



MAX-PLANCK-GESELLSCHAFT

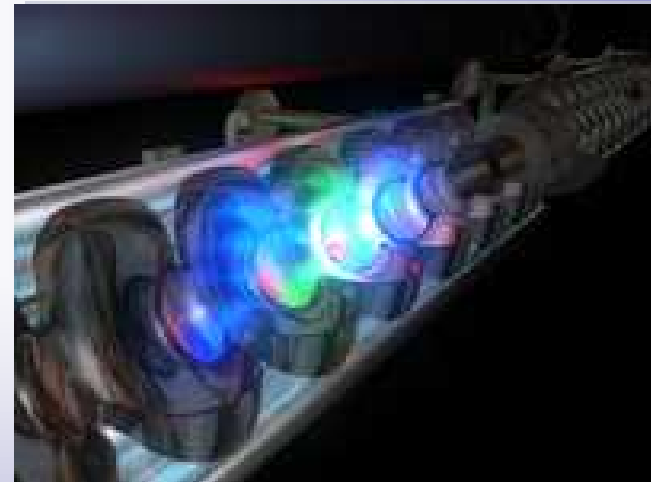
MAX-PLANCK-INSTITUT FÜR METALLFORSCHUNG  
Abteilung Dosch



M. Delheusy

## SXRD IN-SITU INVESTIGATION OF THE O/Nb(110) INTERFACE

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dapnia  
SACM

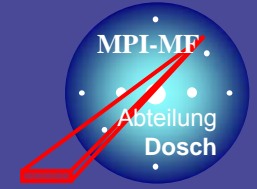
cea

saclay

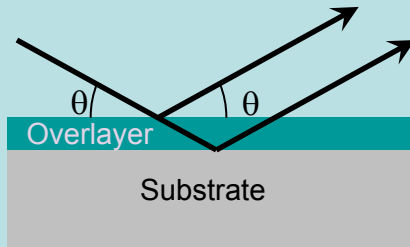
Département d'Astrophysique, de physique des Particules, de physique Nucléaire  
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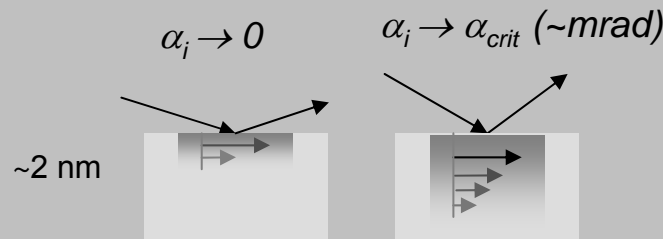
# 3 complementary Surface X-ray techniques



