ISBN: 978-3-95450-207-3 doi:10.18429/JACoW-MEDSI2018-WEOPMA06

# A COMPACT AND CALIBRATABLE VON HAMOS X-RAY SPECTROMETER BASED ON TWO FULL-CYLINDER HAPG MOSAIC CRYSTALS FOR HIGH-RESOLUTION XES

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*Abstract*

In high-resolution X-ray emission spectroscopy (XES) crystal-based wavelength-dispersive spectrometers (WDS) are being applied for the characterization of the electronic structure of matter in various research fields like geosciences, chemistry or material sciences. Thereby, the von Hamos geometry provides high detection efficiency of spectrometers due to sagittal focusing using cylindrically bent crystals. To maximize the detection efficiency, a full-cylinder optic can be applied [1].
Based on this idea, a novel calibratable von Hamos X-ray spectrometer based on up to two full-cylinder optics was developed at the PTB. To realize the full-cylinder geometry, Highly Annealed Pyrolytic Graphite (HAPG) [2] was used. Besides its good bending properties, this mosaic crystal shows highly integrated reflectivity while offering low mosaicity, ensuring high resolving power [3]. The spectrometer enables chemical speciation of elements in an energy range from 2.4 keV up to 18 keV. The design and commissioning of the spectrometer will be presented. The spectrometer combines high efficiency with high spectral resolution (ten times better than in commercial WDS systems) in a compact arrangement also suitable for laboratory arrangements.

## INTRODUCTION

Constant development of novel micro- and nanomaterials in the industry is a huge challenge for metrology. As the research and development cycles are sometimes less than four months, reliable correlations of material functionalities and properties are called for while only few or no reference materials available. Therefore, reliable analytical methods for characterization of new material systems are needed that are not dependent on reference materials. An appropriate method is the reference-free X-ray spectrometry, which is based on the so-called fundamental parameter approach [4, 5, 6, 7, 8]. This method requires calculable synchrotron radiation and radiometrically calibratable instrumentation. In this work we present a novel compact wavelength-dispersive spectrometer that can be calibrated to ensure reference-free X-ray emission spectroscopy (XES). Due to its compactness and its high efficiency, it can be used in different experiments and at different beamlines. A laboratory X-ray source can be used as an excitation source as well.

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## VON HAMOS SPECTROMETER

To ensure high efficiency, the von Hamos geometry was used. In the von Hamos geometry, the radiation source – which, in the case of the XES experiment, is the fluorescence radiation from the sample – and a CCD camera are placed along the cylinder axis of a sagittally bent crystal, see Figure 1. While the original design was based on a cylindrically bent crystal covering only a segment of a ring, the use of full-cylindrical optics allows maximizing the solid angle of detection [9, 10].

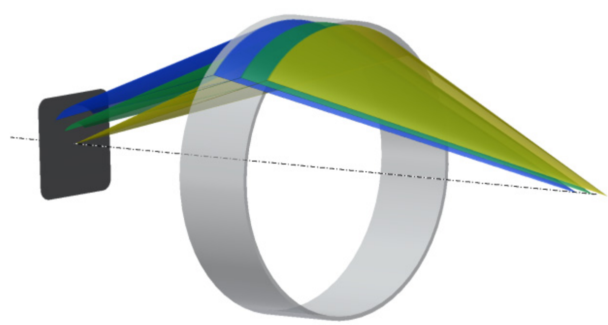

Figure 1: Von Hamos geometry.

