Flat Field Soft X-ray Spectrometry with Reflection Zone Plates on a Curved Substrate

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Abstract: We report on the first experimental results obtained with a newly designed instrument for high-resolution soft X-ray spectroscopy, using reflection zone plates (RZPs) on a spherical substrate. The spectrometer was tested with a fluorescence source. High-resolution flat field spectra within ±50% around the design energies were measured at an interval of 150–750 eV, using only two RZPs: the first RZP, with its design energy of 277 eV, covered the band of 150–370 eV, and the second RZP, with a design energy of 459 eV, covered the band of 350–750 eV, where the upper boundary of this energy range was defined by the Ni coating of the RZPs. The absolute quantum efficiency of the spectrometer, including the optical element and the detector, was, on average, above 10%, and reached 20% at the designed energies of the RZPs. The resolving power E/∆E exceeded 600 for energies E inside the core range of 200–550 eV.

Keywords: soft X-ray spectroscopy; ultra-fast spectroscopy; reflection zone plate

1. Introduction

With the advent of novel soft X-ray sources that provide noticeably short pulses, the need for their temporal resolution in ultra-fast spectroscopy has been emerging rapidly over the past 10 years. Besides synchrotrons and free-electron lasers, relatively small state-of-the-art systems on the laboratory scale are meanwhile capable of producing femtosecond (fs) pulses, and the accessibility to a wide range of photon energies from regions near, or even below, the water window within (0.28–0.53) keV to about 1 keV could be verified in experiments.

In contrast to large facilities like the X-ray free-electron laser (XFEL), with an emission up to ~20 keV at an average rate >10^{12} photons per pulse, however, compact sources for the laboratory fall short of that number of photons—by orders of magnitude. To maintain both the spatiotemporal pattern of the pulses and a sufficient count rate on the detector of the spectrometer, the efficiency of the optical components plays a crucial role. Conical diffraction, for instance, is known as a convenient concept that fulfills those criteria in the (extreme) ultraviolet regime. Beyond ~0.2 keV, the varied line space grating (VLSG) proved as a valuable device for soft X-ray spectrometry in the past [1]. Further development led to a “holographic” VLSG, the off-axis reflection zone plate (RZP) that integrates two-dimensional focusing and wavelength dispersion in a single diffractive optical element. Manifold experiments demonstrated the appropriateness of this approach and good performance, especially in the domain of time-resolved measurements on ultra-short pulses [2].

The RZP [3], as a wavelength-dispersive optical element, has been successfully used in several designs of laboratory spectrometers for soft X-rays [4]. While demonstrating its evident advantages in comparison with conventional VLS gratings, like a high resolving power at the design energy and an excellent signal-to-noise ratio, RZPs on planar substrates suffer from a narrow energy range in parallel...
spectra registration, limiting the applications of this type of optics. Recent developments in theory, technology, and metrology of RZPs make it possible to fabricate RZPs on spherical substrates with a small radius of curvature down to 2 m. To compensate for the slope error of the substrate, an algorithm for diffractive wavefront correction [5] was used in the calculation of the groove structure.

The development of theory and technology for RZPs on figured substrates opens new possibilities for considerable improvement of the instrumental performance in soft X-ray spectroscopy, pushing forward the frontiers in pulse duration, energy resolution, and the efficiency of wavelength dispersive optical elements. Successful results of tests enabled NOB Nano Optics Berlin GmbH (NOB) to start customer-oriented production of RZPs on spherical substrates for spectroscopic applications.

The concept and theoretical calculations of the RZP properties on spherical or generally figured substrates were published, for example, in the paper [5] and several conference presentations. Unfortunately, up to now, all these theoretical results were not supported by experimental measurements, especially in the soft X-ray range. The novelty of our results lies in the first experimental measurements of properties of the RZP, fabricated on a spherical substrate in a practically useful soft X-ray spectrometer. The measurements became possible due to the latest achievements in technology, made by NOB, enabling the manufacturing of RZPs on a spherical surface with a relatively small radius. This paper is the result of joint efforts between the Institut für angewandte Photonik e.V. (IAP) and NOB in the calculation of the theoretical structure, modeling of the optical layout of a novel spectrometer, and finally, the precise production of RZP structures on a spherical mirror. The advanced technology, as developed at NOB, relies on company-internal innovations, and has no analog in the field of fabricating soft X-ray diffraction gratings so far.

