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Differential reflectance spectroscopy on molecular layers: experimental aspects

Laurent Nony

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Institut **M**atériaux **M**icroélectronique **N**anosciences **P**rovence

Spectroscopie de réflectivité différentielle sur des couches moléculaires: aspects expérimentaux

Laurent Nony

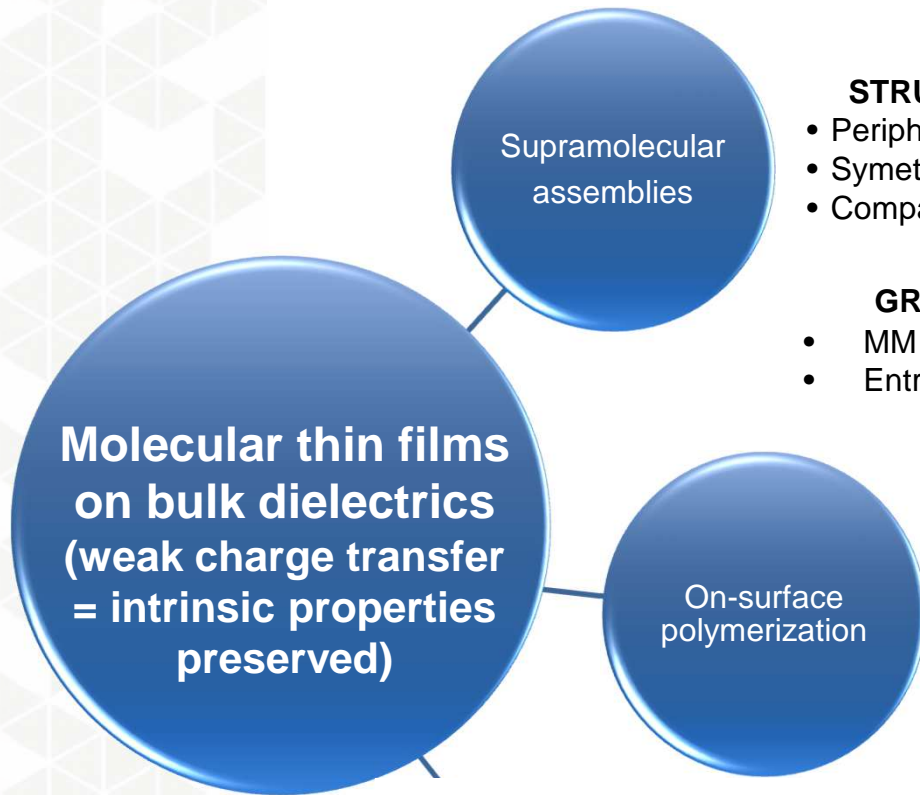
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Ecole thématique « Couplage entre les techniques de microscopies à sondes locales et l'optique »
18-19 Mars 2019, Carry le Rouet



Research topics of the Nanostructuration group

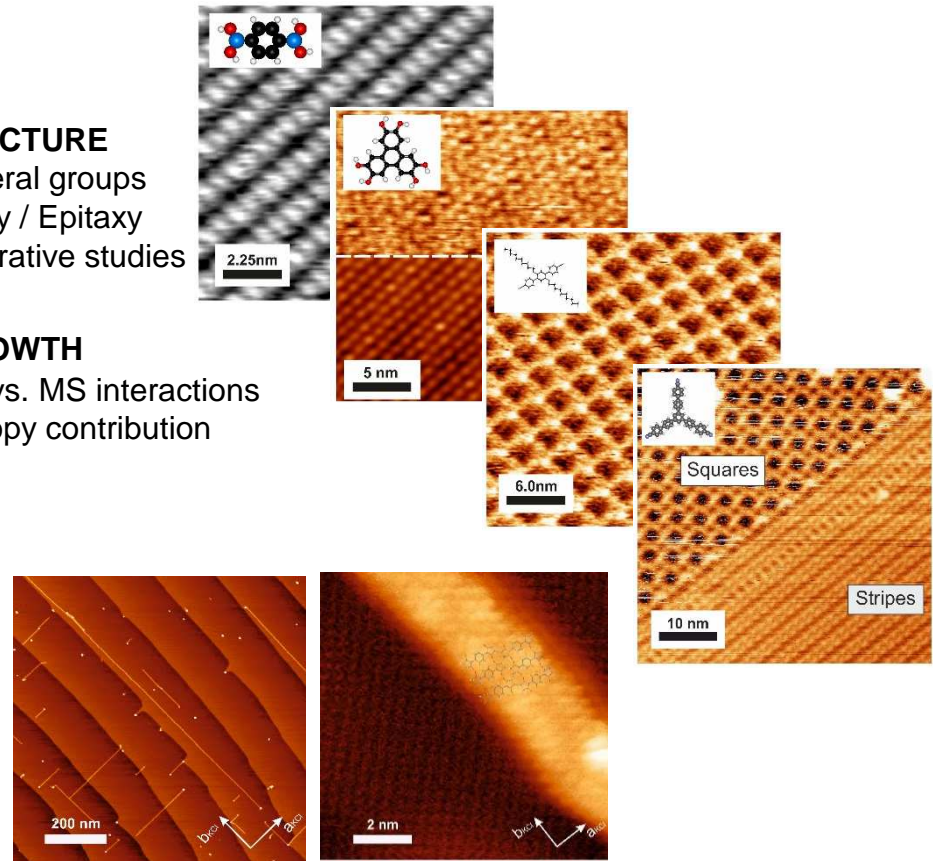


STRUCTURE

- Peripheral groups
- Symetry / Epitaxy
- Comparative studies

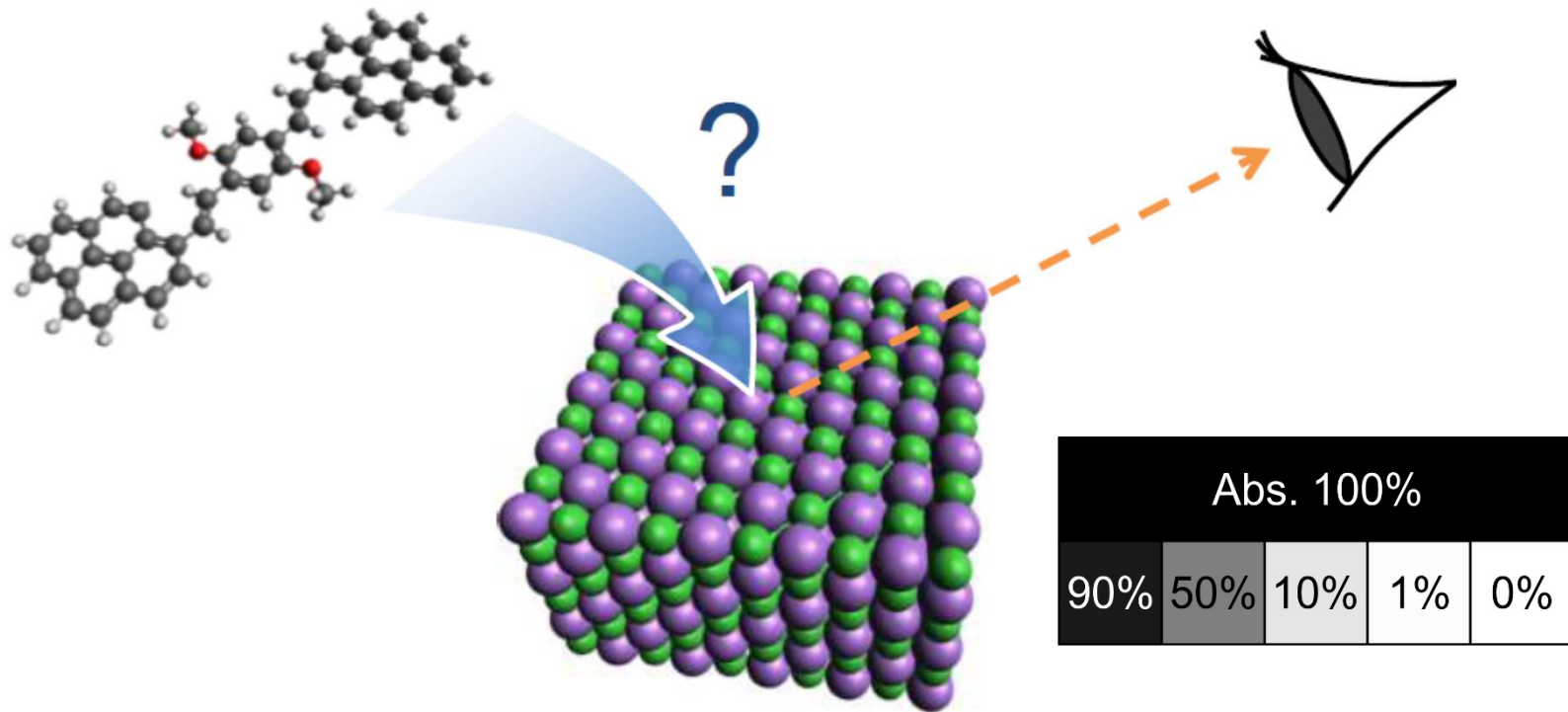
GROWTH

- MM vs. MS interactions
- Entropy contribution



Towards optical properties

Motivation: completing structural measurements with **optical spectroscopy**
structure ↔ **property**



Differential Reflectance Spectroscopy (DRS):

- *in situ* (UHV) & real time UV-vis absorption spectra
- sensitivity ~ 0.1% (~ sub-ML)

Outline

- I. Reminder, context of DRS on molecular films
 - II. Tutorial video: experimental aspects of DRS on molecular films
 - III. Connection to the structural properties of the molecular films by nc-AFM
 - IV. DRS fonctionnal for fitting the DR spectra
 - V. Conclusion
- List of publications*

