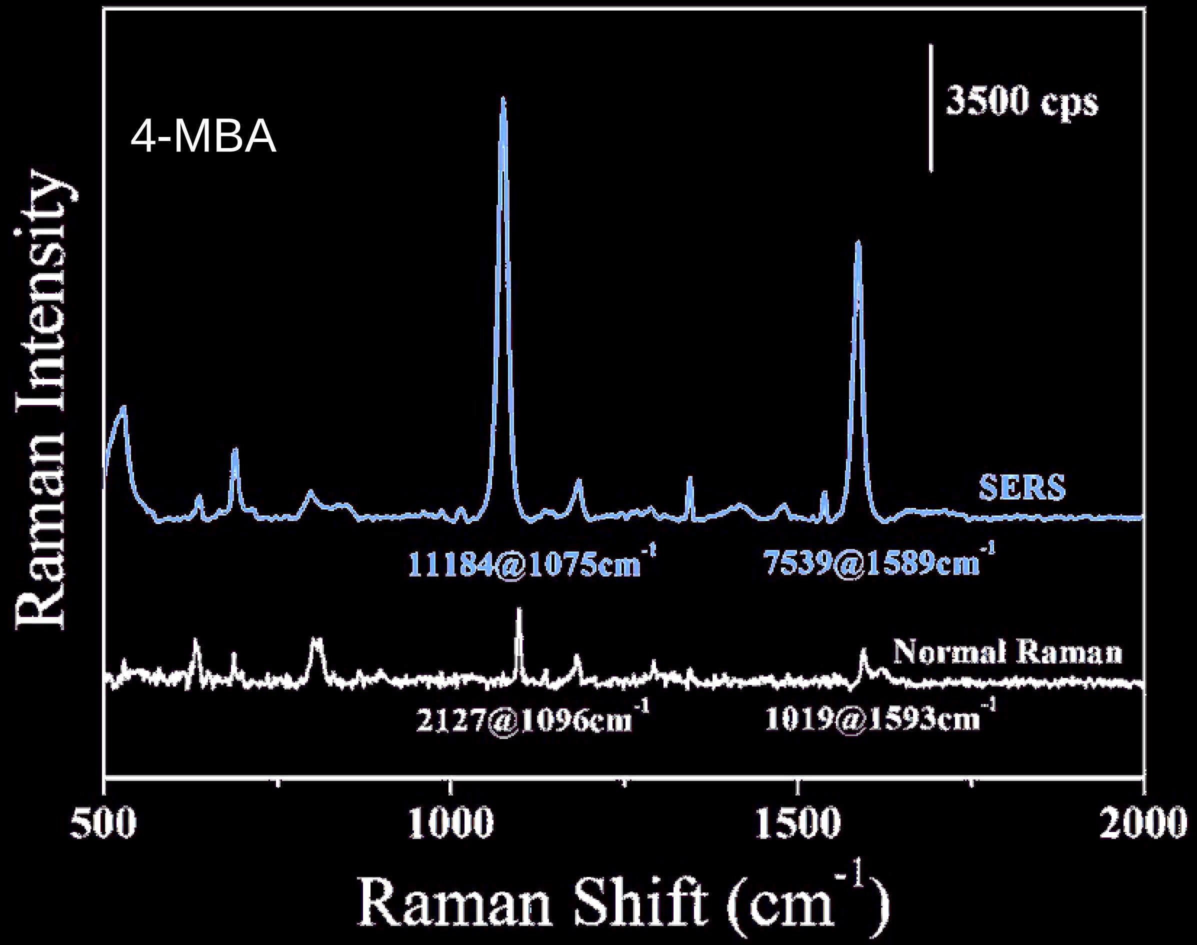
Typically achieved with corrugated gold/silver surface or gold/silver nanoparticles with molecules of interest attached.

