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Characterisation of the microstructure of thin films deposited on glass by optical techniques

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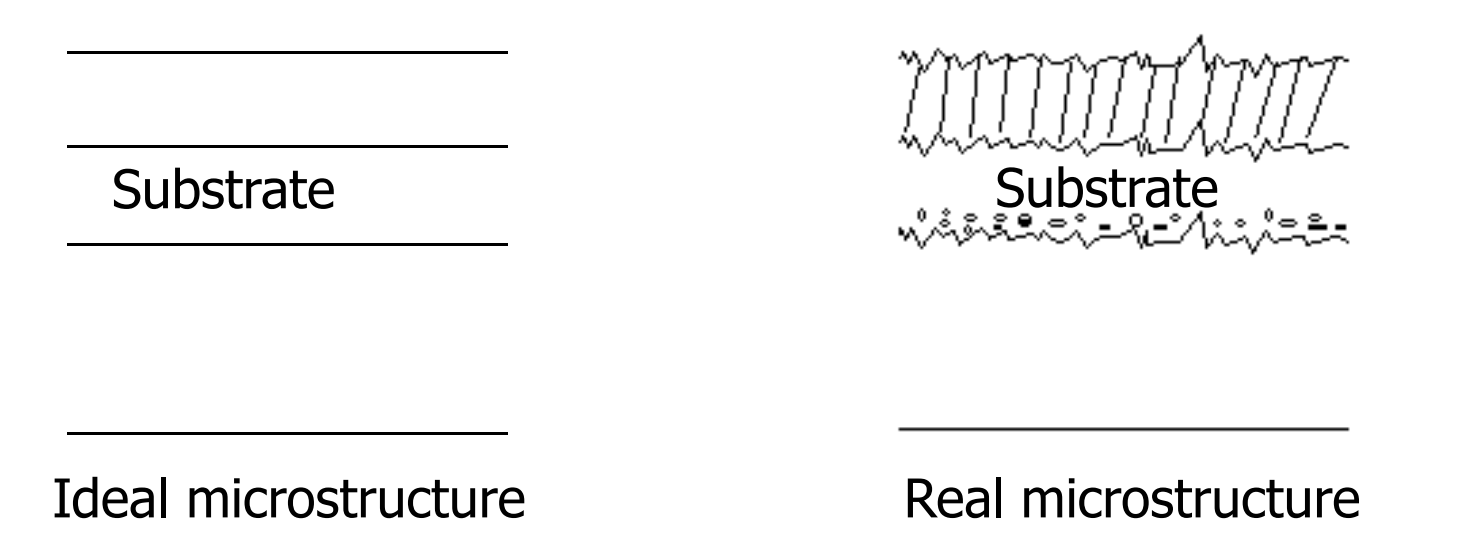
Speech contents

- Aim of the research
- Experimental techniques
- Models
- Real microstructures
- Study of the film, application to ZnO layers on glass
- Conclusions

Aim of the research

- Possibility of using optical techniques to determine the microstructure of thin films
- What is the microstructure of a thin film ?



- Causes : variation of the compacity (voids), of the crystalline structure, of the roughness and/or of the material composition of the film

Aim of the research

- Why studying the microstructure of thin films ?
- Thin films are used in many applications, e.g. :

CDs

Packaging

Glass

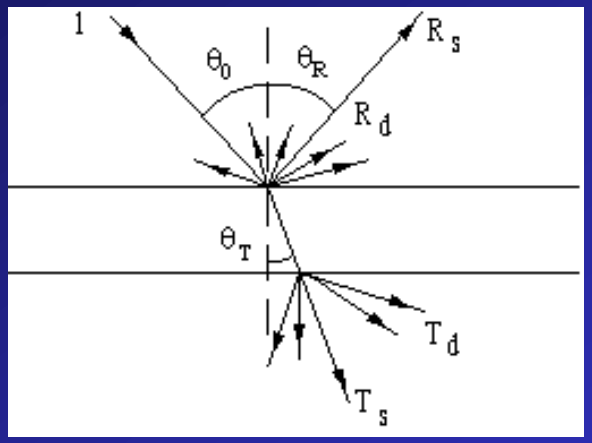
- Their microstructure influences their final properties, e.g. roughness in antireflective coatings

Aim of the research

- 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 used in this research : Spectrophotometry, X-Ray reflectometry, Ellipsometry

Experimental techniques

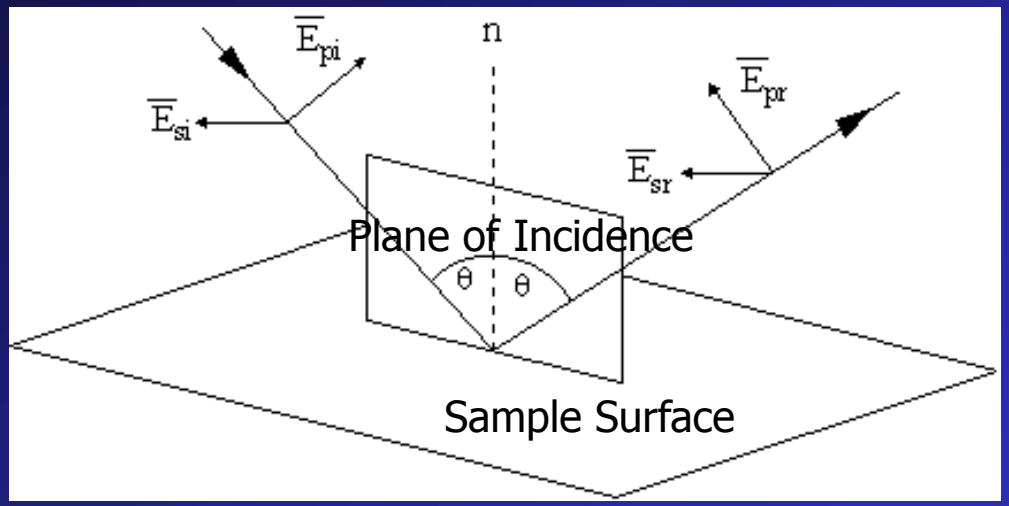
- Measurement of specular and scattered reflectivity and transmitivity of light : R_S T_S R_D T_D