Outline

I. Reminder, context of DRS on molecular films

II. Tutorial video: experimental aspects of DRS on molecular films

III. Connection to the structural properties of the molecular films by nc-AFM

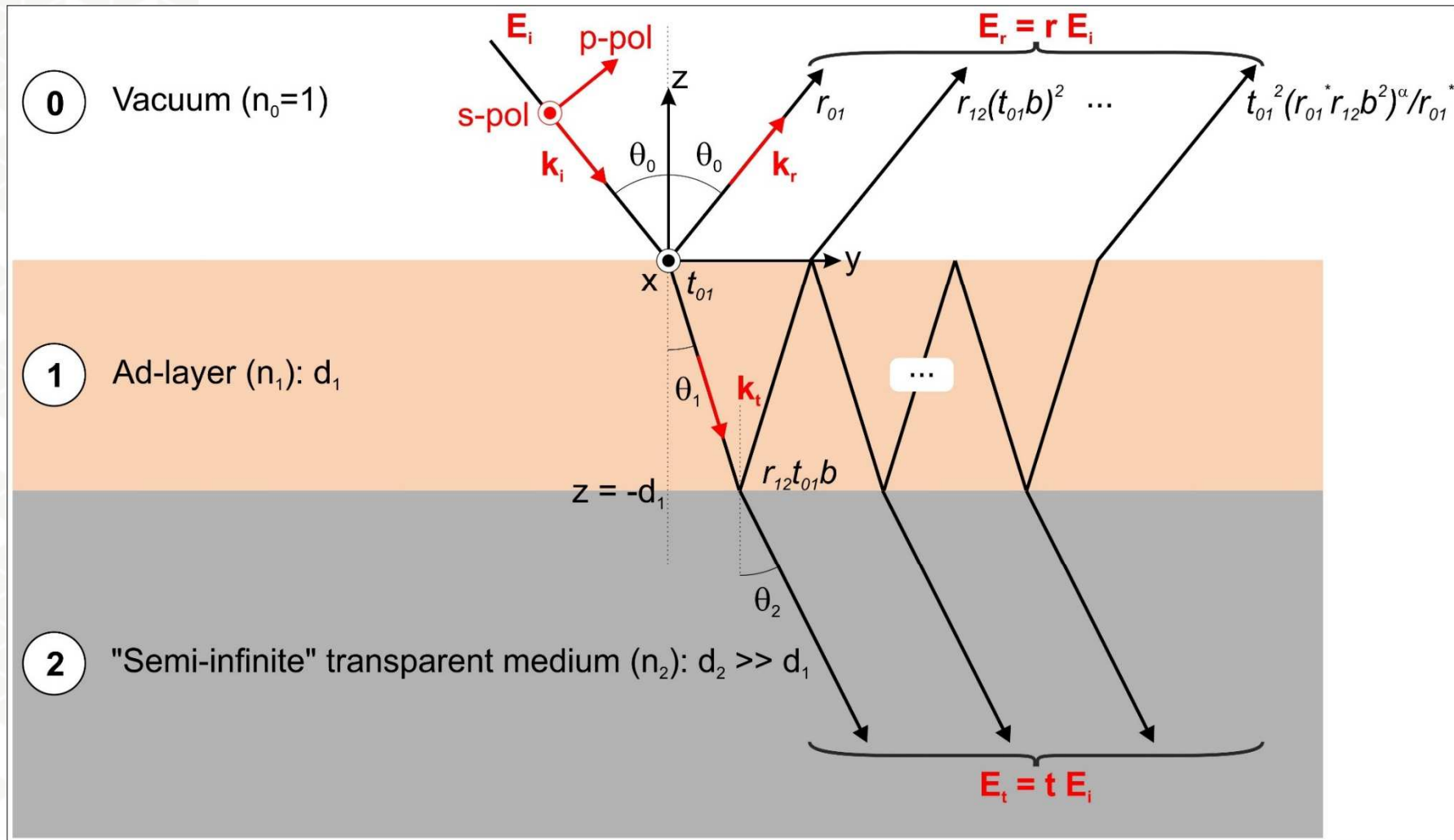
IV. DRS fonctionnal for fitting the DR spectra

V. Conclusion

List of publications

Reminder, context of DRS

- Differential Reflectance Spectroscopy:



Reminder, context of DRS

- Differential Reflectance Spectroscopy:
 - The combination between both Snell law, Fresnel equations and a linearization procedure¹ ($d_1 \ll \lambda$) yields to the so-called **Mc Intyre's approach** to DRS:

$$\text{DRS}^{(s)} = \frac{R(d_1) - R(0)}{R(0)} = \frac{|\tilde{r}(d_1)|^2 - |\tilde{r}(0)|^2}{|\tilde{r}(0)|^2} = \frac{8\pi d_1 \cos \theta_0}{\lambda(n_2^2 - 1)} \text{Im}\{\tilde{n}_1^2\}$$

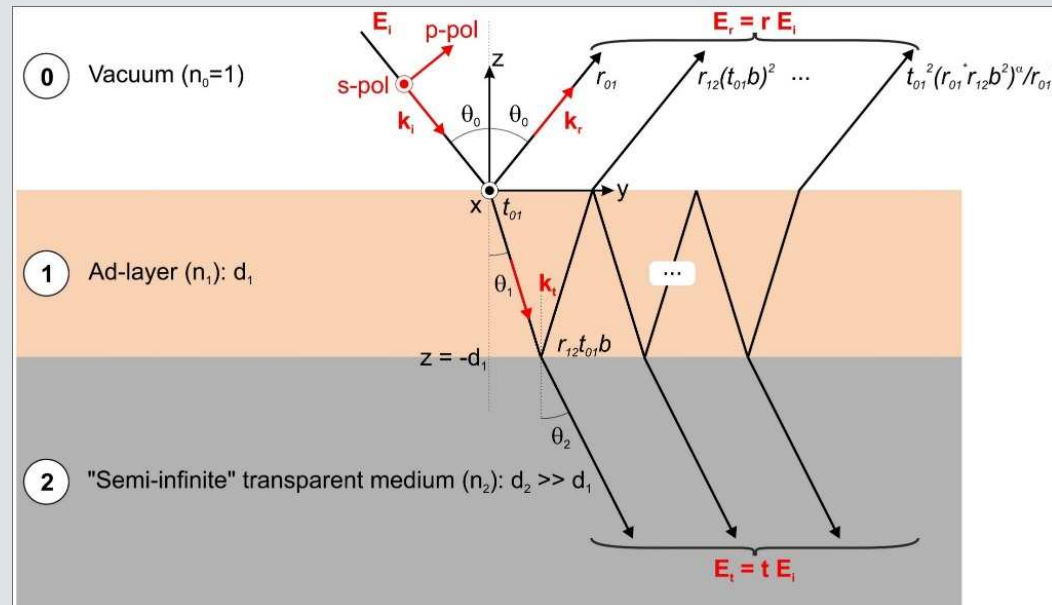
- Relates to the **optical absorption of the molecular film**
- **Goal: quantitative determination of the molecular film's dielectric function $\epsilon(\lambda)$** (or its refractive index: $n(\lambda) = \sqrt{\epsilon(\lambda)}$)

¹J.D.E.McIntyre et al., Surf.Sci. 24, 417 (1971)

Reminder, context of DRS

- Franck Bocquet's lecture on « Theoretical aspects of DRS on molecular films »:

Dans le principe: c'est « simple », car on analyse un multicouche optique!



Equations de passage aux interfaces → Relations de Fresnel → Intensité spéculaire:

- Plusieurs interfaces: *approche matricielle des milieux stratifiés*
- Couches anisotropes: *approche tensorielle, complexe voire très complexe!*
- Rugosités interfaciales : complexe mais faisable (*modèle Gaussien, milieu effectif...*)

NECESSITE D'INJECTER L'INDICE $n(\lambda)$ DU FILM : MAIS en général $n(\lambda)$ est INCONNU

Reminder, context of DRS

- Franck Bocquet's lecture on « Theoretical aspects of DRS on molecular films »:

Calcul de la DRS et fit de $n(\lambda)$ simultanément

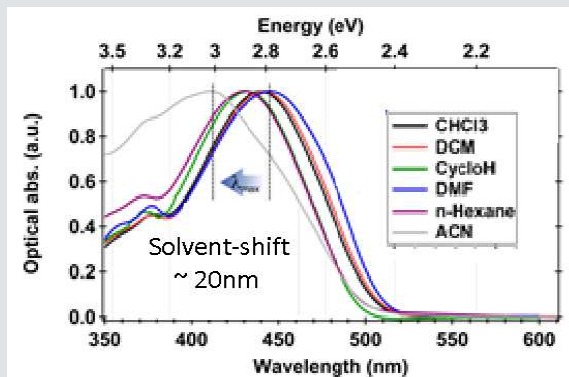
Intuiter correctement le $n(\lambda)$ d'input du fit:

- *Dépendance en énergie*
- *Partie réelle et imaginaire*
- *Nombre de pics et écartement*
- *Rapport d'intensité des pics*

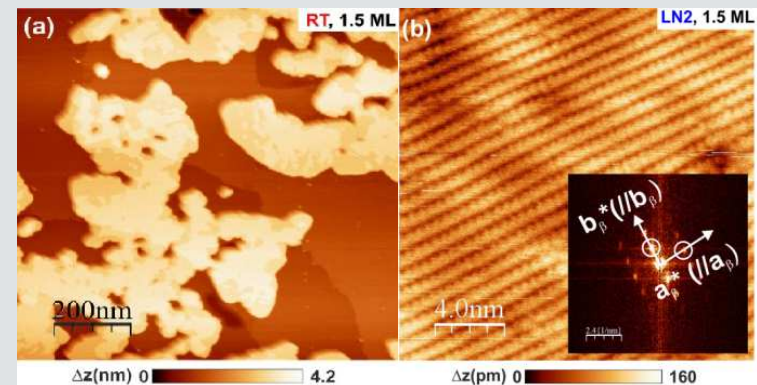
Caractérisation exhaustive du dépôt:

- *Épaisseur, homogénéité*
- *Rugosité*
- *Nombre de couches*
- *Orientation cristalllographique*

