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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

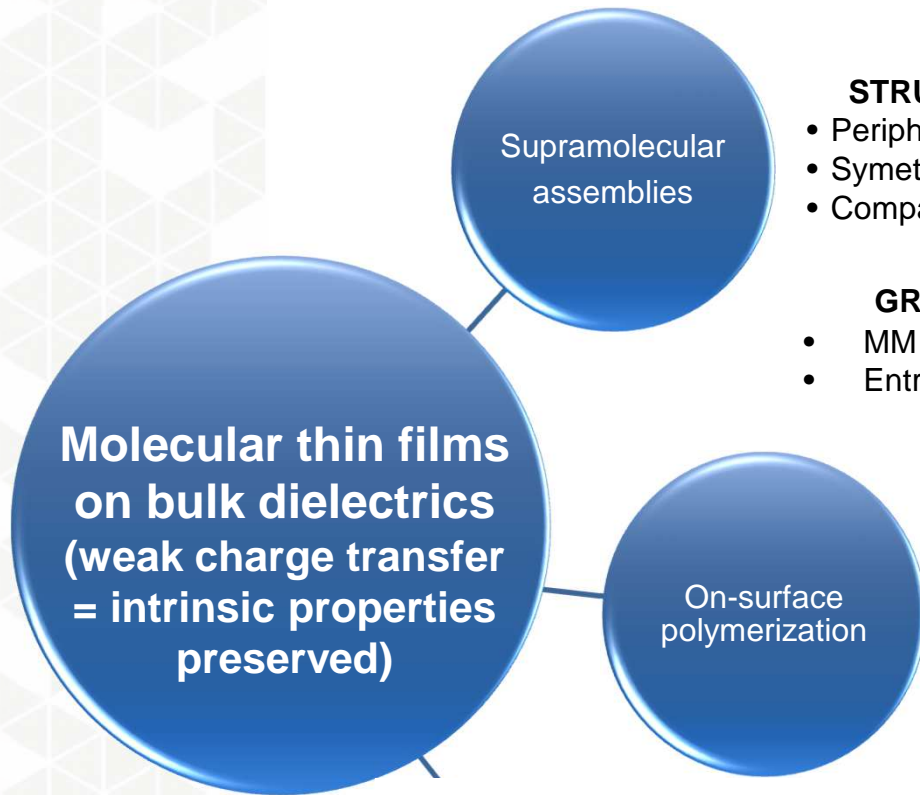
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Ecole thématique « Couplage entre les techniques de microscopies à sondes locales et l'optique »  
18-19 Mars 2019, Carry le Rouet



