In-situ Grazing Incidence Small-Angle-Xray-Scattering Studies Using High Brilliance Microfocus Sources

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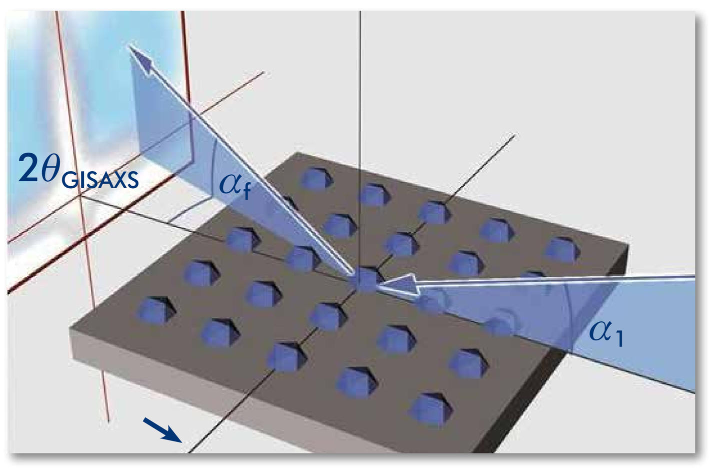
Grazing Incidence Small Angle Scattering (GISAXS)

Measures diffuse scattering in reflection geometry at grazing incidence (α_i) and grazing reflectance (α_f) angles



Diffuse scattering contains information about
- surface and subsurface structure
- island dimensions and 3-D arrangements
- roughness
- pore diameter

Advantages:
- non-destructive, allowing inspection of buried clusters and interfaces
- in contrast to microscopy average over sample surface/volume
- working under all conditions (enables in-situ screening/control)
- changing the incidence angle controls the penetration depth

Disadvantages:
- transformation from reciprocal to real space needs an appropriate model
- we always measure statistical averages over ensemble of entities

The potential of our microfocus source IμS in GISAXS studies in the home-lab is demonstrated in an overview of representative experimental setups and results. The studies take advantage of the brilliance and outstanding beam quality of the low-maintenance IμS. It is shown how the IμS can be used to achieve excellent results in the investigation of in-situ thin film deposition in UHV chambers by using GISAXS or of the structure of oriented two-dimensional liquid crystalline samples.

Upgrading Existing Diffractometers with the Incoatec Microfocus Source IμS

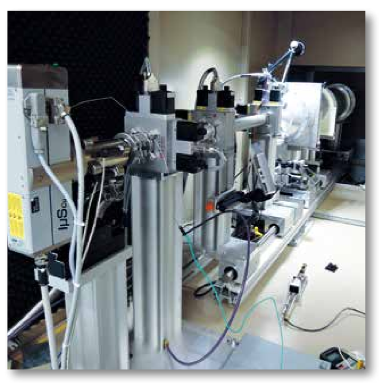
You have a Bruker AXS, Marresearch, Nonius, Rigaku, Huber or some other system?

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

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
- 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, XRR, (GI)SAXS and many more applications
- Dual wavelength setup by adding IμS as complementary source
- Cu, Mo, Ag, Co and Cr radiation (others on request)

The three generations: IμS, IμS High Brilliance and IμS 3.0



IμS + SCATEX upgrade on a customized SAXS setup in Hamburg

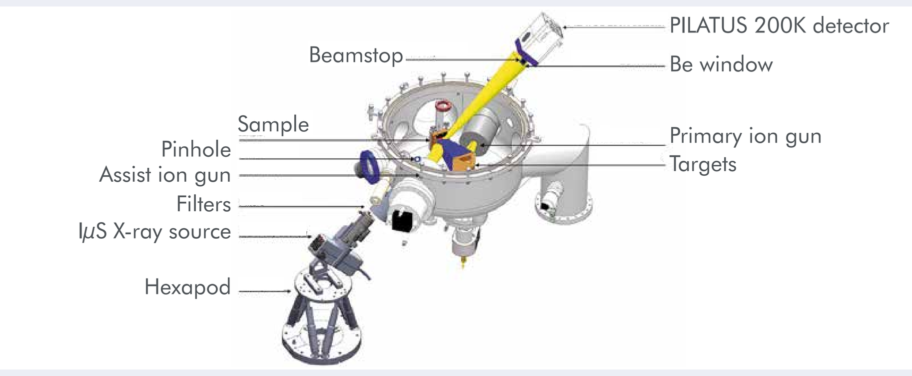
Replacement of Rigaku RU-200 generator in Boulder, USA

