Silicon Photodiodes: Experiences and Guidelines

J. Keister, Nov. 2004

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I. Noise issues on U3c.

A. Problems observed.

U3c monitor diode AXUV-100 92-2 #2 has ~33 pA or more dark current as measured with K617s. This value appeared to depend on GPIB activity, PC state, Heidenhain power source (ground loop noise sensitivity). Measured shunt resistance per IRD method (average of current with ± 10 mV source voltage, with dark box and ground isolated PD leads) to be ~2.4 MΩ. (Measured dark current with K6514 to be ~51 pA, indicating higher noise sensitivity of the newer model electrometer. Some trouble was observed with K6514 autoranging when the current is near the range break – electrometer active electronics affects measured result for low source resistance – as periodic or oscillating on U3c monitor diode). The graph below shows comparative 617 and 6514 filter norm scans. The large difference in the error bars is attributable to the large variation in the dark current measurement which is roughly 20-80 pA (60% peak to peak) on the 6514 and 30-40 pA (14% peak to peak) on the K617. The resulting (2-sigma) errors near 300 eV, where the flux is lowest, are 25% and 2% for the K6514 and K617, respectively, for the standard deviation analysis performed by the beamline calibration software.

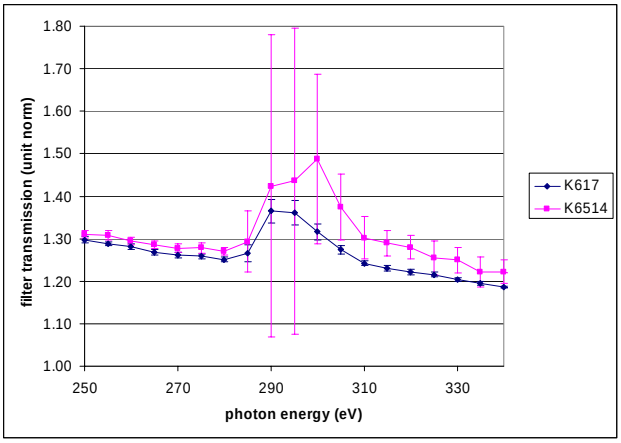

Figure 1 U3c relative detector normalization with Keithley 617 and 6514 before AXUV update. Standard 2-sigma error bars are shown.

B. U3c new diodes and observed improvements.

Both monitor and detector diodes were replaced with matching AXUV-100G 02-2s (#15 and #17). They each have ~210 MΩ measured at NSLS (~250 MΩ at IRD) shunt resistance per IRD method and 0.3 pA dark current. The calibration for the monitor diode was achieved by transfer responsivity from calibration standard AXUV-100G 02-5 #20 as described in a separate document. Filter normalization for the "back" diode was measured and found to prove matching of the two detectors. The sensitivity to GPIB and PC state was observed as fluctuation of less than ± 0.15 pA relative the nominal dark current. Filter norm scans measured with K617 and K6514 electrometers were compared, on both beamlines (relative differences are shown in the figure below). The peak in the (1-sigma) error bars near 300 eV (~1.5%) is attributable to the low signal there (as expected). At 115 eV, a peak is attributed to damage to the upstream B/Ti filter. At both beamlines, the two electrometers give the same result, agreeing within 0.2 % RMS. The RMS 1-sigma error for both beamlines is 0.3% on the 617 and 0.4% on the 6514. Thus the K6514 can be used since the noise sensitivity (dark current) is significantly reduced with the PD upgrades on U3c.