# Research topics of the Nanostructuration group

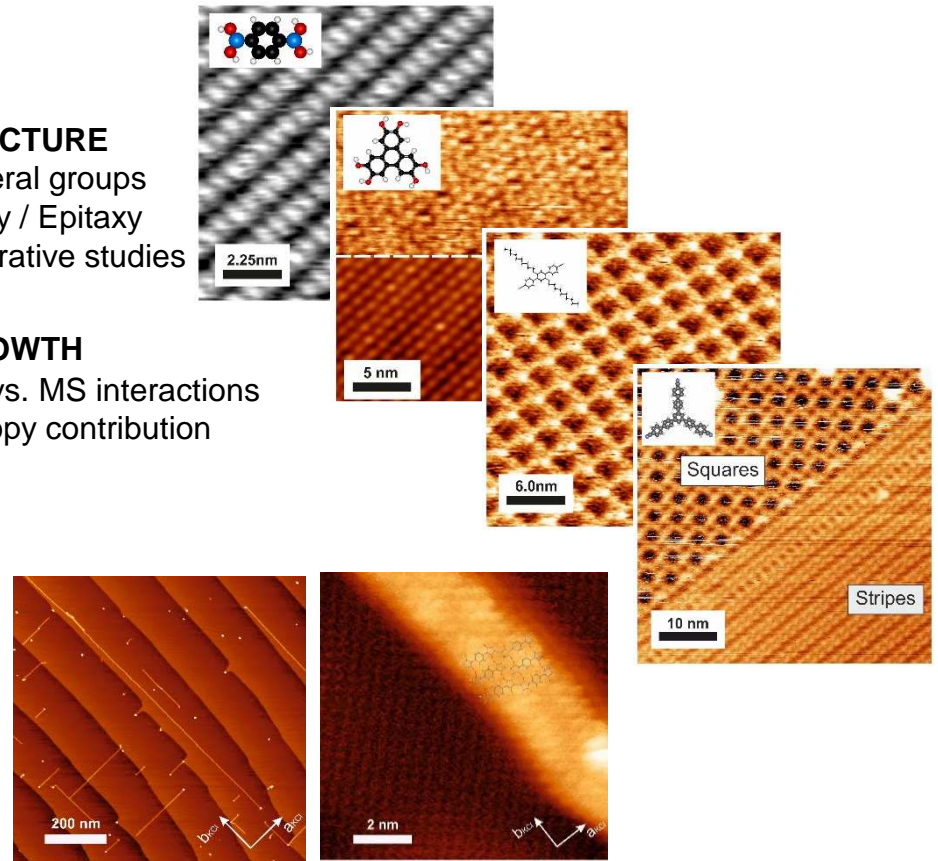


## STRUCTURE

- Peripheral groups
- Symetry / Epitaxy
- Comparative studies

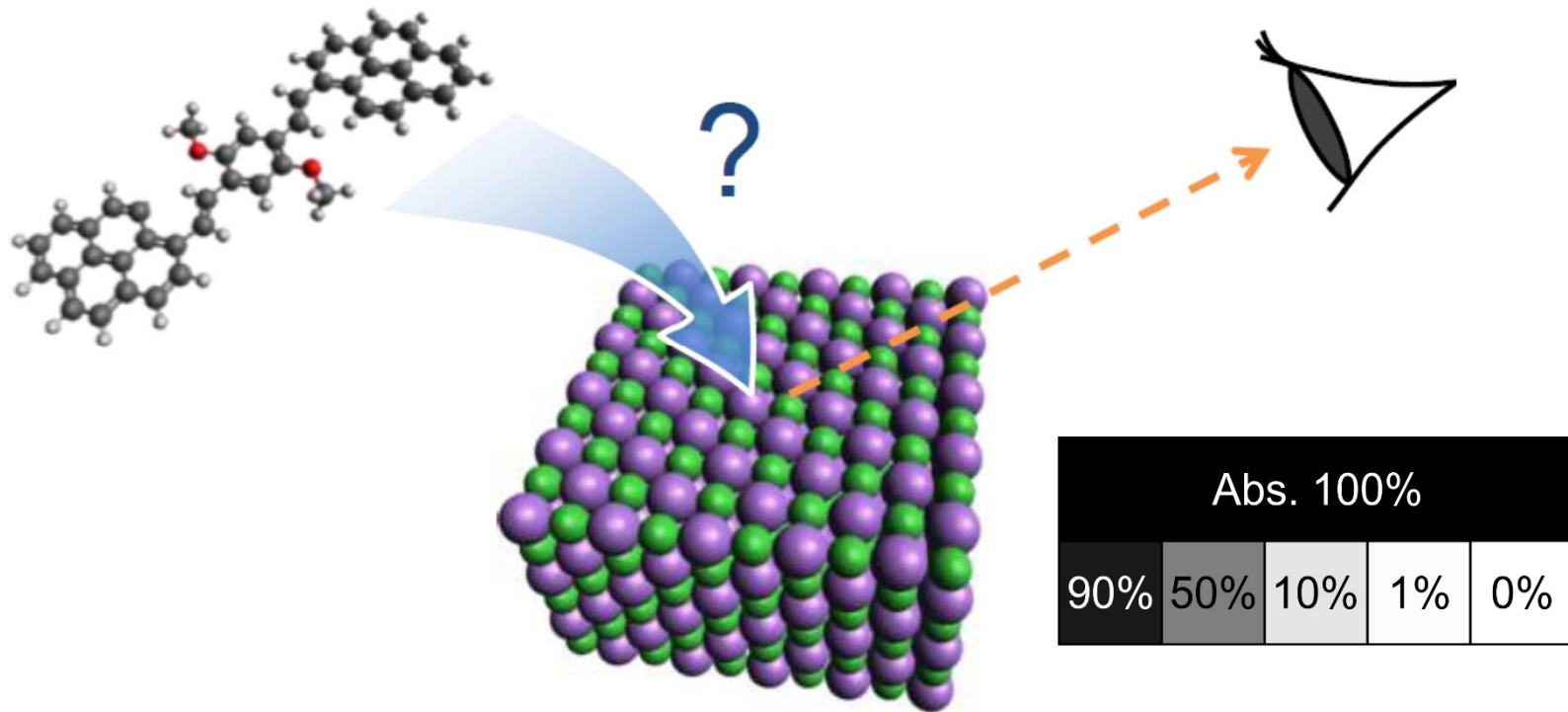
## GROWTH

- 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

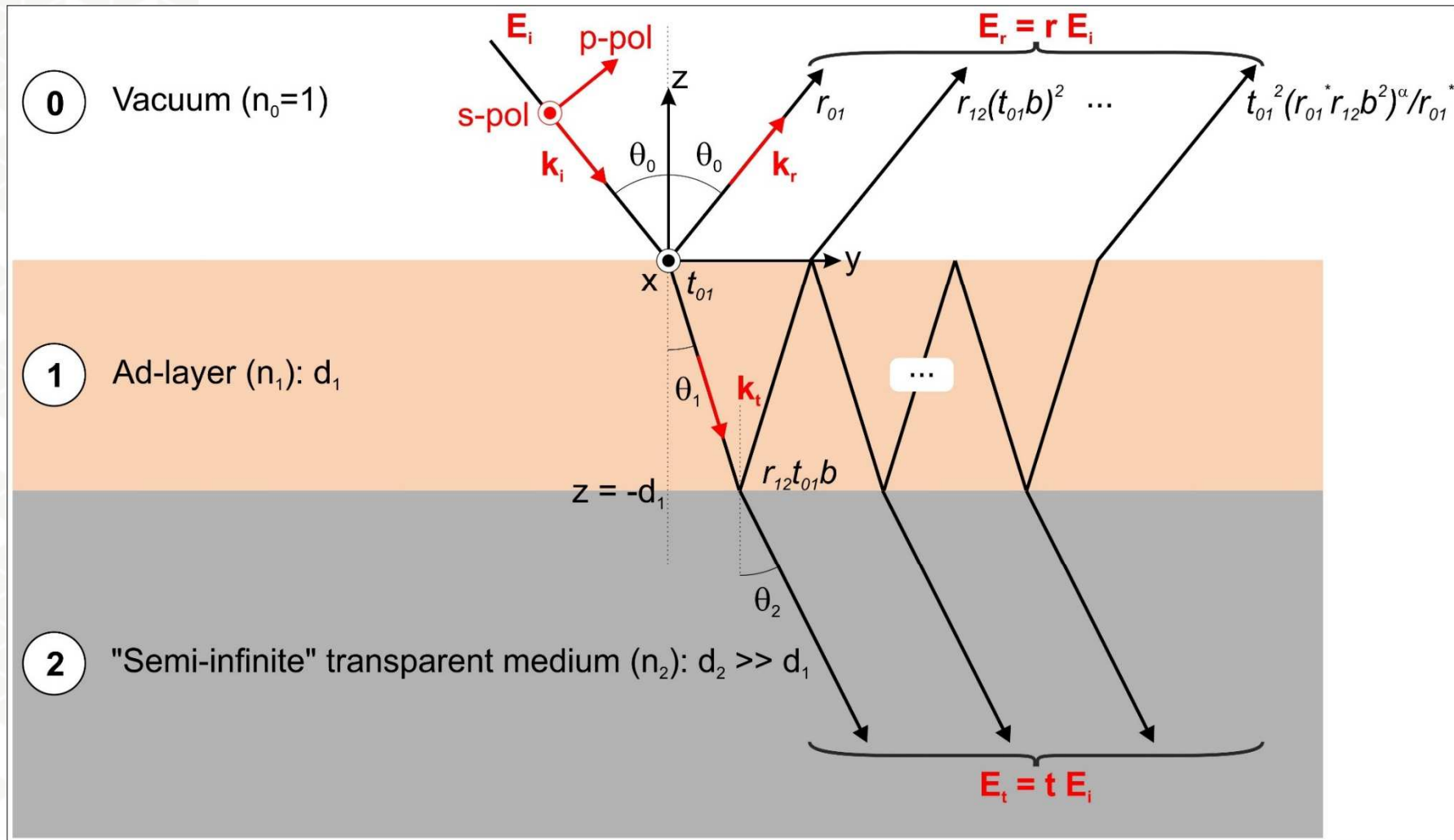
IV. DRS fonctionnal for fitting the DR spectra

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 procedure<sup>1</sup> ( $d_1 \ll \lambda$ ) 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:  $n(\lambda) = \sqrt{\epsilon(\lambda)}$ )

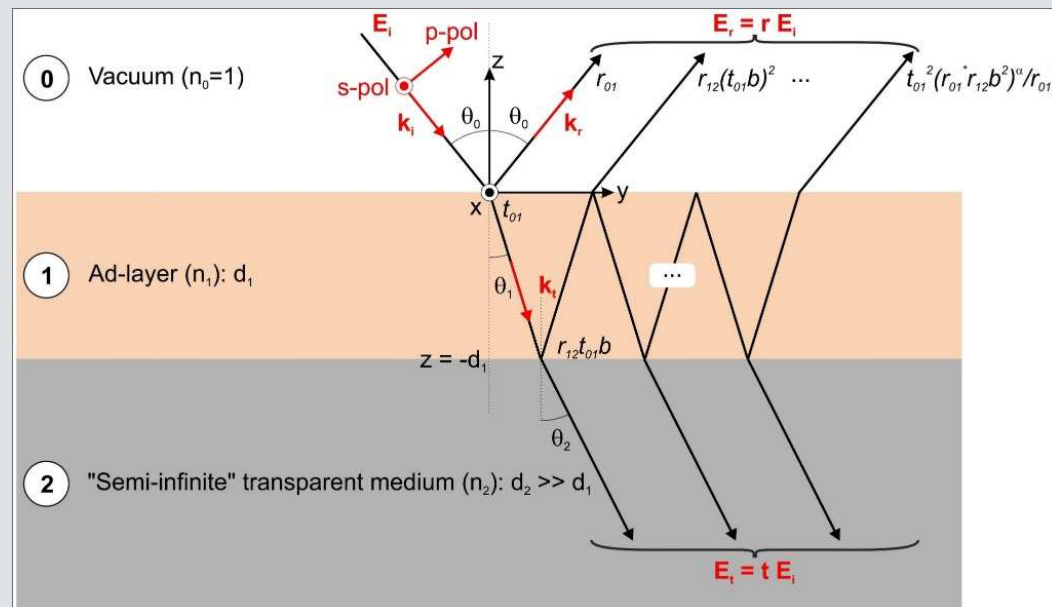
<sup>1</sup>J.D.E.McIntyre et al., Surf.Sci. 24, 417 (1971)



# 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  $\rightarrow$  Relations de Fresnel  $\rightarrow$  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

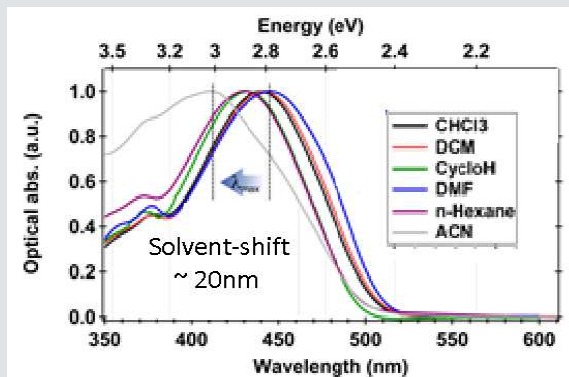
Intuiter correctement le  $n(\lambda)$  d'input du fit:

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

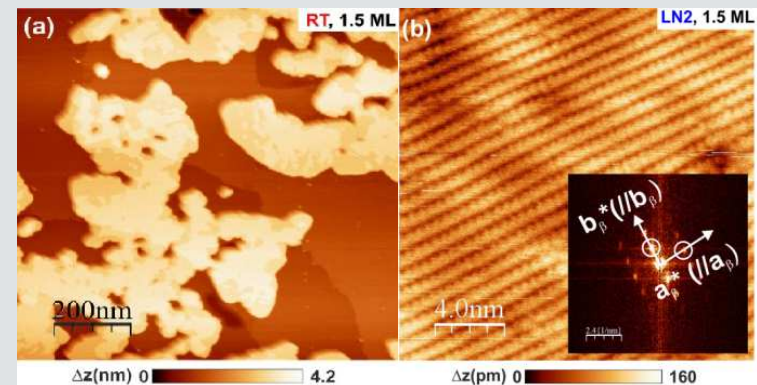
Caractérisation exhaustive du dépôt:

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

Etude préalable de la  $DO(\lambda)$

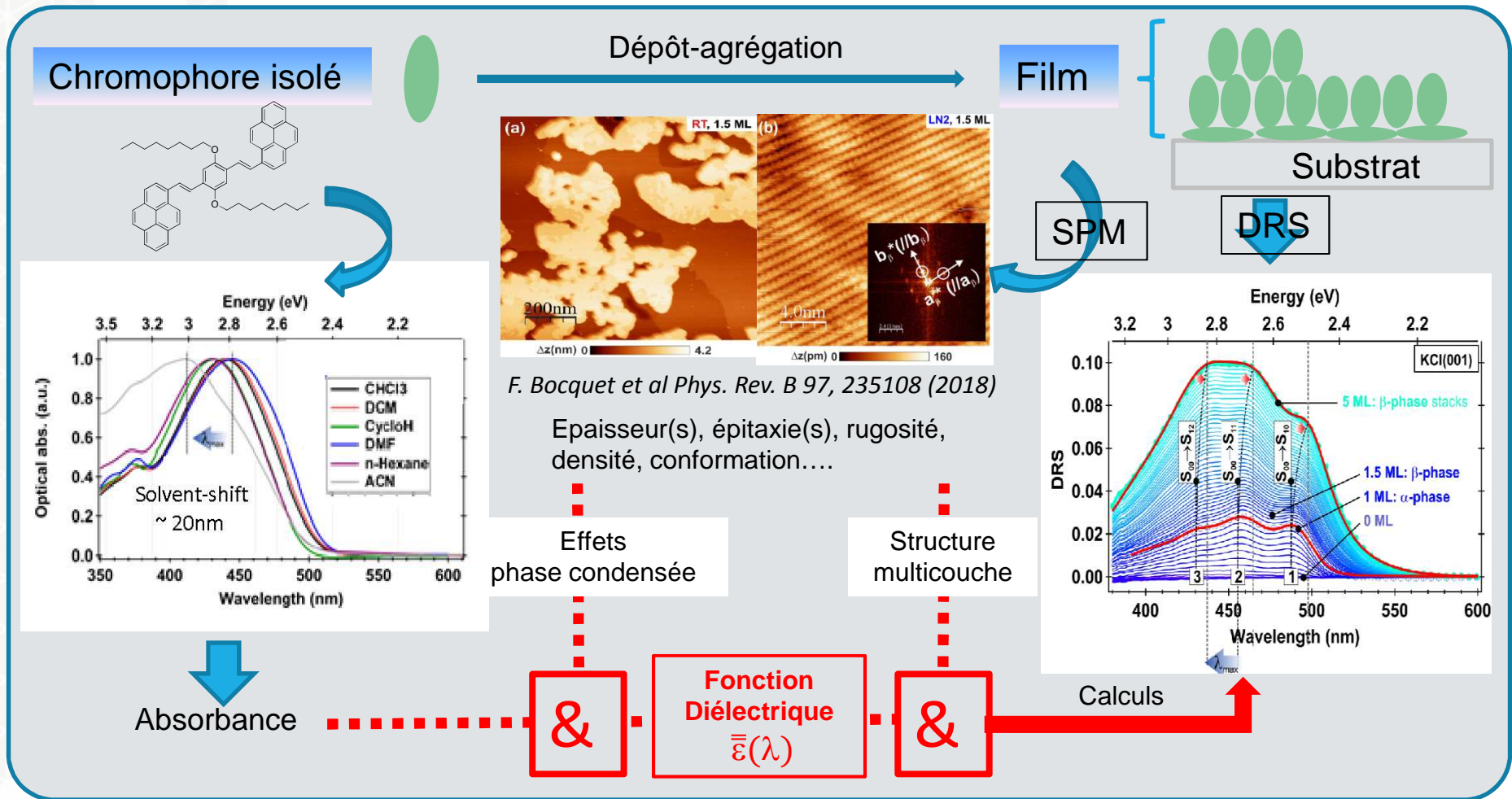


Analyse SPM « poussée »



# Reminder, context of DRS

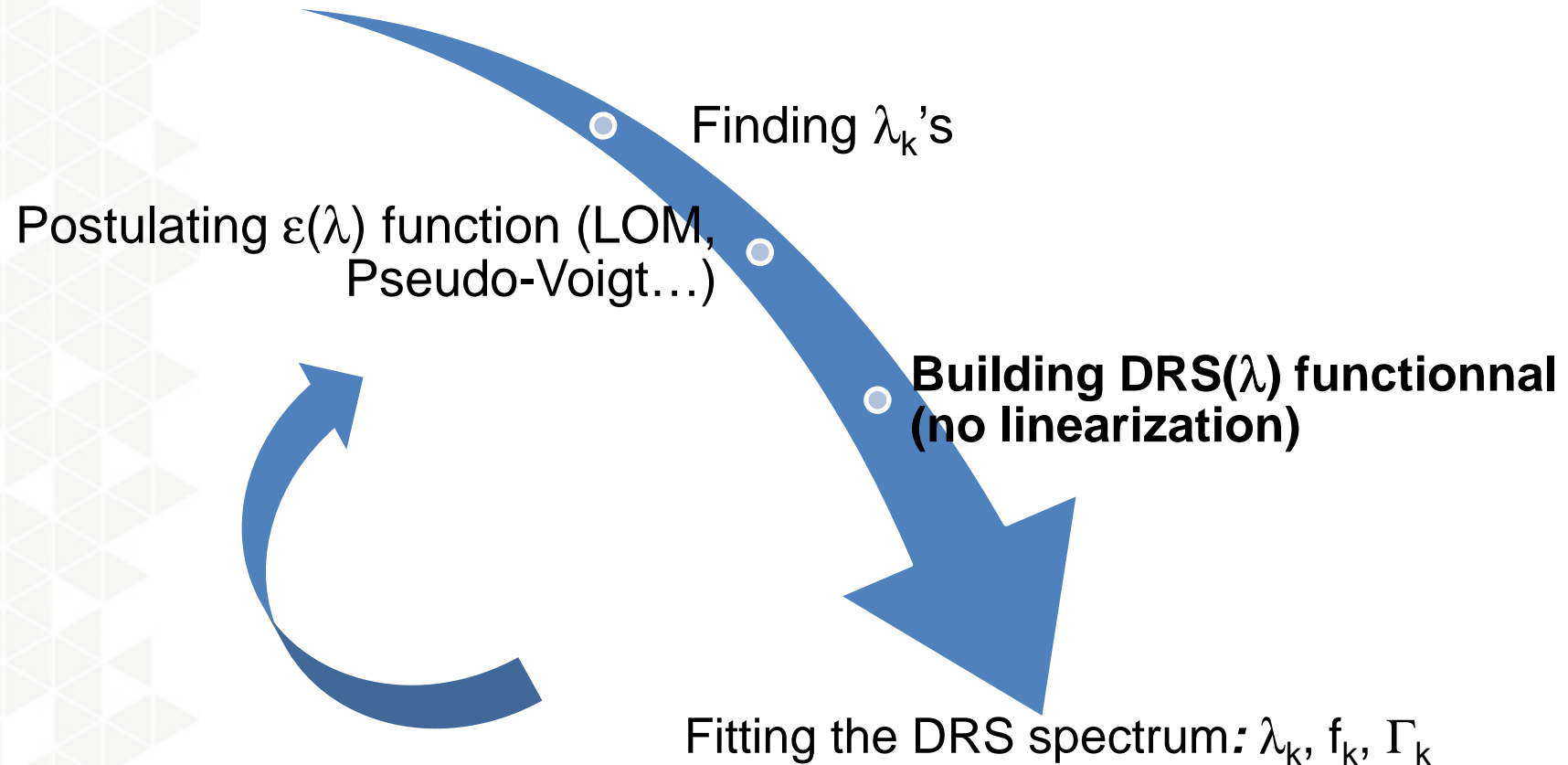
- Franck Bocquet's lecture on « Theoretical aspects of DRS on molecular films »:



# Reminder, context of DRS

11

- Ultimate methodology to extract the dielectric function :  
**DRS spectrum**



# Outline

I. Reminder, context of DRS on molecular films

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

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*List of publications*

# 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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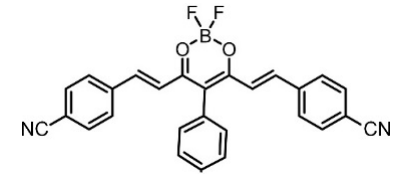
V. Conclusion

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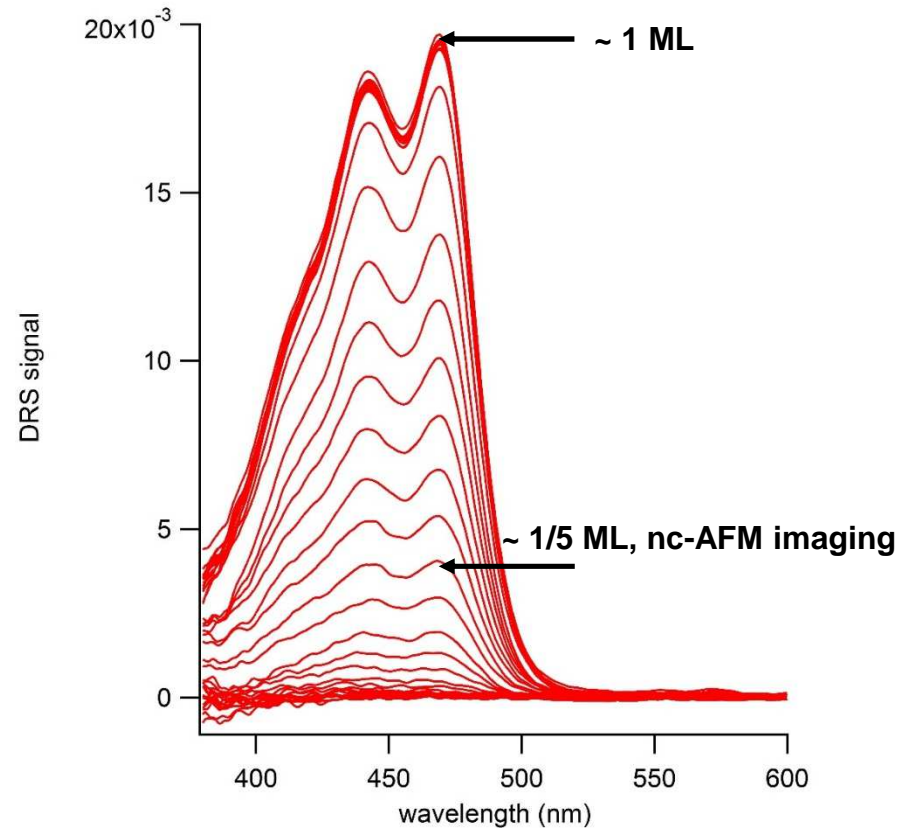
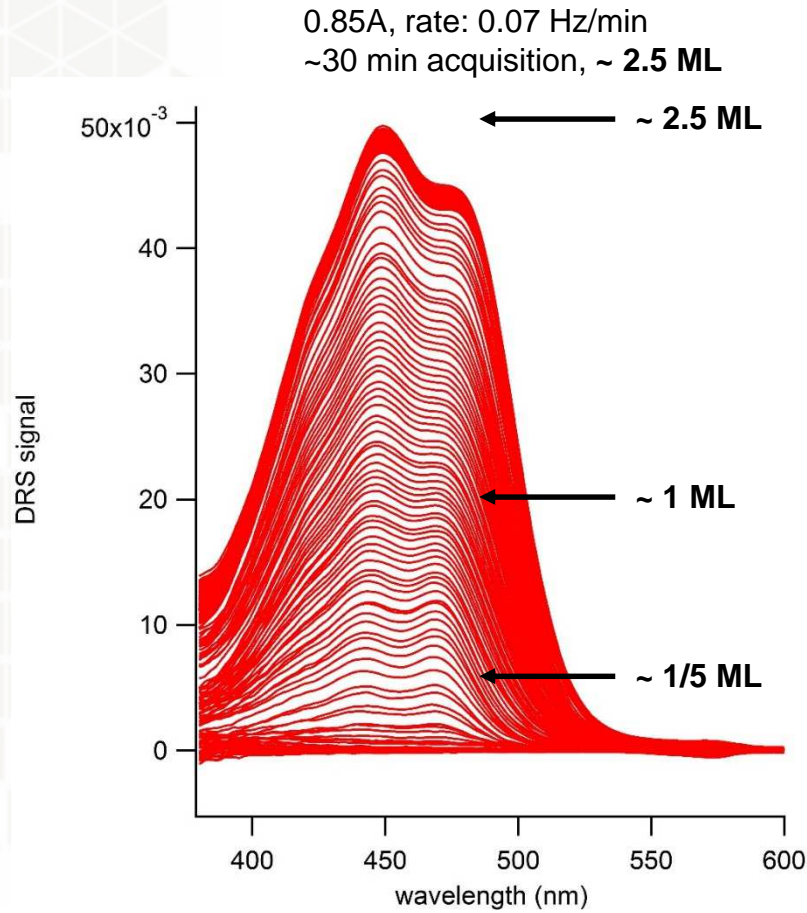
# Connection to the structural properties of the molecular films by nc-AFM