Etude préalable de la $DO(\lambda)$

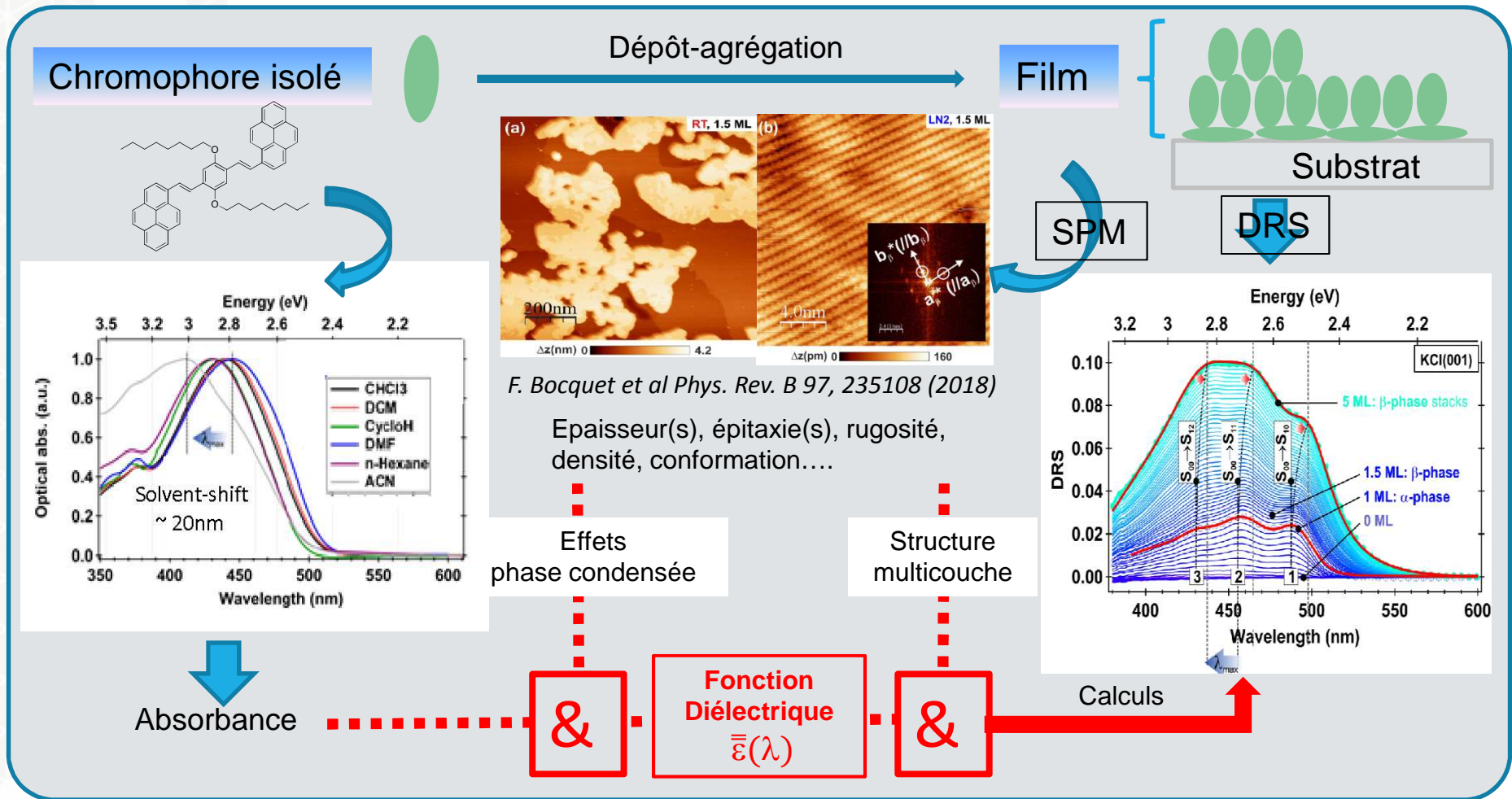


Analyse SPM « poussée »



Reminder, context of DRS

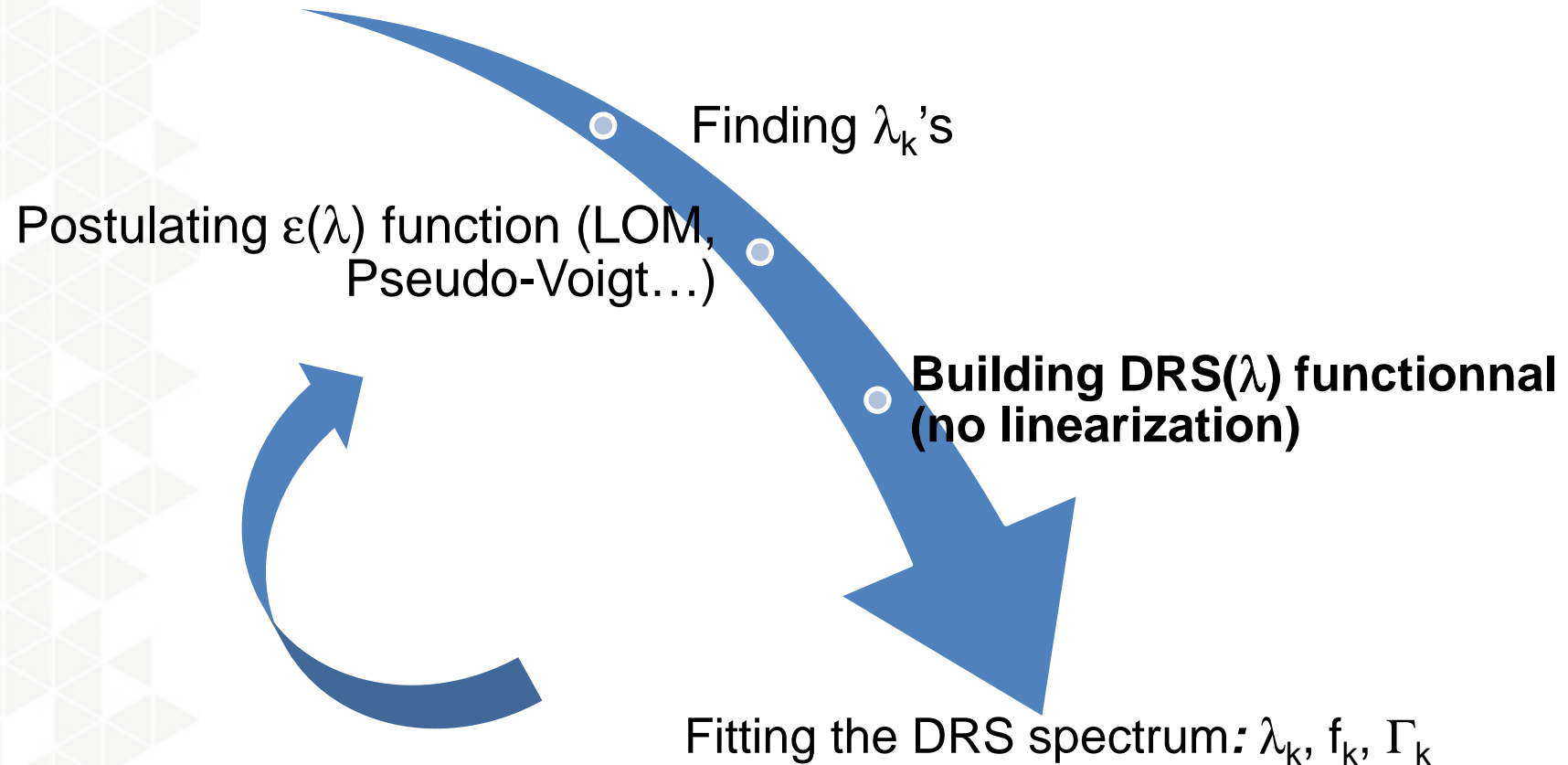
- Franck Bocquet's lecture on « Theoretical aspects of DRS on molecular films »:



Reminder, context of DRS

11

- Ultimate methodology to extract the dielectric function :
DRS spectrum



Outline

I. Reminder, context of DRS on molecular films

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Tutorial video: experimental aspects of DRS

- **Part 1:** UHV setup for DRS and non-contact AFM
- **Part 2:** Preparing and introducing molecules
- **Part 3:** Preparing and introducing samples
- **Part 4:** Calibrating the molecular flux
- **Part 5:** Annealing the sample
- **Part 6:** DRS setup
- **Part 7:** Checking the optical alignment and the stability of the detection
- **Part 8:** DRS acquisition
- **Part 9:** Post-processing
- **Part 10:** Data analysis...

Tutorial video: experimental aspects of DRS

- The link to the video:

<https://amubox.univ-amu.fr/s/4YRR2StjtGoaPGm>

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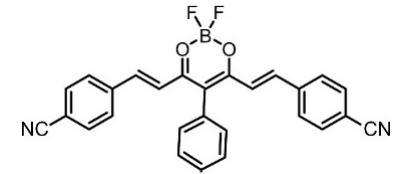
IV. DRS fonctionnal for fitting the DR spectra

