

Thin film based Optical Elements for Analytical X-ray Applications

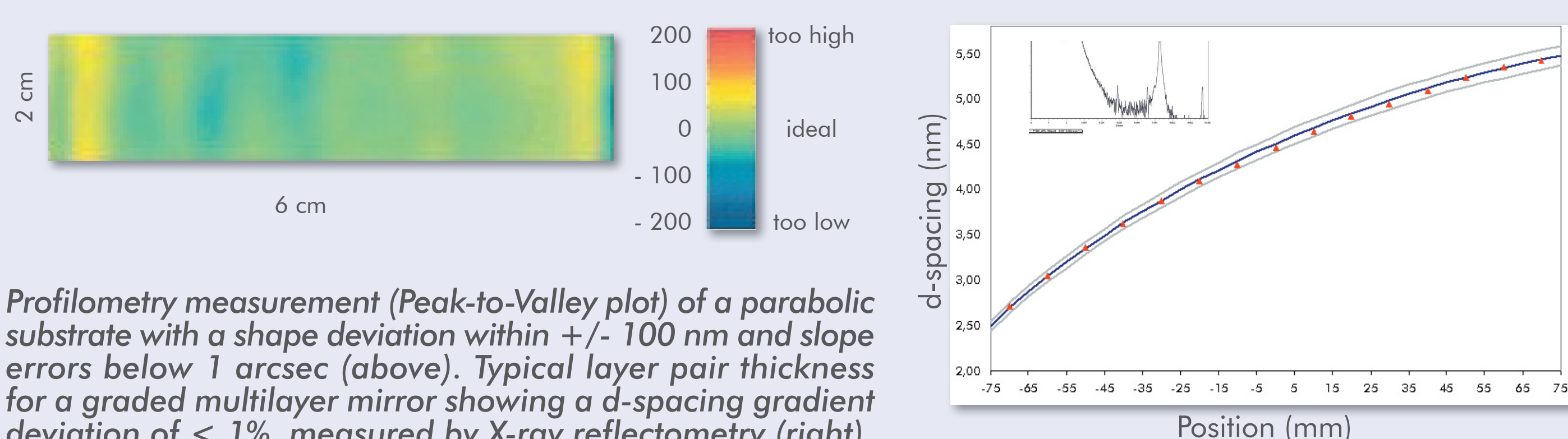
Jörg Wiesmann¹, Frank Hertlein¹, Uwe Heidorn¹, Carsten Michaelsen¹, Karol Vegso², Martin Hodas², Peter Siffalovic²

¹Incoatec GmbH, Max-Planck-Str. 2, 21502 Geesthacht, Germany; ²Institute of Physics, Slovak Academy of Sciences, Dubravska cesta 9, 845 11 Bratislava, Slovakia

We give an overview on developments in multilayer coatings up to 500 mm and Montel optics for Synchrotron applications and for microfocus high brilliance x-ray sources in the home-lab. We present how multilayers and thin films can be characterized in-situ during their deposition by the method of grazing incidence small-angle X-ray scattering (GISAXS). This kind of experiments was done only at synchrotrons. With modern microfocus sources like the μ S it becomes feasible in the home-lab. Further experimental set-ups demonstrate the versatility of the μ S.

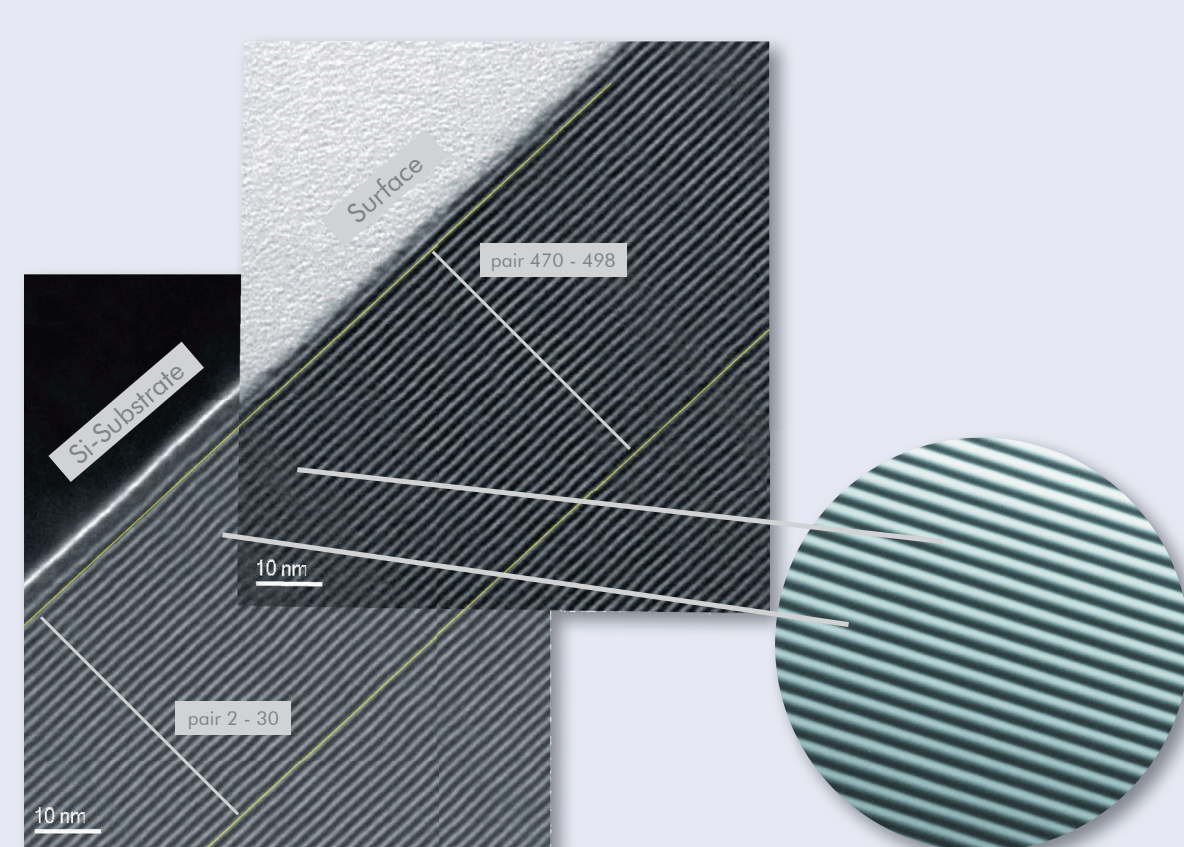
Multilayer Mirrors for Home Lab Sources and Synchrotrons