## X-ray Reflectivity

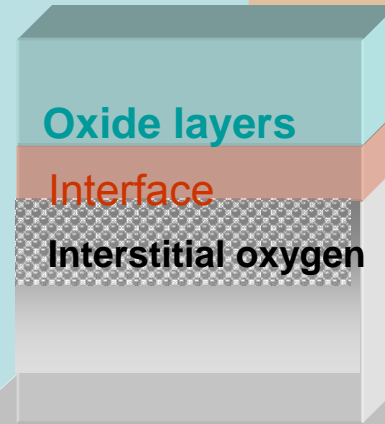


## Evolution of the oxide layers

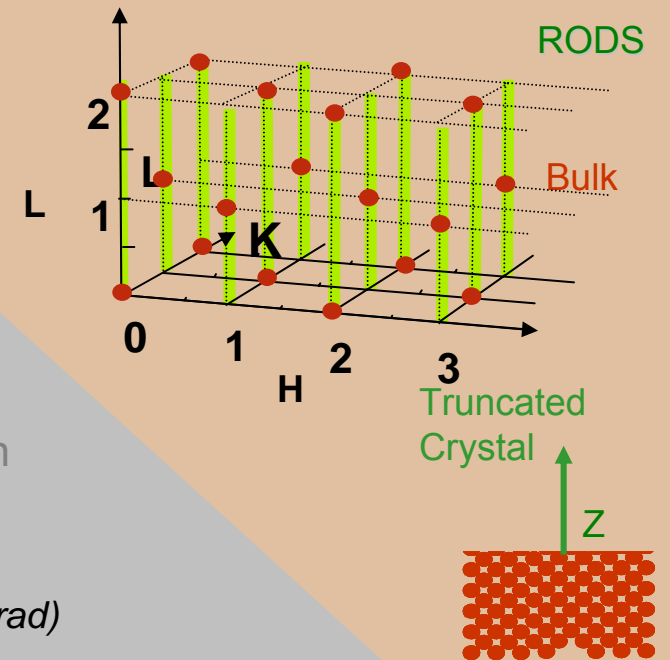


Depth-resolved diffuse scattering induced by interstitial oxygen

Depth-distribution evolution of the interstitial oxygen

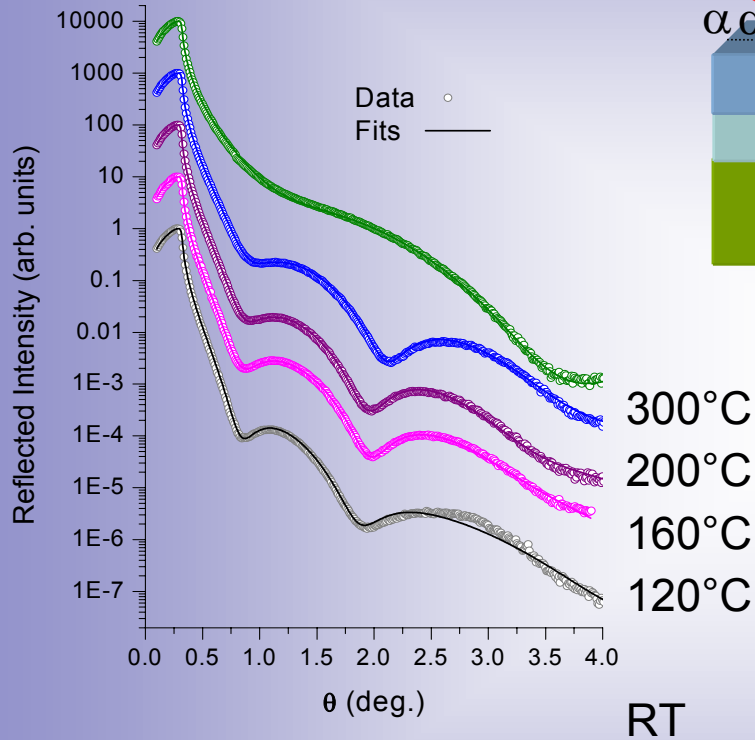
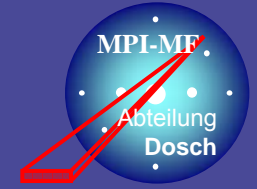


