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X 射线辐射测量

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摘要：在过去的几年里，人们使用同步辐射技术在 X 射线波段实现了基于辐射源和基于探测器的辐射测量，电子储存环被用作主辐射源标准，而低温辐射计（ESR）被用作主探测器标准。因为低温辐射计需要强的单色辐射，所以，这种方式也必须使用同步辐射。文中对能量色散探测器和硅光电二极管的探测效率和响应度做了校准，其相对不确定度约 1%，所得结果与模型计算值做了比较。

关　键　词：X 射线辐射测量；同步辐射；辐射源；探测器
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X-ray radiometry

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Abstract： Source-based and detector-based radiometry has been realized in the X-ray range during the last few years by using synchrotron radiation. Electron storage rings are operated as primary source standards and cryogenic Electric Substitution Radiometers (ESRs) as primary detector standards. As ESRs require intense monochromatic radiation, synchrotron radiation is also necessary for this approach. Energy-dispersive detectors as well as silicon photodiodes have been calibrated with relative uncertainties of the detection efficiency or responsivity down to about 1%. The results are compared to model calculations.

Key words： X-ray radiometry；synchrotron radiation；radiation source；detector

1　Introduction

Quantitative measurements of X-rays require absolutely calibrated detectors. The calibration has to be based on primary standards，either directly or via secondary (transfer) standards. As in the visible spectral range，two complementary approaches have been realized in the X-ray range：source-based radiometry using a calculable radiation source，and detector-based radiometry starting from an absolute detector[1]. Both approaches require the use of synchrotron radiation. Examples of the instrumentation as well as overviews of the facilities existing worldwide are presented，together with applications

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for the calibration of energy-dispersive and non energy-dispersive detectors such as silicon photodiodes.

2 Source-based X-ray radiometry

In the visible spectral region, the well-established source of calculable radiation is the black-body radiator. As shown in Fig. 1, it covers the infrared, the visible and the UV region, but even at the highest possible temperatures in the range between 3 000 K and 3 500 K, the radiation does not extend into the X-ray range. The only available primary source standards in the X-ray range are electron storage rings. As an example, Fig. 1 shows the spectral radiant power of a bending magnet at the storage ring BESSY II in Berlin. The radiation covers the entire UV, VUV and soft X-ray range and can be used up to about 20 keV. If higher photon energies are required at the same facility, a superconductive Wavelength-shifter (WLS) can be used. At the BAM-WLS - which is a 7 T device installed at the beamline of the German Federal Institute for Materials Research and Testing (MRT)-the useable spectral range can be extended up to about 150 keV for radiometric purposes[2]. While the spectrum of any bending magnet or WLS can be calculated according to the Schwinger formula[3], the main requirement for a primary source standard is the precise determination of all parameters entering into this formula. These parameters can be divided into two groups:

the storage ring parameters:

- electron energy
- electron current
- magnetic induction in the bending magnet or the WLS
- vertical extension and divergence of the electron beam

and the geometrical parameters:

- size of the flux-limiting aperture
- the distance of the aperture from the source point
- vertical emission angle to the orbital plane

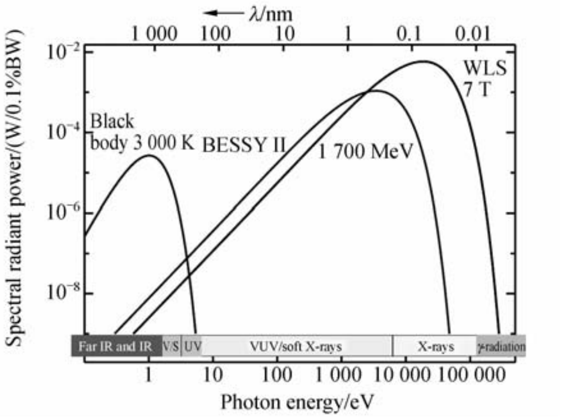

Fig. 1 Calculated spectral radiant power of primary source standards: black-body radiator, BESSY II dipole magnet and 7 T WLS at BESSY II