Incoatec designs and manufactures X-ray optics with properties, which are optimized for individual applications. The multilayer materials, the layer thickness profile and the substrate shape are designed by simulation with ray tracing methods. Our X-ray optics for lab instruments consist of graded multilayers which are deposited by magnetron sputtering. The precision is usually within $\pm 1\%$ of the d spacing for standard optics and up to of $\pm 0.2\%$ for high performance mirrors.



Profilometry measurement (Peak-to-Valley plot) of a parabolic substrate with a shape deviation within ± 100 nm and slope errors below 1 arcsec (above). Typical layer pair thickness for a graded multilayer mirror showing a d-spacing gradient deviation of $< 1\%$, measured by X-ray reflectometry (right).

As standard substrates bent silicon wafers are used which are glued onto backing plates and show slope errors of about 5-10 arcsec. For high-end applications (e.g. high resolution XRD, synchrotron applications, applications with high brightness microfocus sources), we use prefigured substrates which achieve slope errors below 1 arcsec. During the development phase of multilayer optics the method TEM (transmission electron microscopy) is applied.

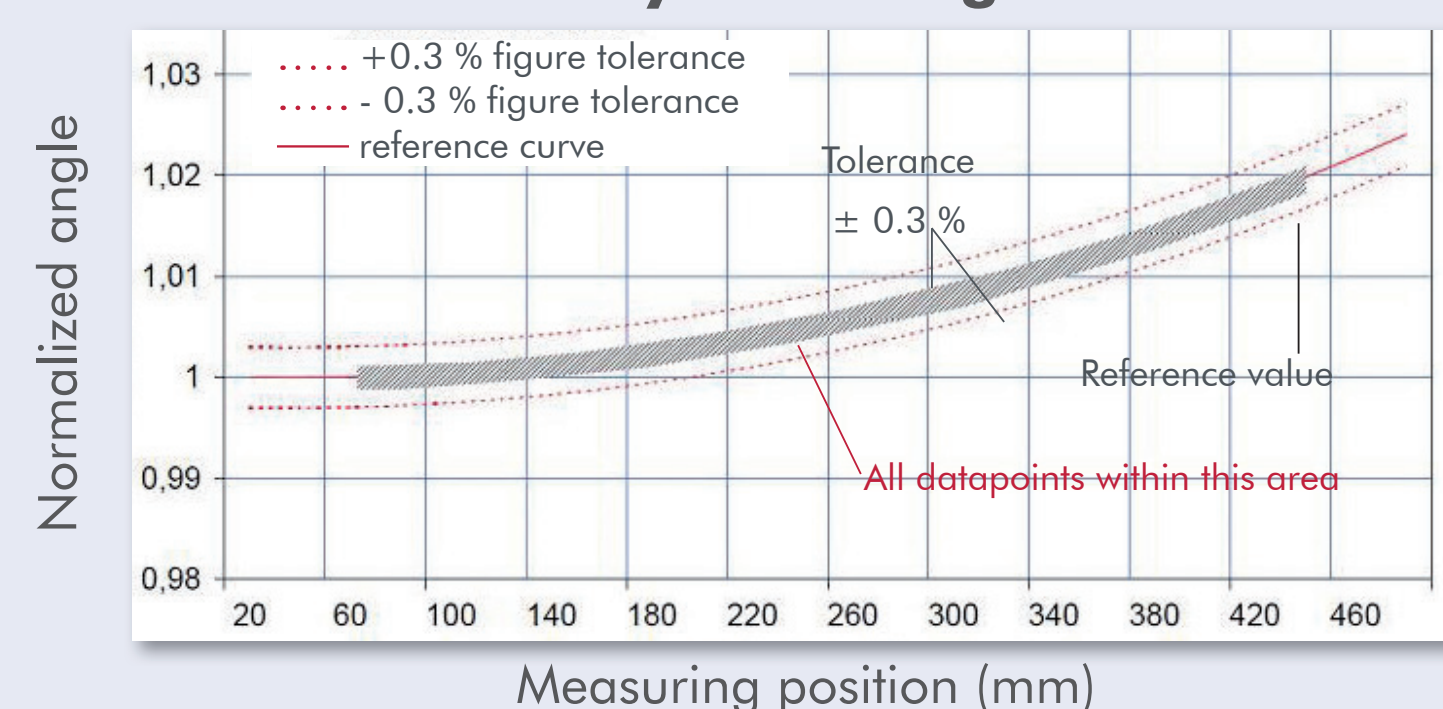


TEM image of a multilayer with 500 pairs of layers. (Prof. Jäger, University of Kiel)

Synchrotron Optics

Incoatec offers different types of X-ray optics for synchrotron and XFEL applications. In our deposition facilities, we can coat substrates of up to 150 cm in length and have experience with more than 40 different types of layer materials.