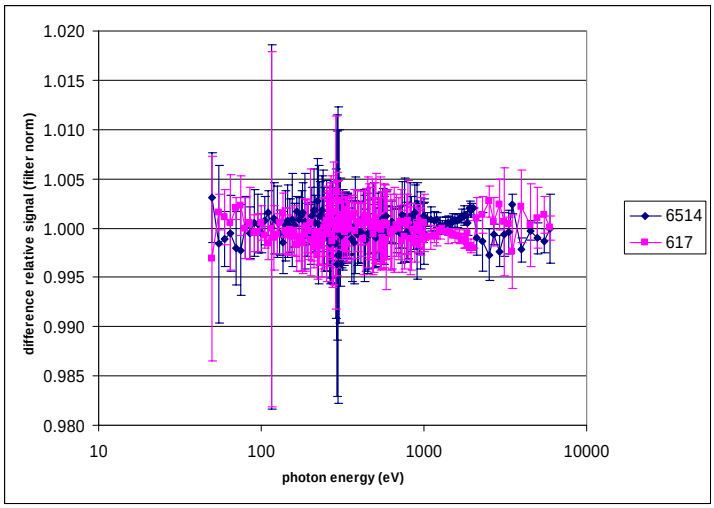

Figure 2 U3c and X8a relative detector normalization with Keithley 617 and 6514 after AXUV update on U3c. Error bars indicate 1-sigma error estimates.

II. AXUV characteristics and figures of merit (FOM)

A. Shunt resistance (dark current)

The rule of thumb in place at NSLS is that shunt resistance must be 200 MΩ minimum, to provide < 1 pA dark current (typically 0.5 pA or lower). This is consistent with the IRD claim that 1 GΩ or greater is needed for measurements to 100 fA or lower. However, the high-impedance PDs must be cherry-picked by IRD. For example, only 5 of 11 from the AXUV-100G 02-2 batch received in FY04 are 200 MΩ or greater.

B. Matching (within batches)

This turns out to be quite useful for calibrations. IRD guarantees 2% matching; this is confirmed for 02-2 and 02-5 batches on different photon energy ranges. Below is the unit norm scan comparing reference diode 02-5 #20 to x8a monitor diode 02-5 #18. One-sigma error bars indicate the diodes match within 2% over the energy range of 1-6 keV.

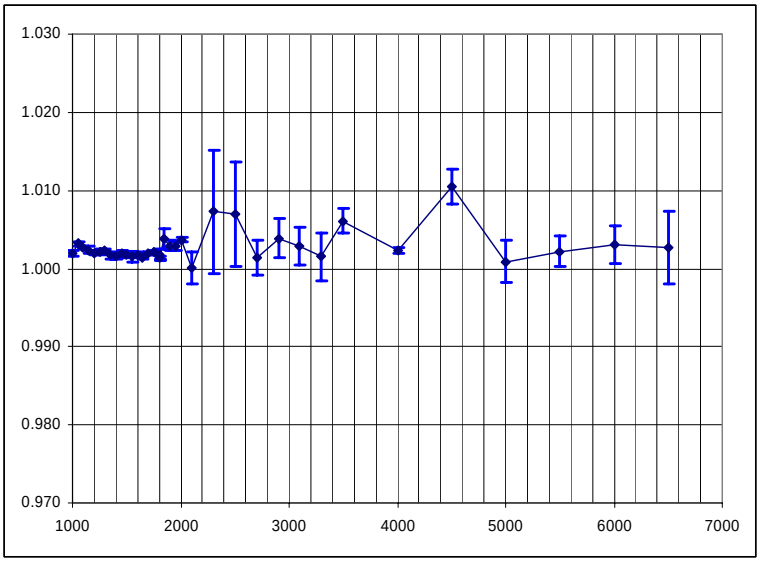

Figure 3 Relative detector response for AXUV-100G 02-5 #20 (reference diode) with respect to AXUV-100G 02-5 #18 (X8a monitor diode) demonstrating response matching to within 2%

Similarly the monitor (02-2 #15) and back diode on u3c (02-2 #17) can be compared on the range of 50-1000 eV. One-sigma error bars indicate the diodes match within 2% over this energy range (with the exception of 115 eV which is known to be affected by a separate beamline problem, namely a damaged filter).

