The new ultraviolet spectral responsivity scale based on cryogenic radiometry at Synchrotron Ultraviolet Radiation Facility III

Ping-Shine Shaw,[a] Thomas C. Larason, Rajeev Gupta, Steven W. Brown, Robert E. Vest, and Keith R. Lykke
National Institute of Standards and Technology, Gaithersburg, Maryland 20899

(Received 29 September 2000; accepted for publication 4 February 2001)

The recently completed upgrade of the Synchrotron Ultraviolet Radiation Facility (SURF III) at the National Institute of Standards and Technology (NIST) has improved the accuracy of radiometric measurements over a broad spectral range from the infrared to the soft x ray. The beamline 4 at SURF III is a cryogenic-radiometer based radiometric facility for the ultraviolet (UV) spectral range. The upgrade of SURF III has allowed us to use beamline 4 to improve the detector spectral power responsivity scales in the wavelength range from 125 to 320 nm. The achieved combined relative standard uncertainty is better than 0.5% over most of this spectral range. This is a significant improvement over the more than 6% relative standard uncertainty in this spectral range of the current scales maintained at the Spectral Comparator Facility (SCF) in the Optical Technology Division and the Far UV Calibration Facility in the Electron and Optical Physics Division. The new UV scale of beamline 4 was subsequently intercompared and transferred to the SCF and to the Far UV Calibration Facility to improve their UV scales and ensure consistency within NIST. The new scale established at beamline 4 improves NIST's calibration capabilities for environmental monitoring, astrophysics, and the UV industry. The new scale also includes wavelengths such as 193 and 157 nm excimer laser wavelengths, which are of particular interest to the semiconductor photolithography industry. [DOI: 10.1063/1.1361081]

I. INTRODUCTION

The National Institute of Standards and Technology (NIST) currently has two calibration facilities for ultraviolet (UV) detector spectral power responsivity measurement. One is the UV Spectral Comparator Facility (SCF)[1,2] in the Optical Technology Division and the other is the Far UV Calibration Facility[3–7] in the Electron and Optical Physics Division. The spectral responsivities of the SCF working standard detectors are derived from the High Accuracy Cryogenic Radiometer facility (HACR)[8,9] and extended to the UV range by comparison with a spectrally flat pyroelectric detector. The overall spectral range is from 200 to 500 nm and the relative standard uncertainty is less than 0.2% except near 200 nm where the relative standard uncertainty increases to 6%.[2] On the other hand, the scale of the Far UV Calibration Facility is derived from a combination of thermopile and the use of the Synchrotron Ultraviolet Radiation Facility (SURF) as an absolute source. The spectral range of the Far UV Calibration Facility is from 50 to 260 nm and the overall measurement uncertainty is about 5%.[7]

In response to rising needs from photolithography[10] and other UV industries for detector calibrations in the UV range with lower uncertainties than currently available, a cryogenic-radiometer based system was constructed on beamline 4 of SURF at NIST.[11,12] The system at beamline 4 was designed and constructed as a general purpose UV facility with functions including detector spectral responsivity measurements, detector radiation damage studies, and UV material characterization. For detector responsivity measurements, the response of a test detector is directly compared with the response of a cryogenic radiometer, thus eliminating any intermediate scale transfer. The spectral range of beamline 4 is from 125 to 320 nm, complementing the SCF and the Far UV Calibration Facility, and including the 193 and 157 nm excimer laser wavelengths that are currently being actively explored by the semiconductor industry for microelectronic device processing.

The primary standard for detector responsivity measurements on beamline 4 is an absolute cryogenic radiometer (ACR), which measures the incident radiant power by using electrical substitution at liquid helium temperature.[9] Cryogenic radiometers are the most accurate radiant power detectors currently available and are widely established at national laboratories as primary standards. For example, HACR is the primary standard of NIST from the near UV to the infrared.[8,9] The combination of a cryogenic radiometer with synchrotron radiation makes it possible to extend the spectral range of radiometric power measurements to as far as the x-ray regime. This was demonstrated at the Berlin electron-storage ring by the Physikalisch-Technische Bundesanstalt.[13–15]

At SURF, a major upgrade was performed shortly after the commissioning of the new radiometry facility at beamline 4. The upgrade from SURF II to SURF III[16,17] improved many aspects of the radiometric properties of SURF, e.g., the uniformity of the magnetic field along the electron trajectory, higher electron energy, higher electron beam current, and a more stable electron beam. For beamline 4, SURF III pro-

