Off-axis reflection zone plate for quantitative soft x-ray source characterization

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A compact system for high-resolution spectroscopy and quantitative photon flux and brilliance measurements of pulsed soft x-ray sources is described. The calibrated system combines a novel elliptical off-axis reflection zone plate with charge-coupled device detection for simultaneous spectral and spatial measurements. Experiments on a water-window droplet-target laser-plasma source demonstrate $\lambda/\Delta\lambda \geqslant 1000$ spectral resolution and absolute flux and brilliance measurements. © *1997 American Institute of Physics.* [S0003-6951(97)04828-6]

The rapid development of compact pulsed soft x-ray sources paves the way for table-top applications of, e.g., lithography[1] and microscopy.[2] For such applications, accurate quantitative characterization of source parameters is increasingly important. In the present letter we describe a calibrated compact soft x-ray spectrograph, which is based on a novel grazing-incidence optical element in combination with charge-coupled device (CCD) detection. The spectrograph allows high-resolution spectral characterization in combination with quantitative flux and brilliance measurements.

X-ray sources are usually characterized by their spectral brilliance, i.e., photons/(s×sr×μm^2×0.1% BW).[3] For pulsed sources, it is more useful to define the pulse brilliance as photons/(pulse×sr×μm^2×0.1% BW). For multi-shot exposures the required exposure time for a given experiment may be calculated by combining the brilliance with, e.g., the repetition rate and the collection efficiency of the optical system. To determine the brilliance of an x-ray source, the emitted photon number, spectral distribution, and source size have to be measured simultaneously. For investigation of pulsed sources in the soft x-ray range (e.g., $\lambda \approx 1-10$ nm) this is a challenging task. Commonly used detectors that may be accurately calibrated, e.g., proportional counters, are not suitable for short pulses.[4] Apart from very expensive and large instruments, most grating spectrographs show relatively poor spectral resolution.[5] To our knowledge, crystal optics with reasonable performance are not yet available for this wavelength range.

For quantitative soft x-ray pulse brilliance measurements we designed a calibrated compact spectrograph based on a single grazing-incidence diffractive optical element. Grazing-incidence diffractive elements[6] have previously been employed for focusing hard x rays using crystal substrates[7] and soft x rays with multilayer substrates.[8] In the spectrograph described in the present letter, the flat glass-substrate element disperses the radiation emitted by a small x-ray source and focuses it onto a calibrated soft x-ray-sensitive CCD, as illustrated in Fig. 1. The basic pattern of this new x-ray optical element is given by nonconcentric ellipses, which, in grazing incidence reflection, act as a plane diffraction grating with highly curved lines. The projection of the pattern towards the source is a circular ring system, which follows the rules of fresnel zone plates in the first order approximation. Therefore, we label the element ''off-axis reflection zone plate'' (ORZ). For the design parameters (i.e., wavelength and geometry) the ORZ forms a diffraction-limited image on the CCD in the image plane. Since the image formation is due to diffraction, chromatic aberration results in defocused and displaced images of each spectral component in the x direction (cf. Fig. 1) on the detector—the spectrum. The image size of the focused wavelength in the spatial y direction is used to determine the source size, which is necessary for calculating the brilliance.

Line-emitting laser-plasma x-ray sources in the $\lambda \approx 2.3$–4.4 nm wavelength range are attractive for microscopy. For characterization of such sources the ORZ may be designed for optimal focusing of a selected spectral line. In the experiments described below the ORZ was designed to image the N VII $1s-2p$ line at $\lambda_0 = 2.478$ nm. This ORZ has an active area of 1×8 mm^2, consists of 6770 elliptical zones with a grating constant varying from 1168 to 1200 nm along the major axis of the ellipses. The object distance is 750 mm and image distance 1500 mm, resulting in 500 mm focal length and 2× magnification in the spatial imaging direction for the design wavelength. The grazing-incidence input angle is $\alpha = 4°$, leading to a diffraction angle $\beta = 1.5°$ for λ_0. Thus, this ORZ is operated in the -1 diffraction order, resulting in an angular dispersion of $d\beta/d\lambda = 32$ mrad/nm and a linear dispersion of $dx/d\lambda = 48$ mm/nm in the image plane at λ_0. The aperture is 6.7×10^{-4} and 3.7×10^{-4} in the spatial and spectral directions, respectively. The collected solid angle is 1×10^{-6} sr. Assuming a point source and perfectly aligned system, this results in a theoretical diffraction-limited spatial resolution of ~ 3 μm and spectral resolution of $\lambda/\Delta\lambda \approx 3300$.