Capable of boosting Raman signal up to **14 Orders of Magnitude** or more! *Science* **275**, 1102 (1997)



*Chem. Rev.* **117**, 5002, (2017)

# Surface Enhanced Raman Spectroscopy (SERS)

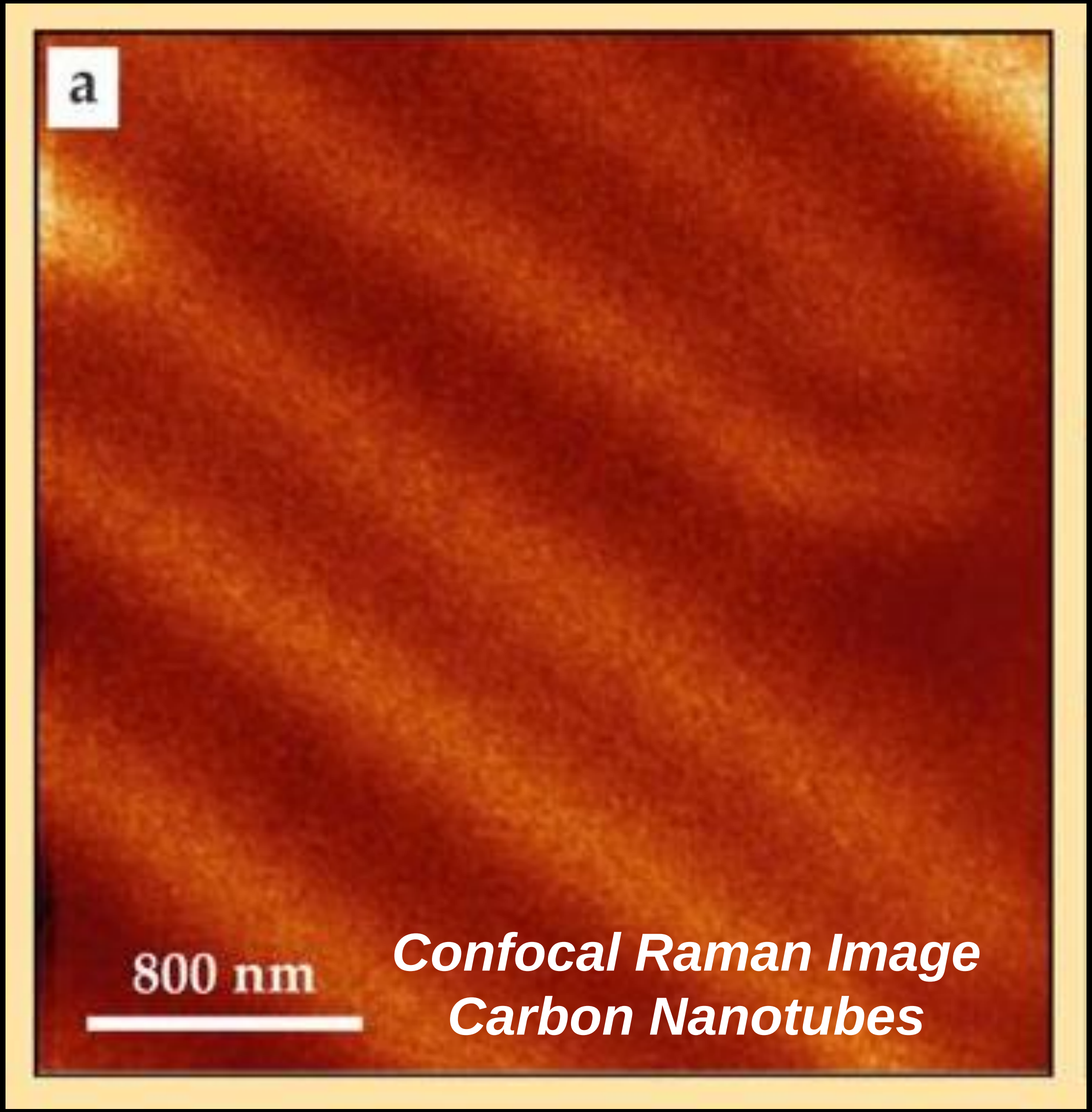


*Anal. Methods*, **6**, 9547 (2014)

# Confocal Raman Microscopy

**That’s cool, but what about …**

- Limited spatial resolution (diffraction limited).

