Installation and commissioning the monochromator for studies of liquid interface at the high energy beamline

DESY Summer Student Programme 2010

students:

Oleksiy Troshyn[1]

Marlena Kochel[2]

supervisor:

Dr. Martin von Zimmermann

[1]Ukraine, Lomonosov Moscow State University

[2]Poland, Wroclaw University of Technology

Hamburg, 9th September 2010

Table of contest

1. Introduction

During the DESY summer student programme our task has been performed in HASYLAB in Hard X-ray Scattering group. It consisted of setting up the monochromator for studies of liquid surfaces/interfaces in PETRA III at the high energy beamline P07. Molecular ordering at, for example, liquid-liquid interfaces is not well studied yet, owing to the difficulties in probing deeply buried interfaces at conventional X-ray energies [10]. Use of high energy X-rays and the tilt double crystal monochromator gives the unique opportunity to peep into buried interfaces under centimeters of matter.

2. PETRA III

PETRA III (Fig.1) is one of accelerators at DESY site in Hamburg, the new high-brilliance synchrotron radiation source. It is the accelerator of 3rd generation. To convert it into a brilliant radiation source, it was necessary to rebuild completely nearly 300 meters of the 2.3-kilometre long PETRA ring and to erect a new experimental hall. The plan calls for 14 experimental stations (Fig.2) with up to 30 instruments. Excellent experimental capabilities are ensured by the installation of undulators – long arrays of magnets that generate X-ray radiation of exceptionally high brilliance. In simple terms, this means that a very large number of photons are emitted from a very small area to form an extremely collimated beam of X-rays. As a result, PETRA III delivers a photon flux within an area of a single square millimeter.

Fig.1. PETRA III – view from the top

Parameters of PETRA III:

circumference: 2304 m
energy: 6 GeV
energy range of 50–150 keV.
emittance: 1 nmrad
emittance coup.: 1% (10 pmrad!)
current: 100 (200) mA
bunches 40 / 960
straight sections: 9
undulators: 14
undulator length: 2, 5, 10 (20) m

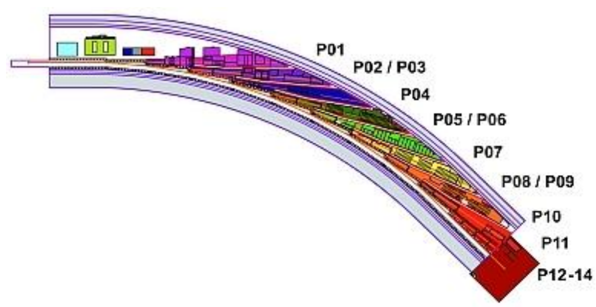


Fig.2. Schematic layout of the installations on the PETRA III experimental floor including the ring tunnel, the optics and experiment hutches as well as control cabins.

3. P07 beamline at PETRA III

P07 beamline at PETRA III is high energy beamline (60-150 keV) with little beam dimensions (0.7mm*0.3mm) and high momentum transfer (~30 1/Å). The experimental station consists of several principal parts (Fig.3). Incident beam goes through focusing optics, the diameter of the beam becomes 0.7mm*0.3mm mm. Passing through monochromator system beam interacts with the sample, posited on the sample tower. Scattered beam comes to detector.

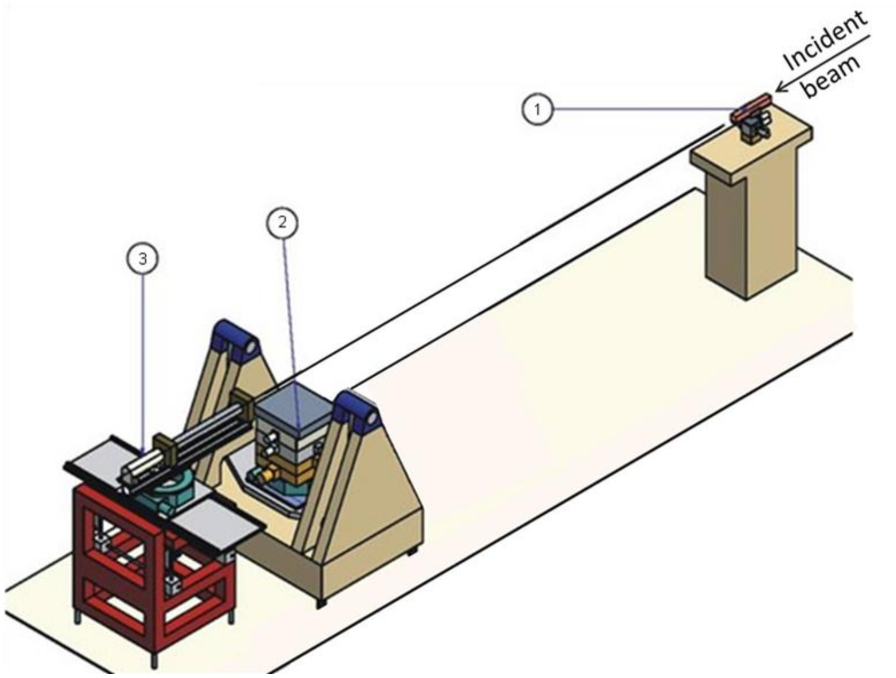


Fig.3. High Energy Micro Diffraction instrument (scheme) at P07 beamline at PETRA III, 1 - focusing optics, 2 - sample tower, 3 - detector tower