- Exemplary case : curcuminoid chromophores



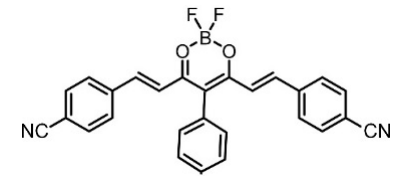
I: DRS in the multi-layer regime

III: DRS in the sub-ML regime

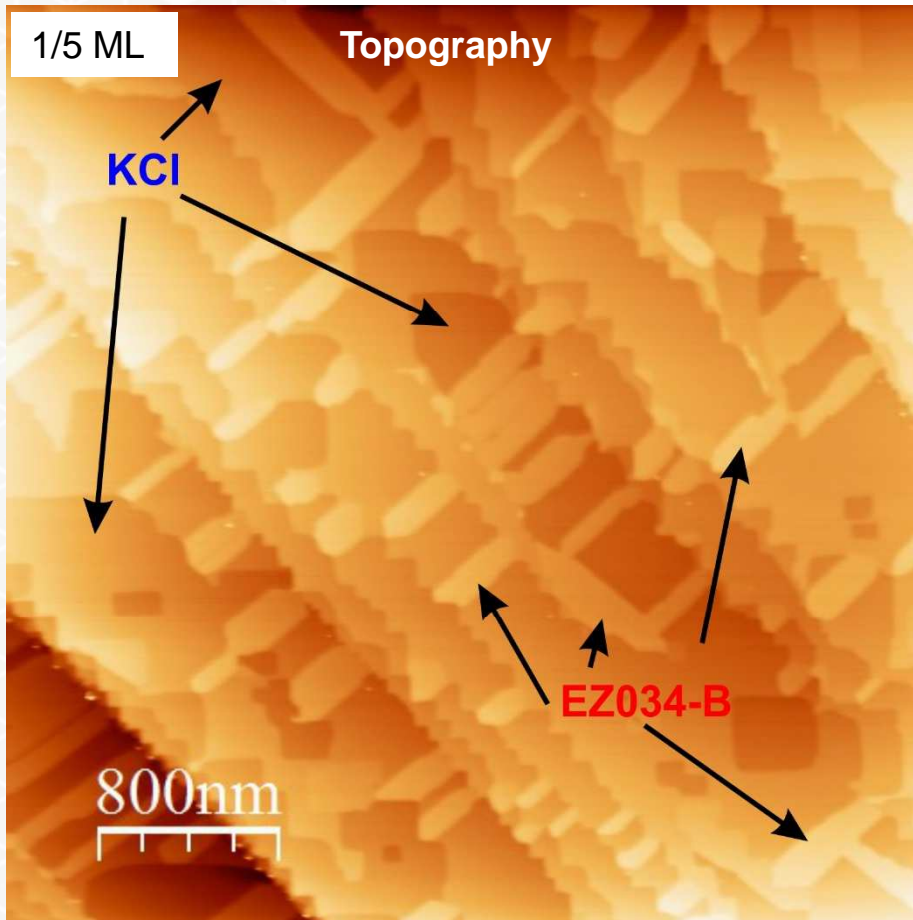


# Connection to the structural properties of the molecular films by nc-AFM

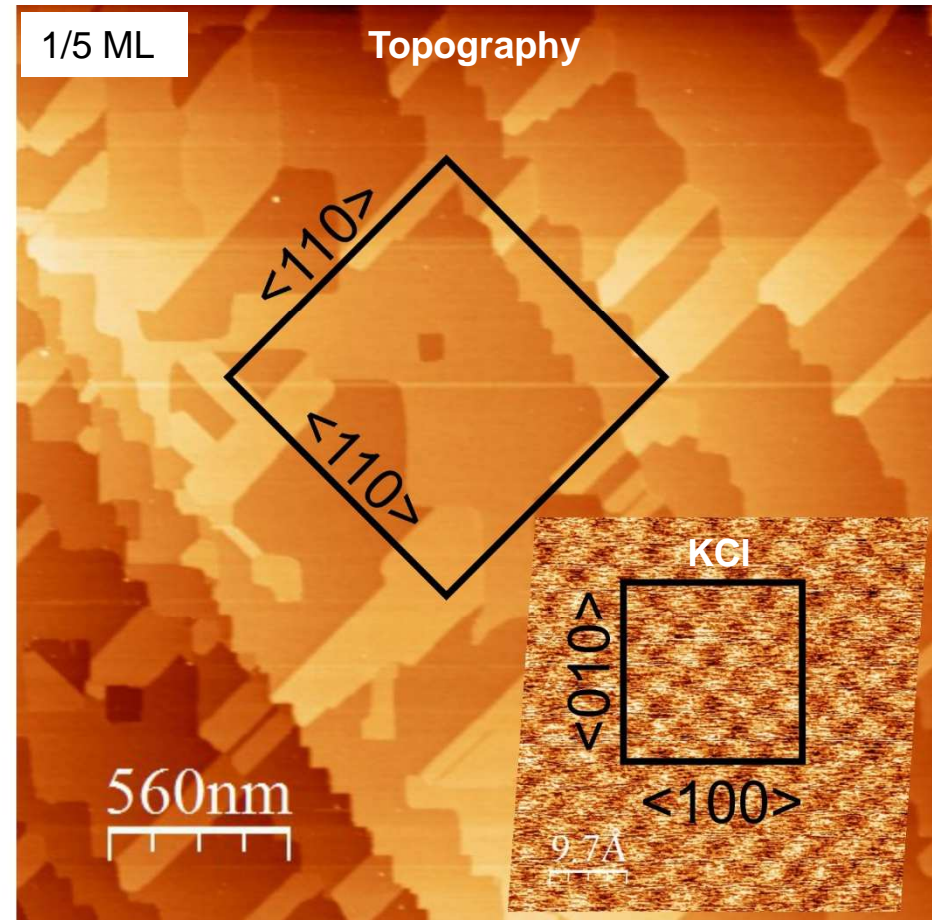
- Exemplary case : curcuminoid chromophores



Large crystallites of equivalent domains

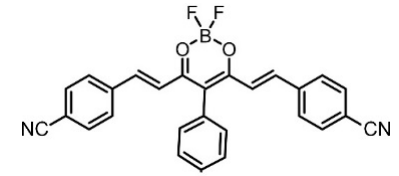


Orientations of ML @ +/-45° w.r.t. <100> direction



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

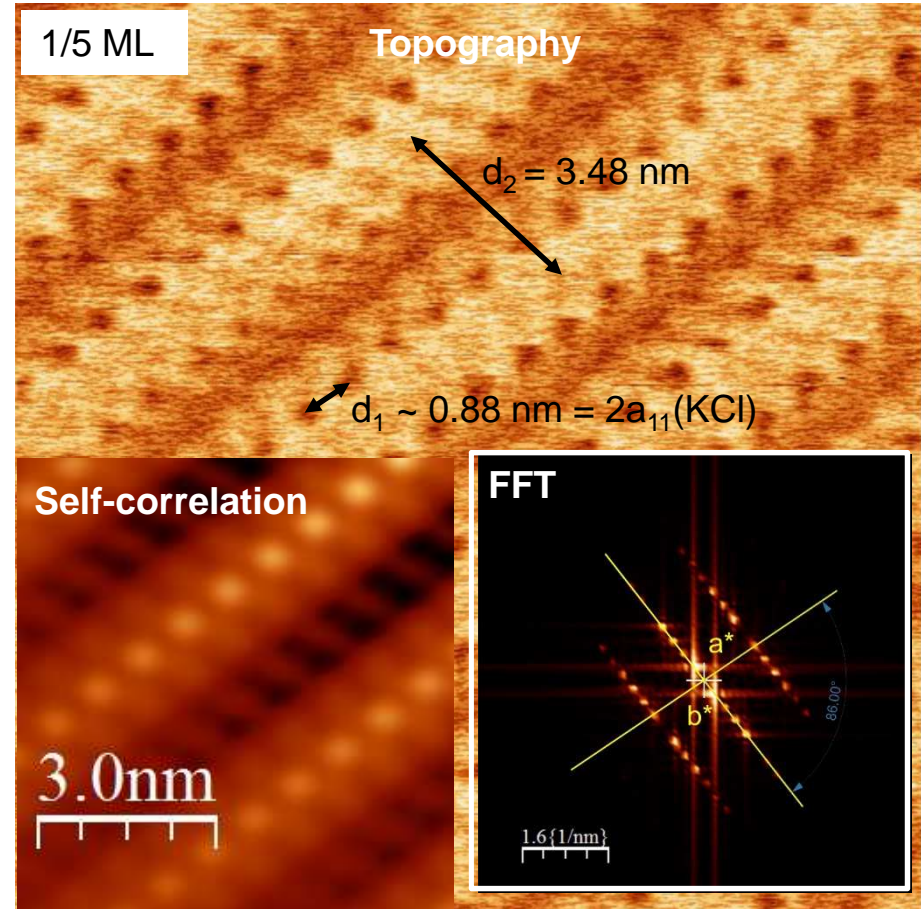
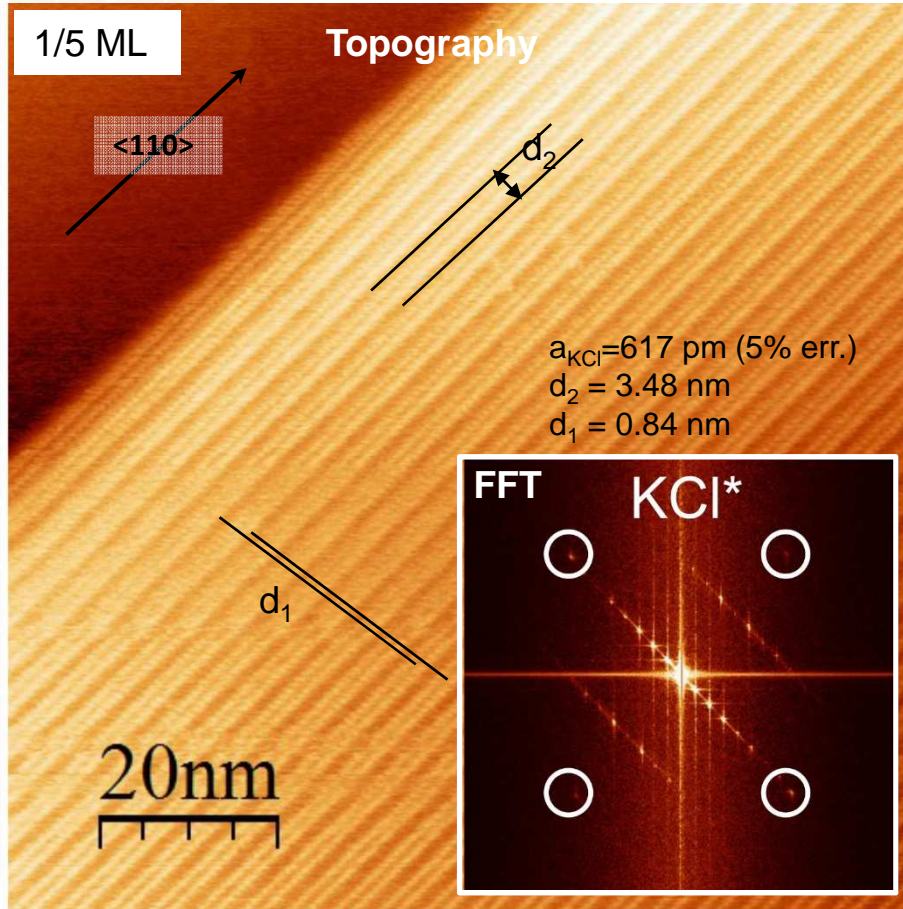
- Exemplary case : curcuminoid chromophores