[a]Electronic mail: shaw@nist.gov

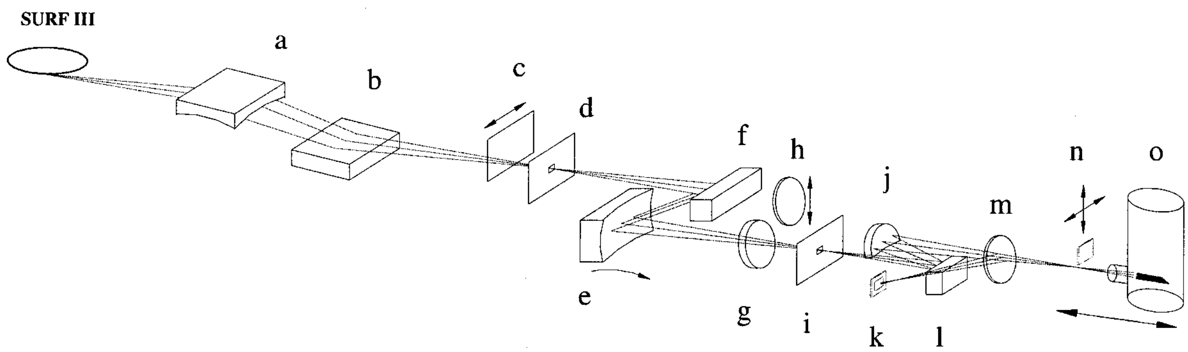

FIG. 1. Schematic diagram of beamline 4 for UV radiometry at SURF III showing grazing incidence preoptics [(a) and (b)], shutter (c), monochromator entrance slit (d), grating (e), plane mirror (f), CaF_2 window (g), quartz filter (h), monochromator exit slit (i), spherical mirror (j), monitor photodiode (k), plane mirror (l), CaF_2 beam splitter (m), test detector (n), and absolute cryogenic radiometer (ACR) (o).

vides more radiation power with improved stability that leads to a larger signal-to-noise ratio and an improvement in the accuracy of radiometric measurements. We have performed detector spectral responsivity calibrations on beamline 4 with a relative standard uncertainty of less than 0.5% over most of the beamline 4 spectral region. This uncertainty is significantly lower than the up to 6% uncertainties obtained at the SCF and the Far UV Calibration Facility. With the new UV scale at beamline 4, a round of intercomparison was conducted among beamline 4, the SCF, and the Far UV Calibration Facility to ensure UV scale consistency. Subsequently, the UV scale at beamline 4 was transferred to the SCF and the Far UV Calibration Facility to reduce their uncertainties.

In this article, we describe the results and the uncertainty analysis of the new scale set by beamline 4 with SURF III radiation and the intercomparison results with the SCF and the Far UV Calibration Facility. We also present the calibration results of a variety of UV detectors with beamline 4.

II. BEAMLINE DESCRIPTION

The schematic diagram of the beamline 4 components is shown in Fig. 1. A more general discussion of the operation and performance of the preoptics and the monochromator system was given elsewhere.[18,19] Briefly, synchrotron radiation from SURF III is collected and imaged onto the entrance slit of a monochromator by two grazing incidence mirrors, one plane and one toroidal. A shutter in front of the monochromator entrance slit can block the beam for background measurements by downstream detectors. The monochromator is a 2 m normal-incidence monochromator with a 600 line/mm grating blazed at 200 nm. A CaF_2 window in front of the exit slit separates the downstream beamline from the storage ring. This reduces the pump-down time for detector calibrations since ultrahigh vacuum ($\sim 10^{-9}$ Torr) is not required for operation in the end station where test detectors are positioned. A movable quartz window acts as an order-sorting filter for wavelength scans longer than 200 nm. Typical resolution of the monochromator for this work was approximately 1.4 nm at 200 nm, which corresponded to a slit opening of 2 mm$\times$2 mm.

With the upgrade of SURF III, the wavelength calibration of the beamline 4 monochromator system was performed using a holmium oxide solution wavelength standard [NIST standard reference material (SRM) 2034].[20] The transmission bands of the wavelength standard were measured and each measured band was identified and the corresponding published wavelength value was used to correct the wavelength scale of beamline 4. The resulting wavelength uncertainty of the 2 m monochromator in the UV region is ± 0.2 nm.