- 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

Experimental techniques

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



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

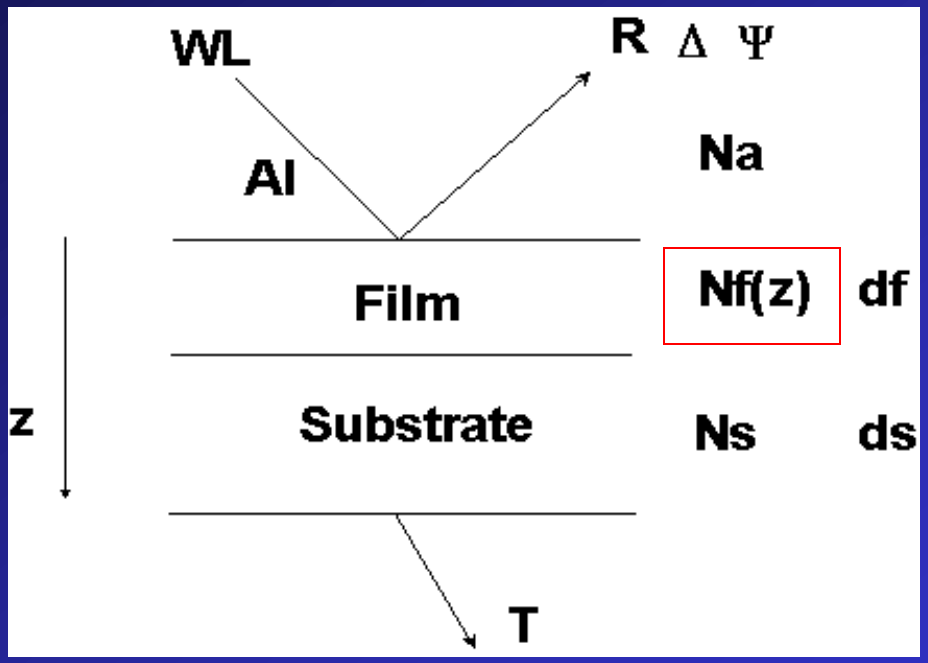
$$R_s = E_{sr} / E_{si}$$

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

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

Models

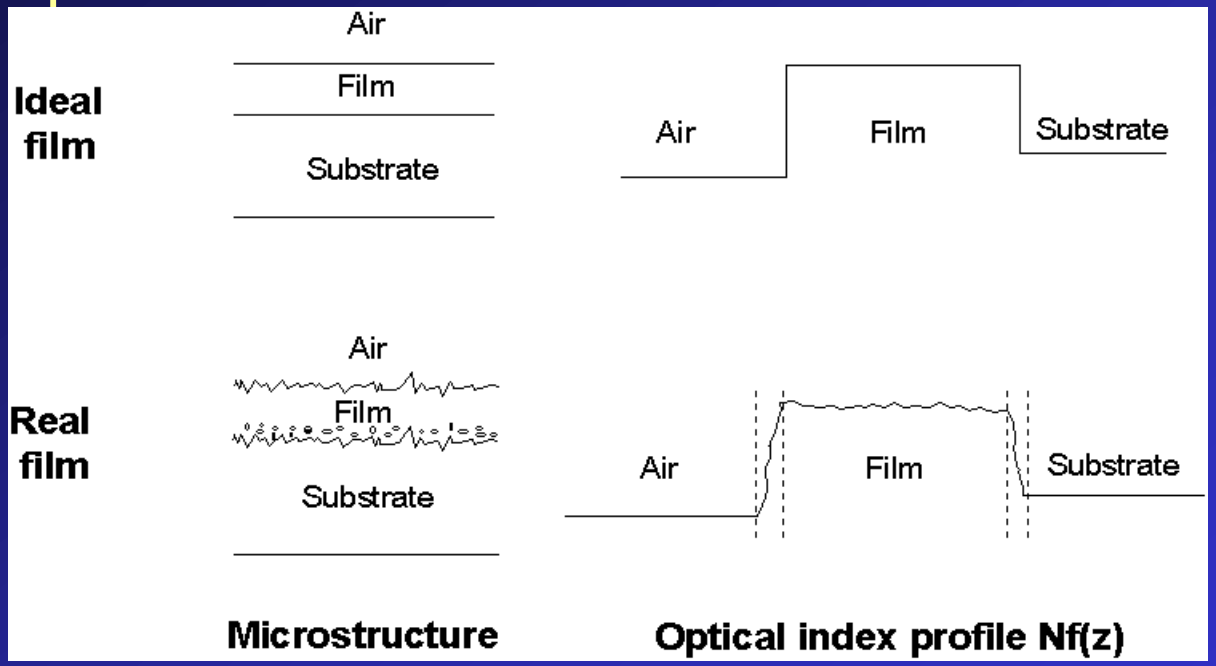
- Optical measurements depend on : experimental parameters and physical parameters of a film



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

Models

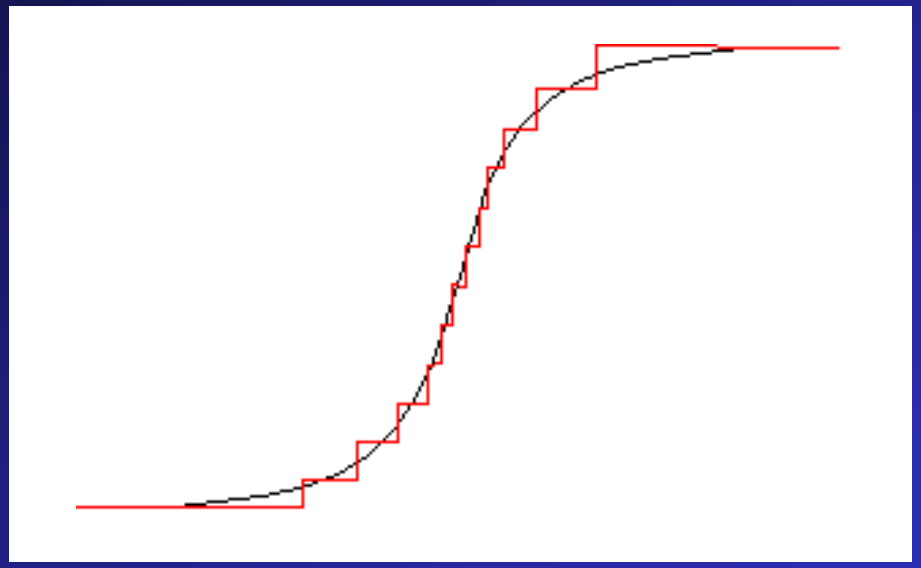
- Relation between the microstructure and the optical index profile of a thin film



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

Models

- How to model an optical index profile ?



- Optical index profile described by a packing of homogeneous sublayers which follows the profile
- Optical properties R, T, Δ and Ψ then easily computed by Fresnel equations

Models

- How to calculate the optical index profile $N_F(z)$ of a thin film ?
- 1) create a mathematical model with adjustable parameters to describe $N_F(z)$
- 2) compute R, T, Δ and Ψ for starting values of the parameters of the model
- 3) compare the computed values with experimental values of R, T, Δ and Ψ measured on a sample and adjust the parameters in order to minimize the differences thanks to a χ^2 method

Models

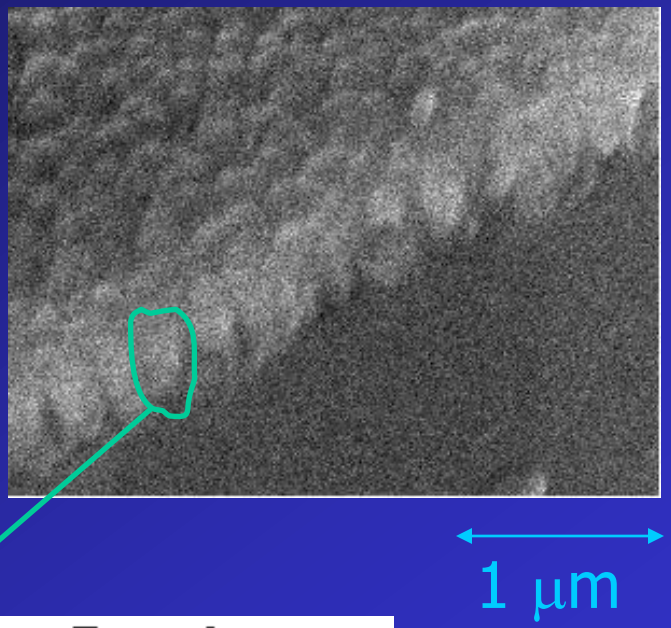
- The optical index profile depends on the wavelength wl
- ↑ creation of a « new model » based on a volume fraction of material profile $F_v(z)$ not dependant on wl
- ↑ $N_F(z, wl) = f(N_{material}(wl), F_v(z))$

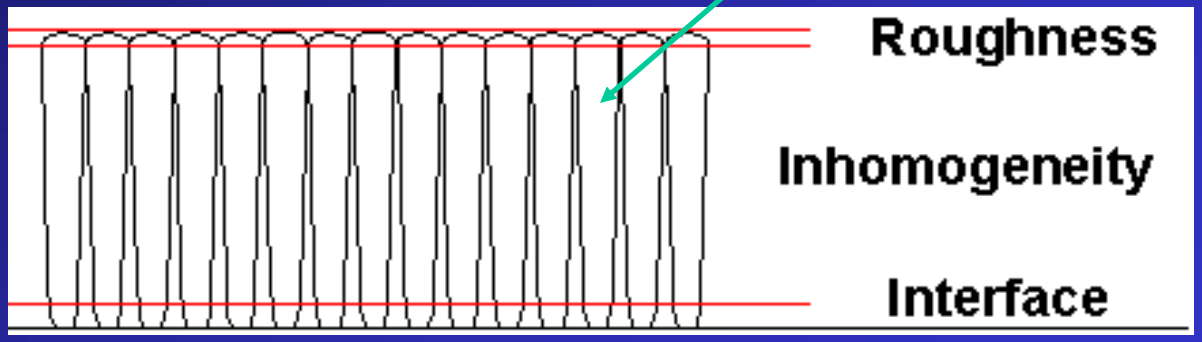
  Visible wavelengths : $N_{material} = n - j\,k$

  X-Ray wavelengths : $N_{material} = 1 - \delta - j\,\beta$
- ↑ determination of the microstructure = calculation of $F_v(z)$

