# Development Of A New Device For Angular Dependent Conversion Electron Yield XAFS Measurements

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**Introduction**

X-ray absorption fine structure (XAFS) measurements in the soft X-ray region are widely conducted with X-ray fluorescence yield (XFY) method and electron yield method owing to the difficulties in preparing samples of appropriate optical thickness for the transmission method. We have been employing conversion electron yield (CEY) method [1] that utilizes the ionization of surrounding gas molecules by the energetic Auger electrons from a sample. CEY is advantageous in the sense of S/N compared to XFY owing to the extremely small fluorescence yield in the soft X-ray region. However, the use of an electrode that prevents created electron-ion pairs from the recombination has made the angular dependent measurements difficult. We have developed a new device that can make the angular dependent XAFS measurements using CEY method. The device was applied to the fractional determination of orientations in polythiophene thin films.

**2. Experimental**

2.1 CEY device

Principles of the CEY measurements are similar to those of X-ray detection with an ionization chamber except for the ionization source of gas molecules. Typically He gas is employed to reduce the direct ionization with X-rays, and a planer electrode has been adopted in BL-11 to avoided the recombination of the created electron-ion pair. To overcome the interference of the electrode with incident X-rays, we have adapted a rod-shaped electrode that is similar to the proportional counter (PC) for X-ray detection, and the PC could be utilized for measuring CEY from the sample placed in the counter when operated with the mixture of He and methane [2].

Figs.1 show schematic side view and the photograph of the new device. Two electrodes of semicircle rod shape were arranged below and above the beam height. The diameter of the electrode, *b*, was 6 mm, and the
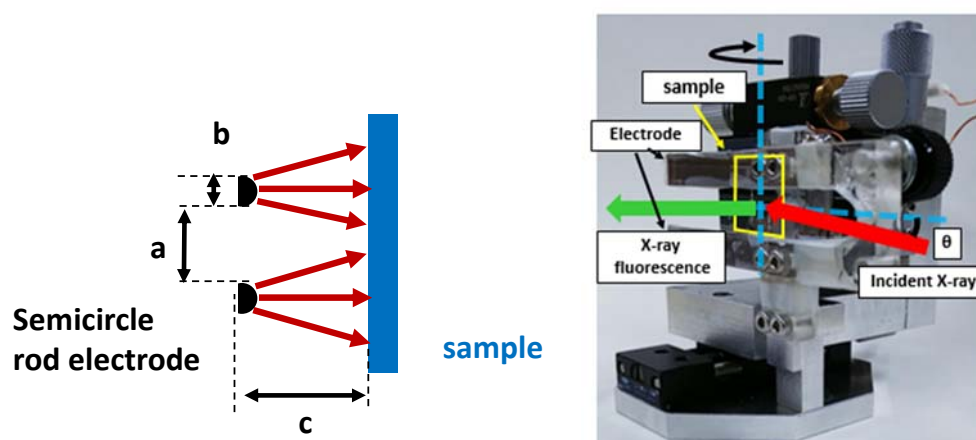


**FIGURES 1.** Schematic side view around the electrodes and the photograph of the new device

size was determined to make the strength of the electric field around the sample relatively uniform within the region of the ionization chamber while the electron cascade around the electrode was prevented. The clearance between the electrodes, $a$, was 8 mm, and the distance between the sample surface and the center of the electrode, $c$, was 8 mm. A sample was mounted on the rotational stage or a rotational axis of a motor for in plane rotation.

2.2 P3HT samples

Several types of poly (3-hexylthiophene-2,5-diyl) (P3HT) were spin coated onto Si or ITO glass substrates. Among them a special P3HT sample was prepared with a commercial P3HT reagent, and the surface of the ITO substrate was previously brushed with a velvet fabric. The sample showed uniaxial edge-on orientation, and the majority of the backbone of P3HT molecules were aligned along the brushing direction [3].

XAFS measurements were performed on BL-11, and both XFY and CEY were measured simultaneously. The developed new device was utilized for the XAFS measurements with the various glancing and azimuthal angles.

## 3. Results and discussion

Fig. 2 shows a series of sulfur K-edge XAFS spectra from the uniaxial edge-on sample. The glancing angle was 80 deg, and the incident beam was almost normal to the surface. It was clear that the XAFS spectra have azimuthal angular dependence, and the uniaxial character of the film could be explained with the angular dependences of σ* and π* peaks in the spectra.

Figs. 3 and 4 show azimuthal angular dependence of σ* and π* peak intensities. Fitting procedures with the theoretical model provided fractions of orientations in the sample.

In this presentation the principles and the advantages of the new device will be explained、 and the results of the fractional determination of the P3HT orientations will be presented.
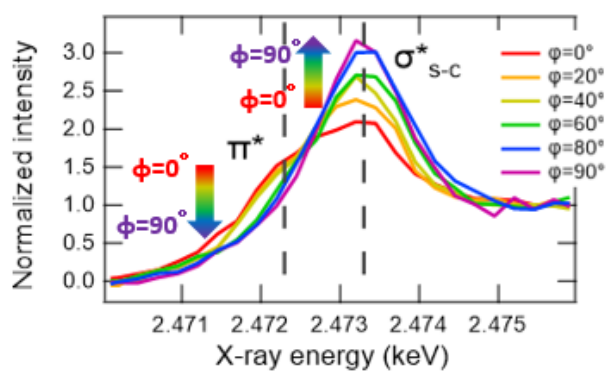
**FIGURE 2.** Polarization (azimuthal angle) dependence of Sulfur K-edge XAFS spectra of a P3HT film of uni-axial edge-on orientation.
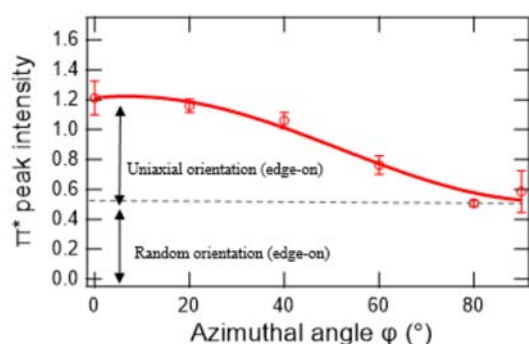
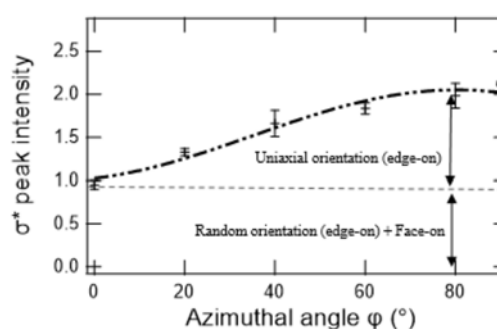**Figure 3.** Azimuthal angle dependence of π* peak intensities

**Figure 4.** Azimuthal angle dependence of σ* peak intensities

## REFERENCES

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3. M Imanishi et al., Sci.Rep.,7(2017)514,