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High-accuracy detector calibration in the 3–1500 eV spectral range at the PTB radiometry laboratory

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State-of-the-art detector calibration in the UV/VUV and soft X-ray spectral ranges at the Physikalisch-Technische Bundesanstalt (PTB) is based on the primary detector standard SYRES, a cryogenic electrical substitution radiometer capable of measuring radiant power of a few μW. At the PTB radiometry laboratory at the synchrotron radiation facility BESSY, two dedicated beamlines are operated, providing monochromatic radiation of high spectral purity, high radiant power and tunable photon energy in the 3–1500 eV range. The spectral responsivity of detectors, *e.g.* photodiodes, can be measured with a relative uncertainty of about 1% by direct comparison with SYRES, as will be demonstrated for PtSi/Si and GaAsP/Au Schottky and silicon n-on-p photodiodes. The calibration of photon-counting detectors traceable to SYRES can by accomplished by exploiting the unique capability to scale the spectral photon flux over several orders of magnitude by changing the stored electron current. Calibrations of CCDs and photomultipliers are presented as examples.

Keywords: photodiodes; detector calibration; soft X-rays; primary detector standards.

1. Introduction

During the past decade, the UV/VUV and soft X-ray spectral ranges have acquired growing importance in basic research and applications. This development has created an increasing need for radiometric standards and quantitative radiometric measurements. In order to meet these demands, the PTB radiometry laboratory at the electron storage ring BESSY has pursued the characterization and calibration of radiation detectors in the VUV and soft X-ray spectral regions as a major task (Ulm & Wende, 1995, 1997).

The straightforward way to determine the absolute spectral responsivity of a detector is a comparison with the known responsivity of a primary detector standard. At other national laboratories, radiometry in the VUV and soft X-ray spectral regions is mainly based on rare-gas double-ionization chambers and proportional counters, which are used as primary detector standards (Canfield & Swanson, 1987; Saito & Onuki, 1995/1996). Room-temperature electrical substitution radiometers have also been used recently (Saito *et al.*, 1996). The spectral responsivity of photodiodes can be determined with 4–15% relative uncertainty.

Significant progress has been achieved in the radiometry laboratory of PTB by using a cryogenic electrical substitution radiometer, SYRES, optimized for use of monochromated synchrotron radiation, as a primary detector standard for the VUV and soft X-ray spectral ranges (Rabus *et al.*, 1997). With SYRES it is now possible to measure radiant power in the 1–10 μW range with an uncertainty of about 1%, and the spectral responsivity of detectors can be determined with that uncertainty by direct comparison with SYRES, a prerequisite being a sufficient stability and homogeneity of the detectors. The capacities of the system were recently demonstrated in an accurate determination of the mean energy required to produce an electron–hole pair in silicon (Scholze *et al.*, 1996).

The operation principle of SYRES can be briefly described as follows: a cavity absorber of heat capacity C transforms the incident radiant power Φ, which comprises the monochromated synchrotron radiation as well as the thermal background radiation, into a heat flow conducted through the heat link of thermal resistance R to the heat sink, which is kept at a constant temperature T. The cavity geometry ensures an absorbance sufficiently close to unity so that the temperature difference between absorber and heat sink is given by $\Delta T = \Phi R$.

If the power load changes, for example when the synchrotron beam is switched on, the cavity absorber temperature would approach a new equilibrium value if the radiant power were constant. The synchrotron radiation source, however, provides a monotonically decreasing radiant power output so that it is advantageous to work in the dynamic substitution mode.

2. Measurement of the spectral responsivity of photodiodes

The calibration of photodiodes is performed by measuring the radiant power behind an aperture using SYRES and the diode. Two beamlines in the PTB radiometry laboratory at BESSY are specially designed for detector calibration with monochromatic radiation. A 1 m McPherson-type normal-incidence monochromator (NIM) covers the energy range 3–35 eV (Lau-Främbs *et al.*, 1995/1996), and a SX700-type grazing-incidence monochromator (GIM) covers the range 40–1500 eV (Scholze *et al.*, 1994).