50 cm Graded Multilayer Coating with 200 Pairs



Multi-stripe Multilayer Optics

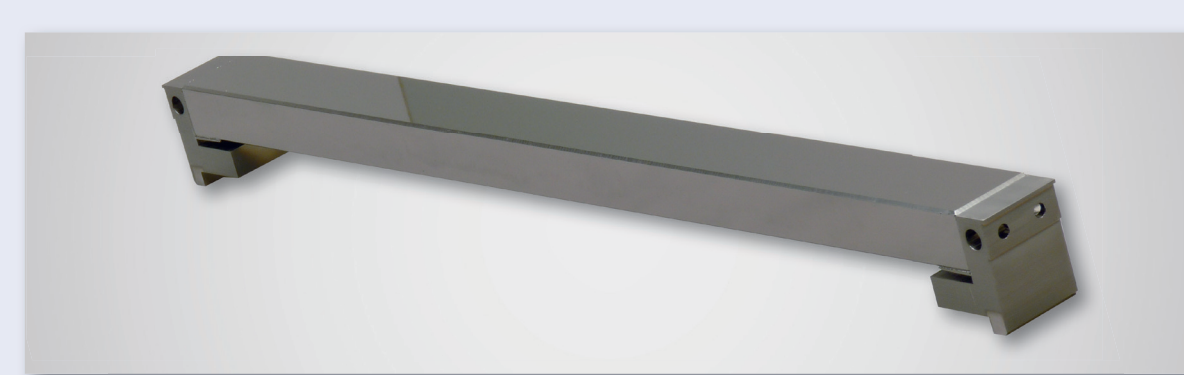


Three-stripe multilayer optics for tomographic microscopy and coherent radiology (TOMCAT at SLS; M. Stamparoni, PSI).

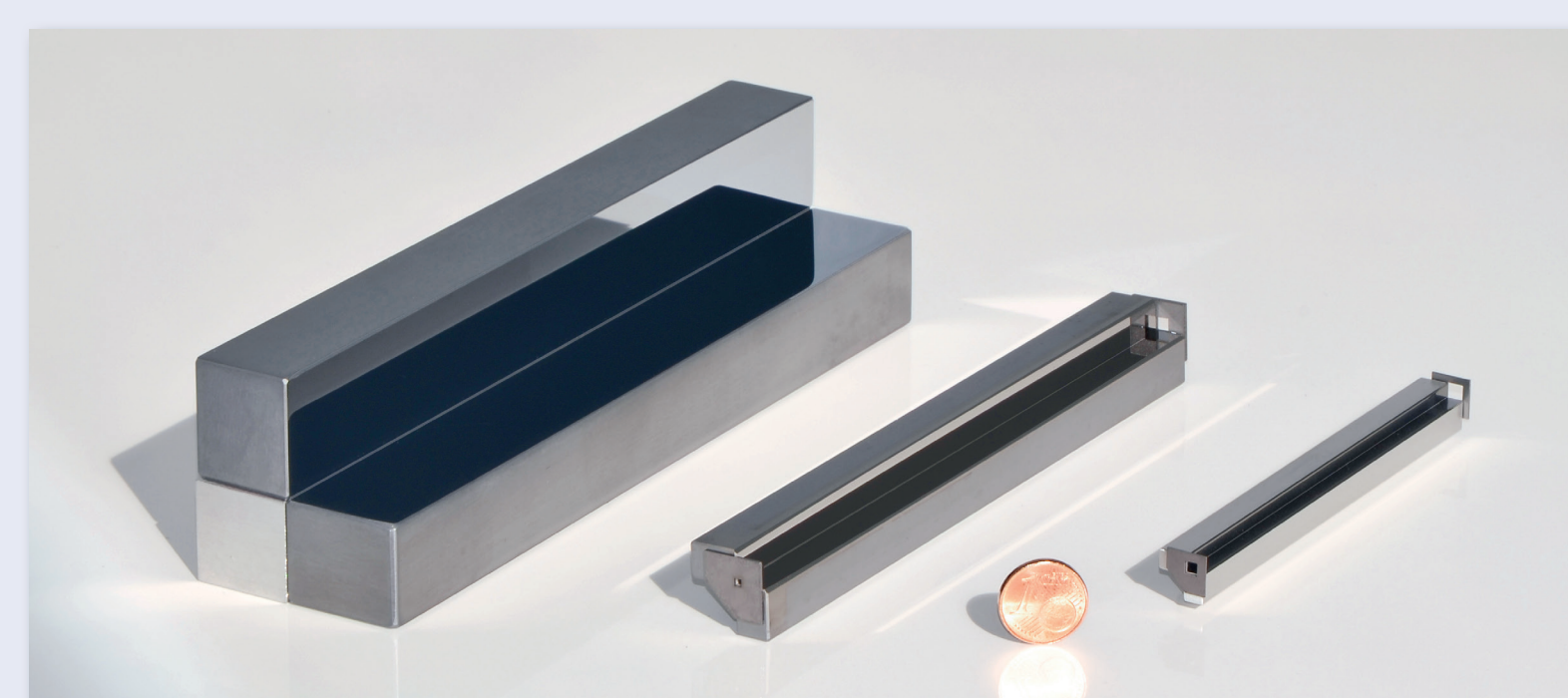
Stripe A: [Ru/C] 100, $d=4$ nm, $\Gamma=0.5$, $R > 80\%$ for $10 < E < 22$ keV

Midspace: Si $\langle 111 \rangle$, slope error 0.04"

Stripe B: [W/Si] 100, $d=30$ Å, $\Gamma=0.5$, $R > 80\%$ for $22 < E < 45$ keV



Bendable 400 mm silicon mirror



Montel Optics - 10-15 cm in length

Different cross sections from 4 x 4 cm to 1 x 1 cm; some were sold to NSLS and DLS. They are used at inelastic scattering beamlines.