4. Motivation

Many natural and technologically important interfaces are hidden under exposed surfaces of materials. The investigation technique should be non-destructive, structural, in-situ probe with atomic-scale resolution. These demands are fulfilled with the help of high-energy x-rays. The basic principle relies on the deep penetration of high-energy x-rays so that only the one interface f interest is illuminated with x-rays, as opposed to standard x-ray reflectivity. (Fig. 4). This reduces the number of interfaces that contribute to the scattered signal and makes a wide variety of buried interfaces accessible for structural studies. Solid-solid, solid-liquid, and liquid-liquid interfaces can now be probed.

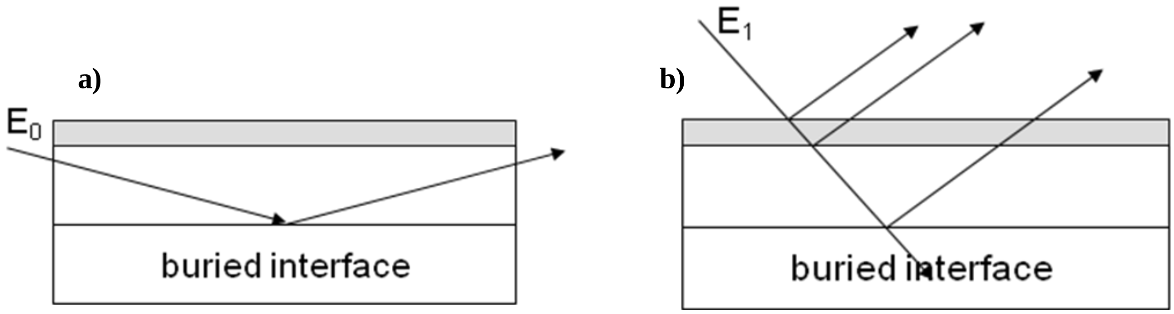


Fig.4. a) High-energy x-ray reflectivity; only one interface of interest is illuminated, **b)** Standard x-ray reflectivity; all interfaces contribute to the scattered signal

Buried interfaces require particularly specialized instrumentation to satisfy geometrical constraints. Since a liquid surface cannot be tilted with respect to a fixed incident beam, we have to tilt the primary beam with high precision with respect to the surface. It becomes possible, supplying conventional diffractometer with double crystal monochromator in special geometry. Thereby the task included installation and commissioning such monochromator for studies of liquid interfaces at P07 beamline (Fig.5).

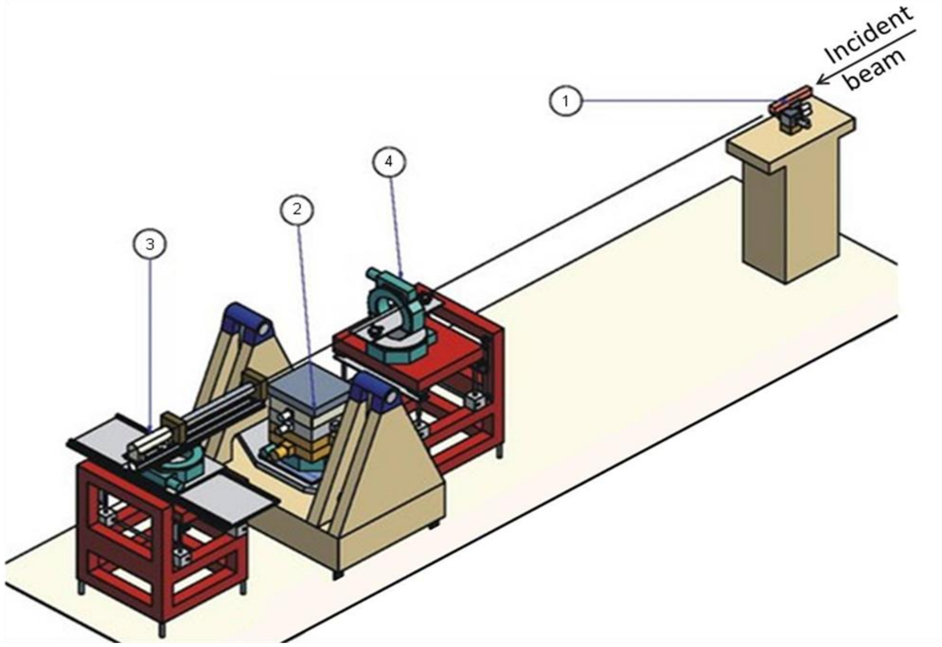


Fig.5. High Energy Micro Diffraction instrument with the tilt double crystal monochromator (4) (scheme) at P07 beamline at PETRA III, 1 - focusing optics, 2 - sample tower, 3 - detector tower, 4 - the tilt double crystal monochromator