Not only large scale research facilities, like the X-ray free-electron laser (XFEL), are in the scope of applications, but also laboratory sources; our spectrometer can be used as well with laser-produced plasma (LPP), high harmonic generation (HHG)—potentially from a relativistic oscillating mirror (RHHG), isolated attosecond X-ray pulses from a gas jet, and the fluorescence provided by an electron beam material analysis (EBMA).

We begin with an outline of our experimental idea with a short description of RZPs on spherical substrates in Section 2. A detailed view of the instrumental setup by which the spectrometer was realized is presented in Section 3, and Section 4 specifies the fabricated RZPs. Section 5 presents measured test spectra, followed in Section 6 by a discussion of our findings. Conclusions are combined with perspectives for a potential roadmap towards next steps in Section 7.

2. Reflection Zone Plate on Curved Substrates

We suggest using an RZP on a spherical substrate, based on its theoretical description [5]. In comparison with an RZP on a planar substrate, it has evident advances, but is much more difficult to fabricate. With the RZP on a spherical substrate, one can benefit considerably from the energy range, across which the diffracted beam is still sharply focused (Figure 1a). In the domain of that so-called “flat field” spectrometry, the resolving power $E/\Delta E$ depends only moderately on the energy of the photons, which were emitted by a sample. The difference could be illustrated by means of a numerical example, as depicted in Figure 1b. We took the technical parameters for RZP 1 from Table 1: $E_0 = 277$ eV, $R_1 = 838$ mm, $R_2 = 2500$ mm, $\alpha = 2.59^\circ$, $\beta = 4.11^\circ$, $r \to \infty$ (planar) or 28.617 mm (sphere).

With the program RAY-UI [6], ray tracing simulations were performed to retrieve the energy resolving power $E/\Delta E$ as shown in Figure 1b. An effective spatial resolution of 25 μm (FWHM) was assumed for the CCD with a pixel size of 13.5 μm.

The full width at half maximum (FWHM) is indicated in Figure 1b as 3.1 eV for the planar substrate, and 114 eV for the spherical one.
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**Table 1.** Technical data of the two reflection zone plates (RZPs). See Figure 2 for the meaning of the geometrical parameters.

| Parameter                        | RZP 1               | RZP 2               |
|----------------------------------|---------------------|---------------------|
| Energy range                     | 150–370 eV          | 350–750 eV          |
| Designed energy                  | 277 eV (C Kα)       | 458.6 eV (Ti Lβ, NIST) |
| Designed distances               | R₁ = 838 mm, R₂ = 2500 mm |
| Grazing angles                   | α = 2.59°, β = 4.11° |
| Grazing deviation angle          | θ = 6.7°            |
| Radius of spherical substrate    | r = 28.617 m        |
| Coating                          | Ni                  |
| Groove profile                   | laminar, 18.5 nm    | laminar, 10 nm      |
| Meridional line density          | 341.7–346.3–349.7 l/mm | 565.7–573.4–578.9 l/mm |
| Optical aperture (mer./sag.)     | 80 mm/6 mm          | 80 mm/4 mm          |
| Angular acceptance (mer./sag.)   | 4.3 mrad/7.2 mrad   | 4.3 mrad/4.8 mrad   |

3. Test of the Experimental Arrangement

In a transmission fs spectrometer, X-rays were emitted by a femtosecond source (HHG, XFEL, etc.) and focused by a toroidal or elliptical mirror onto a sample foil, which was additionally irradiated with a short infrared pulse in the so-called design of a “pump-probe” experiment. The X-ray transmission spectrum was subsequently analyzed by the spectrometer. For reasons of simplicity and high efficiency, the spectrometer had only one optical element: a focusing RZP array on a spherical substrate. Finally, the spectra were recorded on a charge-coupled device (CCD) detector.

