Optical materials characterization

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Why optical characterization?

- Material properties that affect its optical response:
  - Temperature
  - Structure
  - Composition
  - Stress
  - Thickness
  - Electronic structure
Why optical characterization?

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Optics Communications, Vol 284(9), 2376-2381 (2011)
Why optical characterization?

Material properties that affect its optical response:

- Temperature
- Structure
- Composition
- Stress
- Thickness
- Electronic structure

Humic acid makes Negro river waters dark
Mud makes Solimões river waters light brown.

Solimões and Negro rivers joining to form the Amazon river.
Why optical characterization?

- Material properties that affect its optical response:
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  - Composition
  - Stress
  - Thickness
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Why optical characterization?

Material properties that affect its optical response:

- Temperature
- Structure
- Composition
- Stress
- Thickness
- Electronic structure

Optics Express 12, 5789 (2004)
Why optical characterization?

- Material properties that affect its optical response:
  - Temperature
  - Structure
  - Composition
  - Stress
  - Thickness
  - Electronic structure
  - Etc.
Why optical characterization?

Many of the fundamental properties of matter were discovered using optical methods and spectroscopy.

- Evidence of energy levels in atoms.
  - Helium was discovered from its absorption lines in the emission spectrum of the sun, from which it got its name.

Helium Emission spectrum

<table>
<thead>
<tr>
<th>Energy Level</th>
<th>Energy Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>3p</td>
<td>-2.43×10^{-19}</td>
</tr>
<tr>
<td>3s</td>
<td>-2.67×10^{-19}</td>
</tr>
<tr>
<td>2p</td>
<td>-5.81×10^{-19}</td>
</tr>
<tr>
<td>2s</td>
<td>-6.37×10^{-19}</td>
</tr>
<tr>
<td>1s</td>
<td>-3.94×10^{-18}</td>
</tr>
</tbody>
</table>
Many of the fundamental properties of matter were discovered using optical methods and spectroscopy.

- Evidence of energy band formation in solids.
Why optical characterization?

- The photoelectric effect

**The Nobel Prize in Physics 1921**

Albert Einstein

"for his services to Theoretical Physics, and especially for his discovery of the law of the photoelectric effect"

The Nobel Foundation

---

The diagram shows the interdependence between light frequency and the maximal energy of electrons emitted from metal. It shows the interdependence for three different metals. See that it clearly shows the limiting frequencies - different for different metals.

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**The Nobel Prize in Physics 1923**

Robert A. Millikan

"for his work on the elementary charge of electricity and on the photoelectric effect"

The Nobel Foundation
Why optical characterization?

Black body radiation, which lead to quantum physics…

The Nobel Prize in Physics 1918
Max Planck

"in recognition of the services he rendered to the advancement of Physics by his discovery of energy quanta"

Copyright 2001 B.M. Tissue

The Nobel Foundation
Why optical characterization?

1907
Albert Abraham Michelson
for his optical precision instruments and the spectroscopic and metrological investigations carried out with their aid

1908
Gabriel Lippmann
method of reproducing colours photographically based on the phenomenon of interference

1918
Max Karl Ernst Ludwig Planck
discovery of energy quanta

1921
Robert Andrews Millikan
Albert Einstein
the photoelectric effect

1922
Niels Henrik David Bohr
investigation of the structure of atoms and of the radiation emanating from them

1923
1930
Sir Chandrasekhara Venkata Raman
discovery of the Raman effect

1931
Nicolaas Bloembergen
Arthur Leonard Schawlow
contribution to the development of laser spectroscopy

1953
Frits (Frederik) Zernike
demonstration of the phase contrast method, especially for his invention of the phase contrast microscope

1964
Charles Hard Townes
Nicolay Gennadiyevich Basov
Aleksandr Mikhailovich Prokhorov
fundamental work in the field of quantum electronics, which has led to the construction of oscillators and amplifiers based on the maser-laser principle

1966
Alfred Kastler
discovery and development of optical methods for studying Hertzian resonances in atoms

1966
Steven Chu
Claude Cohen-Tannoudji
William D. Phillips
development of methods to cool and trap atoms with laser light

1977
Gabriel Lippmann
development of methods to cool and trap atoms with laser light
Why optical characterization?

2005
Roy J. Glauber
John L. Hall
Theodor W. Hänsch

Contributions to the quantum theory of optical coherence and the development of laser-based precision spectroscopy

2009
Charles Kuen Kao
Willard S. Boyle
George E. Smith

Transmission of light in fibers for optical communication and invention of the CCD

2014
Isamu Akasaki
Hiroshi Amano
Shuji Nakamura

The invention of efficient blue light-emitting diodes which has enabled bright and energy-saving white light sources

Eric Betzig
Stephan W. Hell
William E. Moerner

The development of super-resolved fluorescence microscopy
The interaction of radiation with matter
• Interrogating the material properties with light.

  – Transmission/Reflection spectroscopy (identification, structure, concentration, speed, etc.)
    • Spectrophotometry, FTIR
  – Ellipsometry (dielectric function, layer thickness, carrier density, etc.)
  – Raman (identification, structure, phase, crystallinity, stress, drug distribution, diagnosis, etc.)
  – PL/PLE (electronic structure, carrier life-time, defect levels, carrier concentration, size distribution, etc.)
  – Modulation spectroscopy (electronic structure, carrier life-time, defect levels, internal electric fields, thermal properties, mechanical properties, etc.)
    • PR, ER, TDTR, FDTR
  – Optical microscopy
What is measured?
The transmission, and reflection of light as a function of the incident photon energy.