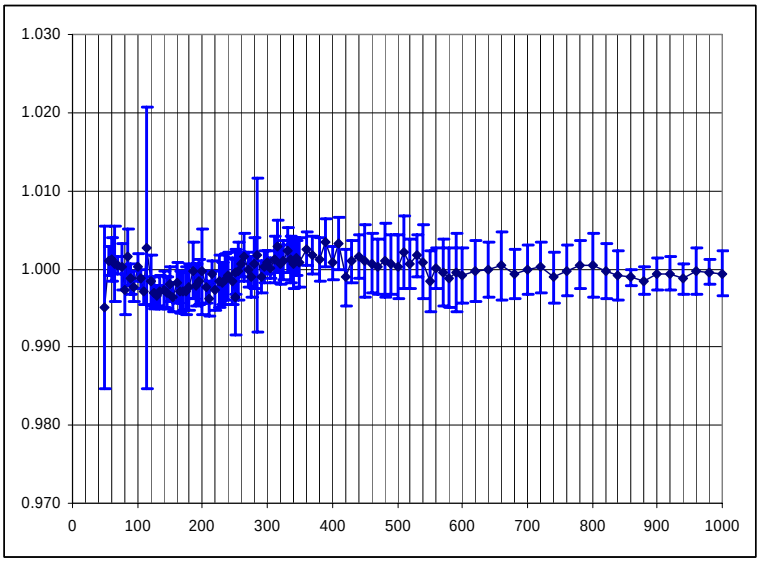

Figure 4 Relative detector response for AXUV-100G 02-2 #17 (U3c back diode) with respect to AXUV-100G 02-2 #15 (U3c monitor diode) demonstrating response matching to within 2%

C. Active area thickness

Depends on batch. For example, the 94-4 batch is thicker (48 µm instead of the ~25 µm seen on other batches). IRD can provide thicknesses in the range of 10-50 µm for the AXUV product.

D. Silicon dead layer thickness and migration probability
Also depends on batch. These fitting parameters turn out to be useful even for thin or near-zero dead layer silicon thickness (92-2 had high thickness 61 nm and low probability 0.68; newer batches have variable thicknesses 107 nm for 02-5 / 1 nm for 02-2 and probability 0.95 02-5 / 0 02-2). See AXUV cal procedure document for more details.

III. NIST calibration: useful?

The PD AXUV-100G 02-2 #29 was calibrated at NIST prior to delivery to NSLS. Self-calibration of that diode as well as transfer calibrations for presumably matching diode 02-2#15 is also compared to the NIST result. The NIST calibration source is cryogenic radiometer (ACR) between 11 and 35 nm. Between 5 and 11 nm, the source is a calibrated Ne ion chamber. Errors are roughly 10-20% over these two ranges.

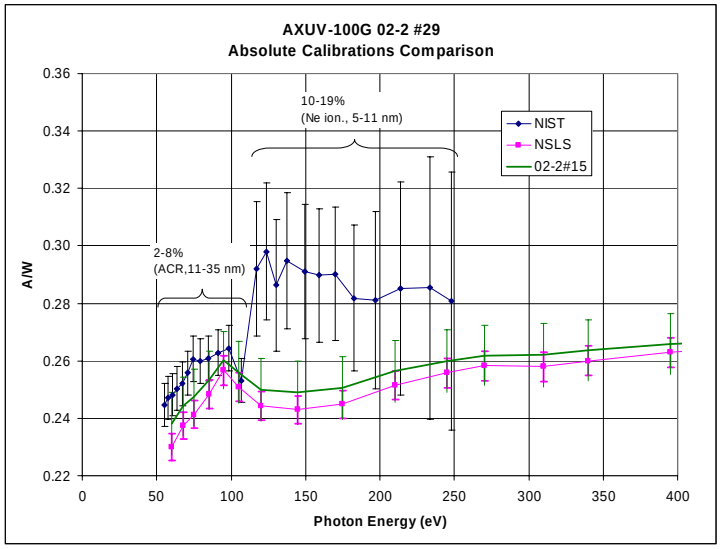

Figure 5 NIST calibration responsivity for AXUV-100G 02-2 #29, compared to NSLS self-calibration for #29 and #15 diodes from the same batch

The self-calibration procedure utilized at NSLS is also compared to PTB transfer calibrations (from LURE) and previous NSLS self-calibration source transfers for the 02-5 #20 reference diode.