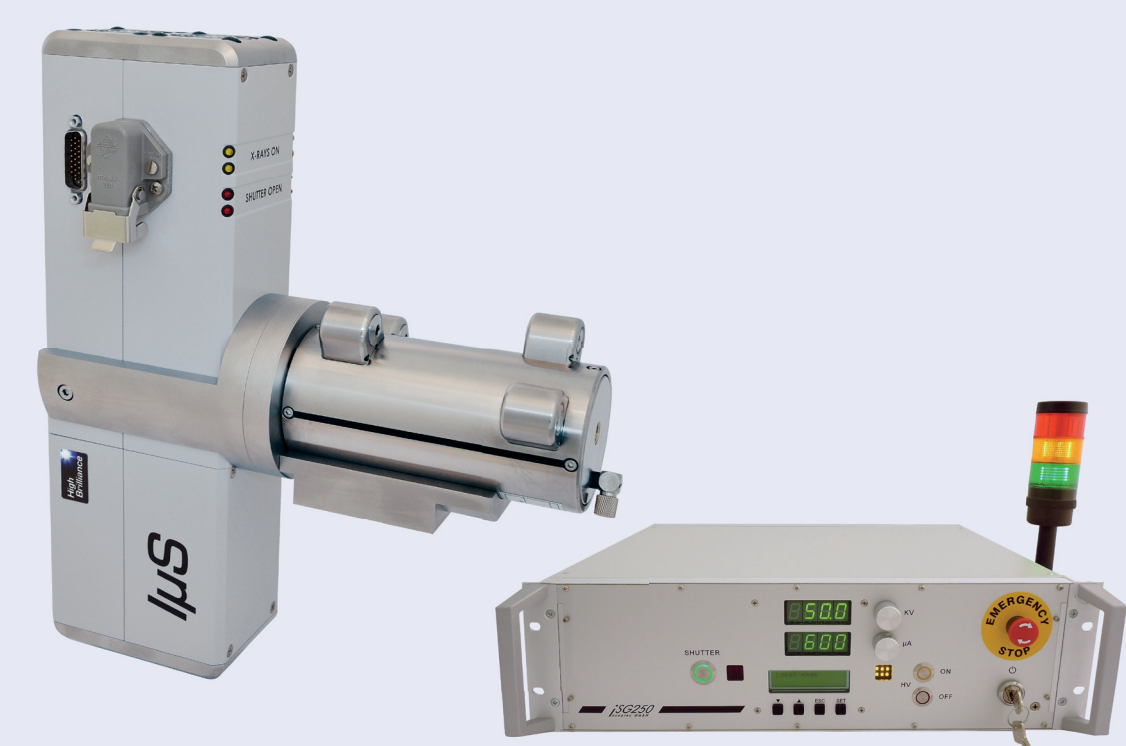
Incoatec Microfocus Source μ S for X-ray Diffraction

The μ S is a low power air cooled X-ray source for diffractometry applications. The source is equipped with a Montel optics. Montel optics consist of two mirrors mounted side-by-side in an L-shape enabling a 2-dimensional beam shaping. Therefore, we can form either a highly collimated beam with a low divergence (below 0.5 mrad) or a focusing beam with higher divergence (up to 10 mrad) and very small focal spots at the sample (diameters down to 100 μ m). The Cu- μ S with collimating optics can be used for (GI)SAXS and X-ray diffraction studies. With focusing optics experiments can be carried out in transmission geometry, especially in powder diffraction applications. With the Mo- and Ag- μ S highly absorbing and radiation-damage sensitive materials can be investigated. Consequently, these sources are often used for chemical crystallography and become more and more interesting for investigation of soft matter samples or for XRD measurements during the growth of nanosized materials.

Upgrading Existing Diffractometers with the Microfocus Source μ S

You have a Bruker AXS, Marresearch, Nonius, Rigaku, Huber or some other system?

