Journal of large-scale research facilities, 2, A49 (2016) http://dx.doi.org/10.17815/jlsrf-2-65

Published: 19.02.2016

KMC-2: an X-ray beamline with dedicated diffraction and XAS endstations at BESSY II

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Abstract: The KMC-2 beamline is dedicated to provide X-ray radiation with high energy stability and resolution. The experimental setup is optimized towards offering a wide range of methods and sample environments. Two permanent endstations can be used in alternation. DIFFRACTION is a flexible multi-purpose diffractometer, based on a Huber six circle diffractometer in psi geometry. XANES provides the possibility for EXAFS, XANES and X-ray fluorescence measurements at-air.

1 Introduction

The beamline KMC-2 (Fig. 1) is located at the synchrotron light source BESSY II, HZB, Berlin. It provides X-ray radiation with medium photon flux between 4 keV and 15 keV and linear photon polarization, with high energy stability and resolution (Erko et al., 2000). This is achieved by an optical design described in detail in section 3. No capability for experiments in continuous vacuum is currently offered, as this would negatively affect the desired flexibility in sample environment and experiment geometry. The beamline is permanently equipped with two endstations, named XANES and DIFFRACTION. It is possible to switch between these two stations within hours, if the experiment calls for the application of methods from both. Both stations allow for mounting of comparatively large sample environments.

KMC-2 XANES (Fig. 2) is a dedicated endstation to investigate the short-range environment around selected atomic species in condensed matter by X-ray Absorption Spectroscopy. This station provides the possibility for EXAFS, XANES and X-ray fluorescence measurements at-air. The detector system consists of three ionization chambers, Si-PIN photodiode, energy-dispersive detector (BRUKER X-Flash)

*Cite article as: Helmholtz-Zentrum Berlin für Materialien und Energie. (2016). KMC-2: an X-ray beamline with dedicated diffraction and XAS endstations at BESSY II. *Journal of large-scale research facilities, 2*, A49. http://dx.doi.org/10.17815/jlsrf-2-65

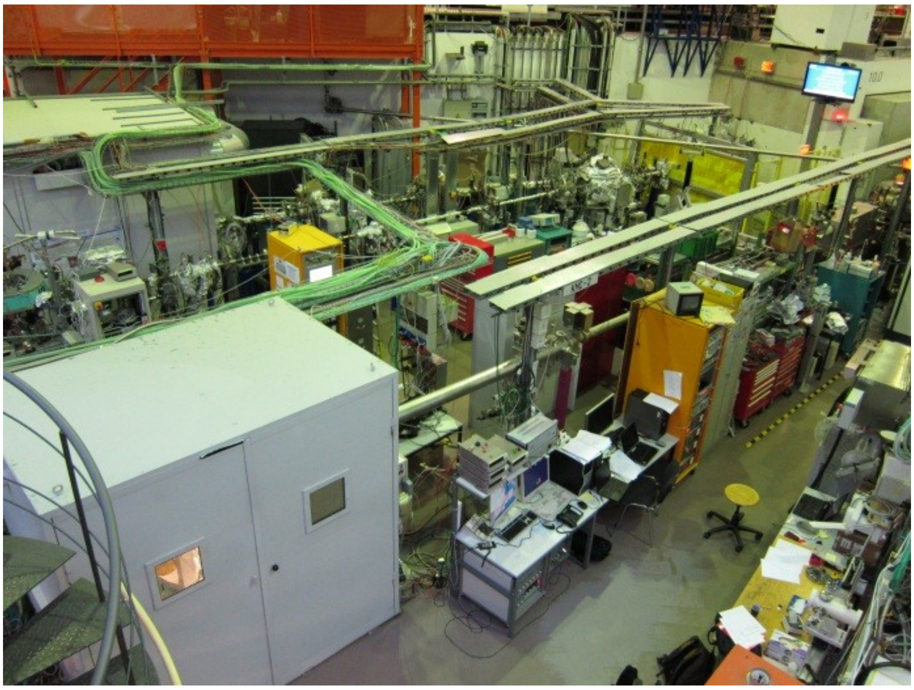

Figure 1: Top-view of beamline KMC-2. Both endstations are contained in the hutch.

