Université du Maine

LPEC

CNRS

Institut de Recherche en Ingénierie Moléculaire et Matériaux Fonctionnels

IRIM2F
FR CNRS n° 2575

Laboratoire de Physique de l'Etat Condensé

UMR CNRS 6087

Equipe Nanostructuration et Fonctionnalisation (Resp. Alain Gibaud)

Systèmes

- Systèmes hybrides mésostructurés et fonctionnalisés
- Surfaces fonctionnalisées
- Hybrides polymères / nanoparticules SiC
- Films minces de polymères conducteurs

Techniques d'analyse des couches minces et nanoobjets

- Réflectivité X / SAXS / GISAXS
- Microscopie nano-objet (Sarfus)
- Raman couches minces
- Electro-optique de couches minces
- Optoacoustique
- Capteurs bioluminescents

Laboratoire des Rayons-X (Resp. Guillaume Brotons et Alain Gibaud)

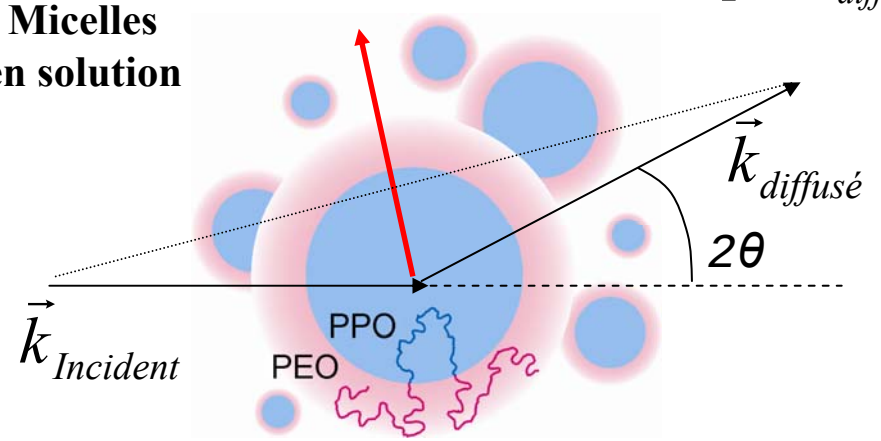
guillaume.brotons@univ-lemans.fr *(Tel: 02.43.83.32.93)*

Equipe Nanostructuration et Fonctionnalisation.

Permanents: D. Ausserré, J.-F. Bardeau, A. Bulou, G. Brotons, P. Daniel, N. Delorme, A. Désert, N. Errien, A.Gibaud, A. Kassiba, C. Launay O. Noël, M. Tabellout.

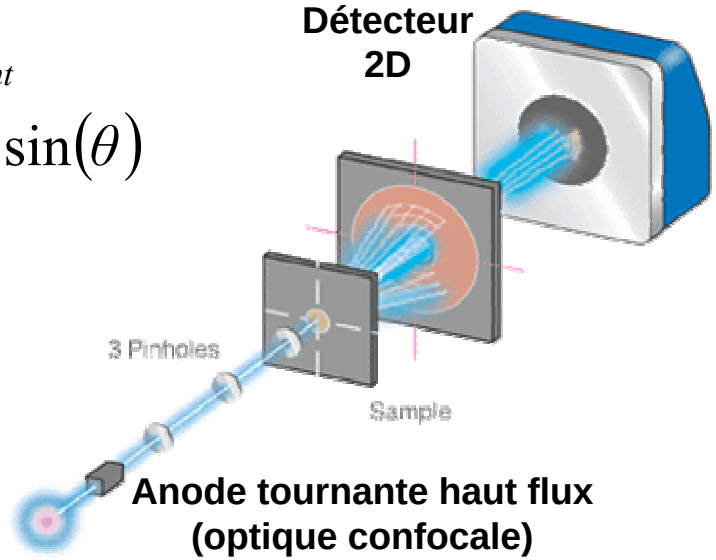
Exemples SAXS au LPEC.

Micelles en solution



Vecteur Diffusion: $\vec{q} = \vec{k}_{diffusé} - \vec{k}_{Incident}$

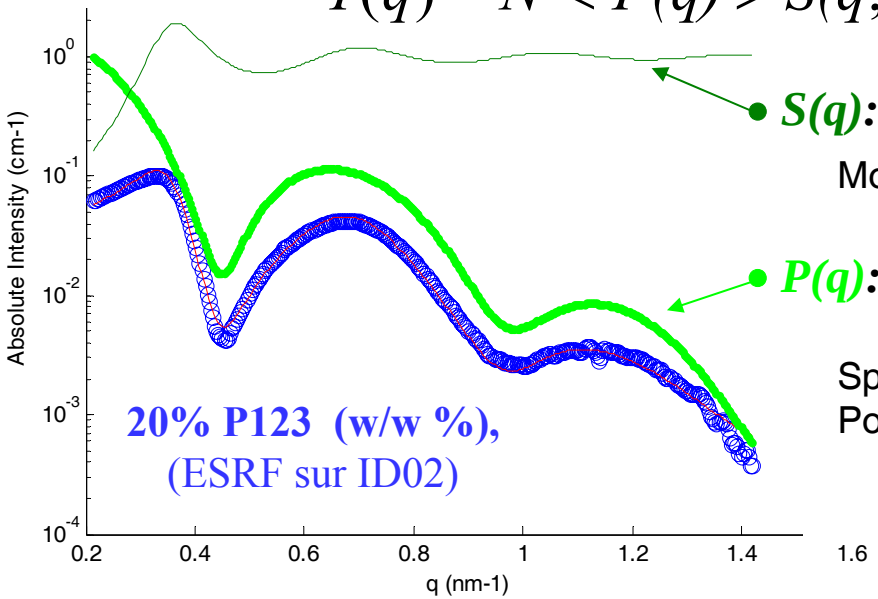
$$= (4\pi/\lambda)\sin(\theta)$$



Anode tournante haut flux (optique confocale)

10^7photons/s sur 300μm^2
divergence < 0,5 mrad.

$$I(q) \sim N < P(q) > S(q, R_{HS})$$

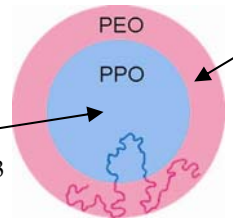


- ***S(q)*: Facteur de Structure** (*corrélations entre micelles*)

  Model sphères dures (approximation Perkus-Yevick)

- ***P(q)*: Facteur de Forme** (*profil micelles*)

  Sphère cœur-coquille
  Polydispersité faible



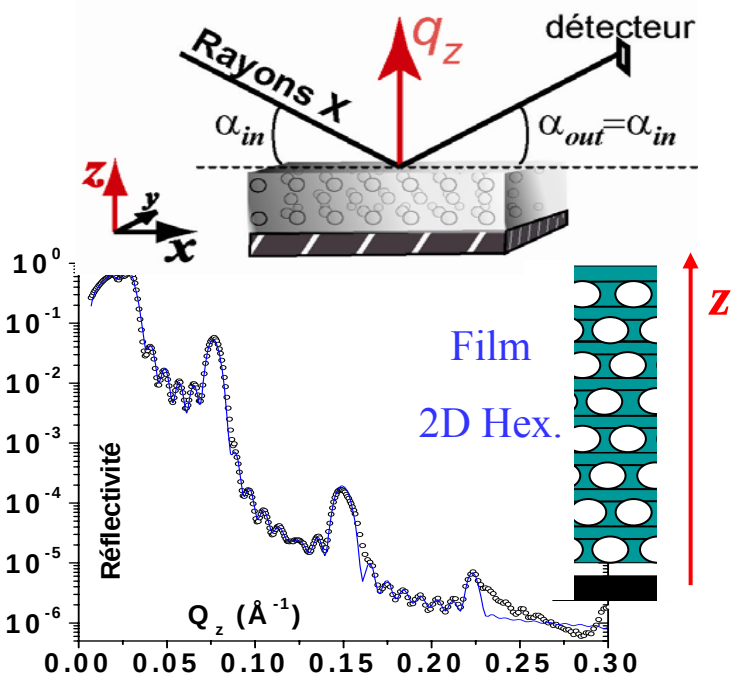
PEO~H_2O =334,5 é/nm^3

PPO~ 338,2 é/nm^3

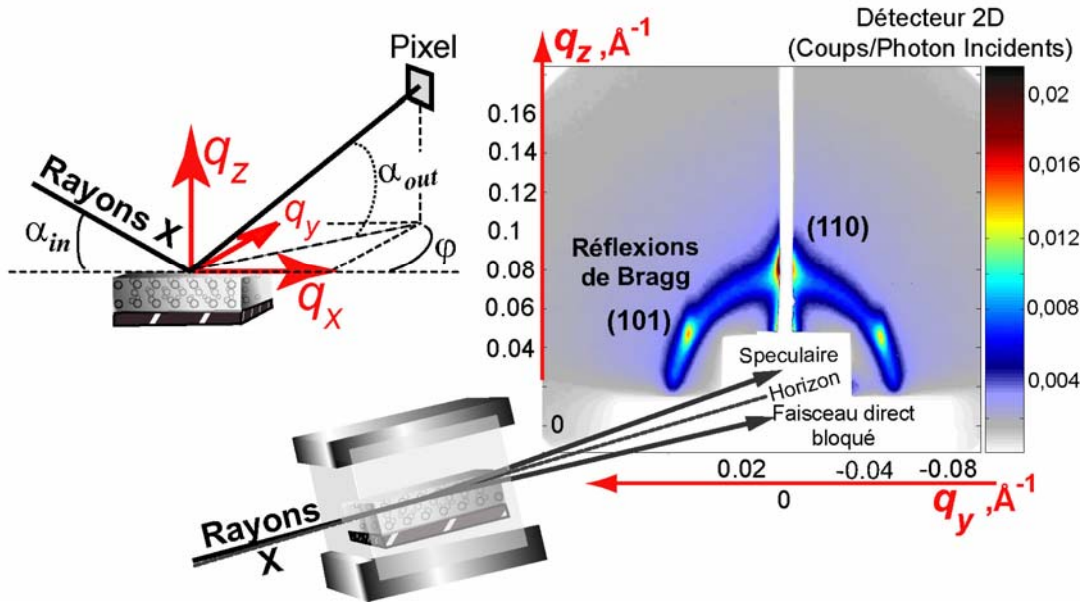
Soni, Brotons, Bellour, Naranayan & Gibaud. J.Phys.Chem.B., 2006.