Basic principle:
The absorption, reflection and transmission of light by a material depends on its electronic, atomic, chemical and morphological structure.
Transmission, Reflection, Absorption

UV-VIS-NIR

FTIR
Transmission, Reflection, Absorption

- **Ground state**
- **Excited states**
- **Vibrational states**
- **Impurities**

- **Donor levels**
- **Acceptor levels**

IR, UV/VIS
Transmission, Reflection, Absorption

(a) Electronic absorption transition

(b) H$_2$ emission spectrum (top), H$_2$ absorption spectrum (bottom)
Vibrational modes

Normal vibrational modes in molecules:

- **CO₂ (4 modes)**
  - \( v_1 = 1388 \text{ cm}^{-1} \) symmetric stretch
  - \( v_2 = 667 \text{ cm}^{-1} \) bending
  - \( v_3 = 2349 \text{ cm}^{-1} \) asymmetric stretch

- **H₂O (3 modes)**
  - \( v_1 = 3652 \text{ cm}^{-1} \)
  - \( v_2 = 1595 \text{ cm}^{-1} \)
  - \( v_3 = 3756 \text{ cm}^{-1} \)

- **PO₄ (9 modes)**

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

Normal vibrational modes in solids:
IR active vibrations

The intensity of a vibrational absorption depends on the strength of the transition dipole moment, so a vibration mode $j$ will be “IR active” only when $\left( \frac{\partial \mu}{\partial Q_j} \right)_0 \neq 0$. 

Transmission, Reflection, Absorption
Spectrophotometry (UV-VIS-NIR)

Instrumentation:

Transmission

Diffuse reflectance

Specular reflectance
Instrumentation:
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"

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

$$\Delta L = n\lambda \Rightarrow \text{constructive interference}$$
$$\Delta L = (n+1/2) \lambda \Rightarrow \text{destructive interference}$$
Fourier Transform IR spectroscopy (FTIR)

The Nobel Prize in Physics 1907
Albert A. Michelson

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

\[ \Delta L = n\lambda \Rightarrow \text{constructive interference} \]
\[ \Delta L = (n+1/2)\lambda \Rightarrow \text{destructive interference} \]

Detector voltage vs. time graph:

- Detector voltage
- Time

-10 -8 -6 -4 -2 0 2 4 6 8 10
Spectrum formation:

\[ I(v) = \int_{-\infty}^{\infty} S(t) e^{2i\pi \nu t} dt \]
An example of an interferogram and its corresponding FTIR spectrum.

The signal is detected as intensity vs. time (interferogram).

The Fourier transform of the interferogram gives the spectrum, as intensity vs. wave number.
Advantages:
• Multiplexing (all wavelengths are measured simultaneously).
• No chromatic aberrations or artifacts.
• Higher throughput.
• Improved S/N ratio.

Disadvantages:
• More complex system.
• Linearity of detectors (working closer to saturation levels).
• Stray light scattering at low signal level.

FT vs. Dispersive optics spectrometers
Using absorption to determine Au/Hg concentration in water solutions

As Au relative concentration rises, the absorption peak shifts toward shorter wavelengths, increase in intensity, and its FWHM decreases. The intensity increase follows Beer-Lambert’s law.

Beer-Lambert Law

\[ \text{Abs} = K \ell c = a \ell \]
Using transmission interference fringes to determine thickness

Two sets of interference fringes are present on the spectrum, corresponding to each film that composes the system.

\[ \lambda m = \frac{m}{\nu} = 2nd \sin \theta \]
Excitations in materials

- Plasmons

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.
Plasmonic crystal Brillouin zone from the transmission spectra measured for many different angles of incidence.
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
Fingerprinting:

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

Complementary characterization techniques, like XRD can provide conclusive evidence for the identification.

J. of Archaeological Sci. 39 (2012), 1227
Applications of UV-VIS-NIR spectrophotometry/spectroscopy:

- Quantitative analysis in analytical chemistry and pharmaceutical industry
  - Solute concentrations in solutions
  - Composition of gases
- Structure elucidation of organic compounds (saturated/unsaturated)
- Chemical kinetics, monitoring of chemical reactions
- Analysis of colors in dyes, paints, and raw materials
- Dissociation constants of acids and bases
- Detection of impurities
- Molecular weight determination
- Qualitative analysis
- Chemical analysis of biological processes
- Thin film thickness measurements
Applications of IR spectrophotometry/spectroscopy:

- Quantitative analysis in analytical chemistry and pharmaceutical industry
- Molecular structure analysis
- Thermal analysis
- Characterization of biological molecules
- Disease diagnosis
- Detection of impurities
- Differentiation of microbial cells
- Clinical chemistry
- Qualitative analysis
- Quality control
- Fingerprinting (forensic, art conservation, process monitoring, etc.)
Strengths:

• Very little to no 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).
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.

Complementary techniques:

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

http://www.photophysics.com/

Guimond and Elmore - Oemagazine May 2004
Polarization

**Figure 2**
- Light Waves Vibrating Perpendicular to the Highway
- Light Waves Vibrating Parallel to the Highway

 Incident ray (unpolarised)

 Refracted ray (slightly polarised)

 Reflected ray (polarised)

[www.bobatkins.com](http://www.bobatkins.com)
Polarization

Seven-Segment Liquid Crystal Display (LCD)

Liquid Crystal Sandwich

Positive Electrodes

Negative Electrodes

Polarizer 1

Display

Figure 3

A

B

A

B

C

D

E

F

G

H

I

3D box size: 100 x 100 x 20.3 μm

3D box size: 25 x 25 x 6 μm

3D box size: 25 x 25 x 6 μm

5 μm

3 μm

5 μm

5 μm
What is measured?