In the laboratory of the Physikalisch-Technische Bundesanstalt (PTB) at BESSY II, most of these parameters can be determined with relative uncertainties of about 10^{-4}[4]. The resulting uncertainty in the spectral radiant power of a bending magnet varies between about 2×10^{-4} in the photon energy range below 1 keV to 5×10^{-4} at 10 keV. While the uncertainty at low energies is dominated by the contribution of the geometrical quantities and the electron current, it is limited at high photon energies by the uncertainty of the electron energy and the magnetic induction. The uncertainty of the magnetic induction is also responsible for the higher uncertainty at the WLS[5] as shown in Tab. 1, which lists all electron storage rings used as primary source standards. As can be seen in Tab. 1, several facilities are operated in the US, Germany, Japan and China with characteristic photon energies well below 1 keV so that they cannot be used in the X-ray region. This region is only covered by two facilities: the BESSY II storage ring (with bending magnets and a WLS) used by PTB, and the VEPP-3 storage ring operated by the Budker

Tab. 1 Primary source standards based on calculable synchrotron radiation, ordered by characteristic energy. Typical relative uncertainties are extracted from available references. The Metrology Light Source (MLS) will come into operation in 2008

Ring name	Used by	Location	E_c/ keV	u_{typical}/ % (at E_c)
SURF III	NIST	Gaithersburg, USA	0.17	0.25[18]
MLS (2008)	PTB	Berlin, Germany	0.31	
HLS	NSRL	Hefei, China	0.52	
TERAS	NMIJ	Tsukuba, Japan	0.57	0.3[19]
BESSY II	PTB	Berlin, Germany	2.5	0.03[4]
VEPP-3	INP	Novosibirsk, Russia	4.8	4[6]
BESSY II WLS	PTB	Berlin, Germany	13.0	0.3[5]

Institute in Novosibirsk, Russia[6]. However, the typical uncertainties at the facilities differ by orders of magnitude.

The calculable undispersed radiation of the primary source standards can be used to calibrate a spectrometer by placing it behind the flux-limiting aperture. In the X-ray range, the ′spectrometer′ is typically an energy-dispersive detector. However, these detectors have to be operated in the photon-counting mode with photon fluxes of 10^3 s^{-1} to 10^4 s^{-1}, while synchrotron radiation in the normal operation of a storage ring is known to be very intense. By drastically reducing the electron current, the photon flux can be reduced without modifying the spectrum. While the normal electron current at BESSY II ranges between 150 mA and 250 mA, detector calibrations are typically performed in the pA range. Even single electrons can be stored for hours in the ring having 240 m circumference where one electron corresponds to a current of 0.2 pA.

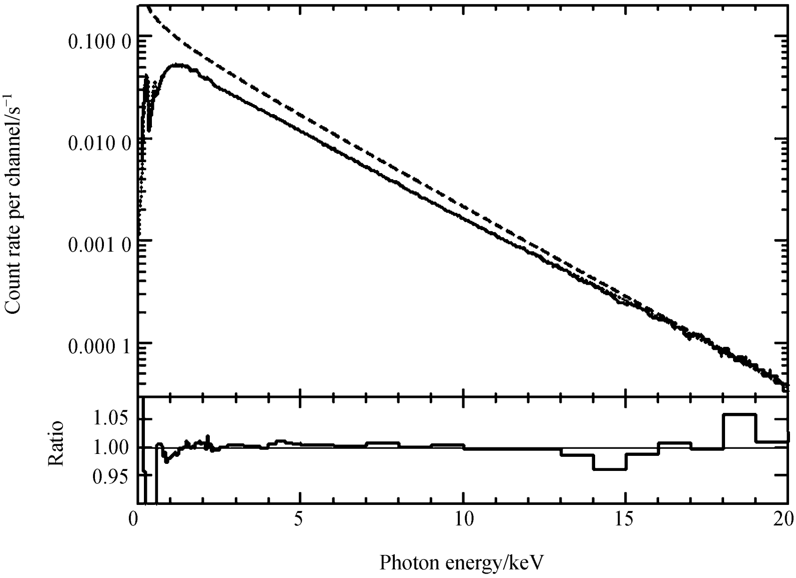

Fig. 2 Measured spectrum of a Si(Li) detector in bending magnet radiation from BESSY II (solid line), calculated incident spectrum (dashed line) and calculated spectrum, including the transmittance of the detector entrance window and a convolution with the response function (dotted line). The lower part shows the ratio of this calculation to the measured spectrum.

