

## Spec Sheet SAXS 31

# NANOSTAR

## Innovative Components for Enhanced Capabilities

### Keywords

Small Angle X-Ray Scattering (SAXS), X-ray source, X-ray optic, Wide Angle X-Ray Scattering (WAXS), Image plate IP, VÅNTEC-2000, sample handling

### Introduction

In the reciprocal world of x-ray scattering the instrument for investigating nanometer structures by SAXS is quite impressive. However, even the NANOSTAR of second generation offers potential of further improvements to an advanced access to more information about the sample properties. This potential concerns the source and beam path, the sample environment, and the detection units.

### SAXS Detector

The 2-dimensional detector is the most essential component of a SAXS instrumentation. The detector needs a large active area while providing a good spatial resolution and a very low noise level. For a laboratory instrument a real photon counter with negligible read-out time is the preferable choice. The collection of requirements for an affordable price can be matched by a gas detector only.

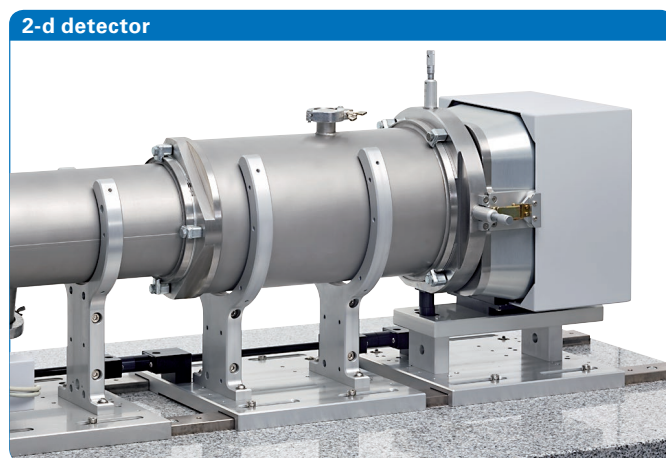


Figure 1: VÅNTEC-2000 integrated into a NANOSTAR.

The NANOSTAR now offers the possibility to be equipped with the newest 2-d detector technology: the patented MikroGap VÅNTEC-2000 detector (figure 1). This mature technology, hundredfold proven with the VÅNTEC-1 detector, overcomes the limitations of the conventional 2-d multi-wire proportional counters (MWPC). The large 14 x 14 cm<sup>2</sup> active area enables larger 2theta coverage than accessible with the HI-STAR when using the identical sample-to-detec-

tor distance. Simultaneously the VÅNTEC-2000 offers an improved spatial resolution with increased dynamical range far above conventional MWPC detectors. Furthermore, the overall data quality is superior due to the MikroGap technology intrinsic feature of more uniform sensitivity across the entire active area (figure 2).

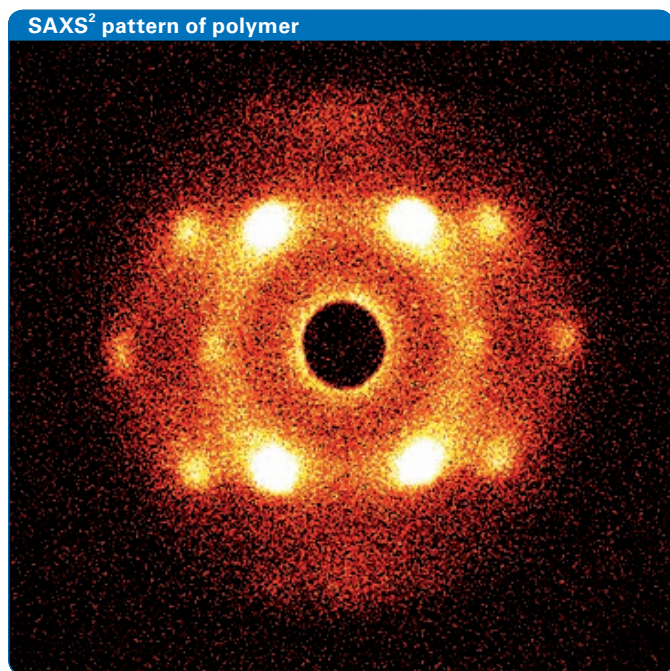


Figure 2: SAXS² pattern of a polymer sample recorded with a VÅNTEC-2000, measurement time 100 seconds.

