GISAXS with Nanoparticles on Liquids and with Multilayer Films on a Lab Source

Jörg Wiesmann[1], Bernd Hasse[1], Carsten Michaelsen[1], Peter Šiffalovič[2], Karol Végsö[2], Eva Majková[2], Jozef Keckes[3], Günther Maier[4]

[1] Incoatec GmbH, Geesthacht, Germany; [2] Slovak Academy of Sciences, Bratislava, Slovakia; [3] Österreichische Akademie der Wissenschaften, Leoben, Austria; [4] Material Center Leoben, Leoben, Austria

The Microfocus Source IµS is a versatile device providing a very high X-ray flux within a small spot size. With this lab-source we have collected (GI)SAXS data of outstanding quality on liquid and thin-film samples with structures in the nanometer scale. In conclusion the IµS now enables (GI)SAXS measurements in the home-lab with a quality which was in the past only reachable at Synchrotrons. With the new IµS$^{\text{High Brilliance}}$ the flux could be increased by 30% for Cu, 50% for Ag and 60% for Mo radiation.

Incoatec Microfocus Source IµS and 2D Montel Optics

The IµS is the perfect upgrade for older equipment in X-ray diffractometry. It combines the advantages of a sealed-tube system with the superior data quality of conventional rotating anode systems. The IµS is also included in the new equipment of Bruker AXS: for SAXS in the NANOSTAR and for XRD in the D8 family with 2-dim detectors. The IµS consists of a microfocus source and a specially designed multilayer optics named Quazar.

The source

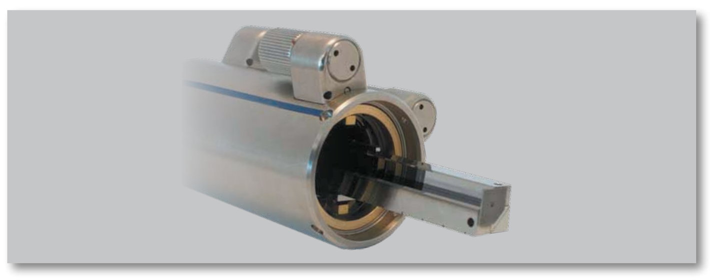
- high brilliance & best spectral purity
- Cu, Mo, Cr and Ag radiation available
- air cooled
- < 40 µm spot size at the anode
- convenience of sealed-tube and three-year warranty
- high voltage generator

Quazar Optics

- 2-dim beam shaping
- collimating, focussing or collimating + focusing (hybrid)
- patented housing for high stability and easy alignment
- motorized alignment (optional)

Optics	Divergence (mrad)	Beam size (mm)	Flux (10^8 ph/s)
ELM23	5.1	0.25	> 7
ELM32	1.0	0.8	> 2.5
PAM42	0.5	2	> 4

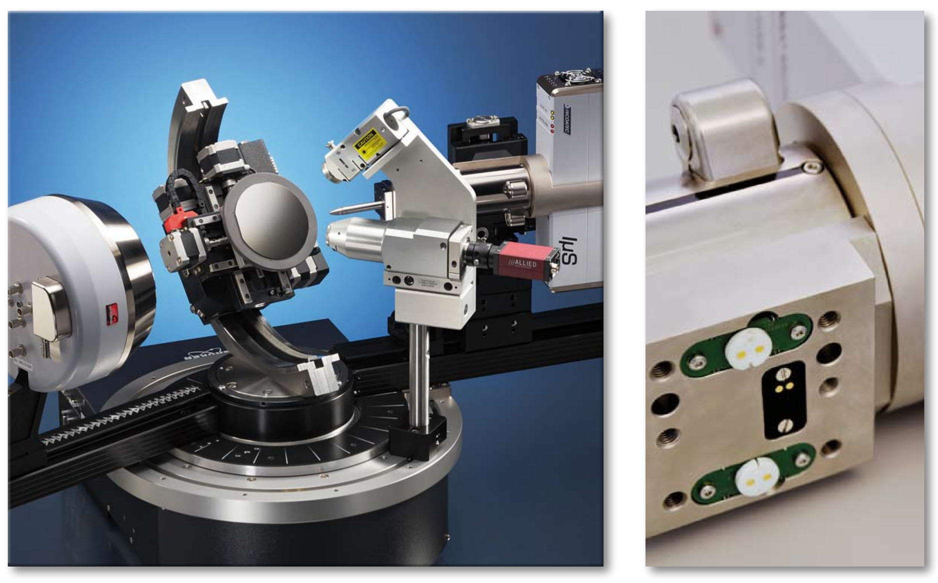
Different types of Quazar Optics for the IµS with Cu radiation for SAXS applications



