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Silicon photodiode with selective Zr/Si coating for extreme ultraviolet spectral range

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Abstract. **The procedure of manufacturing silicon photodiodes with an integrated Zr/Si filter for extreme ultraviolet (EUV) spectral range is developed. A setup for measuring the sensitivity profile of detectors with spatial resolution better than 100 μm is fabricated. The optical properties of silicon photodiodes in the EUV and visible spectral ranges are investigated. Some characteristics of SPD-100UV diodes with Zr/Si coating and without it, as well as of AXUV-100 diodes, are compared. In all types of detectors a narrow region beyond the operating aperture is found to be sensitive to the visible light.**

Keywords: *EUV radiation, silicon photodiode, detector, EUV filter.*

1. Introduction

The progress of projection lithography in the extreme ultraviolet (EUV) range at the operating wavelength 13.5 nm [1], diagnostics of high-temperature plasma, controlled thermonuclear fusion [2], X-ray microscopy [3], and astronomy [4] gives rise to increased requirements to detectors of EUV radiation.

The AXUV silicon photodiodes (IRD Inc., USA) based on n–p structures are well-known and have been used for a long time to register the soft X-ray and EUV radiation [5]. The opposite in structure SPD-100UV p–n photodiodes (Ioffe Physical Technical Institute, St. Petersburg) were developed for using in the same regions of the spectrum [6, 7]. In Russian-language literature the SPD photodiodes are traditionally referred using the abbreviation FDUK. In Ref. [8] the experimental data obtained at the VEPP-4 synchrotron are reported about the sensitivity of these detectors being uniform with respect to aperture and resistance to radiation damage in the soft X-ray and EUV ranges. The radiation resistance of the SRD-100UV photodiodes in the EUV range appeared to be higher than that of the AXUV photodiodes, which makes the SPD-100UV detectors promising for registration of high EUV radiation doses.

In some cases of exploiting silicon photodiodes their high sensitivity to near-IR, UV, and visible radiations appears to be a serious drawback. In such cases the detectors should be equipped with special external thin-film filters. In the X-ray spectral region by choosing appropriate external filters one can provide high selectivity of a photodiode with suppression of sensitivity to long-wavelength radiation by several orders of magnitude. However, manufacturing of such filters for the EUV range is difficult, since in order to keep the quantum efficiency of the device high the thickness of the films should be as small as tens or hundreds of nanometres [9]. Low mechanical strength of such filters gives rise to problems in the course of their exploitation, particularly, during the vacuum pumping of the devices, as well as in the case of acoustical and local thermal perturbations. A convenient way to solve this problem is to deposit thin filtering films directly onto the sensitive surface of the photodiode.

In the present paper we study the properties of SPD-100UVZr/Si photodiodes with multilayer Zr/Si filters, deposited directly onto the active area, optimised for registration of radiation in the vicinity of the wavelength 13.5 nm. The films were deposited using the method of magnetron sputtering at the Institute for Physics of Microstructures, Russian Academy of Sciences (Nizhnii Novgorod) onto the wafers with already fabricated diode structures. Before film deposition, a photoresist was deposited onto the silicon wafer, and the following photolithography opened 10.1 × 10.1 mm windows in the photoresist. This operation provided deposition of filter layers onto the entire active area of the detector. Thus, the filter layers coated only the active area of the photodiode, margining the current-collecting electrode only by 0.05 mm.

We also describe the setup developed to study the quantum efficiency of detectors in the solt X-ray and EUV ranges over the entire sensitivity area of the photodiode with spatial resolution better than 100 μm. The results of comparing the characteristics of SPD-100UVZr/Si and AXUV-100 silicon photodiodes are presented.