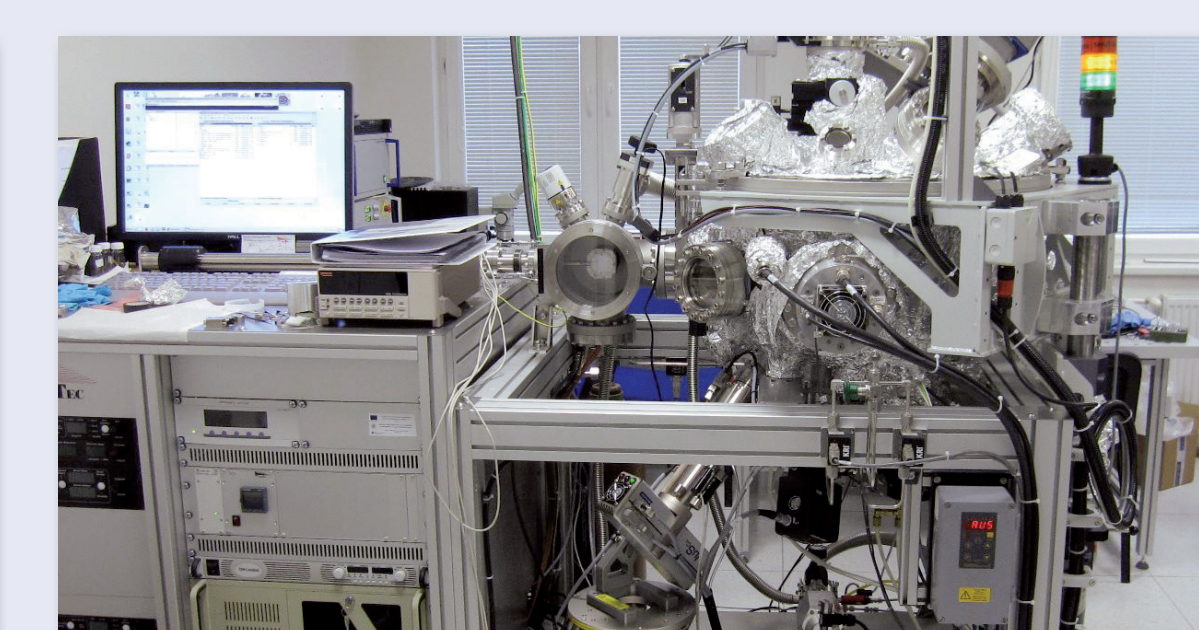
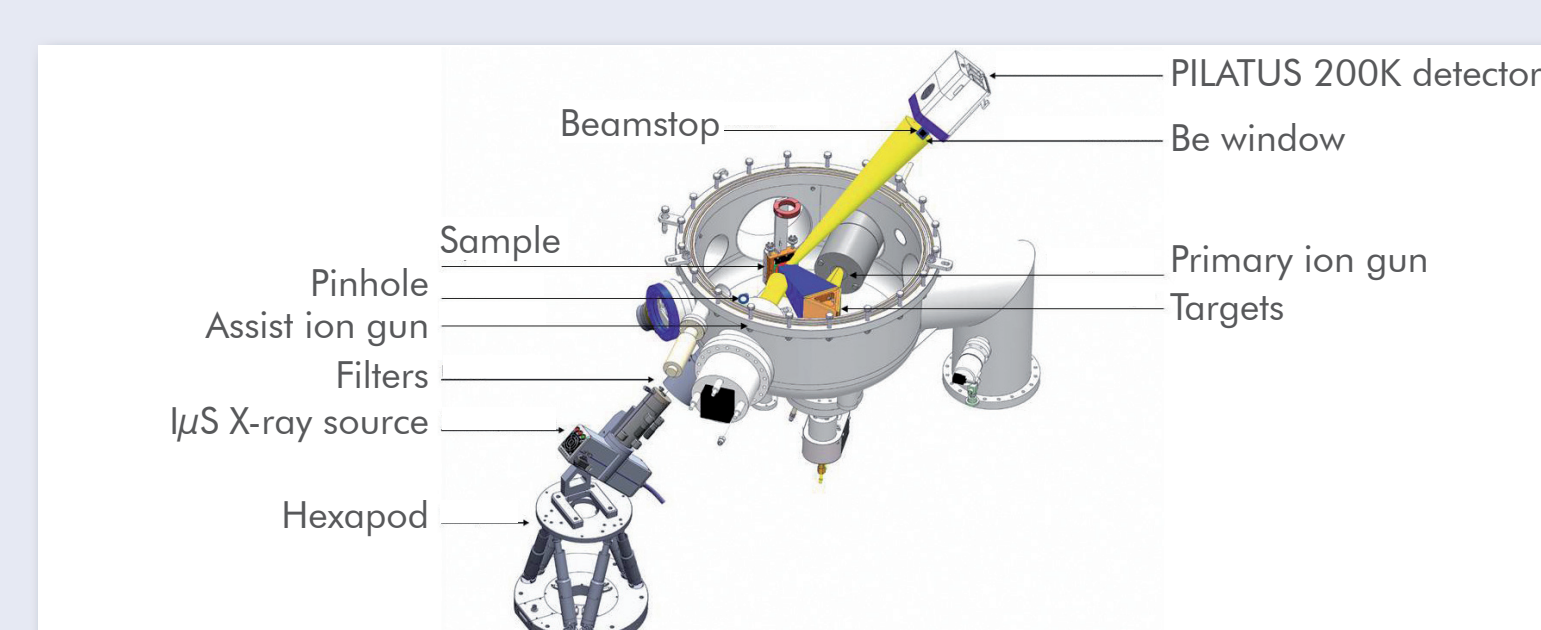
Incoatec offers a unique possibility to upgrade your existing diffractometer by installing the high-performance, air-cooled and low-power microfocus source μ S.



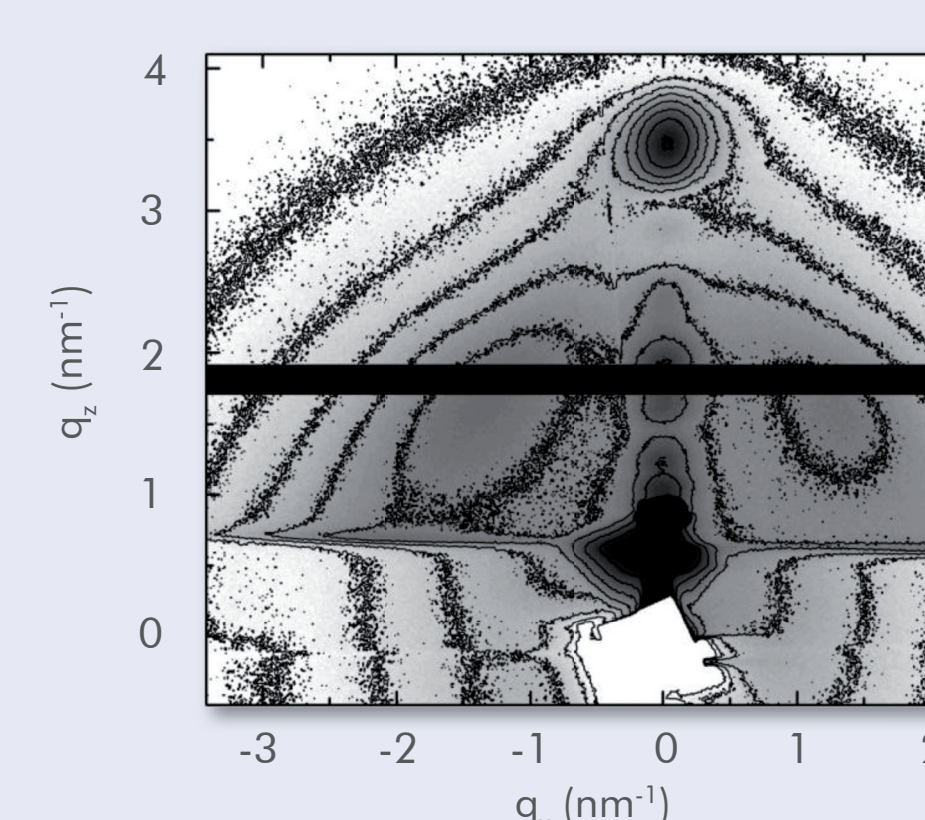
In-situ GISAXS during Thin Film Growth

By using in-situ GISAXS in the home-lab we investigated how multilayers grew during thin film deposition. This kind of experiments is typically done only at synchrotrons. With an μ S it is now also feasible in the home-lab.