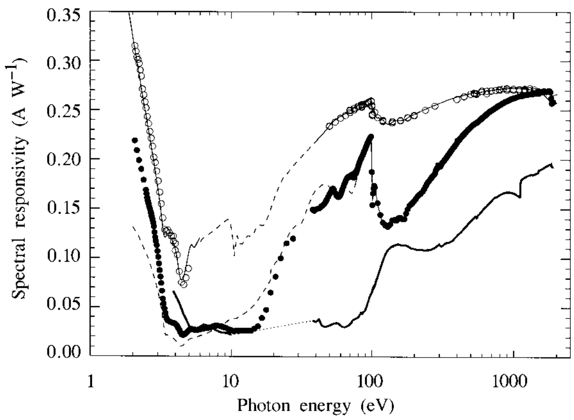

Figure 1
Spectral responsivity of a silicon n-on-p diode (AXUV100G; open circles) and PtSi/Si (closed circles) and GaAsP/Au (G2119; thick solid line) Schottky barrier diodes measured against SYRES. The thin lines represent a model calculation for the silicon diodes; dashes indicate the spectral regions where the model is not reliable due to a lack of optical data.

At the GIM, the storage-ring current can be used as a reference signal to follow the monotonic decrease of the storage-ring output. The ratio of the spectral radiant power, measured with SYRES at a photon energy of 140 eV in the focal point, to the stored electron current is constant within 0.07% (1σ) (Rabus *et al.*, 1996). In the UV/VUV spectral range covered by the NIM, due to source-size effects and heat-load problems at the condenser mirror, the radiant power does not scale linearly with the beam current. Therefore, a flux monitor has been established. The radiant power at the detectors is proportional to the flux monitor signal within 0.2% (1σ) (Lau-Främbs *et al.*, 1995).

Typical results for the spectral responsivity of n-on-p silicon photodiodes (AXUV100G) (Korde *et al.*, 1993) and PtSi/Si (Solt *et al.*, 1996) and GaAsP/Au (G2119) Schottky barrier photodiodes at room temperature are shown in Fig. 1. The typical uncertainty of measurements at about 1 μW radiant power is 1%.

The use of photodiodes as transfer detector standards requires the spectral responsivity to be stable under irradiation. The results of a recalibration of an AXUV photodiode against SYRES in the soft X-ray spectral region after one and two years of storage in air are shown in Fig. 2. We obtained a reproducibility of the calibration within 1% over that period. The stability under irradiation by soft X-rays of 140 eV photon energy, however, decreased significantly after two years (Fig. 3). The degradation was more severe for the diodes kept in the dark and under vacuum for the two-year period. These stability problems are even more pronounced in the VUV spectral range (Fig. 3). To overcome these problems, PtSi/Si Schottky barrier photodiodes have been developed for the UV/VUV spectral region (Solt *et al.*, 1996). No instability of these diodes was detected for exposure levels of up to 400 mJ cm^{-2} at 8.3 eV photon energy (150 nm) (Fig. 3).

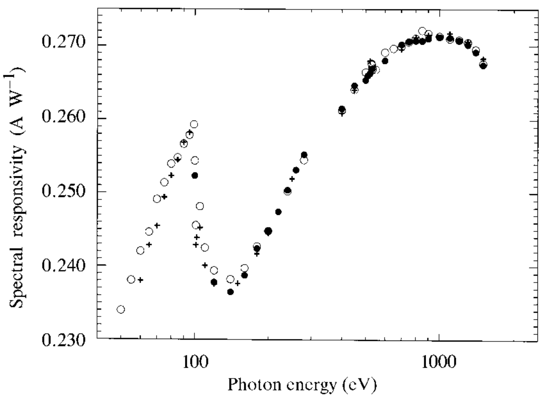

Figure 2
Measurement of the spectral responsivity of an AXUV silicon diode against SYRES over a period of two years. February 1995: closed circles; January 1996: open circles; March 1997: crosses.