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

\[ \tilde{n} = n + ik \]

\[ \tilde{n}_1 \sin \phi_1 = \tilde{n}_2 \sin \phi_2 \]

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

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

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

\[ \tan(\Psi) e^{i\Delta} = \frac{\tilde{R}_p}{\tilde{R}_s} \Rightarrow \begin{cases} \tan(\Psi) = \frac{\left| \tilde{R}_p \right|}{\left| \tilde{R}_s \right|} \\ \Delta = \delta^r - \delta^i \end{cases} \]
Ellipsometry

Applications

- Film thickness

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>21.9 Å</td>
</tr>
<tr>
<td>Si</td>
<td></td>
</tr>
</tbody>
</table>

SiO₂ thickness: 2.19 ± 0.04 nm

$\chi^2 = 4.31$

Model fit

Experiment

- $\Psi$
- $\Delta$

Wavelength (nm)

Ψ (degrees)

Δ (degrees)
Ellipsometry

Applications

- Composition
- Surface roughness
- Film thickness
- Band gap energy

Ellipsometric $\Psi(\lambda)$ and $\Delta(\lambda)$ spectra of Cd$_{1-x}$Zn$_x$S thin films deposited under the different concentration of ammonia: 0.19, 0.38, 0.56, and 0.75 M. The solid lines represent the best fit to the theoretical model. (a) and (b) were measured under 68° and 72°, respectively.

Ellipsometry

Applications

- Composition
- Surface roughness
- Film thickness
- Band gap energy

Parameters of the Cd1-xZnxS thin films as a function of different ammonia concentrations obtained from SE analysis.

<table>
<thead>
<tr>
<th>[NH4OH] (M)</th>
<th>Thickness (nm)</th>
<th>Roughness (nm)</th>
<th>ZnS (%)</th>
<th>Band-gap (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.19</td>
<td>42.12</td>
<td>23.77</td>
<td>99.7</td>
<td>3.49</td>
</tr>
<tr>
<td>0.38</td>
<td>73.79</td>
<td>7.15</td>
<td>45.5</td>
<td>2.52</td>
</tr>
<tr>
<td>0.56</td>
<td>50.89</td>
<td>5.94</td>
<td>32.3</td>
<td>2.45</td>
</tr>
<tr>
<td>0.75</td>
<td>18.59</td>
<td>4.54</td>
<td>5.2</td>
<td>2.43</td>
</tr>
</tbody>
</table>

Ellipsometry

Applications

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

Refractive index (n) and extinction coefficient (k) as a function of wavelength of Cd$_{1-x}$Zn$_x$S thin films under different ammonia concentrations.

Plots of $(\alpha h \nu)^2$ versus photon energy $h$ for Cd$_{1-x}$Zn$_x$S thin films under different ammonia concentrations.

Ellipsometry

Optical Hall Effect

Layer model calculations provide DF:

Electronic contribution

\[ \varepsilon_j(\omega, H) = \varepsilon_{\infty} \cdot \prod_{i=1}^{l} \frac{\omega^2 + i\gamma_{LO,j} \omega - \omega_{LO,j}^2}{\omega^2 + i\gamma_{TO,j} \omega - \omega_{TO,j}^2} \cdot \prod_{k=1}^{m} \left( 1 + \frac{i\delta \gamma_{k,j} \omega - \delta \omega_{k,j}^2}{\omega^2 + i\gamma_{AM,k,j} \omega - \omega_{AM,k,j}^2} \right) - \varepsilon_{j}^{(FC-MO)}(\omega, H) \]

THz ... IR modes

Disorder-induced modes

Free-carrier contribution

Static dielectric constant

Phonon mode frequencies and broadening parameters

Impurity mode parameters

Magnetic field \( H \) causes non-symmetric properties of the IR Dielectric Function tensor:

\[ \varepsilon^{(FC-MO)}(\omega) = -\left( \omega_p^* \right)^2 \left( \omega^2 + i\omega \gamma \right) I - i\omega \langle \omega_c \rangle \begin{bmatrix} 0 & -h_3 & h_2 \\ h_3 & 0 & -h_1 \\ -h_2 & h_1 & 0 \end{bmatrix} \]

Decoupling of \( m_{\text{eff}} \) and free charge carrier concentration

Identification of carrier type (n/p-type)

Determination of free charge carrier concentration, mobility and effective mass possible!!

Plasma (frequency) tensor

\[ \langle \omega_p^* \rangle \equiv \frac{N q^2}{m_e} \]

Cyclotron (frequency) tensor

\[ \langle \omega_c \rangle \equiv \frac{H q}{m_e} \]
Electrical properties

From the fit for the C-face sample:

Two graphene layers with distinctly different properties:

- a bottom \( p \)-type channel with \( N_s=(5.5\pm0.4)\times10^{13} \) cm\(^{-2} \) and \( \mu=1521\pm52 \) cm\(^2 \) V\(^{-1} \) s\(^{-1} \)

- a top \( p \)-type channel with \( N_s=(3.4\pm0.6)\times10^{14} \) cm\(^{-2} \) and \( \mu=18\pm4 \) cm\(^2 \) V\(^{-1} \) s\(^{-1} \)

From electrical dc Hall effect measurement:

\( p \)-type conductivity with \( N_s=(3.0\pm0.5)\times10^{13} \) cm\(^{-2} \) and \( \mu=3407\pm250 \) cm\(^2 \) V\(^{-1} \) s\(^{-1} \)
Ellipsometry

Applications

- Electrical properties

From the fit for the Si-face sample:

A $p$-type channel with $N_s=(1.2\pm0.3)\times10^{12}$ cm$^{-2}$ and $\mu=794\pm80$ cm$^2$ V$^{-1}$ s$^{-1}$

From electrical dc Hall effect measurement:

$p$-type conductivity with $N_s=(1.9\pm0.2)\times10^{12}$ cm$^{-2}$ and $\mu=891\pm250$ cm$^2$ V$^{-1}$ s$^{-1}$

The correspondence between electrical and OHE data is very good.
Ellipsometry

Applications

- Electrical properties

Hole-channel conductivity in epitaxial graphene determined by terahertz optical-Hall effect and midinfrared ellipsometry


C-face:
- Top layer: \( m^* = (0.19 - 0.08\sqrt{B}) m_0 \)
- Bottom layer: \( m^* = 0.035 m_0 \)

Si-face:
- \( m^* = 0.03 m_0 \)
**Ellipsometry**

**Strengths:**
- Fast.
- Measures a ratio of two intensity values and a phase.
  - Highly accurate and reproducible (even in low light levels).
  - No reference sample necessary.
  - Not as 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 usually required to obtain useful information.

Complementary techniques:

PL, Modulation spectroscopies, X-Ray Photoelectron Spectroscopy, Secondary Ion Mass Spectroscopy, XRD.
Lord Rayleigh (John William Strutt)

The Nobel Prize in Physics 1904 was awarded to Lord Rayleigh “for his investigations of the densities of the most important gases and for his discovery of argon in connection with these studies”.

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".
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.
Excitations in materials

- Phonons
- Molecular vibrations


Phonons are the quanta of Collective lattice vibrations
Raman spectroscopy

Inelastic scattering:

The dependence of the polarizability tensor $\tilde{\alpha}$ on the normal coordinate $Q$ associated with a normal vibrational mode of a material, for small amplitude oscillations near the equilibrium can be written:

$$\alpha = \alpha_0 + \left( \frac{\partial \alpha}{\partial Q} \right) Q = \alpha_0 + \alpha' Q$$

For a harmonic oscillation ($Q = Q_0 \cos \omega t$) and $E = E_0 \cos \omega_0 t$, the time dependence of the induced dipole momentum $\mu'$ will be:

$$\mu' = \alpha_0 E_0 \cos \omega_0 t + \frac{1}{2} \alpha' Q_0 E_0 [\cos(\omega_0 - \omega)t + \cos(\omega_0 + \omega)t]$$

meaning that the dipole oscillates with three frequencies simultaneously, corresponding to the three possible scattering modes (Rayleigh, Stokes Raman and anti-Stokes Raman)
The intensity of a vibrational absorption depends on the strength of the transition dipole moment, so a vibration mode \( j \) will be “IR active” only when \( \left( \frac{\partial \mu}{\partial Q_j} \right)_0 \neq 0 \).
Raman active vibrations

The intensity of the Raman scattering linked to a vibrational state depends on the change in the polarizability tensor \( \left( \frac{\partial \alpha}{\partial Q_j} \right)_0 \neq 0 \).
Like the FTIR, Raman spectroscopy measures the interaction of photons with the vibration modes of materials, but the physical processes behind the two techniques are fundamentally different and so are the selection rules that apply to each.

The two techniques are complementary, rather than equivalent.
Molecular and crystalline structure characterization

Raman spectroscopy

Raman is sensitive to the atomic structure of the material.

Physics Reports, 409 (2005), 47
Chemical composition & component identification

Components distribution at micron & sub-micron scale

![Graph showing Raman shifts for Salbutamol and BDP]

Distribution of ingredients in a pharmaceutical tablet

The AAPS Journal 2004; 6 (4), 32
Molecular and crystalline structure characterisation

Presence of N vacancies yields poor crystallinity

Substitutional C fills N vacancies improving the crystallinity

C incorporates interstitially causing a degradation of the crystal lattice

Raman spectroscopy

Raman intensity (arb. units)

GaN: C
10 K
λ<sub>L</sub> = 514.5 nm

400 600 800
Raman Shift (cm<sup>-1</sup>)

G H I J

< C

> C

PHYSICAL REVIEW B 68, 155204 (2003)
Raman spectroscopy

Stress measurements

Mapping the Raman peak position of a micro indentation in a silicon wafer.

Phase transition monitoring

Fig. 1. Temperature dependence of the low frequency Raman modes for the tungsten trioxide $\text{WO}_3$ powder across the monoclinic-to-orthorhombic phase transition.

Renishaw, Inc.

A complete Raman mapping of phase transitions in Si under indentation


(a) Different Raman Modes

<table>
<thead>
<tr>
<th>Si-I (cm$^{-1}$)</th>
<th>Si-III (cm$^{-1}$)</th>
<th>Si-XII (cm$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>300, 520</td>
<td>166, 171</td>
<td>182</td>
</tr>
<tr>
<td>a-Si (cm$^{-1}$)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>384</td>
<td>352</td>
<td></td>
</tr>
<tr>
<td>475, 510</td>
<td>432, 463</td>
<td>397</td>
</tr>
</tbody>
</table>

$^\dagger$ Ref 2,3
Raman spectroscopy is able to clearly distinguish areas of differing numbers of layers in thin graphene sheets.

