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Caractérisation de la microstructure de couches minces non homogènes par techniques optiques Application au ZnO et au $LiNbO_3$

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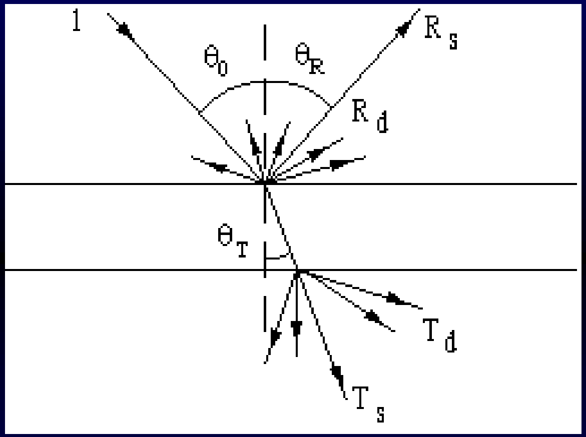
Optical techniques

- Why using optical techniques for the determination of the microstructrure of a thin film ?
- Advantages : non-destructive techniques, no sample preparation, measurements in the air
- Drawbacks : use of complex mathematical models to determine this microstructure
- Optical techniques : Spectrophotometry, X-Ray reflectometry, Ellipsometry

Optical techniques

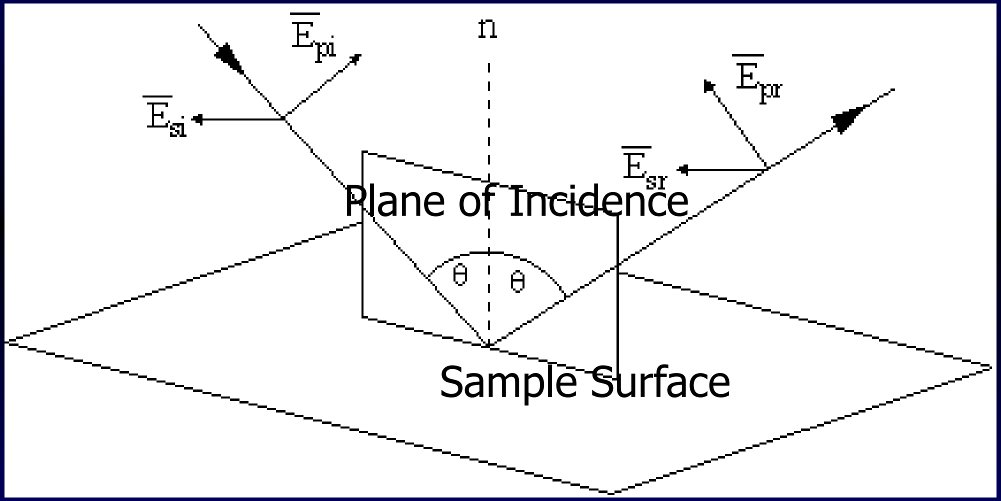
- Measurement of specular and diffuse reflectivity and transmitivity of light : R_S T_S R_D T_D



- R_S T_S : Spectrophotometry : wl =190 - 2500 nm
- R_S : X-Ray Reflectometry : wl = 0.15418 nm
- R_D T_D : Spectrophotometry with integration sphere : wl =190 - 2500 nm

Optical techniques

- Measurement of the change of the polarisation state of polarised light by specular reflection : Δ and Ψ



