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To cite this article: A Firsov *et al* 2013 *J. Phys.: Conf. Ser.* **425** 152013

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Novel wavelength-dispersive X-ray fluorescence spectrometer

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Abstract. A new spectrometer, utilizing a reflection zone plate based grating, for the Mn L fluorescence line was recently designed, manufactured and tested at Helmholtz Zentrum Berlin. The angular acceptance of the grating is $\sim 0.011 \text{ rad}^2$, the absolute efficiency at 640 eV is 16%, and the energy resolution, for a detector slit size of 120 μm and in simultaneous spectra registration mode, is about $\lambda/\Delta\lambda \sim 100$ FWHM.

1. Introduction

One of the aims of the new Institute for Nanometre Optics and Technology, founded at Helmholtz Zentrum Berlin (HZB), is the development and production of diffraction gratings for synchrotron radiation and space applications. Recently, a new design for X-ray fluorescence spectrometer has been suggested and successfully tested [1,2,3]. The design explores focusing and dispersive properties of the off-axis part of a reflection zone plate (RZP). Unlike the existing grating spectrometers, based on a grazing incidence spherical grating Rowland circle design [4] or on a variable line spacing (VLS) grating [5], the RZP spectrometer consists of only one optical element. The element is built onto a plane substrate and combines reflection, focusing and dispersion, all in one. Such a combination of optical properties offers possibilities to design spectrometers with very high efficiency and spectral resolution. The merger of spherical or toroidal mirror with a grating into one element on a plane substrate considerably reduces absorption (losses) in an optical system.

This paper reports on the manufacture and preliminary tests of highly efficient spectrometer optics designed for the detection of a low flux Mn fluorescence source. The radiation is generated by the interaction of an X-ray laser pulse with a jet of liquid containing very low concentrations of Mn atoms. The RZP was optimized for the detection of the Mn fluorescence lines $L\alpha_1$ (637 eV) and $L\beta_1$ (649 eV). The design energy 640 eV (1.94 nm) was chosen to cover both lines and enhance the signal. The main goal of the design was not to achieve a high spectral resolution, which was kept at ~ 100 , but to maximize the angular acceptance and efficiency of the optic. The total reflection efficiency of an RZP rapidly decreases with increasing incident and diffraction angles, and so to increase the efficiency we selected the negative first order of diffraction, where the diffraction grazing angle is smaller than the incident angle and offers higher reflectivity than the positive order. The structure of the grating, with a period of $\sim 140 \text{ nm}$ and very large angular acceptance of $3 \times (27 \times 40 \text{ mrad}^2)$ was produced using nano-structuring technology.

2. The spectrometer principle

A schematic of the optical layout of the spectrometer is shown in figure 1. The main dispersive element of the spectrometer is a 2D VLS grating. The grating is formed from an off-axis, periphery

part of an RZP generated by the projection of a Fresnel zone plate onto a plane surface [3]. The working area, shown between x_1 and x_2 in the figure, provides wavelength dispersion in the focal plane of the RZP. The incident radiation emitted by the source, S , is reflected and diffracted from the surface of the grating and focused at the focal plane, F , perpendicular to the optical axis, R' . The focal distance of the grating depends linearly on the design wavelength, λ , of the radiation. The dispersive property of the RZP can also be utilized to record spectra with a spatially resolving detector placed in the focal plane of the optic.