Downstream from the monochromator, two Al/MgF_2 mirrors, one spherical and one flat, image the exit slit of the monochromator onto the test detectors and the ACR with one-to-one magnification. Before the monochromatized beam irradiates the test detectors and ACR, a CaF_2 beam splitter reflects a portion of the beam onto a monitor photodiode. The signal from the monitor photodiode is used to normalize the signal either from the test detector or from the ACR to eliminate the effects on the measurements of the gradual decay of the electron beam in the storage ring and any fluctuations of the beam.

Further downstream, the test detectors and ACR are

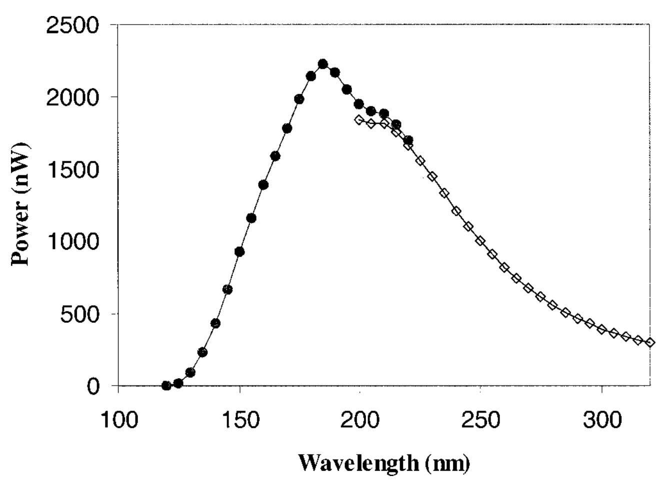

FIG. 2. Measured optical power output from beamline 4 with an exit slit size of 2 mm by 2 mm. For wavelengths longer than 200 nm (open diamonds), a quartz window was used as an order-sorting filter. At shorter wavelengths (closed circles), only the CaF_2 window provided filtering.

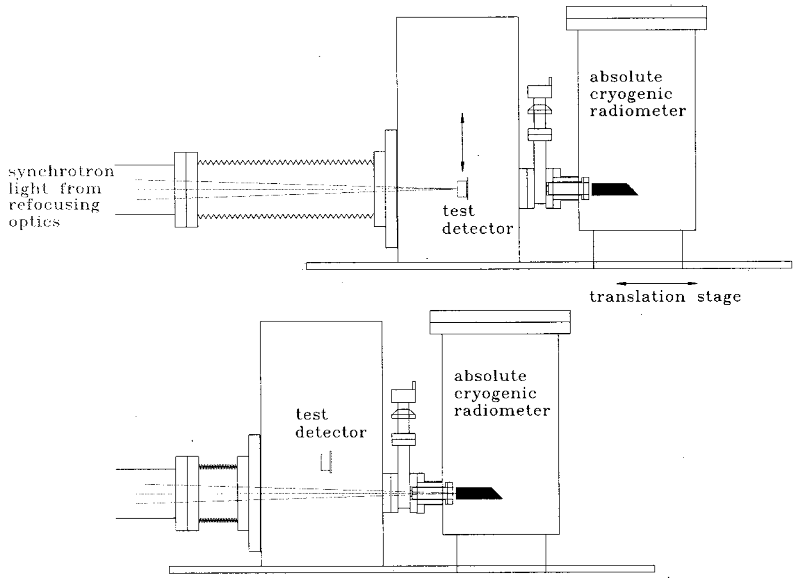

FIG. 3. Experimental configurations for responsivity measurement of the test detector.

placed in line with the monochromatized beam. The test detectors are mounted on an $x-y$ translation stage. The linear motions are used to raster scan the test detectors for spatial response mapping and to move the test detectors in and out of the optical path during calibration. Both the ACR and test detector assembly are mounted on a separate translation stage that moves parallel to the light path so that the ACR and test detectors can be placed at the focal plane sequentially. Movements of all stages are controlled and automated by a computer.

A typical spectral power output of beamline 4 is shown in Fig. 2 with SURF III operated at an energy of 331 MeV and a current of 180 mA. For wavelengths longer than 200 nm, the fused silica filter was inserted into the beam to suppress high-diffraction-order contributions.

III. DETECTOR CALIBRATION METHOD

The measurement of absolute spectral responsivity of test detectors involves two steps, as shown in Fig. 3:

First, the ACR is moved to the focal position and the test detector is moved out of the optical path for absolute power measurement. The data from the ACR is continuously recorded with beamline shutter closed, opened, and closed again. This measurement sequence provides a measurement of the slow drift of the ACR background signal due to the changes in thermal environment. The ACR background drift is approximately linear during the duration of the ACR measurements of typically 2 min at a particular wavelength. An example of the data measured using this sequence is shown in Fig. 4. To determine the ACR background level during the full beam measurement, a line is fitted to the background signal measured with shutter closed. This best-fit line is used as the ACR background level during the full beam measurement with shutter opened.