Exemples Réflectivité et GISAXS au LPEC.

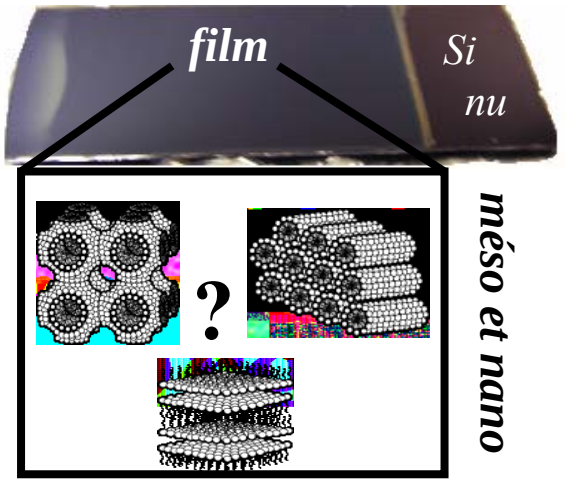
Réflectivité Spéculaire

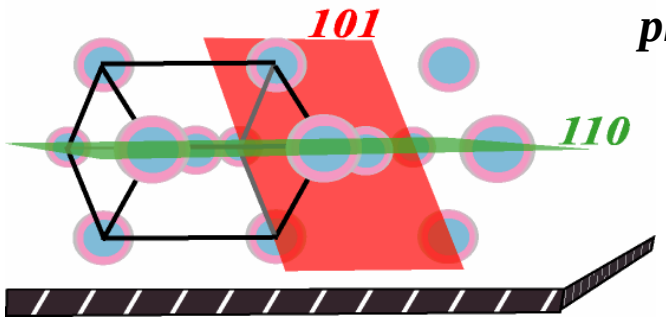


Diffusion des X en incidence rasante (GISAXS)

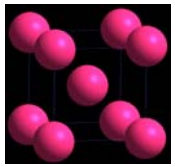


TiO_2/Pluronics

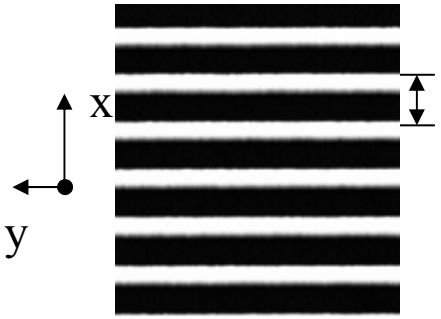




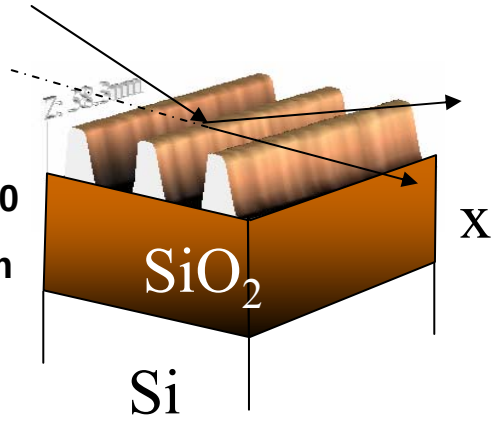
phase cubique centrée:

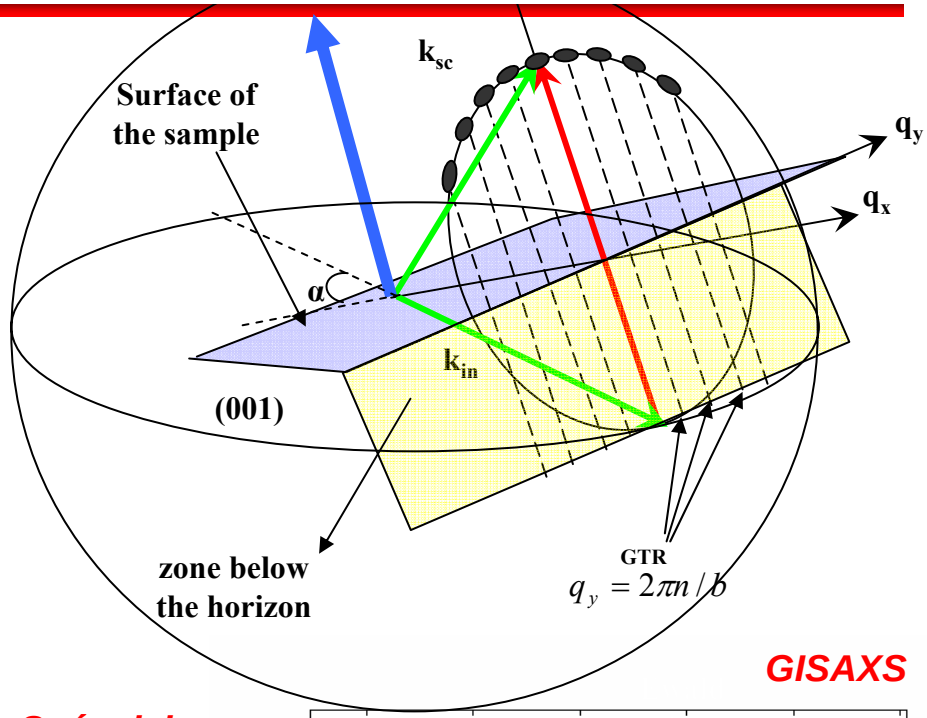


Réflectivité et GISAXS sur Réseaux de diffraction.



