Quantitative study of the transition layers in Mo/Si multilayer from the analysis of the Si Kβ x-ray emission band

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GROUPEMENT NATIONAL DE
MICROSCOPIE ELECTRONIQUE A BALAYAGE
ET DE MICROANALYSES

En convention de coopération avec la Société Française de Physique

M&M 2005
Honolulu, Hawaii

X-UV multilayer interferential mirrors (MIM)

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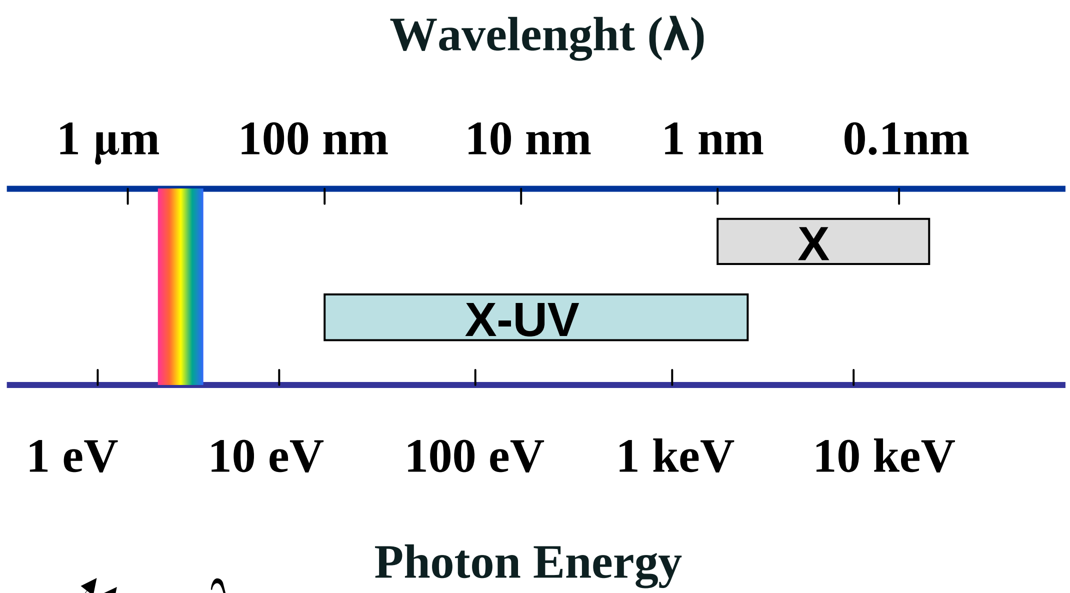
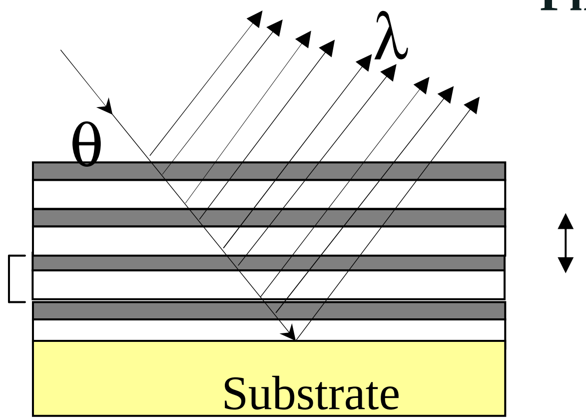
Optical components in the X-UV range

Consist in periodic stacks of ultrathin layers with nanometer thickness



Absorbing material (Mo, W…)

Light material (Si, C, B4C…)



d = d absorbing + d light

$$2d\sin\theta\sqrt{1-\frac{2\delta}{\sin^2\theta}}=k\lambda$$

Bragg's law corrected by the effects of refraction

δ is the bilayer weighted real part of the refractive index

X-UV Multilayer interferential mirrors (MIM)

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Structures at the basis of various devices because :

