Detector-based Radiometry with Cryogenic Radiometers and Monochromatized Synchrotron Radiation

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Introduction

Detector-based radiometry is based on the use of primary detector standards for the measurement of absolute radiant power. For this purpose, electrically calibrated substitution radiometers have – since their introduction more than 100 years ago – been established for optical radiometry [1]. These radiometers are thermal detectors which are based on the equivalence of electrical heating and the radiation heating of an absorber. In the 1980s, the measurement uncertainty was clearly reduced by an operation at the temperature of liquid helium and in connection with lasers [2–7]. Cryogenic electrical substitution radiometers today are used by many national metrology institutes for the realization of the scale of the spectral responsivity of photodetectors from the infrared to the ultraviolet spectral range, whereby the smallest relative uncertainties can be achieved in the range of $10^{-4}$ when the detectors are used in combination with intensity-stabilized lasers as radiation sources [8].

In the spectral ranges of the vacuum-ultraviolet (VUV), the extreme ultraviolet (EUV) and X-ray radiation, where no lasers exist, monochromatized synchrotron radiation from storage rings offers very good preconditions for detector-based radiometry, in particular due to the broadband radiation and the resulting continuous tunability of the wavelength (in combination with suitable monochromators). In contrast to laser radiation, in the case of which radiant powers of a few milliwatts can typically be used, the power of the monochromatized radiation, which is available at the bending-magnet beamlines, amounts, however, to not more than 50 µW (partly to even less than 1 µW). The stability of the synchrotron radiation is not limited by stochastic variations, but by the continuous, nearly exponential decay of the electron current in the storage ring with typical time constants in the range of a few hours. In contrast to this so-called decay mode, many storage rings (also at BESSY II) allow operation today in the top-up mode, in the case of which the electron current is kept almost (better than 1 %) constant. To achieve small uncertainties in radiation measurements in the decay mode, the measurements must be performed within a few minutes. Due to the – compared to lasers – relatively low power of synchrotron radiation, ionization chambers have been used for a long time as primary detector standards here [9]. These allow, however, measurements of the radiant power to be performed only with relative uncertainties of a few percent [10].

Conventional cryogenic electrical substitution radiometers are suitable for measurements with synchrotron radiation only after considerable adaptations. In its laboratory – first at the storage ring BESSY I [11], since 1999 at BESSY II [12], and since 2008 also at the Metrology Light Source (MLS) [13] – PTB has continuously developed and improved detector-based radiometry with synchrotron radiation on the basis of specially adapted cryogenic radiometers. By now, this technique can be regarded as established and is meanwhile also used by other national institutes, such as NIST and NMIJ [14, 15]. Based on the use of cryogenic electrical substitution radiometers as primary detector standards, and by using monochromatized synchrotron radiation, PTB has established a scale of the spectral responsivity which extends from the UV range to the X-ray range [16–19]. The scale is disseminated by semiconductor photodiodes which have been calibrated against the primary standard, using – as a function of the spectral range – different photodiode types as secondary detector standards.
PTB’s cryogenic radiometers for synchrotron radiation

PTB has two cryogenic radiometers at its disposal which have been especially developed for use with monochromatic synchrotron radiation [20–22]. These thermal detectors, which are called SYRES I and II (SYnchrotron Radiation Electrical Substitution Radiometer) and are based on cavity absorbers, are operated at temperatures slightly above the boiling point of liquid helium. Both systems are windowless and, thus, directly connected to the beamlines for synchrotron radiation [23, 24] under ultra-high-vacuum conditions. Use of a metallic cavity absorber guarantees high absorptance – compared to other absorber types (e.g. disk absorbers) – to be achieved while secondary effects such as photoemission or fluorescence are, at the same time, effectively suppressed. Although the metallic absorbers have – due to the contribution of the conducting electrons – a higher thermal capacity than insulators, they prevent non-thermal energy losses such as, for example, the formation of color centers in insulating absorbers, like sapphire.