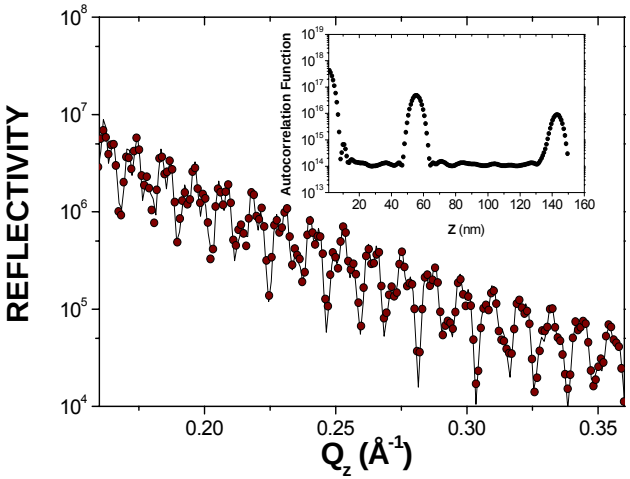
Motif 2



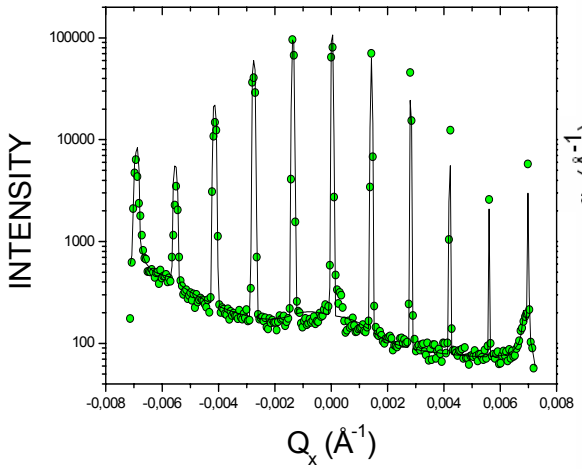


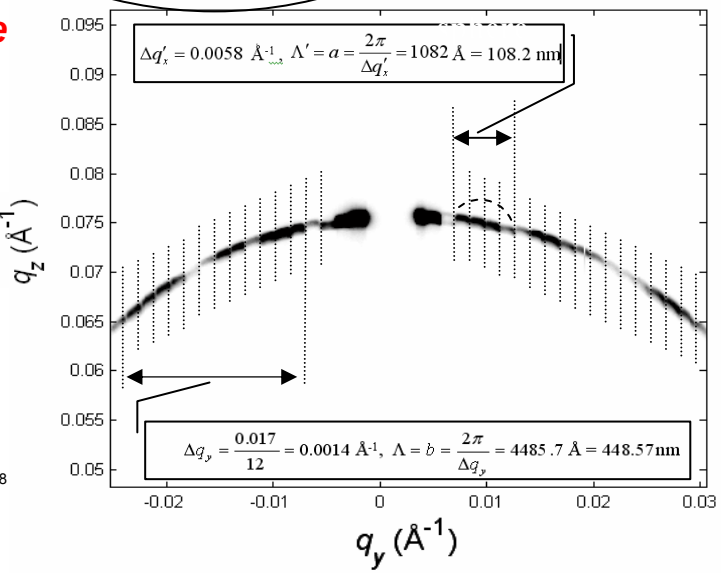
GISAXS

Réflectivité Spéculaire



Réflectivité Hors-Spéculaire





M.Yan, M.J.Henderson, J.-F. Bardeau, G.Brotons and A. Gibaud

Instrument SAXS/GISAXS Rigaku Molecular Metrology

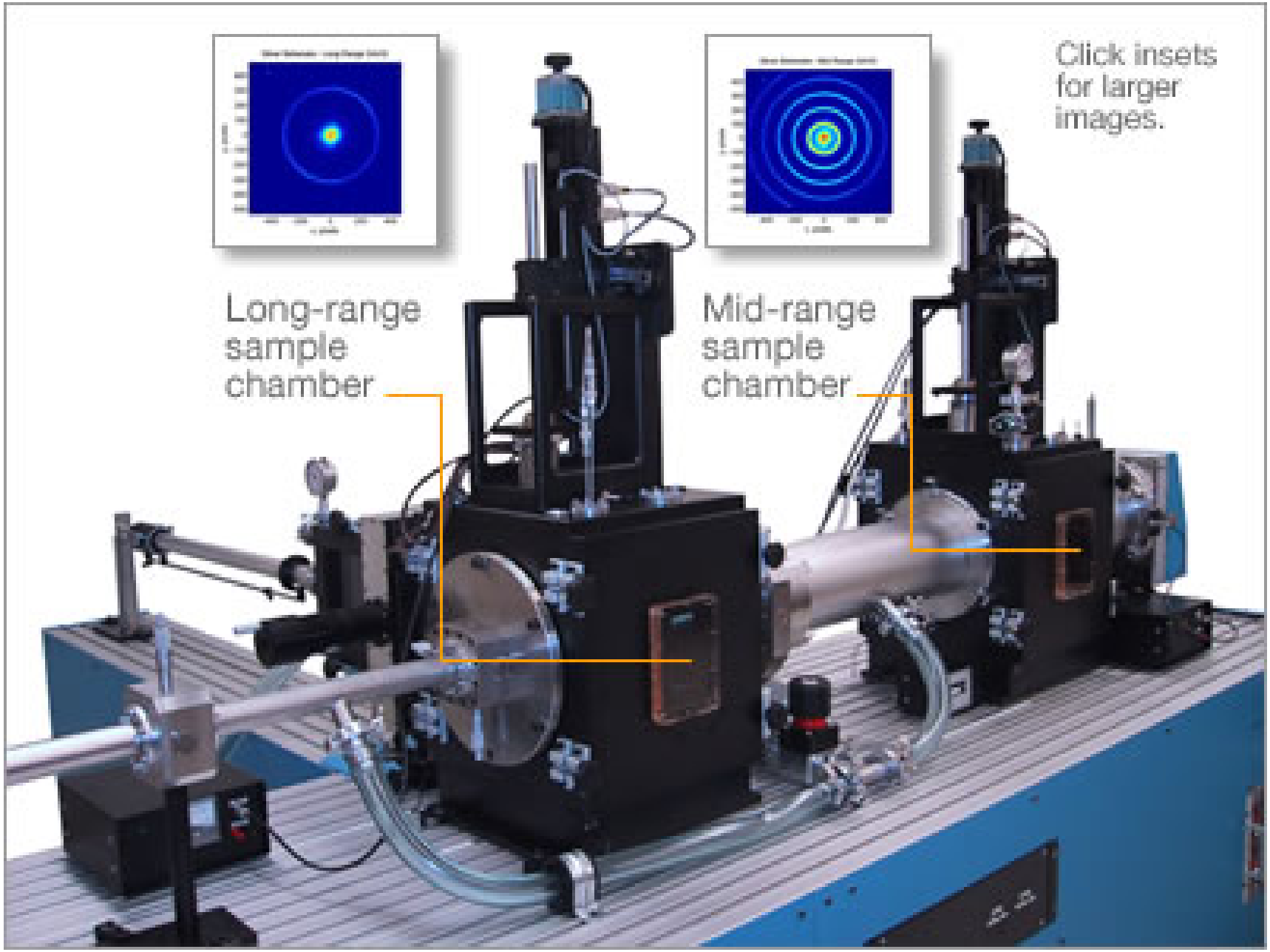
Revendeur France: www.elexience.fr (Pascal Clément) .

- Flexibilité du montage, Conception avec l'utilisateur

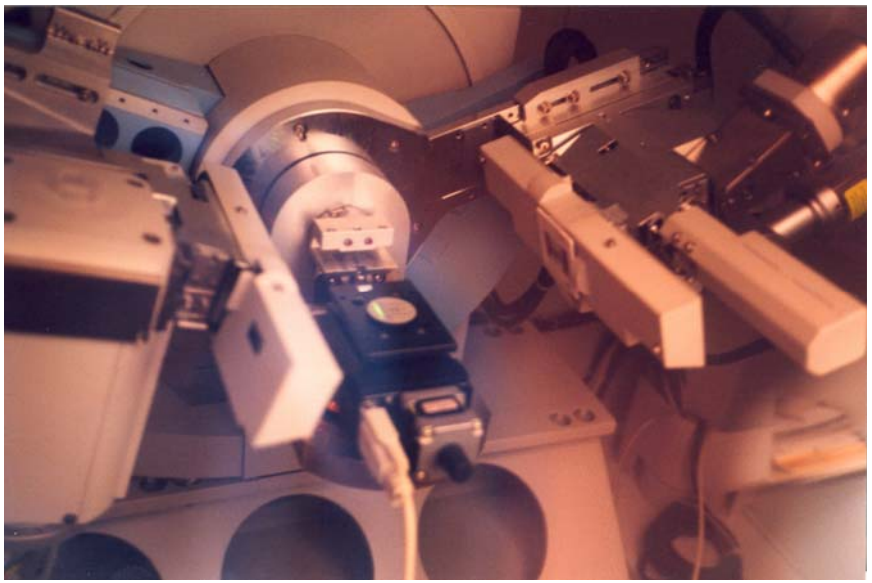
MicroMax™-002

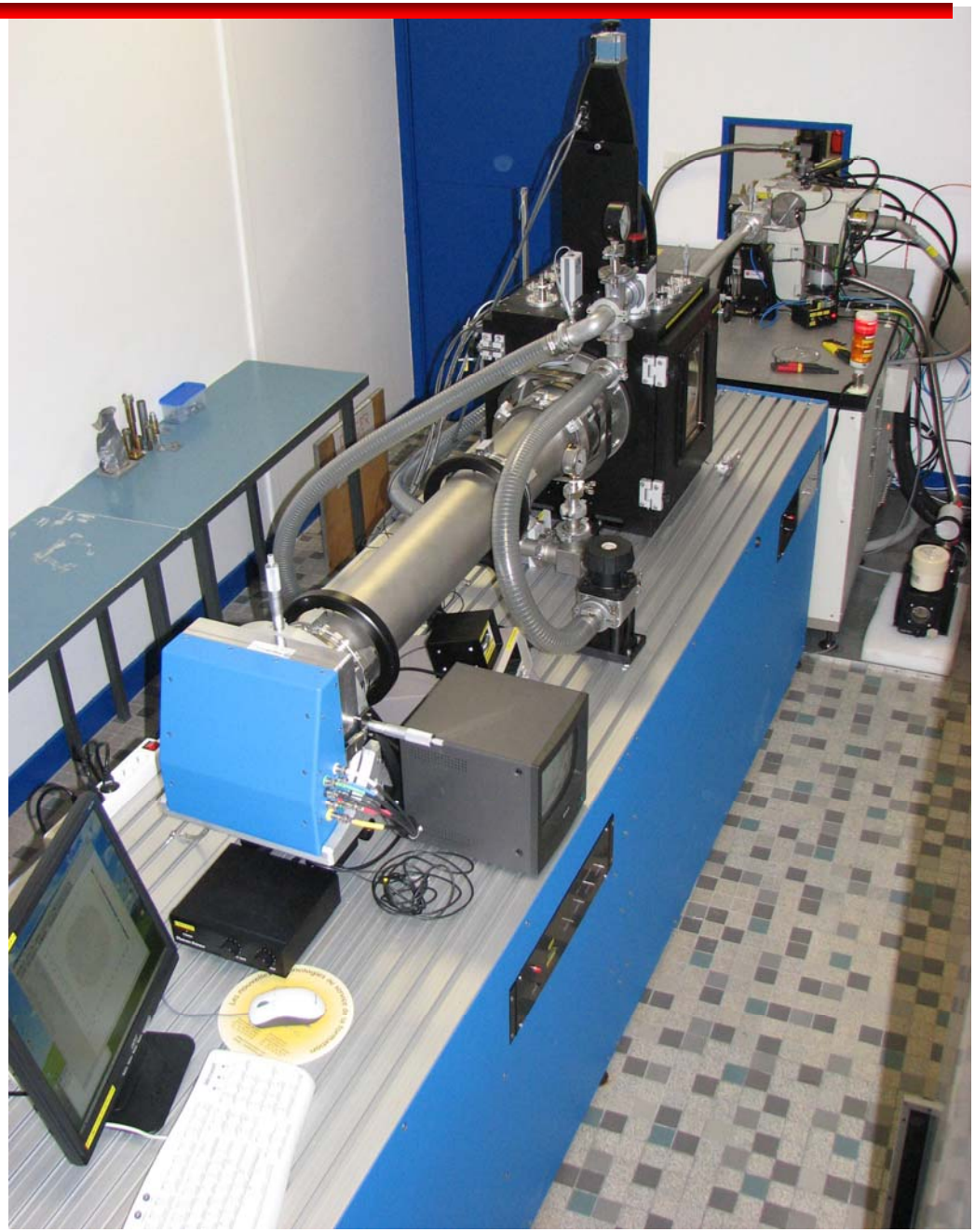
Source size:	20 μm
Source power:	30 watts
Weight:	220 lbs
Source-to-optic distance:	65 mm
Source-to-focus distance:	700 mm
Length of optic:	80 mm
Convergence angle:	2.91 mR