Optics in high-stability housing

NEW with IµS$^{\text{High Brilliance}}$

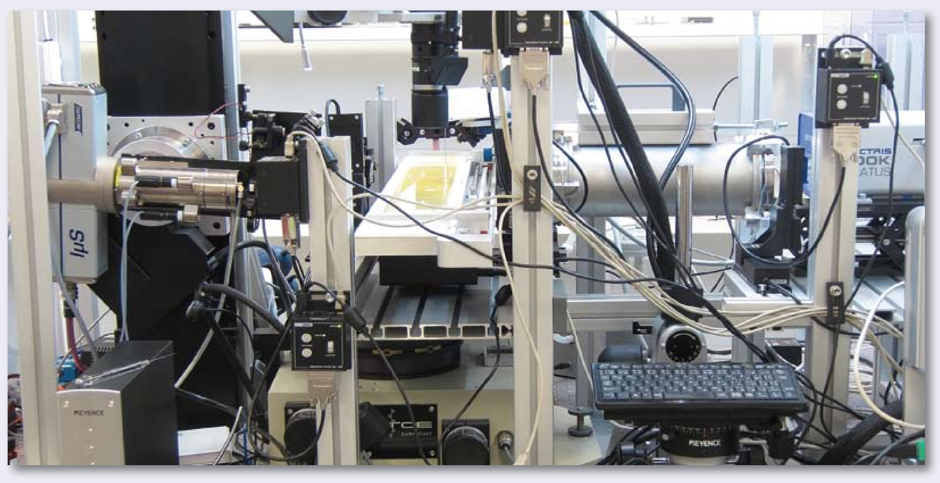
- up to 60 % more flux due to improved optical design
- for Cu, Mo & Ag
- component recognition
- improved safety features
- compliant with Machinery Directive 2006/42/EC



IµS integrated in a new Bruker D8 DISCOVER (left) and component recognition (right)

New IµS$^{\text{High Brilliance}}$ System with optics housing and 19" wide stand-alone generator

IµS with Dectris Pixel Detector



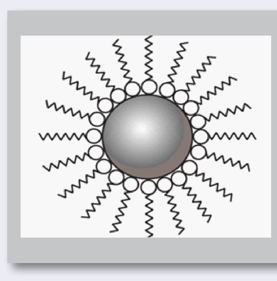
For rapid grazing incidence small-angle-scattering measurements (GISAXS) of liquid samples our IµS equipped with an ELM 23 optics was combined with a Dectris Pilatus detector. The set-up including the alignment can be changed from liquid to capillary samples in less than 30 min (picture and data: P. Šiffalovič, Slovak Academy of Science).

IµS with NANOSTAR

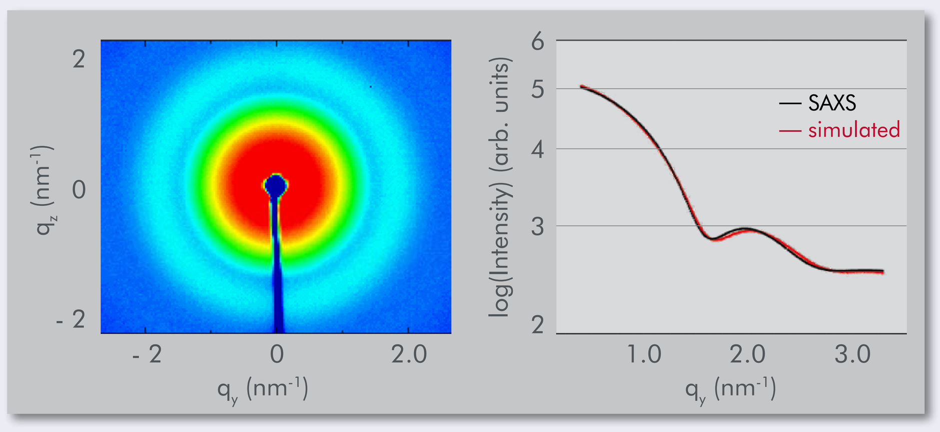
Incoatec´s Microfocus Source, the IµS, provides the Bruker AXS NANOSTAR with a high-flux, collimated primary beam combined with the convenience of a sealed tube. The NANOSTAR uses the VÅNTEC-2000 detector which enables virtually noise-free, real-time 2D detection. In combination with the IµS the NANOSTAR is designed like a synchrotron SAXS beamline and gives the opportunity to perform WAXS measurements simultaneously.

