# Application Note – Large Area CCD Cameras for X-ray Scattering

## Introduction

A group of scientist lead by Dr. Eliot Gann from the National Institute for Standards and Technology (NIST) based at the NSLS-II in Brookhaven (New York, USA) have built a facility for Resonant Soft X-ray Scattering (RSoXS). It was primarily built for the purpose of measuring nano-scale soft matter structures. A key component of this setup is a low noise greateyes CCD detector. The groups findings were presented in their recent publication: Eliot Gann et al 2021 J. Phys.: Condens. Matter (in press).

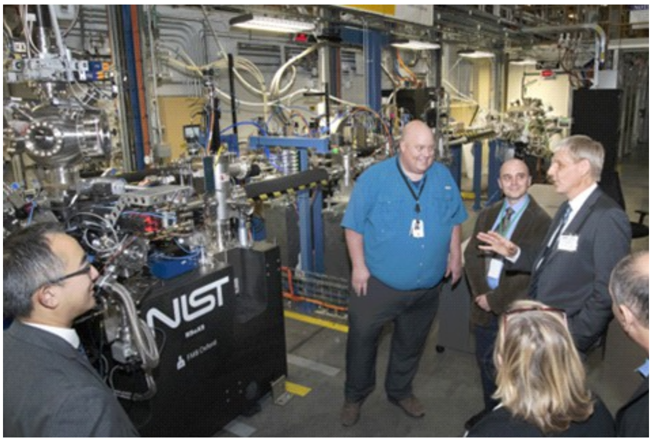


***Image 1:*** *picture of the official commissioning of the RSoXS station at NSLS-II*

RSoXS is a technique that is gaining a lot of traction for research evolving nanostructure characterization of soft matter. By utilising X-ray radiation near the absorption edges of carbon, nitrogen and oxygen, materials containing these elements can be probed. In particular their molecular bonds and the orientation [1-3]. The technique already gained a lot of popularity in the characterization of polymers, primarily to better understand the nanoscale structure in organic electronics materials [4, 5].

## RSoXS Station with greateyes CCD camera at NSLS-II, New York, USA

RSoXS experiments present several challenges. One of them is caused by the lower energy of the applied X-rays. Soft X-rays are defined by having energies less than 2 keV. This results in a shorter penetration depth through most materials and even through air. As a result RSoXS stations require a high vacuum environment for both the sample and the detector. This in turn limits the range of samples that can be probed by such a setup. The relatively low X-ray energy also means that the penetration depth through samples is limited and that special detectors must be used which are particularly sensitive in this energy range. The layout of the RSoXS station at NSLS-II is shown in Figure 1.

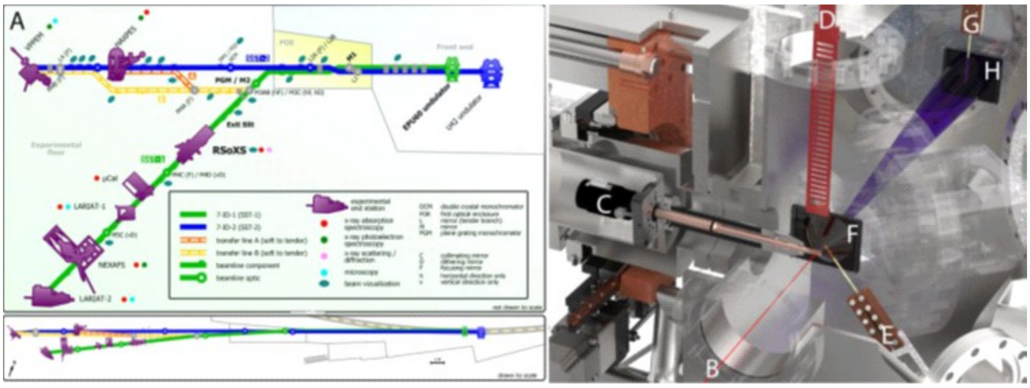


***Figure 1:*** RSoXS layout and schematic. (A) (left frame) a "Subway" schematic of beamline and in the right frame, a 3D rendering of the inside of the RSoXS chamber: (B) X-ray beam (C) sample holder (D) alternative multi-sample holder (E) Wide Angle Beamstop, with (F) marking the WAXS detector CCD and (H) the SAXS detector CCD.

As a very unique feature of this setup it is possible to partially withdraw the WAXS CCD detector and measure both small and wide angle scattering simultaneously (by utilizing the SAXS detector at the same time). After careful consideration the two detectors chosen for the RSoXS station were customised CCD-based cameras from greateyes GmbH in Berlin (Germany). Both CCD detectors are having a resolution of 4096 pixel × 4096 pixels and a 15 µm pixel pitch. They are flange-mounted and their sensitivity is optimised for the detection of soft X-rays. The angled positioning of the sensor (allows for) enables ideal positioning of the detector focal plane with regards to the sample. The cameras light sensitive area is approximately 60 mm square with a 100% fill factor. In operation, these back-illuminated CCD surfaces are cooled to -80 °C via thermoelectric cooling. Image 2 shows a greateyes ALEX 4k4k camera which is similar to the one used at the NSLS-II in Brookhaven.