Version double chambre et image plate



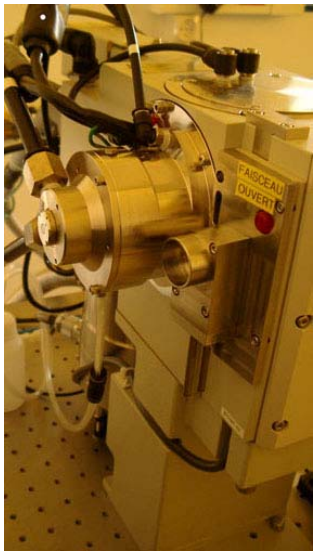
Montages laboratoire des Rayons-X du LPEC, Le Mans.

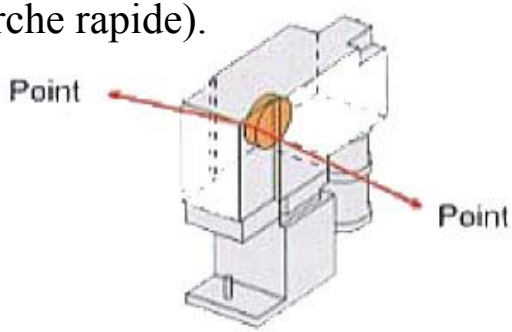




Anode tournante Rigaku UltraX 18 (Anode Cuivre) .

- Tête orientable et déplaçable sur le banc (Direct-Drive Rotating Anode).
- Volume intérieur tube divisé par deux (mise en marche rapide).





Anode Cuivre:

- 10 cm de diamètre.
- rotation à 6000 tours/min.
- Joint eau mécanique;

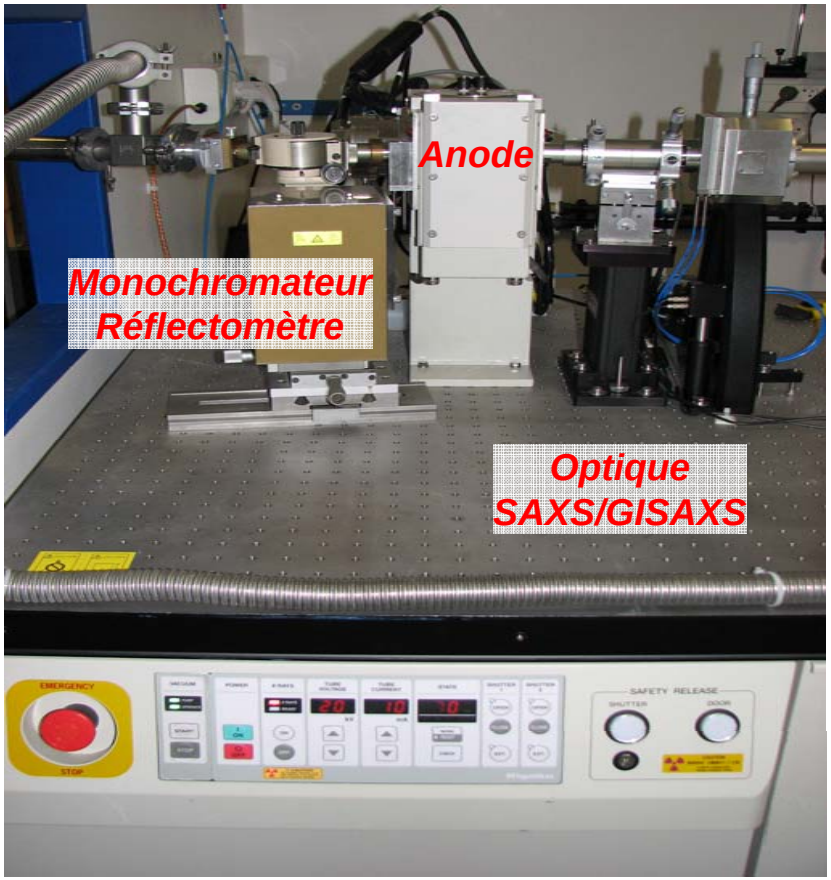
Gaine céramique et
Joint vide de type ferrofluide.

Générateur:

- **Voltage et courant:** 20-60kV (paliers de 1kV)
  10 à 300mA (paliers de 1mA)
- **Puissance maximum: 18kW (=60kVx300mA)**

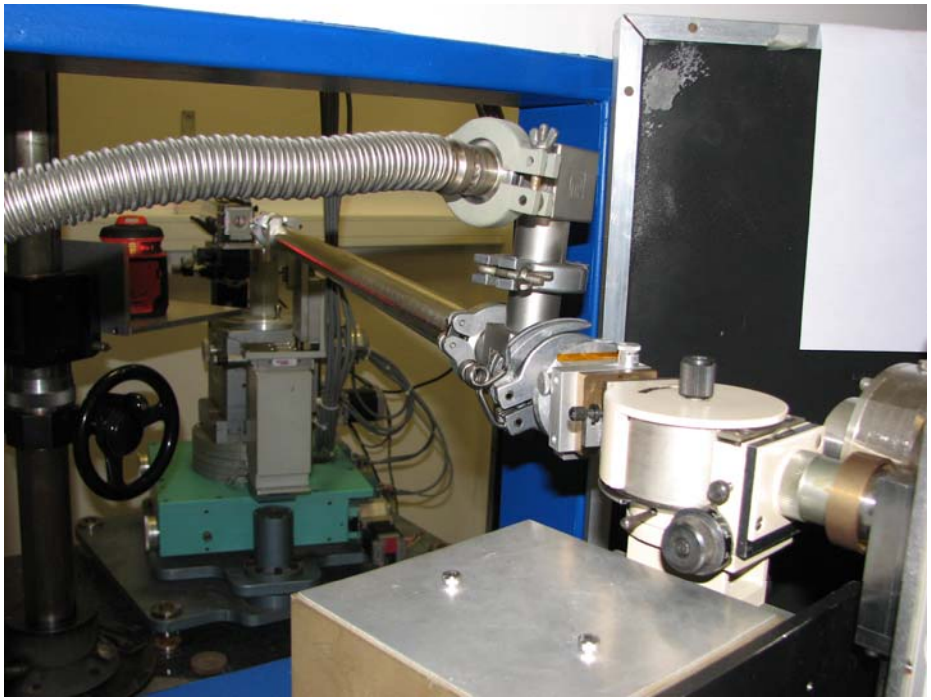
On travaille avec optique à:

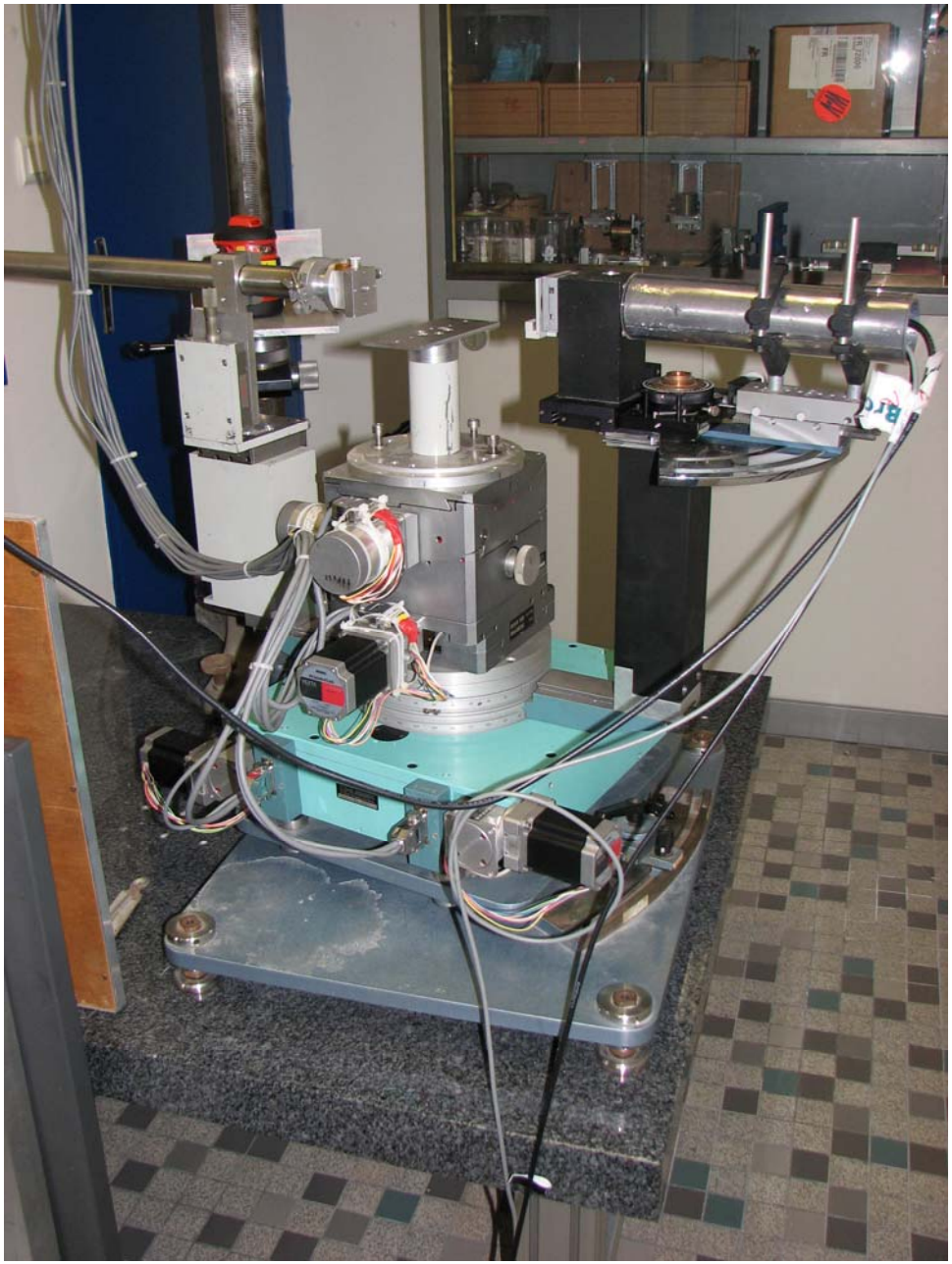
- **Stabilité tension: ± 1% rms à (18kW=60kVx300mA)**



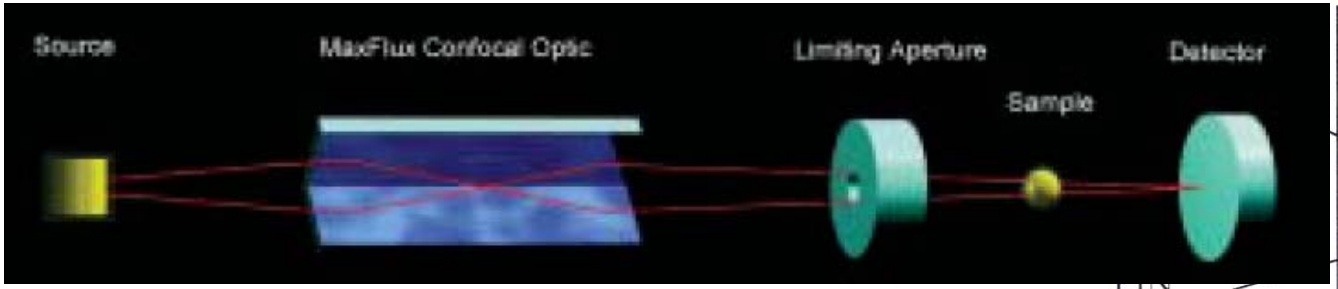
Rillettes

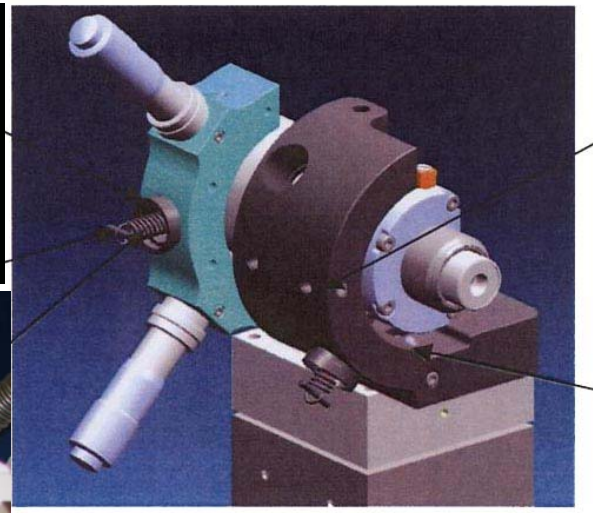
Réflectomètre Interdisciplinaire à Longue Lorgnette
Et Tromblon Télescopique pour l'Étude des Surfaces…