- reflect
- image
- focus
- polarize
- monochromatize

X-rays → **Many applications**

Mo / Si

λ	3 nm	13 nm	30 nm	46 nm	60 nm
E	413 eV	92 eV	41 eV	27 eV	20 eV

X-ray Microscopy

EUV Lithography

X-ray Astronomy

X-ray Lasers, Plasma diagnostics

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Problematics

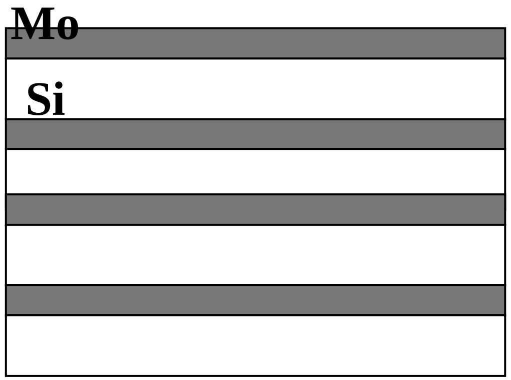
MIM's performances depend on the quality of their interfaces

Thermal treatments enhance the diffusion processes at the interfaces

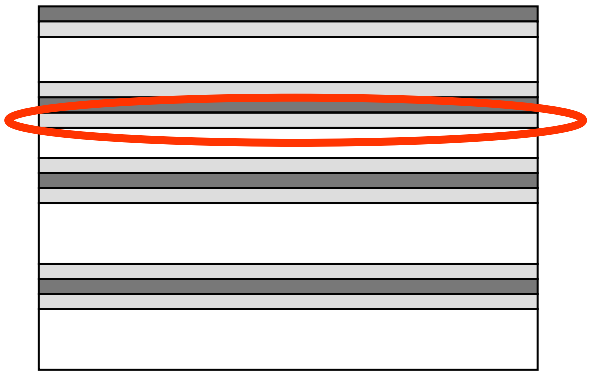
GOAL

to probe the transition layer

to determine its nature and thickness



Perfect Mo /Si MIM



Mo / Si MIM affected by interdiffusion

G N M E B A

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Analysis method

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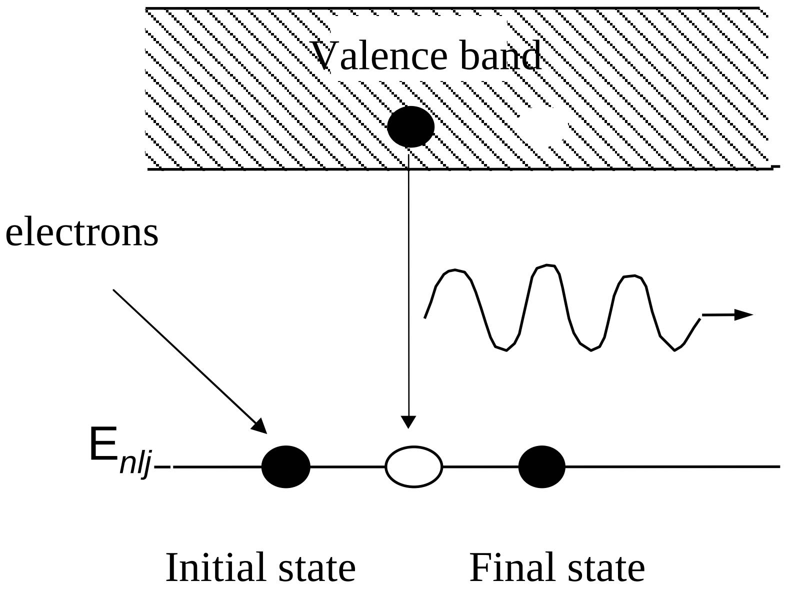
X-RAY EMISSION SPECTROSCOPY INDUCED BY ELECTRONS (XES)



Study of buried layers and interlayers non destructively

Studied emission : Si Kß (3p→1s)