2. Description of a photodiode with absorption filter

Figure 1 presents the structures of the SPD-100UVZr/Si silicon photodiode with a Zr/Si filter, deposited onto its surface, and those of the AXUV-100 photodiode. The dimensions of the active area of both photodiodes were 10 × 10 mm. In order to select the spectral region in the vicinity of the wavelength 13.5 nm by suppressing the long-wavelength (IR, visible and UV) and short-wavelength parts of spectrum, a filter coating

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based on a pair of Zr and Si materials was deposited onto the surface of the photodiode. The spectral region under study lies near the $L_{II,III}$ edge of absorption of silicon (12.3 nm), while for the radiation with the wavelength 13.5 nm the silicon is transparent. To enhance the blocking ability of the filter in the long-wavelength region of the spectrum, it is necessary to include in the structure a metal with high transparency at the operating wavelength. One of the most transparent metals near 13.5 nm is zirconium. That is why we have chosen the multilayer Zr/Si structure as a protective coating. Since the studied objects possess strongly different emission characteristics, while the detector is supposed to be used for in a wide scope of applications (i.e., to be universal), the total thickness of the filter and the fraction of each material in the multilayer structure were taken to be the same as in free-standing Zr/Si filters, which have shown themselves to advantage as parts of different devices used in the studies of the Sun, high-power laser plasma, and gas discharge sources of X-ray radiation [9, 10]. The filter consisted of $N = 47$ periods (bilayers), each containing a layer of zirconium with the thickness $d_{Zr} = 4.15$ nm and a layer of silicon with the thickness $d_{Si} = 1.96$ nm. In Fig. 2 the solid curve shows the calculated dependence of the filter transmission coefficient in the soft X-ray and EUV ranges. It is seen that the transmission at the wavelength 13.5 nm attains 44%. In accordance with the calculations, the suppression of UV, visible, and IR radiation up to the long-wavelength boundary of the detector sensitivity (~1 μm) exceeds 10^{10} (100 dB).

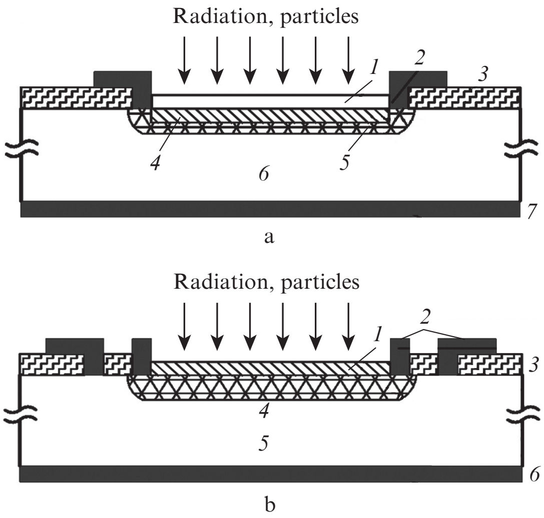

Figure 1. Structure of SPD-100UVZr/Si (a) and AXUV-100 (b) photodiodes: (a) (*1*) multilayer Zr/Si filter; (*2*) metallic coating, (*3*) protective thermal oxide; (*4*) passivating layer of silicon–boron compound; (*5*) p-type doped region; (*6*) n-type substrate; (*7*) metallic coating; (b) (*1*) passivating layer of silicon dioxide; (*2*) metallic coating; (*3*) protective thermal oxide; (*4*) n-type doped region; (*5*) p-type substrate; (*6*) metallic coating.

The films were deposited using the method of magnetron sputtering, the equipment and technology being described elsewhere [11]. The uniformity of the coating thickness over the detector area was determined by measuring the position of Bragg reflection peaks in the X'Pert Pro MRD (PANalytical) X-ray diffractometer equipped with a six-axis goniometer, and was no worse than 0.2%.