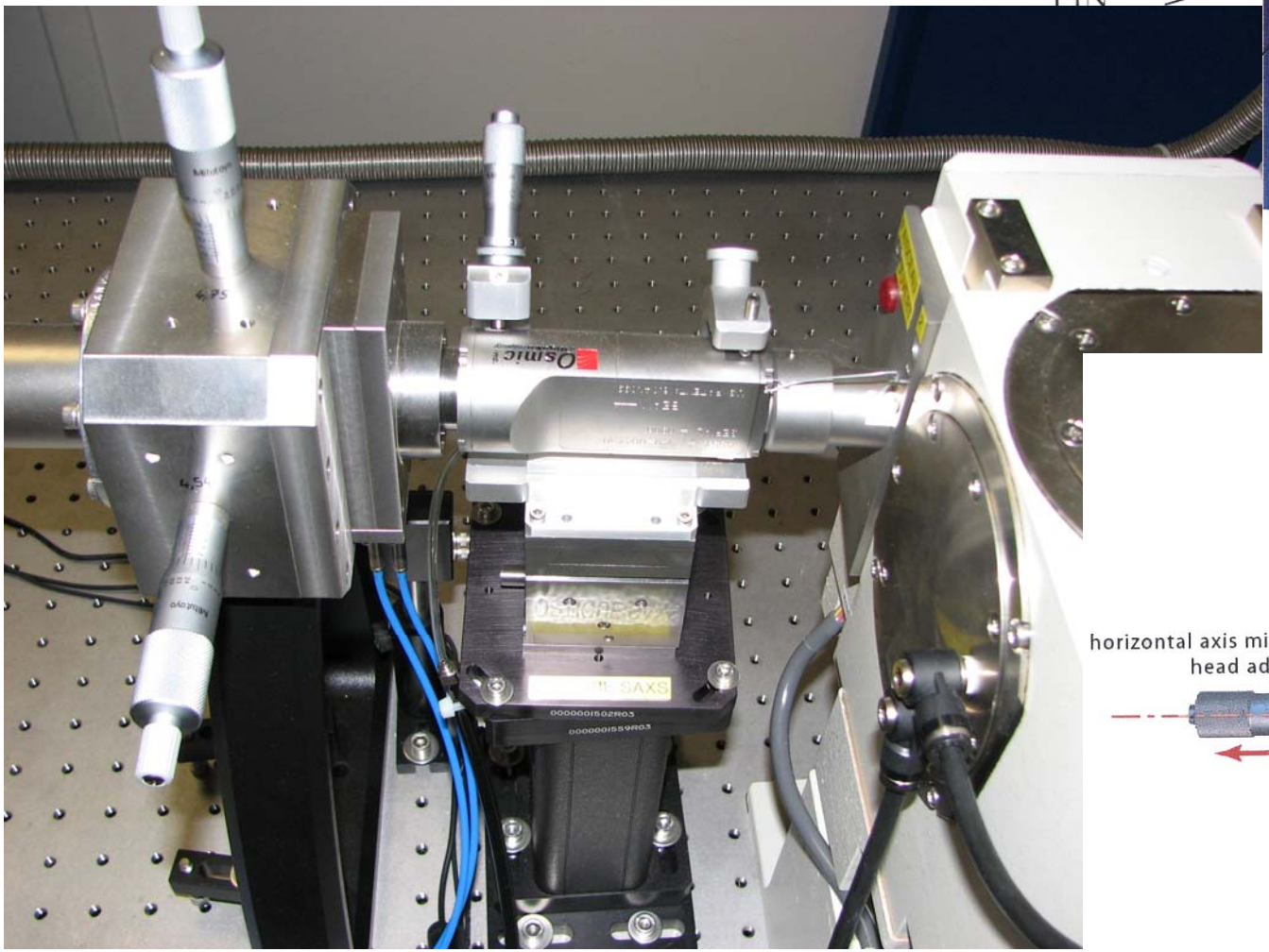




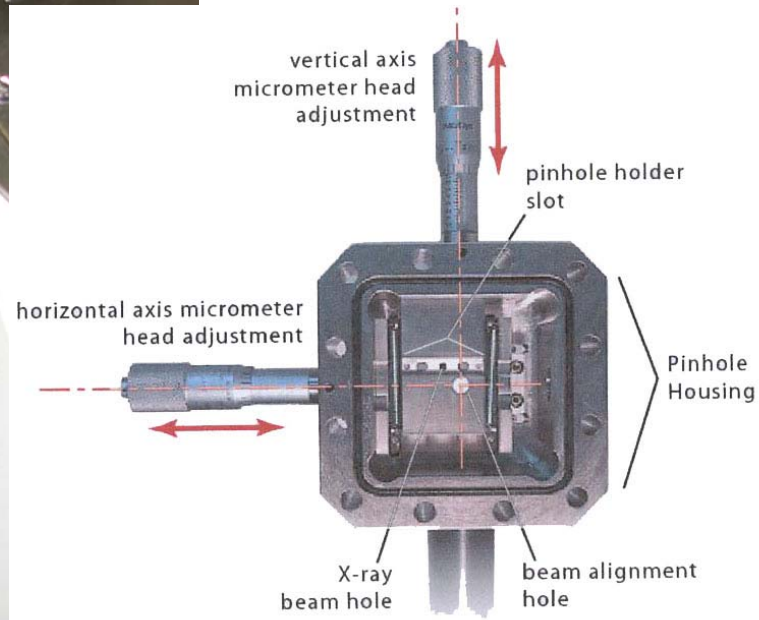
Optique Osmic Confocal Max-Flux (CMF Blue).







- 3 pinholes échangeables et ajustables.



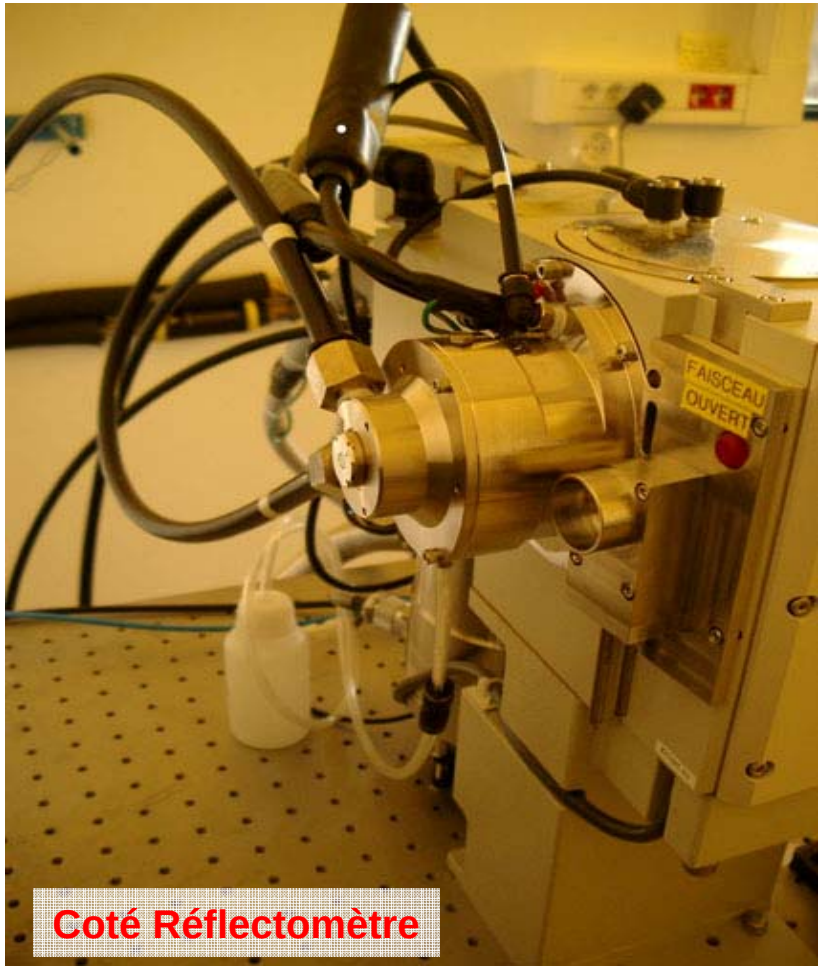
Filtres pneumatiques automatiques.

Générateur:

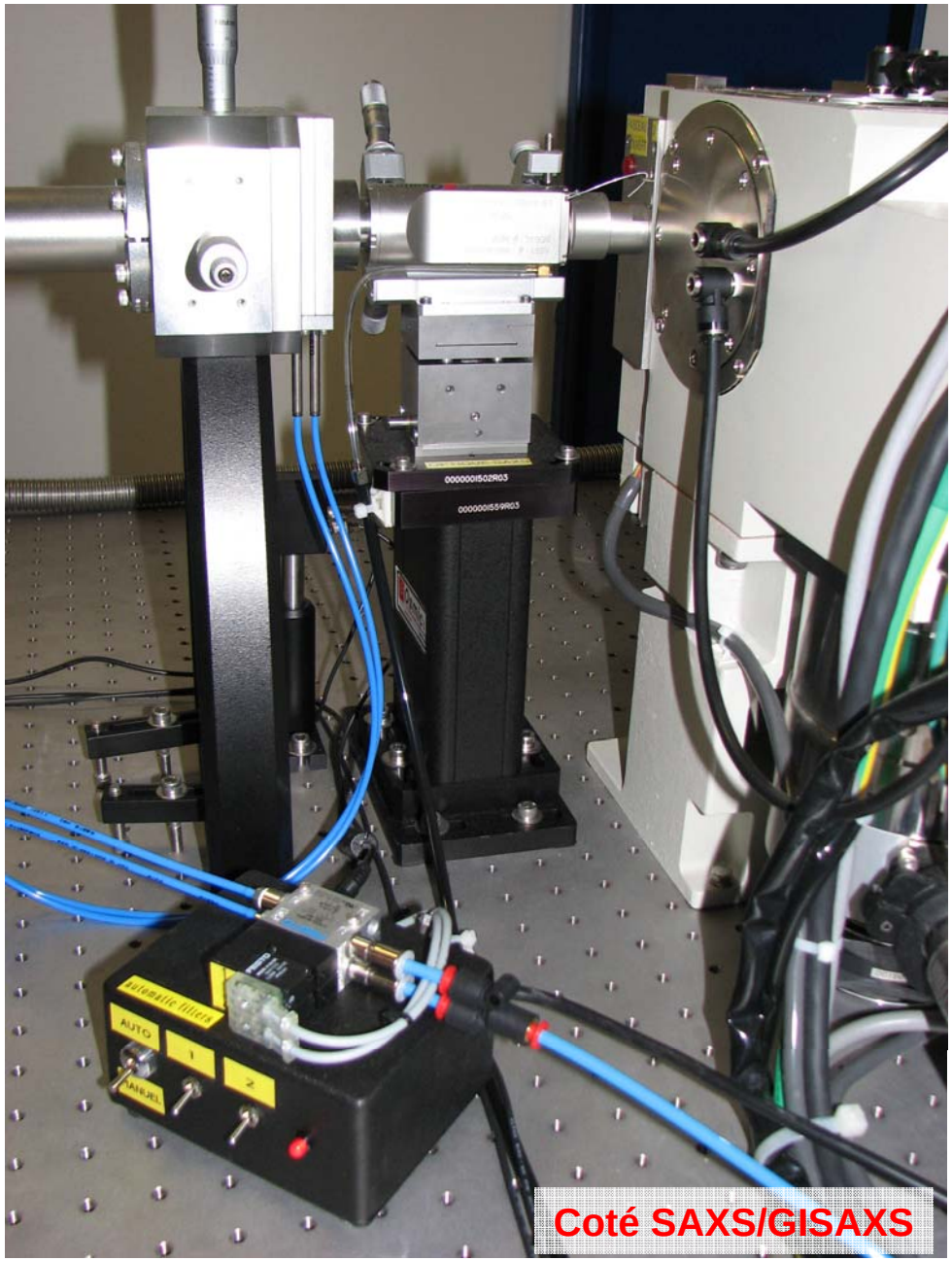
● Contrôlée par RS-232 (logiciel Spec ou autre).
Cycles montée descente, shutters, …

Filtres automatiques pneumatiques:

● Contrôlée sur carte NI et logiciel Spec.