To realize full-cylinder geometry, Highly Annealed Pyrolytic Graphite (HAPG) is especially suitable for this purpose, as it can be bent up to a 50 mm radius without any structural impact on the resolution. In the spectrometer presented here, 40 µm thick HAPG layers on cylindrical glass ceramic (Zerodur®) substrates were used. The HAPG is a mosaic crystal that consists of 50 µm to 80 µm mosaic blocks, which again consists of several approximately 1 µm sized crystallites that are tilted against each other [11]. Its mosaic structure leads to the highest integrated reflectivity of all known crystals, as all crystallites that are lying on the Rowland circle and fulfil the Bragg equation contribute to the crystal reflectivity. Simultaneously, HAPG has an especially small mosaic spread, the angle distribution function of the crystallites, and high resolving power.
For an additional increase of the resolving power, two HAPG optics are used in the beam path. An increase of the resolving power of around 40 % is expected [12]. This double reflection concept also increases the discrimination capability and improves the tailing of the spectral peaks.

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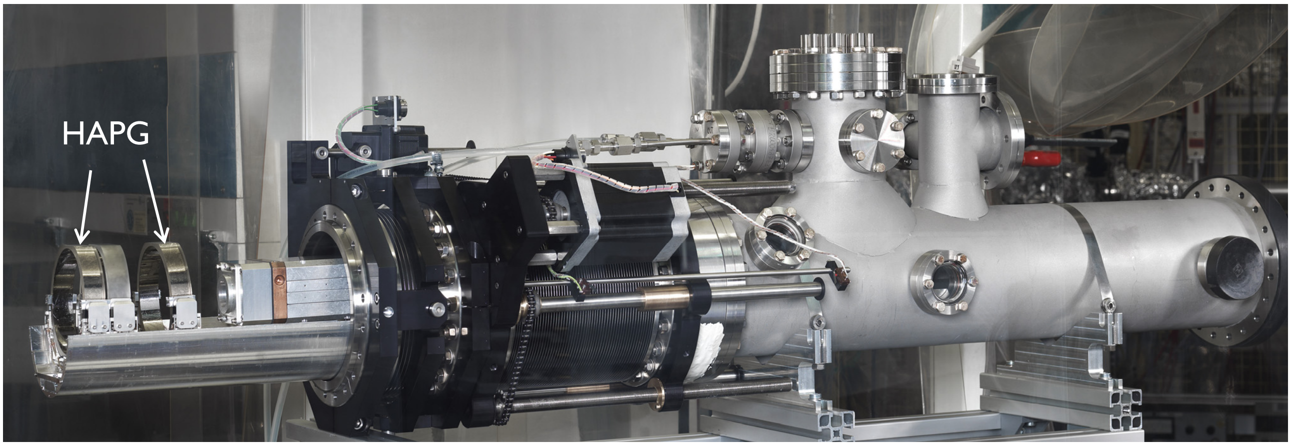


Figure 2: Picture of the presented X-ray von Hamos spectrometer.

Using the double Bragg reflection concept lowers the efficiency of the spectrometer compared to using one Bragg reflection.

In Figure 2 the spectrometer is shown. The whole front part goes into an experimental chamber. The flange inner diameter is 150 mm. The main components are three cylindrical optics and a CCD camera. The first and the third optics are HAPG optics. The second optic will be an optic based on a perfect crystal in the future. To prevent the icing of the CCD chip of the camera, an ultra-high vacuum (UHV) environment is necessary. The CCD camera is a water-cooled Princeton Instruments camera with 2048 x 2048 pixels. The pixel size is 13.5 µm. Furthermore, there is a port aligner, a linear feedthrough, and the ultra-high vacuum chamber.

## THE 17-AXIS MANIPULATOR

As only a small assembly space was available to move and to adjust the 100 mm inner diameter optics in a 150 mm inner-diameter tubular chamber, piezo manipulators were used.