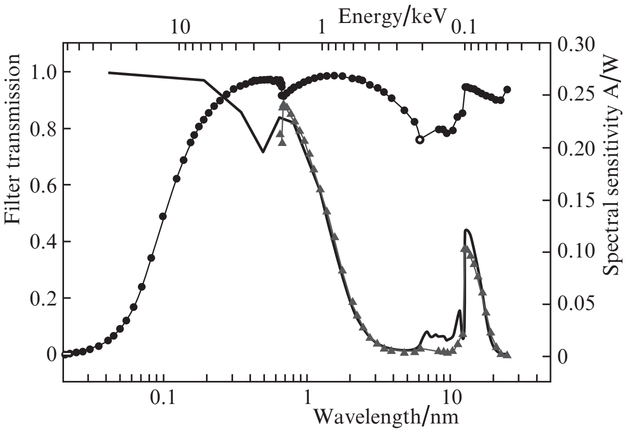

Figure 2. Spectral dependences of the sensitivity of SPD-100UVZr/Si and SPD-100UV detectors with a filter (△) and without a filter (○), measured at the PTB Metrology Centre (Berlin, Germany) and the theoretical transmission spectrum of Zr/Si filter with the number of periods $N = 47$, each of which contains the layers of Zr and Si with the thickness $d_{Zr} = 4.15$ nm and $d_{Si} = 1.96$ nm (solid curve).

3. Results of the measurements

3.1. Measuring the sensitivity of the SPD-100UVZr/Si photodiode in the EUV region

The study of sensitivity of the detectors in the EUV range was implemented using the specially designed multifunctional laboratory reflectometer, the schematic diagram of which is presented in Fig. 3. The optical scheme is based on the two-

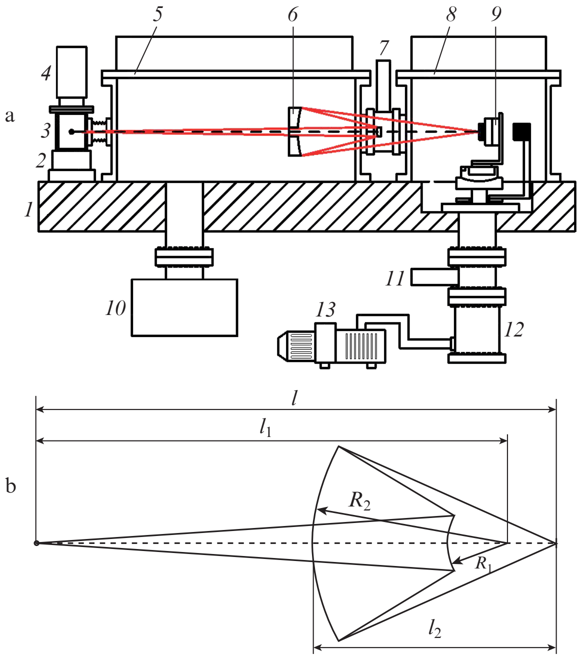

Figure 3. Schematic diagram of the reflectometer (a) and optical scheme of the objective-monochromator (b):
(*1*) base plate; (*2*) alignment element of the X-ray tube; (*3*) X-ray tube; (*4*) ion source; (*5*) monochromator chamber; (*6*) monochromator; (*7*, *11*) vacuum valves; (*8*) sample chamber; (*9*) goniometer; (*10*, *12*, *13*) magnetic discharge, turbo-molecular, and fore-evacuation pumps, respectively.

mirror Schwarzschild objective-monochromator, formed by two spherical multilayer mirrors, optimised to the relevant wavelength. The parameters of the objective-monochromator are listed below.

The total length l of the system (from the source to the image)/mm	1143.17
The distance l_1 from the source to the centre of curvature of the mirrors/mm	1036.16
The distance l_2 from the second (concave) mirror to the image/mm	534
Curvature radius R_1 of the first (convex) mirror/mm	133.38
Curvature radius R_2 of the second (concave) mirror/mm	427.0
Image size (calculated without diffraction) for the 'zero' source/nm	20
Image size (calculated) for the size of the source 0.5 mm/mm	0.05
System demagnification	9.86
Diameter of the concave mirror/mm	115
Diameter of the aperture in the concave mirror/mm	40
Diameter of the convex mirror/mm	20
Operating wavelength of the monochromator/nm	13.5
Transmission bandwidth/nm	0.41
Transmission coefficient of the objective	0.36
Transmission coefficient of the filter at the X-ray tube output	0.41