In-situ GISAXS of Nano-Particles on Liquid Surfaces

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



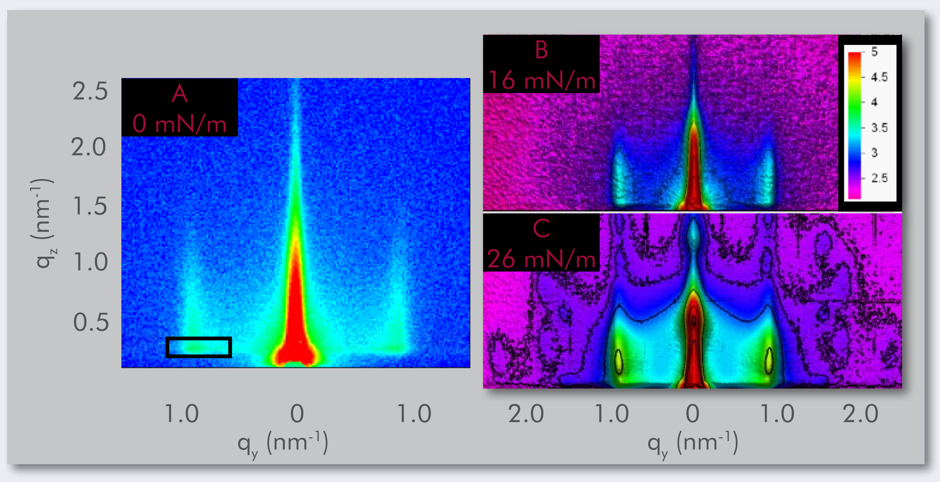
Sample: Langmuir film with silver nanoparticles: Organic enveleope (oleic-acid, oleylamin)



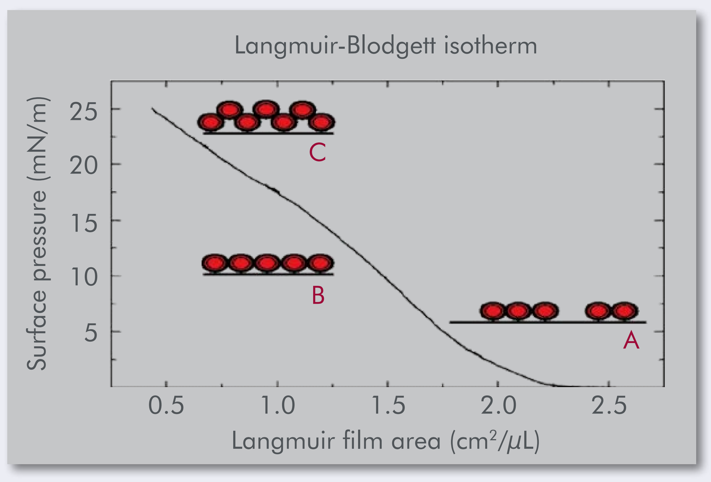
Analyzing the particle diameter: SAXS of diluted solution of Ag nanoparticles. Result: Metal core, radius = 2.9±0.3 nm

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

It was possible to study the formation process in-situ and time-resolved from unordered islands to ordered layers by increasing the pressure on the surface of the liquid film.