Second, the test detector is moved to the focal position and the response of the detector is measured using a calibrated electrometer. The responses of the test detector and ACR are both normalized by *in situ* measurements of the

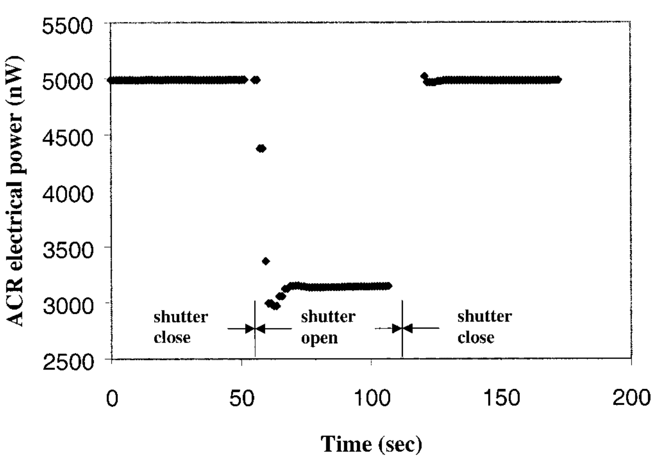

FIG. 4. Time sequence of data acquisition for power measurements by the ACR. The incident optical power is the difference in electrical power supplied to the ACR cavity when the shutter is closed versus when the shutter is open.

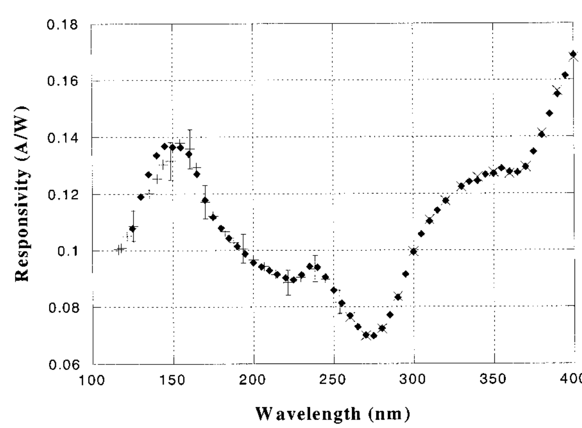

FIG. 5. Spectral responsivity of the Far UV Calibration Facility working standard 94-103 measured by beamline 4 (filled diamonds), the SCF (X), and the Far UV Calibration Facility (+). The representative error bars show the measurement uncertainty of the Far UV Calibration Facility.

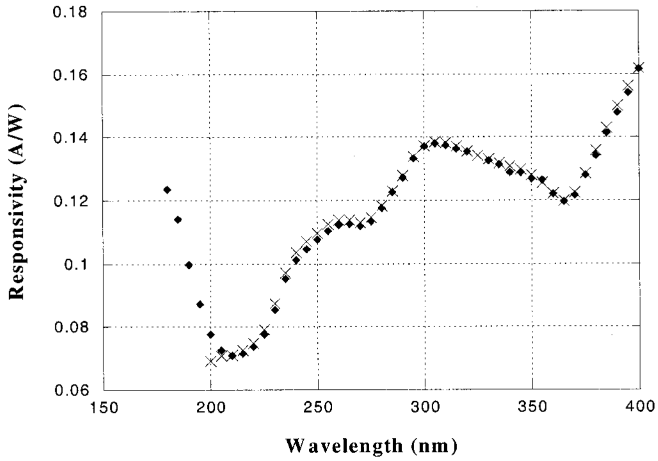

FIG. 6. Spectral power responsivity of the SCF working standard $u503$ measured at beamline 4 (filled diamonds) and from the SCF (X).