### Specifications of VÅNTEC-2000

- Active area: 14 cm x 14 cm, corresponding  $\geq \pm 3.7^\circ$  2theta at 1,079 mm sample-to-detector distance
- Operational mode: 2048 x 2048, 1024 x 1024, or 512 x 512 channels
- Spatial resolution at 8 keV:  $\leq 230 \mu\text{m}$  FWHM ( $\leq 100 \mu\text{m}$  RMS), equivalent to  $2\theta = 21.32$  (9.27)  $\mu\text{rad}$  corresponding  $1.74$  (0.756)  $\text{m}\text{\AA}^{-1}$  at 1,079 mm sample-to-detector distance)
- Usable wavelength range: from Cr-K $\alpha$  to Cu-K $\alpha$ , factory setting is Cu-K $\alpha$
- Background noise:  $< 5.0 \cdot 10^{-4}$  c/s/mm<sup>2</sup>
- Dynamic range (local maximum count rate / local noise rate):  $> 10^8$
- Response uniformity:  $< 2.5\%$

### Sample Chamber

The sample chamber of the NANOSTAR accommodates an XY-translation for sample positioning or mapping (Nanography) and a reference sample wheel. Additionally, non-ambient sample environments can be placed in the chamber. For the extended requirements on sample manipulation the NANOSTAR can be provided with an extended sample chamber (figure 3). The inner dimensions of the extended chamber allow to mount a Linkam CS 450 shear cell e.g.. For supply connections of the sample manipulation devices the extended chamber offers two NW 65 flanges plus one NW 40 flange.

### Specifications extended sample chamber:

- Inner dimensions: 287 mm x 501 mm x 380 mm, W x H x D
- Motorized x-y stage: travel 80 x 150 mm (horizontal x vertical)
- Motorized reference sample wheel
- Available flanges: two NW 65, one NW 40
- Operation under vacuum or ambient conditions with additional vacuum tight exit funnel
- Access: via interlocked transparent and radiation tight front door

The NANOSTAR offers different mounting positions for the sample allowing different sample-to-detector distances. This feature enables the capability to adapt the resolution and the 2theta-range recorded simultaneously to the experimental requirements. Positioned at the smallest possible distance – the detector-WAXS position – a 2theta range of  $40^\circ$  can be captured with the real photon counting detector (HI-STAR or VÅNTEC-2000). A further expansion to more than  $80^\circ$  2theta can be realized by mounting a  $20 \times 25 \text{ cm}^2$  large image plate (IP) detector (figure 4) into the extended chamber close behind the sample. The IP has a central hole (figure 4) for the SAXS signal passing through and being recorded simultaneous with the HI-STAR detector e.g.. After exposure the IP is removed from the chamber for an off-line read-out, converting of the data, and read by the SAXS software (figure 5). For further evaluation such as preferred orientation determination or crystalline phase identification the data can be imported into the 2D SAXS software.

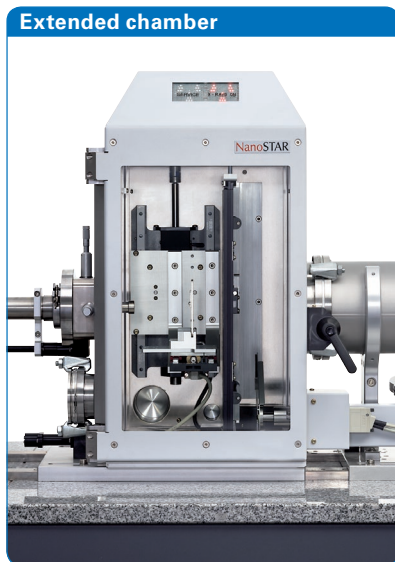


Figure 3: Extended sample chamber of NANOSTAR.



Figure 4: Close view into the extended sample chamber of the NANOSTAR showing standard sample holder and central part of the IP.

### Specifications IP

- Size: 20 cm x 25 cm
- Read-out: 2000 x 2000 pixel 16 bit
- Maximum 2theta coverage: about 83° for 11.5 mm sample-to-IP distance
- Read-out device: Fuji FLA-7000
- Operation: under vacuum or air

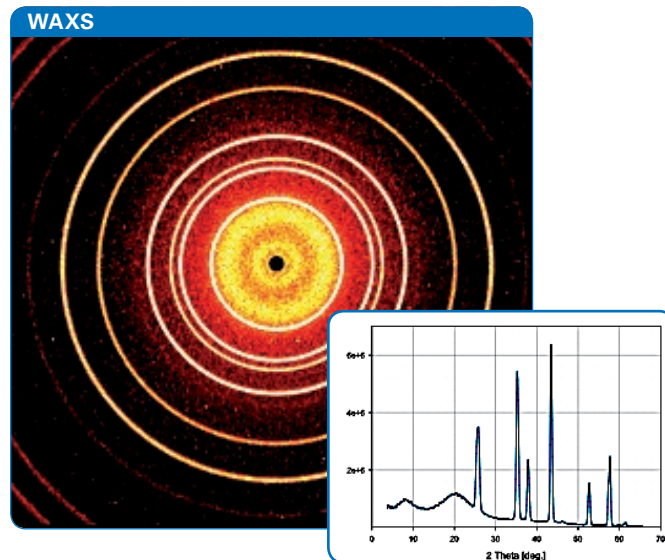


Figure 5: WAXS test pattern of  $\alpha$ - $\text{Al}_2\text{O}_3$  powder sandwiched between two foils taken with the IP; measurement time 300 s, primary attenuation by a factor of 10. The insert shows Intensity versus 2theta angle as obtained after chi-integration.

