

The Extreme Conditions Beamline at PETRA III, DESY: Possibilities to conduct time resolved monochromatic diffraction experiments in dynamic and laser heated DAC.

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Abstract. We present plans for the new Extreme Conditions Beamline at PETRA III, DESY, Germany. The beamline is being designed and built with the specific goal to explore time resolved high-pressure and -temperature x-ray diffraction experiments in the dynamic and laser heated diamond anvil cell. Within we discuss the conceptual design of the optical components and experimental setup to conduct monochromatic high-pressure powder diffraction experiments in the sub-second time regime.

1. Introduction

For many years, laser heated Diamond Anvil Cell's (DAC) have been used to determine the static compression behavior of materials at simultaneous high-pressure and -temperature in order to explore geophysical phenomena [1] and to systematically study the crystal chemistry of elements, oxides, nitrides, carbides, etc. [2]. Most of these experiments require structural analysis of the powder and single crystals by means of x-ray diffraction. While laboratory x-ray sources can provide some information at lower pressure, only the invention of very brilliant x-ray sources such as synchrotrons of the 2nd and 3rd generation have enabled multi Mbar experiments at thousands of Kelvin. In particular the change from energy dispersive to monochromatic diffraction with area detectors has revolutionized structural analysis at simultaneous very high-pressure and -temperature. However, there has been a very large divide between the community that conducts static and dynamic high-pressure, -temperature experiments. Fortunately, that divide is being bridged by new developments such as the dynamic [3] as well as pulsed laser heated DAC [4]. There have been considerable efforts to transfer these laboratory based experiments to the current 3rd generation synchrotron facilities and to optimize beamlines to conduct time resolved high-pressure, -temperature experiments [5-9] that allow the exploration of structural changes in real time.

Within this work we present plans for the new Extreme Conditions Beamline that is currently under construction at the most brilliant synchrotron x-ray source in the world, PETRA III, DESY, Germany. The goal of this beamline is to provide a dedicated tool that will bridge the static and dynamic high-pressure experimental regime enabling sub second time resolved experiments in the dynamic and laser heated DAC.

2. Extreme Conditions Beamline

The Extreme Conditions Beamline, P02.2, is located in Sector 2 of the new 3rd generation synchrotron, PETRA III at DESY. Sector 2 is shared with two other beamlines, “The Micro- and Nanofocus X-ray Scattering Beamline” P03 and the “High-Resolution Powder Diffraction” beamline P02.1. Although located in the same straight section of the storage ring, P02 and P03 each command their own undulator that are positioned in canted configuration and thus enable independent operation. Beamlines P02.1 and P02.2 are sharing the same undulator (U23). Beamline P02.1 will be tuned to 60 keV using the 7th harmonic of the undulator spectrum. The photon beam for this beamline will be provided through a monochromator that is equipped with a (111) diamond and (111) silicon crystal in Laue geometry (Fig. 1), where the (111) diamond crystal is filtering the 7th harmonic. The beam that is passing through the (111) diamond Laue crystal will be used to provide photons for beamline P02.2. While certain operational modes on beamline P02.2 will require control of the undulator there will be an effort to keep the energy of the 7th harmonic tuned to 60 keV to allow optimal operation of beamline P02.1. Hence, the energy for the Extreme Conditions Beamline needs to be fixed during these periods to either the 3rd or 5th harmonic at 25.7 and 42.9 keV, respectively.

Below we describe the different optical components that are going to be used to provide the Extreme Conditions Beamline P02.2 with monochromatic x-rays. The operation of the beamline with pink beam will be possible at a second stage of the development and is going to be discussed elsewhere. Finally, we will introduce the conceptual design of the experimental station for beamline P02.2 and its proposed configuration into a laser heating and general purpose experiment.