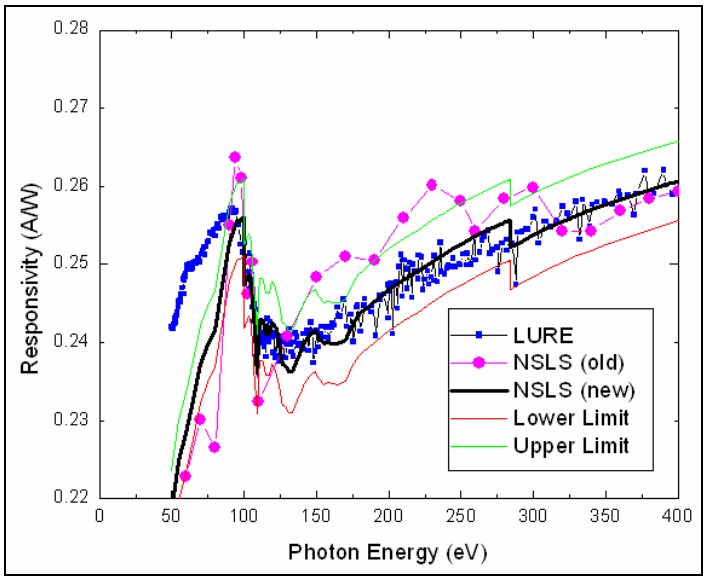

Figure 6 NSLS self-calibration data for AXUV-100G 02-5 #20, compared with NSLS transfer cal and PTB/LURE calibration for the same diode

Finally, example NIST and PTB calibration data is shown for diodes used at X24c (NRL).

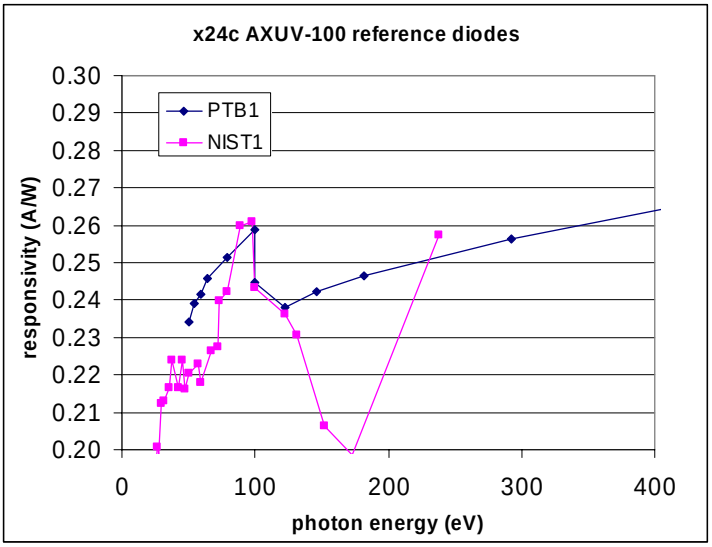

Figure 7 NIST and PTB calibration data compared for AXUV-100 used by X24c (NRL) group, showing deviations relevant to observations seen at U3c

It is interesting to note that the NIST and NSLS data are consistently at odds with the PTB data below 100 eV (showing flatter response / higher responsivity at PTB than the others). With the exception of data below 100 eV, the NSLS values consistently agree with PTB on several different diodes. Also we see large deviations for NIST data above 100 eV, including responsivities in excess of the known maximum of 0.27 A/W. In conclusion, the NIST cal is useful only up to 100 eV and even in that range may be easily

off by as much at 10%. The PTB calibration shows good agreement in general with the NSLS data sets, but should be more closely investigated and understood, and possibly improved, in particular below 100 eV, and maybe even at higher energies (as the self-cal ratio approaches 2). Other absolute standards should also be considered.

IV. Use of silicon PDs for high energy device calibration.

The fundamental limitation of the silicon photodiode for use at high energy is the diminishing responsivity with increasing photon energy, which results from the decreasing x-ray absorption of the silicon. To put this into practical terms, if you want to measure no less than 10 pA on an AXUV with flux as low as 10^6 photons per second, at 30 keV, you need ~ 30 µm active layer thickness (corresponding to ~ 2 A/kW responsivity). This is roughly the practical limit to the use of silicon photodiodes. Thicker active layers are available but must increase exponentially with photon energy to track with transmission of the silicon. Typical thicknesses provide by IRD are in the range of 25-50 µm.