As an example for the detector calibration with bending magnet radiation, Fig. 2 shows the calculated spectrum as well as the spectrum measured with a Si(Li) detector[7]. Also shown is the product of the calculated spectrum and the modelled detection efficiency which in this case is governed by the transmittance of the detector entrance window, consisting of a MOXTEC foil on a support structure. The ratio of this product to the measured spectrum is unity throughout the energy range, proving that the detection efficiency was correctly modelled.

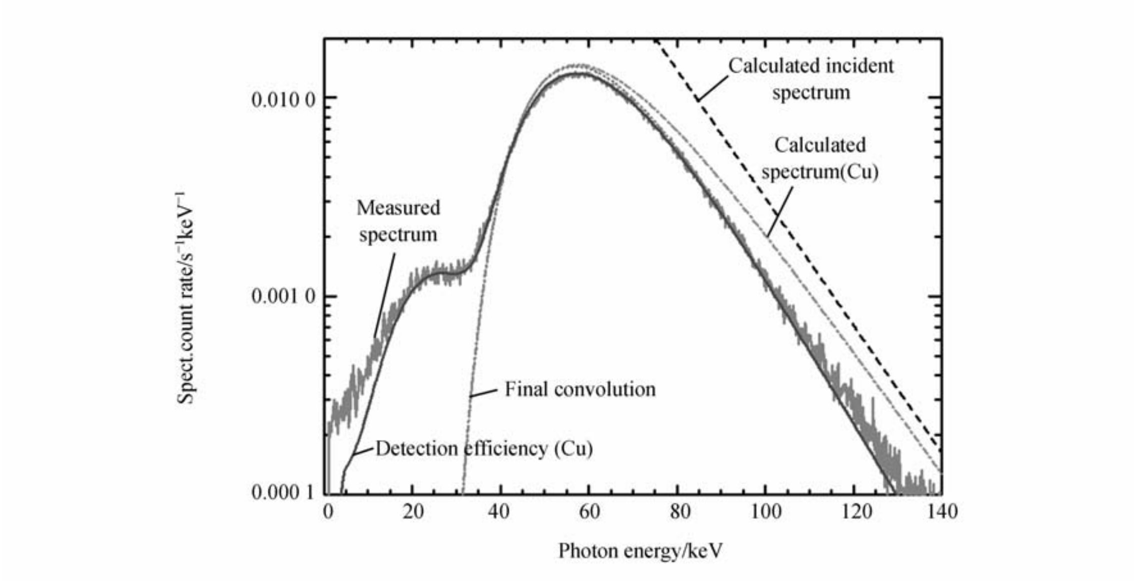

Fig. 3 Measured spectrum of a CdTe detector in WLS radiation(thick line), calculated incident spectrum (dashed line), calculated spectrum including the transmittance of a 1 mm thick Cu filter (dotted-dashed) and detection efficiency (solid line) as well as the final convolution with the response function (dotted line)