$$R_p = E_{pr} / E_{pi}$$

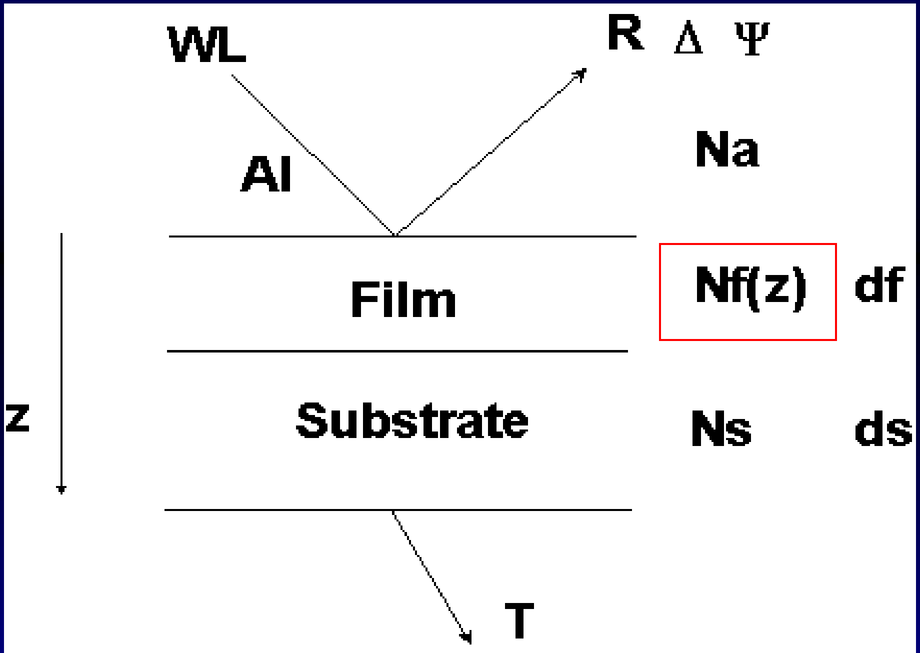
$$R_s = E_{sr} / E_{si}$$

$$R_p / R_s = \tan \Psi \, e^{j\Delta}$$

- Δ and Ψ : Ellipsometry : wl = 300 - 850 nm

Models

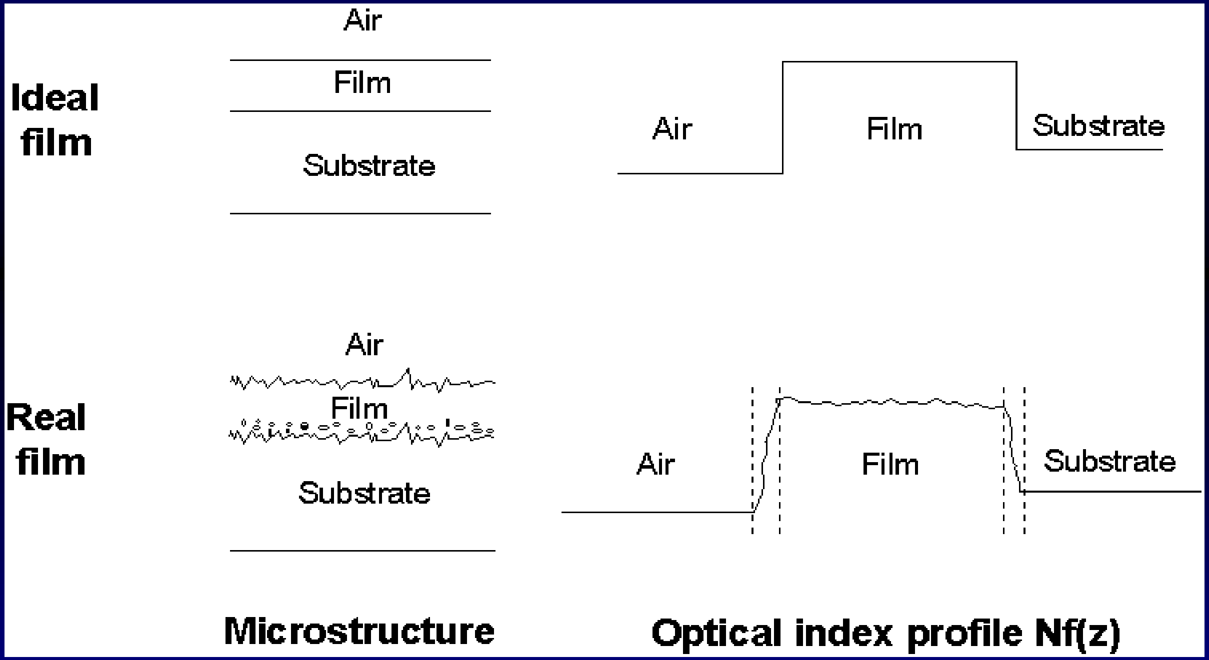
- Optical measurements depend on : experimental parameters and physical parameters of a film



- R, T, Δ and Ψ = f (N_a , N_F (z) , d_F , N_s , d_s , WL, AI)