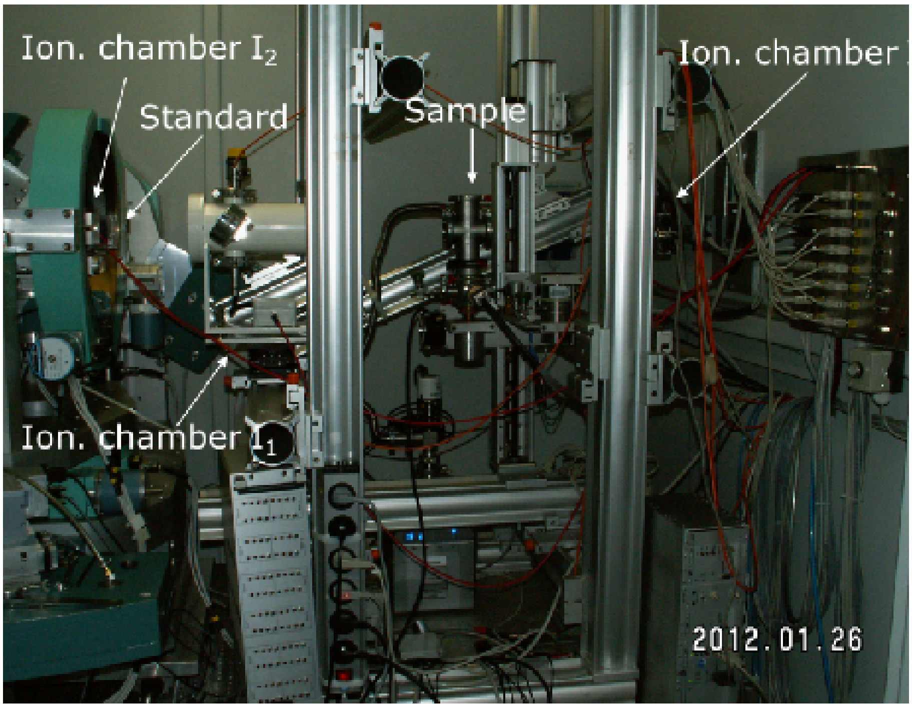

Figure 2: Lateral view on XANES transmission experiment with three ion chambers; Huber goniometer removed to allow large sample chamber.

and scintillation counter, which can be used in any combination. An add-on microprobe capillary system with the spatial resolution of $> 5\mu m$ is available for micro-XAFS and micro-fluorescence experiments. The experimental setup allows for a parallel monitoring of the transmitted intensity as well as for fluorescence yield measurements. A Linkam THMS600 temperature chamber is available onsite, which allows for a temperature range of 77 to 873 K. Various systems for experiments under controlled atmosphere and gas loading can also be provided by the HZB.

The station DIFFRACTION (Fig. 3) is a flexible six circle diffractometer in psi geometry, covering most scattering requirements up to a diffraction angle of 140°. A scintillation point detector (Cyberstar) with motorized detector apertures allows high resolution experiments. A MikroGap area detector (Bruker Våntec 2000) provides reciprocal space coverage with high counting rates and low background. The sample is mounted on a motorized stage allowing movement in all three directions as well as rotation.