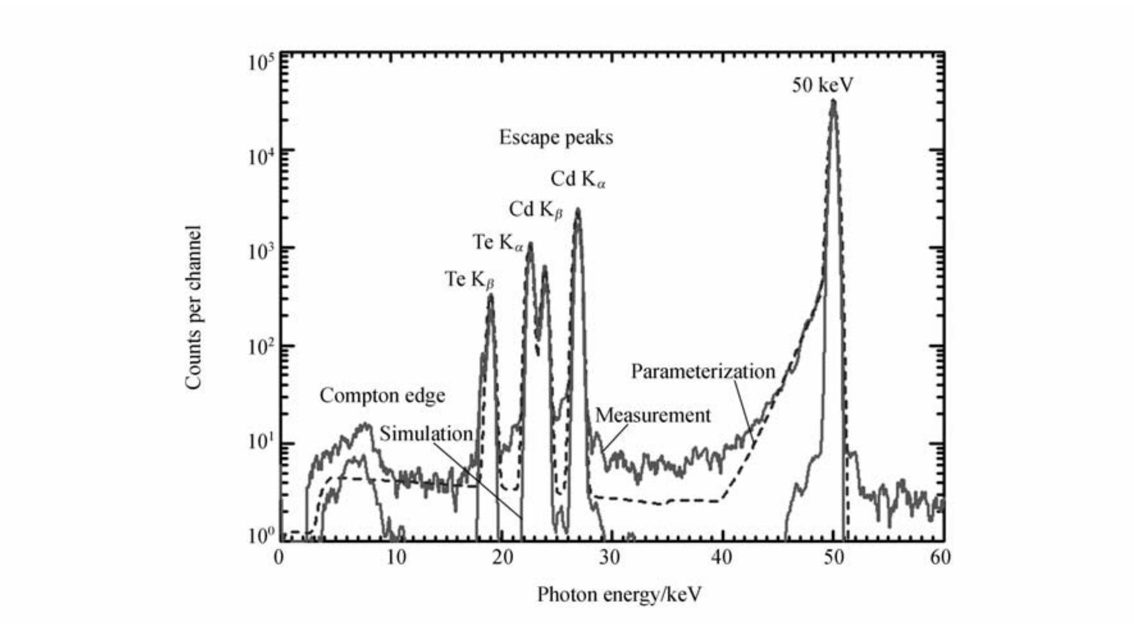

Fig. 4 Response function of a CdTe detector for monochromatic radiation at 50 keV: measurement, simulation with GEANT4, and parameterization using an analytical model

For higher photon energies, CdTe detectors are becoming more and more popular because, in contrast to high-purity Ge detectors, they only require thermoelectric cooling instead of more demanding cooling with liquid nitrogen. A spectrum measured with a CdTe detector at the BAM line WLS is shown in Fig. 3[8]. Here again, the calculated incident spectrum is shown, but this time also multiplied with the transmittance of a 1 mm thick Cu filter which was used to block the low energy radiation. The efficiency of the CdTe detector in this energy range is mainly determined by its thickness, which turned out to be around 1 mm as specified. However, the measured spectrum can only be reproduced if the product of incident spectral photon flux, filter transmittance and detection efficiency is convoluted with the detector response function. The response function has to be determined independently with monochromatic radiation. Fig. 4 shows the response function which was measured with monochromatized radiation of 50 keV at the BAMline[2]. Apart from the photoelectric peak at 50 keV, which has a Gaussian shape and a low energy tail due to incomplete charge collection in the CdTe crystal, the spectrum exhibits pronounced escape peaks due to fluorescence radiation leaving the detector volume and resulting in peaks at the energy difference to the incident radiation. Also shown are calculations, which are either based on a Monte Carlo simulation using Geant4[9] or on an analytical model. Whereas in the analytical model, Compton scattering is not included, in the Monte Carlo program the incomplete charge collection in the semiconductor is not calculated. The dominating escape peaks are correctly described by both calculations.

Source-based radiometry has been successfully applied to calibrate a large variety of energy-dispersive X-ray detectors with relative uncertainties of about 1%. Some of the X-ray detectors of the space observatories Chandra[10], operated by NASA, and XMM-Newton[11], operated by ESA, have been calibrated by PTB at BESSY I, the precursor of BESSY II.