Beyond the minimum sensitivity requirement, it is desirable to avoid the diode calibration issue altogether (need to accurately know the active layer thickness) by making the material thick enough to absorb essentially all of the incoming x-rays. In this case, the thickness must be even higher. Expect 1 mm to 10 cm to be needed for 98% absorption in the range of 10-50 keV.

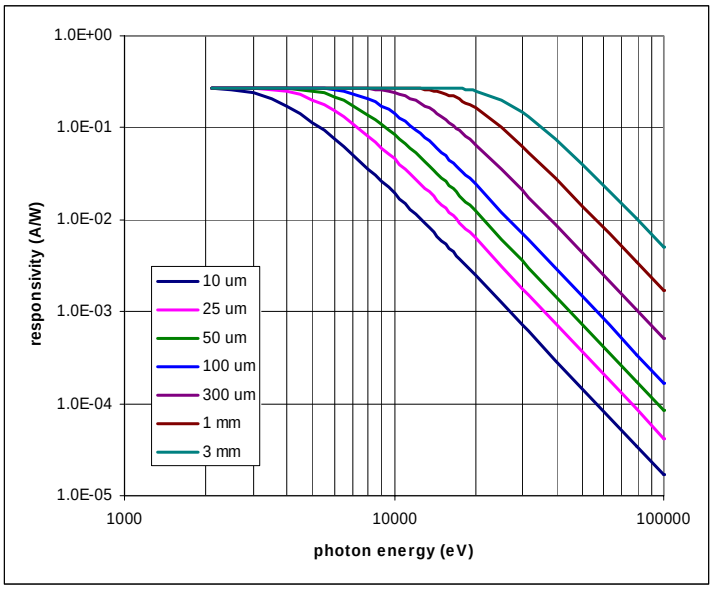

Figure 8 Modeled responsivity for AXUV-like silicon photodiodes of various active layer thickness

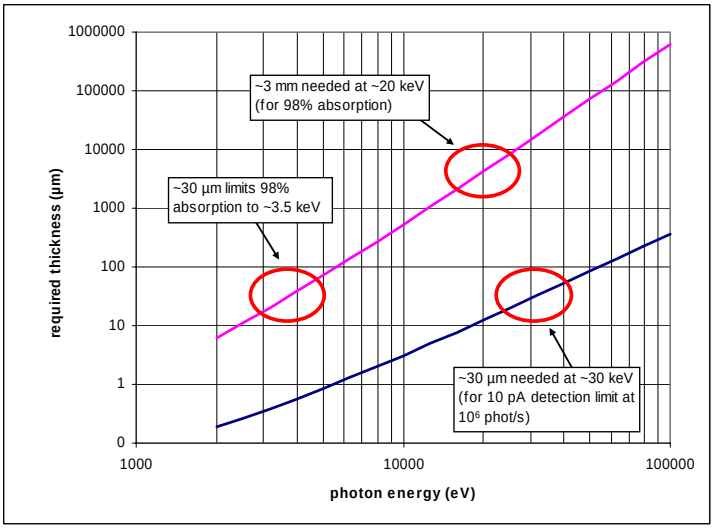

Figure 9 Estimated active layer thickness required for A) 98% absorption and B) 10 pA signal at 10^6 photons / second flux

For work up to this point, all the PD active layer thicknesses have been less than 3 mm and absorption therefore has been < 98% at 20 keV or higher. This has required calibration of the PD. Essentially this calibration amounts to measuring the active layer thickness and diffusion length, and making use of the high-energy silicon PD responsivity model:

$$S(\theta) = \left(\frac{e}{w}\right) \cdot \left(1 - \frac{\exp\left(-\frac{t_s}{\lambda_{Si}(E)\cos(\theta)}\right)}{\left(\frac{L}{\lambda_{Si}(E)\cos(\theta)} + 1\right)}\right)$$

