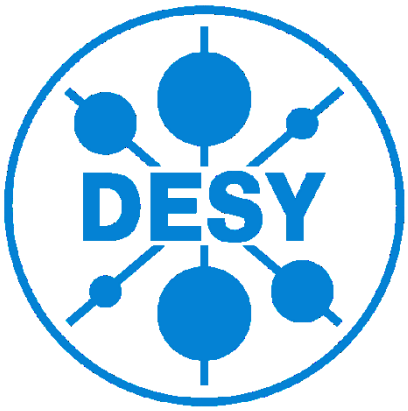

In-situ X-ray diffraction on fast compressed iron at extreme condition beamline P02.2 at Petra III

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Abstract

This report describes work that has been done under the supervision of Zuzana Konopkova during the Summer Students Programme 2013 at DESY. Our study was aimed on experiments with iron under high pressure at extreme condition beamline P02.2, which also include preparation of diamond anvil cells as well as samples loading. Fast compression was realized with compression rate 20, 40, 60 bar/min up to pressure 40 GPa in membrane. Phase transformation of iron from bcc to hcp took place at a pressure of 13 GPa. There was no relaxation from stress in samples during the relaxation on constant pressure. Decompression was not evaluated in this study.

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1 Introduction

Iron is the most common element (by mass) forming the planet Earth as a whole, forming much of Earth's outer and inner core. The pressure in Earth's inner core is from range about 330 to 360 GPa. The idea of high-pressure experiments is to simulate conditions in a laboratory as closely as possible to the conditions of planetary interiors. In DAC it is possible to achieve the pressure almost as in the Earth's core [1].

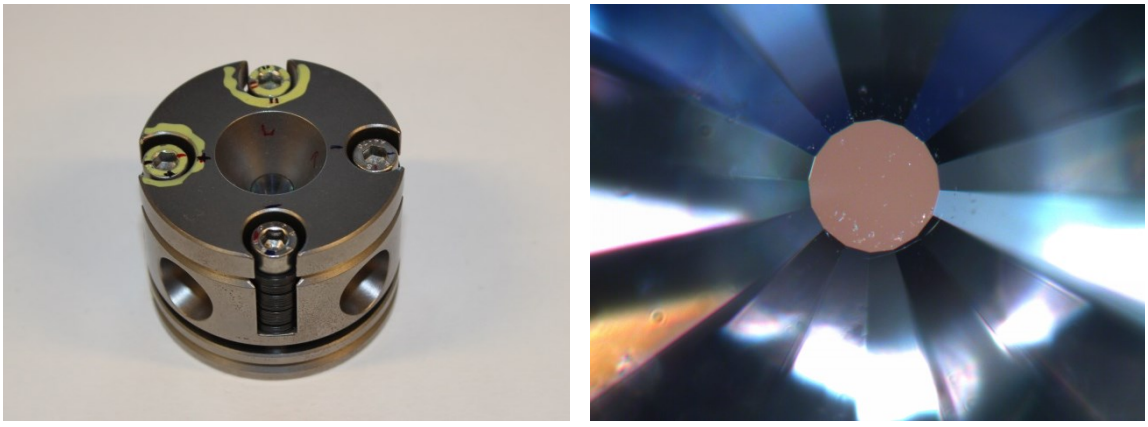
The following chapters describe diamond anvil cell and its preparation. Chapter 2 deals with a description of the beamline and chapter 3 focuses on the experimental part.

2 Diamond anvil cell

Diamond anvil cell (DAC) is the most versatile tool for static high-pressure research. When coupled with infrared lasers, pressures over 200 GPa and temperatures of thousands of kelvins can be reached in a controllable way. In planetary science, these conditions correspond to the Earth's outer core. Using DAC can be created condition with quasi-hydrostatic pressure. Various measurements can be performed in such experiments thanks to the unique physical properties of the diamonds. They are transparent over the visible, ultraviolet, infrared and X-ray spectral range [2].