5. Principle and design parameters

The principle of the tilt double crystal monochromator in geometry, acceptable for liquids, relies in the variation of vertical angle with respect to the surface without changing the sample position. (Fig.6). The monochromator crystal 1 (Si 111) is used to deflect the incident beam. Subsequently the beam is reflected by the crystal 2 (Si 220) such that double-reflected beam crosses the incident beam at the fixed sample position. In order to change the incident vertical angle to the surface, the two crystals and must be rotated in a coupled motion by the angle **φ**. And also detector should follow this rotation. The vertical angle **α** with respect to the sample surface and the angle **φ** between the reflection plane and the horizontal plane is then determined by

$$\sin\alpha = \sin\varphi \cdot \sin 2\Delta\theta$$

where $\Delta\theta$ is the difference between the Bragg angles of the two crystals [2]

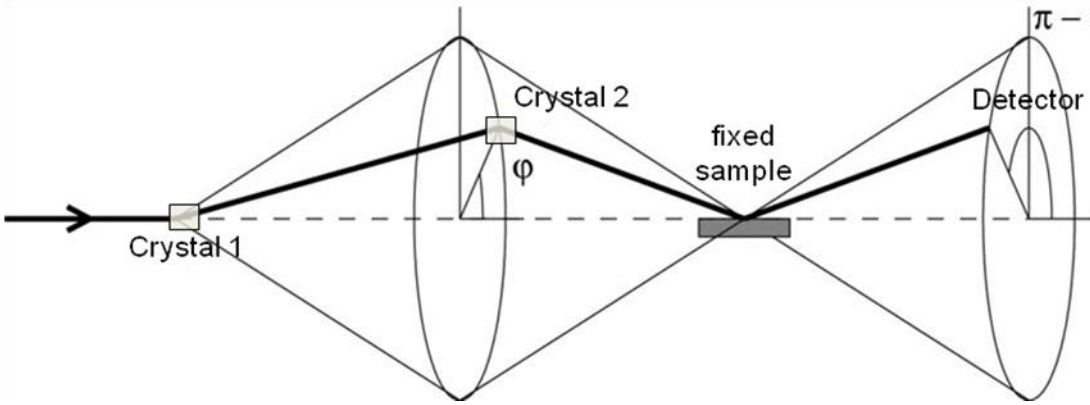


Fig.6. The incident beam is reflected by crystal 1 Si (111) and than by crystal 2 Si (220), crossing the incident beam in the sample position. The vertical angle of the incident beam on the sample changes, due to coupled rotation of the two crystals around the incident beam.

6. Thermal gradient crystals as tuneable monochromator

If the crystal is cooled on one side and heated on the other, the lattice spacing is changing from one side of the crystal, which causes a bending of lattice planes (Fig.7). Relaxated crystal will have a gradient proportional to the temperature difference. After cooling and heating reflectivity is increased up to a factor of 2 (crystal is not perfect but becomes distorted). We can achieved reflectivity of up to 100%[1]

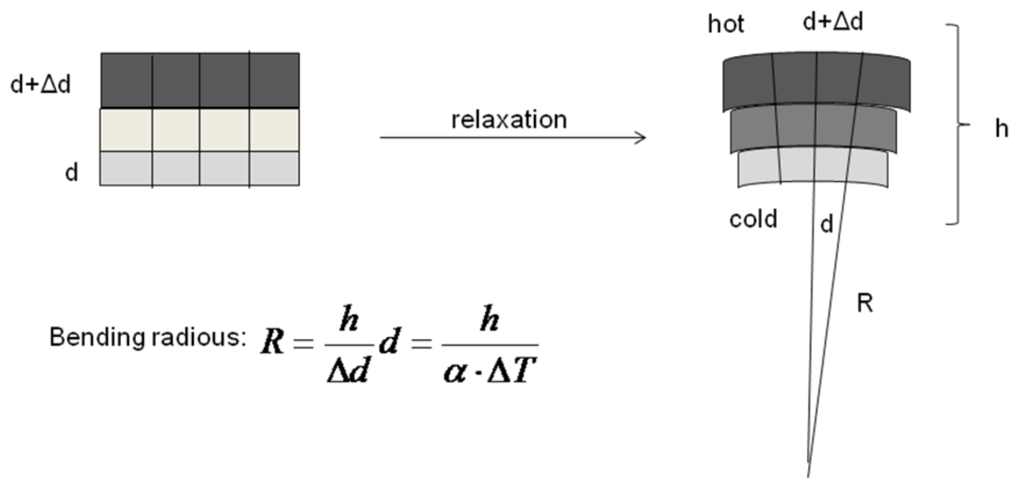


Fig.7. Schematic drawing of the bendig of the lattice sparing gradient crystal.The bending radius can be given by the difference in lattice spacing and thickness of crystal