Models

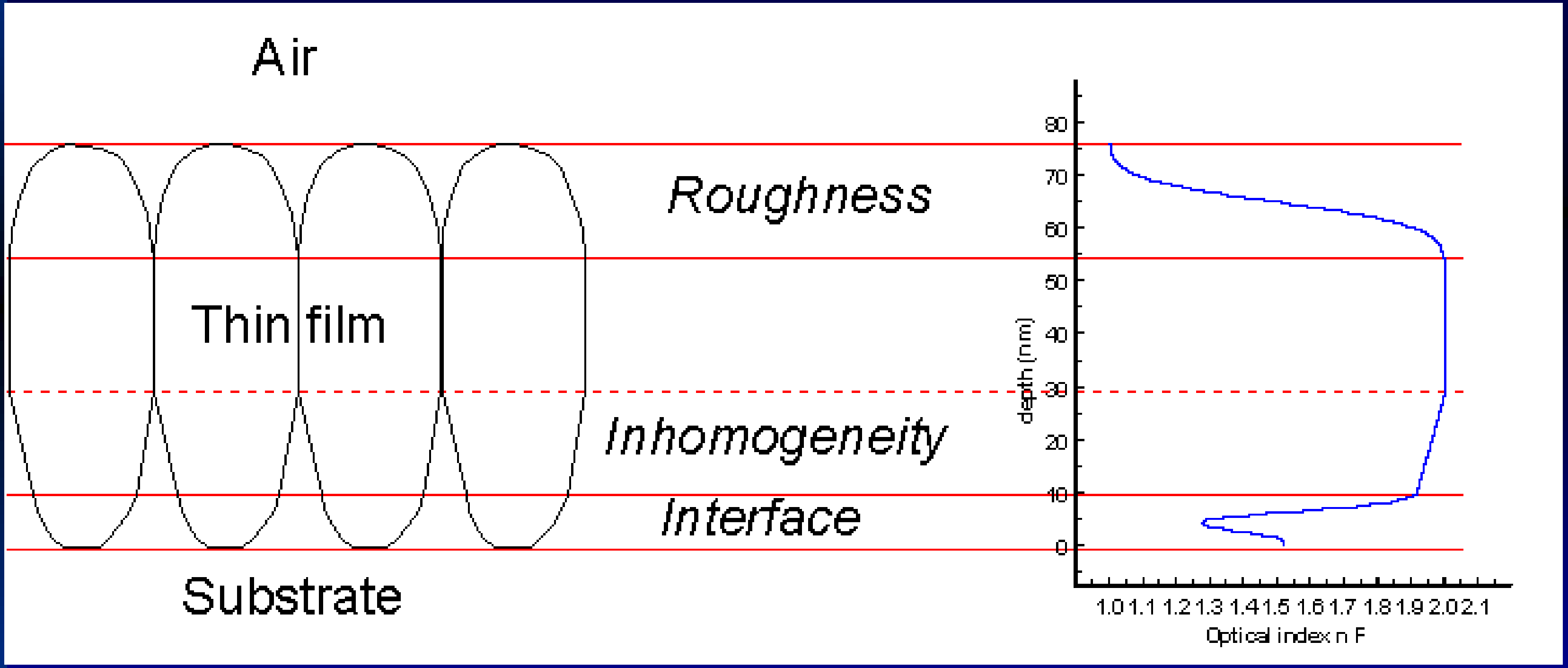
- Relation between the microstructure and the optical index profile of a film



- Determination of the microstructure = calculation of the optical index profile $N_F(z)$

Real microstructures

- Relation between the index profile and the real structure of a film :