Figure 1 shows the essential components of the cryogenic electrical substitution radiometer: A cavity absorber of the thermal capacity \( C \) is coupled via a thermal resistance \( R \) to a heat sink of the temperature \( T \) which is kept constant by means of an electronic control. The heat sink is in thermal contact with a helium-bath cryostat. When irradiated with the radiant power \( \Phi \), the temperature of the absorber increases by \( \Delta T \) compared to the original temperature. The time constant \( \tau = R \times C \) of the temperature change is given by \( R = \Delta T / \Phi \).

The same temperature increase can be achieved by electrical heating, i.e. the radiant power \( \Phi \) can be substituted by the electric heating power \( P \). This substitution is based on the assumption that electrical heating and radiation heating are equivalent. Generally, the radiometers are operated in the so-called dynamic substitution mode: Already before the irradiation, the temperature of the absorber is regulated to a constant value \( T + \Delta T \) by electric heating using a resistance heating element. If the radiant power to be determined now is absorbed, the electric heating power must be reduced by exactly the amount to keep the temperature constant (Figure 2). The measurement of the radiant power is, thus, traced to a difference measurement of the electric heating power and to the electrical quantities associated with it.

To adapt the conditions to the special use of synchrotron radiation, some essential modifications are required compared to conventional cryogenic radiometers with cavity absorbers, in particular in view of the sensitivity and the suppression of the thermal radiation background. Due to the low radiation power available, the thermal resistance \( R \) must be large to achieve sufficient sensitivity in the range of some ten mK/\( \mu \)W. In addition, the thermal capacity must be small to achieve a justifiably small time constant \( \tau \) in the range of less than one minute in order to keep the change of the beam intensity due to the monotonously decreasing ring current in the storage ring smaller than the measurement uncertainty aimed at. To implement these requirements, the cavity absorbers of the cryogenic radiometers are made of electrolytically formed copper with a wall thickness of approx. 100 µm. To achieve a high value for the thermal resistance in the coupling to the heat sink, the absorbers are suspended on thin nylon threads. The defined thermal coupling
is realized by a wire, respectively, made of copper or constantan which allows the thermal resistance to be varied to a certain extent by an adaptation of the length.

The uncertainty which can be achieved in the determination of the radiant power is limited by the capacity of the absorber to completely absorb the radiation incident on it (i.e. to have an emissivity close to unity), and to convert it (without losses) into heat. Whereas in the UV and VUV spectral ranges, it is the reflection of the radiation on the surface of the absorber which represents the essential loss factor which must be taken into account, it is the transmission losses (due to insufficient wall thickness) as well as fluorescence and scattering processes which come to the fore in the X-ray range. To allow measurements at photon energies of up to 60 keV to be performed, the cylindrical cavity absorber (40 mm in length) whose wall consists of copper (90 µm in thickness) with a gold layer (approx. 1 µm in thickness) was closed with a solid gold plate (730 µm in thickness) as back wall which is inclined by 30° to the perpendicular (Figure 3a) [23]. This absorber is used in the SYRES-I instrument and enables the radiometric traceability of detector-based calibrations in the photon energy range between 30 eV and 60 keV. The SYRES-II radiometer has, however, been developed for dedicated use in the photon energy range between 3 eV and 30 eV (corresponding to wavelengths of 400 nm to 40 nm). Here, back plate and cylindrical walls of the cavity absorber have almost the same wall thickness; the copper has also been covered with a gold layer (approx. 1 µm in thickness). This layer is to generate as high a directed (specular) reflection as possible. With a length of 80 mm, the cavity absorber is twice as long as the absorber used in the X-ray range. In addition, a so-called retro-reflector, which consists of two truncated cones set with the basis against each other (slope: 30°, length: 9 mm), is installed at the entrance (Figure 3b) [16]. By means of this geometry, a beam, which is completely directed at the back wall of the absorber, is again reflected into the cavity. Leaving of the cavity is possible only after more than 15 reflections on the surface. For an assumed reflectivity of 0.4, the reflection losses are, thus, smaller than 10⁻⁶. In the case of the two absorbers used, the electrical heater and a germanium resistance thermometer are applied to the outside of the beveled back wall. To guarantee the equivalence of electrical heating and heating by radiation as well as possible, uncontrolled heat flows must be suppressed. The radiation absorber (cavity absorber) is located in the high vacuum to rule out heat derivation by convection. In these temperature ranges, thermal radiation, too, plays no role in the heat transport. The current leads for heater and resistance thermometer consist of superconducting materials, so that hardly any Joule’s heat is generated in the range of the working temperatures. In addition, an inhomogeneous temperature distribution is minimized by the high thermal conductivity of the absorber material at these low temperatures.