Graphene monolayer, bilayer and other multiple-layer regions identified

Oriented CNT between two electrodes
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.

Complementary techniques:
FTIR, EELS, Mass spectroscopy, EXAFS, XPS, AES, SIMS, XRD, SFG.
Raman spectroscopy

- Ground state
- Excited states
- Virtual states
- Donnor levels
- Acceptor levels
- Impurities
- Luminescence
- Raman scattering
- Vibrational states
- Excitation

Diagram showing transitions between ground and excited states, involving impurities, donnor and acceptor levels, and various vibrational states leading to luminescence and Raman scattering.
Raman spectroscopy

Impurities

Donor levels

Excited states

Virtual states

Vibrational states

Ground state

Luminescence

Raman scattering

Excitation
Luminescence

Lifetime: Phosphorescence, fluorescence
Mechanism: Photoluminescence, bioluminescence, chemoluminescence, thermoluminescence, piezoluminescence, etc.
Photoluminescence

Charles Hedgcock © University of Arizona, Tucson, AZ

http://uclagettypogram.wordpress.com

http://www.evidentcrimescene.com

http://kevincollinsphoto.smugmug.com

Visible Light

UV Light ($\lambda_{\text{exc max}}=365\text{nm}$)

CrimeScope ($\lambda_{\text{exc max}}=415\text{nm}$)

http://uclagettypogram.wordpress.com
Photoluminescence

www.glofish.com
What is measured?

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

**Basic principle:**

The impinging light promote electrons from the less energetic levels to excited levels, forming electron-hole pairs. As the electrons and holes recombine, they may release some of the energy as photons. The emitted light is called luminescence.
Excitations in materials

- Excitons
- Bound excitons
- Excitonic complexes

Exciton describes the bound state of an electron-hole pair due their mutual Coulomb attraction

Peter Abbamonte et al., *Proc. Nat. Acad. Sci.* 105 (34)
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).

Solid lines show the theoretical fitting curves based on a model of interband recombination in degenerated semiconductors. As a result, the true value of InN band gap $E_g \sim 0.7$ eV was established.

Photoluminescence

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.

Photoluminescence

Conduction band

Valence band

Photoluminescence

GaAs 4.2K

PL INTENSITY (arb. unit)

WAVELENGTH (nm)

PHOTON ENERGY (eV)

Shigenori Takagishi and Hideki Mori

Japanese Journal of Applied Physics

Using PL to determine the width and quality of InGaAsN/GaAs quantum wells.

<table>
<thead>
<tr>
<th>Layer</th>
<th>Material</th>
<th>Thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>100 nm</td>
<td>GaAs</td>
<td>cap</td>
</tr>
<tr>
<td>3 nm</td>
<td>InGaAsN QW</td>
<td></td>
</tr>
<tr>
<td>35 nm</td>
<td>GaAs</td>
<td>barrier</td>
</tr>
<tr>
<td>5 nm</td>
<td>InGaAsN QW</td>
<td></td>
</tr>
<tr>
<td>35 nm</td>
<td>GaAs</td>
<td>barrier</td>
</tr>
<tr>
<td>9 nm</td>
<td>InGaAsN QW</td>
<td></td>
</tr>
<tr>
<td>100 nm</td>
<td>GaAs</td>
<td>buffer</td>
</tr>
<tr>
<td></td>
<td>GaAs (001) SUB</td>
<td></td>
</tr>
</tbody>
</table>
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.
What is measured?

The reflectance variation with temperature.

Basic principle:

The dielectric function of a material, and thus its reflectance, is a function of temperature. By modulating the material’s temperature, we can measure the variation in its reflectivity, caused by that modulation. With time resolved measurements, we can calculate the thermal conductivity of the sample.
What is measured?
The reflectance variation with temperature.

Basic principle:
The dielectric function of a material, and thus its reflectance, is a function of temperature. By modulating the material’s temperature, we can measure the variation in its reflectivity, caused by that modulation. With time resolved measurements, we can calculate the thermal conductivity of the sample.
**Instrumentation:**

- fs Ti:Sapphire laser operating in the range 700-800 nm.
- A variable delay line in the pump beam path for time resolution.
- Scanning sample stage.
- Cryostats.
- Magnetic field.

**Applications of TDTR include:**

Spatially resolved thermal and elastic properties of materials:

- Thermal effusivity ($\Delta C$) and conductivity.
- Mechanical properties (by determination of the speed of sound).

Ultralow Thermal Conductivity in Disordered, Layered WSe$_2$ Crystals

Catalin Chiritecu, David G. Cahill,$^1$ Ngoc Nguyen,$^2$ David Johnson,$^2$ Arun Bodapati,$^3$ Pawel Keblinski,$^3$ Paul Zschack$^4$


The lowest thermal conductivity so far observed for a fully dense solid. (48 mWm$^{-1}$K$^{-1}$)
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Catalin Chiritescu, David G. Cahill, Ngoc Nguyen, David Johnson, Arun Bodapati, Pawel Keblinski, Paul Zschack


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Taeyoon Lee, K. Ohmori, C.-S. Shin, David G. Cahill, I. Petrov, and J. E. Greene

PHYSICAL REVIEW B 71, 144106 (2005)
Elastic constants of single-crystal TiN$_x$(001) (0.67 ≤ x ≤ 1.0) determined as a function of x by picosecond ultrasonic measurements

Taeyoon Lee, K. Ohmori, C.-S. Shin, David G. Cahill, I. Petrov, and J. E. Greene
PHYSICAL REVIEW B 71, 144106 (2005)
**Strengths:**

- Thermoconductivity over a broad range can be measured.
- Can measure interface thermal conductance.
- Spatial resolution.
- Measurements can be made over a wide range of temperatures.
- Fast.