Real microstructures

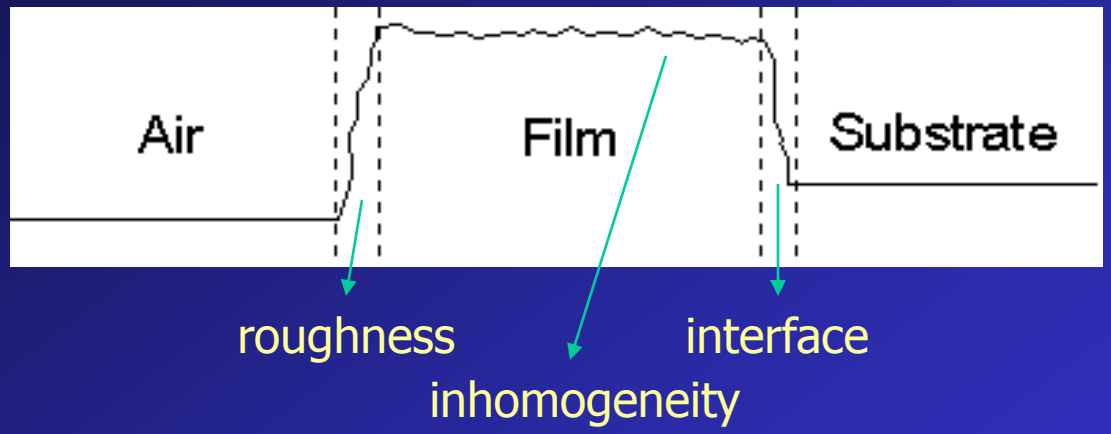
- Studies by scanning electron microscopy (SEM) show that in real microstructures, we observe three different features in the optical index profile : inhomogeneity, roughness and interface :





Real microstructures

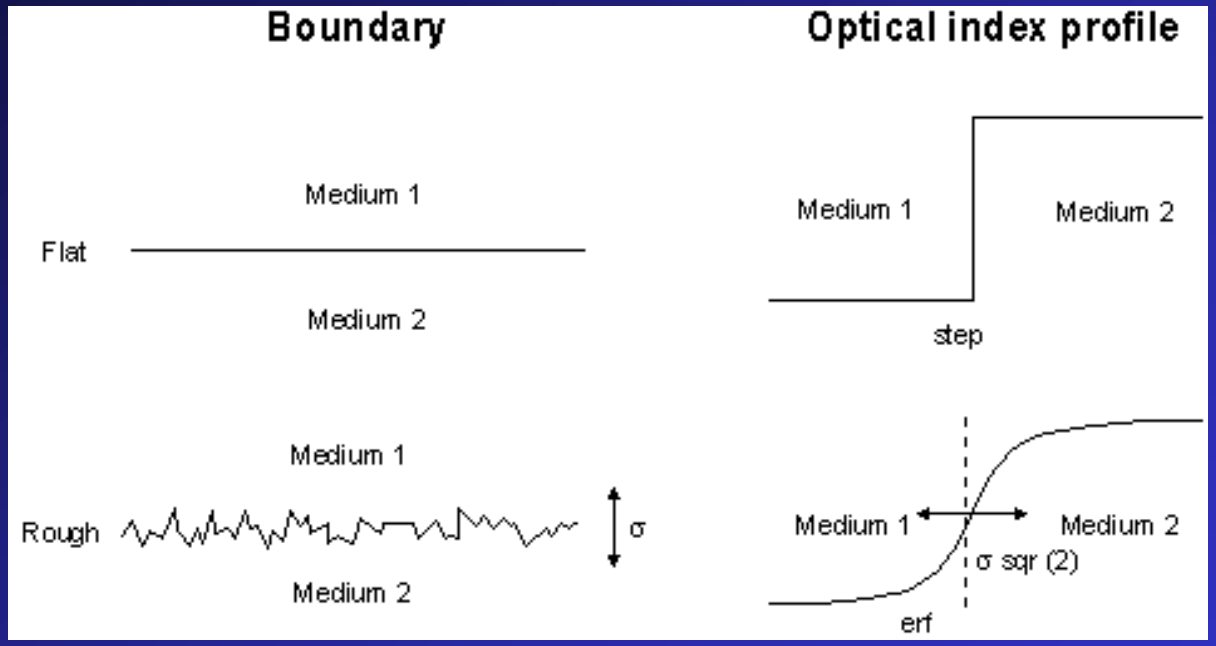
- In the optical index profile, these features are also present :



- Roughness and interface : rapid variation of the optical index at the boundaries
- Inhomogeneity : slight variation of the optical index along the thickness of the film

Real microstructures

- How to describe roughness ?



- Roughness described by its rms value σ
- Optical index profile modeled by the error function erf (z, σ)

Study of ZnO films

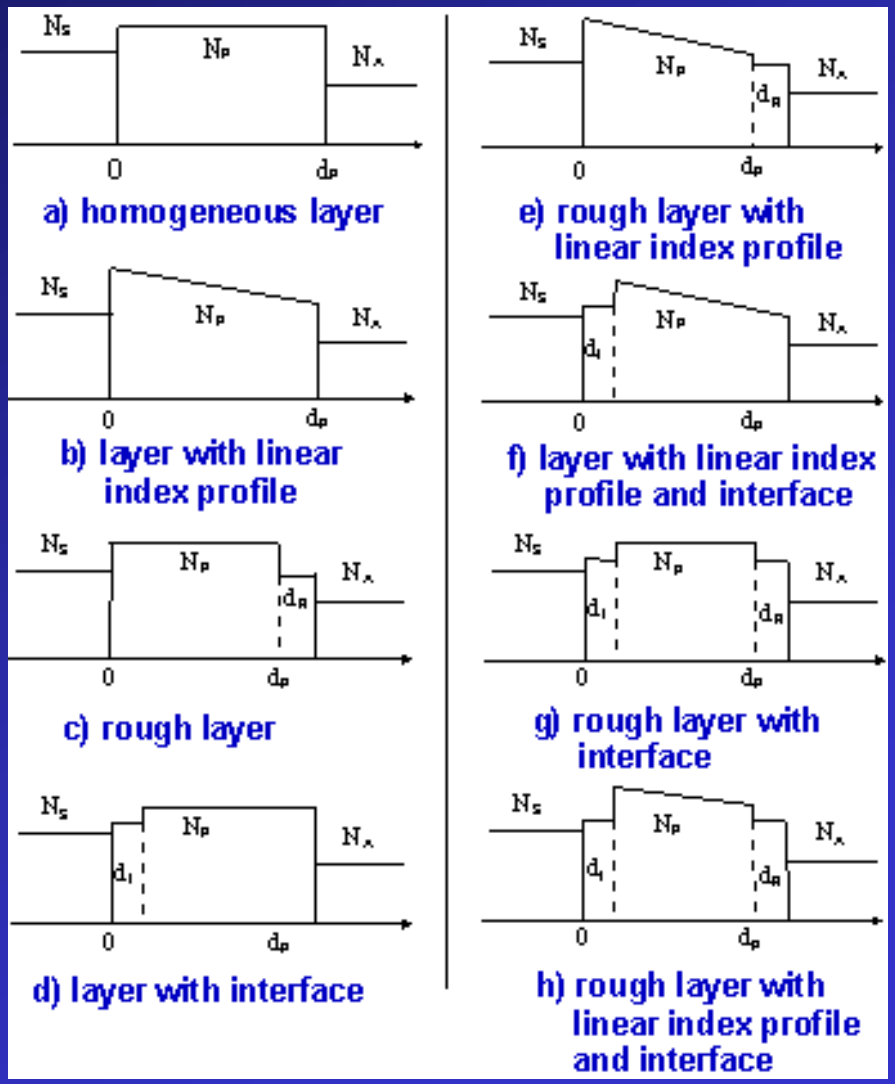
- Determination of the volume fraction profile $F_v(z)$ of ZnO thin films