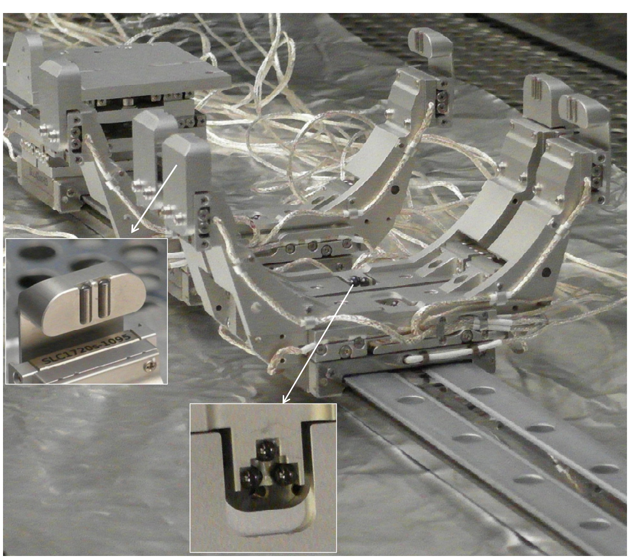

Figure 3: 17-axis manipulator from SmarAct GmbH.

As a result of a joint development of the PTB and SmarAct GmbH, a 17-axis manipulator was manufactured, which allows for the alignment of each optic and the CCD camera. The 17-axis manipulator is shown in Figure 3.

Each component can be moved by one slide, which can be used independently from one another. To ensure high reproducibility, all components were guided on one common linear slide. For each optic, two translator axes in x and y directions are included in addition to the feed motion. The rotation around the vertical axis of the optic is realized by two linear motors that are positioned on the optic sides and move against each other. Therefore, both sides of the optic as well as the bottom of the optic are equipped with a ball joint, see Figure 4.

Figure 4: HAPG optics with three ball joints.

On the manipulator, the optics holders are also equipped with ball or roll joints. At the bottom of the holder, three ceramic balls are included, Figure 3 zoomed view below, in which the below ball joint of the optics is positioned. To ensure a rotation around the horizontal symmetry axis of the optic perpendicular to the feed direction, both linear alignment-axes are moving parallel to the feed direction and in the same direction. Simultaneously the optic is tilted around the below ball joint. Thereby the horizontal

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cylinder axis is shifted downwards in the direction of the tilt. The principle is shown in Figure 5. To perform a rotation of the optic around the horizontal axis in an angle α, both linear motors, labeled with number *1* in Figure 5 left, have to move about $d_x$ which is calculated by the distance from the rotation point of the optic to the rotation point of the ball joint (=55 mm) of the optic multiplied with the sine of α. This movement has to be offset with the linear axis number *3* in the opposite direction about $d_x$. To compensate the vertical position change, motor number 3 has to be moved about $d_y$. *Dy* is calculated by $d_x$ multiplied with the tangent of half α. This vertical movement is possible due to the lateral slide of the optic ball joints along the roll joints of the manipulator, see Figure 3 zoomed view left.

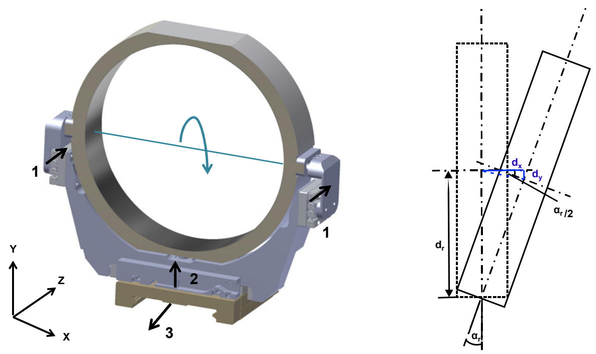


Figure 5: Principle of the rotation around the horizontal axis (perpendicular to the feed direction) using four linear motors.

This translation of the linear movement into polar coordinates enables the integration of two translational as well as two rotatory axes in limited space.

## THE ENERGY CHAIN

To ensure the water supply for the Peltier element of the CCD camera a considerable challenge was to ensure a guidance of the hoses over a large travel range of around one meter. Also, the guide of the energy cable for the piezo manipulators was necessary. Because of the limited available space in the chamber it was not possible to use a conventional energy chain here, which is usually made of separate chain links. Furthermore, it has been shown that during the movement of the chain, the chain links tilt abruptly when they are bent, so that can have an impact on the CCD camera position and thereby on your spectral image [13].