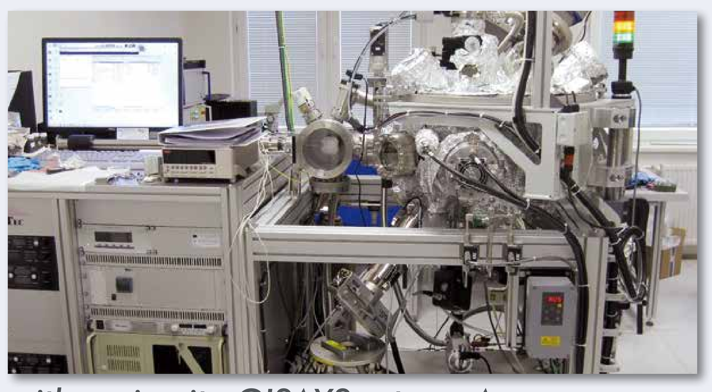
Huber system for SAXS in Tamkang, Taiwan

In-situ GISAXS during thin film growth

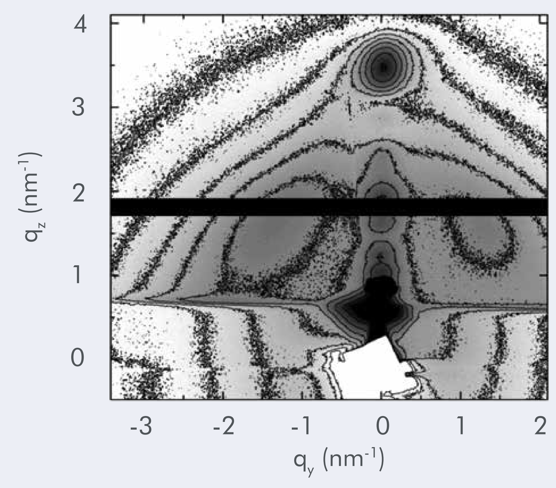
By using in-situ GISAXS in the home-lab we investigated how multilayers grew during thin film deposition. This kind of experiments is typically done only at synchrotrons. With an IμS it is now also feasible in the home-lab.