⇓

Analysis of the density of occupied Si 3p states

Identification and quantification

of the Si-containing compounds

APPARATUS

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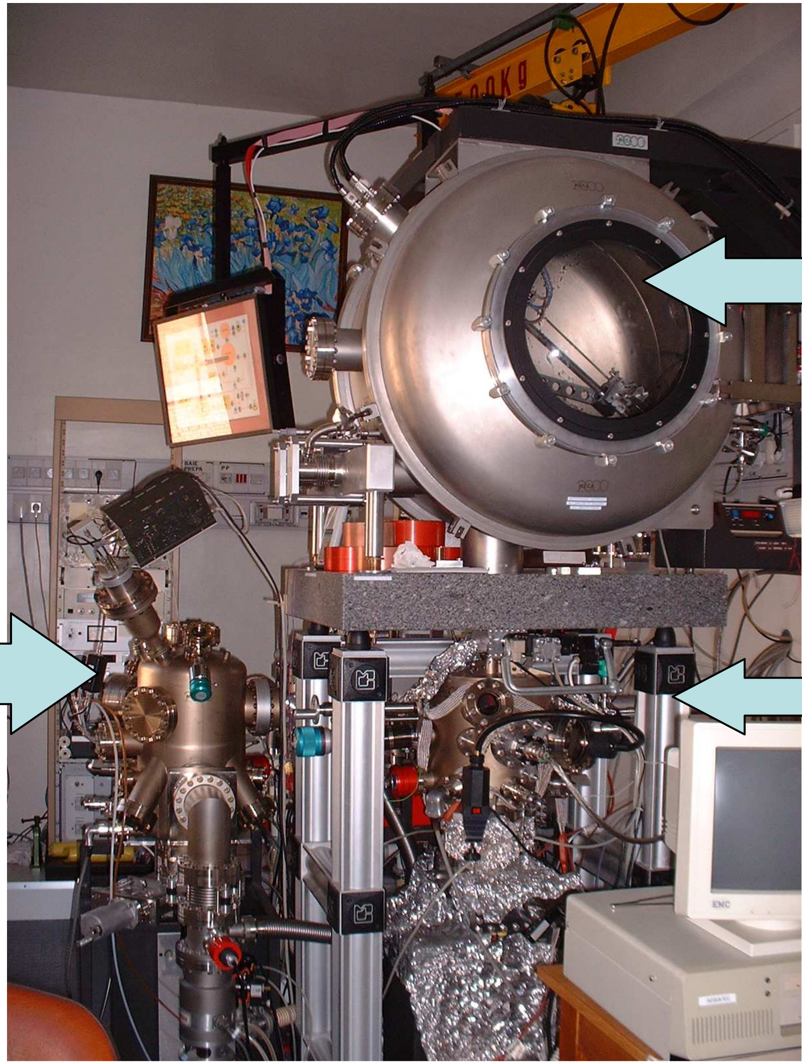
IRIS : Instument de Recherche sur les Surfaces et les Interfaces



Spectrometer

Preparation chamber

Electron gun

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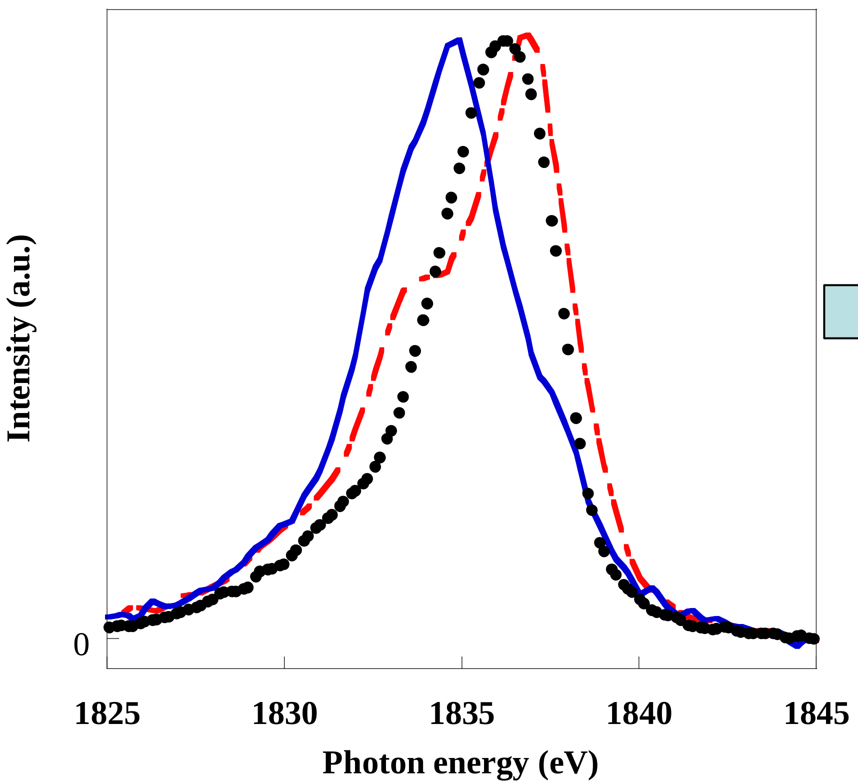
Si Kβ spectra of the reference compounds