The analytical part of the spectrometer was tested in the IAP laboratory using a fluorescence calibration source. Below, the results of that spectrometer part test are reported.

The optical layout of the absorption transmission spectrometer is shown in Figure 2. Only one optical element, a reflection zone plate on a spherical substrate, was used to obtain the emission spectrum of a sample target irradiated by a focused electron beam with an energy of 4.4 keV.
Figure 2. Optical layout of the spectrometer. The spherical substrate (red rectangle) carried two RZPs in a parallel layout, and one of them was used to select the desired energy range. See Table 1 for a definition and specification of the design parameters.

In this arrangement, we used an EBMA instrument from the company JEOL (model no. 6400) with an electron beam current of ~10 µA at an acceleration voltage of 4.4 kV, focused to a beam size of ~10 µm in diameter. The RZP substrate was mounted on a parallel robot (“SmarPod”, SmarAct GmbH) with 6 degrees of freedom (Figure 3). The camera, a CCD detector with an aperture of 27.6 × 6.9 mm² (2048 × 512 pixels), was located 2500 mm away from the RZP substrate. The photon transport line was a KF40 vacuum tube, connected with the vacuum chamber that contained the SmarPod (Figure 4). The RZP aperture served as the input aperture for the spectrometer. No slit was placed in between the source and RZP position. The total optical aperture and angular acceptance are shown in Table 1.

Figure 3. Substrate (100 mm × 30 mm) with two RZPs in its holder, mounted on the SmarPod in the vacuum chamber.

The parameters of the RZPs were chosen to cover the energy range between 200 eV and 550 eV at a high resolving power of E/ΔE > 500 (RZP 1) and E/ΔE > 1000 (RZP 2), without RZP rotation and detector translation (see Figure 5). With the central energies of 277 eV (RZP 1) and 459 eV (RZP 2), where the spectra were recorded with maximum resolution, the detector aperture with 2048 pixels covered overlapping energy ranges of 200–370 eV and 350–550 eV. To get an entire spectrum within
200–550 eV, it was sufficient to translate the RZP substrate only in the sagittal direction; the detector did not need to be moved.

![General view of the spectrometer. For the alignment, an ANDOR CCD camera was used.](image)

Figure 4. General view of the spectrometer. For the alignment, an ANDOR CCD camera was used.

![Energy resolving power E/ΔE for RZP 1 (black) and RZP 2 (red) in the aperture of the CCD detector with 2048 pixels. Ray tracing [6] was used for the simulation, assuming an effective spatial resolution of 25 μm for the detector.](image)

Figure 5. Energy resolving power E/ΔE for RZP 1 (black) and RZP 2 (red) in the aperture of the CCD detector with 2048 pixels. Ray tracing [6] was used for the simulation, assuming an effective spatial resolution of 25 μm for the detector.

Furthermore, the CCD detector could be translated along the focal plane to cover a larger energy range in the dispersion plane, e.g., 150–750 eV. We could also fabricate up to five RZPs on one substrate, covering the wide energy range of 50–1000 eV.

The user-friendly software RAY-UI [6] allowed for the convenient design and simulation of complete beamlines with several types of optical elements. Among them, RZPs on plane and curved substrates were implemented as specialized sub-routines that were applied to our design.

### 4. Optical Element

The RZPs were fabricated on a spherical substrate with a radius of curvature of 28.617 m. The substrate profile was carefully measured by two different methods, as was reported in [5]. The results, which we used for the correction of the RZP structure from aspheric and local slope (or figure) errors, provided a high spatial resolution. Table 1 summarizes the main parameters of the RZPs.

The efficiency of the RZPs was maximized by a proper depth of profile, as it is shown in Table 1. A special technology called “vector-scan ion etching technique” was used [7] for fabrication. In Figure 6a, the absolute efficiency of RZP 1 and RZP 2 is shown. The dips near 0.28 keV and 0.53 keV...
refer to the inevitable contamination of the Ni coating with a natural layer composed of carbon (C) and oxygen (O). Figure 6b presents the quantum efficiency of the CCD camera (greateyes GmbH) with a BI UV1 sensor.