- Substrate : Corning 7059 glass
- Film : r.f. sputtered ZnO

- Why ZnO ?
  - not well known
  - transparent electric conductor

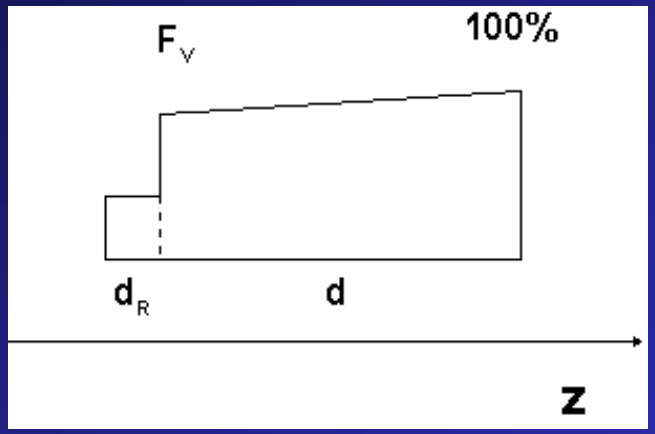
Study of ZnO films

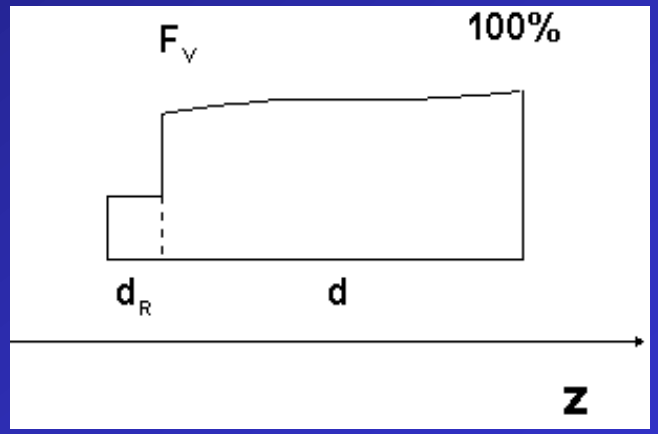
- Step 1 : use of state of-the-art models in spectrophotometry and ellipsometry
- Spectrophotometry : models b-c-e-f-g-h are good ↑ not very sensitive to the microstructure
- Ellipsometry : models e and h good ↑ not very sensitive to « interfaces »



Study of ZnO films

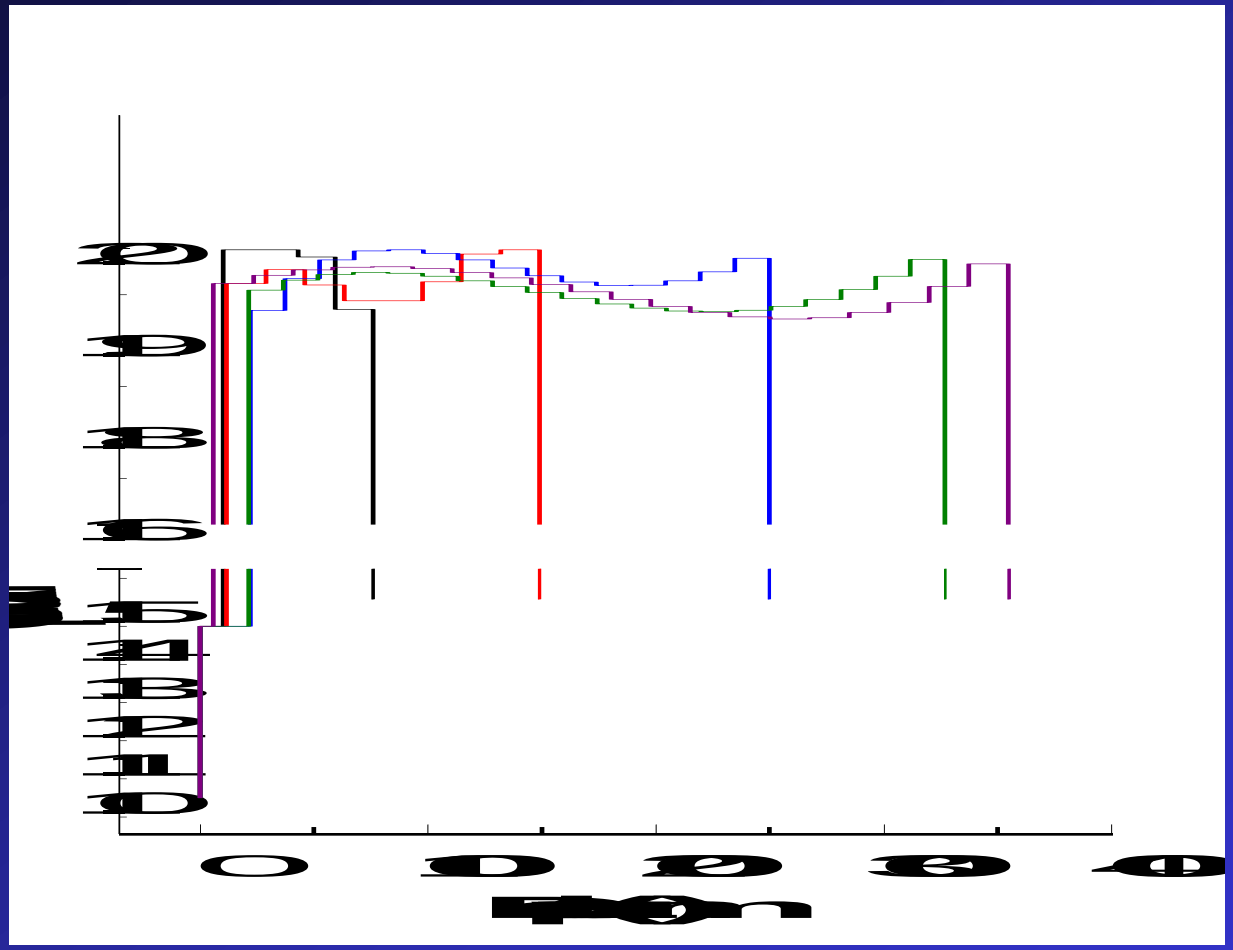
- Step 2 : use of improved models in ellipsometry
- linear index profile replaced by polynomial index profile, with Chebyshev polynomials





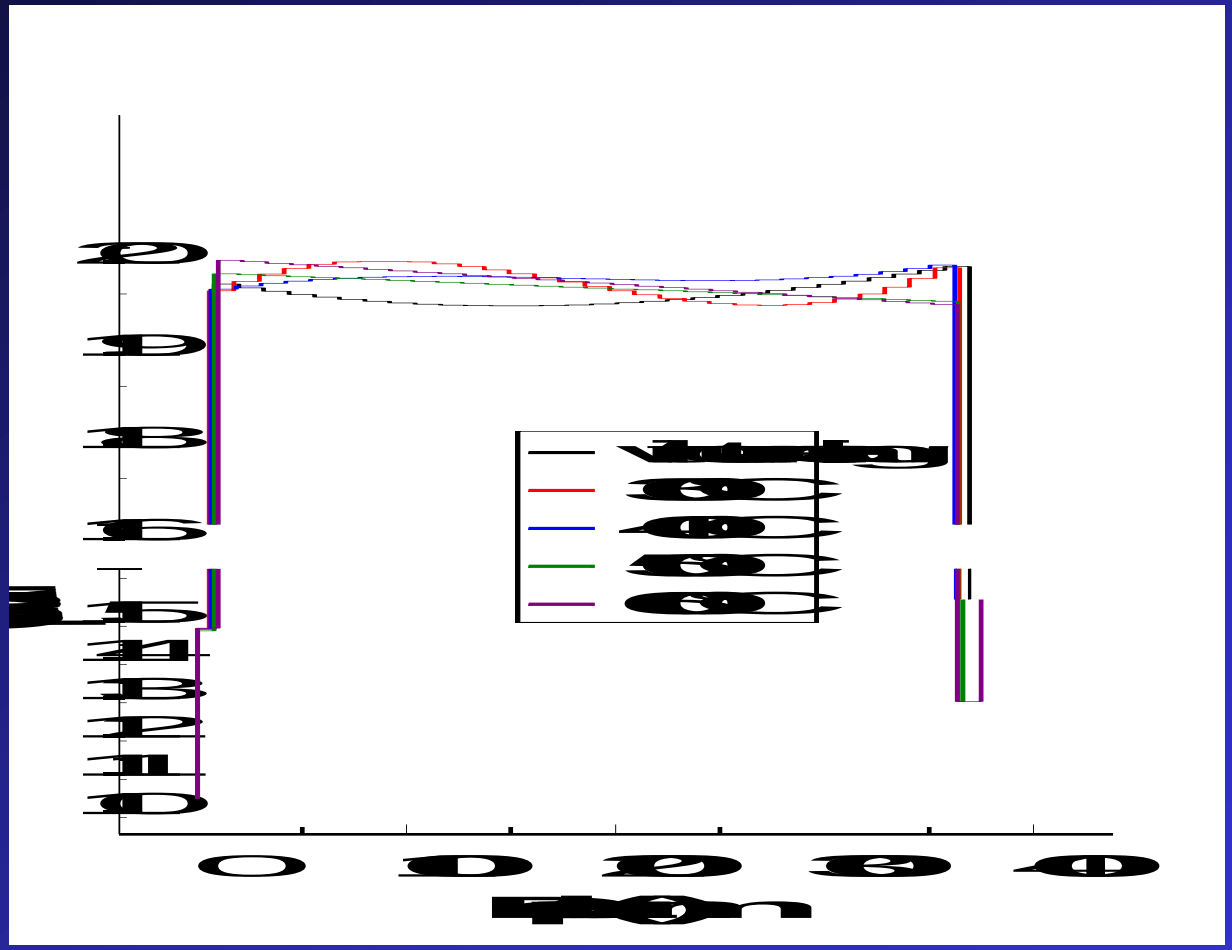
- Application to multisample analysis and to the analysis of annealing of ZnO layers