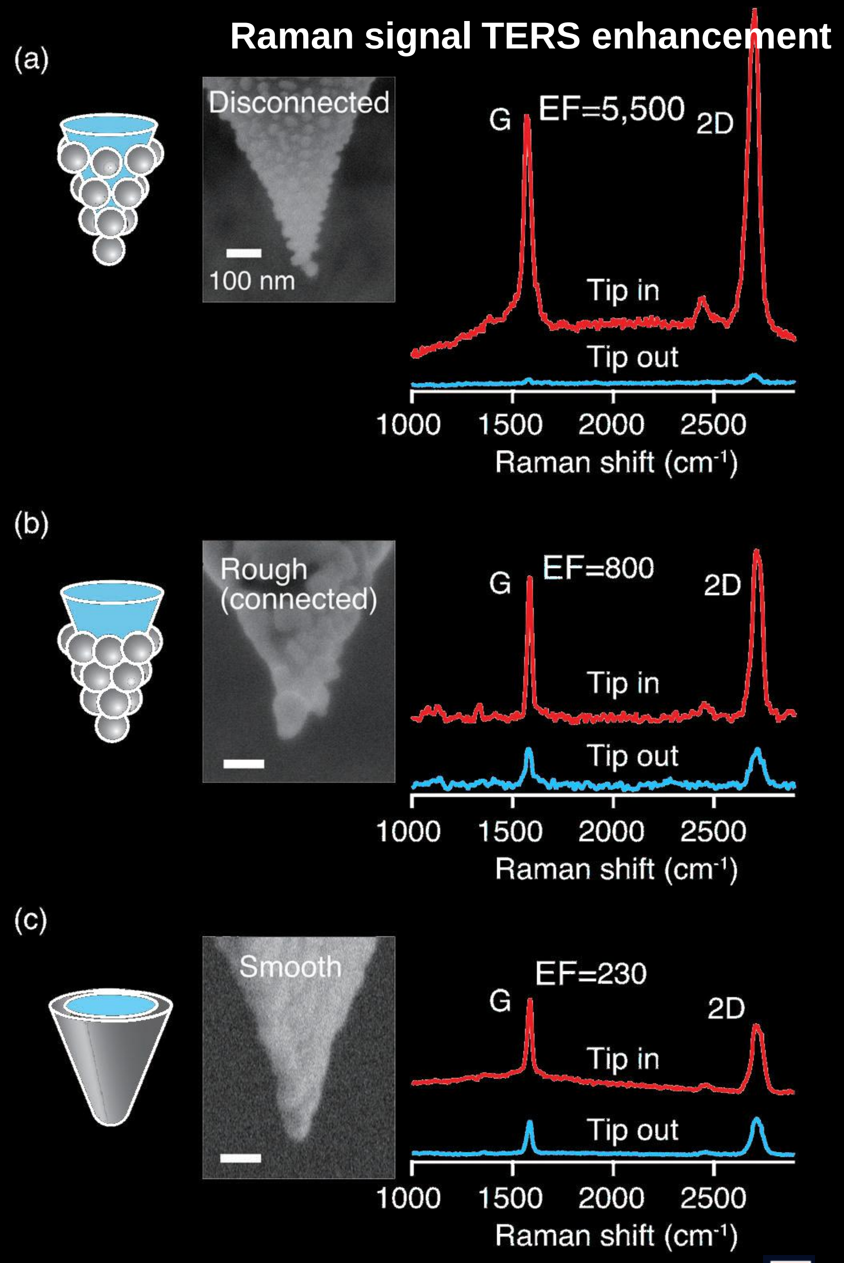


*Confocal Raman Image*
*Carbon Nanotubes*

*Phys. Rev. Lett.***103**, 186101 (2009)