The main advantages of multilayer interference normal-incidence mirrors as compared with traditional diffraction gratings are large geometric aperture ratio and high reflection coefficients of the mirrors, as well as the possibility to combine in one device the functions of a monochromator and a shaper of geometric characteristics of the probe beam. Particularly, in [12] the use of such mirrors allowed one to increase the probing signal intensity by 800 times as compared with the case of a traditional grating monochromator [13]. The formation of the transmission spectral band of the device is provided by optimising the fraction of strongly absorbing substance in a period of the multilayer structure [14], depositing nonperiodic structures (which provides the maximal intensity of the probing beam) [15], or shifting the resonance wavelengths of the mirrors (which provides the minimal spectral bandwidth of the probing beam). If testing at several wavelengths is required, then the multilayer reflecting structures, optimised for appropriate wavelengths, may be deposited onto different sectors of the substrate. In the process of operation the idle sectors are simply shielded using a metallic shutter. In the present work we used multilayer Mo/Si mirrors, optimised for the wavelength 13.5 nm. The reflection coefficients of the mirrors amounted to 60%, the total transmission spectral bandwidth being 0.41 nm. To reduce the scattered light in the device, a free-standing Zr/Si filter was installed at the input of the monochromator chamber at the distance ~90 mm from the objective. The parameters of the filter layers were similar to those used in deposition of the filters onto the photodiodes. The operating aperture of the filter was 30 mm.

The role of radiation source was played by an X-ray tube with a silicon target having the L-line fluorescence intensity maximum at the wavelength 13.5 nm [16]. The energy of electrons amounted to 7 keV, the beam current was equal to 1.5 mA. The registered detector current was 0.4 nA. With the quantum efficiency of the detector and the transmission of filters taken into account, this corresponded to the EUV probing beam radiation power of 8.2 nW (4..8 × 10^8 photons s^{-1}) (see Fig. 2). The diameter of the electron beam (the source size) was equal to 0.5 mm. Taking the demagnification into account, one could expect the diameter of the spot on the sample under study to be ~50 μm (at half-maximum level). Figure 4 presents the X-ray radiation beam profile in the sample plane, obtained by gradual blocking of the beam with a 'knife' placed in the plane, perpendicular to the optical axis (Foucault method). It is seen that the true beam size (at half-maximum level) amounts to nearly 54 μm, which is close to the theoretical value.

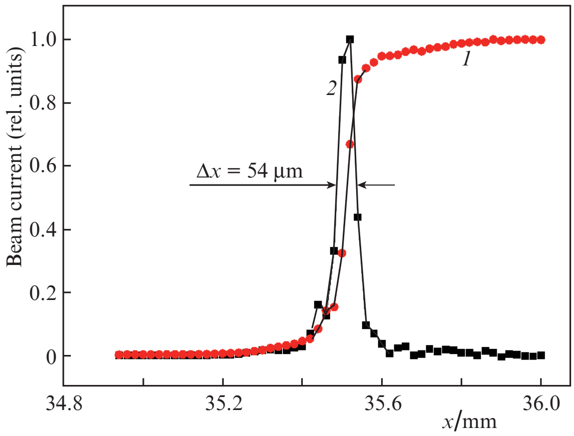

Figure 4. Dependence of the detector current on the position x of the 'knife' (*1*) and the EUV radiation intensity distribution in the beam (*2*).

In the present work we studied the relative sensitivity of three SPD-100UVZr/Si detectors with multilayer Zr/Si ~300-nm-thick absorption filters, deposited onto the surface for selecting the operating spectral region near 13.5 nm and suppressing the long-wavelength background. The etalon, with respect to which the detector sensitivity was evaluated, was the AXUV-100 photodiode without coating, calibrated at the PTB Metrology Centre (Berlin, Germany).

The studied detectors were fixed at the goniometer table and then scanned in the plane, perpendicular to the beam axis, with the step of 0.5 mm. Analogous procedure was performed with the AXUV-100 diode. Figure 5 shows typical profiles of sensitivity for the detectors SPD-100UVZr/Si and AXUV-100, whose maximal sensitivity was taken for unity, measured along the lines, parallel to the detector faces.