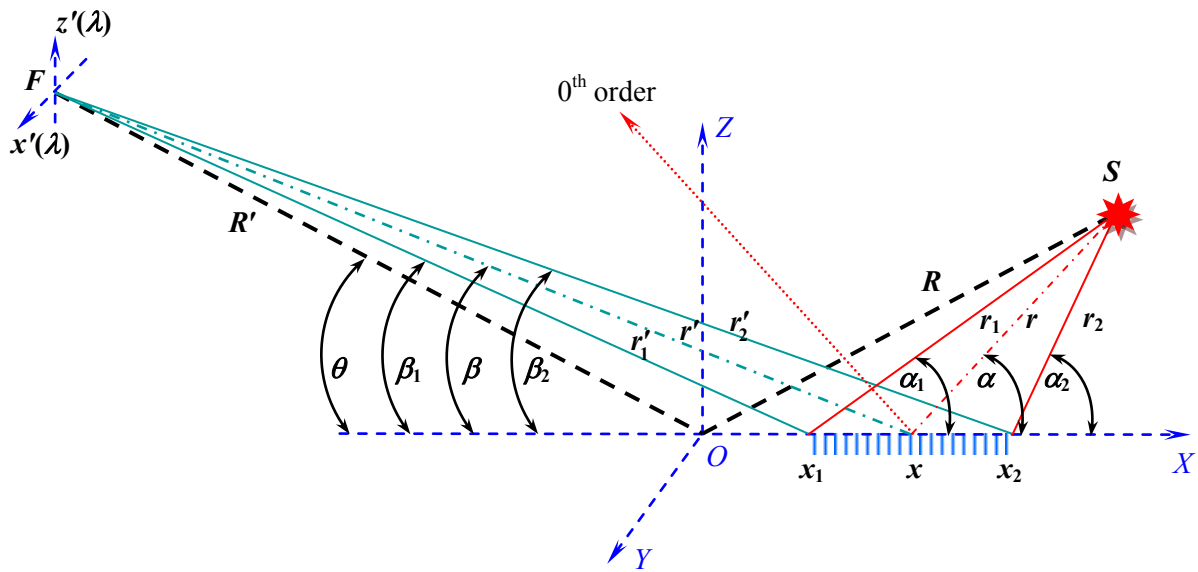


Figure 1. A schematic of the optical layout of the spectrometer. The 2D VLS grating is located between x_1 and x_2 , with the centre at x . The optical axis is along R' .

The off-axis location of the working area of the RZP brings two main advantages to the spectrometer: 1) the zero diffraction order is directed away from the optical axis and hence does not contribute to the background noise of a detector, and 2) the spectral selection can be done with a slit placed in the focal plane perpendicular to the optical axis. Unwanted wavelengths are dispersed but not transmitted through the slit.

From the standard grating equation, written as

$$m\lambda = d(\cos \alpha - \cos \beta) \quad (1)$$

where m is the diffraction order, λ is the incident wavelength, d is the grating period, α and β are the incident and diffraction angles respectively, the limiting incident angles, α_1 and α_2 , and the limiting diffraction angles, β_1 and β_2 , of the spectrometer (see figure 1) can be calculated. Also, with the help of the equation the expression for the angular dispersion of the grating, at the centre of its working area (x), can be written as

$$\frac{\Delta \beta}{\Delta \lambda} = \frac{1}{d \sin(\beta)} \quad (2)$$

where $\Delta \lambda$ is the variation of the incoming wavelength and $\Delta \beta$ is the corresponding variation of the angle of diffraction. The linear dispersion in the focal plane can be calculated using the equation

$$\frac{\Delta z'(\lambda)}{\Delta \lambda} = \frac{r'}{d \sin(\beta)} \quad (3)$$

where $\Delta z'(\lambda)$ is the vertical displacement of the focal spot for different wavelengths and r' is the distance from the centre of the grating to the focal spot (figure 1). From equation (3) the wavelength

(energy) resolution, which corresponds to the vertical displacement and is synonymous with the slit width, can be calculated. The expression for the local period, d_x , of the grating can be written as

$$d_x = \frac{\lambda}{\sin(\alpha)} \left[\sqrt{1 + \cot^2(\alpha) + \left(\frac{r'}{\Delta h} \frac{\Delta \lambda}{\lambda} \right)^2} - m \cot(\alpha) \right] \quad (4)$$

where Δh is the slit (pixel) width. The equation indicates the role of the geometrical magnification factor, which defines the minimum width of the slit. From equations (3) and (4) it can be seen that the resolution depends on the value of the diffraction angle, β , local period, d_x and slit size, Δh .

Equation (4) can also be used to calculate the design parameters of an RZP—the angle, θ , between the RZP's surface and the optical axis, and the distances, R and R' , from the centre of the RZP to the source and to the image, respectively (figure 1).

3. Design parameters of the spectrometer

The design parameters of the spectrometer were calculated and optimized to achieve the maximum angular acceptance at the working wavelength $\lambda = 1.94$ nm (640 eV). The grazing incidence angle $\alpha = 2.18^\circ$ yielded the maximum efficiency at this wavelength. The parameters are shown in table 1. Here, $\lambda/\Delta\lambda$ is the wavelength resolution, d is the local period at the centre of the grating, E is the design energy, the length of the grating is $L = x_2 - x_1$ and the other symbols have their usual meaning (see figure 1).

Table 1. Design parameters of the spectrometer at the centre of the grating.

$\lambda/\Delta\lambda$	α [deg]	β [deg]	Δh [μm]	r [mm]	r' [mm]	E [eV]	d [μm]	L [mm]
100	2.15	1.0	120	90	400	640	3.5	80

The limiting incidence angles, α_1 and α_2 , and the corresponding limiting diffraction angles, β_1 and β_2 , for the length L may also be calculated from geometrical considerations

$$\alpha_{1,2} = \text{atan} \left(\frac{r \sin \alpha}{r \cos \alpha \pm L/2} \right) \quad \text{and} \quad \beta_{1,2} = \text{atan} \left(\frac{r \sin \beta}{r \cos \beta \pm L/2} \right). \quad (5)$$

The limiting angular ranges, with fixed $\beta = 1^\circ$, and the corresponding grating local frequencies are shown in table 2.