3 Detector based X-ray radiometry

Non energy-dispersive detectors such as semiconductor photodiodes or ion chambers, operated in the current mode, cannot be calibrated in undispersed synchrotron radiation. Here, monochromatic radiation of known radiant power is required. As monochromatic primary source standards do not exist in the X-ray range and as the radiant power at a monochromator beamline is no longer calculable with small enough uncertainties, a primary detector standard has to be used to measure the radiant power at such a beamline. The most important requirement for the radiation is high spectral purity, especially the absence of higher-order radiation. As in the visible spectral range, Electrical Substitution Radiometers (ESRs) are used as a primary detector standard[12]. This implies that the radiant power has to be in the nW- or, better, μW-range. As monochromators in combination with X-ray tubes deliver a much lower radiant power, monochromatized synchrotron radiation has to be used.

As shown in Fig. 5, the main component of an ESR is a cavity absorber which is coupled to a heat sink by a heat link. All components are in UHV and are cooled down to liquid helium temperatures. On the cavity absorber, a thermometer and an electrical heater are mounted. An electrical power is applied to heat the absorber to a selectable temperature in the range between 4.2 K and 7 K. If radiation enters the absorber, the electrical power is reduced by a control loop to keep the temperature constant. The difference in electrical heating power which can easily be measured equals the radiant power. This assumes that the incident radiation is fully absorbed and transformed into heat. For the hard

X-ray range, a dedicated cavity has been produced by electroforming. The cavity consists of a 40 mm long Cu tube of 8 mm diameter with a 500 μm thick gold bottom to absorb all photons with energies up to 50 keV[13]. This cavity has recently been installed in an ESR called SYRES I in the PTB laboratory at BESSY II.

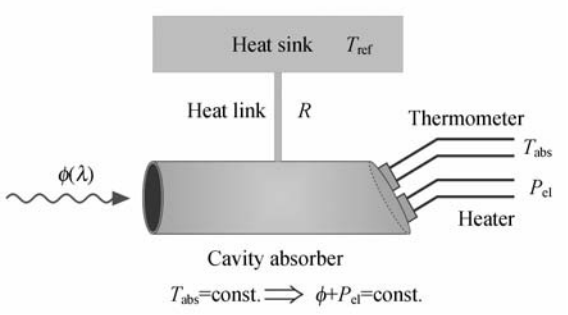

Fig. 5 Operating principle of a cryogenic ESR

All existing ESRs which are operated with monochromatized SR are listed in Tab. 2. While the ESR of NIST as well as SYRES II of PTB are used in the UV and VUV region only, the instruments of CEA in France and NMIJ in Japan are not yet in regular operation, but some test measurements have recently been made[14-15]. Currently SYRES I of PTB is the only ESR that is used throughout the X-ray range at different beamlines: in addition to a soft X-ray plane grating monochromator beamline for the range from 50 eV to 1.8 keV, a Four-crystal Monochromator (FCM) beamline is used to provide monochromatic radiation in the range from 1.75 keV to 10 keV, while a beamline with a Double Crystal Monochromator (DCM) and a Double Multilayer Monochromator (DMM) at a WLS (BAMline) is used in the range from 8 keV to 60 keV.

Tab. 2 ESRs operated as primary detector standards with monochromatized synchrotron radiation. The ESRs of CEA and NMIJ have been used for test experiments. The metrology beamline at SOLEIL will begin operation until 2009.

Radiometer, Facility	Used by	Location	Energy range / keV
ESR, SURF III	NIST	Gaithersburg, USA	0.004 to 0.1
BOLUX, SOLEIL (2009)	CEA	Saclay, France	?
ESR, PHOTON FACTORY	NMIJ	Tsukuba, Japan	0.1 to 4
SYRES II, BESSY II / MLS	PTB	Berlin, Germany	0.003 to 0.03
SYRES I, BESSY II	PTB	Berlin, Germany	0.05 to 60

At the FCM beamline, either four InSb(111) crystals or four Si(111) crystals monochromatize the radiation. A Pt-coated toroidal mirror is used for horizontal focussing, while a plane mirror with bender focuses the beam vertically. This plane mirror is equipped with two coating stripes: Pt and MgF_2. Both crystal sets and the mirror coatings can be changed under vacuum according to the desired photon energy range. The spectral purity of the monochromatized radiation is depicted in Fig. 6 for the Si(111) crystals. The contribution of higher orders, which were measured with an energy-dispersive Si(Li) detector, are always below 10^{-4}. It decreases rapidly towards higher energies and remains below 10^{-6} above 3.5 keV. This high purity is due to the four-crystal design and the use of the MgF_2 coating for energies below 4 keV[16].