IμS for in-situ GISAXS with Pilatus 200K

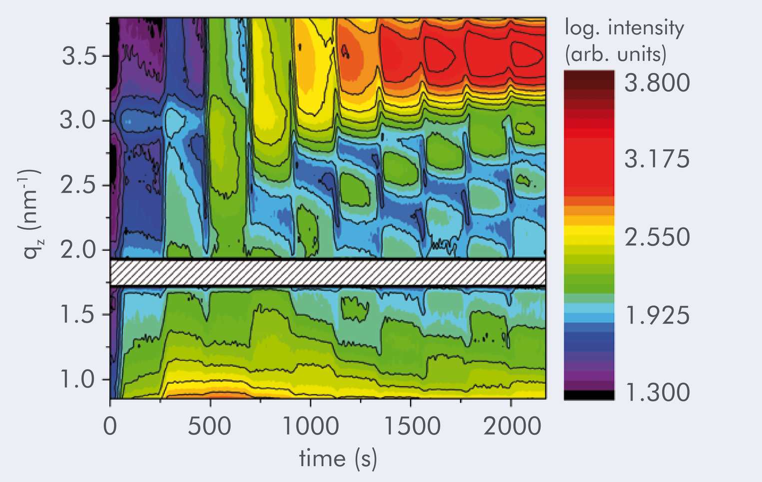




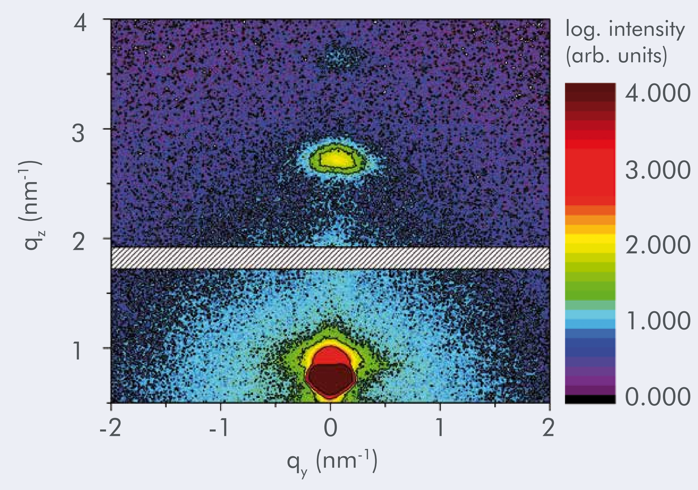
The Dual Ion-beam sputtering unit in Bratislava was upgraded with an in-situ GISAXS set-up. As a source an IμS with a special collimating optics for SAXS is mounted on a Hexapod. Together with the 2-dim detector Dectris Pilatus 200K dynamic measurements during thin film growth become feasable.



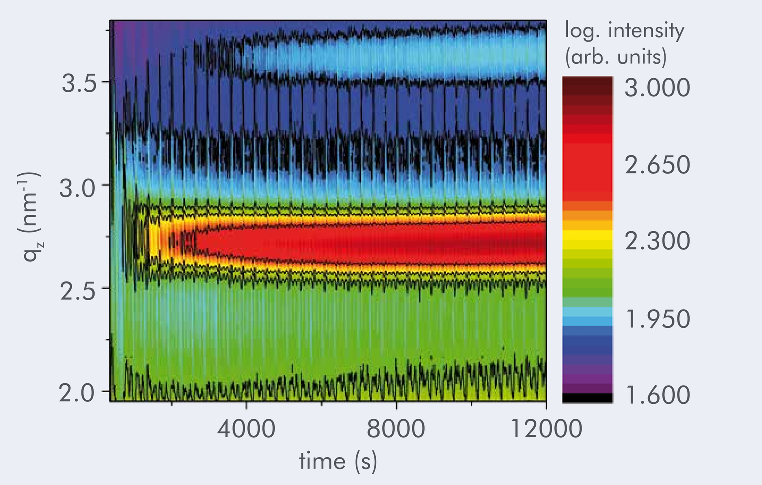
Reciprocal space map of 10 periods W/B4C multilayer mirror with 1.5 nm period thickness measured ex-situ by GISAXS in deposition chamber



Time resolved evolution GISAXS reciprocal space map of the 10x W/B_4C multilayer mirror with visible Bragg peak and Kiessig fringes



GISAXS reciprocal space map of the 40 x 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

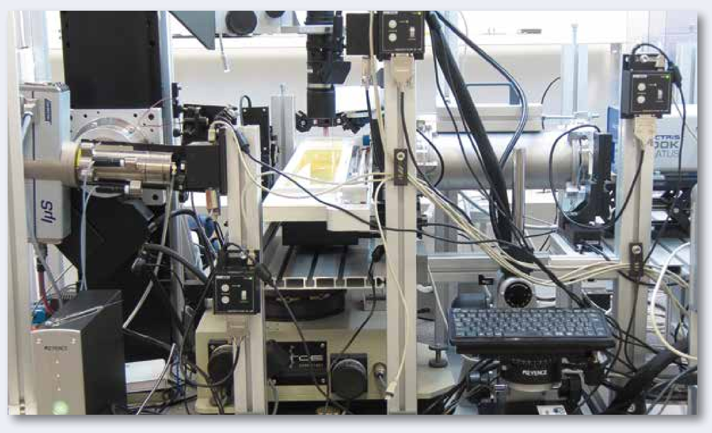
GISAXS plots show the perfect growth of the multilayers. Even thin films with a total thickness in the range of 15 nm could be measured. The time resolved evolution of the specular signal enables the measurement of the Bragg peaks and the Kiessig Fringes dynamically.