## Crystal Truncation Rods (CTR)

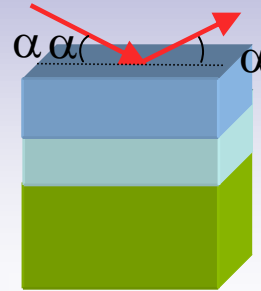




# X-Ray Reflectivity



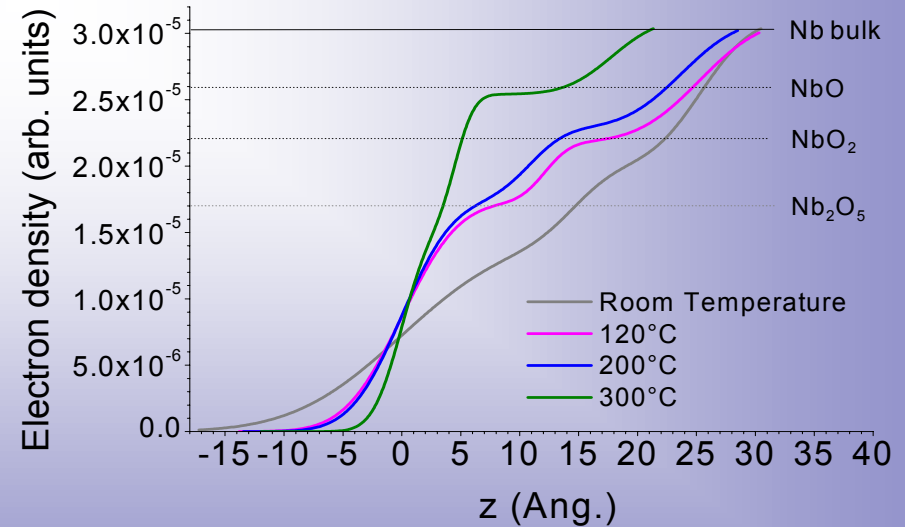
Nb(100) oxidized 14 days in air



## Optical properties of the material

Parrat<sup>1</sup> formalism layers model :

- Electron density profiles
- Thickness
- Roughness



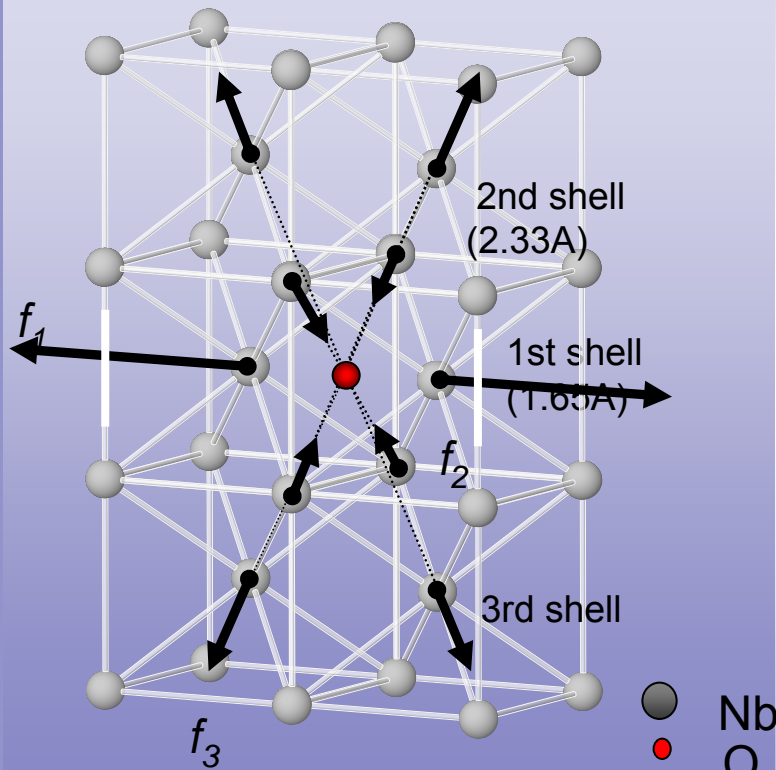
<sup>1</sup>Formalisme de L.G. Parrat, *Phys. Rev.* **95**, 359 (1954)