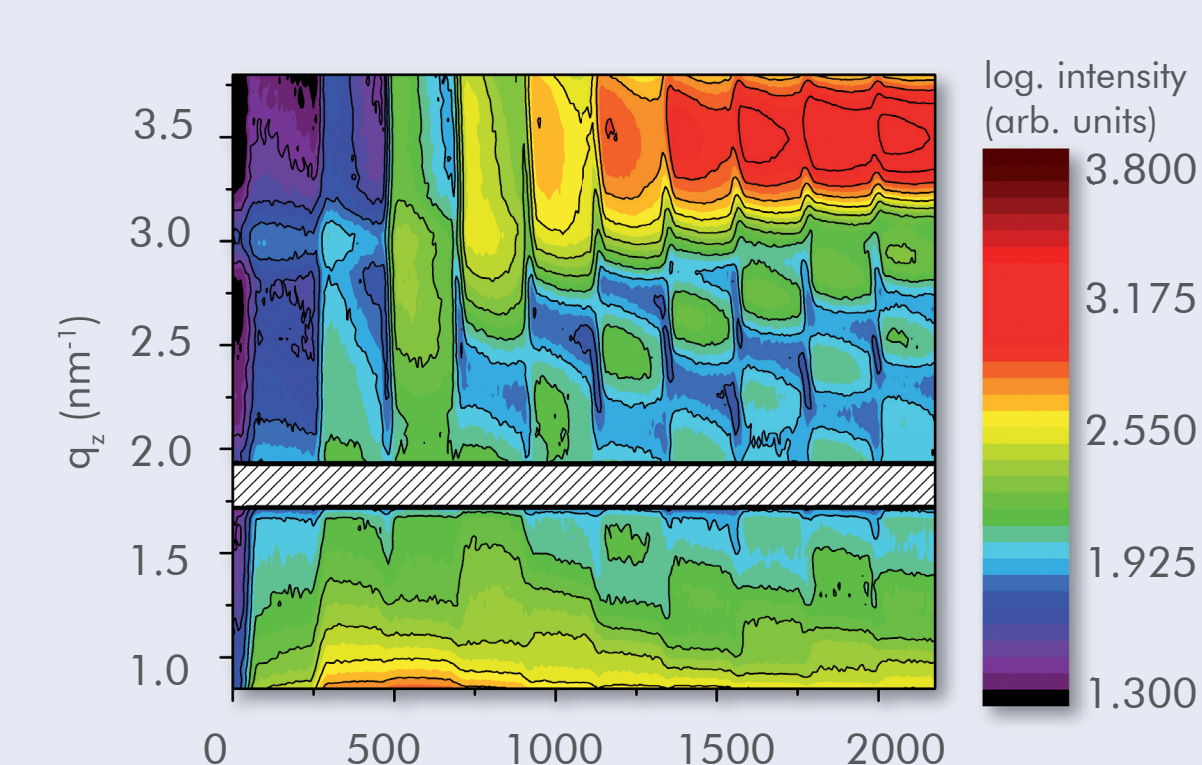
μ S for in-situ GISAXS with Pilatus 200K



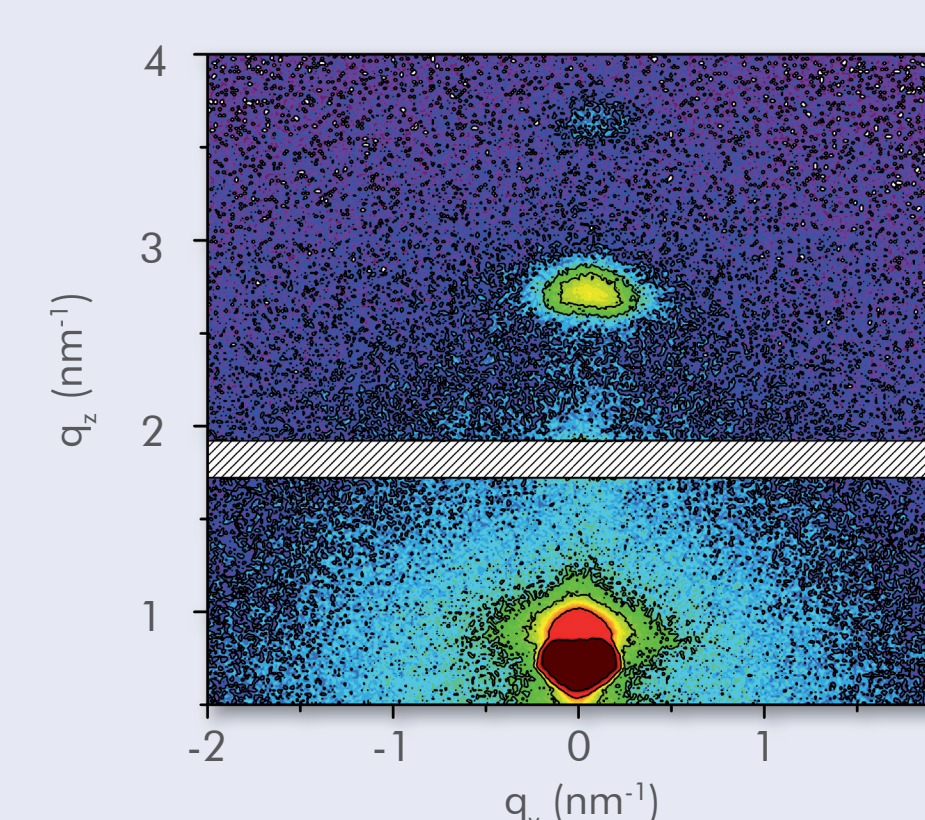
The Dual Ion-beam sputtering unit in Bratislava was upgraded with an in-situ GISAXS set-up. As a source an μ S with a special collimating optics for SAXS is mounted on a Hexapod. Together with the 2-dim detector Dectris Pilatus 200K dynamic measurements during thin film growth become feasible.