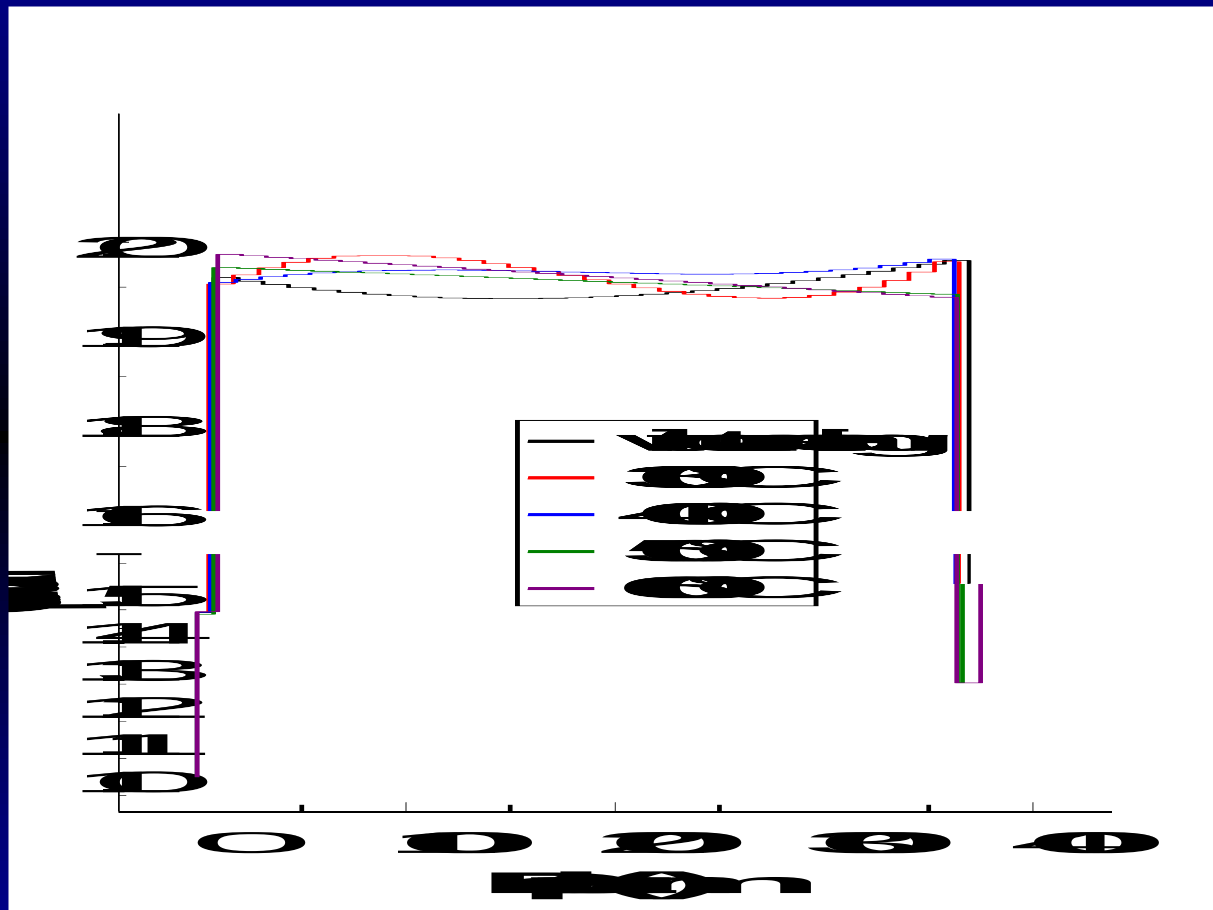
Fields of application

- Each technique has its own field where it works well, different from the two other ones
- Sensitivity :
  - spectrophotometry not very sensitive to the microstructure of the films ↑ can only be used to determine the thickness of a film
  - ellipsometry not very sensitive to the presence of interfaces ↑ can only be used to determine the roughness and inhomogeneity of a film
  - X-Ray reflectometry not very sensitive to inhomogeneity ↑ can only be used to determine the roughness or presence of interface in a film