2.1 Preparation of DAC

The DAC designs used in these experiments is symmetric piston-cylinder diamond cell (Fig. 1.). The core of the DAC consists of two diamonds, tips of which generate high pressures. Diamond is placed on the seat with bigger area facing down. Both parts are connected and aligned using lateral screws. Base of the diamonds is then glued to the seats. One of these seats is made from tungsten carbide and the second one is made from boron nitride material. Boron nitride is transparent to X-rays; hence it is used on the detector side so that the seat does not limit the diffraction cone. These seats are tightened with little screws to the rest of the cell body. Culets of the diamonds must be precisely aligned because any inaccuracy can cause diamond fractures. The alignment is achieved by tightening one of the seats until the diamond culets perfectly coincide. After alignment a stainless-steel gasket is pre-indented couple of times until the alignment holds firm.



a) b)

Fig. 1 (a) Symmetric piston-cylinder diamond cell, (b) Working face (culet) of the diamond

The rhenium gasket is positioned between the diamonds and compressed to a pressure corresponding to about half of the target pressure. A ruby chip is placed between diamond tip and gasket to measure the pressure. After the indentation, diamond tips leave imprints in the gasket from both sides. Thickness of the gasket at this point should not exceed 50 microns. Gasket provides support for the anvils and it contains the sample. Commonly used materials for gasket are rhenium, tungsten and beryllium. Next, a hole is drilled inside the indentation that serves as pressure chamber for the sample. For this purpose an electro-erosion machine or infrared laser is used. Diameter of the hole should be about half of the indentation hole diameter. The gasket is then cleaned in an ultrasonic cleaner and it is placed back on the diamond in the same position as during the indentation.

2.2 Loading samples

The samples (pure iron in form of powder and foil) were loaded with and without a pressure medium. In non-hydrostatic experiments (without the pressure medium), the powdered iron was compressed and added in separate layers. The second sample (Fig. 2.) that was loaded for laser-heating experiment, was an iron foil placed between 2 insulating layers of NaCl. The insulating layers of NaCl are necessary due to extraordinary high thermal conductivity of the diamonds. This causes that the heating is not effective and the heat is evacuated from the pressure chamber. At the same time, NaCl serves as pressure transmitting medium, creating quasi-hydrostatic conditions at high pressures [2].

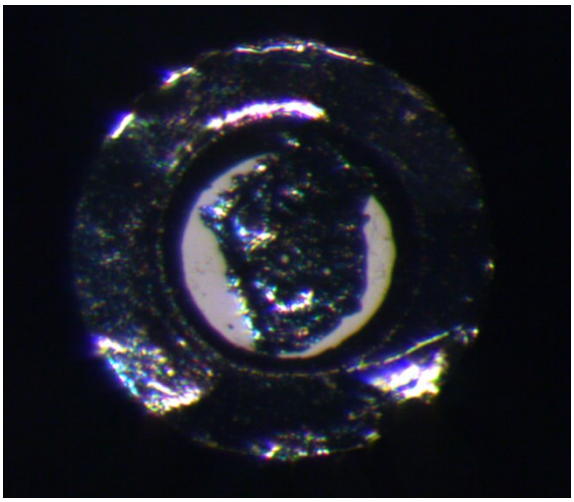


Fig. 2 Loaded sample for leaser heating

3 Description of beamline

The Extreme Conditions Beamline, P02.2, is located in sector 2 of the new 3rd generation synchrotron PETRA III at DESY. This beamline is focused on extreme conditions (high pressure and high temperature). 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.

The beam that is passing through the (111) diamond Laue crystal is used to provides photons for Beamline P02.2. The beam monochromatization is carried out using double crystal monochromator Si (111) and Si (311). This beamline operate with fixed energy 25.7 and 42.9 keV [DB]. The beamline utilizes two separate systems for focusing, a Kirkpatrick-Baez (KB) mirror system and a Compound Refractive Lens (CRL) changing system. The endstation is equipped with detector in forward geometry (PerkinElmer, MAR345IP). Using the flat panel detector XRD1621 from PerkinElmer enables collection of 2D diffraction data with a sub-second repetition rate (max. 15 frames per second). Size of the focused beam is less than 1.8 (H) x 1.6 (V) μm^2 [3]. This setup is schematically shown at Fig. 3.