# Tip Enhanced Raman Spectroscopy (TERS)

Raman signal TERS enhancement

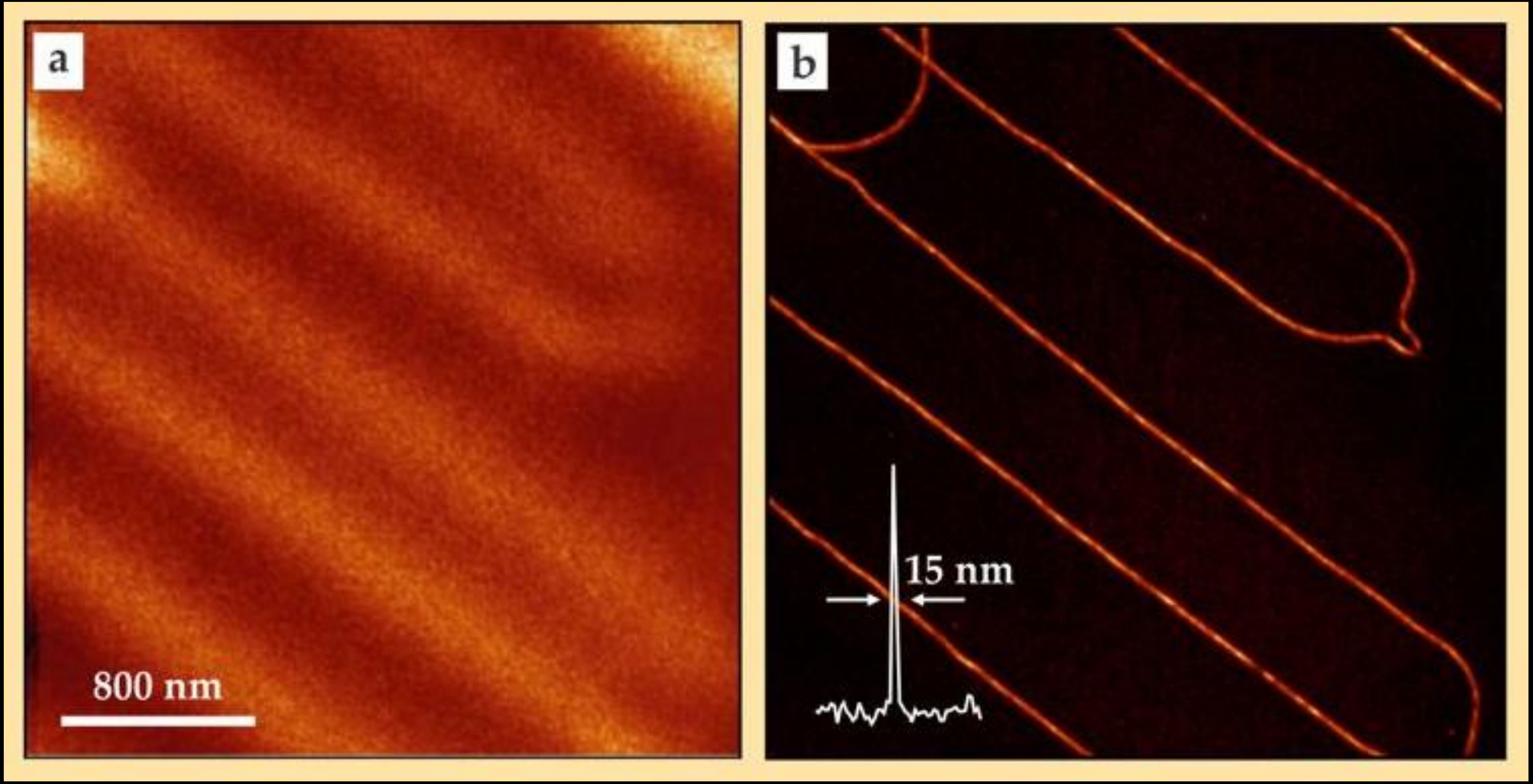


**What is really cool is that this also works with a single metalized sharp tip, such as an STM or AFM tip!**

**Not only do you get the electric field enhancement, but now the source of the Raman signal is extremely localized.**

*Chem. Soc. Rev.* **46**, 4077 (2017)