***Image 2:*** *greateyes ALEX 4kx4k soft x-rax camera*

As the RSoXS station at NSLS-II covers an energy range between 0.1 keV and 2.2 keV back-illuminated CCDs are ideally suited as they offer superior sensitivity in this range. In regular operation, both detectors are read out in a 4×4 binning mode (now with effective 60 µm square pixels) in approx. 1s. The binning is used to increase the readout speed while maintaining low noise levels. The resolution is still much smaller than the beam size on the detector, which is 250 µm under normal conditions. Because binning is important for the experiments it is very advantageous to avail of the 18-bit digitisation of the greateyes CCD cameras. This enables the detection of signals with high photon flux – without saturating the detector. At the same time the camera still provides excellent sensitivity for signals just above the detection limit. CCD technology is used for the RSoXS station because it allows the best signal to noise and flux efficiency with the largest detection area. This in turn gives scientists the best chance of measuring their samples (sample nanostructures and sample chemistry) before the beam has a chance to alter them. Even faster detection systems are desirable and in use on other soft X-ray beamlines at NSLS-II [6]. These systems currently come with the drawback of increased noise and therefore would require increased flux on the samples before the same signal to noise ratio could be reached. The majority of users have also found that the exposure times generally required to obtain statistically relevant measurements do indeed match the speed capabilities of the greateyes CCD detector very well. But to make proper use of the increased flux of future sources, faster, highly efficient, low noise soft X-ray detectors will be advantageous.

## First Experimental Results

Popular samples that could already be studied at the RSoXS station were photovoltaic double cable polymers. Organic electronic materials offering possibilities to study resonant polarisation and diffraction effects at X-ray energies between 90 eV and 2,200 eV. The RSoXS station is ideally suited for these applications. For this to succeed the high angle scattering performance of the experimental setup is critical. Gann et.al. demonstrated the high angle performance of the scattering station on these organic electronic materials. Please note the 360 º of azimuthal scattering achievable with the WAXS detector in the closest position. By this the characterization of the energy dependence of a device relevant length scale diffraction peak is demonstrated. The ring observed in Figure 6B under resonant conditions corresponds to the first order side chain stacking peak, which exhibits notable anisotropy. This in turn also demonstrates that the RSoXS station can collect a full ring of scattering corresponding to size scales approaching the wavelength of the probing X-rays, (4.35 nm at 285 eV, 13.8 nm at 90 eV and 0.56 nm at 2,200 eV), This is opening up the possibility of studying resonant and polarization diffraction effects at soft X-ray energies near the wavelength limit.

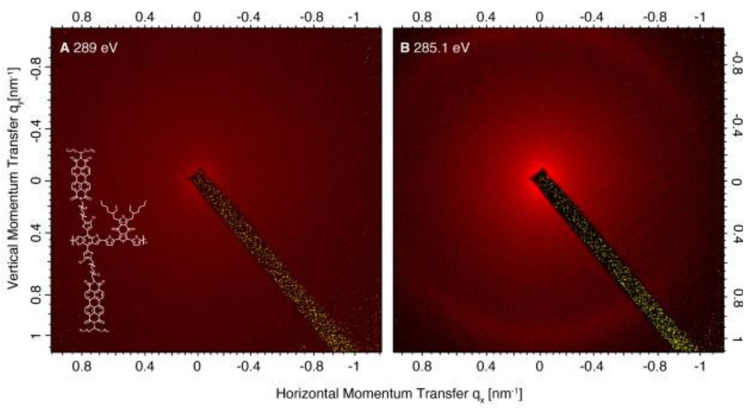


***Figure 6:*** *Wide angle resonant scattering example*

**Summary and Outlook**

An advanced RSoXS station at NSLS-II has been built primarily as a tool to conduct soft matter measurements, focusing on mitigating the beam damage and making maximal use of every photon interacting with the sample. This led to a unique layout for a scattering beam line with the data collected by low noise large area CCD detectors from greateyes GmbH in Berlin. This experimental setup has already allowed for data capture with near complete removal of the carbon dip, while maintaining stable, high-flux and high-resolution spectroscopy.

**Keywords:** Resonant Soft X-ray Scattering, X-ray scattering, Beamline, Soft Matter, greateyes CCD camera, spectroscopy

For more information on the cameras used in this study please contact the greateyes team at info@greateyes.de or tel: +49 30 912 075 250

**References:**

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