# DIFFUSE SCATTERING IN THE NEAR SURFACE REGION



## Previous study on Nb bulk

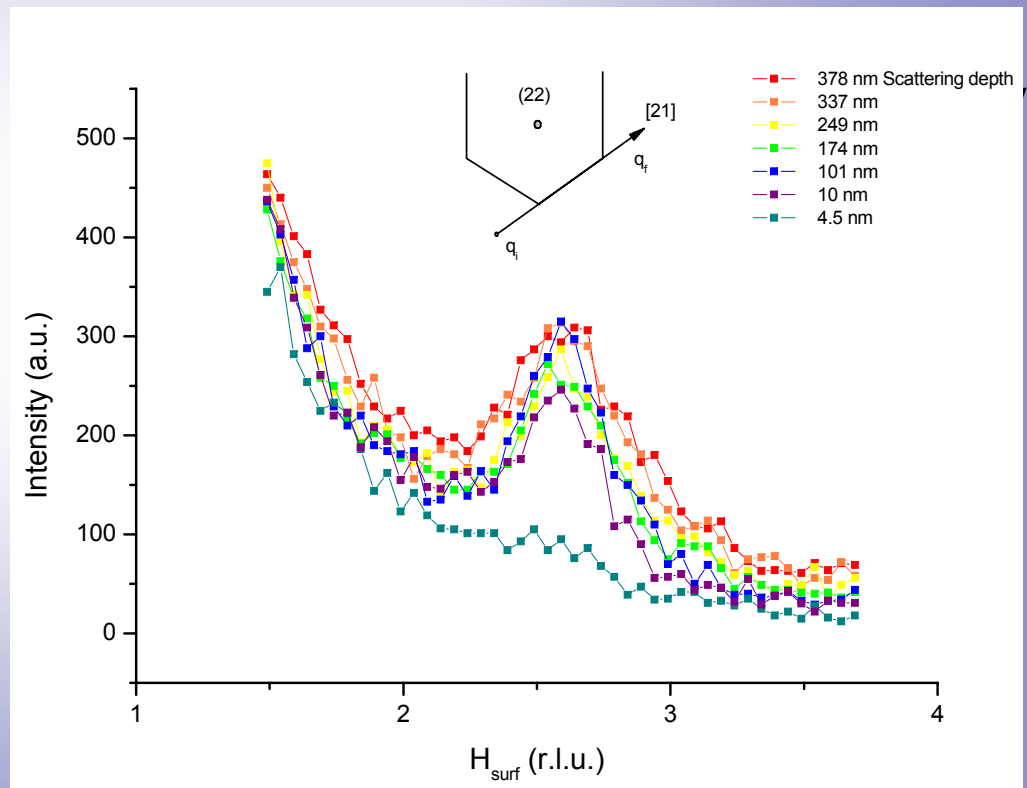


Interstitial O atom on octahedral site

## Near Surface region :

Reciprocal map for  $\alpha_i: 0.6^\circ$ ,  $\alpha_f: 0.5^\circ$  ( $\Lambda \sim 150\text{nm}$ )

Nb(110) oxidize in air (atm. conditions)