# Tip Enhanced Raman Spectroscopy (TERS)



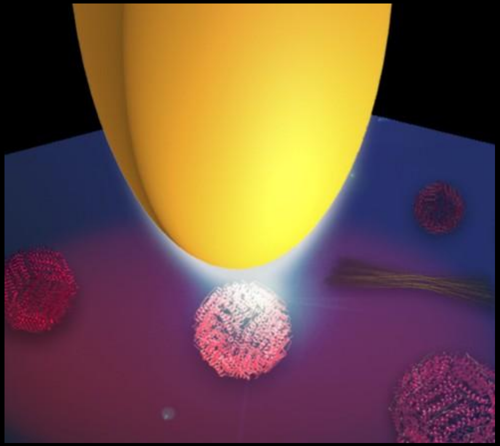
*Confocal Raman Image* *Tip Enhanced Raman Image*

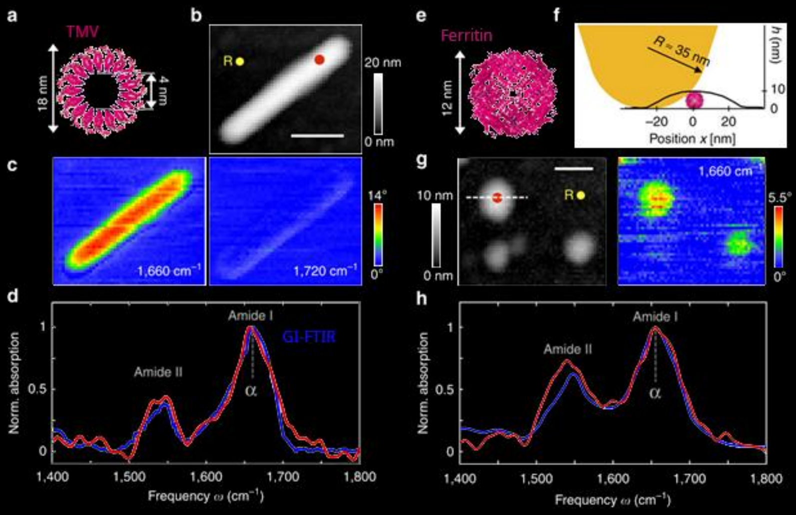
***Carbon Nanotubes***

*Phys. Rev. Lett.* **103**, 186101 (2009)

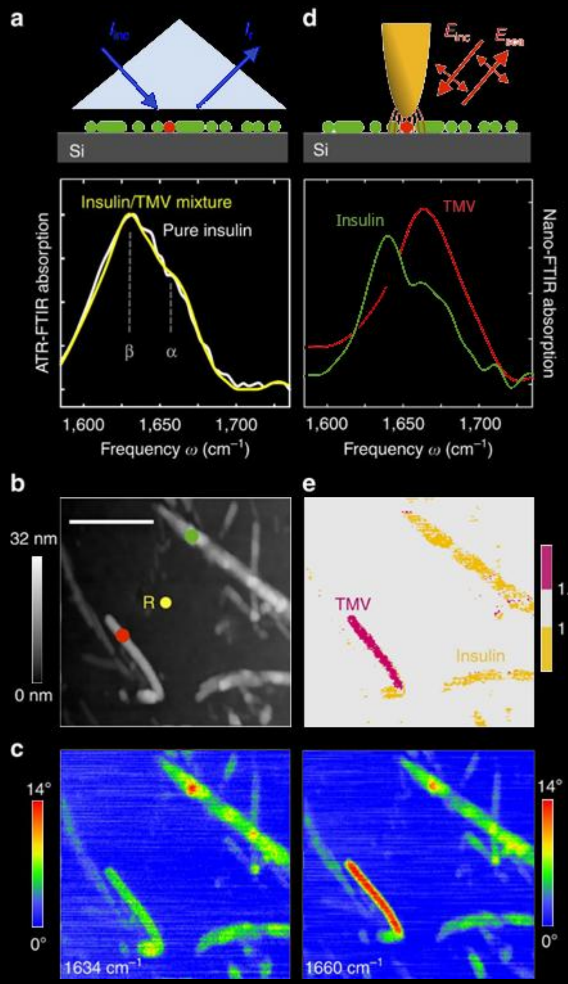
# Near-field scanning optical nanospectroscopy