At the BAMline, the DCM is usually equipped with two Si(111) crystals, but Si(311) is also available. Above 30 keV, the spectral purity is already high due to the spectral distribution of the WLS radiation, but at lower photon energies, the radiation from the DCM contains significant higher-order radiation which has again been measured with an energy-dispersive detector (Fig. 7). To suppress these higher or-

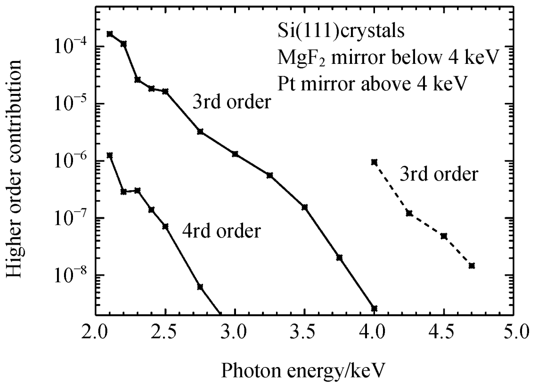

Fig. 6 Measured higher-order contributions at the FCM beamline for the Si(111) crystals and different mirror coatings

ders, a DMM consisting of two 300 mm long W/Si multilayers can be inserted in front of the DCM. Both monochromators can be operated in a fixed offset mode. The combination of the two monochromators ensures higher order contributions below 2×10^{-5} in the entire energy range by keeping the high spectral resolving power of the DCM. In addition, the heat load on the first DCM crystal is reduced by the DMM[2].

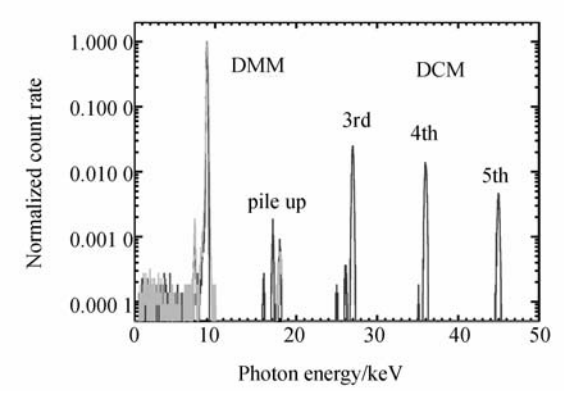

Fig. 7 Spectrum at the BAMline of the monochromatized radiation at 9 keV: the DCM provides high resolving power but significant higher-order contributions, while higher orders from the DMM are almost absent.

Many photodiodes have been calibrated against SYRES I at these beamlines. The results for three different diode types are shown in Fig. 8 in the energy range from 1. 75 keV to 60 keV. While the measured responsivities in the few-keV-range are almost identical, they differ by more than one order of magnitude at higher energies. This is due to the differences in the thickness of the active layer, as can be seen from the good agreement with the calculated responsivity based just on the energy absorption in a silicon layer. For the IRD AXUV 100 diodes, large sample-to-sample variations were observed at higher energies as only an epitaxially grown layer is depleted. The investigated Hamamatsu and Canberra diodes are fully depleted even without applying a bias voltage. The Canberra PIPS 500 diode has the thickest Si substrate and therefore the highest responsivity at high energies. Furthermore, it can be used in transmission as there is no mounting structure behind the active area. In contrast, the Hamamatsu PIN diode S3590, which is widely used in the X-ray range, is mounted on a ceramic support. This mounting is also the reason for the increased responsivity above 25. 5 keV: the Si substrate is obviously attached to the ceramic by conductive silver and the Ag-K fluorescence radiation - excited by the radiation that penetrates the Si substrate - contributes to the photocurrent. Mapping of the responsivity over the active area revealed pronounced inhomogeneities above the Ag-K edge due to the distribution of the conductive silver