<sup>1</sup>H. Dosch et al., Phys. Rev. B, vol. 34 n°3, 1986

<sup>2</sup>J.M. Rowe and A. Magerl, Phys. Rev. B 21(4) (1980), 1706

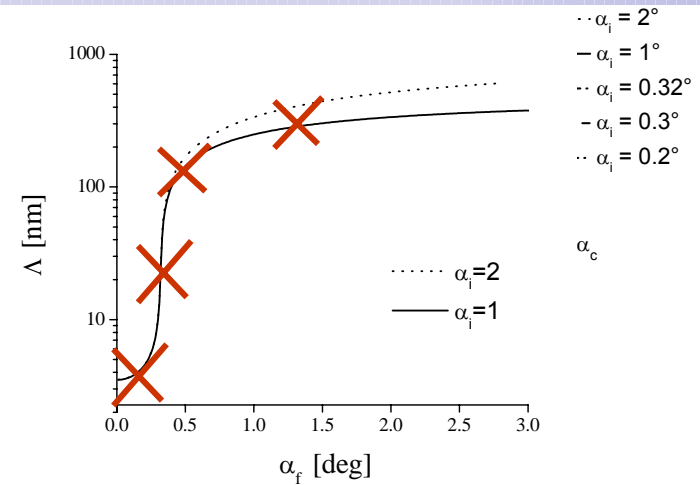
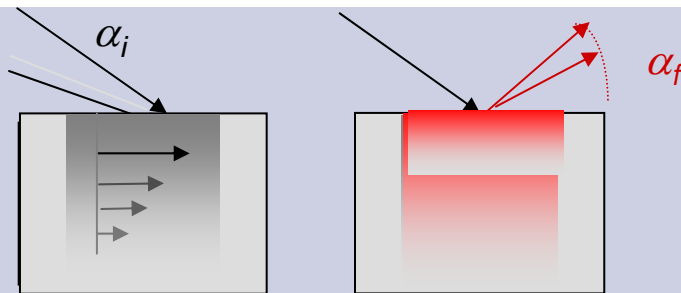
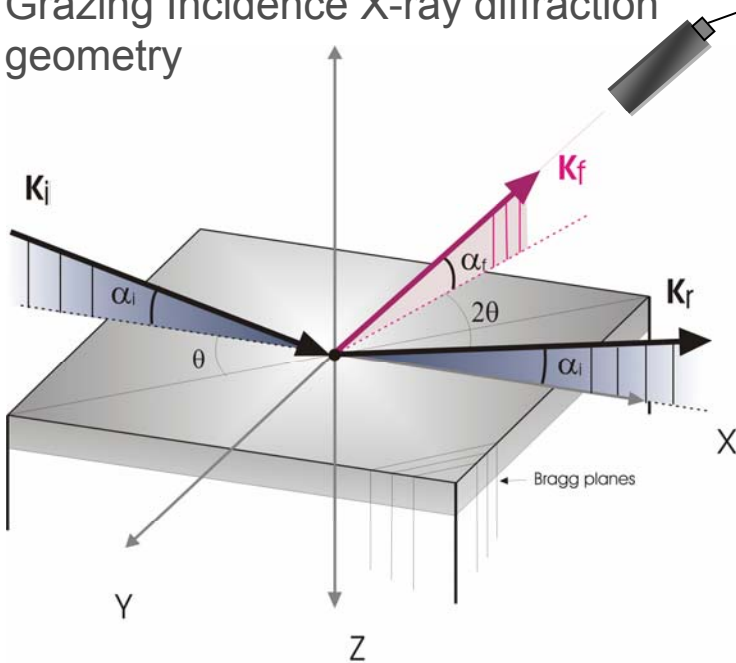


# DEPTH-SENSITIVE SURFACE STUDY



M. Delheusy

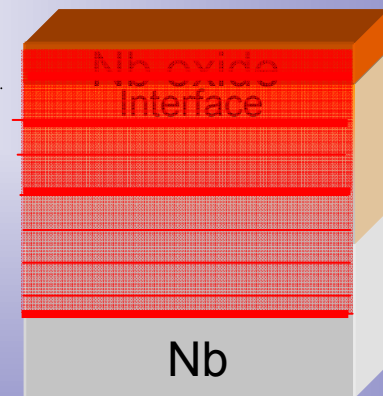
## Grazing Incidence X-ray diffraction geometry