Home-Lab Small Angle X-ray Scattering

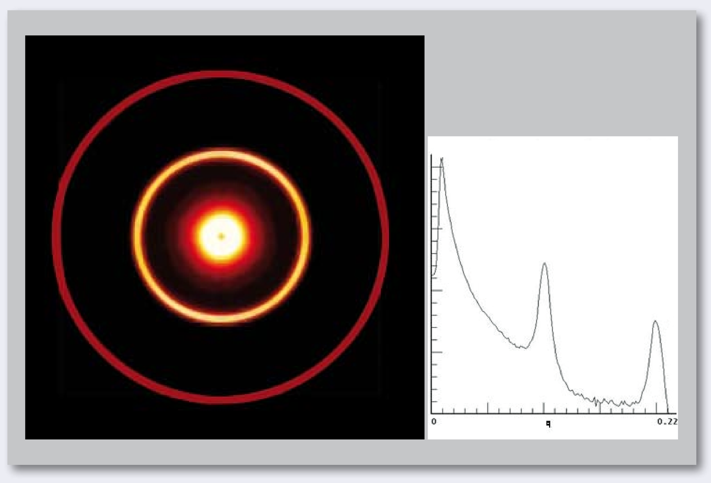
For the following measurements we used a NANOSTAR with IµS. The beam was collimated by a Quazar optics with a small divergence of 1mrad. A typical standard measurement shows the superior quality in comparison to former set-ups with standard sealed tubes.

Experimental for SAXS:

- 3-Pinhole-geometry
- 1035 mm detector distance
- 1000 s exposure time

Sample:
Silver behanate (H_3C-$(CH_2)_{20}$-COOAg)

Total integrated Intensity (left) and detector image with corresponding I-q plot (right):
With an IµS at 30W we achieved 17.000 cps.
The intensity of „old" Nanostar: equipped with sealed tube+ cc-Göbel mirrors at 1.4 kW was only 3.300 cps

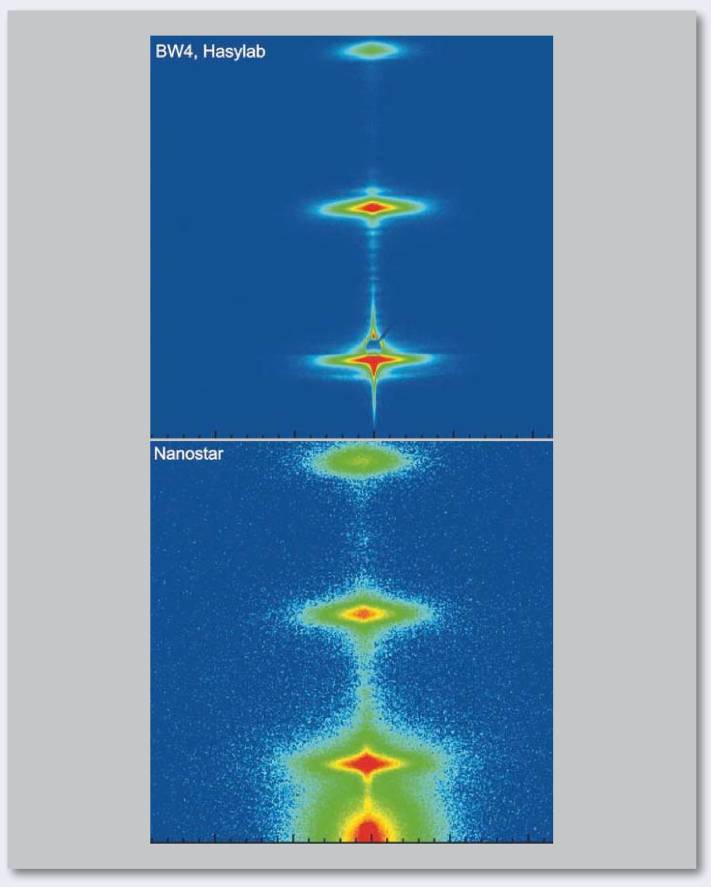


Grazing Incidence SAXS of Thin Films

The integration of the IµS into the NANOSTAR allows measurements, which usually need synchrotron facilities, to be performed in the home-lab. The figures show the results of a Mo/Si multilayer measured with the NANOSTAR and the synchrotron beamline BW4 at the Hasylab. Both measurements clearly show three Bragg sheets. In comparison the NANOSTAR results have a higher background and thus a lower resolution. However, all features required for data processing are visible.

This striking result gives the opportunity to plan future experiments without the restriction of beamline time. With an IµS the NANOSTAR enables a more efficient use of synchrotron beamline time and gives the opportunity to envision e.g. time- or temperature-resolved long-term experiments.

For more information see the publication:
P. Šiffalovič et al., Vacuum 84 (2010) 19–25



www.incoatec.de

innovative coating technologies gmbh