Fields of application

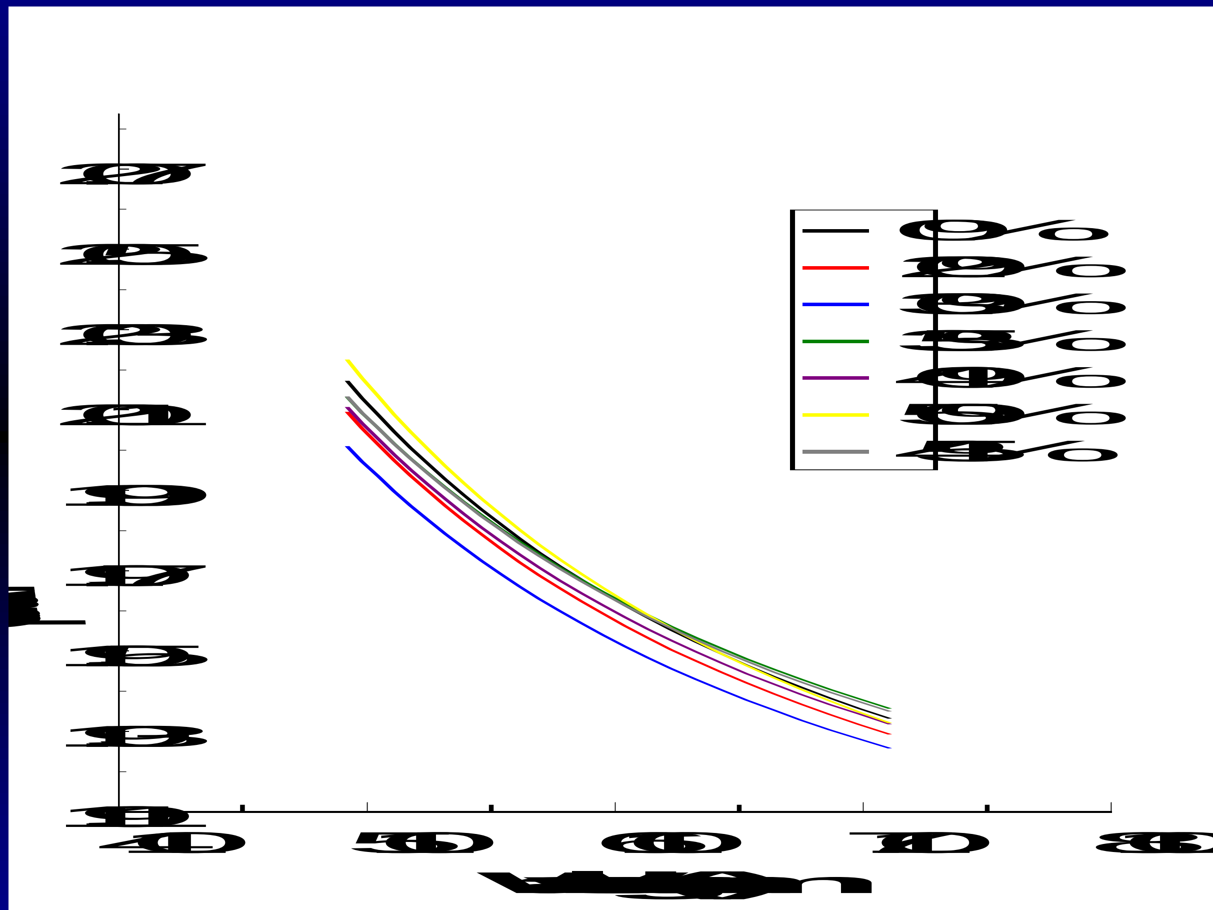
- Each technique has its own field where it works well, different from the two other ones
- Thickness range :
  - spectrophotometry and ellipsometry : $d_F > 50$ nm
  - X-Ray reflectometry : $d_F < 100$ nm, sample not too rough