Scattering depth  $\Lambda = f(\alpha_f, \alpha_i)$

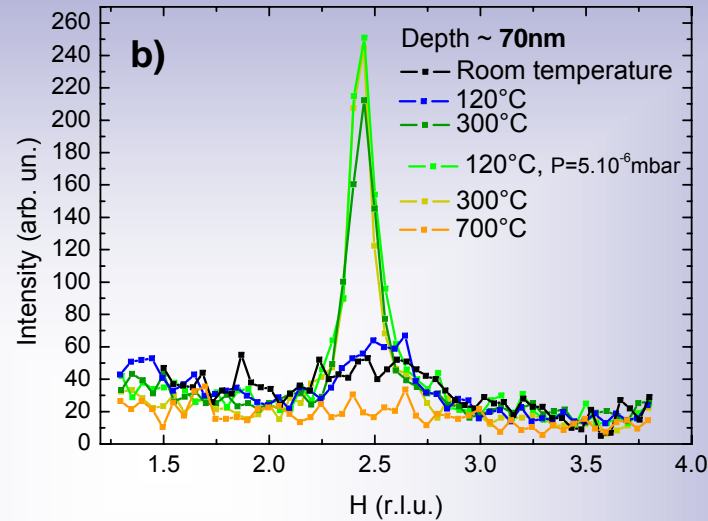
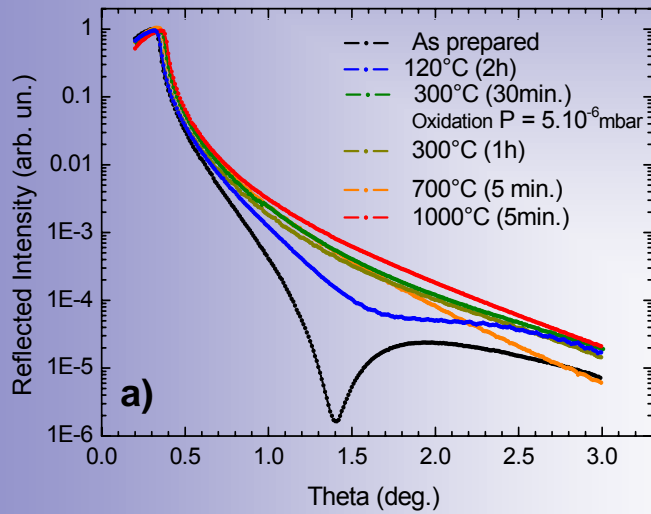
nm resolution

- ~ 5nm
- 10nm
- 50-100nm
- 200nm
- 400nm

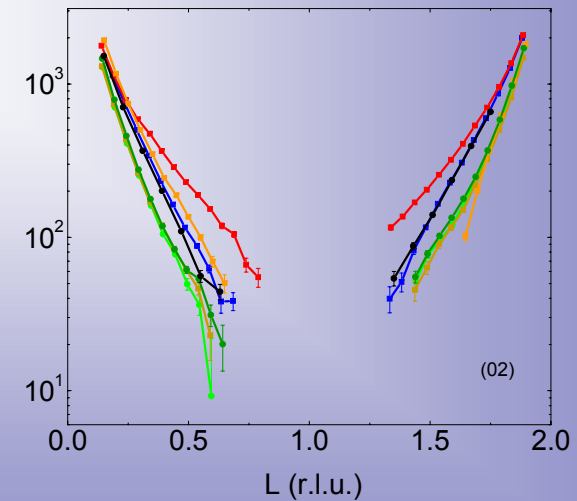
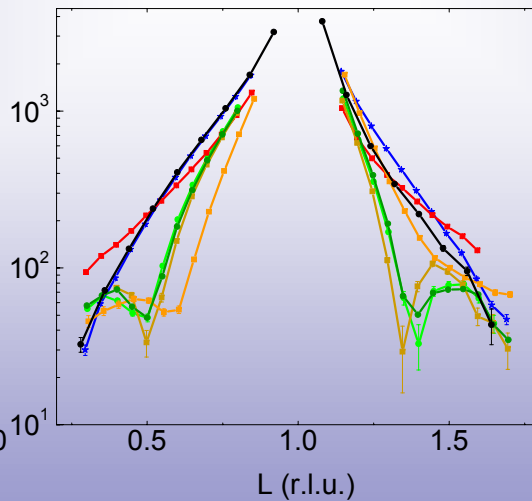
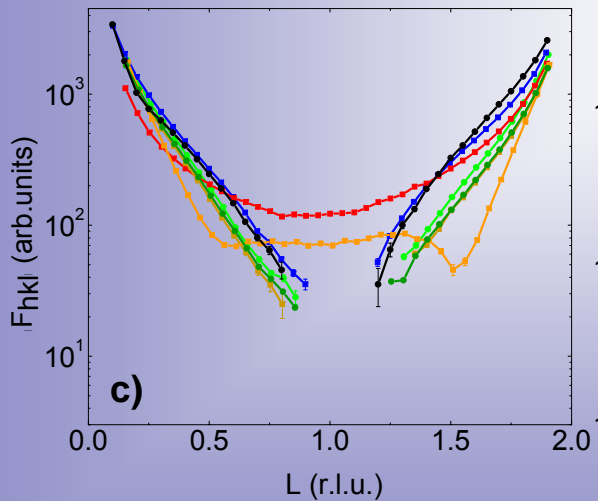