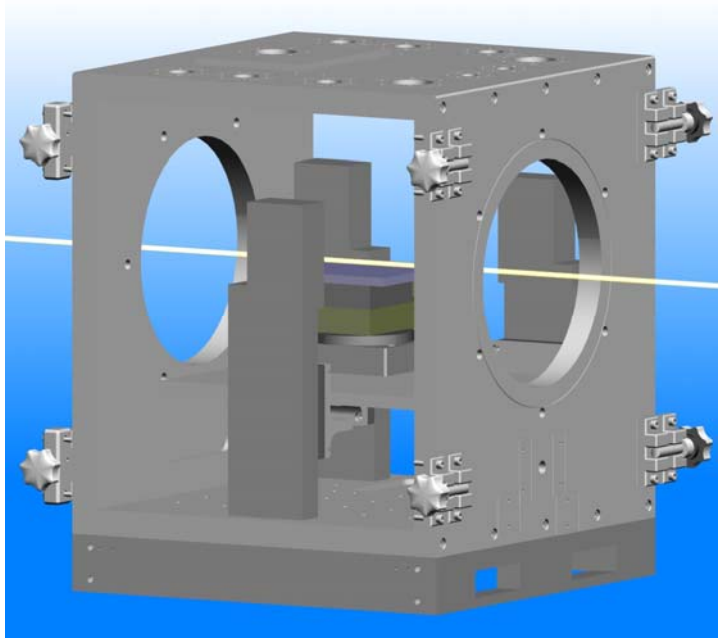
Coté Réflectomètre

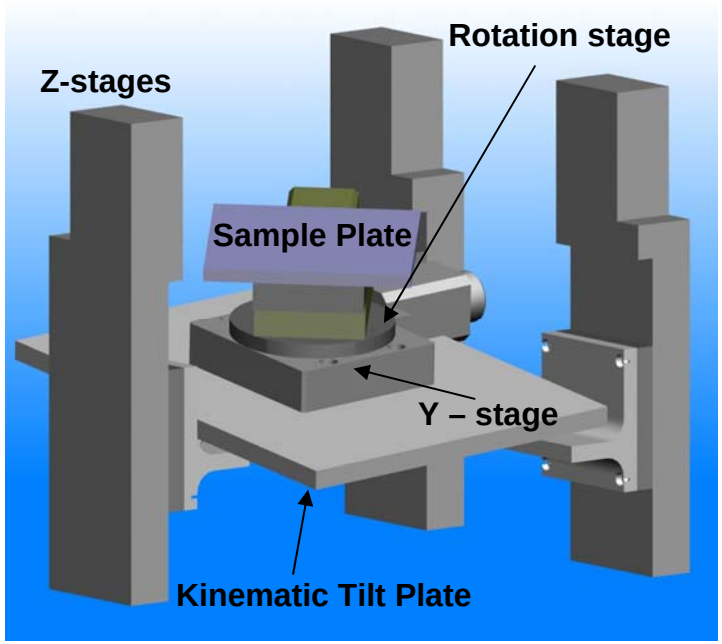


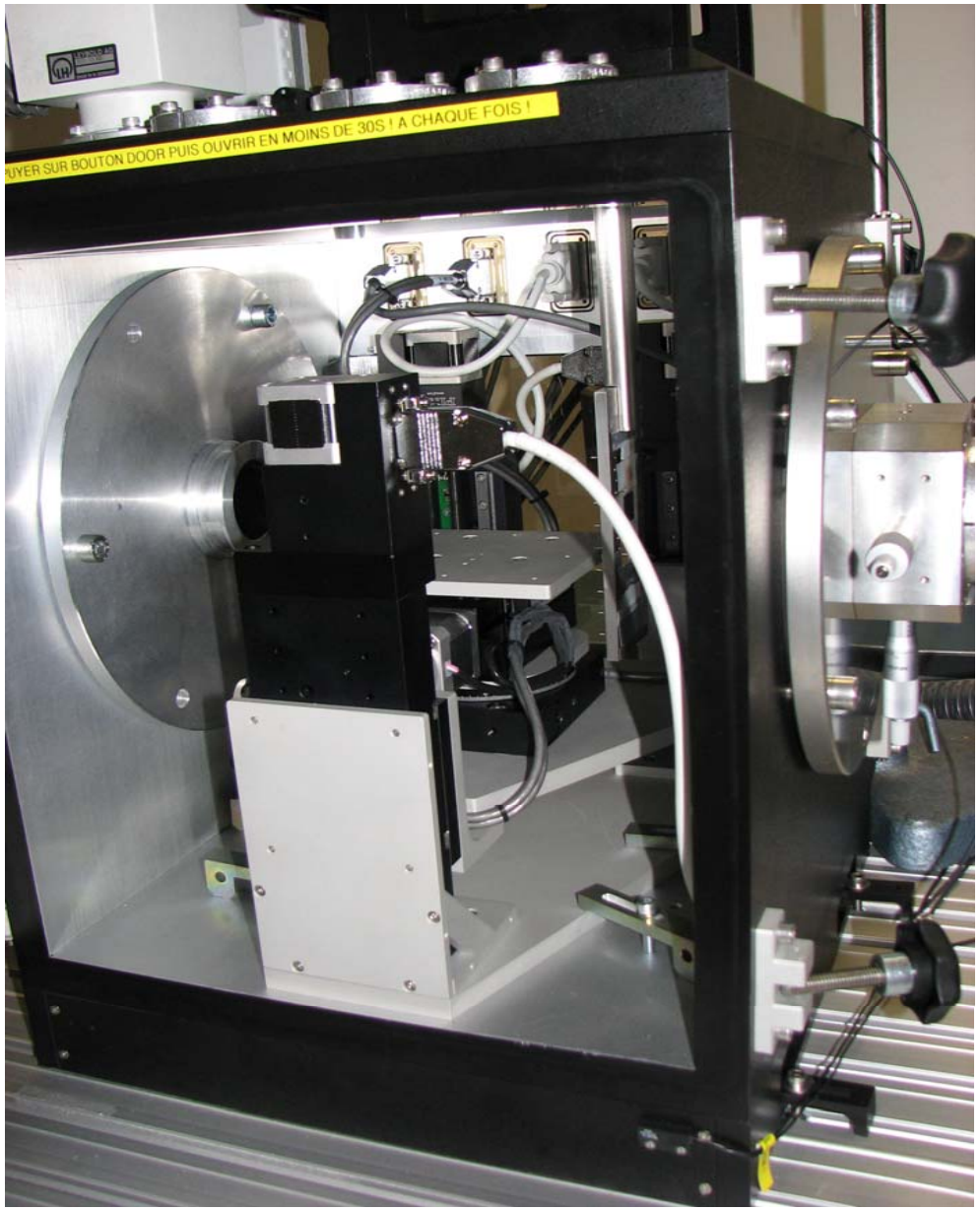
Coté SAXS/GISAXS

Goniomètre de type tripode.

Independent Stages Provide All Required Translations
Z-stages (Physik Inst. M-403.42S)
Y-translation +/- 25 mm (Danaher/Neat RM-200)
Full 360° Rotation (Thorlabs NR360/M)







Goniomètre.

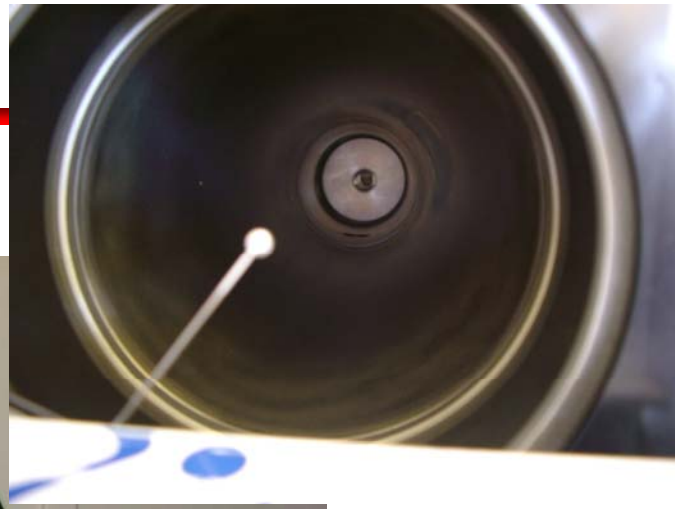
Résolution Angle d'incidence: 1mm/rev avec 200pas/rev soit une résolution de 0.005mm descendue à **0.0007mm/micro-pas**, avec plateau à 11cm du centre de rotation on obtient:

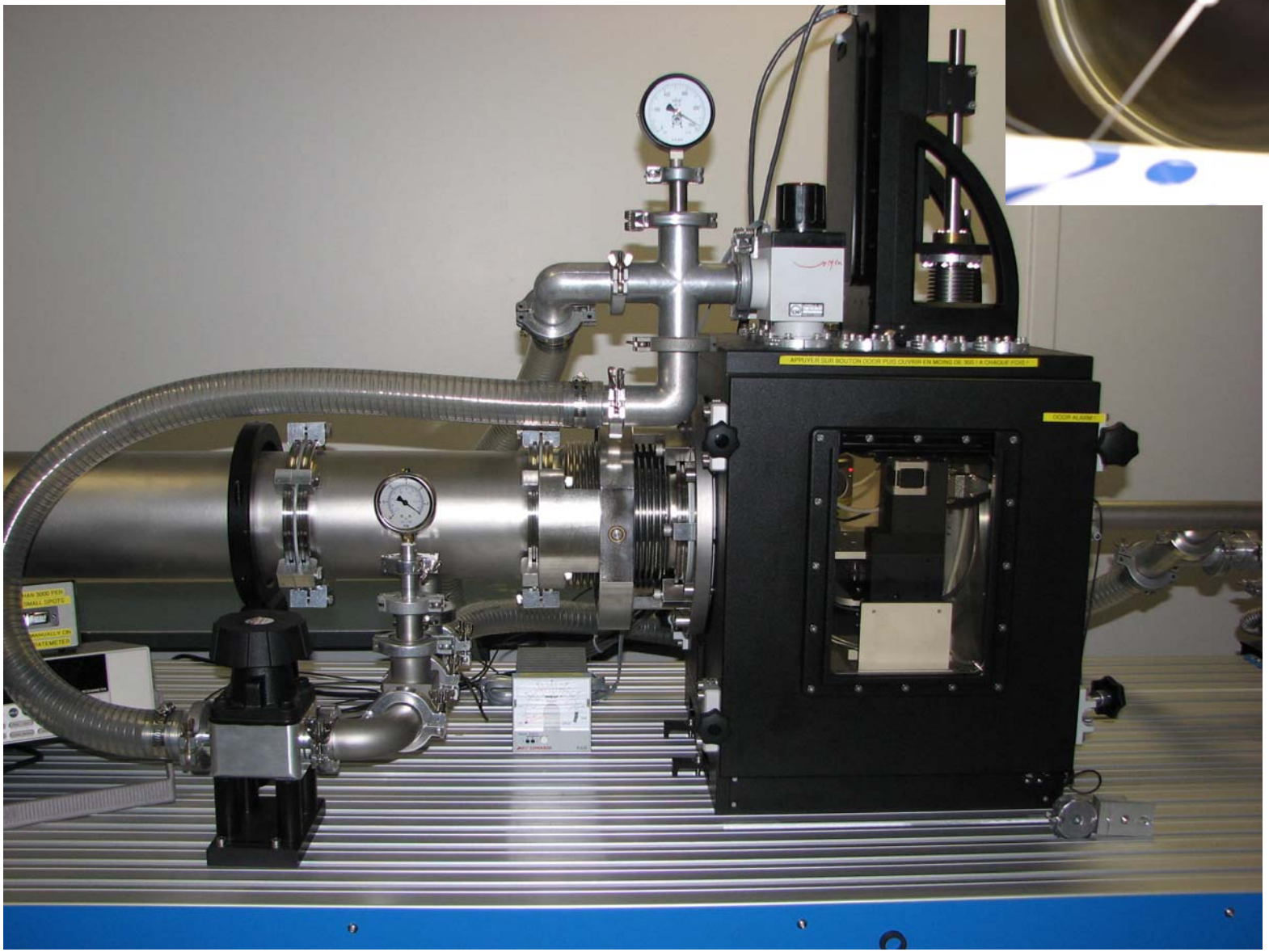
~ arctan(0.005 *à* 0.0007 mm/110) soit 0.0026° à 0.00033° en résolution.

TEST	RESULTS TO OBSERV	PRECISION
Rotation about y-axis (Grazing Incidence Angle)	-1 to 5 degrees	0.001 degree
Rotation about x-axis (Perpendicular to plane of incidence)	-2 to 2 degrees	0.001 degree
Z translation	Total Course 20mm	10 microns
Total Y translation	Minimum 20mm (+/- 10mm) (50 mm based on best design efforts – depends highly on the final room available within the system).	25 microns
Rotation about sample normal axis	360 degrees rotation	0.01 degree
Sphere of confusion	Desired equal or below 25 microns based on best design effort and manufacturer's specifications. Not larger than 100 microns.	
Load capacity Guaranteed to be at least 5 kilograms	Mounting of sample with a limit of 5 kg onto the stage. The correct motions will be observed.	

Beam Stop avec compteur interne.

- Beam stop avec Pin-diode silicium, position micro-contrôle.



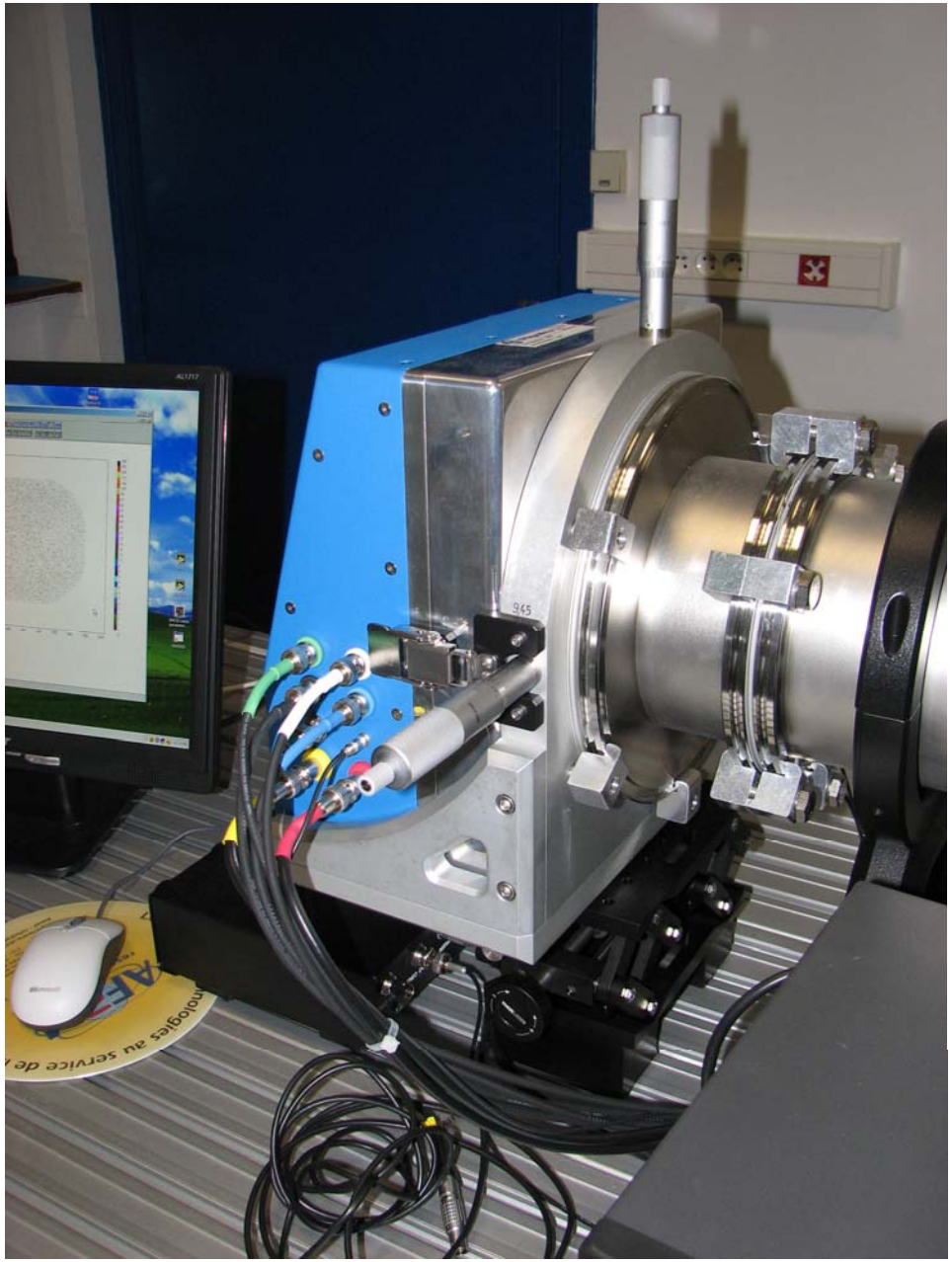


Détecteur 2D à Gaz de type chambre à fils "Gabriel".

licence Molecular Metrology, Rigaku USA .

- 12 cm de diamètre et existe en 20cm.
- Résolution 200 microns (512x512 à 4096x4096).
- Dynamique: 100 coups/seconde homogènes.
- Électronique type NIM avec MCA Fastec (à retard).
- Fenêtre Carbone composite fibré.

- Contrôlé sur PC indépendant LabView (Windows) et SPEC (Linux interface utilisateur).
- Logiciel Fastec Image Vlewing Software.
- Applications Matlab Windows et LINUX fournies (Karsten Joensen, JJ X-ray systems ApS Danemark).



N°	PARAMETER	TEST CONDITIONS	RESULTS TO MEET	Réglage Haute résolution	Réglage Basse résolution
D- 1	Source optic distance (mm)	150			
D-2	Source - sample distance (mm)	1475			
D-3	Source - detector distance (mm)	3000			
D-4	Length of the optic (mm)	80			
D-5	Source - 1st pinhole (mm)	250			
D-6	1st pinhole - 2nd pinhole (mm)	690			
D-7	2nd pinhole - 3rd pinhole (mm)	485			
D-8	Diameter of 1st pinhole (mm)	0.4			
D-9	Diameter of 2nd pinhole (mm)	0.2			
D-10	Diameter of 3rd pinhole (mm)	0.65			
D-11	Diameter of the beamstop (mm)	4.0			
D-12	3rd pinhole - sample distance (mm)	50			
D-13	Sample - detector distance (mm)	1525			
D-14	Q_{min} (angstrom^{-1})		0.006613		**0.0117**
D-15	d_{max} (angstrom)		$\geq$ 950		**> 540**
D-16	Divergence (arcmin)		2.8		
D-17	FWHM at sample (mm)		0.32		**0.37**
D-18	FWHM at detector (mm)		1.07		**1.73**
D-19	FW10% at sample (mm)		0.58		
D-20	FW10% at detector (mm)		1.78		
D-21	Expected Flux on sample (cps)		9.35^{E}+06		**9 10^{7} photons/second**
D-22	**Guaranteed flux (cps)**		**8.00^{E}=06**		
D-23	Maximum Kβ/Kα ratio		1.36E-04		