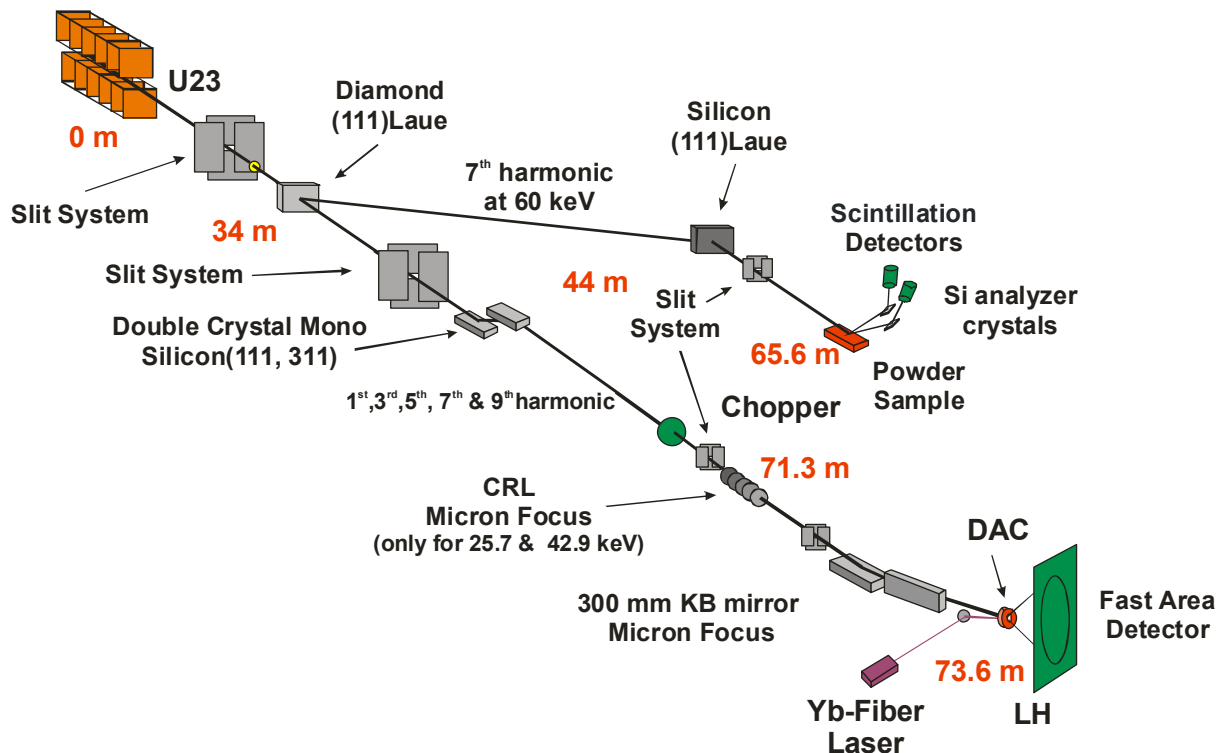


Fig. 1: Proposed optical train for monochromatic experiments at the Extreme Conditions Beamline P02 at PETRA III, DESY. CRL: Compound Refractive Lenses, KB: Kirkpatrick-Baez mirror, LH: Laser Heating Experiment. Red numbers indicate distance from the source.

2.1. Storage Ring and Optics for the Extreme Conditions Beamline

The storage ring parameters of PETRA III are described in [10] and are listed in Table 1. The unique feature of the rebuild storage ring is the very low emittance of the electron beam of 1 nmrad that will be achieved through the use of damping wigglers in the north and west octant of the storage ring. As a result the electron beam creates in the undulator a very low divergence photon source that can easily be focused even at large distances from the source. The monochromatic optics planned for the Extreme Conditions Beamline will consist of a standard monochromator developed at DESY for PETRA III and a combination of 300 mm long Kirkpatrick-Baez (KB) mirrors as well as Compound Refractive Lens (CRL) systems.

2.1.1. Undulator

All of the 14 undulator devices that are currently being built and installed in the new storage ring of PETRA III are described in [10]. Beamline P02.2 will be using an undulator U23 in high- β configuration that has been described in [11] and listed in Table 2.

Machine Parameters		Insertion Device Parameters	
Energy	6 GeV	Device	U23
Circumference	2304 m	Minimum magnetic gap [mm]	9.5
Harmonic Number	3840	Period length λ_U [mm]	23
HF	500MHz	Length L [m]	2
Horizontal Emittance	1 nmrad	Peak field B_o [T]	0.61
Coupling Factor	1%	Deflection parameter	1.3
Beam Current	100 mA	1 st Harmonic E_1 [keV]	8.0
Number of bunches	960 or 40	Total power P_{tot} [kW]	1.7
Number of DBA cells	9	On-axis power density [kW/mrad ²]	71
DBA Cell Length	23.013 m	Power in 1x1mm ² at 40m [W]	44
β_x	20.12 m	High- β source size (10keV)	140 (H) x 5.6 (V) μm^2
β_y	2.36 m	High- β source diver. (10keV)	7.9 (H) x 4.1 (V) μrad^2

Table 1: Machine parameters from [10]. Table 2: Parameters of the planar undulator for 100 mA from [11].