As the beamlines are at ambient temperature, an infrared radiation power of a few milliwatts falls from the semi-infinite space onto the absorber. The whole absorber module must, therefore, be surrounded by a cooled cryocollimator to reduce the solid angle for the incident infrared radiation approximately to that of the synchrotron radiation. In the case of SYRES II, the absorber module can, in addition, be vertically lifted under vacuum from the beam path, together with the associated cryostat, to carry out measurements with the absorber and the detector to be calibrated at exactly the same beam position. In doing so, it is ensured that also exactly the same amount of radiant power falls on the primary detector standard and on the detector to be calibrated, independent of the divergence and of the stray light halo of the beam.

Calibration of transfer detector standards

In the laboratories of PTB at BESSY II and at the MLS, the cryogenic radiometers described in the preceding section are used as primary detector standards to measure the spectral responsibility of transfer detector standards [16–19, 25–32]. The calibration is performed by comparing the measured photocurrent of the transfer detector with the incident radiant power which is measured with the cryogenic radiometer. By the use of monochromatized synchrotron radiation, PTB realizes a scale of the spectral responsibility from the ultraviolet spectral range up to the X-ray

![Figure 3](image-url)

**Figure 3:** Cavity absorber of the two SYRES cryogenic radiometers. (a) SYRES-I absorber, length \(l = 40\) mm, diameter \(d = 8\) mm; top: photo, bottom: simulated X-ray scattering and fluorescence [23]. (b) SYRES-II absorber \(l = 80\) mm, \(d = 8\) mm; top: photo, bottom: geometric beam path for direct (specular) reflection of the incident beam [16].
range (Figure 4, top). It is applied to different detector types (semiconductor photodiodes) depending on the respective spectral range. The following detectors are used here: windowless semiconductor photodiodes of the Schottky type (PtSi-nSi-photodiodes) [31], detectors of the nSi-pSi type or Passivated Implanted Planar Silicon detectors [23]. Decisive for the selection of the detectors is aspects such as irradiation stability, linearity and homogeneity across the detector surface [30, 31]. Depending on the detector type and even more on the wavelength range and the corresponding instrumentation, different uncertainties are obtained for the scale and for the calibration of transfer detector standards (Table 1).

In the UV, VUV and soft X-ray ranges, the largest contribution to the total uncertainty comes from scattered light and higher grating diffraction orders of the monochromatized synchrotron radiation. These contributions differ considerably, depending on the optical configuration of the beamlines in the spectral sub-ranges (filter, grating, etc.) so that the achievable measurement uncertainty is different and shows an abrupt change at the respective range limits (Figure 4, bottom, and Table 1).

The scale of the spectral responsivity represented in this way has been validated by internal and external comparisons. As primary detector standard, SYRES II has, for example, been directly compared against the storage ring MLS as primary source standard [33, 34], whereas for SYRES I in the soft X-ray range, an energy-dispersive Si(Li) detector was used as transfer standard [35]. Further detector-based comparisons were performed against an open-air ionization chamber as another primary detector standard [23] and, with semiconductor photodiodes, against the radiometric scales of other national institutes [36–38].

In the dissemination of the scale, also the properties of the detectors, in particular the homogeneity of the responsivity across the detector surface, contribute essentially to the uncertainty budget. The irradiation stability and the general aging of the transfer detectors are, as a matter of principle, a problem in the dissemination of the scale and do not only depend on the detector type, but also on the wavelength and on the power of the incident radiation. Thereby, the aging of a detector relates to both the homogeneity and also to the spectral responsivity [39, 40]. Particularly relevant here is the aging of semiconductor photodiodes in the wavelength range of the VUV, especially for wavelengths between approx. 50 nm and 150 nm [41, 42]. The cause of it lies in the high radiation absorption of all materials in this wavelength range so that the penetration depth of the radiation amounts to only a few nanometers. The whole energy of the incident photons is, thus, absorbed in the thin top layer of the detector. This means, on the one hand, that the quality of the top layer (uniformity, lack of structure errors) essentially determines the properties of the detector and, on the other hand, that this top layer is subject to strong aging processes by radiation damage. A distinction must be made between aging processes and changes in the spectral responsivity by contamination of the detector surfaces. Secondary electrons are emitted from the detector surface – in particular at a sufficiently high energy of the photons (larger than approx. 10 eV, corresponding to wavelengths smaller than 120 nm) – whose kinetic energies are sufficient to chemically break surface-absorbed hydrocarbons and, thus, to generate a constantly increasing, strongly absorbing carbon layer.