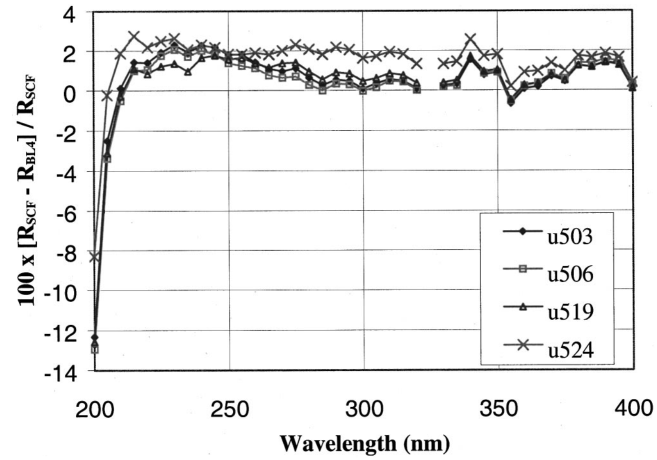

FIG. 7. Difference in spectral responsivities measured by the SCF and beamline 4 for four UV SCF working standards. The difference is calculated by subtracting the responsivity measured with beamline 4 from the responsivity measured with the SCF and then ratio the result with the responsivity measured with the SCF.

monitor diode output to eliminate the effects of beam fluctuations during the measurements.

The spectral power responsivity S_x (ampere/watt) of the test detector can be calculated using the following measurement equation derived similarly to the procedure described in Ref. 21:

$$S_x=\left(\frac{I_x-I_{d,x}}{I_{mx}-I_{d,mx}}\right)\cdot\left(\frac{I_{ms}-I_{d,ms}}{P_{\text{off}}-P_{\text{on}}}\right), \tag{1}$$

where I_x and I_{mx} are the measured current from the test detector and the monitor photodiode; $I_{d,x}$ and $I_{d,mx}$ are the background current from the test detector and monitor photodiode measured with shutter closed; P_{on} is the electrical power (SI unit W) applied to the ACR cavity heater with the incident light (shutter opened) and P_{off} is the interpolated ACR thermal background derived from the measurements performed with shutter closed; and I_{ms} and $I_{d,ms}$ are the measured current and its background from the monitor photodiode simultaneously recorded with the ACR measurement.

IV. INTERCOMPARISON WITH CURRENT NIST SCALE

An intercomparison was performed among beamline 4, the Far UV Calibration Facility, and the SCF by measuring the spectral responsivity of an AXUV-100G photodiode from International Radiation Detectors (IRD).[22] The AXUV-100G photodiode is used as a working standard at the Far UV Calibration Facility and it was also calibrated at the SCF from 260 to 400 nm. The measurement results from these three facilities are shown in Fig. 5. The agreement between the SCF and beamline 4 is better than 1%. For the Far UV Calibration Facility, the agreement with beamline 4 is better than 2% for wavelengths longer than 160 nm. Such agreement is well within the 5% overall uncertainty for the Far UV Calibration Facility.

In addition, we measured the absolute spectral responsivities of four United Detector Technologies UV-100 photodiodes.[22] These photodiodes are used at the SCF as UV working standards with their scale derived from HACR and a pyroelectric detector.[2] The spectral responsivity of working standard $u503$ measured at beamline 4 is shown in Fig. 6 along with the spectral responsivity derived from the SCF scale. The differences in spectral responsivities of these working standards between the SCF scale and those measured at beamline 4 are shown in Fig. 7. In general, the measurements between beamline 4 and the SCF agree with each other within about 2% except at wavelengths near 200 nm where the SCF measurement is significantly lower in responsivity than that of the beamline 4 measurement. This is in accordance with the increase in uncertainty near 200 nm

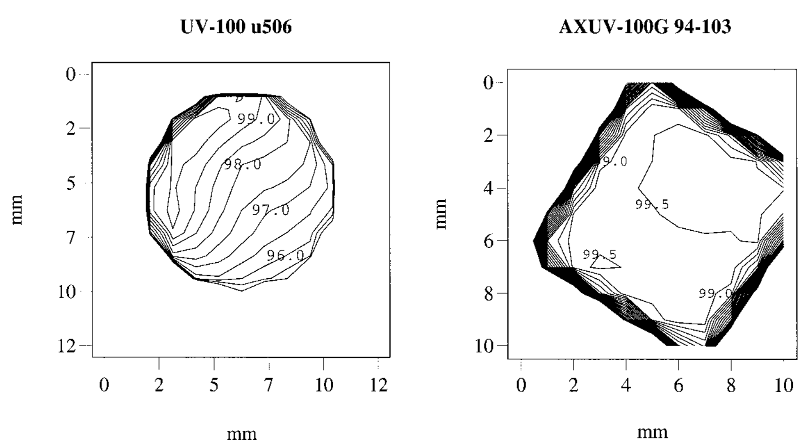

FIG. 8. Spatial response uniformity with 0.5% contours at 240 nm for the SCF working standard $u506$(UV-100) and the Far UV Calibration Facility working standard 94-103 (AXUV-100G) measured at beamline 4.