2.1.2. Monochromator

The monochromator for P02.2 will be a high heat load instrument that was developed at DESY in collaboration with *FMB Oxford*. Its prototype is described in [11]. The directly driven Bragg axis holds a silicon (111) crystal for the energy range from 2.4 to 54.0 keV and a Si (311) for 4.6 to 103.3 keV, both attuned to a fixed exit of 21 mm from the incident beam. Calculation with the ray tracing program SHADOW indicate that the (111) silicon crystal is large enough (25 (H) x 50 (V) mm) to capture large portions of the beam in the entire energy range up to 53 keV. Latter is due to the small divergence of the x-ray beam resulting in a beam size of ~ 0.500 (H) x 0.35 (V) mm at 44.5 m from the source, just before the high heat load monochromator. Hence, there is no need to use the (311) silicon crystal pair that can be attuned to a much steeper angle capturing more of the x-ray beam before reaching 53 keV. $\Delta E/E$ for both silicon crystal pairs stay in the range of $1-2 \times 10^{-4}$ throughout the entire energy range of the envisioned operation, i.e. from 24 (minimum gap of the 3rd harmonic) to 80 keV. For the calculations we assumed that the heat bump on the first and second crystal is kept to a minimum since the crystals are cooled with liquid nitrogen.

2.1.3. Focusing Optics

As with all Extreme Conditions Beamlines, focusing the beam to a size of 1-5 μm is a necessity in order to conduct high-pressure experiments in the Mbar range and to avoid diffraction from the gasket. We have decided to use two separate focusing systems, a Kirkpatrick-Baez (KB) mirror system [13] and a Compound Refractive Lens (CRL) changing system developed at DESY.

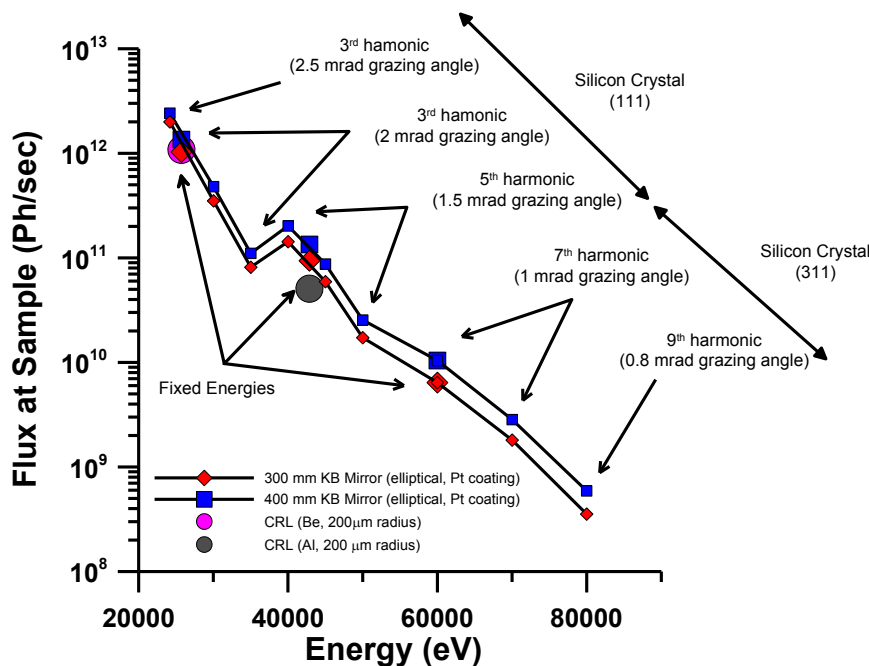


Fig. 2: Results from ray-tracing calculations with SHADOW to predict the flux at the sample position for a 300 and 400 mm long KB mirror system. For comparison we also plot the flux at the sample position expected for a set of Be and Al CRLs.