Ellipsometry



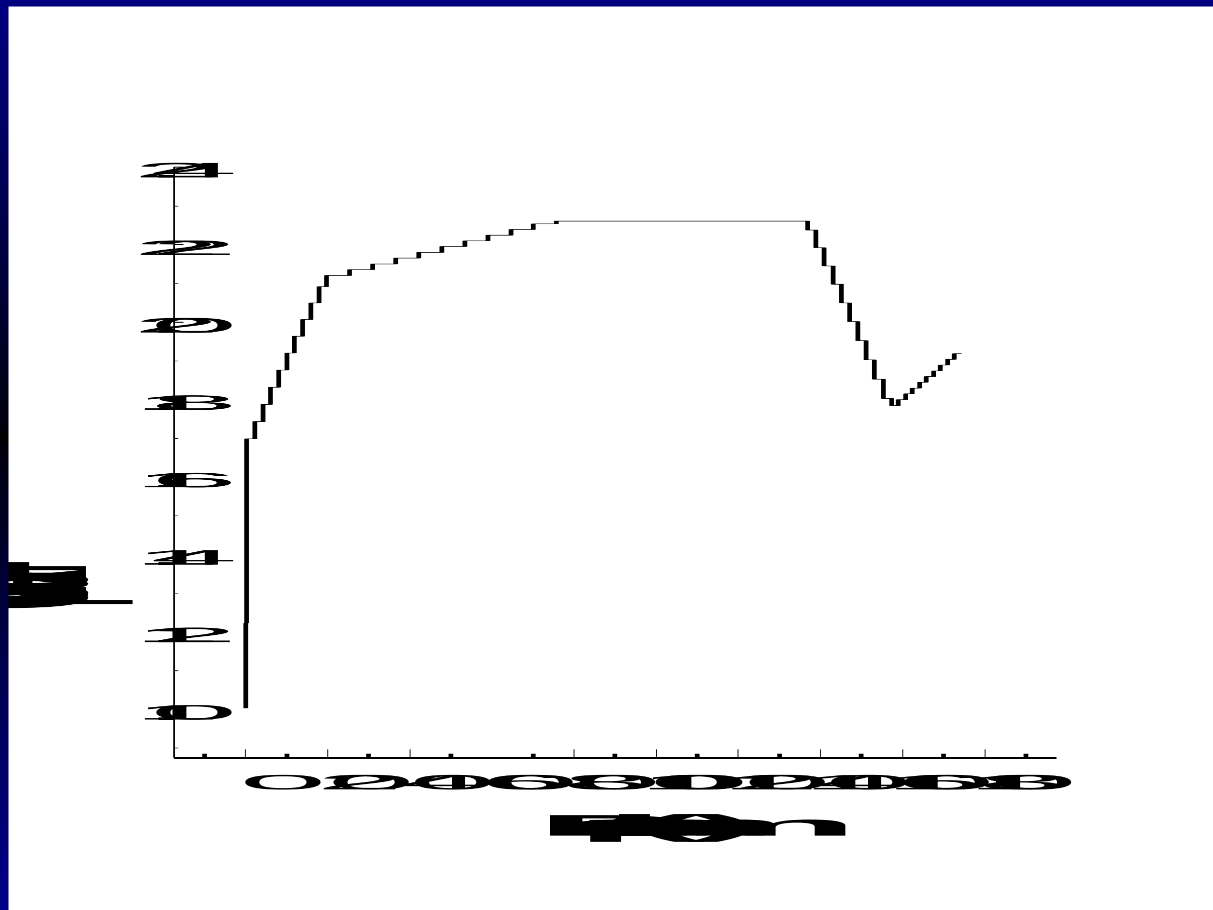
Annealing of a ZnO layer

Ellipsometry



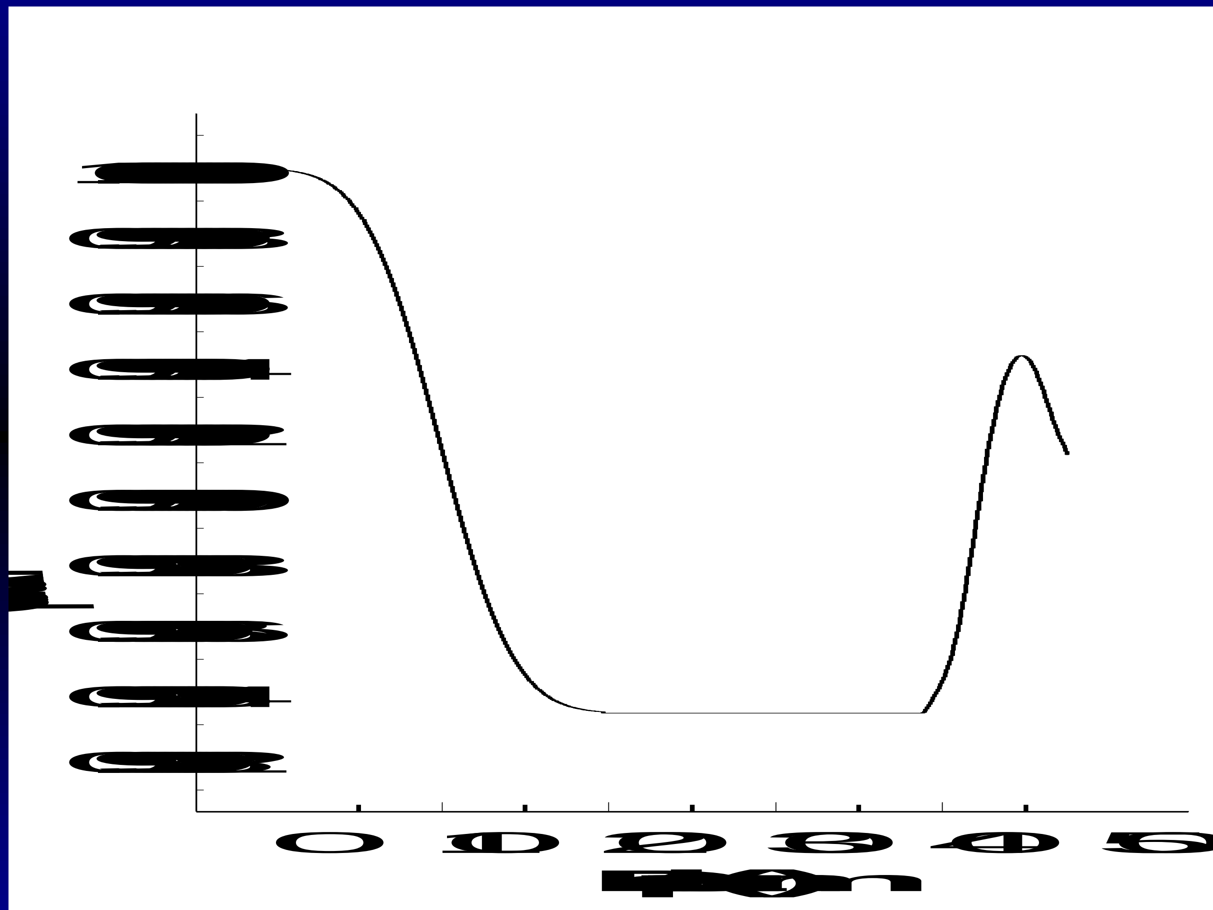
Influence of the O_2 percentage in ZnO films

