Nanoscale X-ray structural characterization instrument

NANOPIX

1. Introduction

SAXS (Small Angle X-ray Scattering) is a powerful tool for nano-scale structural analysis covering a broad range of applications from research and development to quality control. The technique targets a broad range of materials, from periodic and non-periodic structures including solid, liquid, liquid crystal and gels to research in advanced functional materials such as carbon fiber reinforced plastics (CFRP), nano-particle (NP) sizing, and coarse-structure analysis of macromolecules. Advanced functional materials, used increasingly in today's nanotechnology-focused research, have nanoscale fine structural features that must be well-controlled. SAXS measurement and analysis is used for atomic and molecular-scale structure evaluations, but it can also be used for measurement of diffuse scattering caused from boundaries of electron density inhomogeneity. In the case of a periodic structure having 10 nm spacing, a diffraction peak is observed around 0.4 degrees in two theta using copper $K\alpha$ emission line ($\lambda=0.15418$ nm). This is an example of small angle scattering, or scattering which occurs at lower angles—typically less than 5 degrees.

Rigaku's new product, NANOPIX, gets its name from "Nano Particle Inspection by X-rays". It is optimized for SAXS measurement such as scattering occurs at low scattering angles. Moreover, It has been developed for not only the SAXS but also the atomic-scale structures that are in higher scattering angles around 20–30 degrees. Including both SAXS and WAXS, this new instrument is developed for multi-scale structure analysis of materials which have hierarchical structures.

2. Features of NANOPIX

2.1. Optimized for nano-structure analysis

The X-ray generator MicroMax007HFMR in NANOPIX is the same as in NANO-Viewer and S-Max 3000 (earlier models of dedicated SAXS instruments). It has an output power of 1,200 W and an apparent source size of 0.07 mm in diameter. NANOPIX features high performance optics, OptiSAXS, for which the multilayer confocal mirror, is newly designed, and a newly developed scattering pinhole slit, ClearPinhole, which reduces parasitic scattering and is achieved with two pinhole collimation optics. NANOPIX enables the user to measure ranges up to Q_{min}~$0.02\,\mathrm{nm}^{-1}$, corresponding

Fig. 1. Appearance of beam module (MicroMax007HFMR and OptiSAXS confocal multilayer mirror).

Fig. 2. HyPix-3000 (upper) and HyPix-6000 (lower) in the NANOPIX.

to about 300 nm in d-spacing. These features make it one of the highest performance SAXS instruments available for the home laboratory to date.

NANOPIX is equipped with beam module technology (Fig. 1), in which an X-ray generator and confocal multilayer mirror are integrated into a single module in order to increase the both intensity and positional stabilities of the X-ray beam.

2.2. High performance 2D semiconductor detector: HyPix-3000/6000

The detector is a key component SAXS measurement systems. NANOPIX is equipped with the high performance semiconductor detector, HyPix-3000, in the standard system configuration (Fig. 2). HyPix-3000 has a 775×385 pixels with 100 μm×100 μm pixel size and an active area of 77.5 mm×38.5 mm. For an anisotropic system in the specimen, HyPix-3000's large active area enables measurement of the diffraction/scattering and anisotropy of the system, such as preferred orientation of small grains simultaneously. Moreover, the HyPix-6000, which is a multi-modules configuration (two modules of HyPix-3000) is offered as an option in order to cover wider measurable scattering angles.

2.3. Easy operation

For the investigation of the hierarchical structure of nano-structures in advanced functional materials, wide coverage of scattering angles from SAXS (up to 5 degree) to WAXS (30–40 degrees) requires changing the sample-to-detector distance. It is due to the fixed wavelength of the X-rays in laboratory use X-ray sources. Moreover, a vacuum path between the sample and the detector is required to reduce the additional scattering created by air. When changing the vacuum path (Fig. 3) with NANOPIX, it is not necessary to lift and take off the piece of vacuum path. All pieces of the vacuum path are mounted onto a sliding rail, and can be

Fig. 3. Vacuum path changing system.