## Zoom on a domain:

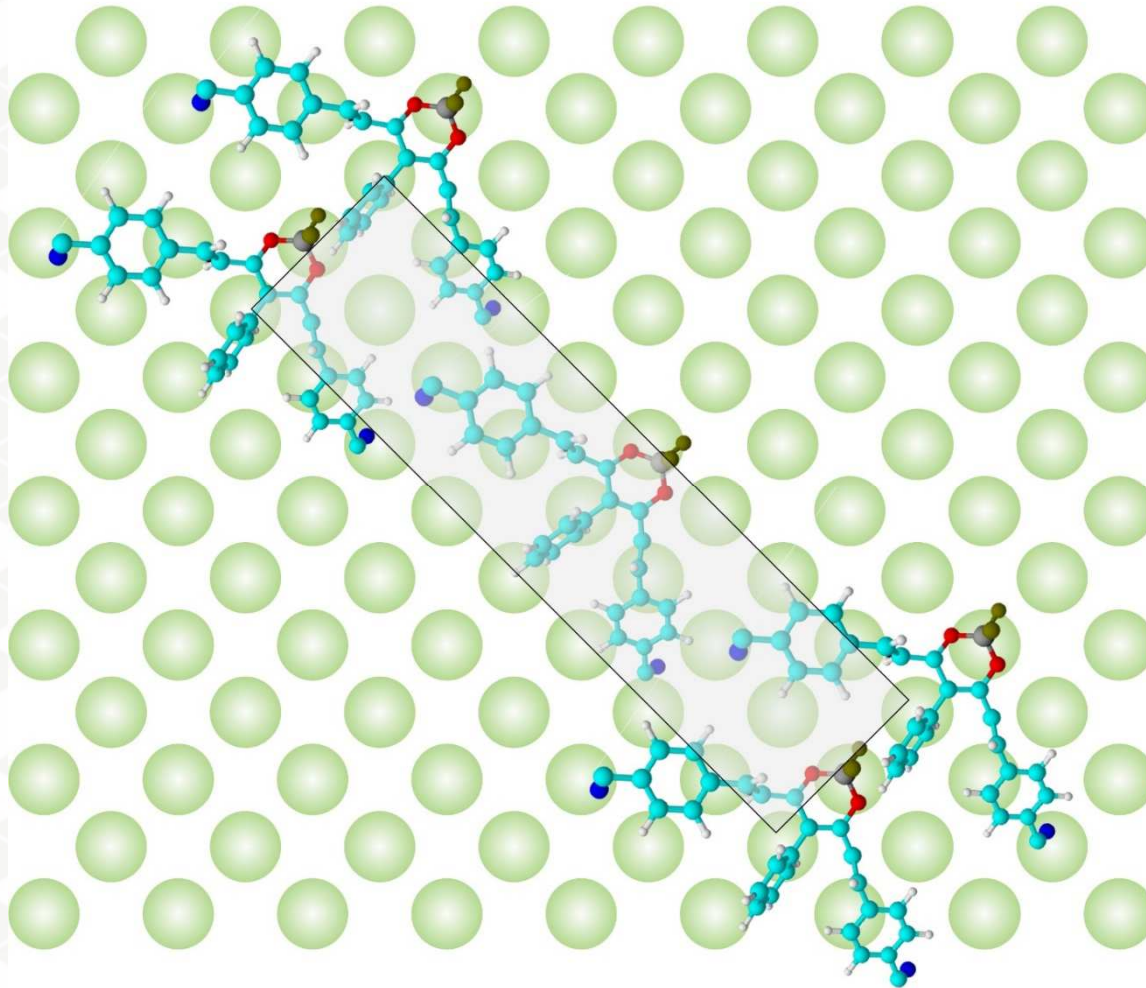
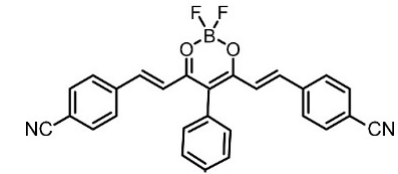
- Molecular rows match substrate's <110> polar lines
- Apparent height : 4.0 Å
- Atomic resolution on KCl (traces in FFT)

## 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**

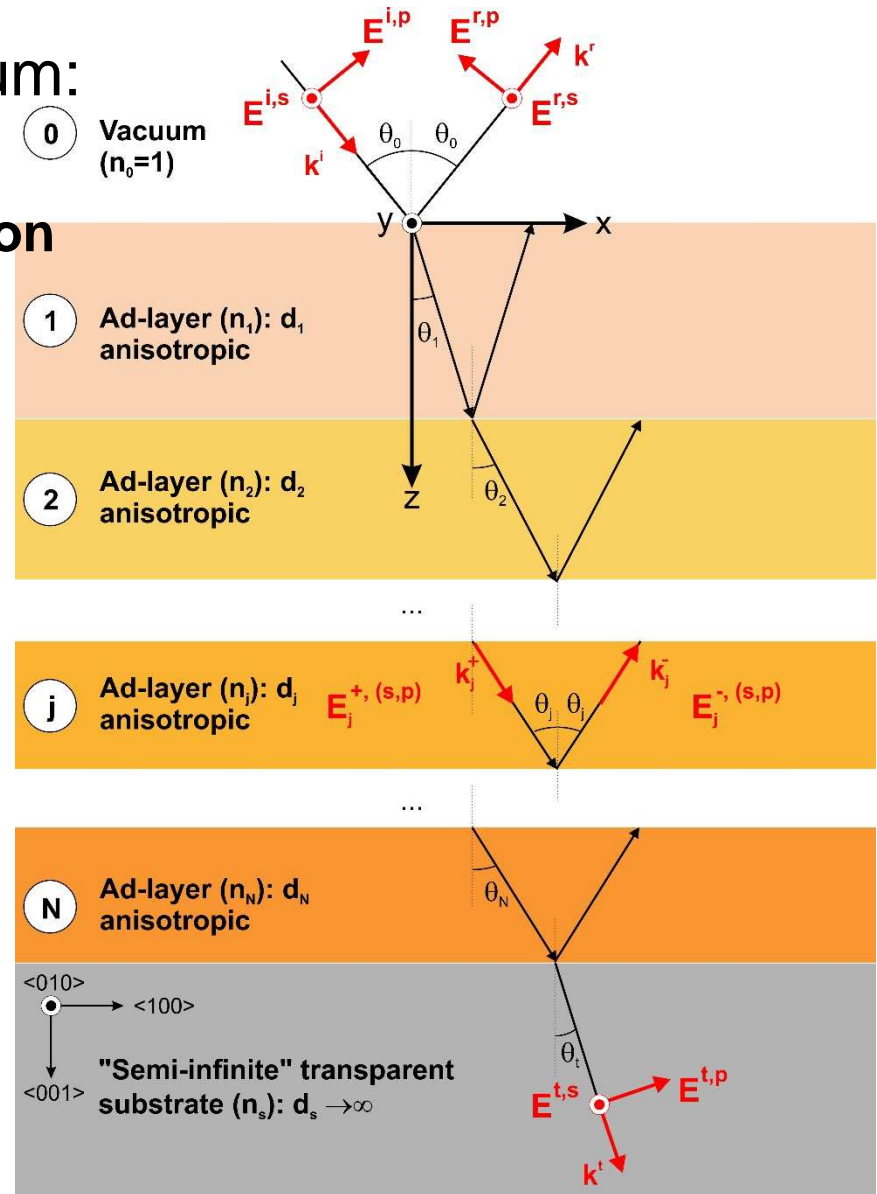
(x,y,z geom. accordingly with cristallographic axes)