Ellipsometry



LiNbO$_3$ on sapphire

X-Ray reflectometry



Index profile of a thin ZnO film

3 techniques together

- The optical index profile depends on the wavelength wl
- ↑ creation of a « new model » based on a volume fraction of material profile $F_v(z)$ not dependant on wl
- ↑ $N_F(z, wl) = f(N_{material}(wl), F_v(z))$

Visible wavelengths : $N_{material} = n - j\,k$

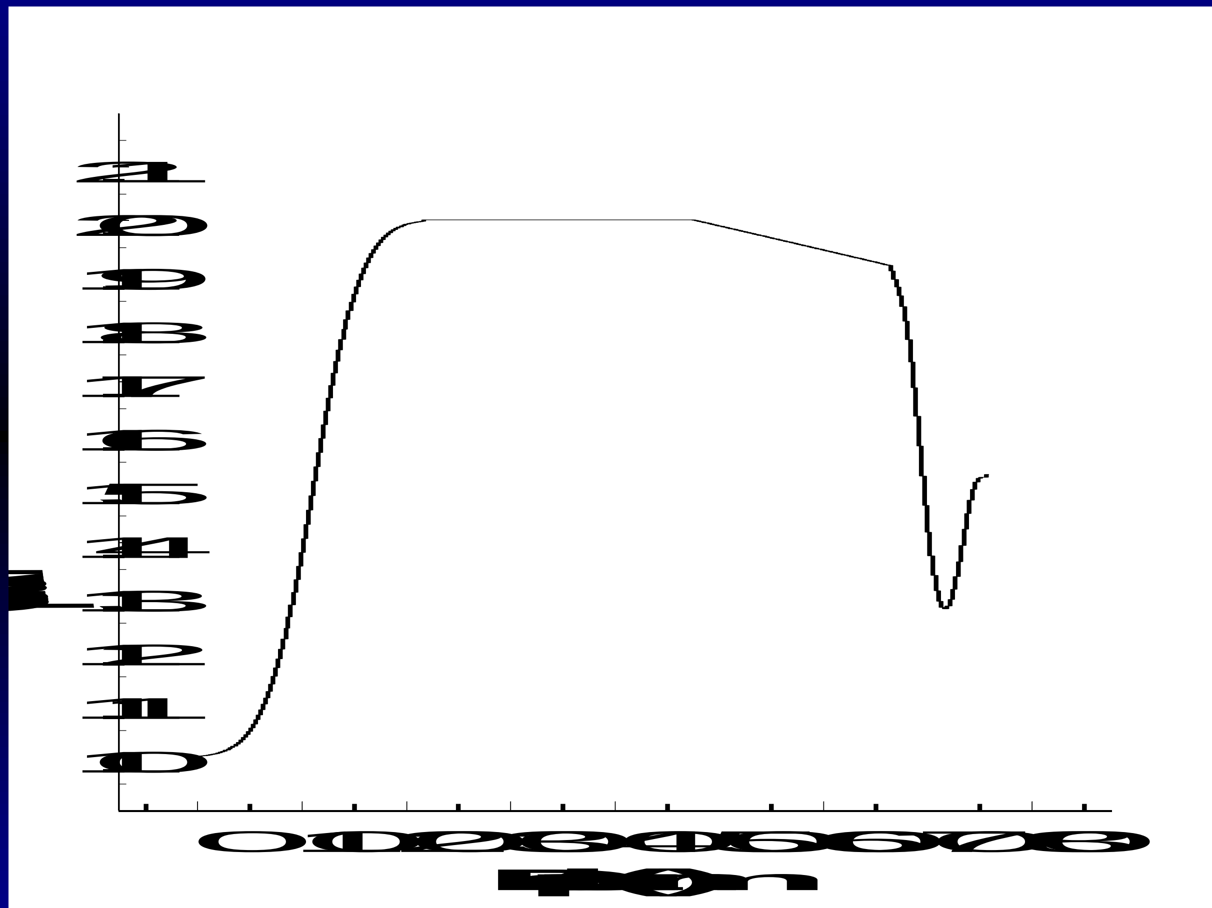
X-Ray wavelengths : $N_{material} = 1 - \delta - j\,\beta$