The ORZ is manufactured by e-beam lithography and microfabrication processes. First, 5 nm Cr, 20 nm Ge, and 80 nm polymethyl methacrylate (PMMA) are deposited on a 12.7-mm-diam, 30 nm flatness and 1 nm rms roughness glass substrate. After lithographic patterning[9] and development of the PMMA, the unprotected Ge is removed by $CBrF_3$ reactive ion etching before the residual PMMA is removed. Fi-

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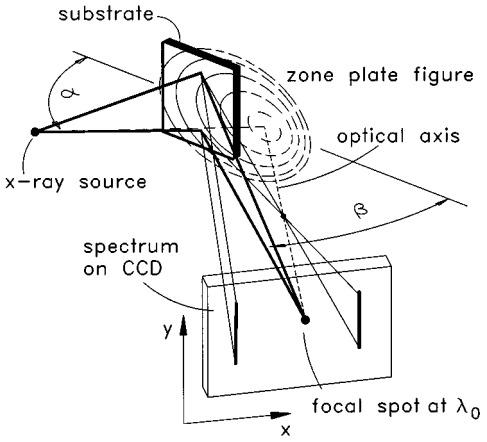

FIG. 1. Experimental arrangement for the off-axis reflecting zoneplate spectrograph.

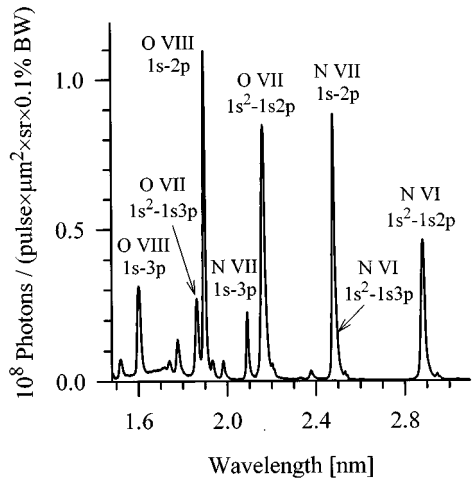

FIG. 3. Calibrated spectrum from ammonium hydroxide droplet-target laser-plasma source.

nally, a 10 nm Ni layer is evaporated onto the Ge, giving a higher reflectivity for the wavelength region $\lambda=1.5$–5 nm.

For quantitative measurements, the optical components and the detector have to be calibrated. The diffraction efficiency of the ORZ was measured at the x-ray microscopy beamline at the Berlin electron storage ring (BESSY). The solid line in Fig. 2 shows the -1 order diffraction efficiency of the ORZ as a function of wavelength. The maximum efficiency is ~ 0.06 at $\lambda \approx 2.2$ nm. The low efficiency at short wavelengths is due to the Ni L_{III} absorption edge at $\lambda=1.45$ nm. Consequently, ORZs designed for $\lambda<1.6$ nm should be coated with a different material. The decreasing efficiency for $\lambda>2.2$ nm is probably due to destructive phase effects, but not fully understood yet. The calibration of the thinned, back illuminated CCD (Photometrics AT200L with Tektronix TK1024AB) was performed at the PTB (Physikalisch Technische Bundesanstalt) radiometry beamline at BESSY.[10] The dashed line in Fig. 2 shows the fairly uniform quantum efficiency of the CCD, which was measured down to $\lambda=1.55$ nm and extrapolated to $\lambda=1.1$ nm (dotted).

The spectrograph consists of two vacuum chambers, containing the ORZ and the CCD, which are connected with vacuum tubes and adapted to the source chamber. In front of the ORZ a 200 nm freestanding Al foil is placed to remove scattered visible light from the plasma and a shutter to control the exposure time. To record a larger spectral range than that of a single CCD image, the ORZ is rotated so that the total deflection angle is kept constant.

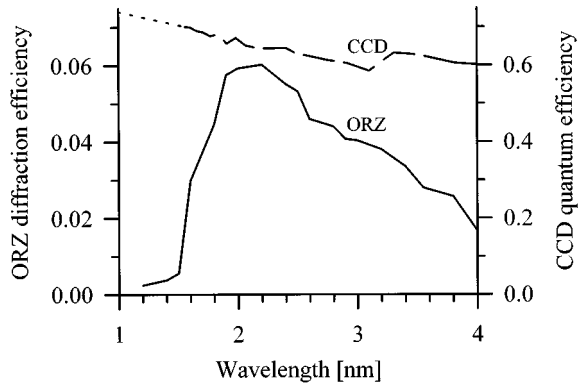

FIG. 2. Absolute calibration of the CCD detector and the ORZ diffraction efficiency.