TABLE I. Components of the combined relative standard uncertainty ($k=1$) of spectral responsivity at beamline 4.

Source of uncertainty	Component of uncertainty (%)
ACR power measurement with 1 μW optical power	0.30
monitor diode current measurement	0.10
test diode current measurement	0.10
wavelength scale and spectral bandwidth	0.30
out-of-band stray light	0.10
test diode positioning	0.20
Relative combined standard uncertainty	0.50

for the SCF. It should be noted that the somewhat different result for working standard $u524$ in Fig. 7 is believed to be caused by the variation of the spatial uniformity of that photodiode.

For the two types of working standards used at the SCF and the Far UV Calibration Facility, we found that the AXUV-100G photodiode has better reproducibility than the UV-100 photodiode and therefore lower overall measurement uncertainty. This is attributed to the better spatial response uniformity of the AXUV-100G photodiode as shown in Fig. 8. The data of Fig. 8 was measured at beamline 4 by rastering the test detectors across the light beam at a wavelength of 240 nm. We will calibrate more detectors with beamline 4 to help the SCF in establishing new UV working standards.

After this round of intercomparison, the UV scale at beamline 4 is transferred to both the SCF and the Far UV Calibration Facility. For the SCF, the uncertainty of the new scale based on the measurements of beamline 4 is 1% near 200 nm whereas the original SCF uncertainty is up to 6%. It also allowed the SCF to extend its wavelength limit to 190 nm to include the excimer laser's 193 nm wavelength that is particularly important for semiconductor photolithography. For the Far UV Calibration Facility, the new scale reduces the measurement uncertainty for wavelengths longer than 130 nm from the original value of 5% down to 1%.

V. MEASUREMENT UNCERTAINTY ANALYSIS

Most of the uncertainty budget for the absolute responsivity measurement on beamline 4 is caused by the components of the measurement equation expressed in Eq. (1). These uncertainties, the power measurements by the ACR, monitor diode, and test detectors, are evaluated by the statistical analysis of repeated observations (type A uncertainties).[23] There are also components (type B) that depend on the systematic effects of beamline instruments, such as the stray light, alignment, detector positioning, wavelength scale, and ACR calibration. The study of stray light, alignment, and ACR calibration have been discussed previously.[12] It was shown that each of these components has a relative standard uncertainty of approximately 0.1%. The uncertainty in detector responsivity caused by wavelength accuracy and detector positioning are dependent on the spectral responsivity and spatial response uniformity of the test detector. For example, the accuracy of the wavelength scale becomes very important when there is a large slope in the spectral responsivity of the test detector. The uncertainties caused by wavelength scale and detector positioning can only be estimated based on measured results of the test detector.

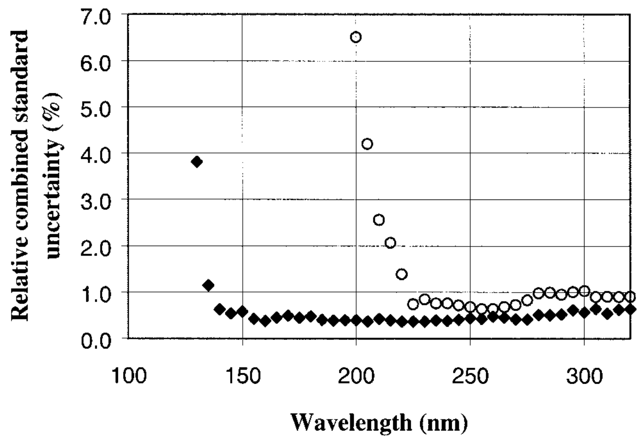

FIG. 9. The relative combined standard uncertainty of an AXUV-100G photodiode responsivity calibration by beamline 4 (filled diamonds) and the SCF (open circles).

To assess the type A uncertainties from a power measurement by the ACR, test detector, and monitor diode, we sampled 50 data points for each wavelength to derive the standard deviation of each measurement. We found that, typically, the ACR had a measurement uncertainty of 0.3% at about 1 μW of incidence radiation power whereas the measurement uncertainty for the monitor diode and test detector was less than 0.1%. Listed in Table I are typical components of the total measurement uncertainty for beamline 4.