The measured nonuniformity of the sensitivity (square root of the mean-square deviation) in the 'plateau' region amounted to ~1%. When determining the detector sensitivity relative to that of AXUV-100, the experimental data were averaged over a large series of measurements in order to minimise the statistical error.

The relative sensitivity of detectors, S, was calculated using the formula

$$S = \frac{I_{\text{SPD}} - I^{\text{d}}_{\text{SPD}}}{I_{\text{AXUY}} - I^{\text{d}}_{\text{AXUY}}}, \qquad (1)$$

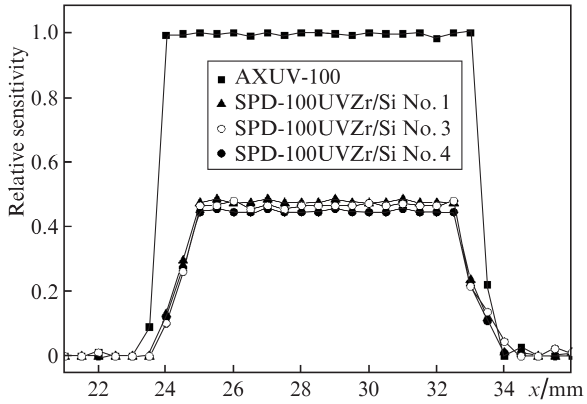

Figure 5. Profiles of sensitivity of SPD-100UVZr/Si detectors relative to the sensitivity of the AXUV-100 detector depending on the coordinate x of the detector surface point, on which the radiation is incident, measured along the lines, parallel to the detector faces.

where I_{SPD} and $I^{\mathrm{d}}_{\mathrm{SPD}}$ is the current and the dark current of the tested detector; I_{AXUY} and $I^{\mathrm{d}}_{\mathrm{AXUY}}$ are the current and the dark current of the ‘etalon’ detector. The relative sensitivities for three samples of SPD-100UVZr/Si photodiodes (in the centre of the sensitive area) at the wavelength 13.5 nm appeared to be 47.7 ± 0.1%, 46.7 ± 0.1%, and 44.8 ± 0.1%. The calculated transmission of Zr/Si filters deposited onto the detector surface at the wavelength 13.5 nm amounted to ~44%.

In contrast to AXUV-100, in SPD-100UVZr/Si we found transition zones near the edges of sensitive area, where the sensitivity of the detector to EUV radiation smoothly changes from zero to maximum. The extent of these zones amounts to ~1 mm, thus limiting the aperture of uniform sensitivity of the detector to the area 8 × 8 mm. The cause of the transition zones being so wide requires further study.

Besides the evaluation of the SPD-100UVZr/Si sensitivity relative to that of AXUV-100, at the PTB Metrology Centre the measurements of absolute sensitivity of SPD-100UVZr/Si and SPD-100UV photodiodes were carried out [17]. One SPD-100UVZr/Si detector was calibrated in the range 50–1900 eV, three SPD-100UV detectors were calibrated in the ranges 50–1900, 1750–10000, and 8000–60000 eV. Based on these measurements, the typical spectral dependences of the SPD-100UV and SPD-100UVZr/Si detectors were plotted (see Fig. 2).

3.2. Evaluation of suppressing the detector sensitivity to visible light

The measurement of diode sensitivity in the visible range of spectrum was performed at the wavelength 633 nm using a He–Ne laser. The signal from the detector was applied to the amplifier based on the OPA827AP operational amplifier, the transmission coefficient of which was set using an external resistor having the rated resistance up to 1 GΩ. Previously, using calibrated optical filters, the linearity of the dependence of the output signal on the rated resistance and the intensity of laser radiation, incident on the detector, was checked.