Equation 1 High-energy responsivity model, function of incident angle and photon energy

where the parameters are active layer thickness t_s and diffusion length L. The average pair creation energy w, and the x-ray absorption length for silicon as a function of energy $\lambda_{Si}(E)$, come from the literature. The "self-calibration" method may be used, measuring the ratio of response at 60° (essentially doubling the path length) to normal (0°). In the case of calibrations recently performed for use at x15a, the diode AXUV-100G 94-4 #4595 was calibrated to determine the active area thickness and diffusion length. This was accomplished in two energy ranges simultaneously. In the range of 2-6 keV, the responsivity was directly estimated by comparison to reference standard AXUV-100G 02-5 #20 (data from NSLS and PTB/LURE). In the range of 8-20 keV, the value A_{60} (S_{60}/S_0) was measured at x15a. Since there were two types of data, the goodness of the fit of the model to this data was estimated by summing the percent errors at each point. The result of this fitting for this particular diode and dataset was an active layer thickness of 48.22 µm and diffusion length of essentially zero, with estimated error bars on the responsivity in the range of 2-4 percent.

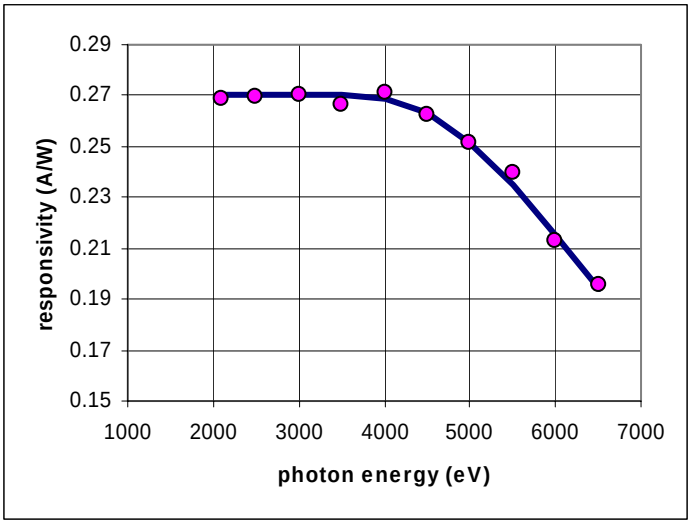

Figure 10 Responsivity measured and fit for AXUV-100G 94-4 #4595. Data from X8a calibration relative to reference diode AXUV-100G 02-5 #20. Fit includes agreement with A_{60} measurement at X15a (Next figure)

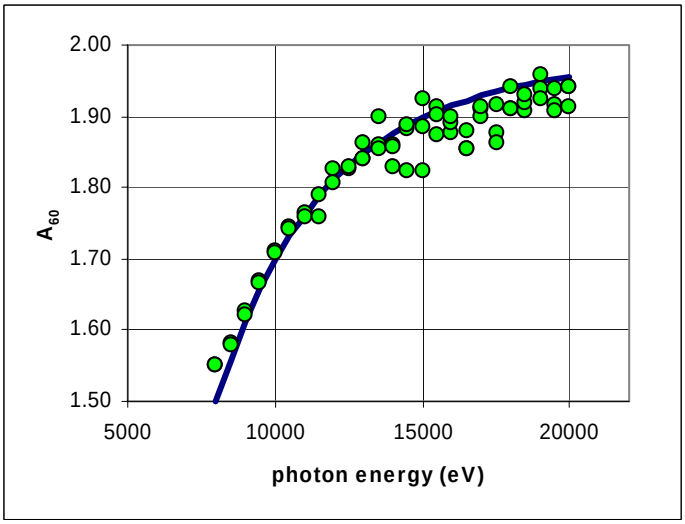

Figure 11 Response ratio A_{60} measured and fit for AXUV-100G 94-4 #4595. Data from X15a. Fit includes agreement with responsivity measurement at X8a (Previous figure)

The self-calibration method is limited in utility by the "saturation" value of 2 for the A_{60} ratio which occurs at high energy. In particular, thicker active layers are needed to preserve significance of the self-calibration ratio measurement in the same way that they are needed to preserve signal level.