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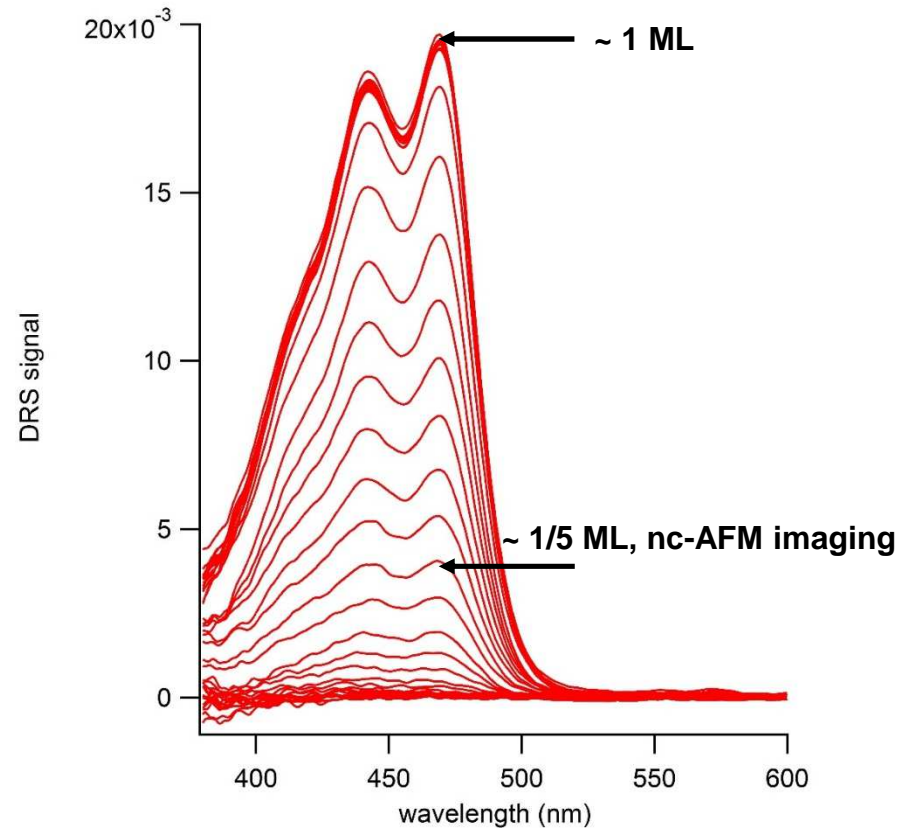
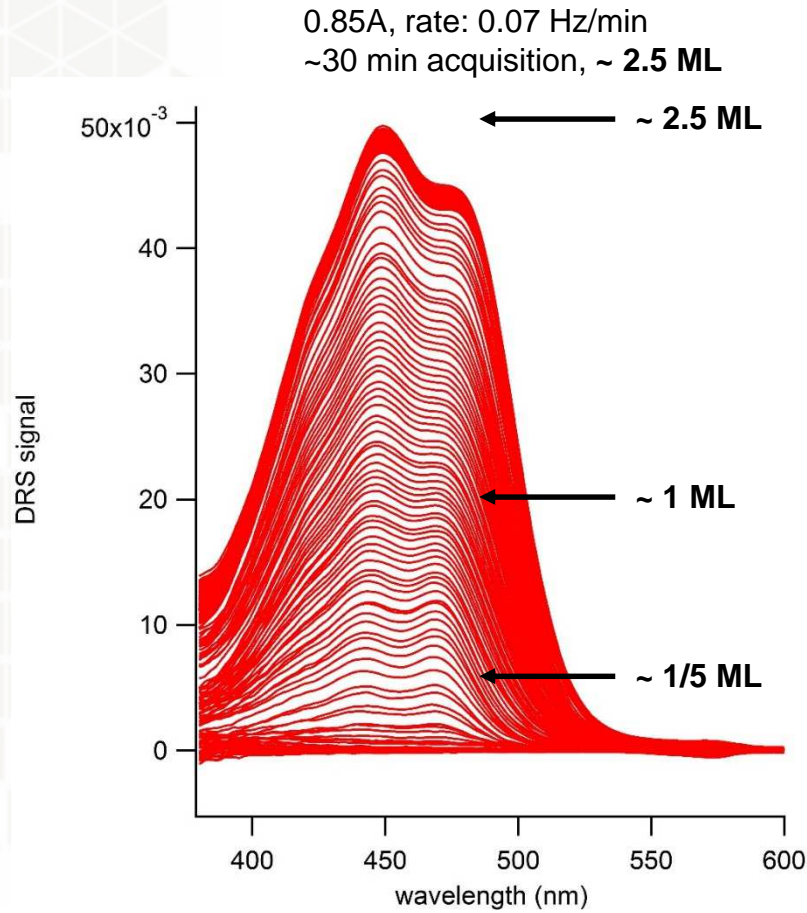
Connection to the structural properties of the molecular films by nc-AFM

- Exemplary case : curcuminoid chromophores



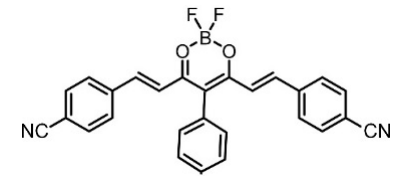
I: DRS in the multi-layer regime

III: DRS in the sub-ML regime

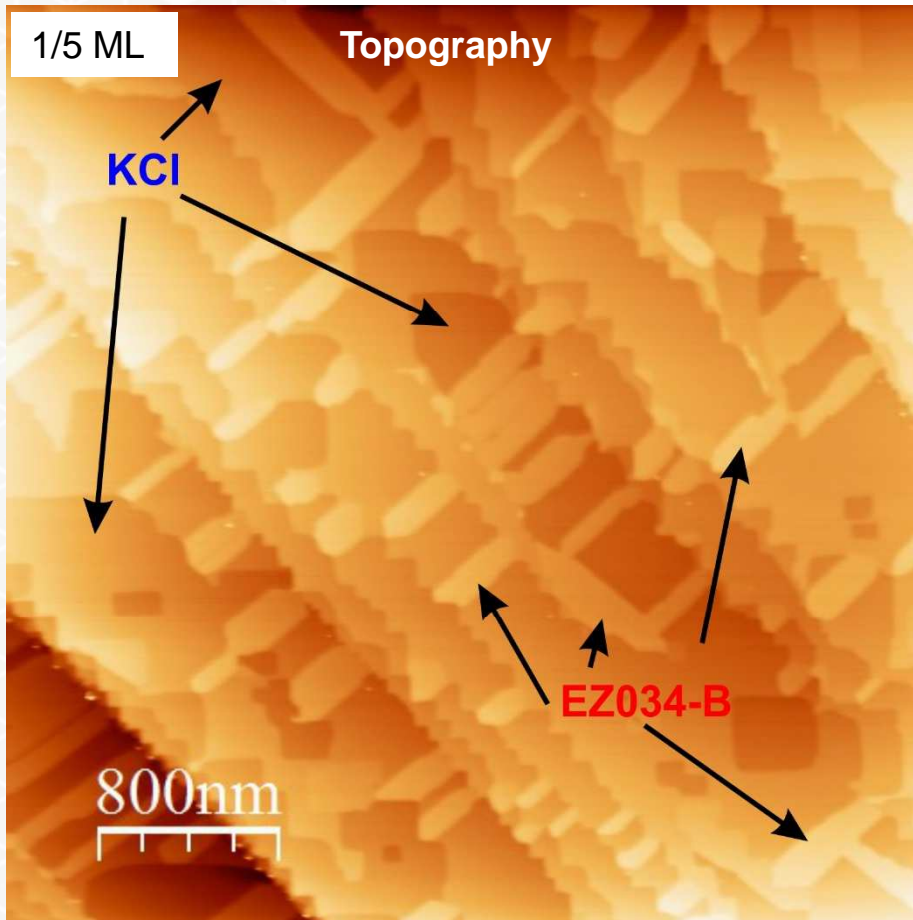


Connection to the structural properties of the molecular films by nc-AFM

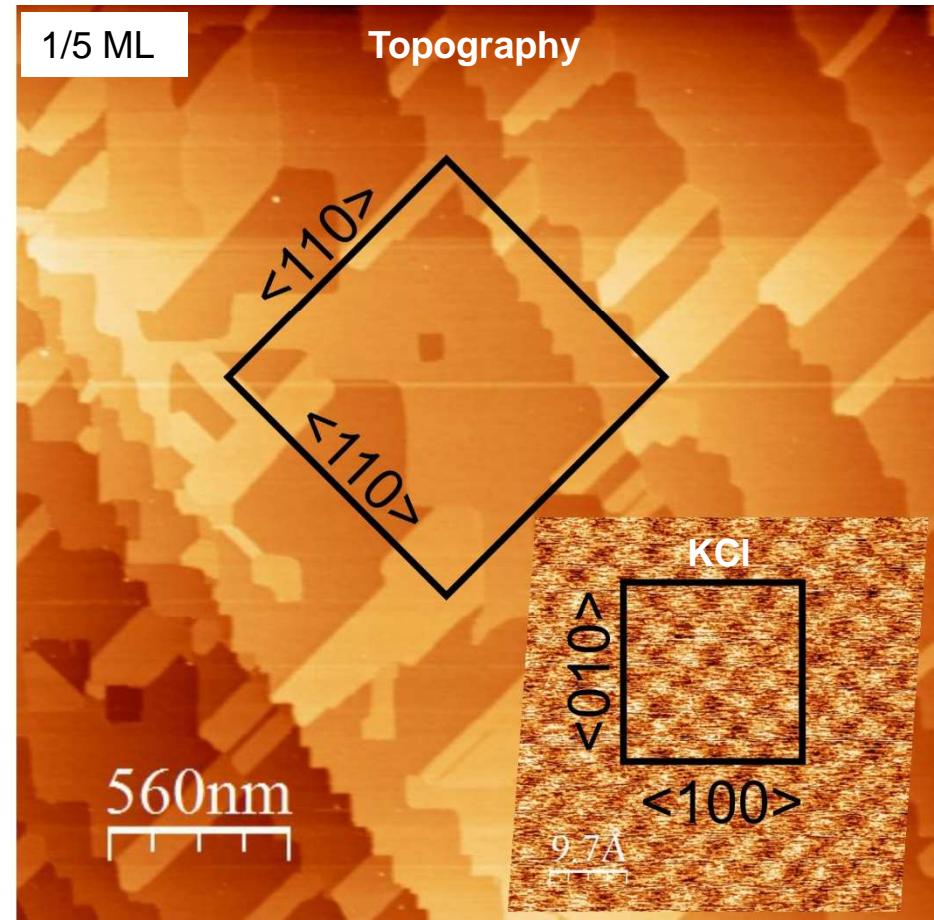
- Exemplary case : curcuminoid chromophores



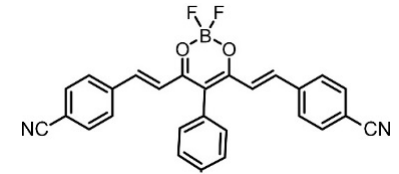
Large crystallites of equivalent domains