Study of ZnO films



Multisample analysis

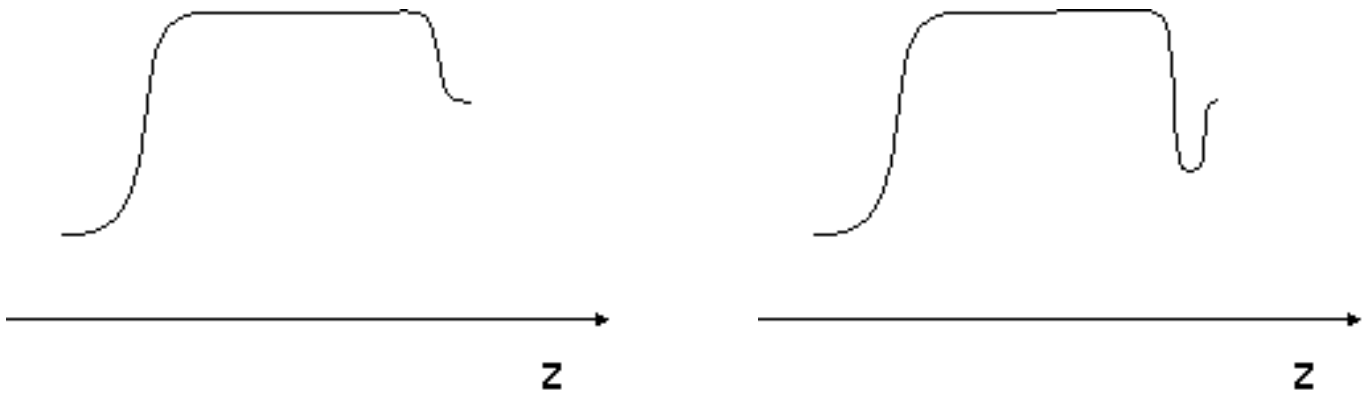
Study of ZnO films



Annealing of a ZnO layer

Study of ZnO films

- Step 3 : use of state of-the-art models in X-Ray reflectometry

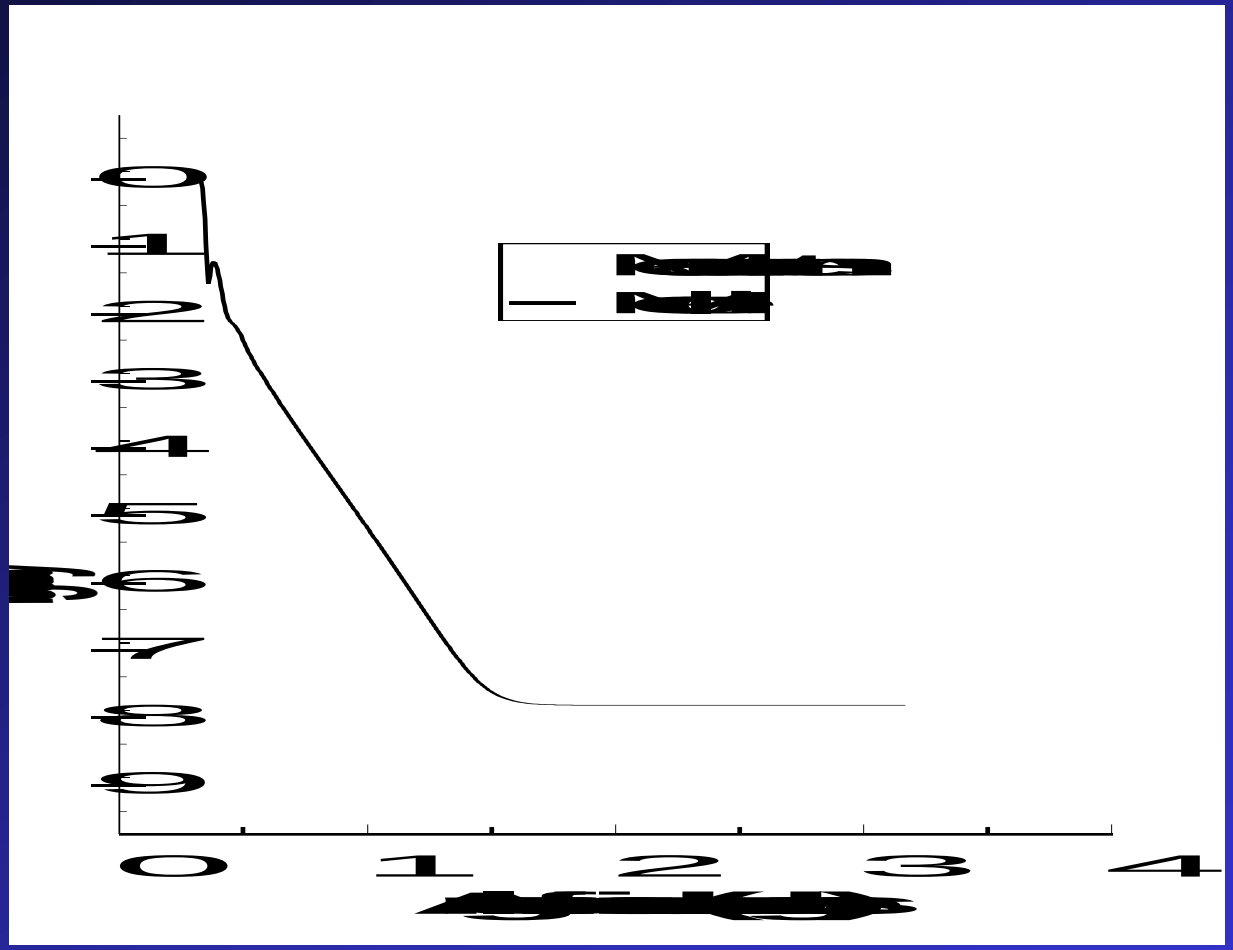


a) rough layer with « standard » interface

b) rough layer with porosity

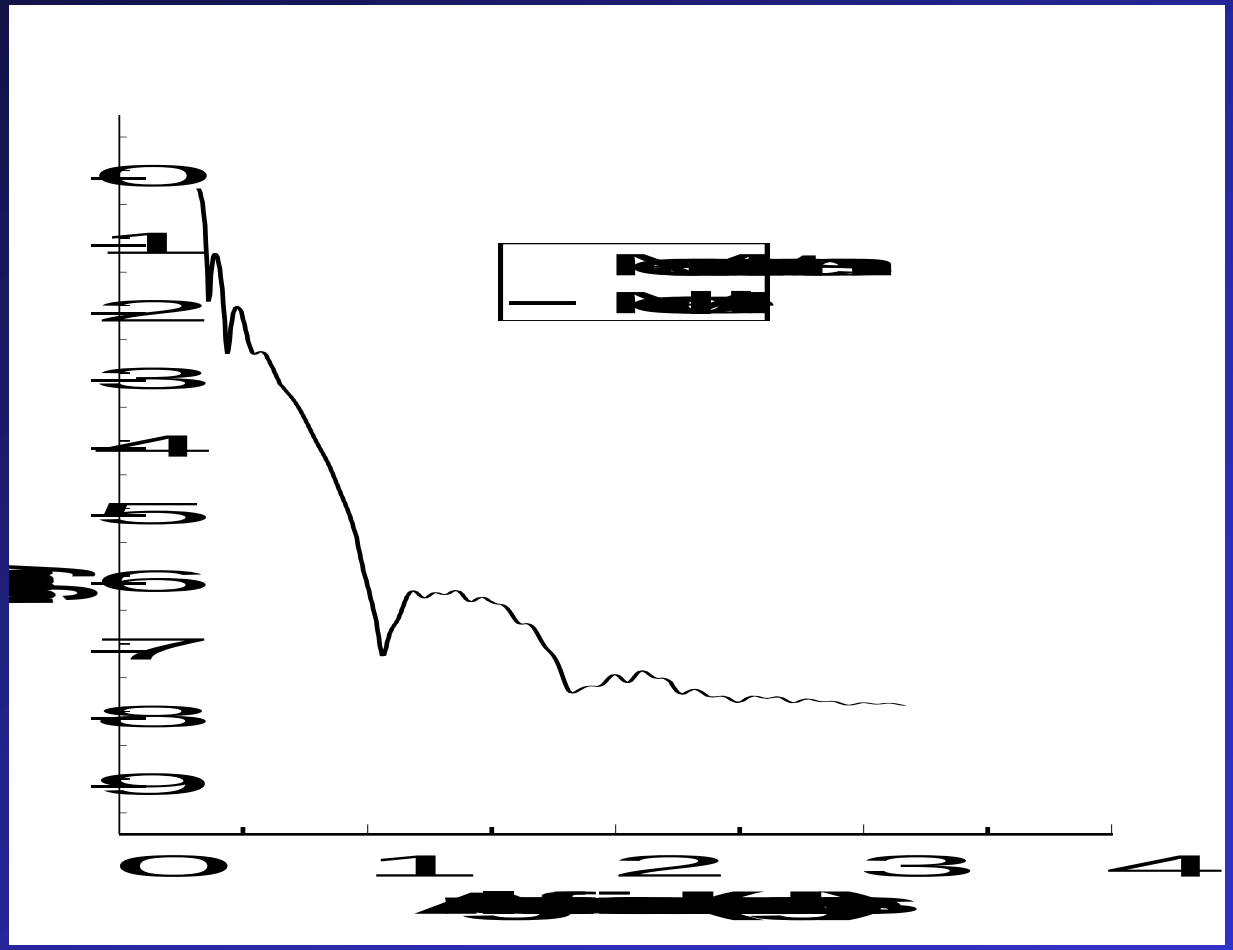
- Model b is the best ↑ X-Ray reflectometry sensitive to roughness and to the presence of « interfaces »