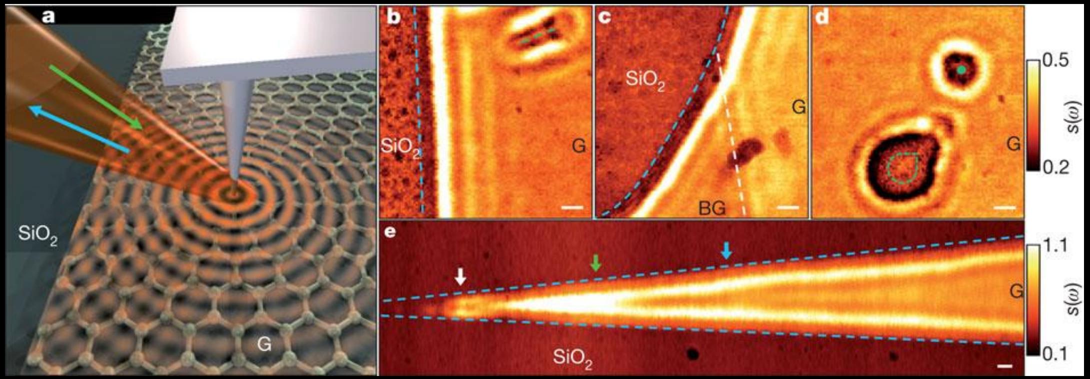
Nano-FTIR



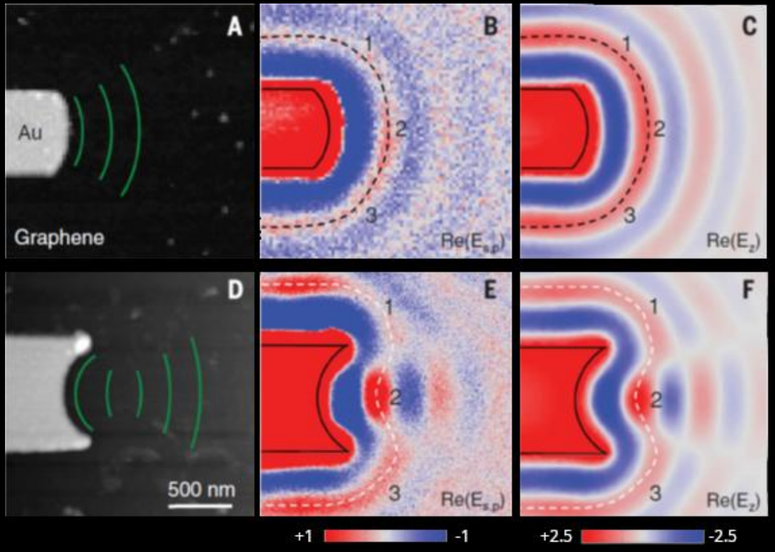


*Nature Communications* **4**, 2890





*Nature* **000**, 1-4 (2012) doi:10.1038/nature11253



Science **344**, 1369

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