Orientations of ML @ +/-45° w.r.t. <100> direction



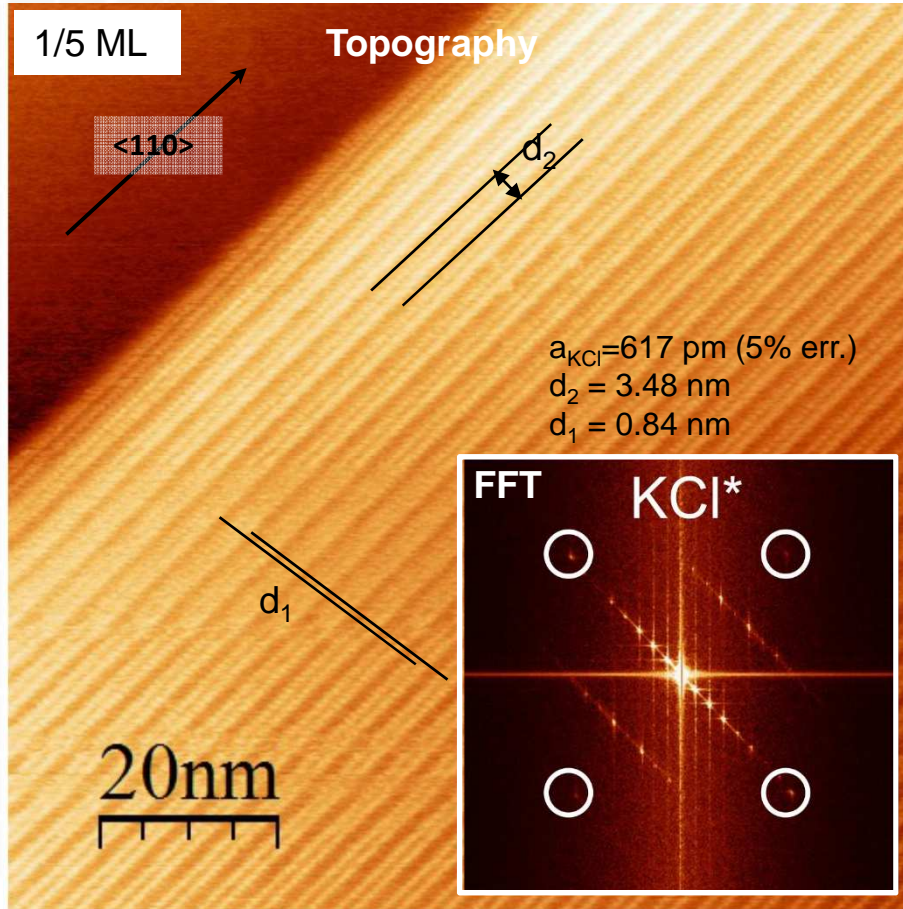
Connection to the structural properties of the molecular films by nc-AFM



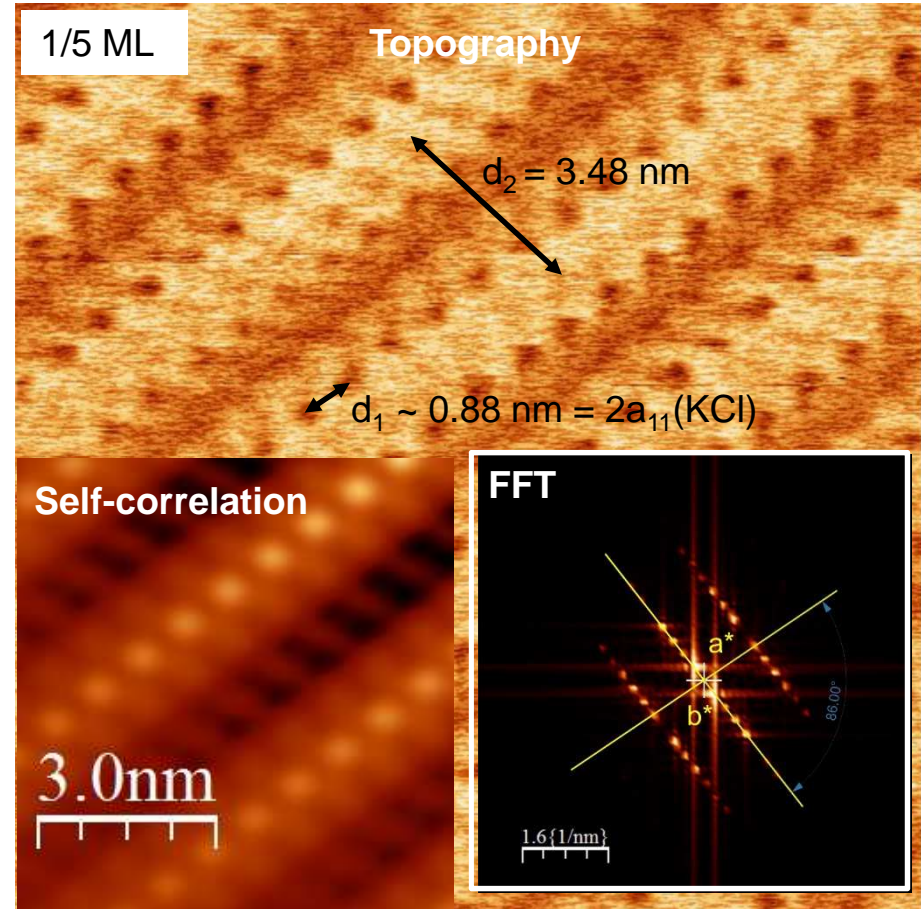
- Exemplary case : curcuminoid chromophores

Zoom on a domain:

- Molecular rows match substrate's <110> polar lines
- Apparent height : 4.0 Å
- Atomic resolution on KCl (traces in FFT)

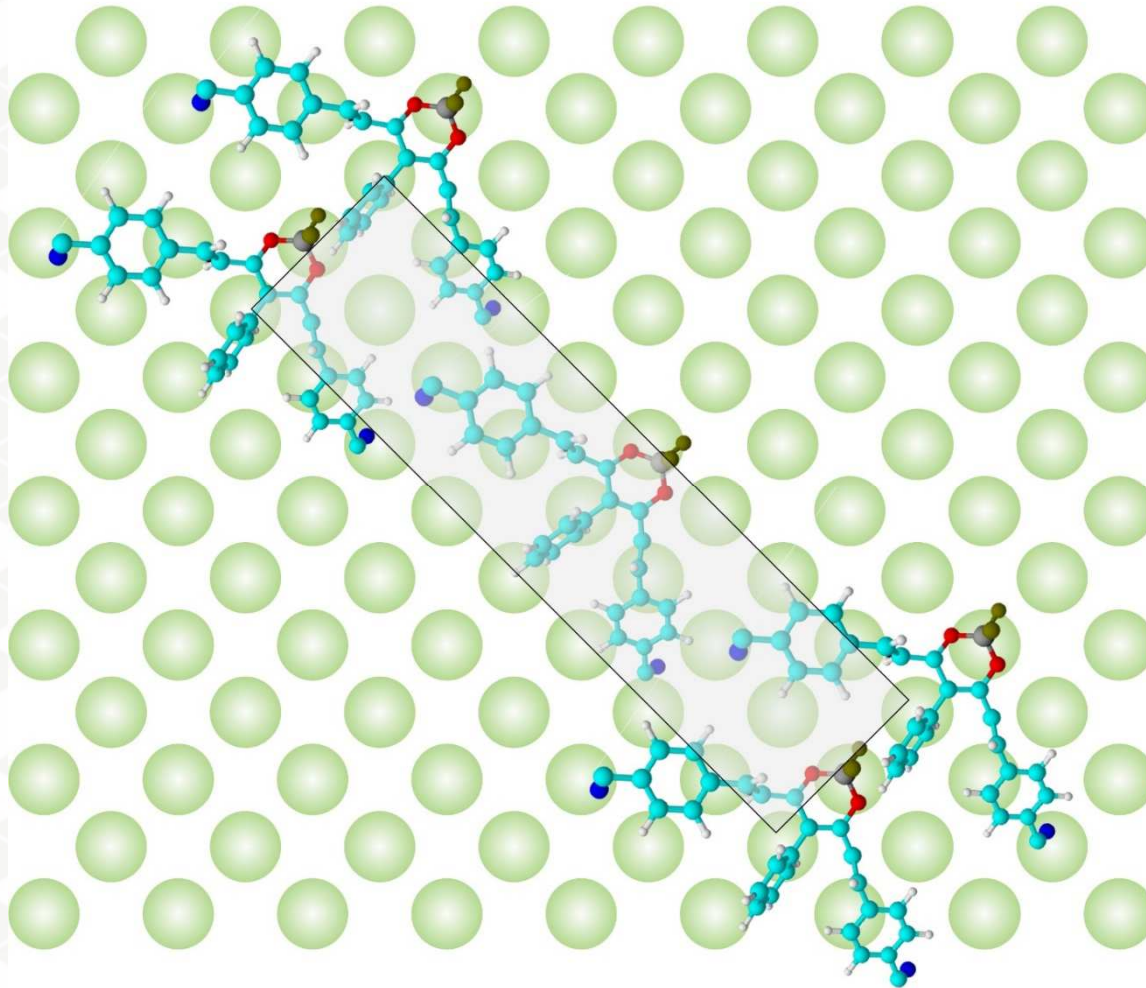
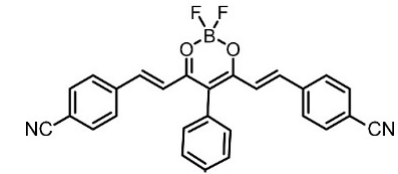


High-resolution imaging



Connection to the structural properties of the molecular films by nc-AFM

- Exemplary case : curcuminoid chromophores



- Epitaxy
- Density
- Molecular conformation
- Dipole transition orientation
- ...