The monochromator can be used at photon energies over 60 keV, thus the measurements are possible in PETRA III. For this energy monochromator in Laue geometry is usually used because of small Bragg angles (about 2^o) (Fig.8)

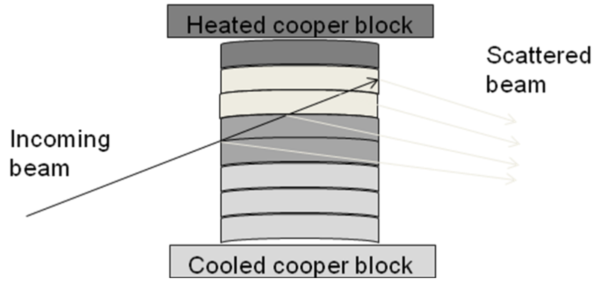


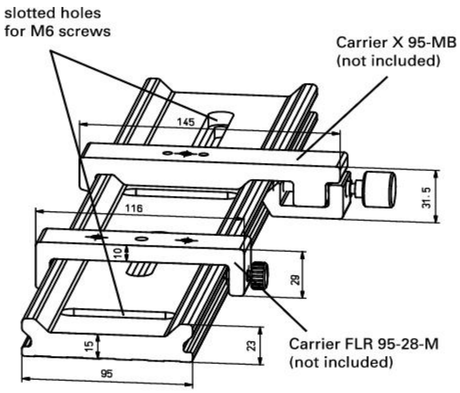
Fig.8. Scattering of an incident white beam on the bent crystal

7. Tasks and difficulties

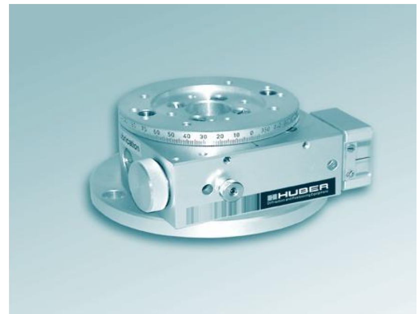
- Carry out the analysis of the devices which would satisfy our demands
- Ordering the devices
- Fitting the devices
- Assembling cooling and heating systems for the crystals
- We needed help of mechanical, electrical and chemical workshops
- People were on holidays

8. Experimental setup

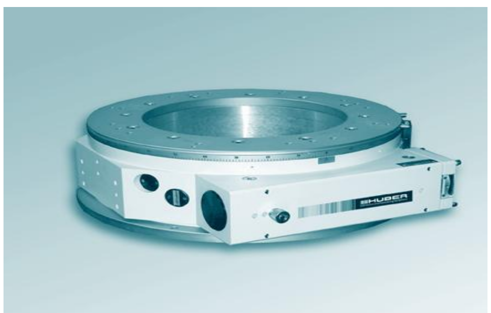
8.1. List of instruments:



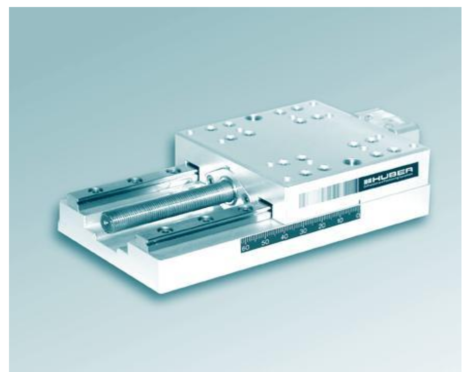
FLS 95 [4]



Huber 408 [3]



Huber 430 [3]



Huber 5101.1 [3]

8.2. The two crystals setup

The geometry of the tilt double crystal monochromator can be achieved by arrangement crystals on the top of two towers, consisted of translation linear stages, metal plates and 1-circle goniometers (Fig.9)