Parameters of the studied Mo / Si MIM prepared by ion beam sputtering

- **40 Mo / Si bilayers**
- **d_{Mo} = 2.92 nm, d_{Si} = 4.04 nm**
- **Substrate : silicon**



Each reference compound has its own shape

Distinction between the physiso-chemical states of the emitting silicon atoms

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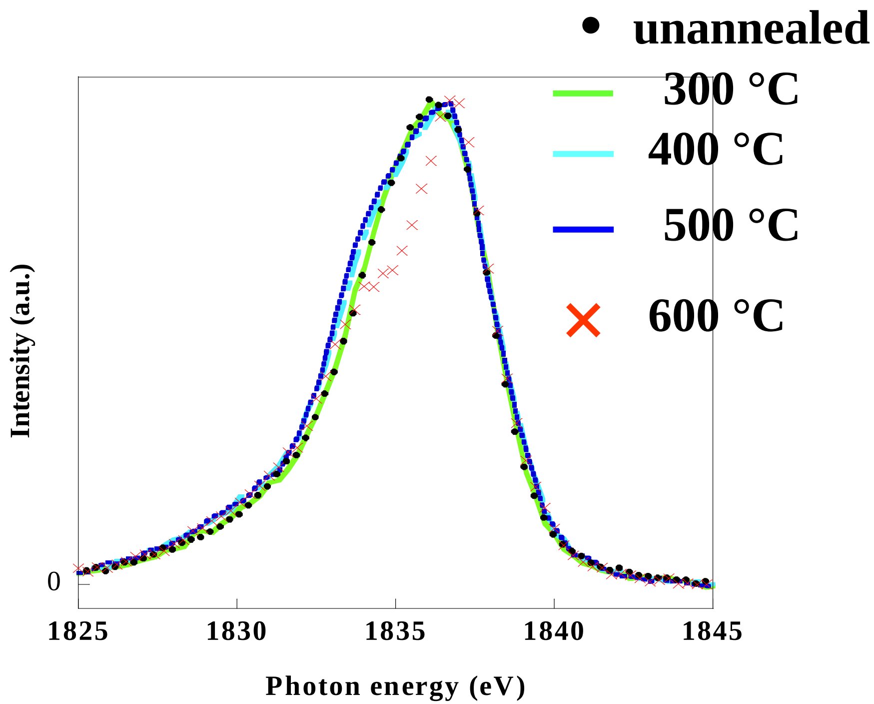
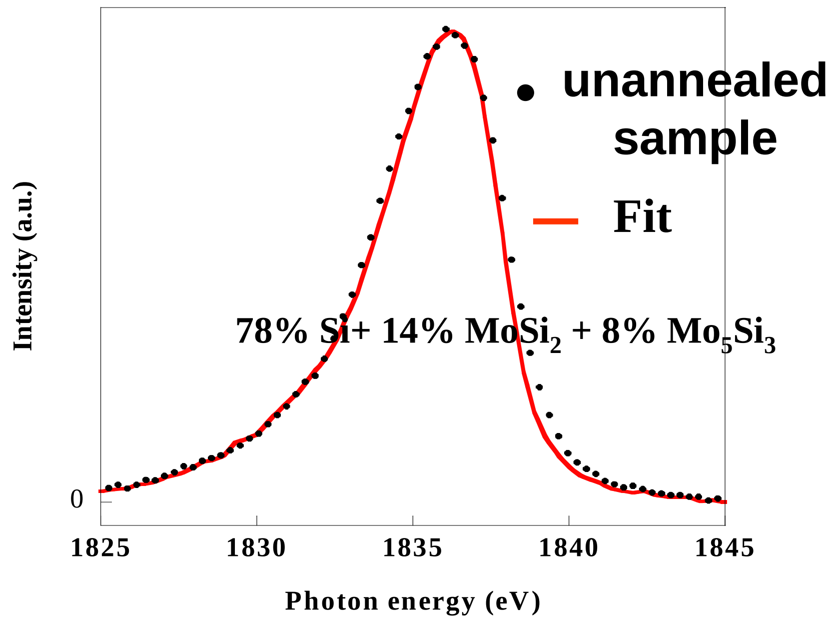
Study of the sample upon annealing