Silicon photodiodes are widely used as radiometric secondary standards. Due to the advanced manufacturing techniques, the spectral responsivity of these photodiodes can reach the theoretical maximum value. A natural oxide growth is – in the case of silicon – however, unavoidable, so that

<table>
<thead>
<tr>
<th>Quantity</th>
<th>Relative standard measurement uncertainty of spectral responsivity ($k = 1$) / %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Photon energy</td>
<td></td>
</tr>
<tr>
<td>Radiant power</td>
<td>0.14                                                               0.11   0.13   0.13</td>
</tr>
<tr>
<td>Measurement signal</td>
<td>0.10                                                               0.10   0.10   0.20</td>
</tr>
<tr>
<td>Calibration factor</td>
<td>0.03                                                               0.06   0.01   0.01</td>
</tr>
<tr>
<td>Wavelengths</td>
<td>0.02                                                               0.01   0.01   0.02</td>
</tr>
<tr>
<td>Spectral bandwidth</td>
<td>0.008                                                              0.001  0.001  0.001</td>
</tr>
<tr>
<td>Scattered light, higher</td>
<td>0.15                                                               0.2    &lt;0.001 &lt;0.001</td>
</tr>
<tr>
<td>diffraction orders</td>
<td></td>
</tr>
<tr>
<td>Spectral responsivity</td>
<td>0.25                                                               0.26   0.17   0.25</td>
</tr>
</tbody>
</table>
a technically generated (possibly ni-trided) oxide is – as a matter of principle – applied as top layer for passivation. Photodiodes for the visible and near UV spectral ranges can, thereby, even have thick (some 10 nm to 100 nm) oxide layers with antireflection properties. However, for the ranges with even shorter wavelengths, only diode types with ultra-thin (a few nm) passivation layers are suited to produce enough electron-hole pairs in the depletion zone which then lead to a measurable signal. The oxide passivation layer itself is not electrically conductive. Due to the (external) photoeffect, the radiation leads to an electrical (positive) charging of the top layer in the range of the irradiation by which the sensitivity of the photodiode is changed. The use of a metallic silicide top layer instead of the oxide for passivation can remedy the problem [43]. However, it reduces the spectral responsivity in the relevant spectral range by almost one order of magnitude due to the higher absorption in the top layer. Although Schottky photodiodes (with platinum silicide as metallic contact) [31] prove to be very insensitive to irradiation, they are – due to their electrical properties (low internal resistance) – suited for the measurement of small photocurrents only to a limited extent. Photodiodes with different implant ions (e.g. phosphor, arsenic or boron) and implant profiles have, in principle, been known for a long time already [44], however, conventional implantation techniques do not generate sufficiently small depletion zones. Only for some years has it been possible to control corresponding techniques in semiconductor laboratories so that photodiodes for the VUV spectral range with high sensitivity and, at the same time, high resistance to radiation [45] have become available.

In addition to the semiconductor photodiodes which are also used internally as transfer standards, also other radiation detector types are calibrated (for example: vacuum photocathodes, photomultipliers and bolometers) [46]. Spatially resolved calibrations of imaging detectors such as, for example, Charged Coupled Devices (CCD) [47] or sensors with individually readable pixels (Active Pixel Sensors, APS) [48] are possible.

For the measurement of highly intensive, pulsed radiation of free-electron lasers, especially developed detectors, based on the atomic photoionization of (noble) gases, have been calibrated traceably to a cryogenic radiometer [49, 50].
References

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