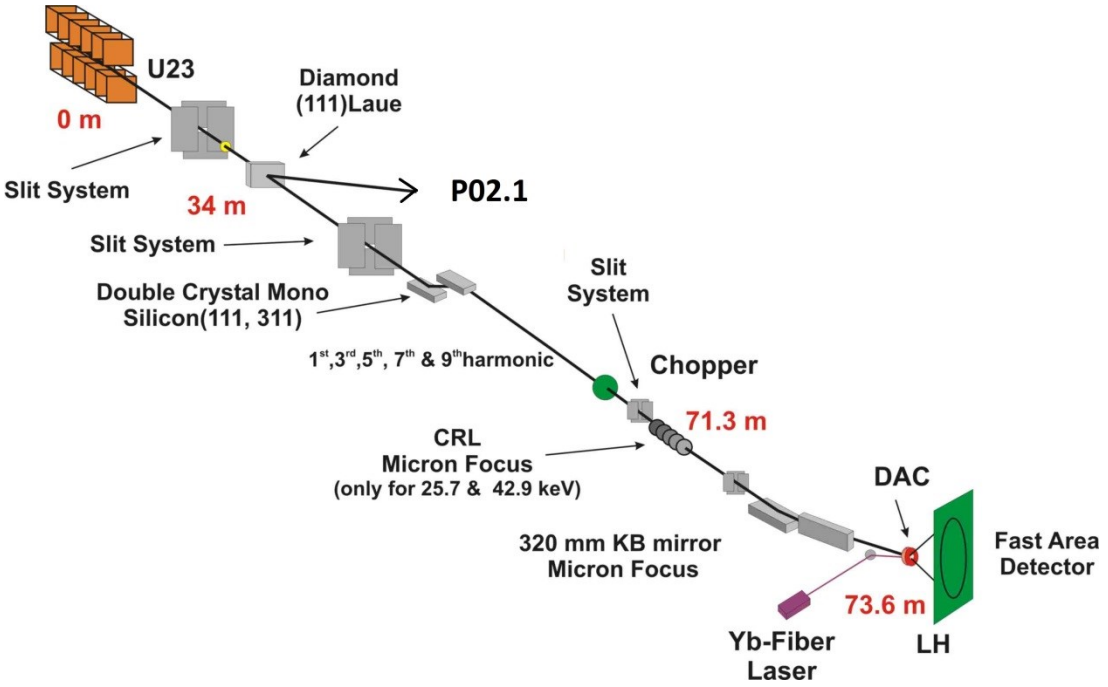


Fig. 3 Optical train for the Extreme Condition Beamline [4]

Present applications are [5]:

- Single crystal diffraction
- Angle-dispersive powder X-ray diffraction
- Membrane system for fine control of applied pressure
- Laser heating system & temperature measurements by spectroradiometry
- Online ruby system for pressure determination
- Gas loading (Ar, He, N, Ne)

4 Experiment

4.1 Methodology of experiments

Diamond anvil cell was prepared by technique, which is described in chapter 2.1. The sample, which was loaded to DAC was iron powder with purity 99,9 %. DAC was inserted into the membrane housing, which enabled a controlled increase of pressure in the DAC.

Sample in mDAC was compressed from the ambient pressure up to pressure 40 bar with compression rate 20, 40, 60 bar/min. The diffraction patterns were taken continously with exposure time of 100 ms, until the pressure in the membrane reached the maximum. After fast compression has been complied with constant pressure, this proces is called relaxation. The sample was relaxing for 3 hours and diffraction patterns were taken every 5 minutes. Then fast decompression took place down to ambient pressure with decompression rate 20, 40, 60 bar/min.

The scheme of the experimental setup, which was used during our experiments is shown at fig. 4. The focused beam (shown in blue in the figure) was going through the X-ray pinhole with size 2 x 2 μm^2 and with energy 42.7 keV. After interaction with the sample, the beam was diffracted and captured by an area detector. The direct beam was absorbed by a beam stop.