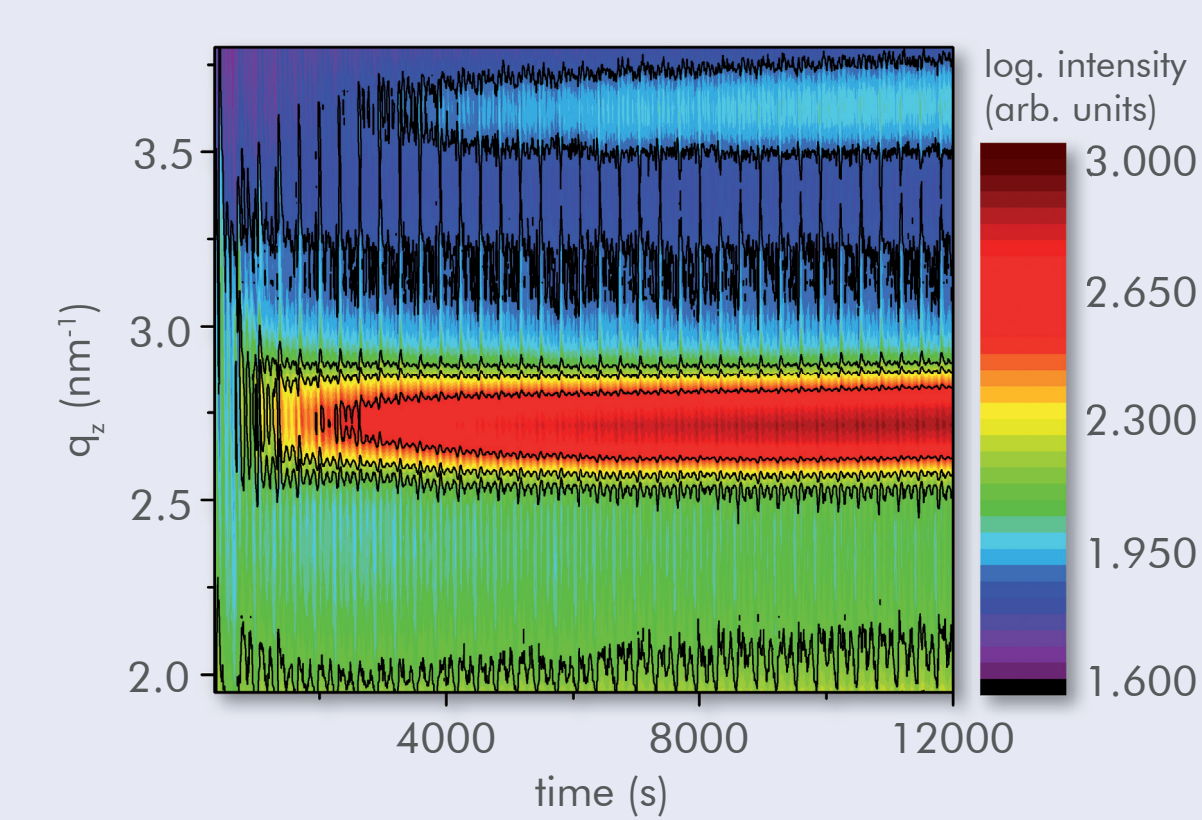
Reciprocal space map of 10 periods W/B₄C multilayer mirror with 1.5 nm period thickness measured ex-situ by GISAXS in deposition chamber



Time resolved evolution GISAXS reciprocal space map of the 10x W/B₄C multilayer mirror with visible Bragg peak and Kiessig fringes



GISAXS reciprocal space map of the 40x Mo/Si multilayer mirror with period 6.9 nm

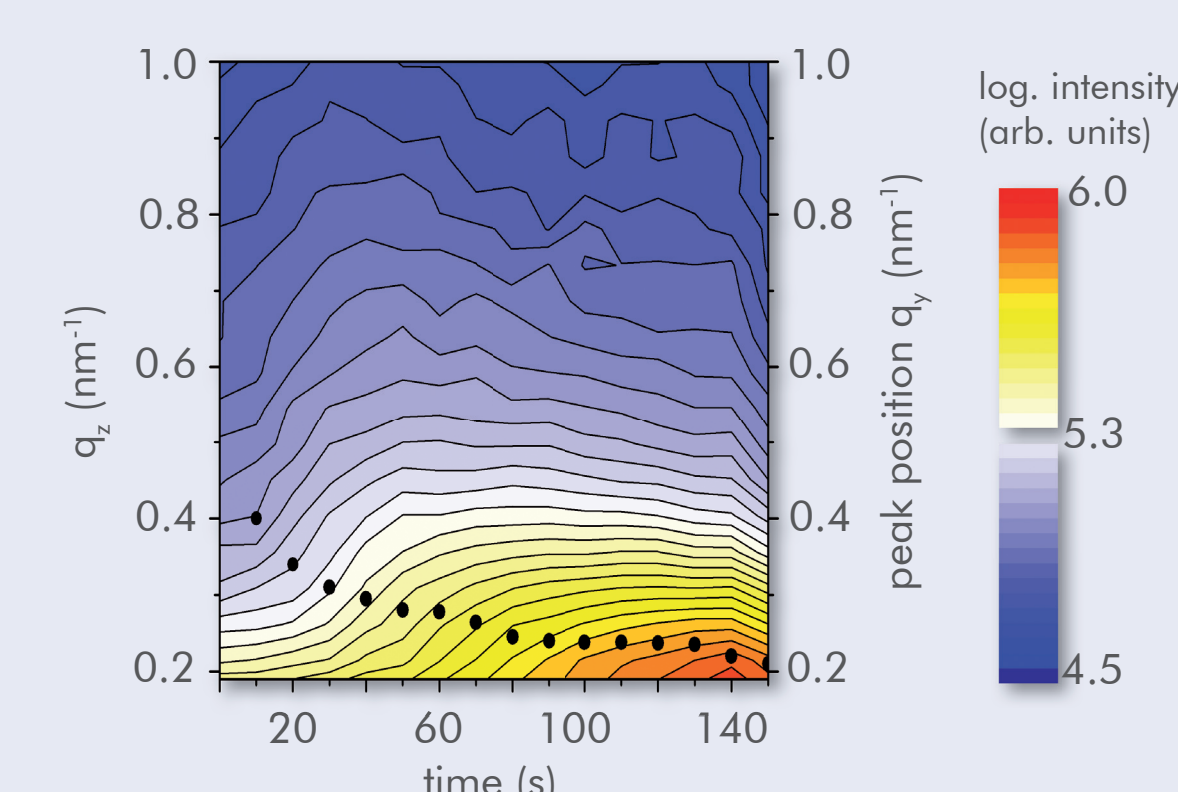


Time resolved evolution GISAXS reciprocal space map of the 40x Mo/Si multilayer mirror with visible Bragg peaks