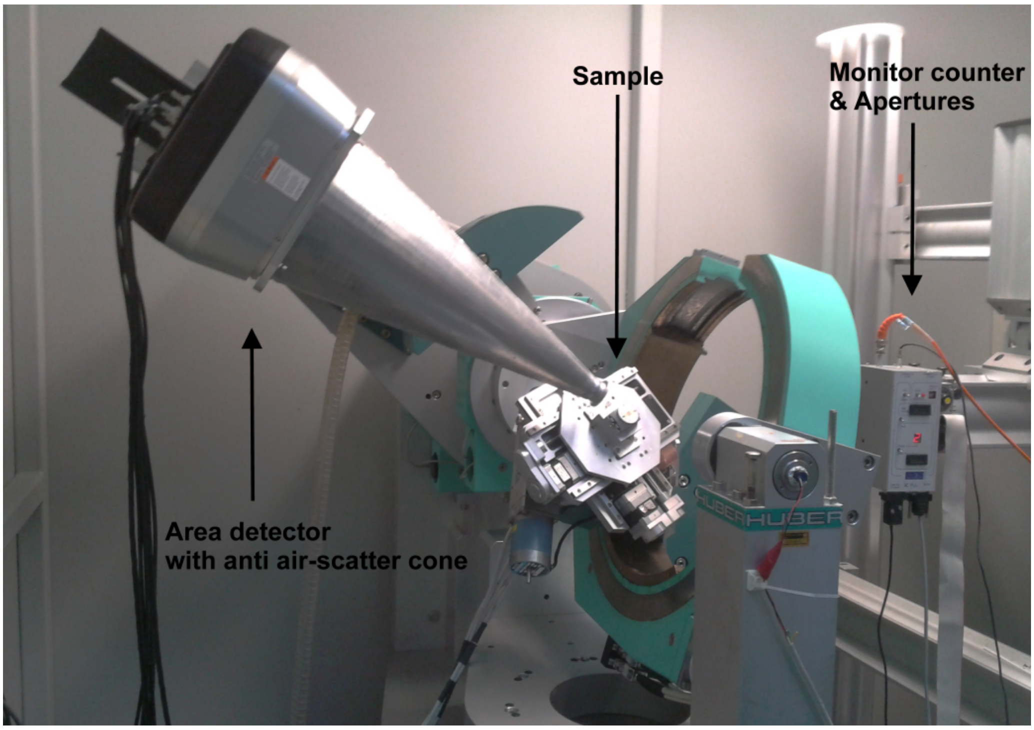

Figure 3: View of the DIFFRACTION station on the KMC-2 beamline, with area detector mounted.

Sample environment available on-site includes an Anton-Paar Domed Hot Stage for experiments up to 1000°C without geometry restrictions and a Be-dome high temperature vacuum chamber, also up to 1000°C, providing vacuum down to 10^{-6} mbar. Again, various systems for experiments under controlled atmosphere and gas loading can be provided by the HZB.

2 Instrument application

Experimental methods feasible at XANES include
- EXAFS (Extended X-ray absorption fluorescence spectroscopy)
- XANES (X-ray absorption near edge structure spectroscopy)
- XRF (X-ray fluoresence)
- μEXAFS
- μXANES
- μXRF

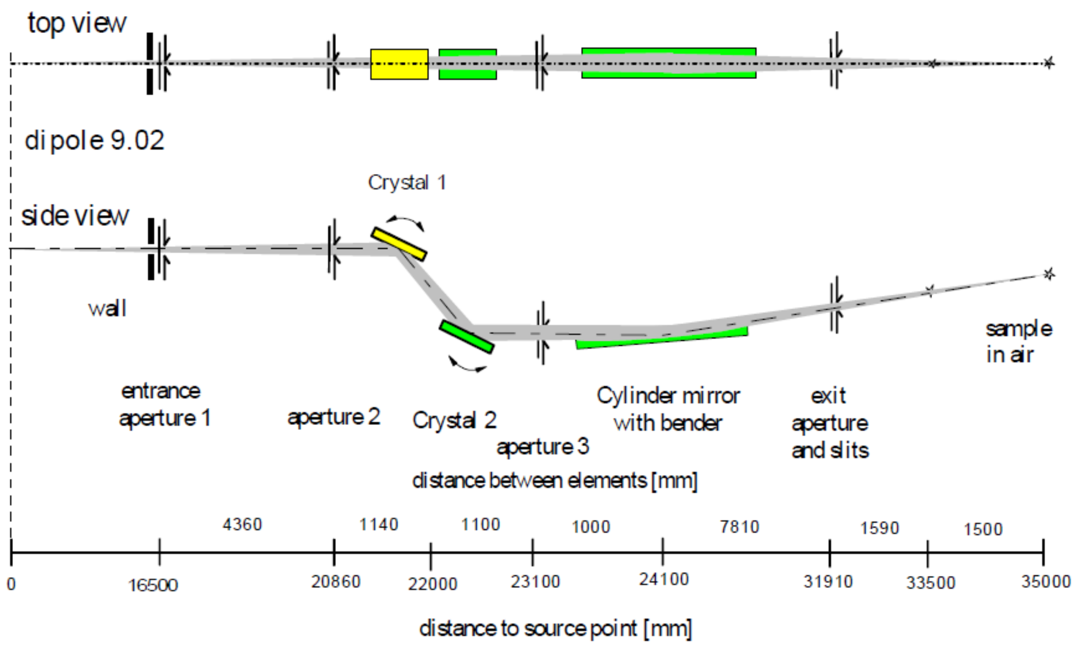

Figure 4: Optical layout of beamline KMC-2.