Limitations:
- Samples need coating with an Al thin film.
- Alignment can be tedious.
- Thermal conductivity cannot be measured for films much thinner than the thermal penetration depth.

Complementary techniques:
$3\omega$ method, Raman spectroscopy, Nanoindentation.
Optical microscopy

Figure 6

Von Leeuwenhoek Microscope (circa Late 1600s)
British Microscope (circa 1865)
Hand-Held Microscope (circa early 1700s)

Zeiss Laboratory Microscope circa 1930s

Hooke Microscope circa 1670

Focus Knob
Sample Translator
Sample Holder
Lens

Eyepiece
Body Tube
Objective
Mechanical Stage
Condenser Mirror
Base

Oil Lamp
Water Flask

Focusing Screw
Objective
Specimen Holder
"Classical" Optical Microscopy

**Image Formation**

- In the optical microscope, light from the microscope lamp passes through the condenser and then through the specimen (assuming the specimen is a light absorbing specimen).

- Some of the light passes both around and through the specimen undisturbed in its path (direct light or undeviated light).
  - The direct or undeviated light is projected by the objective and spread evenly across the entire image plane at the diaphragm of the eyepiece.

- Some of the light passing through the specimen is deviated when it encounters parts of the specimen (deviated light or diffracted light).
Optical microscopy

- **Astigmatism Aberration**
  - Airy Diffraction Pattern
  - Tangential (Meridional) Focal Plane
  - Sagittal Focal Plane
  - Circle of Least Confusion
  - Objective
  - Optical Axis
  - Airy Diffraction Pattern
  - Figure 4

- **Axial Chromatic Aberration**
  - White Light
  - Blue Light
  - Red Light
  - Optical Axis
  - Simple Thin Lens
  - Green Light
  - Focal Point (Red Light)
  - Figure 1

- **Spherical Aberration**
  - Monochromatic Red Light
  - Optical Axis
  - Simple Thin Lens
  - Focal Point 2
  - Focal Point 3
  - Figure 2

- **Field Curvature Aberration**
  - Flat Specimen Surface
  - Concave Spherical Image Surface
  - Simple Lens
  - Figure 5

- **Off-Axis Coma Aberration**
  - Zone 1
  - Zone 2
  - Zone 3
  - Zone 4
  - Optical Axis
  - Lens
  - Coma Blur
  - Figure 3
Optical microscopy

<table>
<thead>
<tr>
<th>Objective Type</th>
<th>Spherical Aberration</th>
<th>Chromatic Aberration</th>
<th>Field Curvature</th>
</tr>
</thead>
<tbody>
<tr>
<td>Achromat</td>
<td>1 Color</td>
<td>2 Colors</td>
<td>No</td>
</tr>
<tr>
<td>Plan Achromat</td>
<td>1 Color</td>
<td>2 Colors</td>
<td>Yes</td>
</tr>
<tr>
<td>Fluorite</td>
<td>2-3 Colors</td>
<td>2-3 Colors</td>
<td>No</td>
</tr>
<tr>
<td>Plan Fluorite</td>
<td>3-4 Colors</td>
<td>2-4 Colors</td>
<td>Yes</td>
</tr>
<tr>
<td>Plan Apochromat</td>
<td>3-4 Colors</td>
<td>4-5 Colors</td>
<td>Yes</td>
</tr>
</tbody>
</table>
Optical microscopy