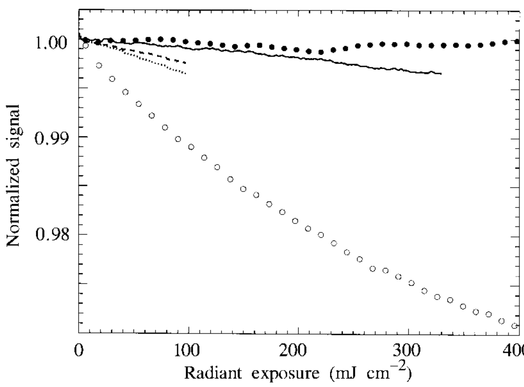

Figure 3
Stability of a PtSi/Si Schottky barrier diode (closed circles) and an AXUV n-on-p diode (open circles) under irradiation by 8.3 eV (150 nm) photons. The normalized responsivity is shown as a function of exposure. The stability of an AXUV diode under irradiation with 140 eV photons is shown additionally: initial performance (solid line) and after two years of storage in air (dashed line) and under vacuum (dotted line).

3. Measurement of the detection efficiency of photon-counting detectors

Many photon detectors for applications of UV/VUV and soft X-ray radiation operate in the photon-counting mode. A comparison between these highly sensitive detectors and the rather insensitive SYRES at the same radiant power is not possible. The PTB radiometry laboratory at the storage ring BESSY, however, offers the unique possibility of running the storage ring according to the requirements of radiometry. The electron current stored during normal operation corresponds to about 1×10^{12} stored electrons. By decreasing the number of stored electrons by up to 12 orders of magnitude, the photon flux can be adjusted to match the dynamic range of different detectors which can thus be compared. At stored electron currents below about 1 mA, the source size does not change and the heat load is no problem, so that also for the NIM, where the flux monitor can be used only at higher radiant power levels, the stored current can be used as a monitor signal for the photon flux.

The calibration of photon-counting detectors is performed in the following way. First, the photon flux behind the monochromator is measured for the photon energies of interest, using a photodiode which was calibrated against SYRES, at a stored electron current of about 1 mA. Afterwards, the electron current needed for the calibration of the counting detector is determined from the measured photon flux per stored current and the count-rate capability of the detector. Finally, the stored current is set to the desired values with a typical uncertainty of about 0.5%. The counting detector is calibrated by comparing the number of measured pulses with the known number of photons.

Two examples are shown to illustrate the results that can be achieved. Fig. 4 shows the detection efficiency of a CCD detector for the Advanced X-ray Astrophysics Facility (AXAF) of NASA

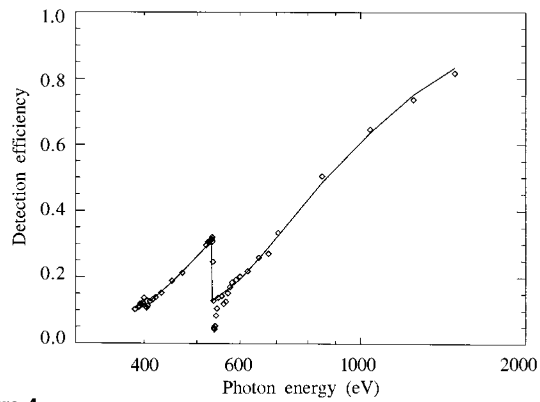

Figure 4
Detection efficiency of a front-illuminated CCD, measured at the GIM. The line represents a calculation using the Henke atomic scattering factors for the front-layer absorption. Note the pronounced fine structure measured at the O *K*-edge at 530 eV.

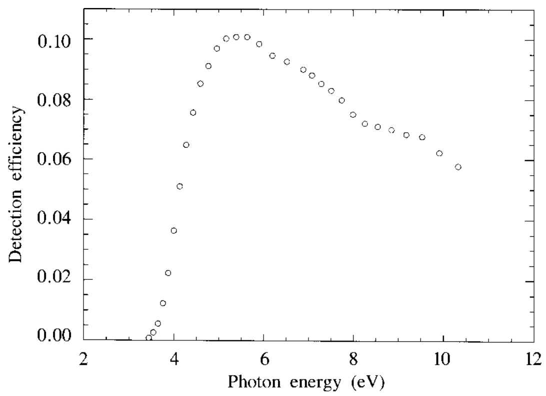

Figure 5
Detection efficiency of a photomultiplier, measured at the NIM.

in the soft X-ray spectral region, measured at the GIM (Prigozhin *et al.*, 1997). The detection efficiency of a photomultiplier (THORN EMI 9412 with CsTe-cathode) in the photon energy range of the NIM is shown in Fig. 5.

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