A more detailed calculation of the overall measurement uncertainty at each wavelength is obtained by combining all the components of uncertainty based on the measurement statistics at every wavelength. Shown in Fig. 9 is the relative combined uncertainty for the calibration of the AXUV-100G photodiode at beamline 4 compared with the SCF. As shown in Fig. 9, beamline 4 has lower uncertainty below 320 nm and it greatly reduces the SCF uncertainty near 200 nm. In

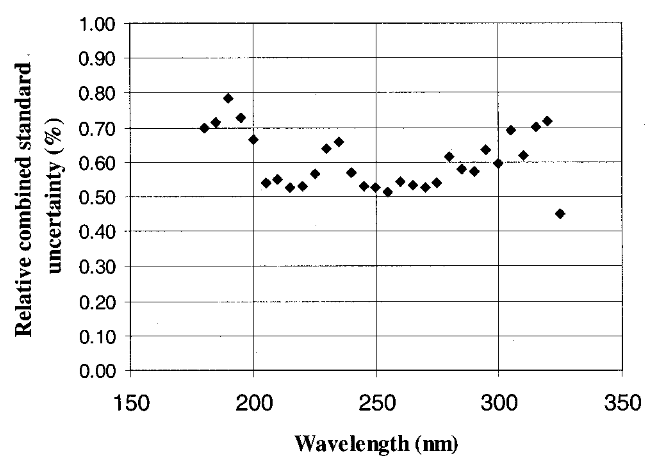

FIG. 10. The relative combined standard uncertainty of a UV-100 photodiode calibration by beamline 4.

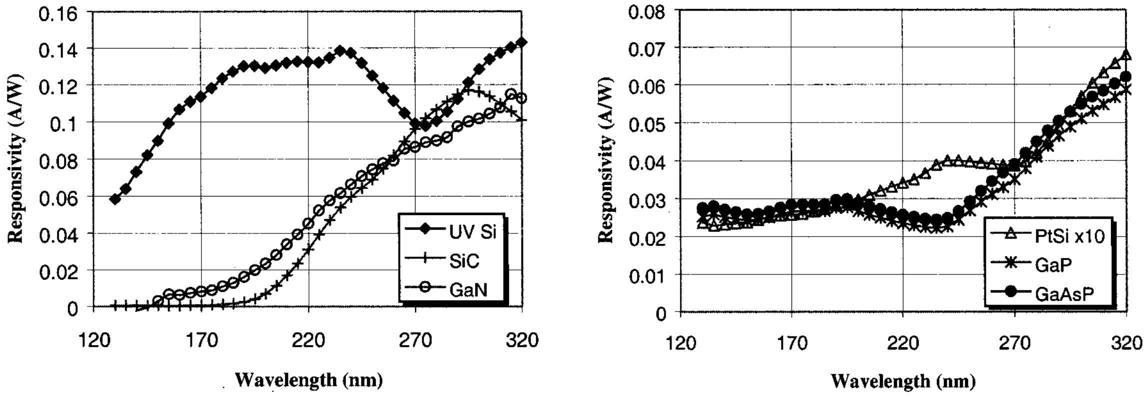

FIG. 11. Measured deep UV spectral responsivities of UV Si, SiC, GaN, PtSi, GaP, and GaAsP photodiodes. The spectral responsivity of the PtSi photodiode shown is shown as 10 times the actual value.

addition, the beamline 4 scale extended the wavelength range to 140 nm with an uncertainty of less than 0.5% whereas the Far UV Calibration Facility is 5% for this wavelength range.

Shown in Fig. 10 is the overall relative combined uncertainty for the SCF UV working standard photodiodes calibrated at beamline 4. This new responsivity scale was subsequently placed on the working standard detectors of the SCF to reduce the uncertainty in the responsivity of the working standard detectors and resulted in the reduction in uncertainty in calibration of test detectors.

VI. CALIBRATION OF UV DETECTORS

The development of UV detectors has drawn intensive research recently due to the dramatic increase in a variety of UV applications ranging from the semiconductor industry to environmental monitoring. The goal of the research is to develop UV detectors with features such as radiation hardness, solar blindness, uniform spatial responsivity, and small size. For the calibration facilities at NIST, the new UV detectors can potentially be used as UV transfer standard detectors to reduce the uncertainty from detector spatial uniformity and radiation damage.