Phase contrast  Bright field  Dark field  Polarizing
Optical microscopy

Difference interference contrast

![DIC Microscope Optical Pathways](image)
Optical microscopy

- Phase contrast
- Bright field
- Dark field
- Polarizing

Fibers in bright field and dark field

Living Cells in Brightfield and Phase Contrast

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

Phyllite Thin Section in Polarized Light
Optical microscopy

Fluorescence
## Optical microscopy

### Contrast-Enhancing Techniques for Optical Microscopy

<table>
<thead>
<tr>
<th>Specimen Type</th>
<th>Imaging Technique</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Transmitted Light</strong></td>
<td></td>
</tr>
<tr>
<td><strong>Transparent Specimens</strong></td>
<td></td>
</tr>
<tr>
<td><strong>Phase Objects</strong></td>
<td></td>
</tr>
<tr>
<td>Bacteria, Spermatozoa,</td>
<td>Phase Contrast</td>
</tr>
<tr>
<td>Cells in Glass Containers,</td>
<td>Differential Interference Contrast (DIC)</td>
</tr>
<tr>
<td>Protozoa, Mites, Fibers, etc.</td>
<td>Hoffman Modulation Contrast</td>
</tr>
<tr>
<td></td>
<td>Oblique Illumination</td>
</tr>
<tr>
<td><strong>Light Scattering Objects</strong></td>
<td></td>
</tr>
<tr>
<td>Diatoms, Fibers, Hairs,</td>
<td>Rheinberg Illumination</td>
</tr>
<tr>
<td>Fresh Water Microorganisms,</td>
<td>Darkfield Illumination</td>
</tr>
<tr>
<td>Radiolarians, etc.</td>
<td>Phase Contrast and DIC</td>
</tr>
<tr>
<td><strong>Light Refracting Specimens</strong></td>
<td></td>
</tr>
<tr>
<td>Colloidal Suspensions</td>
<td>Phase Contrast</td>
</tr>
<tr>
<td>powders and minerals</td>
<td>Dispersion Staining</td>
</tr>
<tr>
<td>Liquids</td>
<td>DIC</td>
</tr>
<tr>
<td><strong>Amplitude Specimens</strong></td>
<td></td>
</tr>
<tr>
<td>Stained Tissue</td>
<td>Brightfield Illumination</td>
</tr>
<tr>
<td>Naturally Colored Specimens</td>
<td></td>
</tr>
<tr>
<td>Hair and Fibers</td>
<td></td>
</tr>
<tr>
<td>Insects and Marine Algae</td>
<td></td>
</tr>
<tr>
<td><strong>Fluorescent Specimens</strong></td>
<td></td>
</tr>
<tr>
<td>Cells in Tissue Culture</td>
<td></td>
</tr>
<tr>
<td>Fluorochrome-Stained Sections</td>
<td></td>
</tr>
<tr>
<td>Smears and Spreads</td>
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</tr>
<tr>
<td><strong>Birefringent Specimens</strong></td>
<td></td>
</tr>
<tr>
<td>Mineral Thin Sections</td>
<td></td>
</tr>
<tr>
<td>Liquid Crystals</td>
<td></td>
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<tr>
<td>Melted and Recrystallized Chemicals</td>
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</tr>
<tr>
<td>Hairs and Fibers</td>
<td></td>
</tr>
<tr>
<td>Bones and Feathers</td>
<td></td>
</tr>
</tbody>
</table>

http://micro.magnet.fsu.edu
# Contrast-Enhancing Techniques for Optical Microscopy

<table>
<thead>
<tr>
<th>Specular (Reflecting) Surface</th>
<th>Brightfield Illumination</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thin Films, Mirrors</td>
<td>Phase Contrast, DIC</td>
</tr>
<tr>
<td>Polished Metallurgical Samples</td>
<td>Darkfield Illumination</td>
</tr>
<tr>
<td>Integrated Circuits</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Diffuse (Non-Reflecting) Surface</th>
<th>Brightfield Illumination</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thin and Thick Films</td>
<td>Phase Contrast, DIC</td>
</tr>
<tr>
<td>Rocks and Minerals</td>
<td>Darkfield Illumination</td>
</tr>
<tr>
<td>Hairs, Fibers, and Bone</td>
<td></td>
</tr>
<tr>
<td>Insects</td>
<td></td>
</tr>
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</table>

<table>
<thead>
<tr>
<th>Amplitude Surface Features</th>
<th>Brightfield Illumination</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dyed Fibers</td>
<td></td>
</tr>
<tr>
<td>Diffuse Metallic Specimens</td>
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</tr>
<tr>
<td>Composite Materials</td>
<td></td>
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<tr>
<td>Polymers</td>
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<table>
<thead>
<tr>
<th>Birefringent Specimens</th>
<th>Polarized Illumination</th>
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</thead>
<tbody>
<tr>
<td>Mineral Thin Sections</td>
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</tr>
<tr>
<td>Hairs and Fibers</td>
<td></td>
</tr>
<tr>
<td>Bones and Feathers</td>
<td></td>
</tr>
<tr>
<td>Single Crystals</td>
<td></td>
</tr>
<tr>
<td>Oriented Films</td>
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</table>

<table>
<thead>
<tr>
<th>Fluorescent Specimens</th>
<th>Fluorescence Illumination</th>
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</thead>
<tbody>
<tr>
<td>Mounted Cells</td>
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</tr>
<tr>
<td>Fluorochrome-Stained Sections</td>
<td></td>
</tr>
<tr>
<td>Smears and Spreads</td>
<td></td>
</tr>
</tbody>
</table>
Resolution

Based on the Airy discs formation Abbé derived a theoretical limit to an optical instrument resolution.

![Airy Discs](image)

He defined the resolving power of an optical instrument as the distance between two point sources for which the center of one Airy disk coincides with the first zero of the other:

\[ d \approx \frac{0.61 \lambda}{NA} \]  Rayleigh criterion (light emitting particle)

\[ d \approx \frac{\lambda}{NA_{col} + NA_{obj}} \approx \frac{\lambda}{2 \times NA} \]  Abbé criterion (illuminated particle)
Optical microscopy

\[ d \approx \frac{0.61 \lambda}{n \sin \mu} \]

Rayleigh criterion (light emitting particle)

\[ d \approx \frac{\lambda}{2 n \sin \mu} \]

Abbé criterion (illuminated particle)
Confocal microscopy

- In confocal microscopy, due to the constrained light path provides an increased contrast, allowing for resolving objects with intensity differences of up to 200:1.
- The in plane resolution, in confocal microscopy, is also slightly increased (1.5 times) while the resolution along the optical axis is high.
- These improvements are obtained at the expense of the utilization of mechanisms for scanning either by moving a specimen or by readjustment of an optical system. Scanning application allows to increase field of view as compared with conventional microscopes.
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%.

\[ r_{\text{resel}} = 0.61 \frac{\lambda}{n \sin \theta} = 1.22 \frac{\lambda'}{D} F \]

\[ r_{\text{conf}} = 0.44 \frac{\lambda}{n \sin \theta} = 0.88 \frac{\lambda'}{D} F \]
LASER SCANNING CONFOCAL MICROSCOPY

Nathan S. Claxton, Thomas J. Fellers, and Michael W. Davidson

Department of Optical Microscopy and Digital Imaging, National High Magnetic Field Laboratory,
The Florida State University, Tallahassee, Florida 32310
Confocal microscopy

Anna-Katerina Hadjantonakis; Virginia E Papaioannou
BMC Biotechnology 2004, 4:33
Confocal microscopy

Strengths:
- Optical sectioning (0.5 µm).
  - three-dimensional images.
- Improved contrast (200:1).
- Better resolution (1.5x).
- Field of view defined by the scanning range.