Study of ZnO films



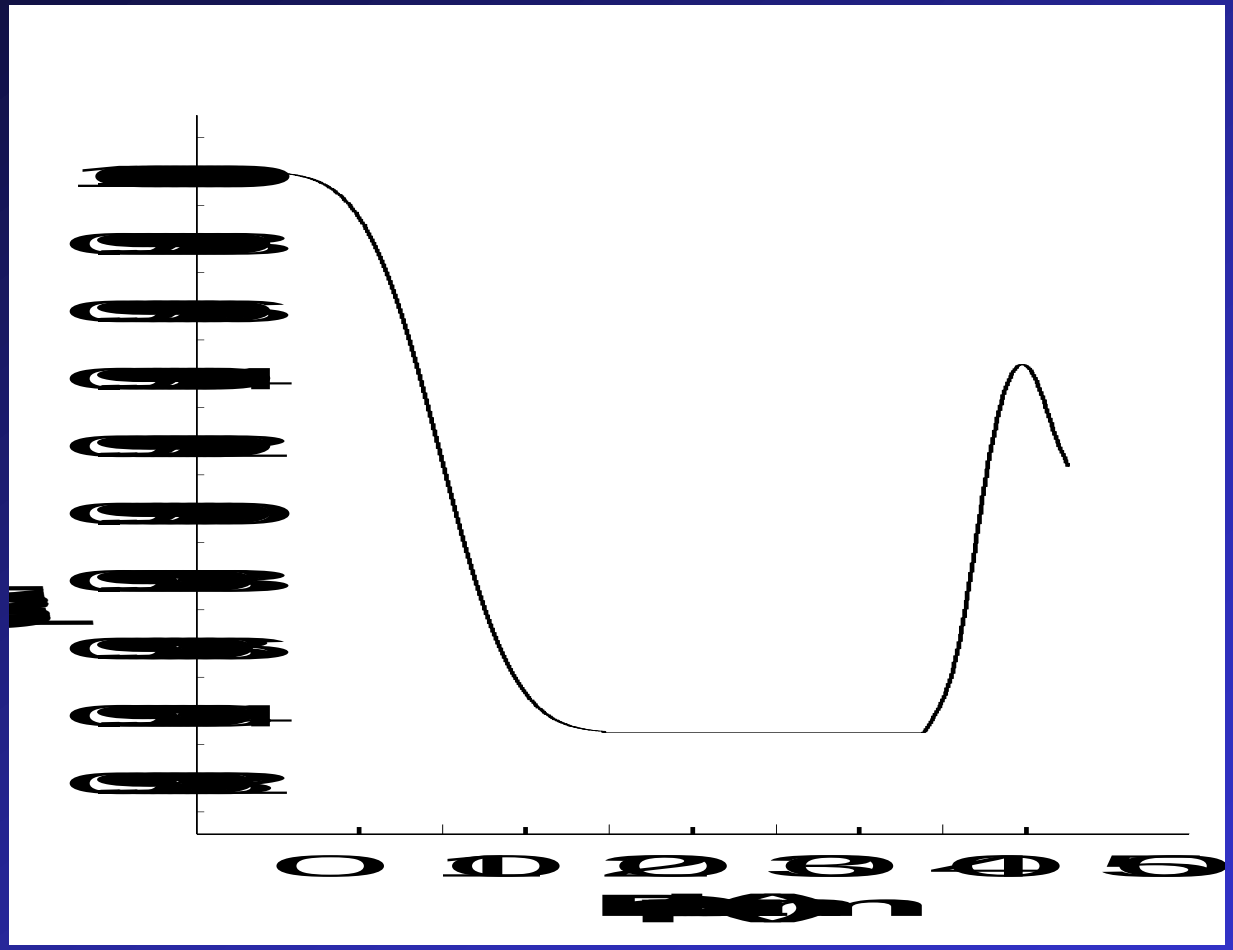
Model with « standard » interface

Study of ZnO films



Model with porosity

Study of ZnO films

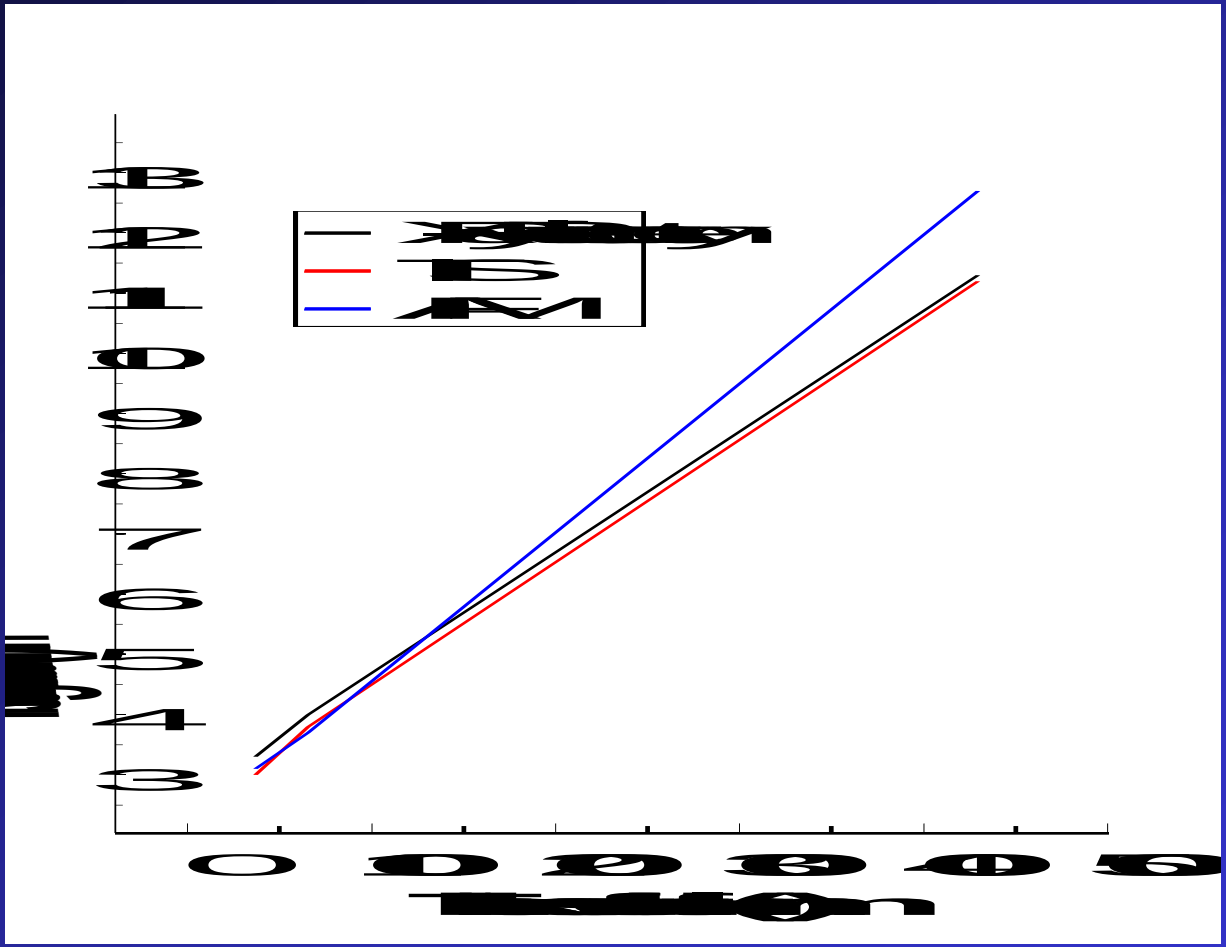


Index profile for the model with porosity

Study of ZnO films

- Use of other techniques to validate the value of the rms roughness σ obtained with X-Ray reflectometry measurements :
- Measurement of the scattered reflectivity R_D and use of the TIS (Total Integrated Scattering) theory
- Measurement by AFM (Atomic Force Microscopy)
- ↑ Same values of σ obtained by the 3 techniques

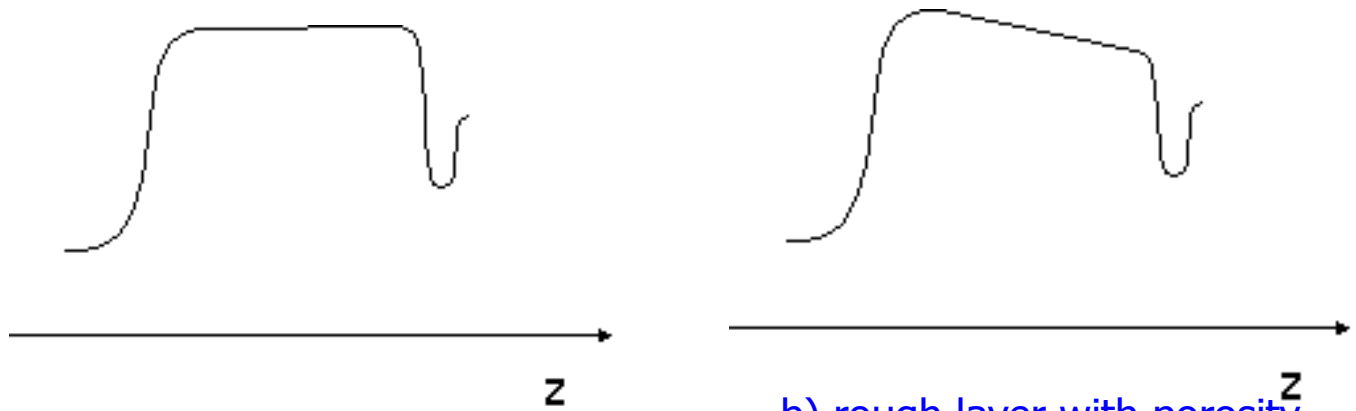
Study of ZnO films



Roughness of films measured with different techniques

Study of ZnO films

- Step 4 : use of improved models in X-Ray reflectometry



a) rough layer with porosity

b) rough layer with porosity and linear index profile

- Models a and b are good ↑ X-Ray Reflectometry not very sensitive to inhomogeneity