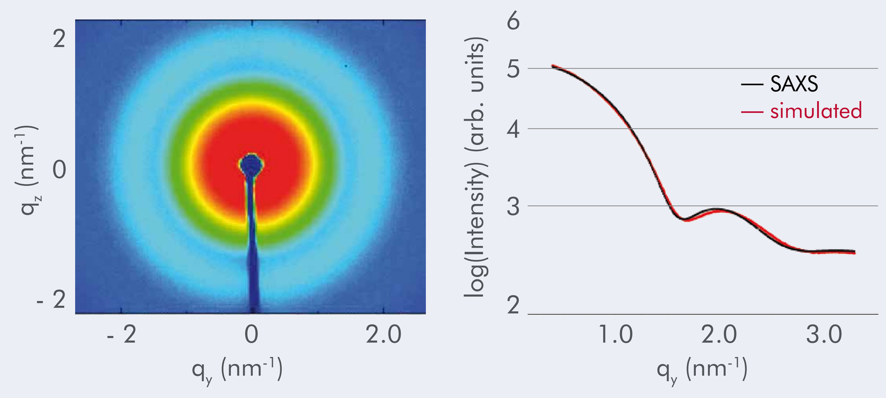
In-situ GISAXS of Nano-particles on liquid surfaces

GISAXS measurements were performed with the IμS/Dectris system at the Institute of Physics at the Slovak Academy of Science inBratislava. Silver particles on a langmuir film were analyzed at different surface pressures which were applied by means of a reduction of the surface area.

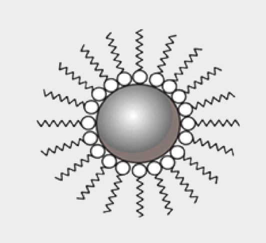
IμS with Dectris pixel detector

For rapid grazing incidence small-angle-scattering measurements (GISAXS) of liquid samples our IμS equipped with an optics with 5mrad divergence was combined with a Dectris Pilatus detector. The set-up including the alignment can be changed from liquid to capillary samples in less than 30 min.

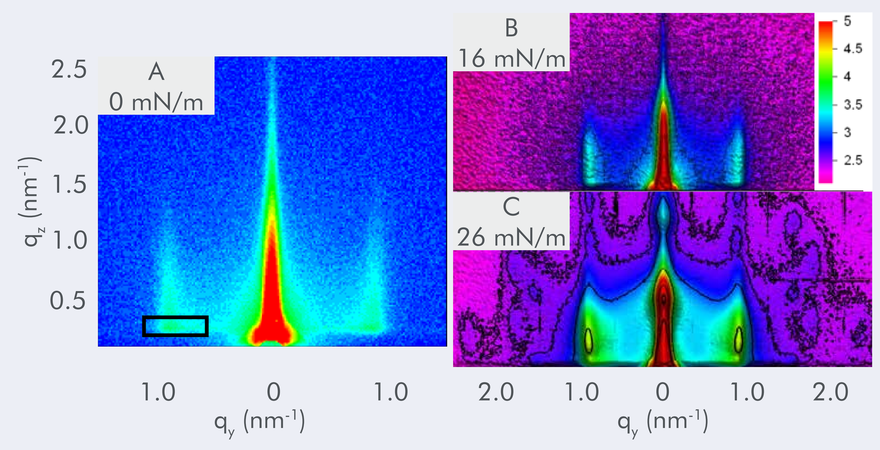




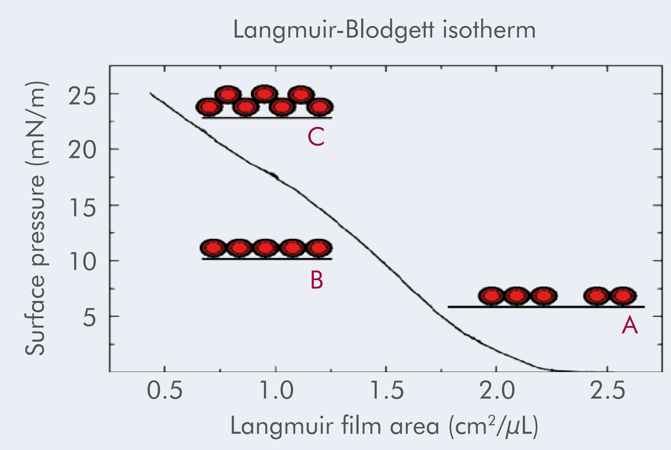
Analyzing the particle diameter: SAXS of diluted solution of Ag nanoparticles.
Result: Metal core, radius = 2.9±0.3 nm