Limitations:
- Image is scanned, resulting in slower data acquisition.
- High intensity laser radiation can damage some samples.
- Cost (typically 10x more than a comparable wide-field system).
Spatial Resolution of Biological Imaging Techniques

- Fluorescence Microscopy
  - Superresolution
    - 4Pi and i^5M
    - High Resolution Structured Illumination
    - Ground State Depletion (GSD)
    - Saturated Structured Illumination (SSIM)
    - Stimulated Emission Depletion (STED)
    - PALM, FPALM and STORM
    - Near-Field (NSOM)
  - Optical Coherence Tomography
  - Widefield and TIRF Microscopy
  - Confocal Microscopy
  - PET
  - MRI and Ultrasound

- Electron Microscopy
Nanoscopy

(a) Single-photon excitation

Laser illumination everywhere
Excitation everywhere

(b) Multi-photon excitation

Laser illumination everywhere
Excitation only at highest photon flux


(photographed by W.B. Amos and M.Cipollone in the LMB, Cambridge)

Microscope Configuration and Point-Spread Function of Opposed Objective Systems

Figure 3

(a) 4Pi and I^5M Microscope Optical Train Configuration

(b) i^5M TPE PSF

(c) 4Pi TPE PSF
Nanoscopy

The Concept of Superresolution with STED Microscopy

(a) Phase Plate  
(b) Excitation + STED Pattern = Effective PSF  
(c) Widefield Image  
(d) STED Image

Figure 6
• Sample is illuminated with a patterned light source.
• The pattern interact with structures finer than the diffraction limit;
• The pattern projection itself is diffraction limited.
• The big advantage of HR-SIM is again its ease of-use.
• Uses common
• Fixation and labeling procedures are similar to normal fluorescence microscopy,
• Dye labeling must be stable enough to survive the acquisition of multiple images.
PALM (photo-activated localization microscopy)

Widefield | PALM | Widefield | PALM

Other similar techniques:
- **STORM** (Stochastic optical reconstruction microscopy)
- **FIONA** (Fluorescence Imaging with One Nanometer Accuracy)
- **SHReC** (Single Molecule High Resolution Colocalization)
- **SPDM** (Spectral Precision Distance Microscopy)

And many variants.
Nanoscopy

<table>
<thead>
<tr>
<th>Method</th>
<th>CLSM</th>
<th>STED</th>
<th>CW-STED</th>
<th>3D-SIM</th>
<th>PALM/STORM</th>
</tr>
</thead>
<tbody>
<tr>
<td>~λ_{em} [nm]</td>
<td>460-670</td>
<td>670</td>
<td>520</td>
<td>620</td>
<td>460</td>
</tr>
<tr>
<td>~D_{x,y} [nm]</td>
<td>180-250</td>
<td>60</td>
<td>70</td>
<td>130</td>
<td>100</td>
</tr>
<tr>
<td>~D_{z} [nm]</td>
<td>500-700</td>
<td>700</td>
<td>560</td>
<td>340</td>
<td>250</td>
</tr>
<tr>
<td>~V_{x,y,z} [10⁻³ μm³]</td>
<td>10-23</td>
<td>1.3</td>
<td>1.5</td>
<td>3.0</td>
<td>1.3</td>
</tr>
</tbody>
</table>

Nanoscopy

For the development of super-resolved fluorescence microscopy

2014

Eric Betzig
Stephan W. Hell
William E. Moerner

For the development of super-resolved fluorescence microscopy

Biophys. J. 105 2013

www.janelia.org/lab/betzig-lab

web.stanford.edu/group/moerner
Near-field microscopy: resolution beyond the diffraction limit!

The near field microscope resolves objects much smaller than the wavelength of light by measuring at very short distances from the sample surface, through sub-wavelength apertures.

Basic principle:

The principle of operation of the NSOM was devised by Synge in 1928 †.

- The sample is kept in the near-field regime of a sub-wavelength source.
- If $z \ll \lambda$, the resolution is determined by the aperture, not wavelength.
- The image is constructed by scanning the aperture across the sample and recording the optical response.

† Philos. Mag. 6, 356 (1928).
Near-field scanning optical microscopy (NSOM)

Instrumentation:
**Near-field scanning optical microscopy (NSOM)**

**Strengths:**
- Powerful, very attractive.
- No sample preparation.
- Non destructive technique.
- Sub diffraction limit resolution (50 nm).
Requirements and limitations:
- Careful alignment required.
- Very low throughput.
- Slow data acquisition.
- Limited to surface features of fairly flat samples (20 μm).
- Interaction between tip and sample may make analysis difficult.

Complementary techniques:
AFM, SEM, TEM, Confocal microscopy.
Spectroscopy at the tip

Structural analysis and mapping of individual protein complexes by infrared nanospectroscopy

Spectroscopy at the tip

Gate-tuning of graphene plasmons revealed by infrared nano-imaging

Spectroscopy at the tip

Resonance enhanced AFM-IR: A new powerful way to characterize blooming on polymers used in medical devices

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Princeton Instruments

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It is Important to use complementary techniques!

Thank you!