Study of ZnO films

- 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

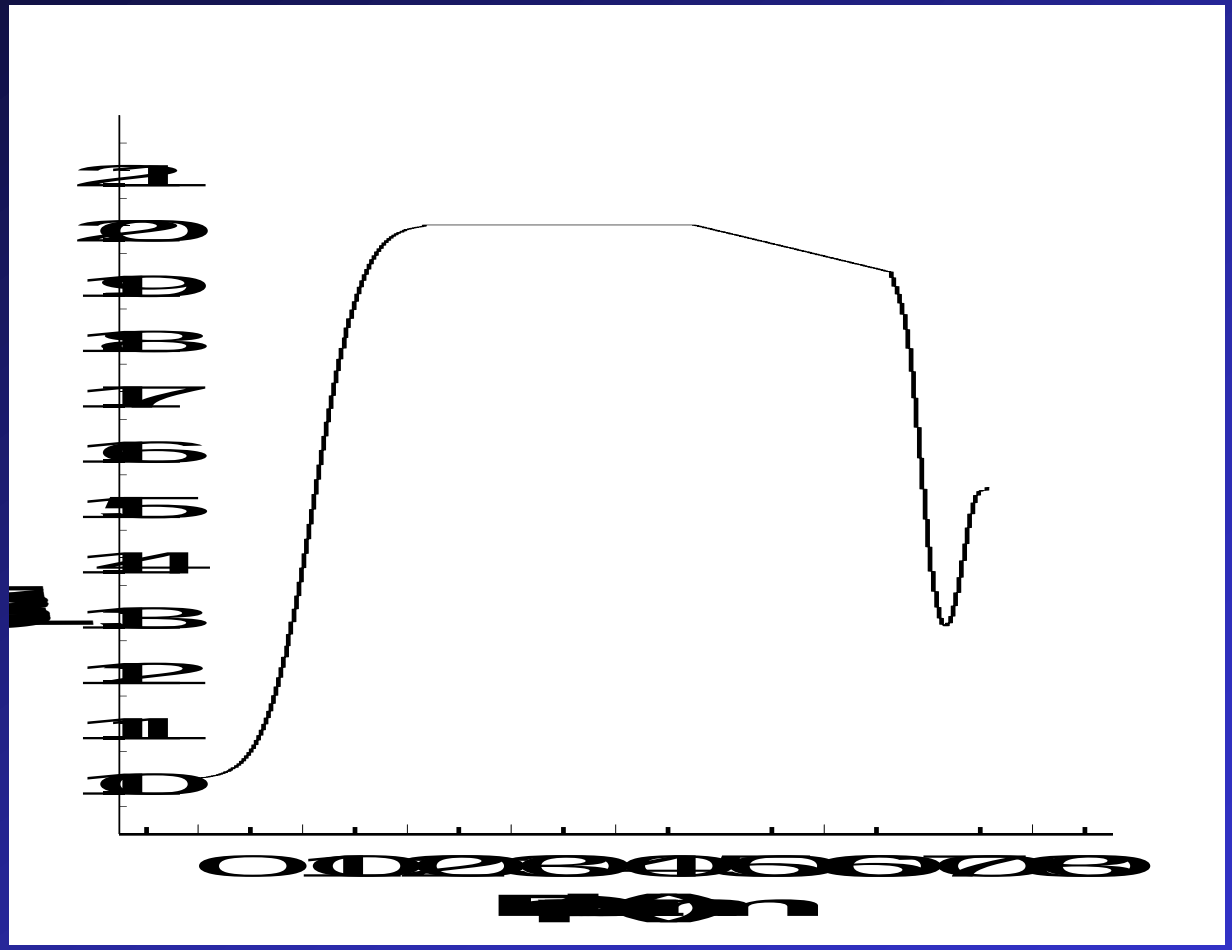
Study of ZnO films

- 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

Study of ZnO films

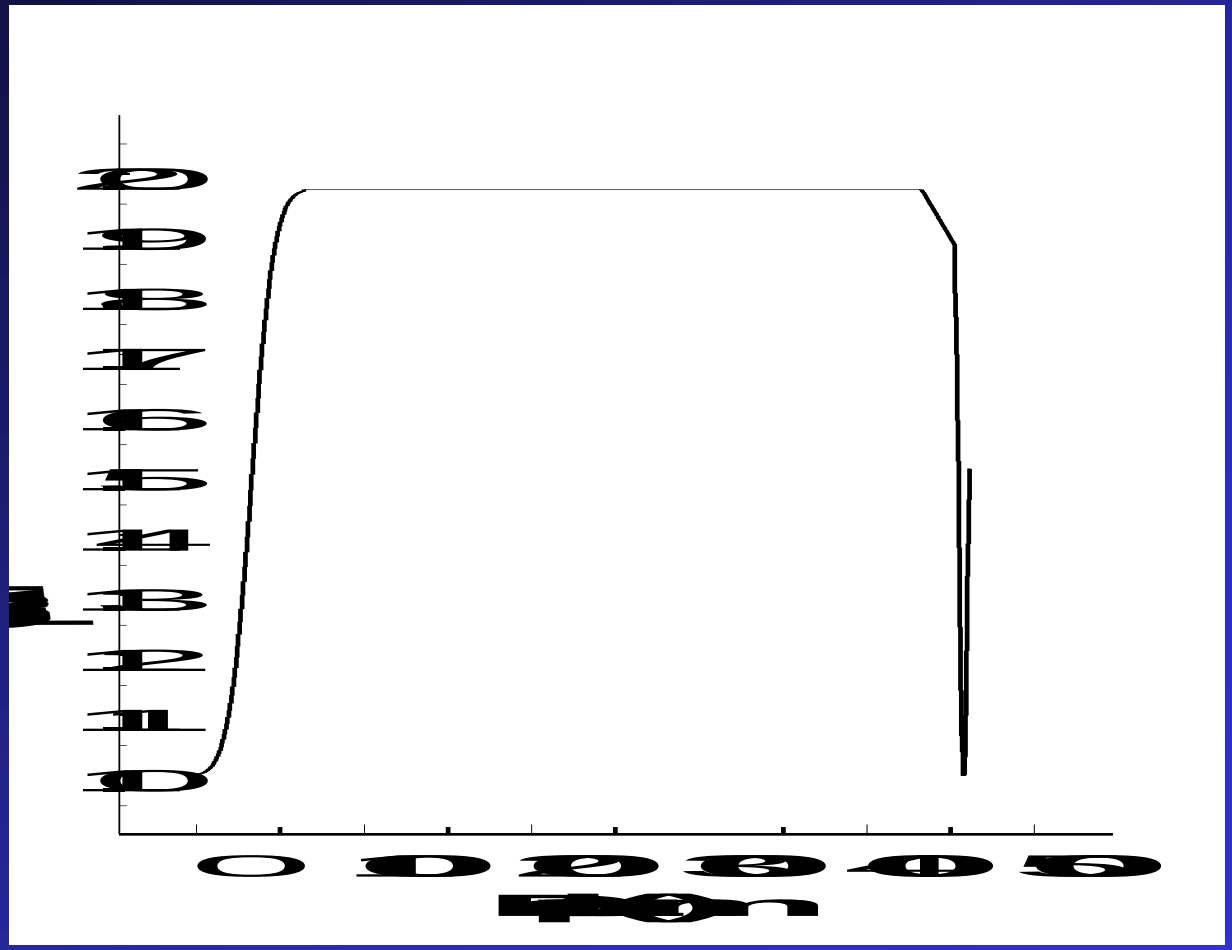
- Step 5 : « new model » used with ellipsometric, spectrophotometric and X-Ray measurements together
- same model for all kinds of measurements :
  - film with roughness, inhomogeneity and interface
  - use of volume fraction profile $F_v(z)$
- Possibility of using the 3 techniques out of their usual range of thickness
- Application to a « thin » (75 nm) and a « thick » (460 nm) film

Study of ZnO films



Thin film

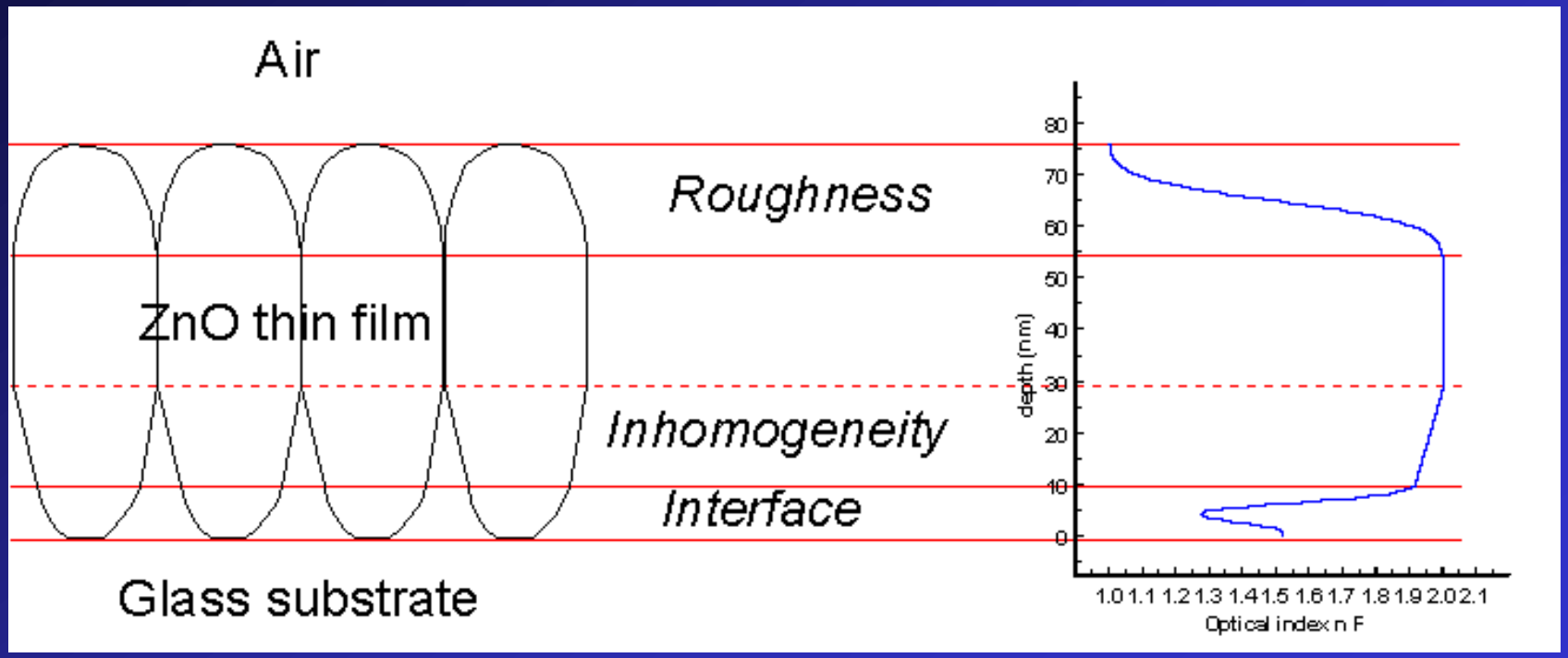
Study of ZnO films



Thick film

Study of ZnO films

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



Conclusions

- Creation of a software able to analyse 3 kinds of optical measurements (spectrophotometry, ellipsometry and X-Ray reflectometry) for the determination of the microstructure of thin films
- Study of the sensitivity of the 3 optical techniques to the various features (roughness, inhomogeneity, interface) of a real microstructure : each technique has its own field of application
- Improvement of the State of-the-art models for the 3 optical techniques

Conclusions

- Creation of a « new model » able to analyse all kinds of optical measurements together
- The « new model » has improved possibilities :
- ↑ possibility to use the optical measurements out of their usual « thickness range »
- ↑ possibility to determine the 3 features of a microstructure simultaneously
- Possibility to use the optical techniques for all kinds of films on all substrates