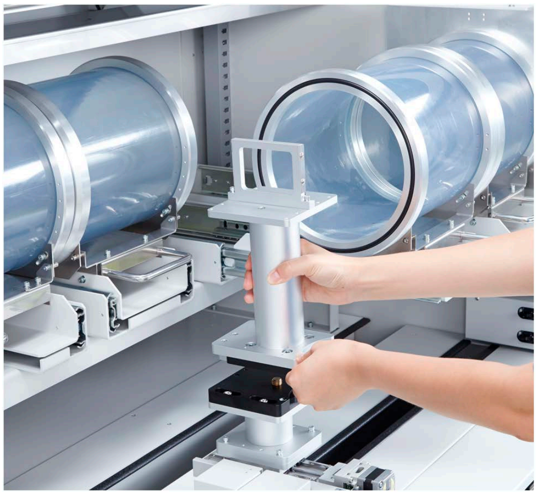

Fig. 4. Sample attachment exchange.

adjusted quickly and easily. This function improves the operability of the system dramatically (Fig. 3).

2.4. Measurement under varied sample environments

The need for "in-situ" SAXS measurements with controlled the temperature and mechanical stress is increasing. A variety of sample attachments including temperature and humidity control, simultaneous differential scanning calorimetry (DSC), and multiple sample holders are available for the NANOPIX as standard options. The kinematic-base mount and the motorized long-traveling length stage (70 mm in horizontal and 50 mm in vertical) enable the user to combine a variety of sample attachments—not only the standard options, but also an array of third-party sample stages and custom stages. Figure 4 shows an example of tool-less sample attachment exchange (multiple sample holder).

3. Applications

3.1. Diffraction of chicken leg collagen

Chicken leg collagen is known to have a 65 nm repeating distance. It is often used as a performance test for SAXS instruments. The SAXS diffraction pattern

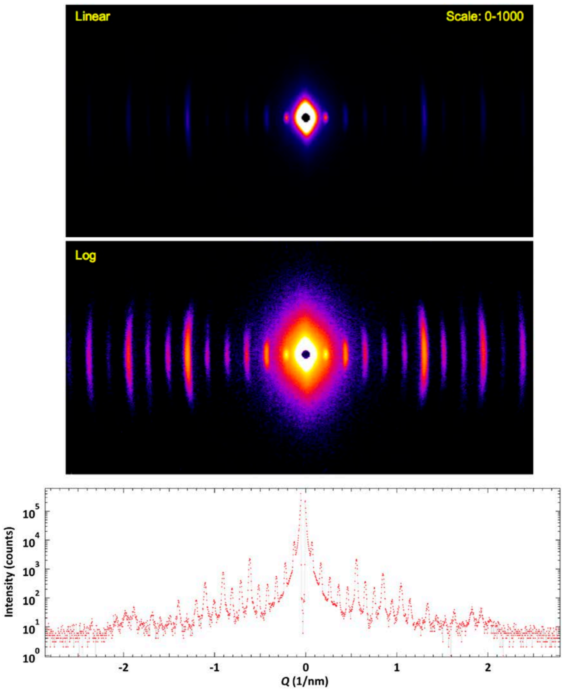

Fig. 5. SAXS patterns from chicken leg collagen with sample-to-detector distance of 1300 mm; (upper) Linear scale, (center) Logarithmic scale, and (lower) cross-sectional profile of the center.

from the chicken leg collagen is shown in the Fig. 5. In Fig. 5, the 1st order peak which corresponds to the 65 nm repeating distance is clearly shown and is close to the beam stop.