- ↑ determination of the microstructure = calculation of $F_v(z)$

3 techniques together

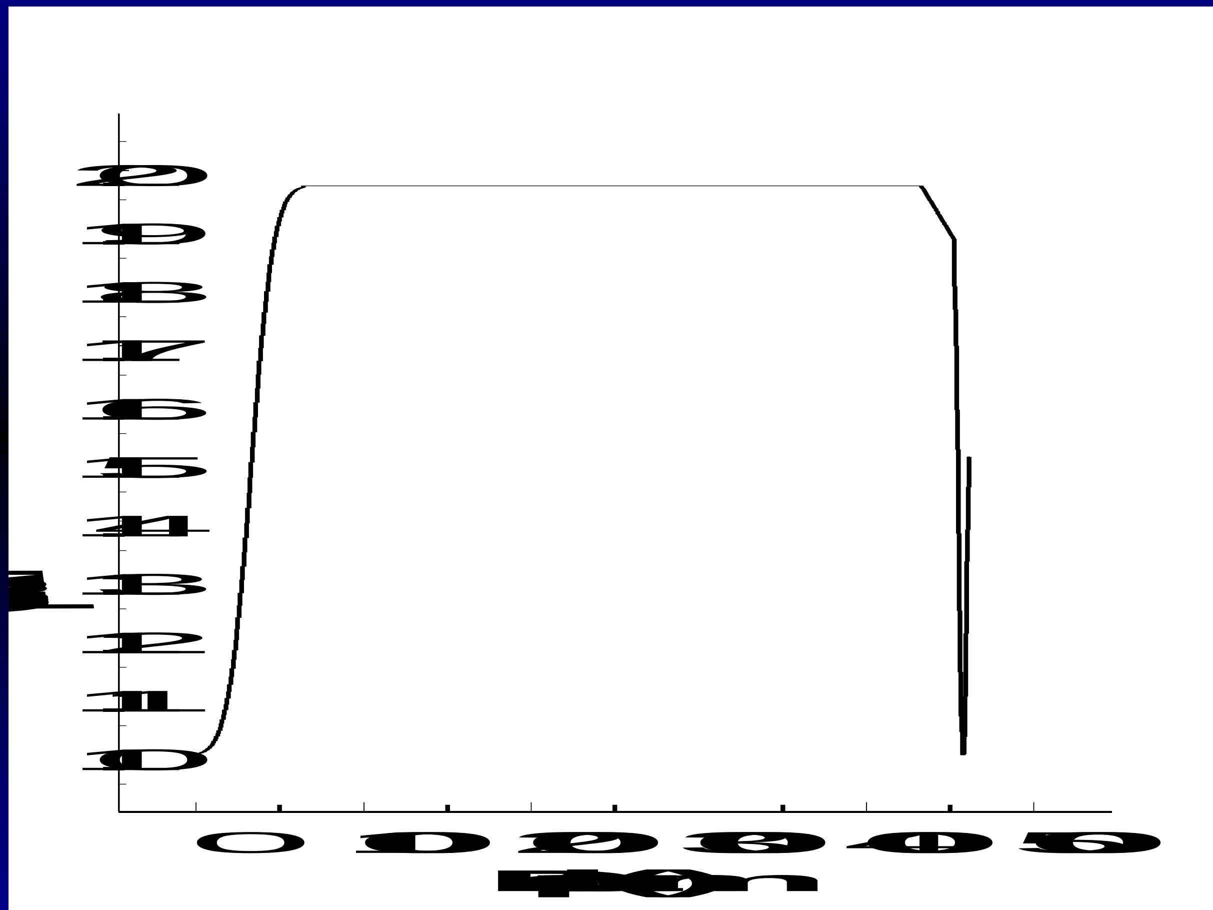
- The « new model » can be used with ellipsometric, spectrophotometric and X-Ray measurements together
- same model for all kinds of measurements :
  - film with roughness, inhomogeneity and interface
  - use of volume fraction profile $F_v(z)$
- Possibility of using the 3 techniques out of their usual range of thickness
- Application to a « thin » (75 nm) and a « thick » (460 nm) film of ZnO

3 techniques together



ZnO on glass (thin film)

3 techniques together



ZnO on glass (thick film)

Conclusions

- Optical techniques are powerfull tools for studying the microstructure of thin films
- Possibility of using the optical techniques for all kinds of films on all substrates
- Possibility of analysing several kinds of optical measurements together with a « new model » :
- ↑ use of the optical measurements out of their usual « thickness range »
- ↑ determination of the 3 features of a microstructure simultaneously