- z axis = optic axis

- Dielectric tensor of the j<sup>th</sup> 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:

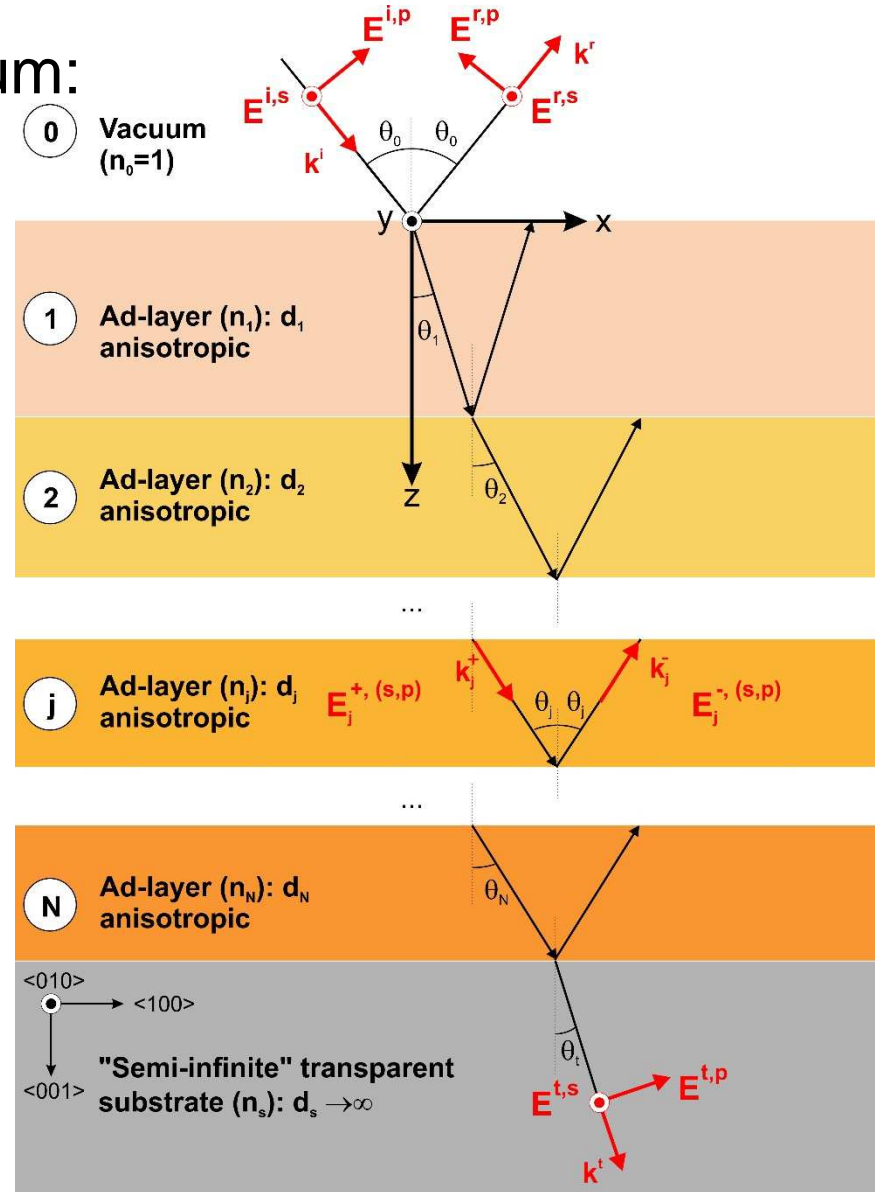
$$\vec{E}^\dagger = \vec{E}^{\dagger,s} + \vec{E}^{\dagger,p},$$

(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)<sup>1</sup>:
  - Wave vector for a j<sup>th</sup> layer

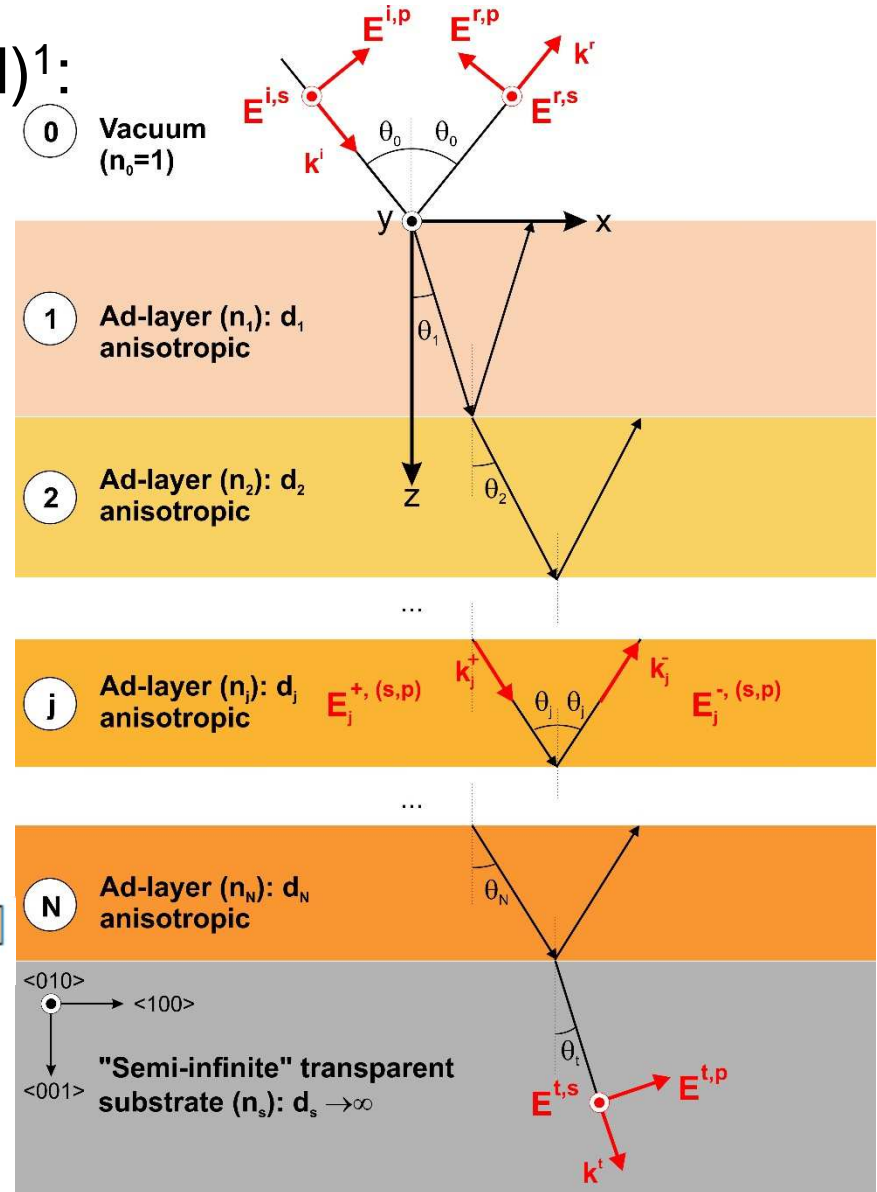
$$\mathbf{k}_j^{(i,r)} = k_j^{(i,r),x} \mathbf{e}_x + k_j^{(+,-),z} \mathbf{e}_z$$