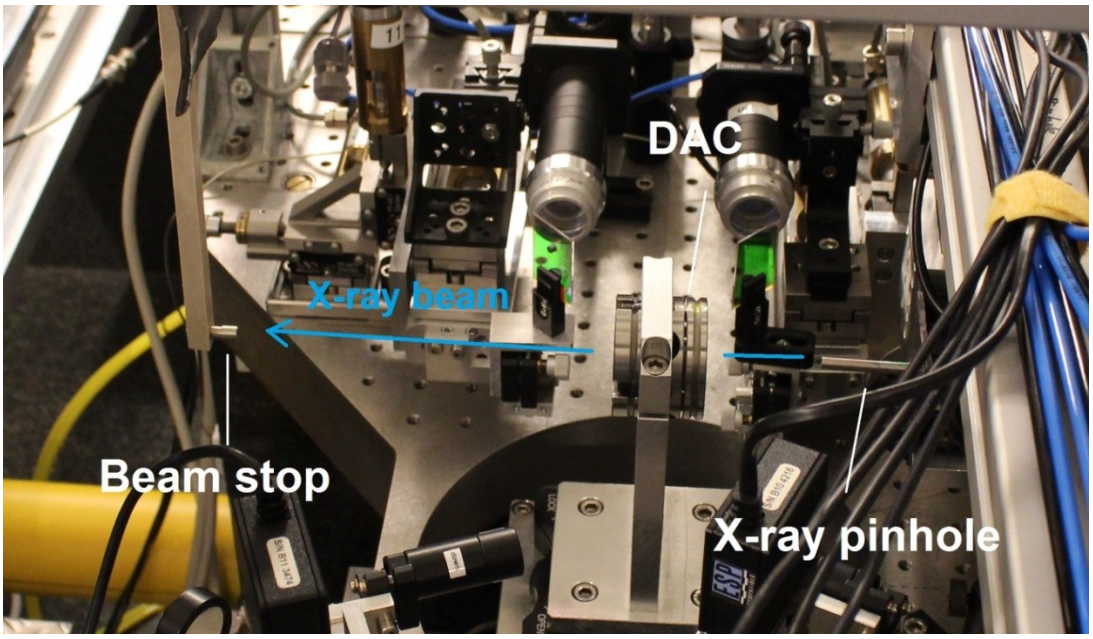


Fig. 4 Experimental setup

4.2 Results

Fig. 5 shows the change of diffraction angle 2θ with increasing pressure p (number of file) up to 40 GPa with compression rate 60 bar/min in membrane. As can be seen in the picture, phase transformation took place with increasing pressure. The structure of iron was changed from body centered cubic to hexagonal close packed. The pressure, in which transformation occured was 13 GPa, which is in line with value in the phase diagram of iron.

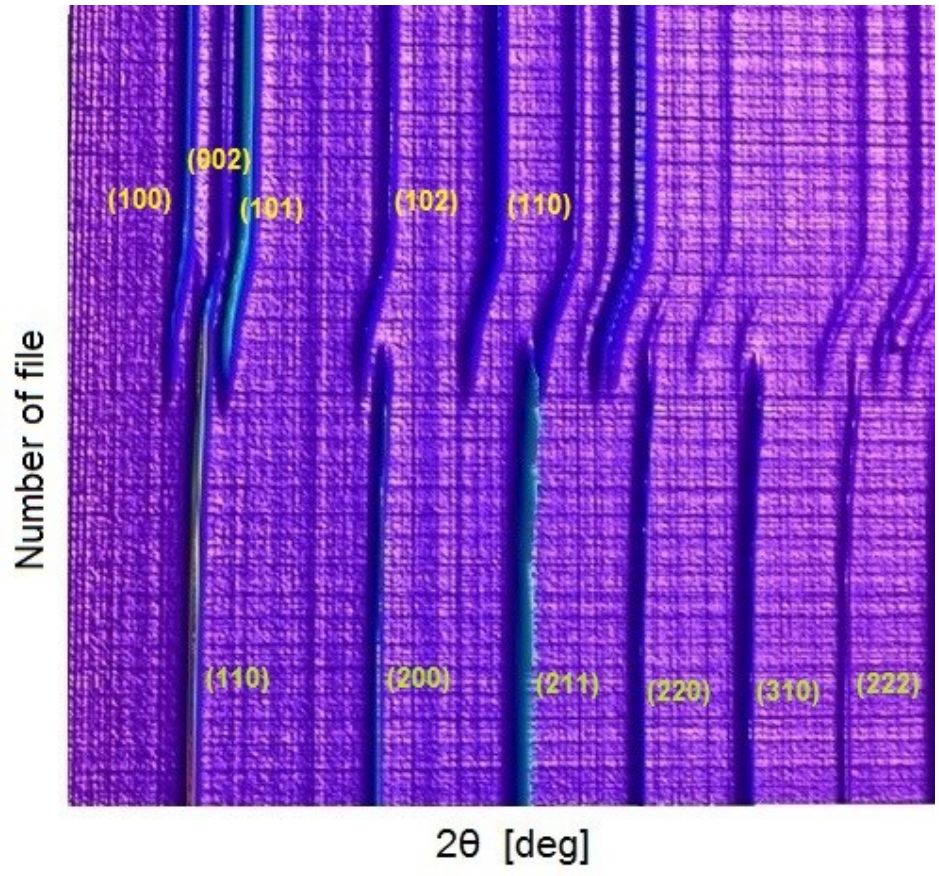


Fig. 5 Diffraction pattern evolution for non-hydrostatic conditions

In the following pictures is shown the relationship between FWHM and time during relaxation at pressure 40 GPa after compression, using the compression rate 60 bar/min (Fig. 6) and compression rate 40 bar/min. (Fig. 7). In these pictures can not be seen a change of FWHM with increasing time of relaxation, which means that there was no relaxation from the stress inside the samples. Values for peak number 7 exhibit a big mistake, because the peak was very small and we encountered fitting problems.

