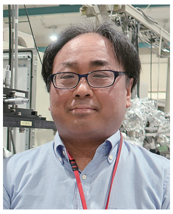
Development of Resonant Soft X-Ray Scattering Spectroscopy for Soft Matter

UVSOR Synchrotron Facility
Division of Advanced Photochemistry



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Soft matter, such as liquid-crystal and polymer, is essential in various technological applications. Interesting soft matter properties come from their mesoscopic structures and dynamics. Small-angle X-ray and Neutron Scattering (SAXS/SANS) are powerful tools for revealing mesoscopic structures. However, since SAXS is sensitive to modifications of electron densities, their contrast difference between similar chemical species is weak.

Resonant soft X-ray scattering (RSoXS) is an emerging characterization technique for mesoscopic structural analysis. Soft matter molecules mainly consist of light elements such as carbon, nitrogen, and oxygen, whose X-rays resonant energies are in the soft X-ray region. Resonant scattering is strongly sensitive to its element, chemical species and the molecular alignment relative to polarization vector. In particular, molecular alignment sensitivity enables us to investigate twisted structure of soft matter in mesoscopic scale.

This year, we developed a new RSoXS spectrometer. The feature of the new spectrometer is that a soft X-ray detector can be rotated along a scattering angle in vacuum. Compared to X-rays used for SAXS measurements, wavelengths of soft X-rays are much longer. Since a scattering vector, q, is proportional to $\sin(\theta)/\lambda$, where 2θ and λ are scattering angle and a wavelength of soft X-ray, a scattering angle becomes wider compared to SAXS. For examples, to analyze the structure from 5 nm to 500 nm, we need to measure the scattering angle 2θ from 0.5 to 60 degrees. To achieve this wide scattering angle measurement, we developed a new RSoXS spectrometer, where a soft X-ray detector can be rotated along the 2θ in the vacuum chamber, as shown in Figure 1.

For the performance evaluation, we investigated twisted bend (TB) liquid-crystal molecules with the new RSoXS spectrometer. The TB liquid-crystal phase is the newest nematic phase, only identified in 2011. There are many outstanding mysteries about the nature of its nanoscale organization and behavior. Our collaborators elucidate how the number of monomer units in a linear TB oligomer influences the structure of its nanoscale helix, an important TB phase structure–property relationship.[1)] Figures 2 show a schematic view of TB liquid-crystal molecules and a scattering image at a photon energy of 285 eV ($\lambda \sim 4.3$ nm), which is corresponding to carbon K-edge resonance. We find a sharp diffraction ring corresponding to the half-pitch periodicity of the helical filaments ($d \sim 128$ nm ($q \sim 0.005$ Å^{-1})). We also confirmed that the detector can be rotated along 2θ and we can measure scattering angles from 0 to 60 degrees. We started to analyze molecular orientation orders of liquid-crystal molecules and crosslinked structures of polymers with the spectrometer.

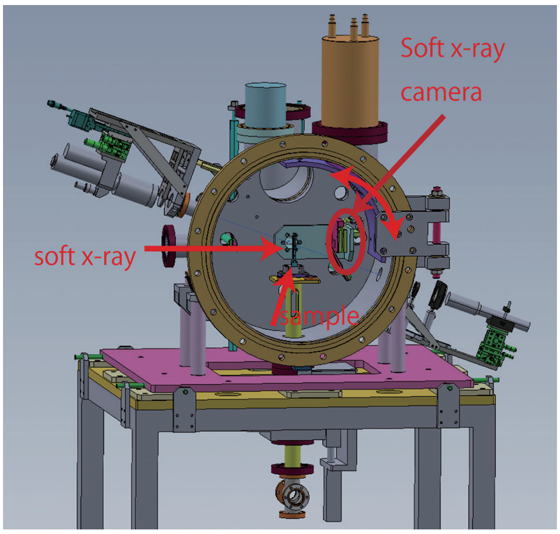

Figure 1. Schematic draw of a new RSoXS spectrometer.

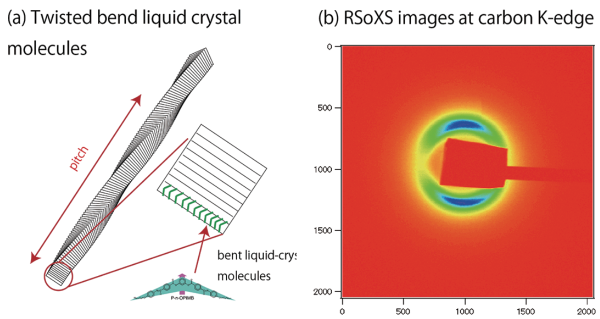

Figure 2. (a) Schematic view of twisted bend liquid crystal molecules. (b) Scattering image at a photon energy of 285 eV.

Reference

1) Y. Takanishi, F. Araoka and H. Iwayama, *RSC Adv.* **12**, 29346 (2022).