Inputs for the $\epsilon(\lambda)$ function

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DRS fonctionnal for fitting the DR spectra

- Beyond Mc Intyre's approach...
 - Back to optics basics...

$$\text{DRS} = \frac{R(d_1) - R(0)}{R(0)} = \frac{|\tilde{r}(d_1)|^2 - |\tilde{r}(0)|^2}{|\tilde{r}(0)|^2} \quad (\text{Equ.0})$$

DRS functional for fitting the DR spectra

- Multi-layers = Stratified medium:

- **Uniaxial material approximation**

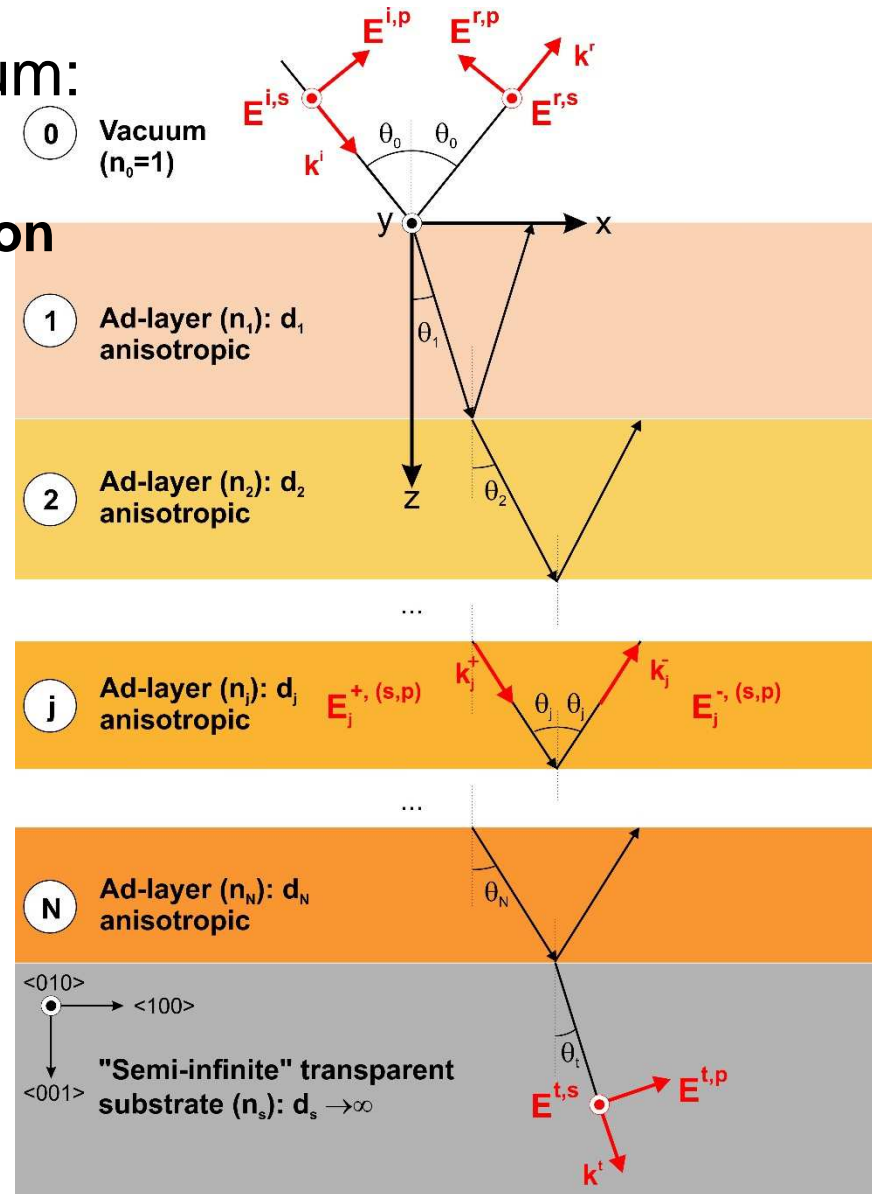
(x,y,z geom. accordingly with cristallographic axes)

- z axis = optic axis

- Dielectric tensor of the jth layer:

(Equ.1)
$$\bar{\bar{\epsilon}}_j = \begin{pmatrix} \tilde{\epsilon}_j^\perp(\lambda) & 0 & 0 \\ 0 & \tilde{\epsilon}_j^\perp(\lambda) & 0 \\ 0 & 0 & \tilde{\epsilon}_j^\parallel(\lambda) \end{pmatrix}$$

Hence, $\bar{n}_j = \Re\{\bar{n}_j\} + i\Im\{\bar{n}_j\} = \sqrt{\bar{\epsilon}_j}$



DRS functional for fitting the DR spectra

- Multi-layers = Stratified medium:

- Electric field may be split into **s** (y axis) and **p** (x,z axes) polarizations:

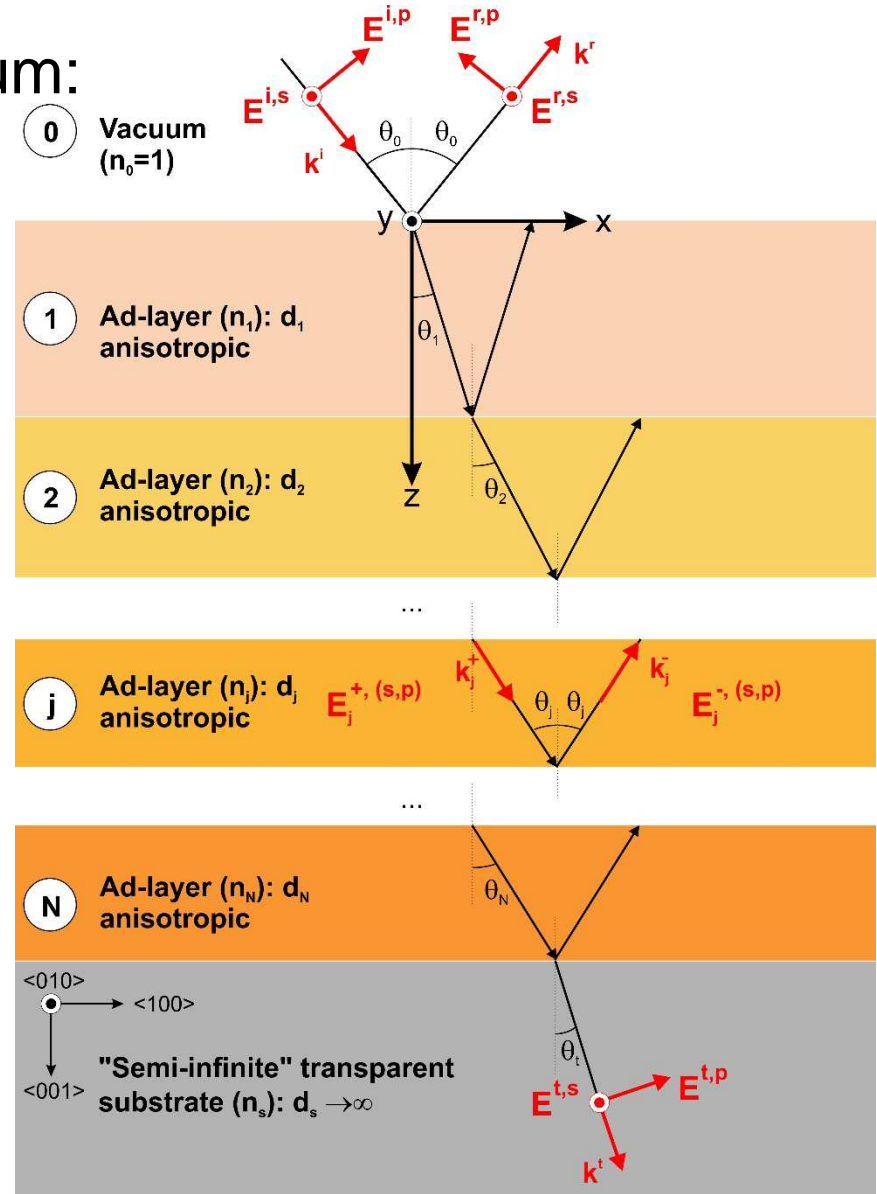
$$\vec{E}^\dagger = \vec{E}^{\dagger,s} + \vec{E}^{\dagger,p},$$

(with $\dagger = i, r, \text{ or } t$)

- Thus:

$$\left\{ \begin{aligned} \vec{E}^{\dagger,s} &= E^{\dagger,y} \mathbf{e}_y e^{i[\omega t - k^{\dagger,x}x - k^{\dagger,z}z]} \\ \vec{E}^{\dagger,p} &= \{E^{\dagger,x} \mathbf{e}_x + E^{\dagger,z} \mathbf{e}_z\} e^{i[\omega t - k^{\dagger,x}x - k^{\dagger,z}z]} \end{aligned} \right.$$

with $\mathbf{k}^\dagger = k^{\dagger,x} \mathbf{e}_x + k^{\dagger,y} \mathbf{e}_y + k^{\dagger,z} \mathbf{e}_z$



DRS fonctionnal for fitting the DR spectra

- Multi-layers = Stratified medium:

- Reflection coefficient:

$$r^{(s,p)}(d) = \frac{\| \mathbf{E}^{r,(s,p)}(d) \|}{\| \mathbf{E}^{i,(s,p)} \|}$$