Figure 6. (a) Diffraction efficiency of RZP 1 and RZP 2, measured at the reflectometer of the synchrotron facility BESSY II [8]. (b) Quantum efficiency of the CCD “GE-VAC 2048 512 BI UV1” (greateyes GmbH) sensor [9].

5. Experimental Results

The spectrometer was tested using standard samples, applied for the calibration of the EBMA instrumentation. Figure 7 shows the spectrum of standard stainless steel, which contains several metals, and is also contaminated by carbon and oxygen.

Figure 7. Measured sample spectrum of stainless steel. The data (red) are not corrected for the total quantum efficiency. X-ray emission lines are identified (blue vertical lines), and the observed diffraction order is noted in brackets.

The spectrum in Figure 7 was obtained as follows: in the focal plane of the RZP, the X-ray CCD camera [9] recorded a two-dimensional (2D) digital image of the diffracted beam. The photon counts of this intensity distribution were integrated along the sagittal direction, i.e., perpendicular to the
dispersive direction of the RZP. This procedure resulted in a raw spectrum, consisting of integrated count rates over the dispersive direction of the RZP. By calibration, with the pixel size of 13.5 μm and the geometrical parameters as given in Table 1, the pixel axis x was then transformed into the energy axis. For the grazing angles α, β, the arm length R₂, and d as the central groove period of the RZP in dispersive direction, the grating equation led to

$$E(\text{eV}) = \frac{1239.8}{d(\text{nm})} \left[ \cos(\alpha) - \cos\left(\tan^{-1}(x/R₂) - \beta\right) \right]$$

Afterward, the calibration could be continued using the known chemical composition of the sample, as in, e.g., Figure 7. The spectra were recorded in the energy range of 150–750 eV. The upper boundary of the energy range was due to the Ni absorption edge of the RZP coating. The entire spectrum was measured for three positions of the detector, at “0 mm” and at “±20 mm”, where 0 mm corresponded to the camera position where the designed energy was focused to the center of the detector. Displacements of ±20 mm referred to respective lateral translations of the detector (see Section 3). It should be mentioned that a camera with an aperture of 60 mm could cover the entire spectral range and would not require a detector movement.

The strong influence of higher-order diffraction on the spectral measurements is notable. For example, in Figure 7 we can identify the first, second, and third diffraction order of Fe Lαβ emission lines, as well as Cr L₁, etc. A disadvantage of this diffraction phenomenon was that more complicated spectra were produced. As an advantage, we gained the possibility of measuring higher energies out of the first order diffraction range, e.g., L lines of metals with a higher atomic weight, like Cu. In this case, the limiting factor was the critical angle of total external reflection.

The Ti/TiN spectra, as shown in Figure 8, demonstrated a high resolution and flexibility of the spectrometer. The Ti spectra were recorded from a pure Ti metal sample and a thin TiN film. The entire spectra of these samples were recorded in parallel using the same RZP 2. No detector or RZP movement was required. The data were not corrected for the total quantum efficiency.

**Figure 8.** (a) Measured L₂ and L₃ spectra of Ti and TiN. (b) Measured L₁₁, L₁, and N Kα spectra of Ti and TiN.

In Figure 9, the spectrum of a metallic boron target is shown. Obviously, the spectrometer could be used as a sensitive detector of boron concentration with simultaneous control of the carbon-oxygen contamination of the sample. In Figure 9, O fluorescence is evidently seen in the second order, beside the first order spectral line of C.
Figure 9. Boron metal spectra measured on RZP 1. The sample was naturally contaminated with a CO layer.

Figure 10 depicts the total quantum efficiency of the entire spectrometer, switching from RZP 1 to RZP 2 near 360 eV (see Figure 6a,b). Quantum efficiency correction was evidently needed as a parameter for quantitative analysis of the sample, but several other factors in the emission process, such as sample thickness, contamination, self-absorption, etc., will play an important role in evaluation.