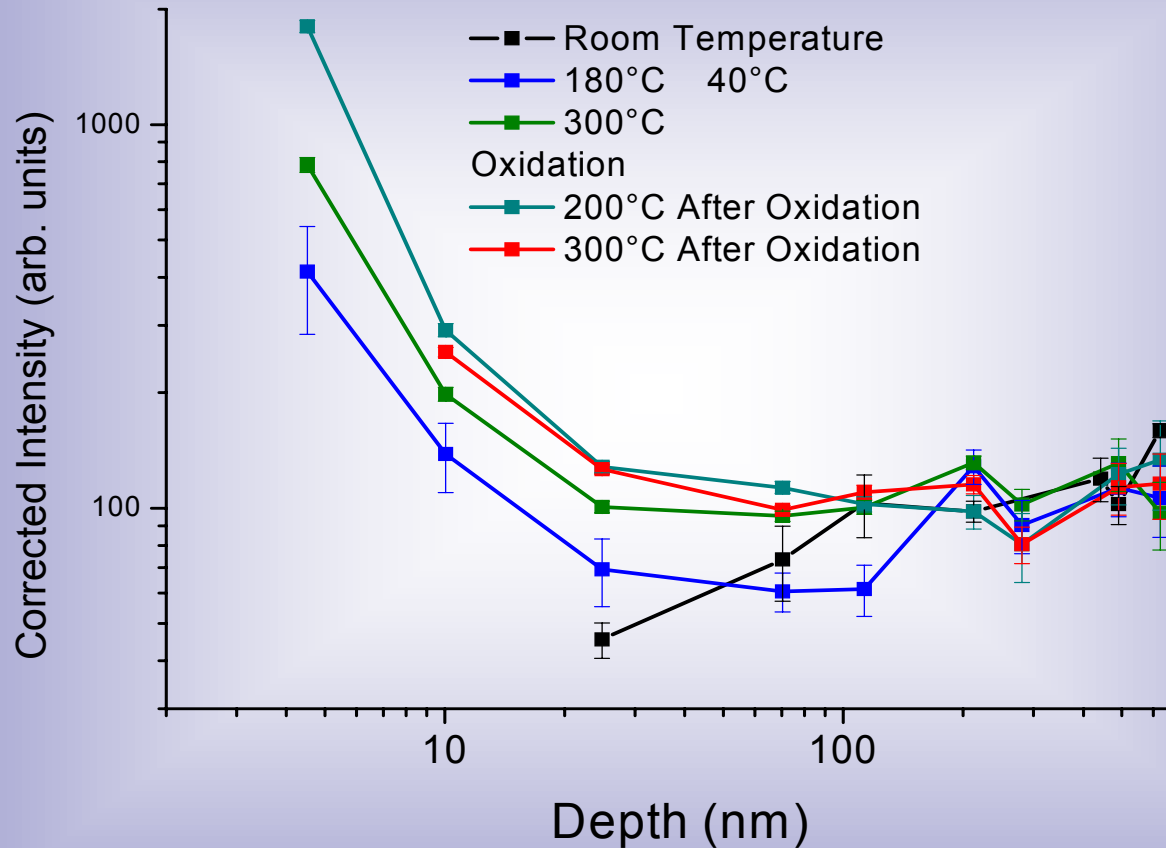
# In-situ results



As prepared  
120°C, 2h (UHV)  
300°C, 30min.  
Oxidation  
120°C,  $P = 5.10^{-6}$  mbar  
300°C, 1h  
700°C, 5min.  
1000°C, 5min.



Figures : a) X-ray Reflectivity, b) grazing incidence diffuse scattering and c) CTR



**First estimation** of the evolution of the **oxygen concentration** as a function of depth and temperature for the Nb(110) oxidized 6h in air.