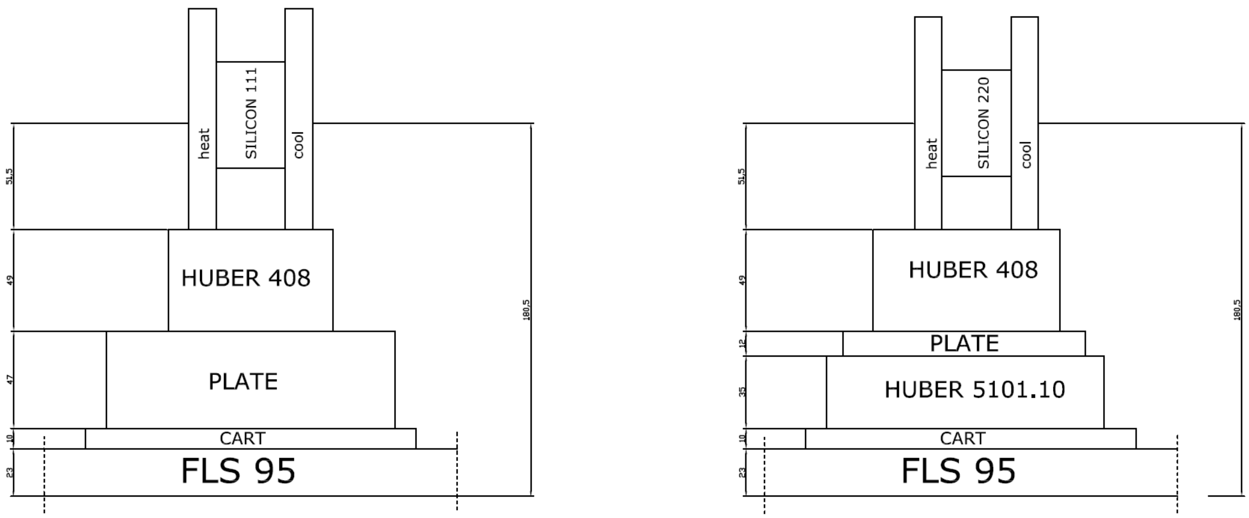


Fig.9. Dimentions of the two towers for crystals

Moreover, these two towers should have the same height and be fixed on the one cart FLS 95 for making them rotate in a coupled way., The construction was improved to fulfill both requirements (Fig.10)

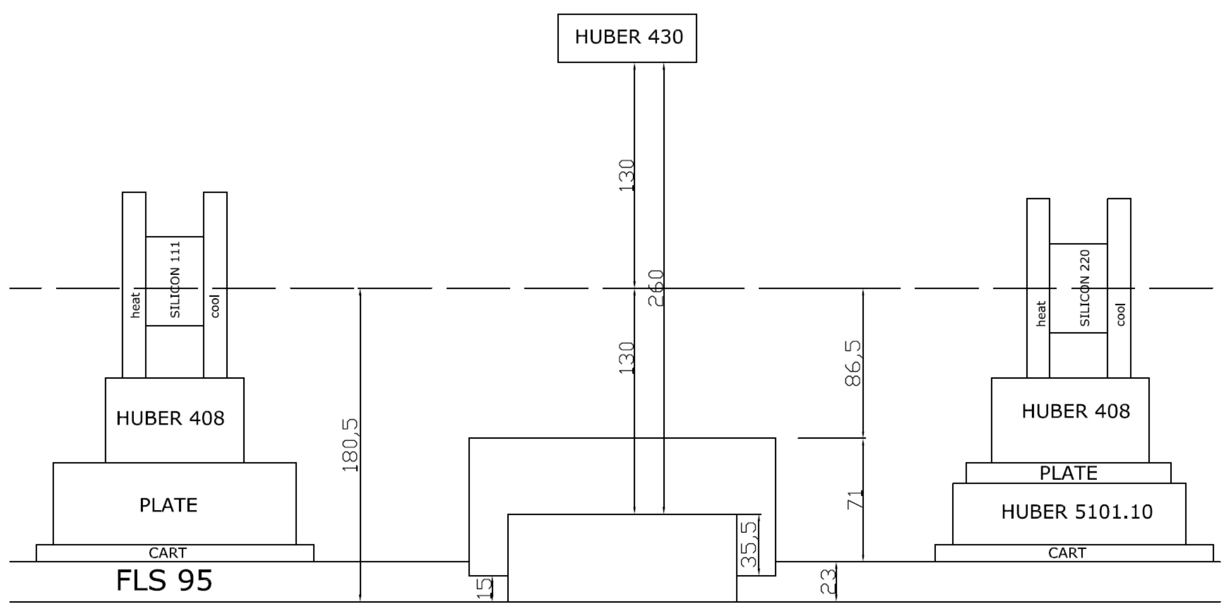


Fig.10. The two crystals setup (scheme). The height of the towers is the same, they are fixed on the one cart

In principle the biggest problem that appeared during our setting up was related with holes because individual elements did not fit. We kept in mind that beam have to pass through the canter of our setup (of course we can regulate this with translation table but just in limited way). As a result we designed an arrangement of holes that matched each other and with completed set of technical drawings we went to workshop. Examples of technical drawings are presented in Fig. 11.