The KB mirrors will be used primarily to focus the beam to less than 2 μm , and to allow rapid changes in the energy without the necessity of refocusing the beam. In order to test what flux

and focus spot one can expect at the sample position using a KB mirror system we conducted ray tracing calculation with the program SHADOW for a 300 mm long mirrors assuming a distance from the source of 73.03 (V) and 73.33 m (H) and a distance of 0.600 (V) and 0.300 (H) m between the center of the mirror and the focal spot. Additionally, we performed calculations for a 400 mm long KB mirror system with distance of 73.03 (V), 73.43 (H), 0.8 (V) and 0.4 (H) m, respectively. The shape of the mirror was assumed to be elliptically, depicting a slope error of 0.5 μrad after coating with platinum. The expected flux at the sample position is illustrated as a function of the energy in Fig. 2. The focal spot for the 300 mm KB mirrors stays below 2 μm whereas that for the 400 mm mirrors stays below 3 μm . The calculations indicate that the 300 mm KB system will be optimal for the operation at the Extreme Conditions Beamline since the focal size is fairly small while collecting large portions of the beam. In contrast, the 400 mm system creates a focal spot of around 3 microns while capturing only slightly more of the beam resulting in an increase of $\sim 1/3$ in the flux at the sample position.

For the operations with a fixed energy of 25.7 or 42.8 keV we plan to use CRLs described in [14] in conjunction with a lens changer developed at DESY. The lens changer allows quick and very reproducible switch of the different lens packages so that a preset focus can be achieved with very little effort. For the fixed energies of 25.7 and 42.8 keV we assumed lens stacks of 80 Be and 140 Al lenses, respectively, resulting in a focal spot of 5.2 (H) and 0.2 (V) μm at a focal distance of 2.45 m from the center of the lens package. Because of the large amount of lenses necessary to achieve a 5 micron focus with a 0.2 mm lens radius (standard) the estimated flux at the sample position is equal to that of the KB mirror system at 25.7 keV and a factor two lower for the focus at 42.8 keV. However, because of the routine usage of these energies and the ease of focusing, the somewhat lower flux seems justified.

2.2. Laser Heating Experiment for the Extreme Conditions Beamline

The experimental hutch for the Extreme Conditions Beamline will be 7.5 x 5 x 4 m (L x W x H) in dimension. Thus, the hutch offers ample space for two separate experimental configurations. The downstream portion of the hutch will be occupied by a dedicated laser heating setup, while the upstream part of the hutch allows space for a second experimental setup, possible for a cryostat and other heavier equipment. The laser heating system for the experiment has been build by the

Department of Crystallography at the University of Frankfurt through funding from the “Bundesministerium für Bildung und Forschung” as part of a “Verbundprojekt”. Currently, the system displays a 100 W Yb-Fiber laser that will be split in two beams to perform double sided laser heating. Double sided temperature measurements are accomplished through standard black body radiation measurements via an Acton Spectrometer (SP2356) equipped with a PI PIXIS 256E. The optical components for the system are located on an optical table that will also carry a sample stack, consisting of two horizontal translation systems, a vertical translation and a rotation (ω). The first translation system is located below the rotation and will have an accuracy/bidirectional reproducibility of less than 1 μm . The stages will be used to position the rotation centre to the focal spot of the x-ray beam. The second translation system will be located above the rotation, with accuracies/bidirectional reproducibility in the submicron range, as will the vertical translation. Latter will allow the position of the sample to the centre of the rotation and hence the x-ray beam. The rotation stage finally should have an eccentricity of less than 1 μm for a 360° rotation. Area detectors for the laser heating experiment will be located perpendicular to the beam on a translation system that allows adjustment of the sample detector distance from 200-1300 mm. Horizontal displacement of the detector system of +/- 1 m provides access to large portion of reciprocal space. For a detector we envision a MAR345 image plate or an amorphous silicon flat-panel detector [15] with a high frame rate of 8 or 30 Hz in order to be able to conduct time resolved experiments.

3. Possibilities to conduct time resolved experiments

Initial calculations using equation (1) described in [16] indicate that high z polycrystalline materials should yield enough counts to conduct sub second diffraction experiments. For example, the (111) reflection of Niobium (BCC-type structure) with a 20-30 μm thickness and 4 μm^2 cross section in the DAC should result in 957 photons/sec on a pixel (200 μm) of an amorphous silicon detector [15]. In the calculation we assumed an x-ray energy of 30 keV and an incident flux taken from the ray tracing calculations in SHADOW, corrected by a factor of fifteen to account for absorption of the diamonds and sample. Considering that the signal needs to be higher than 40 photons to be separated from the background one should be able to collect 4-5 diffraction patterns in one second.

Based on these predictions, we are planning to install a fast shutter that will allow us to administer sub second exposures as well as a chopper to produce repeated single bunch exposures [17] that could maximize the flux while minimize the background during the measurement. In conjunction with new experimental developments such as the dynamic [3] and pulsed laser heating DAC [4], the new Extreme Conditions Beamline will provide the necessary x-ray diffraction tool to start exploring the time resolved regime at extreme conditions.

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