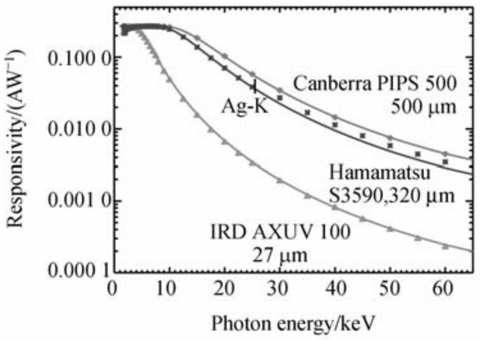

Fig. 8 Responsivity of different Si photodiodes calibrated against the ESR. The solid lines are calculations based on the energy absorption in a given thickness Si layer

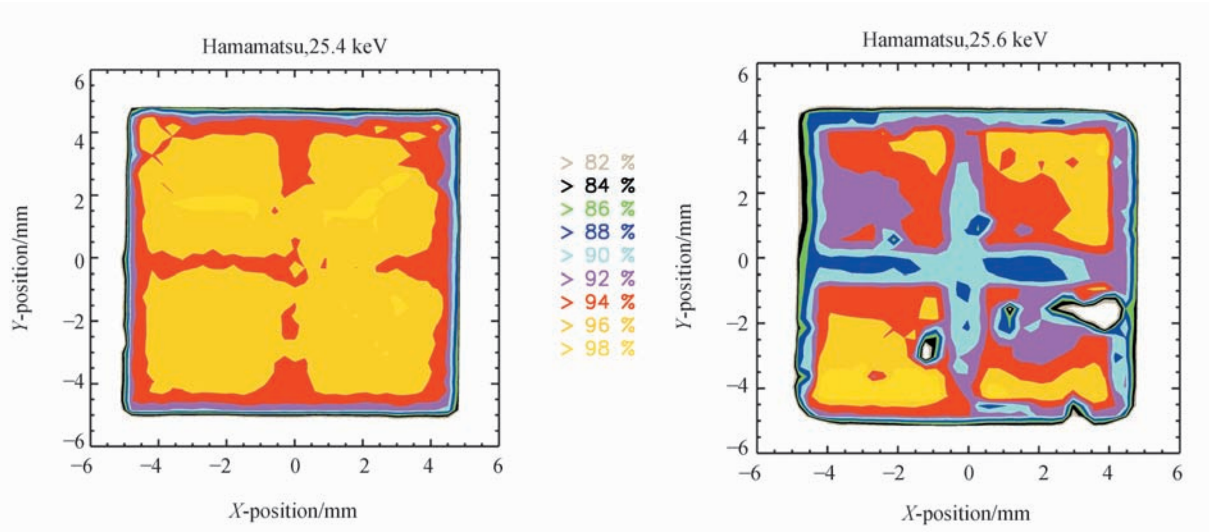

Fig. 9 Measured spatial distribution of the responsivity for a Hamamatsu S3590 photodiode below and above the Ag-K absorption edge at 25.5 keV. The inhomogeneities are due to the distribution of the conductive silver between the Si substrate and the ceramic support.

and less pronounced features below this edge as shown in Fig. 9[17]. For homogeneous photodiodes, the responsivity has been determined with relative uncertainties of about 1%. Calibrations have been performed by PTB for many other synchrotron radiation facilities like ESRF (France), SOLEIL (France), DIAMOND (UK) and APS (USA).

4 Conclusions

As the basis for absolute X-ray radiometry, synchrotron radiation is required: either as calculable radiation for source-based radiometry, or as intense monochromatized radiation in combination with an ESR for detector-based radiometry. However, detectors or spectrometers which were calibrated with synchrotron radiation can also be used at other X-ray sources. Currently, X-ray detector calibrations with relative uncertainties of about 1% are exclusively performed by PTB at BESSY II.

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