Experimental for GISAXS:
- angle of incidence: 0.2 deg
- 180 s measurement time
- aperture 350 μm
- the surface was pressed with 0 up to 26 mN/m



SAXS patterns at three different surface pressures:
A) 0 mN/m: unpressed
B) 16 mN/m: intensity increases
C) 26 mN/m: crystal-like peaks appear

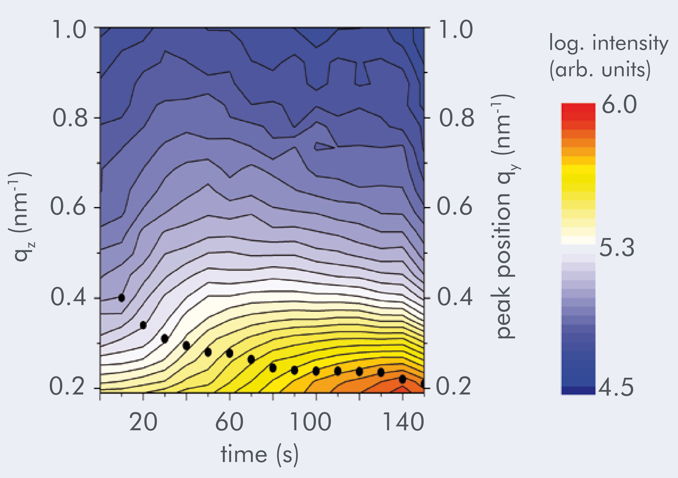


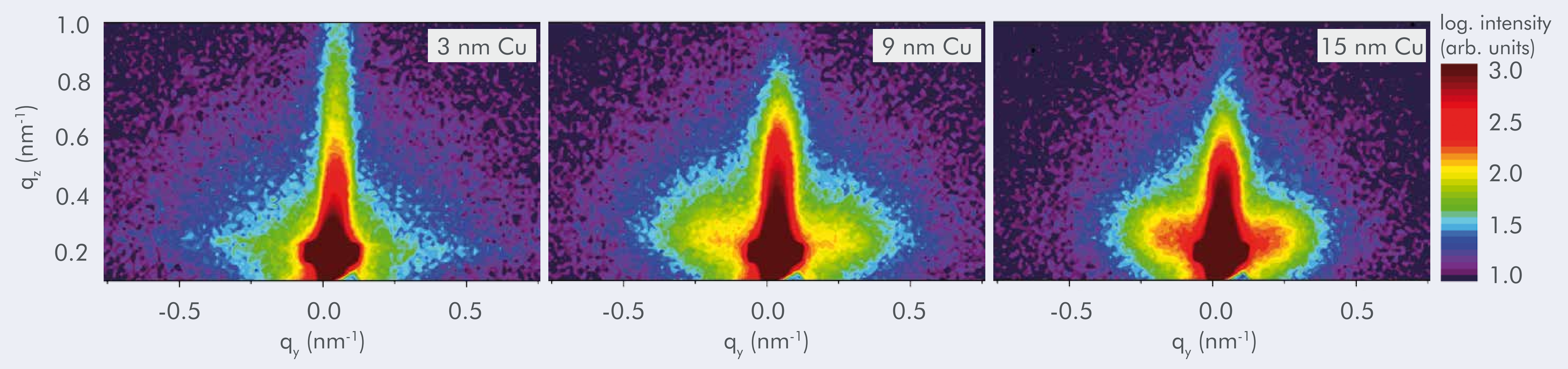
A) Unpressed surface: islands of nanoparticles are swimming on the surface without connection
B) Increasing surface pressure: islands coalescence
C) vertical formation of hexagonal layers

Ordering phenomena could be observed in-situ during a change of surface pressure. The particles were transformed from single islands to an almost vertically ordered structure of connecting particles.

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.





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