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:

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



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

# DRS fonctionnal for fitting the DR spectra

- Transfer matrix method (TMM)<sup>1</sup>:
  - 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

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

# DRS fonctionnal for fitting the DR spectra

- Transfer matrix method (TMM):

- $\alpha_j$  and  $\beta_j$  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):

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

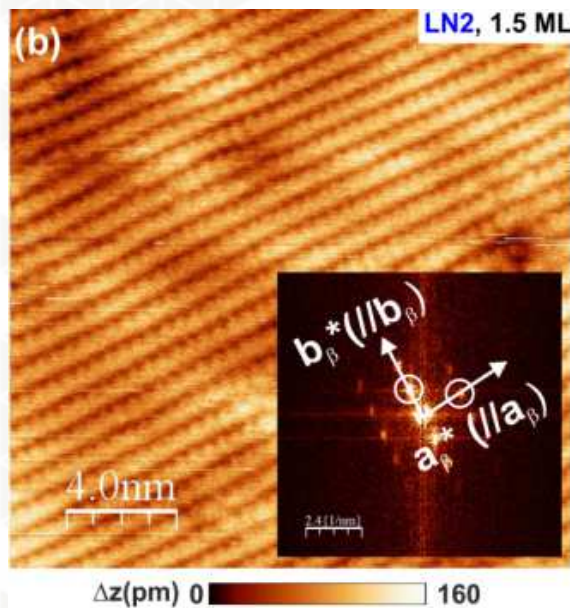
yields the DRS **fitting fonctionnal!**

# 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):

Number of transitions

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

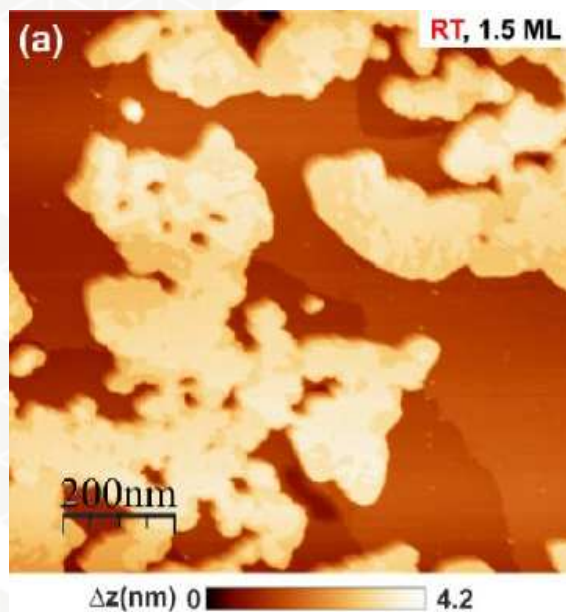


With:

- $f_j$  = oscillator strength
- $\lambda_j$  = absorption wavelength
- $\gamma_j$  = 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 Theory<sup>1</sup> + 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

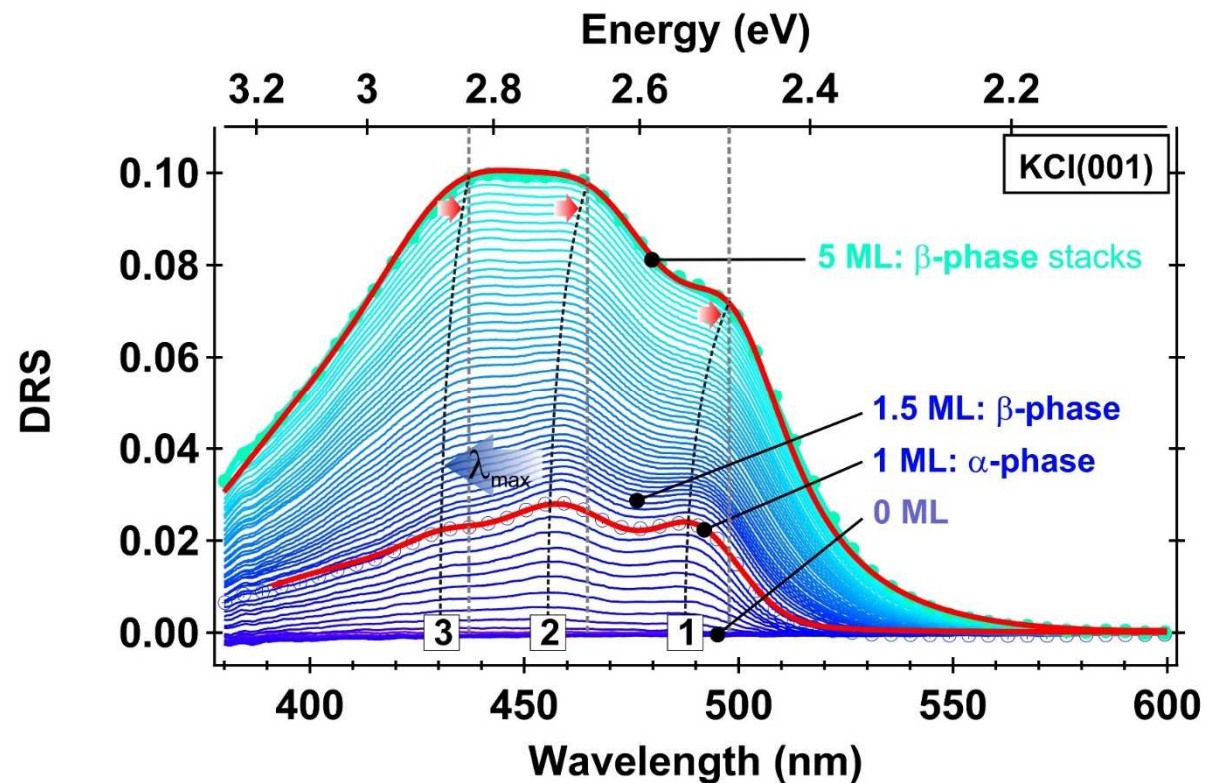
Filling parameter [0;1]

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

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

# DRS functional 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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## V. Conclusion

### *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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- Conrad Becker
- Laurence Masson
- Romain Parret
- Frédéric Fagès
- Elena Zaborova



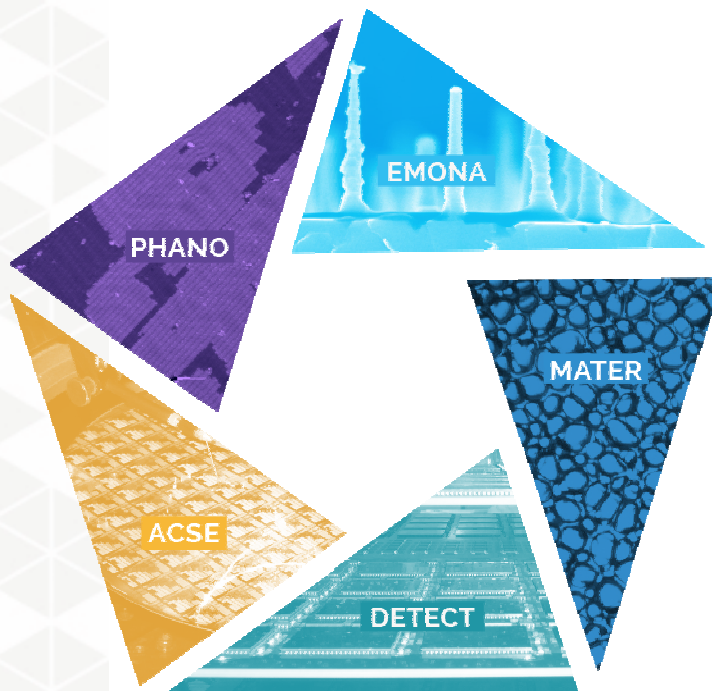
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# Merci pour votre attention



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

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