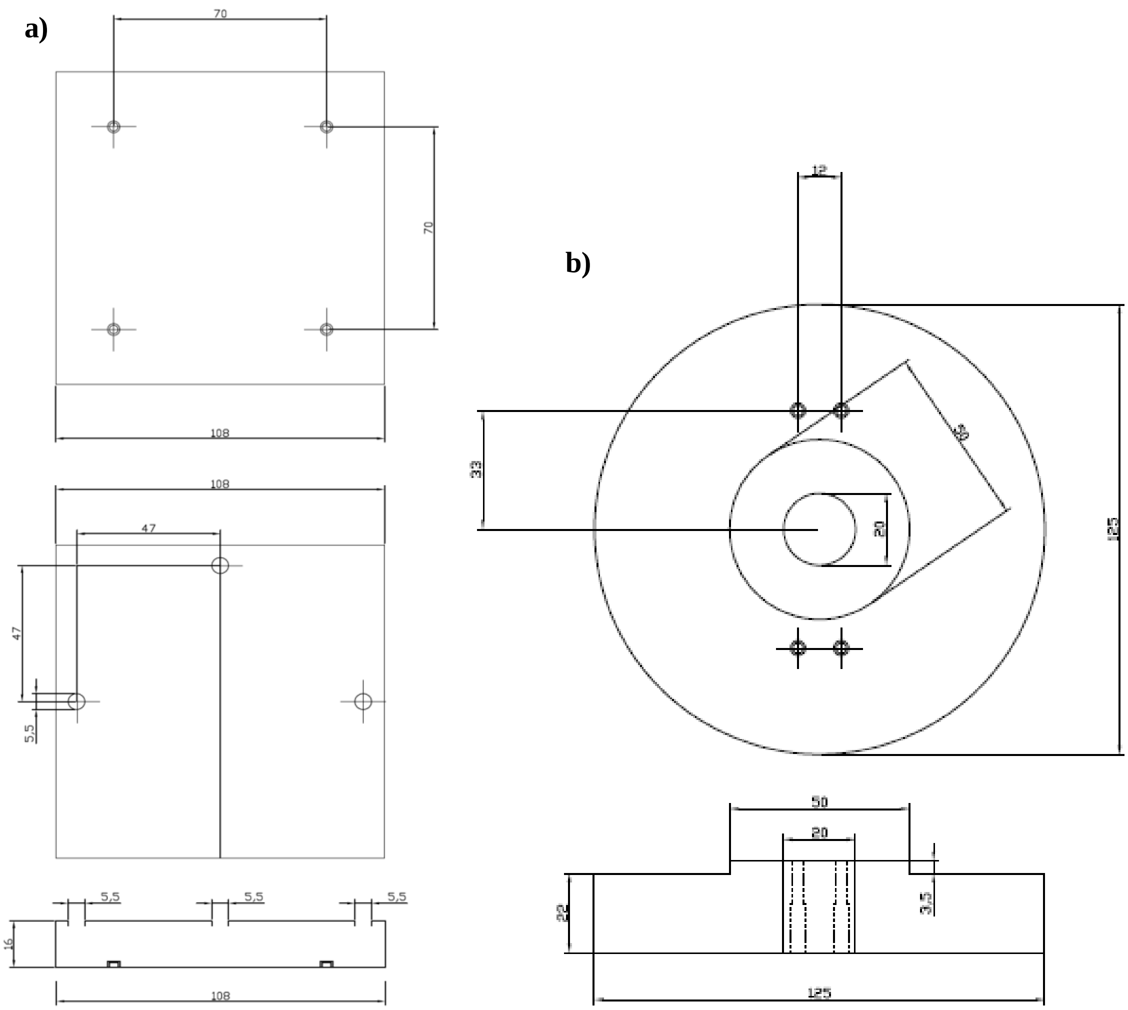


Fig. 11. a) technical drawing of the metal peace between 1-circle goniometer (huber 408) and translation linear stage (huber 5101.1); **b)** technical drawing of the circle stand of monochromator

The photo of the result setup is in Fig.12.