- Reflectance components:

$$R^{(s,p)}(d) = |r^{(s,p)}(d)|^2$$

- DRS components:

$$\text{DRS}^{(s,p)}(d) = \frac{R^{(s,p)}(d) - R^{(s,p)}(0)}{R^{(s,p)}(0)}$$

- Unpolarized DRS (**physical observable!**): $R^u(d) = [R^s(d) + R^p(d)]/2$

$$\text{DRS}^u(d) = \frac{R^u(d) - R^u(0)}{R^u(0)} \quad (\text{Equ.2})$$

DRS functional for fitting the DR spectra

- Transfer matrix method (TMM)¹:
 - Wave vector for a jth layer

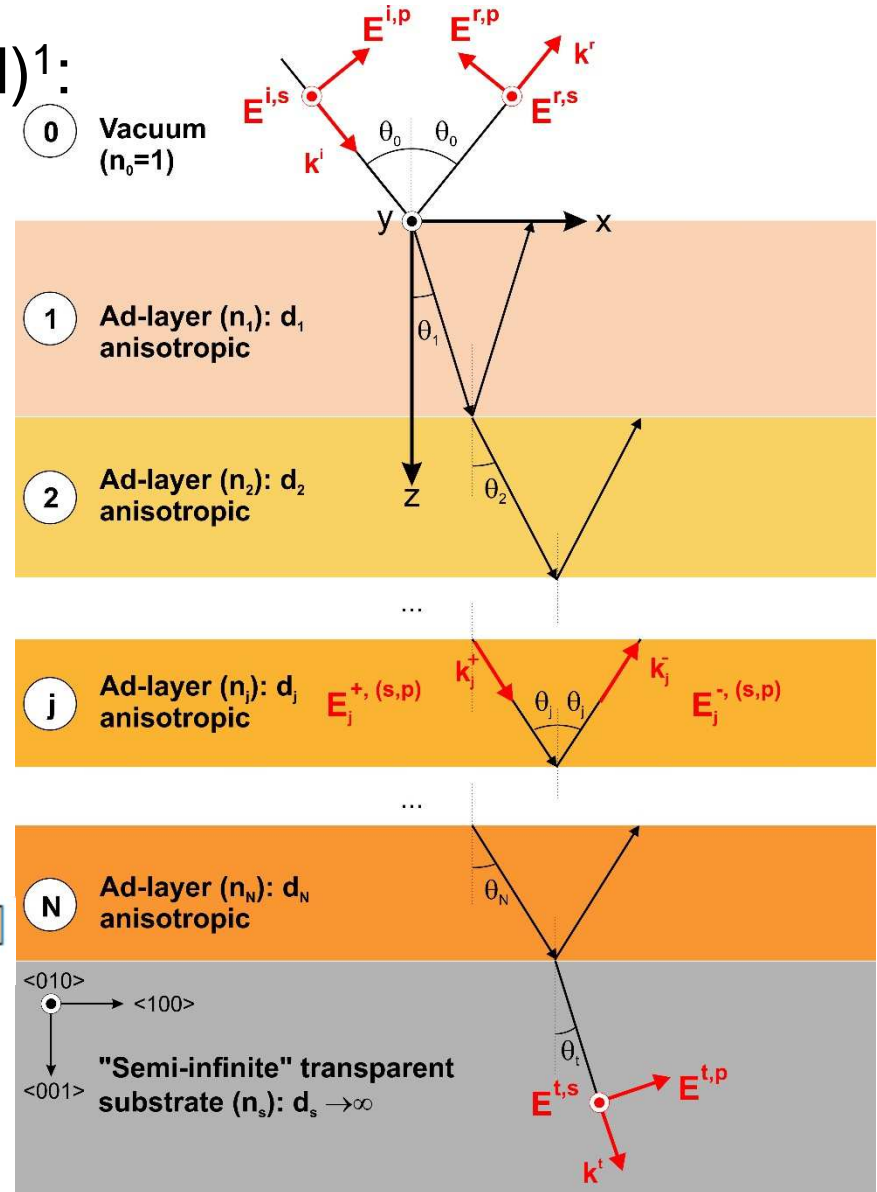
$$\mathbf{k}_j^{(i,r)} = k_j^{(i,r),x} \mathbf{e}_x + k_j^{(+,-),z} \mathbf{e}_z$$

with (Snell's laws):

$$\begin{cases} k_j^{(i,r),x} = \frac{2\pi}{\lambda} n_0 \sin(\theta_0) \\ k_j^{(+,-),z} = \pm \frac{2\pi}{\lambda} \tilde{n}_j(\lambda) \cos(\theta_j) \end{cases}$$

For instance:

$$\mathbf{E}^{(+,-),s} = E^{(+,-),y} \mathbf{e}_y e^{i[\omega t - k^{(+,-),x} x - k^{(+,-),z} z]}$$



¹R. Azzam and N. Bashara, Ellipsometry and Polarized Light (North Holland, Amsterdam, 1977).

DRS fonctionnal for fitting the DR spectra

- Transfer matrix method (TMM)¹:
 - Boundary conditions (Maxwell equs.) at each interface yield in-plane components of **s** and **p** polarizations:

$$\begin{pmatrix} E^{i,(x,y)} \\ E^{r,(x,y)} \end{pmatrix} = \frac{1}{2} \prod_{j=1}^N T_j \begin{pmatrix} \left[1 + \frac{\alpha_s}{\alpha_N}\right] E^{t,(x,y)} \\ \left[1 - \frac{\alpha_s}{\alpha_N}\right] E^{t,(x,y)} \end{pmatrix}$$

with the transfer matrix:

$$T_j = \frac{1}{2} \begin{pmatrix} \left[1 + \frac{\alpha_j}{\alpha_{(j-1)}}\right] e^{i\beta_j} & \left[1 - \frac{\alpha_j}{\alpha_{(j-1)}}\right] e^{-i\beta_j} \\ \left[1 - \frac{\alpha_j}{\alpha_{(j-1)}}\right] e^{i\beta_j} & \left[1 + \frac{\alpha_j}{\alpha_{(j-1)}}\right] e^{-i\beta_j} \end{pmatrix}$$

Hence (removal of the transmitted component):

$$r^{(x,y)}(d) = \frac{|E^{r,(x,y)}(d)|}{|E^{i,(x,y)}|} \quad (\text{Equ.3})$$

p polarization

s polarization

¹R. Azzam and N. Bashara, Ellipsometry and Polarized Light (North Holland, Amsterdam, 1977).

DRS fonctionnal for fitting the DR spectra

- Transfer matrix method (TMM):

- α_j and β_j coefficients:

- **s polarization case:** $\mathbf{E}^{(+,-),s} = E^{(+,-),y} \mathbf{e}_y$ (phase omitted)

Perpendicular to the optic axis = *ordinary direction*

$$\left\{ \begin{array}{l} \alpha_j = \alpha_j^o = \tilde{n}_j^\perp \cos(\theta_j^o) \stackrel{\text{Snell-Descartes}}{=} \sqrt{\tilde{n}_j^{\perp 2} - n_0^2 \sin^2(\theta_0)} \\ \beta_j = \beta_j^o = \frac{2\pi d_j}{\lambda} \alpha_j^o. \end{array} \right. \quad (\text{Equ.4})$$

- **p polarization case:** $\mathbf{E}^{(+,-),p} = E^{(+,-),x} \mathbf{e}_x + E^{(+,-),z} \mathbf{e}_z$ (phase omitted)

Parallel to the optic axis = *extraordinary direction*

$$\left\{ \begin{array}{l} \alpha_j^e = \frac{\tilde{n}_j^\perp \tilde{n}_j^\parallel}{\sqrt{\tilde{n}_j^{\parallel 2} - n_0^2 \sin^2(\theta_0)}} \\ \beta_j^e = \frac{2\pi d_j}{\lambda} \frac{\tilde{n}_j^\perp}{\alpha_j^e}. \end{array} \right. \quad (\text{Equ.5})$$

DRS fonctionnal for fitting the DR spectra

- Summary: the combination between...

$$\left\{ \begin{array}{l} \alpha_j = \alpha_j^o = \tilde{n}_j^\perp \cos(\theta_j^o) \\ \beta_j = \beta_j^o = \frac{2\pi d_j}{\lambda} \alpha_j^o \end{array} \right. \quad \begin{array}{c} \text{Snell-Descartes} \\ \hat{=} \\ \sqrt{\tilde{n}_j^{\perp 2} - n_0^2 \sin^2(\theta_0)} \end{array} \quad \& \quad \left\{ \begin{array}{l} \alpha_j^e = \frac{\tilde{n}_j^\perp \tilde{n}_j^\parallel}{\sqrt{\tilde{n}_j^{\parallel 2} - n_0^2 \sin^2(\theta_0)}} \\ \beta_j^e = \frac{2\pi d_j \tilde{n}_j^\perp}{\lambda \alpha_j^e} \end{array} \right. \quad \text{(Equ.5)}$$

(Equ.4)

(Equ.3)

$$r^{(x,y)}(d) = \frac{|E^{r,(x,y)}(d)|}{|E^{i,(x,y)}|}$$

(Equ.1)

$$\bar{\epsilon}_j = \begin{pmatrix} \tilde{\epsilon}_j^\perp(\lambda) & 0 & 0 \\ 0 & \tilde{\epsilon}_j^\perp(\lambda) & 0 \\ 0 & 0 & \tilde{\epsilon}_j^\parallel(\lambda) \end{pmatrix}$$

And (Equ.2):

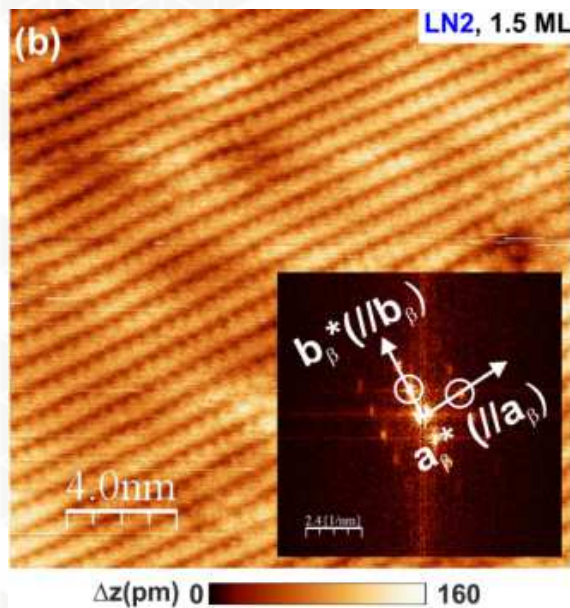
$$\text{DRS}^u(d) = \frac{R^u(d) - R^u(0)}{R^u(0)}$$

yields the DRS **fitting fonctionnal!**

DRS fonctionnal for fitting the DR spectra

- But $\epsilon(\lambda)$ is still to be known as an input of the DRS fonctionnal...
 - In case of a **homogeneous layer**: Lorentz Oscillator Model (cf. Franck Bocquet's lecture):

$$\tilde{\epsilon}_{\text{LOM}}(\lambda) = \epsilon^{\infty} + \sum_{j=1}^{\text{Number of transitions}} \frac{f_j}{1 - \left(\frac{\lambda_j}{\lambda}\right)^2 + i\gamma_j\left(\frac{\lambda_j}{\lambda}\right)}$$

