HARWI-II: A New High Pressure Beamline Equipped with a Large-Volume Press, MAX200x

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For material scientists, chemists and physicists, the in-situ X-ray diffraction experiments under extreme pressure and temperature conditions at synchrotron beamlines become more and more important. These methods are especially important for geoscientists, as mineral interactions within the earth mantle, phase transitions, as well as fluid - rock interaction in this area strongly influence and control the dynamic motions within our whole planet. The conditions of around 25 GPa and 2000 K are required to simulate these processes in the laboratory. The new MAX200x diffraction press at DESY-HASYLAB will be one of the best available apparatus in the world to figure out these challenging experiments.

1. Introduction

Twenty years ago geoscientists from all over the world launched in-situ X-ray diffraction experiments under extreme pressure and temperature conditions at synchrotron beamlines. One of the first apparatus was the MAX80, a single-stage multi-anvil diffraction press, installed at HASYLAB. MAX80 allows in-situ diffraction studies in conjunction with the simultaneous measurement of elastic properties up to 120000 bar and 1600 K. This very successful experiment, unique in Europe, is operated by GFZ Potsdam and is used by more than twenty groups from different countries every year. Experiments for both, applied and basic research are conducted, ranging from life-sciences, chemistry, physics, over material sciences to geosciences. Today new materials and the availability of high brilliant synchrotron sources permit to construct double-stage multi-anvil systems for X-ray diffraction to reach much higher pressures. The newly designed high-flux hard wiggler (HARWI-II) beamline is an ideal X-ray source for this kind of experiments.

2. Research Subject

As only the uppermost few kilometres of the Earth (less than 0.1% of its radius) are accessible for direct observations (*e.g.*, deep drilling), whereas sophisticated techniques are required to observe and to understand the processes in the deep interior of our planet. In-situ studies are an excellent tool to investigate ongoing geodynamic processes within the laboratory. One of the fundamental regions to study geodynamic processes seems to be the so-called transition zone, the boundary between upper and lower Earth‘s mantle, *i.e.* from ≈ 410 to ≈ 670 km depth. Mineral reactions, phase transitions, as well as fluid rock interaction in this area strongly influence and control the dynamic motions within our whole planet. Around 250000 bar and 2000 K are required to simulate these processes in the laboratory. The new MAX200x diffraction press (Fig. 1) will be an excellent tool for these ambitious experiments.

3. MAX200x: Press Tool

The MAX200x is operated in the double-stage compression mode, in which the first stage is the DIA-type apparatus with six anvils (Fig. 2) and the second stage consists of eight anvils (Fig. 3). One corner of each of the eight cubes is stunted orthogonally to the spatial diagonal. The octahedral sample space is compressed by the truncated comer of eight inner cubic anvils, which, in turn, is compressed by the outer first stage six anvils. Either tungsten carbide or cubic boron nitride is used for the second stage anvils. This system has a capability of generating pressures up to 25 GPa and temperatures of 2000 K by a resistance furnace.

4. Detector System

The high purity germanium detector is covered in a 6 mm lead capsule to shield scattered radiation. The detector is cooled by a Kleemenko cycle cooler equipped with a compressor for a refrigerant (a special gas mixture). It is electrically powered and holds the temperature between 85 and 105 K. In front of the detector, there are two motor driven slit-systems (Fig. 4) for focussing the system at the sample. The distance between sample and detector is 2500 mm, so the angle is remindable up to 0.001°.

Figure 1. MAX200x surrounded by aluminium-lead aluminium hutch at the HARWI-II hall. The synchrotron beam position in the HARWI-II – Hall is only 1200 mm above the ground. Thus the multi-anvil press had to be put into a cavity of 3700 mm length, 3220 mm width and 140 mm depth. A 500 mm thick foundation of concrete had to be poured in order to support the weight of 30 tons. This press has a maximal load of 1750 tonnes. It is rotatable +/- 15°, moveable +/- 125 mm in *z*-direction and +/- 50 mm in *x*-direction.