All microprobe methods can also be applied in mapping experiments using the sample stage motors. The energy range 4.0 keV < E < 15 keV is suitable for K-edge studies of elements in the range 21 < Z < 36 and L-edge studies of elements in the range 49 < Z < 83. The sample thicknesses for experiments are in the µm range for transmission experiments. Thin film and/or dilute systems studies are possible in fluorescence mode.

The endstation is frequently used by scientists from the fields of material science, chemistry and biology.

Typical materials under study are:

- Liquids, molecular solutions, liquid crystals
- Single- and poly-crystalline materials
- Amorphous and highly disordered solids
- Molecules and macromolecules containing metallic atoms or partially substituted with heavy atoms

The setup of DIFFRACTION allows a wide range of diffraction and scattering experiments, including

- Reflectometry
- Grazing incidence diffraction (GID)
- Wide-angle X-ray scattering (WAXS)
- Reciprocal space mapping of single crystal Bragg reflections
- Powder diffraction
- Anomalous diffraction
- Texture analysis

Typical applications are:

- Reflectometry
- Phase analysis and texture in thin films
- In situ strain analysis under mechanical or electro-chemical stress
- Structural studies by Rietveld refinement, both in situ and ex situ

3 Source and optical design

The source is the dipole D9.2, with an optical source size of about 144 μm horizontal and 41 μm vertical at 1 mrad horizontal acceptance. The optical layout (Fig. 4) is centered on a double monochromator of two Ge-graded Si(111) crystal substrates (Erko et al., 2001). The crystals can be rotated independently from 2.6° up to 70°. With this the useful energy range of the beamline is limited only by flux from the dipole (upper limit 15 keV) and absorption in air (lower limit 4 keV). Beam intensity is stabilized by MOSTAB electronics with an accuracy better than 0.3%.

4 Technical data

Location	11.1
Source	D9.2
Monochromator	two independent Ge-graded Si(111) crystals (KMC-2)
Beam intensity stabilization	MOSTAB electronics (accuracy better than 0.3%)
Energy range	4 keV - 15 keV
Energy resolution	4000
Flux	10^7 - 10^{10}
Polarization	Horizontal
Divergence horizontal	2.5 mrad
Divergence vertical	0.5 mrad
Focus size (hor. x vert.)	250 μm x 600 μm
Distance Focus/last valve	2500 mm
Experiment in vacuum	No
Free photon beam	No
Control system	EPICS
Data-acquisition software	SPEC
Endstation DIFFRACTION:	
Goniometer	Huber, six circle psi-geometry, open Eulerian cradle
Sample stage	Huber XY-stage (5102.18) + Z-stage
Point detector	Cyberstar Scintillator, motorized & manual apertures
Area detector	Bruker Våntec 2000 MikroGap, 14 cm x 14 cm active area, 2048 x 2048 pixels, 250 μm spatial resolution
Endstation XANES:	
Manipulators	Huber goniometer
Detectors	BRUKER X-Flash, 3 ionization chambers, Si-PIN photodiodes
Microfocusing	5 μm x 5 μm with capillary optics

Table 1: Technical parameters of beamline KMC-2 and the Diffraction and XANES stations .

References

Erko, A., Packe, I., Gudat, W., Abrosimov, N., & Firsov, A. (2001). A crystal monochromator based on graded sige crystals. *Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment*, *467-468, Part 1*, 623-626. http://dx.doi.org/10.1016/S0168-9002(01)00430-2

Erko, A., Packe, I., Hellwig, C., Fieber-Erdmann, M., Pawlizki, O., Veldkamp, M., & Gudat, W. (2000). Kmc-2: the new x-ray beamline at bessy ii. *AIP Conference Proceedings*, *521*(1), 415-418. http://dx.doi.org/10.1063/1.1291824