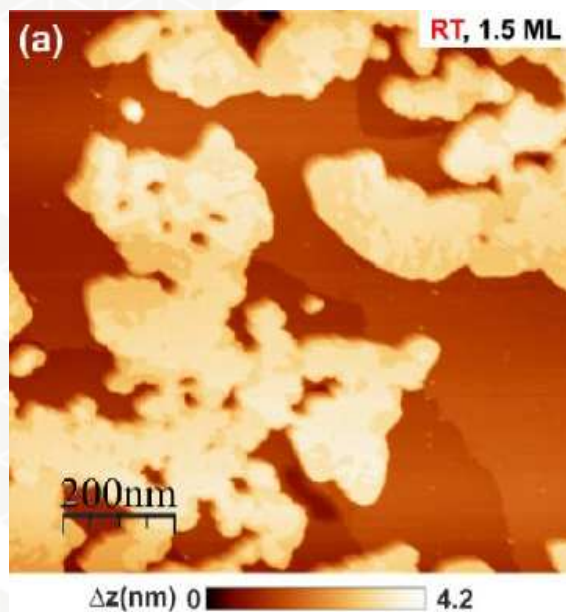


With:

- f_j = oscillator strength
- λ_j = absorption wavelength
- γ_j = peak width (relates to state lifetime)

DRS fonctionnal for fitting the DR spectra

- But $\epsilon(\lambda)$ is still to be known as an input of the DRS fonctionnal...
 - In case of an **inhomogeneous layer**: Effective Medium Theory¹ + Lorentz Oscillator Model:



$$\epsilon_{\text{EMT}}^{\perp, \parallel}(\lambda) = \epsilon_h(\lambda) \frac{\epsilon_h(\lambda) + [v(1-f) + f][\epsilon^{\perp, \parallel}(\lambda) - \epsilon_h(\lambda)]}{\epsilon_h(\lambda) + v(1-f)[\epsilon^{\perp, \parallel}(\lambda) - \epsilon_h(\lambda)]}$$

Dielectric function of the hosting material: vacuum

Form factor

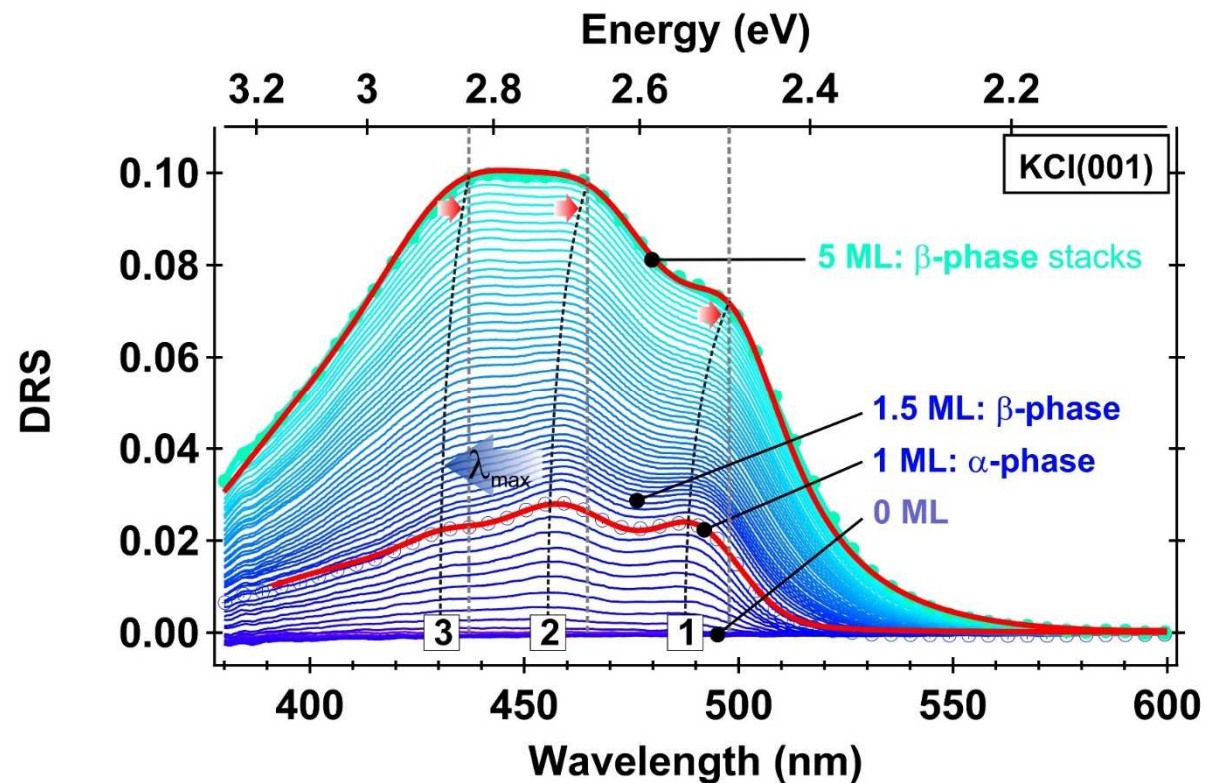
Filling parameter [0;1]

$$\tilde{\epsilon}_{\text{LOM}}(\lambda) = \epsilon^{\infty} + \sum_{j=1}^3 \frac{f_j}{1 - \left(\frac{\lambda_j}{\lambda}\right)^2 + i\gamma_j \left(\frac{\lambda_j}{\lambda}\right)}$$

¹B. J. C. Maxwell Garnet, Philos. Trans. R. Soc., A **203**, 385 (1904); V. Markel, J. Opt. Soc. Am. A **33**, 1244 (2016).

DRS fonctionnal for fitting the DR spectra

- **The fits** (Levenberg-Marquardt algorithm) are then performed on the experimental **DR spectra** with a Mathematica program and allow for the extraction of the dielectric function of the molecular layer, as well as an estimate of its thickness...



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List of publications

Conclusion

- I. DRS on organic layers in UHV is an actual spectroscopic method to measure the UV-vis absorption spectrum of the organic layers
- II. Its experimental implementation is fairly simple
- III. Upon stability achievement, the method is sensitive to a fraction of ML
- IV. The quantitative extraction of the dielectric function of the molecular layers is tedious and rely on many approaches (stratified medium, uniaxial material, LOM, EMT...), whose relevance is to be estimated at best, and based on the molecular structure of the layers, the optical density of the molecules in solution...
- V. The interpretation of DR spectra without further information is useless : this can only be performed in connection with other inputs originating from other characterization methods

Acknowledgments



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Nanosciences de Provence

Nanostructuration group members @ IM2NP:

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- Franck Para
- Sylvain Clair
- Luca Giovanelli
- Younal Ksari
- Jean-Marc Themlin



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- Laurence Masson
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- Elena Zaborova



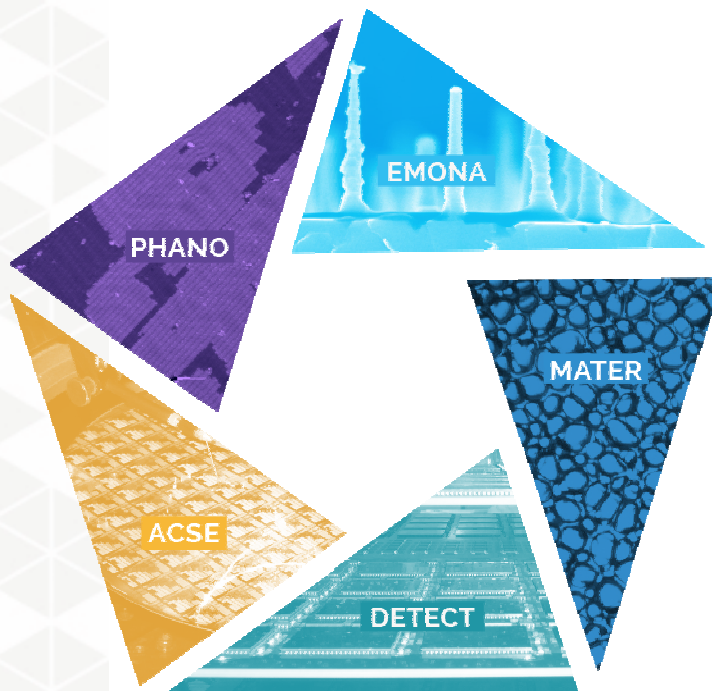
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Merci pour votre attention



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List of publications of the « non-contact AFM » team (2008)

- « *Analytical approach to the local contact potential difference on (100) ionic surfaces: implications for KPFM* », F.Bocquet, L.Nony, Ch.Loppacher and Th.Glatzel, **Phys. Rev. B** 78, 035410 (2008).
- « *Evolution of the Electronic Structure at the Interface between a Thin Film of Halogenated Phthalocyanine and the Ag(111) Surface* », L.Giovanelli, P.Amsalem, J.M.Themlin, Y.Ksari, M.Abel, L.Nony, M.Koudia, F.Bondino, E.Magnano, M.Mossoyan-Deneux, and L.Porte, **J. Phys. Chem. C** 112, 8654-8661 (2008).
- « *Interface dipole formation of different ZnPcCl8 phases on Ag(111) observed by Kelvin probe force microscopy* », P.Milde, U.Zerweck, L.Eng, M.Abel, L.Giovanelli, L.Nony, M.Mossoyan, L.Porte, and Ch.Loppacher, **Nanotechnology** 19, 305501 (2008).
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