GISAXS plots show the perfect growth of the multilayers. Even thin films with a total thickness in the range of 15 nm could be measured. The time resolved evolution of the specular signal enables the measurement of the Bragg peaks and the Kiessig Fringes dynamically.

NEW and in progress: In-situ time-resolved GISAXS of metal films on graphene

- This method revealed kinetics of Cu cluster growth on CVD graphene.
- It allows rapid optimization of metal deposition processes in laboratory conditions.
- Further growth studies of Au, Ag,..., on graphene surface are in progress.



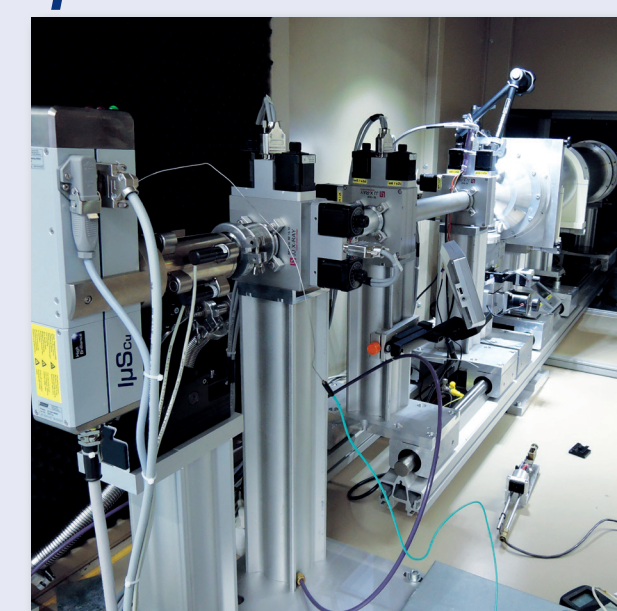
Your upgrade benefits:

- No maintenance, only single phase power and no water cooling required
- 3 years warranty
- Maximum installation down time of only 2-4 days
- Full integration into existing safety circuits for Bruker equipment, new safety concept development on request
- Full compliance with European Machinery Directive 2006/42/EC

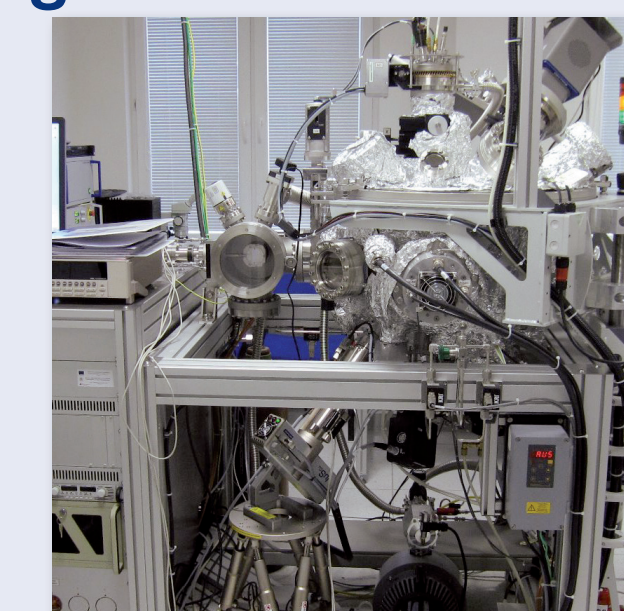
Your upgrade options:

- Source, optics and scatterless slits
- Single source upgrade for XRD, SCD, (GI)SAXS, XRR and many more applications
- Dual wavelength setup by adding μ S as complementary source
- Cu, Mo, Ag, Co and Cr radiation (others on request)

μ S and SCATEX upgrades on SAXS systems



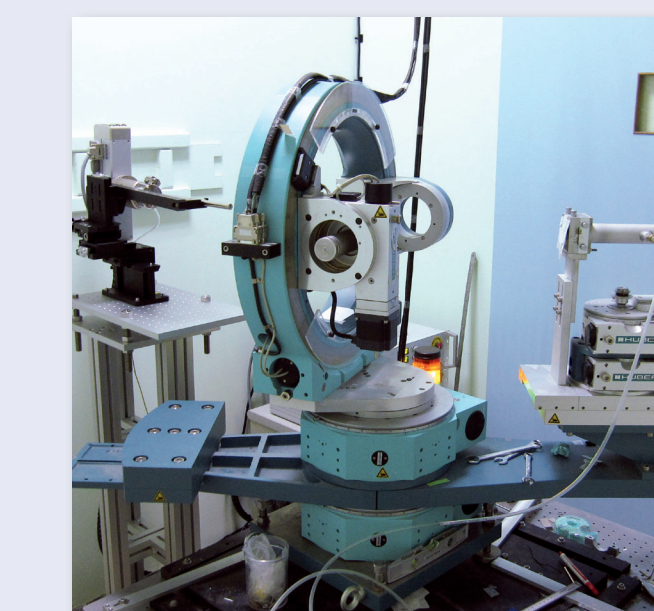
μ S and SCATEX upgrade on a customized SAXS setup in Hamburg



Adaptation to UHV deposition chamber for in-situ GISAXS studies in Bratislava, Slovakia



Replacement of Rigaku RU-200 generator in Boulder, USA



Huber system for SAXS in Tamkang, Taiwan