Results

sample	Unannealed	annealed 300°C	annealed 400 °C	annealed 500 °C	annealed 600°C
silicide contribution (%)	22	38	43	58	87

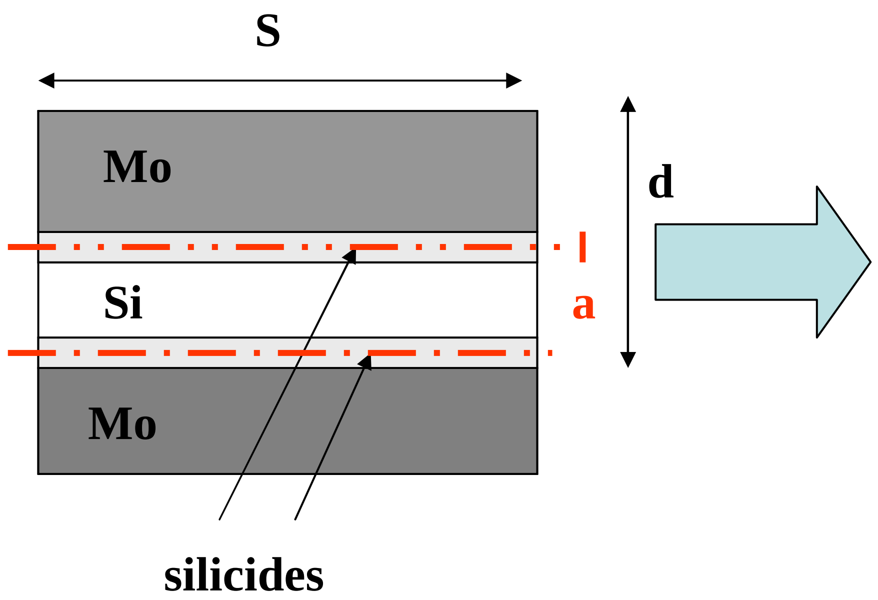
→ **Increasing silicide contribution with increasing temperature**

Determination of the interphase's thickness

$I_{MIM} = x\,Si + y\,MoSi_2 + z\,Mo_5Si_3$

$$\frac{I_{interphase}}{I_{silicon}} = \frac{\text{Number of emitting Si atoms interphase}}{\text{Number of emitting Si atoms Si layer}} = \frac{y+z}{x} = R$$

Interdiffusion model (MIYATA et al.)



$I_{Si} \sim$ **Nb atoms Si/cm^3 x analysed volume**

$$\frac{I_{interphase} \sim \left(\frac{\rho}{M}\right)_{interphase} \times N \times 2a \times S}{I_{Si} \sim \left(\frac{\rho}{M}\right)_{Si} \times N \times (d_{Si} - a) \times S}$$

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Determination of the interphase's thickness

$$\left(\frac{\rho}{M}\right)_{interphase} = 2a \times \left(\frac{\rho}{M}\right)_{MoSi_2} + 3b \times \left(\frac{\rho}{M}\right)_{Mo_5Si_3} \quad \text{with} \begin{cases} I_{silicides} = a\,MoSi_2 + b\,Mo_5Si_3 \\ a + b = 1 \end{cases}$$

$$\left(\frac{\rho}{M}\right)_{Si\ in\ MoSi_2} = 2 \times \left(\frac{\rho}{M}\right)_{MoSi_2}$$

Finally,

$$a = \frac{R(\rho/M)_{Si}\, d_{Si}}{2(\rho/M)_{int} + R(\rho/M)Si}$$

Results

sample	Unannealed	annealed 300°C	annealed 400 °C	annealed 500 °C	annealed 600°C
transition layer (nm)	0.4	0.6	1.0	1.5	3.1

Interlayer thickness increases with the annealing temperature

Conclusion

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1. Methodology Side

- XES suited tool for the study of buried interfaces
- From the silicides contribution → Determination of the transition layer's thickness

2. Material science side

- Increasing of the silicides contribution and thus interlayer's thickness,with the increasing temperature from 300°C to 500°C

Corelation with the optical performances :

- Decreasing of the reflectivity from 300 °C to 500 °C
- Drastic change of the physico-chemical properties at 600 °C.