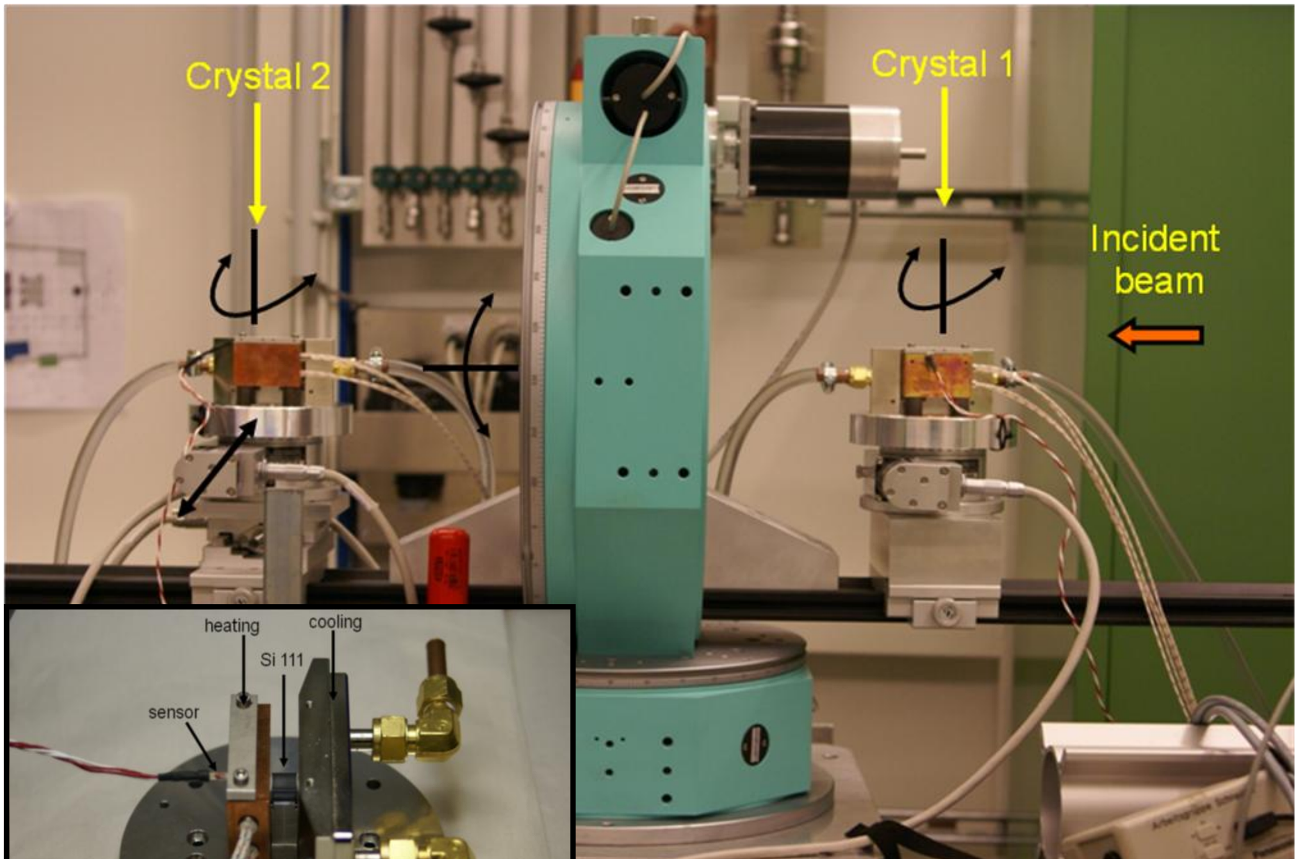


Fig.12. Both crystals are mounted on separate rotation stages along vertical axis (position of crystals can be changed) and crystal 2 can be moved by linear translation stage perpendicularly to the incident beam direction. Incident beam goes from right side to crystal 1 and reflected beam is going to crystal 2 and then to the sample. On the little pictured in the frame thermal gradient crystal Si (111) is shown. It is cooled on the one side (cooling block) and heated on the other. Water is chilled to 20ºC and the other side is heated (heating block) up to 100ºC. Temperature of heating block is read by sensor PT 100.

8.3. Principle of work of heating and cooling systems

During our project we were wondering how in general cooling/heating systems work. The temperature of both systems is monitored. We connected cooler and heater to the crystals. Schemes of the both systems are in Fig.13 and Fig.14. In the case of heating system, heater of the crystal is connected by sensor PT100 with temperature controller and with power supply. Temperature controller sends information to power supply up to which temperature the heater of the crystal should be heated. Sensor reads information from Heater of the crystal, so monitoring real crystal temperature.

In the cooling system cooler blocks in both monochromators are linked and connected together with cooler machine (Julabo F25 HE), where we set the temperature.