Figure 6 represents the sensitivity profiles of the SPD-100UVZr/Si detectors for the radiation at the wavelength 633 nm relative to the sensitivity of the analogous SPD-100UV diode without coating. The laser beam half-maximum diameter was 0.6 mm, the scanning was performed with the step 0.7 mm. Since in the course of studies the sensitivity to visible light beyond the operating aperture (10 × 10 mm) was found both in SPD-100UVZr/Si and in AXUV-100 detectors (see below), for adequate evaluation of the Zr/Si filter transmission, a metal screen with a 10 × 10 mm window in it was placed in front of the studied detector. In the central part of the diode the mean relative sensitivity in the visible range amounted to $\sim 2 \times 10^{-6}$, and considerable nonuniformity over the detector aperture was observed. The observed nonuniformity, as well as the unexpectedly low visible light suppression for a 300-nm-thick film, are due to the presence of microscopic pin-holes in the filtering coating. In Ref. [18] it was experimentally shown that for analogous freely suspended films of Zr/Si 210 nm thick the transmission of visible light amounted to 8×10^{-7}, the films being free of reach-through defects.

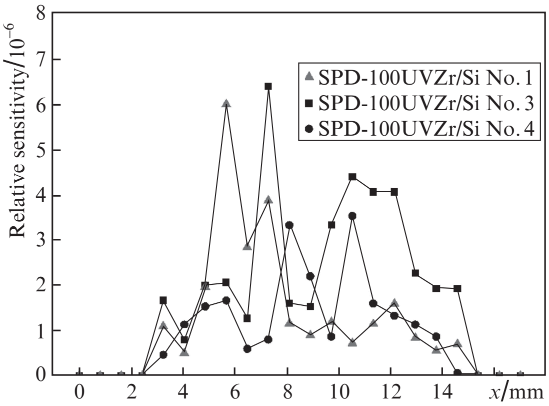

Figure 6. Profiles of sensitivity of the SPD-100UVZr/Si detectors at the wavelength 633 nm relative to the sensitivity of the analogous detector SPD-100UV.

In our case before the deposition of the films onto the wafer with already fabricated photodiode structures the wafer was treated with a flow of dry air in order to clean it from dust particles that get onto the surface in the process of packing, transporting and opening the container. The choice of the ‘soft’ method of cleaning the surface is determined by the small thickness of the passivating coating of the photodiode active area, which amounts to ~10 nm. Mechanical and chemical cleaning of the surface of the passivating coating may lead to its damage and, thus, to irreversible increase in the photodiode dark current, that is why they were not used in the present case. Possibly, the procedure of dry air cleaning of the surface is not sufficient to remove all surface contaminations. Hence, if the conditions for defectless deposition of coatings could be provided, one can expect essential improvement of the filter blocking properties. Nevertheless, the integral coefficient of suppression of sensibility to visible light $\sim 5 \times 10^{5}$ obtained for the SPD-100UVZr/Si diode is not worse than that for the known AXUV-100 detectors with similar Zr/Si filter and is quite sufficient for most applications [5].

It should be noted that silicon detectors may exist with the filter on the surface and without the narrow region, sensible to visible light. This is due to the fact that the charge carriers are generated by the radiation, which is incident onto the area, adjacent to the outer boundary of p–n junction (in the case of AXUV-100) and to the outer boundary of charge-collecting electrode (in the case of SPD-100UVZr/Si). Figure 7 presents the result of scanning the surface of AXUV-100 and SPD-100UVZr/Si with a laser beam at the wavelength 633 nm. The diameter of the beam was ~50 μm, the scanning was performed with the step 100 μm, including the periphery of the active area. The area sensitive to visible light is ~1% of the total photodiode surface, so that without using special apertures the coefficient of suppression of the detector sensitivity to visible light is limited by the value of 10^{-2}. This effect shows the necessity of high-quality collimators and apertures when using silicon detectors with filters in the EUV spectral region.