We have measured the spectral responsivity of a variety of currently available UV detectors using beamline 4. Among them: UV Si *p*-on-*n* (Hamamatsu 5227), SiC, GaN (APA), PtSi (IRD SXUV 100), GaP (Hamamatsu), and GaAsP (Hamamatsu) photodiodes.[22] The results are shown in Fig. 11. The typical measurement uncertainty is less than 0.5% for 157 and 193 nm excimer laser lines that are of the most interest to the UV photolithography industry. This round of calibration is part of our effort to find stable detectors that are candidates for better UV working standards and UV irradiance meters. We are currently in the process of studying the radiation damage effect to a variety of UV detectors by a 157 nm excimer laser using beamline 4.

ACKNOWLEDGMENT

The authors would like to thank the SURF staff members for their help in operating SURF III for this work.

[1] T. C. Larason, S. S. Bruce, and C. L. Cromer, J. Res. Natl. Inst. Stand. Technol. **101**, 133 (1996).
[2] T. C. Larason, S. S. Bruce, and A. C. Parr, Natl. Inst. Stand. Technol. Spec. Publ. **250–41**, (1997).
[3] M. L. Furst, R. M. Graves, L. R. Canfield, and R. E. Vest, Rev. Sci. Instrum. **66**, 2257 (1995).
[4] L. R. Canfield, Appl. Opt. **26**, 3831 (1987).
[5] L. R. Canfield and N. Swanson, J. Res. Natl. Bur. Stand. **92**, 97 (1987).
[6] R. E. Vest, L. R. Canfield, M. L. Furst, R. P. Madden, and N. Swanson, Nucl. Instrum. Methods Phys. Res. A **347**, 291 (1994).
[7] L. R. Canfield, R. E. Vest, R. Korde, H. Schmidtke, and R. Desor, Metrologia **35**, 329 (1998).
[8] T. R. Gentile, J. M. Houston, and C. L. Cromer, Appl. Opt. **35**, 4392 (1996).
[9] T. R. Gentile, J. M. Houston, J. E. Hardis, C. L. Cromer, and A. C. Parr, Appl. Opt. **35**, 1056 (1996).
[10] C. L. Cromer, T. B. Lucatorto, T. R. O'Brian, and M. Walhout, Solid State Technol. **April**, 75 (1996).
[11] P. S. Shaw, K. R. Lykke, R. Gupta, T. R. O'Brian, U. Arp, H. H. White, T. B. Lucatorto, J. L. Dehmer, and A. C. Parr, Metrologia **35**, 301 (1998).
[12] P. S. Shaw, K. R. Lykke, R. Gupta, T. R. O'Brian, U. Arp, H. H. White, T. B. Lucatorto, J. L. Dehmer, and A. C. Parr, Appl. Opt. **38**, 18 (1999).
[13] B. Wende, Metrologia **32**, 419 (1996).
[14] H. Rabus, F. Scholze, R. Thornagel, and G. Ulm, Nucl. Instrum. Methods Phys. Res. A **377**, 209 (1996).
[15] H. Rabus, V. Persch, and G. Ulm, Appl. Opt. **36**, 5421 (1997).
[16] M. L. Furst, R. M. Graves, A. Hamilton, L. R. Hughey, R. P. Madden, R. E. Vest, W. S. Trzeciak, R. A. Bosch, L. Greenler, P. Robl, and D. Wahl, Proceedings of the 1999 Particle Accelerator Conference, 29 March–2 April 1999, New York, p. 2388.
[17] U. Arp, R. Friedman, M. L. Furst, S. Makar, and P. S. Shaw, Metrologia **37**, 357 (2000).
[18] D. L. Ederer, B. E. Cole, and J. B. West, Nucl. Instrum. Methods **172**, 185 (1980).
[19] L. R. Hughey, Nucl. Instrum. Methods Phys. Res. A **347**, 294 (1994).
[20] V. R. Weidner, R. Mavrodineanu, K. D. Mielenz, R. A. Velapoldi, K. L. Eckerle, and B. Adams, NBS Spec. Publ. **260–102**, (1986).
[21] P. S. Shaw, T. C. Larason, R. Gupta, S. W. Brown, and K. R. Lykke, J. Res. Natl. Inst. Stand. Technol. **105**, 689 (2000).
[22] Certain commercial equipment, instruments, or materials are identified in this article to foster understanding. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.
[23] B. N. Taylor and C. E. Kuyatt, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, NIST Tech. Note 1297, 2nd ed. (National Institute of Standards and Technology, Gaithersburg, MD, 1994).