### X-Ray Source

The NANOSTAR offers two types of common X-ray sources: a standard sealed tube running with up to 1.5 kW power load at 0.4 x 0.8 mm focal spot size, or a Turbo X-Ray Source (TXS) with a maximum of 5.4 kW at 0.3 x 3 mm focal spot size. By switching to a 0.1 x 1 mm spot focus running at 1.2 kW max the power density of the TXS can be increased to 12 kW/mm<sup>2</sup> which doubles the primary beam flux. Combined with a Montel-P optic a high-brilliant beam is generated with an ideal match concerning flux and resolution to the NANOSTAR configuration.

The  $\mu$ S micro-focus X-ray source (figure 6) is a brand new power-engine class of source for the NANOSTAR. With only 30 W of power input a photon flux is provided similar to a conventional 0.3 x 3 mm focus rotating anode source running at several kW. Due to the minimized distance between X-ray tube spot and Montel optic the compact  $\mu$ S uses a much larger amount of the x-rays emitted from the anode compared to a sealed tube.

The  $\mu$ S enables high-sample throughput or investigation of samples with low scattering power (figure 7) for attractive running costs because of its low power consumption and the simple air cooling of the x-ray source.

### Low power X-ray source



Figure 6: IµS micro focus X-ray source with integrated Montel optic.

### Specifications IµS

- Power: 45 kV at 0.65 mA
- Anode: Copper
- Integrated Montel optic:
  - 60 mm length
  - 1 mrad output divergence
  - 1 mm output beam dimension
  - $>8 \times 10^7$  cps output flux behind the optic
  - Customized designs on request
  - Radiation tight housing with Be-entrance and exit window enabling evacuation
  - Four manual setting screws

### SAXS<sup>2</sup> pattern of a duck tail tendon

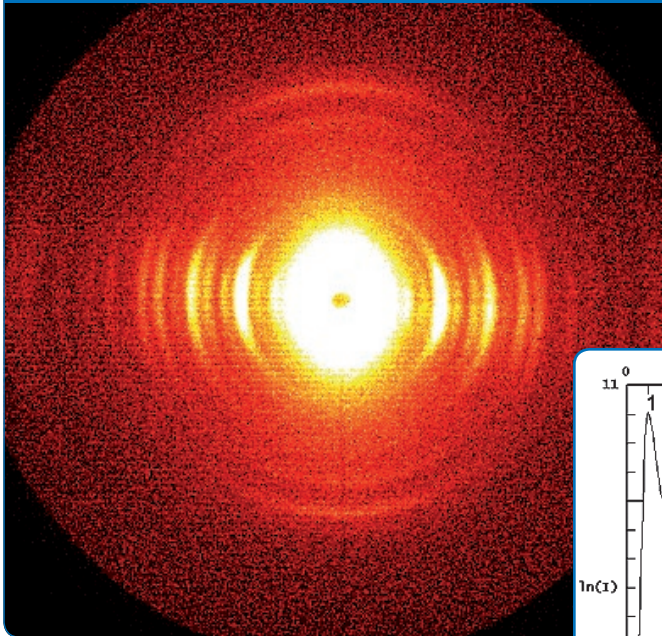
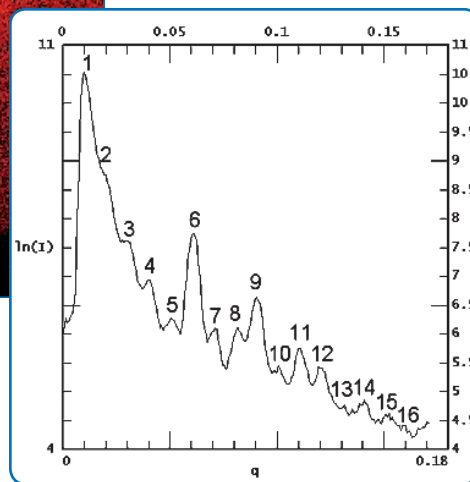


Figure 7: SAXS<sup>2</sup> pattern of a duck tail tendon measured with IµS running at 30 W and HI-STAR detector. The insert shows intensity versus 2theta angle as obtained after chi integration. 16 diffraction orders are observable with a measurement time of 1 hour.



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