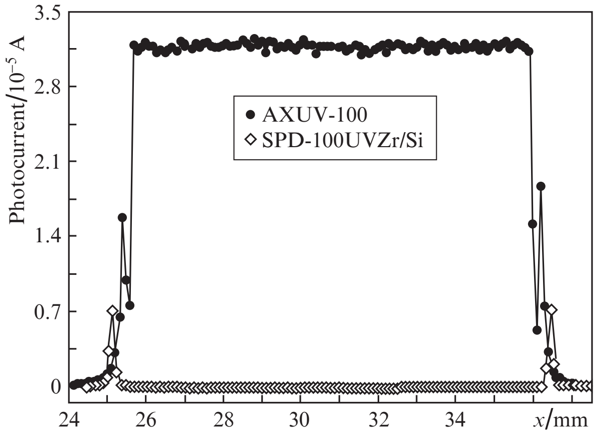

Figure 7. Profiles of photocurrent for SPD-100UVZr/Si and AXUV-100 detectors obtained by scanning their surfaces with a beam of radiation with the diameter 50 mm at the wavelength 633 nm.

4. Discussion of the results

The main result of the work is the developed technology of producing narrow-band silicon photodiodes for detecting soft X-ray and EUV radiation, as well as the determination of spectral properties of these photodiodes. In principal technical characteristics, such as quantum efficiency, the value of dark current, the level of visible light suppression, resistance to radiation damage, these p–n detectors are not inferior to the analogous devices based on n–p structures. Using the developed technology with other coatings, one can optimise the band of detector sensitivity for the solution of particular problems. Finally, an alternative version of a EUV radiation detector has appeared, possessing long-wave background suppression and higher resistance to EUV radiation damage [8].

Further improvement in detectors of the considered type implies, first of all, the search for the ways to reduce the width of the area having lower sensitivity to short-wavelength radiation near the edges of the sensitive zone. Besides, by improving the uniformity of the filtering films, one can most probably achieve additional suppression of detector sensitivity to visible radiation. However, even at the present stage the achieved level of the visible light transmission (2×10^{-6}) is lower than that of AXUV-100 with the analogous Zr/Si coating (10^{-5}) [5]. The suppression by more than five orders of magnitude is achieved for the sensitivity to red light ($\lambda = 633$ nm) in the central part of the sensitive area of photodiodes. It is shown that, with definite aperture limitations, these detectors can be successfully used in research and technological applications, requiring high-selective sensitivity of photodetectors in the EUV region.

An important result of the work is the creation of a wide-aperture laboratory test bench for measuring the detector sensitivity profiles with the spatial resolution better than 100 μm. In the present work the source of X-ray radiation was an X-ray tube with silicon anode having the power of electron beam at the level of 10 W and the efficiency of conversion of electron energy into the EUV radiation at the level of 3×10^{-6} within the monochromator spectral bandwidth and within the solid angle 2π. In the case of equipping the reflectometer with a laser-plasma source (LPS) it is possible to increase the power of the probing EUV beam essentially, by several orders of magnitude. Since the spatial size of LPS radiation is tens of micrometres, the Schwarzschild objective can be used in the inverse geometry and provide magnification, which will automatically increase the EUV beam power by two orders of magnitude. The estimates taking into account the data on the spectral density of radiation power [19] and the efficiency of mirror reflection [11] for the commercially available Nd : YAG laser with the pulse energy of 500 mJ and the repetition rate 10 Hz show that within the spectral bandwidth of the monochromator in the vicinity of the wavelength 13.5 nm it is possible to obtain the photon flux $\sim 10^{13}$ photon s^{-1} on the sample. For the size of probing beam 300 μm at the studied sample and the characteristic absorption length for silicon ~1 μm the absorbed doze will amount to 10^8 rad s^{-1}. For example, to provide the doze of 6×10^{10} rad, at which the degradation of sensitivity of semiconductor detectors was observed [8], in our case the necessary exposure time should be about 10 min. Thus, the developed device, equipped with a laser plasma source, will allow radiation resistance studies in a large class of materials, including semiconductor detectors. At present the development of such source in in progress.

As a final remark, it is necessary to mention one more important result, which, according to the authors' data, was not quantitatively demonstrated earlier, namely, the presence of a narrow, (less than 100 μm) area beyond the detector operating aperture, sensitive to visible light and found both in SPD-100UVZr/SI and in AXUV-100. This effect should be taken into account in designing the systems of registration of EUV radiation on the base of silicon detectors with integrated filters in the active region.

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