5. Goniometer

The goniometer is adjusted to the centre of the press and can rotate from 0° up to 15° in steps of 0.01° (Fig. 4b). It is assembled on a stepper-motor driven table which can be displaced by 150 mm in *z*-direction, 50 mm in *x*-direction and 50 mm in *y*-direction in steps of 0.01 mm (Fig. 5) and rotated around the three orthogonal axes.

6. X-Radiography and Ionization Chamber

For exact sample length measurements as well as deformation experiments and viscosity studies under in-situ conditions, the X-radiography (Fig. 6) is indispensable. This is done by converting the X-ray shadow graphs and decoupling the optical images. The optical system consists of the macroscope and the 1.4 MegaPixels black and white CCD-camera. To measure X-ray absorption as a tool for *in-situ* density measurements of melts, a ionization chamber is available.

7. First experiments

The pressure-transmitting medium was a 10 mm long octahedron prepared from a mixture of amorphous boron, epoxy resin and hardener. The octahedron was filled with fine NaCl powder serving as internal pressure standard. Second setup was a MgO-octahedron. The pressure was determined from the dimension of the unit cell of NaCl using Decker's equation of state [1] and of MgO using Jamieson *et al.* data [2] respectively. Diffraction patterns were recorded in an energy-dispersive mode using white synchrotron X-rays from the storage ring DORIS III. The ring operated at 4.5 GeV and a positron current of 80 - 150 mA [3]. The incident beam has a diameter of 0.7 mm because of radiation security reasons and will be reduced to 100 µm ×100 µm by a motor driven slit system. Spectra were recorded by a germanium solid-state detector with a slits-system and a multi-channel analyser. The resolution of the solid state detector is 153 eV at 5.9 keV resulting in a resolution of diffraction patterns of $\Delta d/d \approx 1\%$. Figures 7 and 8 show the results of the first calibration high-pressure experiments of MAX200x.

Figure 2. DIA-type apparatus with six anvils, four side anvils are shown.

Figure 3. Four tungsten carbide anvils: the lower part mounted with a sample in external octahedral anvils.

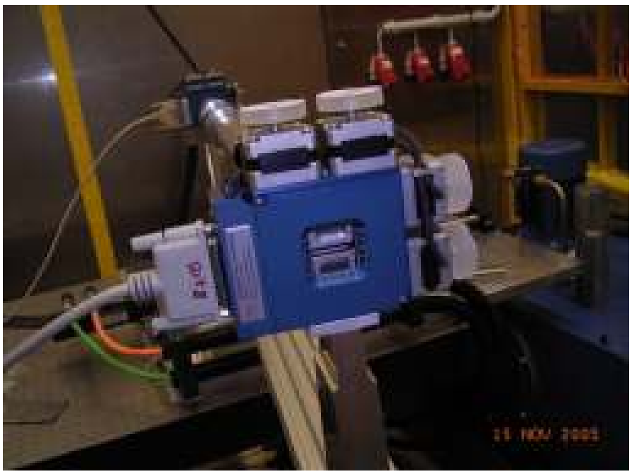

Figure 3. Slit-system with motor driven high precision slits in a distance of 1000 mm from each other to block scattered radiation and diffracted X-rays from non-sampled parts of the set-up. The distance between first slit-system and centre of the sample is 1200 mm.