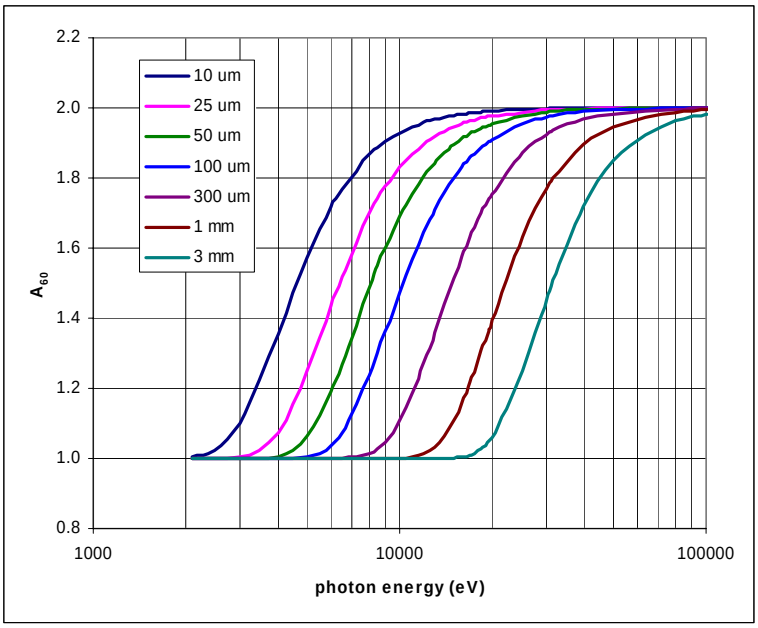

Figure 12 Modeled self-calibration ratio A_{60} as a function of photon energy and active layer thickness

V. Other Limits to self-cal methodology.

A. Reflectivity (low energy)

The number of x-ray photons available for absorption is limited by the reflectivity of the AXUV surface. That is, for the active layer, the reflectivity reduces the signal by a factor of (1-R). When performing the self-calibration, it is important to consider the affect of this factor since it depends on the incident angle. The ratio A_{60} will therefore appear lower than expected if the model does not include the reflectivity. However, the deviation $(1-(1-R_\theta)/(1-R_0))$ is not very large and can probably be neglected. For example, at 60 eV (the lowest energy for selfcals currently for NSLS) the deviation is less than 1%. However, the problem will become severe for lower energies and/or higher normal angles. The A_{60} results for silicon, under p, s and unpolarized x-rays are presented below.

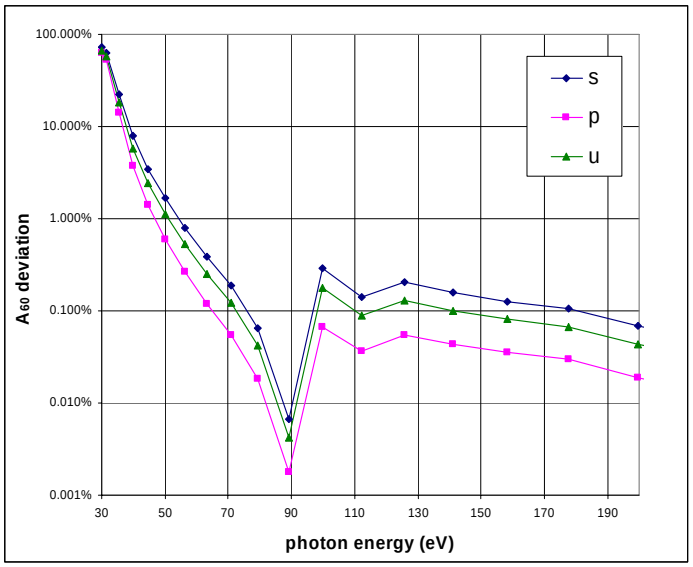

Figure 13 Modeled A_{60} deviation resulting from reflectivity (see text)

B. Available optical constants.

The CXRO database is used almost exclusively to prop up the measurement of the film layer thicknesses in the self-calibration procedure. However, this data set is limited to the range of 30 eV to 30 keV. To go beyond 30 keV, a careful examination should be taken of the available optical data and extrapolation methods.