Therefore, a new concept was realized for the hose as well as for the electrical cables. This concept is based on a steel strapping made of stainless spring steel, which is bent like a tape measure and has a shape of a gutter with a radius *r*. For the guidance of the water hoses the gutter is bent to a radius *R*, so that the gutter inner radius *r* is placed outside, see Figure 6. Thereby both radii are the same *R*=*r*. The bending of the steel strapping leads to tensions in the material so that the steel strapping can hold the weight of the water hoses and is stiff to act as a guide at the same time.

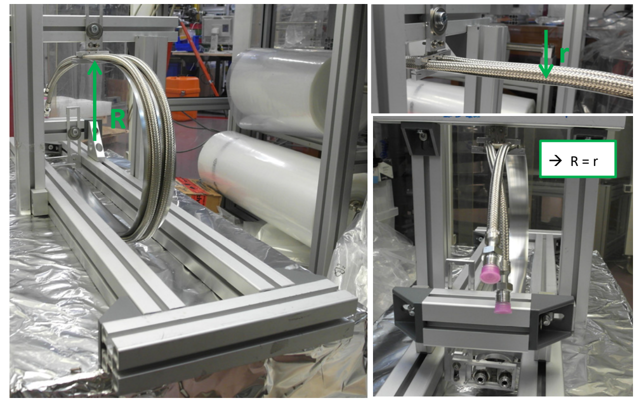


Figure 6: Guiding concept using gutter-type steel strapping.

To ensure that the electrical cable will stay in the guide they were fixed with a copper wire, Figure 7.

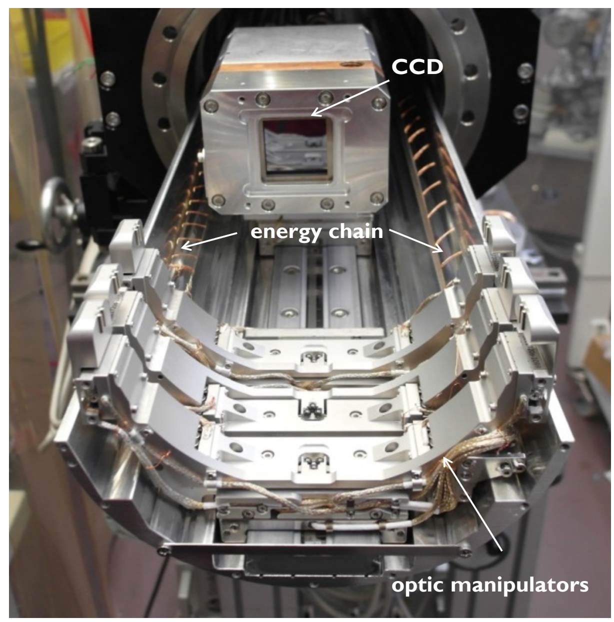


Figure 7: Energy chain for the electric cable of the optic manipulators.

With the here presented guide concept it is possible to carry and guide cables and hoses over a long travel range. At the same time this concept provides an ultra-high vacuum solution.

## CONCLUSION

In this paper a novel calibratable wavelength-dispersive spectrometer was presented. The focus of the development was the compactness of the spectrometer as well as the stiffness of the mechanical system. Due to application of small sized piezo motors and a water hose guide concept two 100 mm inner diameter optics could be integrated in a 150 mm inner diameter tubular UHV chamber. Despite of the compact design a large solid angle was realized due to the full-cylinder optic. The condition for spectrometer calibration were fulfilled from the mechani-

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cal side in respect to the relevant parameters such as detector response function and detection efficiency. On account of the spectrometers compactness, its high efficiency and its high spectral resolving power ($E/\Delta E = 2800$ for titanium $K\beta_{1,3}$) a transfer to a commercial laboratory tool is quite possible. It can be expected that such high-resolution X-ray spectrometers will be applied in different research fields, for ex-situ, in-situ and operando experiments for different material systems.

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