The performance of the spectrograph was tested on the liquid-droplet-target laser-plasma source.[11] In this regenerative and practically debris-free source, x-ray emission is generated by focusing $\sim$70 mJ, $\sim$100 ps, 10 Hz frequency-doubled Nd:YAG laser (Continuum PY61C) pulses onto single ~ 15 μm droplets. For the test measurements concentrated ammonium hydroxide was used as target liquid, resulting in strong N VII and N VI emission at $\lambda=2.478$ and 2.879 nm. A 3 mJ ultraviolet prepulse was employed for enhanced x-ray emission.[12] The source size was determined to be ~ 30 μm full width half maximum (FWHM), which was confirmed by a separate pinhole camera measurement. Figure 3 shows the measured pulse brilliance in the 1.5–3.0 nm wavelength range. The exposure time varies for the different spectral regions from 1 to 10 s. The calibrated spectrum is dominated by line emission from highly ionized oxygen and nitrogen. For $\lambda<2$ nm, the oxygen lines are superimposed on a significant continuous background. In contrast, the $1s^2-1s2p$ N VI line at $\lambda=2.879$ nm is isolated (except for the small satellites at $\lambda=2.95$ nm), with a background intensity of less then 0.4% of the peak intensity within 0.1 nm spectral distance from the line. The low background may be measured due to the high dynamic range of the CCD. When employing the source for x-ray microscopy using zone plates it is important to minimize the background in order to reduce image noise.

Integrating the pulse brilliance over linewidth and source size results in the total photon flux per line. For the $\lambda=2.879$ nm N VI line this number is 3.2×10^{11} photons/(sr$\times$pulse). The accuracy of this value is estimated to be better than 50%, where the major source of uncertainty is in the calibration of the spectrograph. The reported photon flux is approximately a factor of 3 below previous measurements with filters and x-ray diodes.[11] In addition to the 50% uncertainties,[11] the difference may be explained by the influence on the diode measurements by the significant and previously unknown continuum background below $\lambda \approx 2$ nm.

In the above experiments, the spectral resolution is prob-

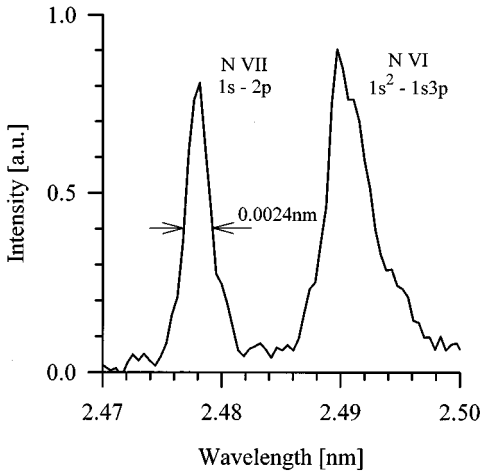

FIG. 4. High-resolution spectrum at the design wavelength employing low laser power.

ably limited by the source size due to the absence of an entrance slit. The spectral resolution of the spectrograph was therefore investigated by applying only the 3 mJ prepulse to the droplets, resulting in a source diameter of less than 10 μm. Figure 4 shows a fraction of the spectrum with the N VII $1s-2p$ ($\lambda=2.478$ nm) and N VI $1s^2-1s3p$ ($\lambda=2.490$ nm) lines. Due to different plasma parameters resulting from the use of only the UV prepulse, the peak intensity of the two lines is now equal. Under these conditions, the linewidth of the N VII $1s-2p$ line was determined to be 0.0024 nm, corresponding to $\lambda/\Delta\lambda\approx1030$. Up to now it is not possible to decide whether this value is limited by the actual linewidth or the performance of the spectrograph.

To estimate the influence of deviations from the design geometry, e.g., finite source size, alignment errors, or defocusing, a ray tracing program has been developed. It shows, e.g., that for the current ORZ a source diameter of ~40 μm restricts the resolving power to $\lambda/\Delta\lambda\approx500$. Thus, for large sources an entrance slit will be added to the optical arrangement. The ray tracing also demonstrates that in order to avoid coma the source needs to be positioned onto the optical axis within $\sim0.05°$. This is accomplished in the present arrangement by rotating the ORZ with a stepper motor.

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