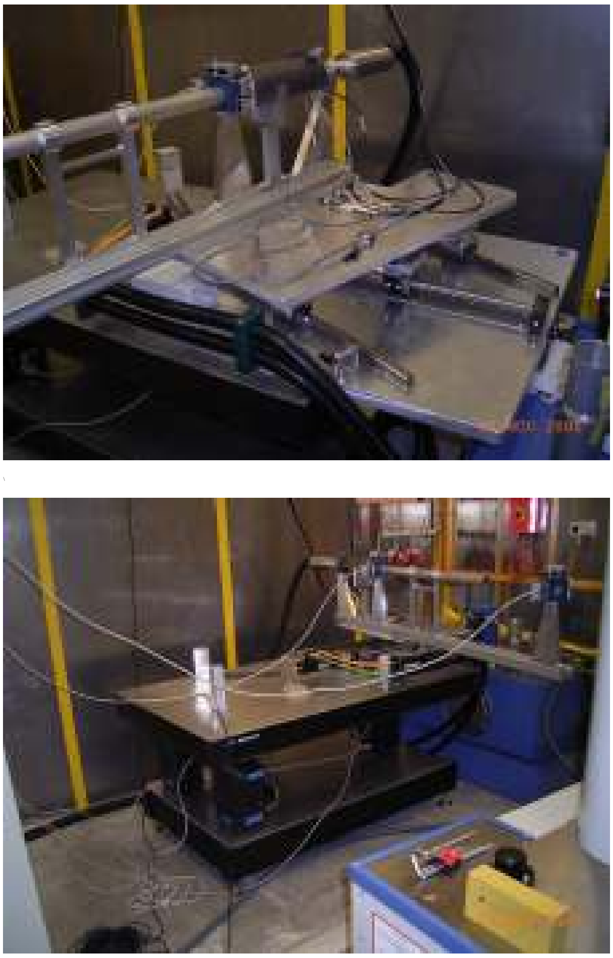

Figure 4. (a) (upper photo) The goniometer in position 10° to the beam. The detector is covered with 6 mm lead to exclude scattered radiation from outside the sample. (b) (lower photo) The table is equipped with six stepper motor and makes feasible a high precision alignment of the detector.

Figure 6. X-Radiography: the Ce:YAG-crystal converts an X-ray shadowgraph to an optical image. The image is magnified by the macroscope, captured by a CCD-camera and finally saved on PC's hard drive. For transmission, Fire-Wire controllers and four repeaters are used. Because of the long cables the images are saved at a PC's hard drive outside the hutch.

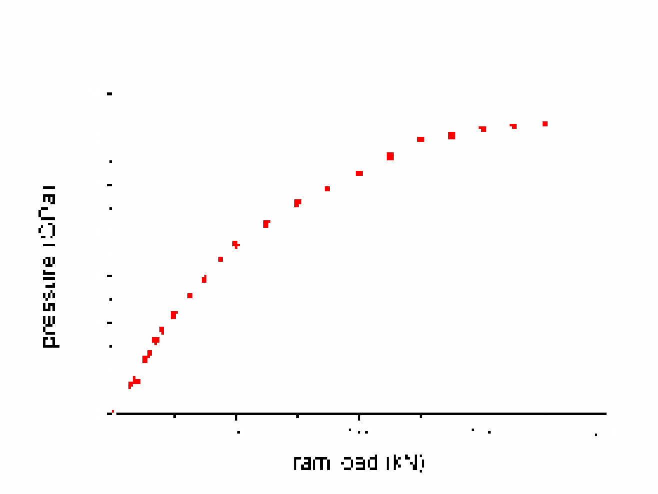

Figure 7 Pressure calibrations using the equation of state for NaCl [1].

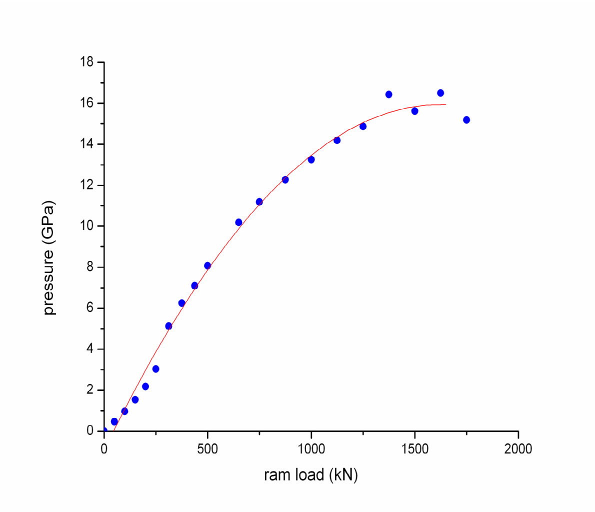

Figure 8. Pressure calibrations using the equation of state for MgO [2].

References

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