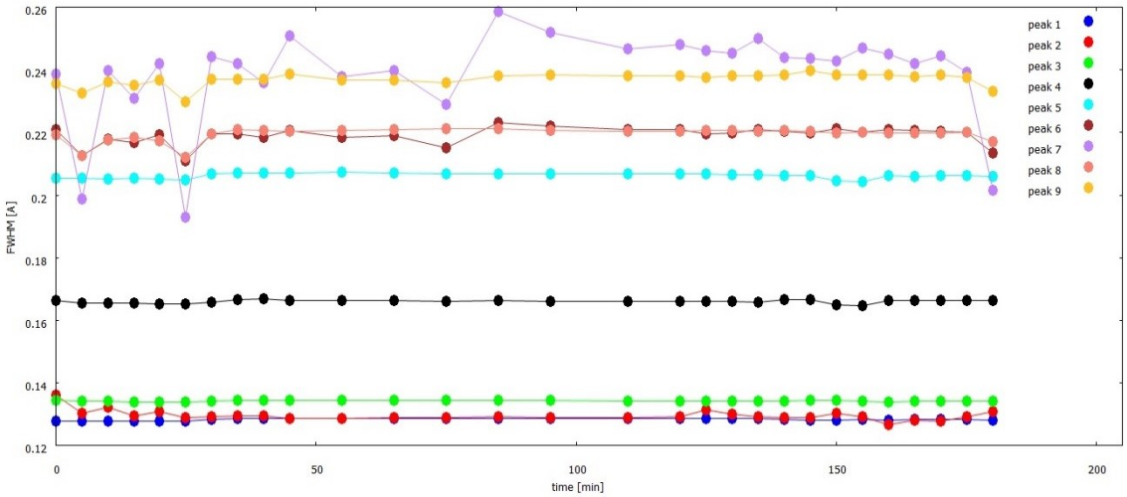


Fig. 6 Relationship between FWHM and time during relaxation after fast compression with compression rate 60 bar/min

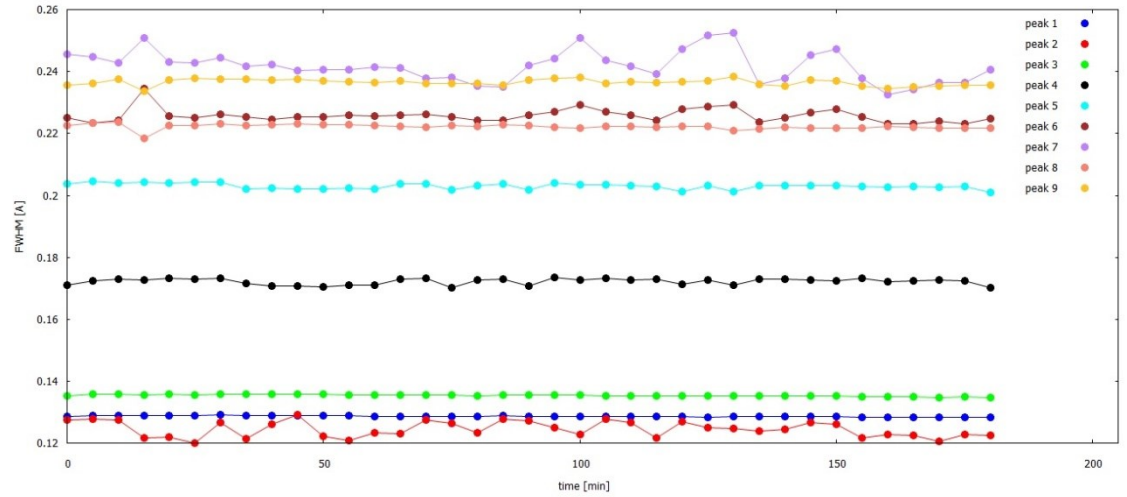


Fig. 7 Relationship between FWHM and time during relaxation after fast compression with compression rate 40 bar/min

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