Table 2. Limiting angular ranges and the corresponding grating local frequencies.

angular range	lines per mm
$\alpha_1 = 1.5^\circ$ $\beta_1 = 1.1^\circ$	79
$\alpha = 2.2^\circ$ $\beta = 1.0^\circ$	285
$\alpha_2 = 3.9^\circ$ $\beta_2 = 0.9^\circ$	1105

The ranges were calculated using the grating equation for the design wavelength of 1.94 nm. The total efficiency of a lamellar diffraction grating depends on the depth of the grating profile. The program REFLEC [6] was used to calculate the optimal profile depth for different local spatial frequencies of the grating.

Most gratings produced at HZB are generated with lamellar profiles on planar substrates made of most common materials. The structure described here was made using high-voltage electron beam lithography (VISTEC EBPG 5000plusES) and reactive ion etching techniques. A super-polished, gold coated silicon substrate, with 0.2 nm rms roughness and slope error smaller than 0.6 mrad rms, was used. An RZP, with lateral dimensions of 80 mm $\times$ 2.4 mm, lamellar profile of 13 nm and the minimum zone width of 70 nm, was produced on the surface of the substrate. Figure 2 shows an image

of the RZP. The optic was tested using the X-ray reflectometer at the BESSY II Optical Test Beamline. At-wavelength measurements of the efficiency are compared with a model simulation in figure 3. The fit parameters of the simulation, the depth of profile (13 nm rms) and the accuracy of the depth (1.2 nm rms), were calculated using program REFLEC. The energy resolution of the spectrometer was measured of $\lambda/\Delta\lambda = 63$ with 150 μm slit size and 10 μm source size.

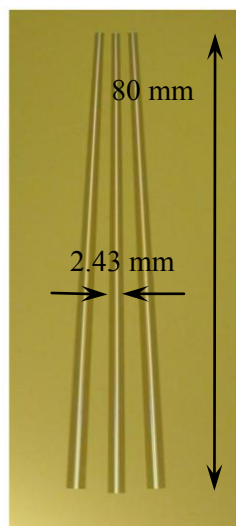


Figure 2. An optical image of the spectrometer structure (Si substrate, Au coating). The size of the substrate is $100 \times 30 \times 10 \text{ mm}^3$.

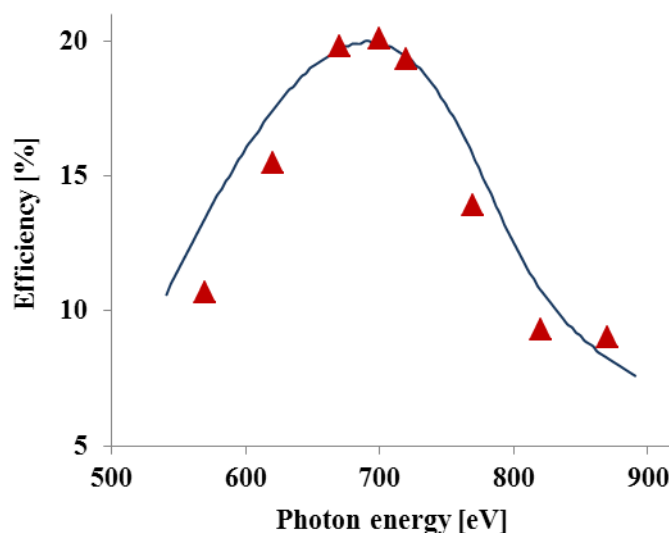


Figure 3. The absolute efficiency of the RZP (red markers) compared with the REFLEC simulation (solid blue line). The measurements were taken at the centre of the RZP (Si substrate, uncoated). At the design energy, 640 eV, the efficiency is 16%.

4. Outlook

The design of the spectrometer for the detection of low-flux Mn L fluorescence radiation is completed. The spectrometer can be used for experimental measurements with the synchrotron radiation sources and X-ray laser facilities. It has an angular acceptance of 2.5 times larger than the existing spectrometers' and 1.5 times higher total efficiency. Three identical RZPs, pointing at the same source, were made on the same substrate surface to improve the total aperture of the spectrometer.

5. Acknowledgement

This work is partially funded by the BMBF project 05K12CB4 "Next generation instrumentation for ultrafast X-ray science at accelerator-driven photon sources".

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