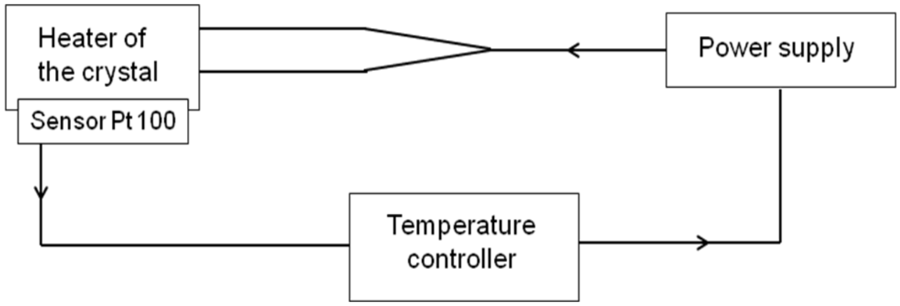


Fig.13 Heating system scheme

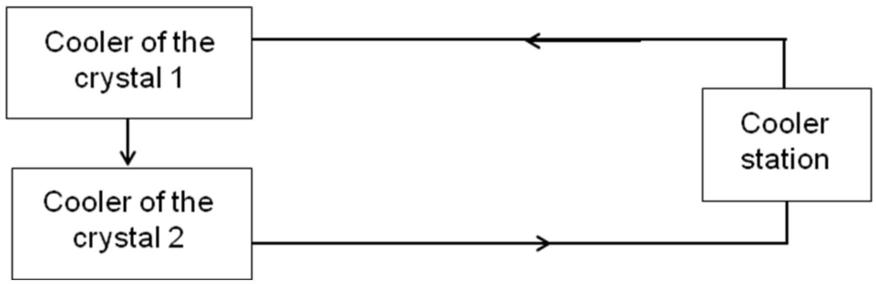


Fig.14 Cooling system scheme

9. Results and discussion

Finally setup is ready for conducting small angle x-ray scattering measurements on liquid-liquid, liquid-solid and solid-solid surfaces/interfaces (Fig.15)

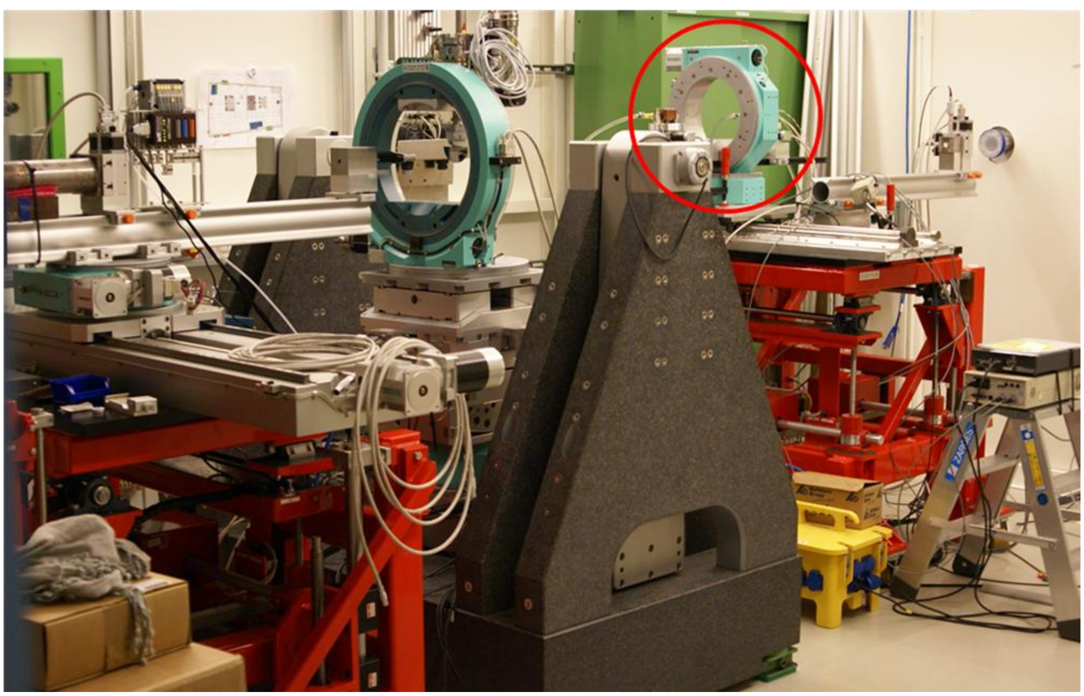


Fig. 15. High Energy Micro Diffraction instrument with the tilt double crystal monochromator (in red circle) at PETRA III at P07 beamline

10. Acknowledgments

We would like to thank DESY for opportunity to participate in Summer Student Programme 2010, our supervisor Dr. Martin von Zimmermann and Dr. Uta Rutt for successful cooperation. Also we thank to Rüdiger Nowak (beamline engineer), Jens Brehling and Tim Jonny Schroder (technical workshop), Markus Kowalski (precise workshop) and personal of electrical workshop for help in supplying us with advises, technical parts and components which were necessary for our work.

11. References

- [1]U.Rutt, H.Schulte-Schrepping,J.Heuer and M. v. Zimmermann, *Thermal gradient crystals as tuneablemonochromator for high energy X-rays*

- [2]V. Honkimaki, H. Reichert, J. S. Okasinskib and H. Dosch,*X-ray optics for liquid surface/interface spectrometers* (2006)

- [3]www.xhuber.de

- [4] www.linos.com