6. Discussion

A classical VLS grating on a plane substrate can also cover such an energy range with good resolution and theoretical diffraction efficiency in the order of 10% [10]. However, the conventional VLS design needs many motion degrees of freedom for the grating, as well as the detector, to maintain a competitive performance. Alternatively, the pattern of a reflection zone plate (RZP) can be inscribed into a curved grating blank, enabling elegant access to the wide range of 150–750 eV, with only two RZPs for adjacent energy bands; to cover the central interval from 200 eV to 550 eV, only one translation in the sagittal direction of the RZP is needed to switch between the channels. The energy range between 200 eV and 370 eV can be covered by RZP 1 without moving. Similarly, RZP 2 covers the interval of 350–550 eV, also without moving. Hence, all optical components are fixed.

As it was already mentioned, the RZP on a plane substrate suffers from an extremely limited high-resolution energy range around the designed energy. To cover a larger range while a highly resolved spectrum is maintained, it is necessary to vary the input angle in proper steps. In this way, the spectrum is recorded sequentially part by part, at a high resolution. As an example, we considered the optical layout as shown in Figure 1a,b. The energy range, defined as the FWHM of the resolving power E/ΔE around E₀ = 277 eV, amounted to 3.1 eV or 1.1%. This means that parts of the spectral band of 200–350 eV must have been recorded in the sequential mode with approximately 50 steps.
The same spectrum, recorded with an RZP on a curved substrate, could be measured in one step. Hence, with the same photon counting statistics, the recording time for the same spectral range is 50 times shorter for the curved RZP compared to the planar RZP.

Moreover, our instrument was a real flat field spectrometer, where the CCD sensor was placed in normal orientation to the beam. In other words, a full spectrum from 200 eV to 550 eV could be recorded “in parallel” with two exposures. Depending on the required spectral resolution, the energy range could be varied and extended to, e.g., 150–750 eV by an additional re-adjustment of the detector, optimizing the application of the spectrometer. The CCD, which was used in our laboratory, featured 2048 \times 512 pixels of 13.5 µm in size, so a meridional angular movement of the detector of 0.46° was needed to access the spectral region outside from 200 to 550 eV. Using more than 2 RZP channels, even 50–1000 eV may be reached. The energy range of parallel spectra registration with one fixed RZP depends mainly on the size of the CCD camera. For detectors with a pixel number >4000 (or better, >6000) in the dispersion direction, the energy range can be as large as 50–400 eV for RZP 1 and 350–1000 eV for RZP 2. Such an expensive detector is available from greateyes GmbH or ANDOR Technology Ltd. To avoid a high cost in our proof-of-principle experiment, we therefore managed with a “synthesized detector aperture” to simulate the large detector, as described in Section 5.

In the present design, we chose an input distance of 838 mm. RZP 1 had an aperture of 6 mm (sagittal, S) \times 80 mm (meridional, M). RZP 2 had an aperture of 4 mm (S) \times 80 mm (M). With the input grazing angle of 2.59°, the angular acceptance for RZP 1 amounted to 7.2 mrad (S) \times 4.3 mrad (M), and for RZP 2 to 4.8 mrad (S) \times 4.3 mrad (M). For further enhancement of the efficiency, each RZP had an optimal depth of profile etched into the same substrate. Based on these features, the overall quantum efficiency of the spectrometer in the range of 150–700 eV was well above 10%, and exceeded 20% at the designed energies of the RZPs, as it is shown in Figure 10.

7. Conclusions

New types of dispersive optical elements, namely reflection zone plates on spherical substrates as an innovative class of 2D VLS gratings with optimized performance, are implemented in the optical design for extreme ultraviolet (XUV) and soft X-ray instrumentation. Our spectrometer covered a core energy range of 200–550 eV with a resolving power E/\Delta E\sim500–2000, without the necessity to move an optical element or the detector. Such a spectrometer can be successfully used for experiments of the EXAFS (extended X-ray absorption fine structure) and XANES (X-ray absorption near edge structure) types on the fs time scale. Due to the flexibility of the scheme, an enlarged spectral range of 150–750 eV can be accessed by means of a translation of the camera.

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Conflicts of Interest: Author J.P. is an employee of the company NOB Nano Optics Berlin GmbH. The other authors declare no conflict of interest.
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