3.2. Scattering from nanoparticles

"Nano" is a unit prefix meaning one billionth (ref. from Wikipedia) and nanoparticles have sizes ranging from 1 nm to 100 nm. Figure 6 (a) shows the 2D scattering pattern from the silica nanoparticles dispersed in the water. In order to improve the signal-to-noise ratio, the 2D data is converted to a 1D profile by using the circular averaging process, and the processed 1D profile is shown in Fig. 6 (b). As a result, the average particle size is calculated to be about 120 nm, shown in Fig. 6 (c).

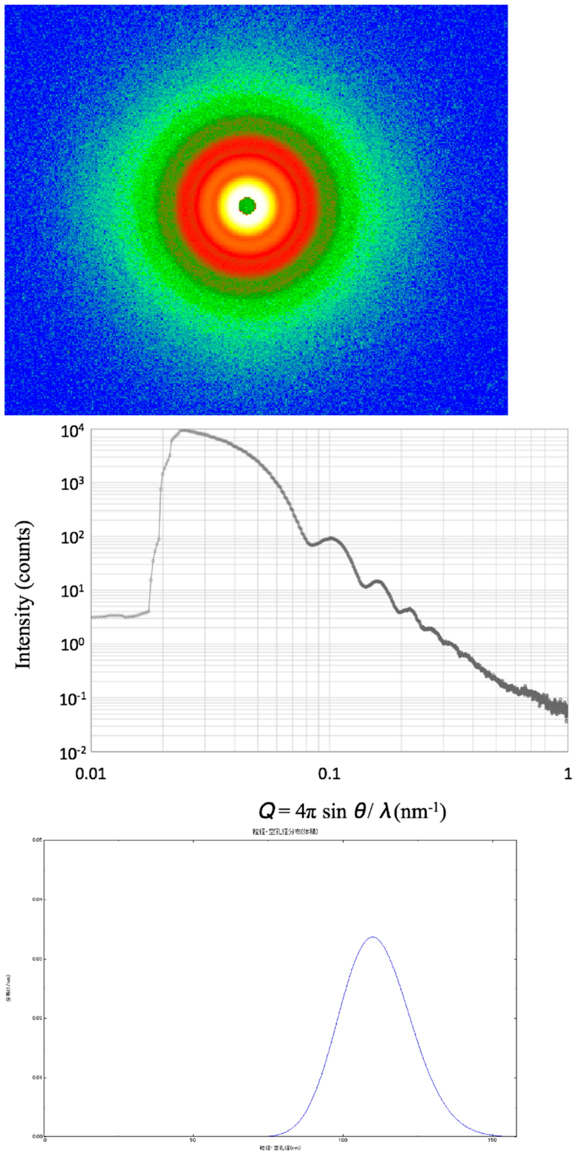

Fig. 6. (a) 2D scattering pattern from the silica nanoparticles dispersed in the water, (b) circularly averaged 1D profile of Fig. 6 (a), and (c) analysis result of particle size distribution from Fig. 6 (b).

4. Conclusion

NANOPIX represents the total integration of advanced technologies in X-ray measurement. The X-ray source and focusing optics, collimation optics, and detector, lead to a broader range of measurable samples and superior SAXS resolution compared to the previous models, NANO-Viewer and S-Max 3000. The dramatically improved operability from the previous models broadens the potential user base from just SAXS specialists to many material scientists and engineers. Moreover, NANOPIX is a dedicated instrument for SAXS measurement, and the SAXS resolution has been upgraded from 200 nm in the previous models to approximately 300 nm. The active area size at the exit window of the receiving optics has been enhanced from 100 mm in diameter to 200 mm in diameter in order to cover larger scattering angles in the WAXS region. The 2 pinhole collimation optics with ClearPinhole technology increases the beam intensity at the sample by approximately 15 times in comparison with the previous models. The HyPix-3000/6000 detectors improve and increase the spatial resolution and amount of information in the 2D image. NANOPIX makes it possible to perform experiments at the home lab that might have previously required beam time at a synchrotron facility. NANOPIX will contribute to and accelerate the research and development of advanced materials, creating a bridge between home laboratory and synchrotron-specific applications.