Summer Student Program 2007
July 25th , 2007 – September 18th, 2007

New approach of tracing a narrow synchrotron X-ray beam by installing a parallel visible laser

Pinit Kidkhunthod

Department of Physics, Faculty of Science
Khon Kaen University,
40002 Khon Kaen, THAILAND

Supervisor

Dr. Oliver H.Seeck

New approach of tracing a narrow synchrotron X-ray beam by installing a parallel visible laser

HASYLAB, Hamburg, 14th of September 2007

P. Kidkhunthod[1], F.Holzner[2]

[1] Khon Kaen University , Thailand
[2] University of Tübingen, Germany

1. Introduction

The new synchroton PETRA III at the DESY site in Hamburg is being under construction until the year 2008. For the design of one new beamline (beamline 8) we wanted to figure out, if there exists a better approach of tracing the X-ray beam than the conventional way. Typically the position of the X-ray beam in a synchrotron beamline is detected by several screens. The screens can tell a computer the exact position of the beam and a program can, if needed, correct the position of the beam very accurately using motors made of Piezo crystals. However, the screens will always absorb partially the intensity of the X-ray beam. Furthermore, for varying photon energies the absorption of one screen changes and might not be optimal.

That is why we tried to find out, if it is possible to determine the position of the X-ray beam without screens, but with a parallel laser beam. If it is possible to install a perfectly parallel laser beam, one can measure the position of the visible laser very easily and accordingly knows the position of the X-ray beam without disturbing it.

The new beamline 8 will be equipped with four Silicon crystals to monochromatize. The X-ray beam will therefore change its direction by diffraction at each of these crystals. The laser needs to be reflected in the same direction. However, the laser is reflected by the surface and the X-rays by a lattice plane of the crystal. The lattice plane is never

perfectly parallel to the surface. This always existing miscut angle is the reason why a separate mirror for the laser is needed.

We tried to find out how difficult it is to align a mirror parallel to a lattice plane very accurately.

First we calculated how exact we have to be in the alignment. These calculations are shown in the next chapter 2.

How we did the experimental trial at the beamline D4 at DORIS III is described in chapter 3.

2. Theoretical Calculation

2.1 Calculation of how small unwanted rotations of the crystals disturb the position of the X-ray beam

Because in this beamline there are four monochromators which are 2 Si(111) and 2 Si(311) or in another setup 2 Si(111) and 2 Si(511), the position and direction of the x-ray beam can be easily disturbed by changing the angles (both θ and χ, see Fig.2) of each monochromator. Thus, we will divide our calculation into two parts, θ and χ, employing EXCEL and the XTRACE raytracer.

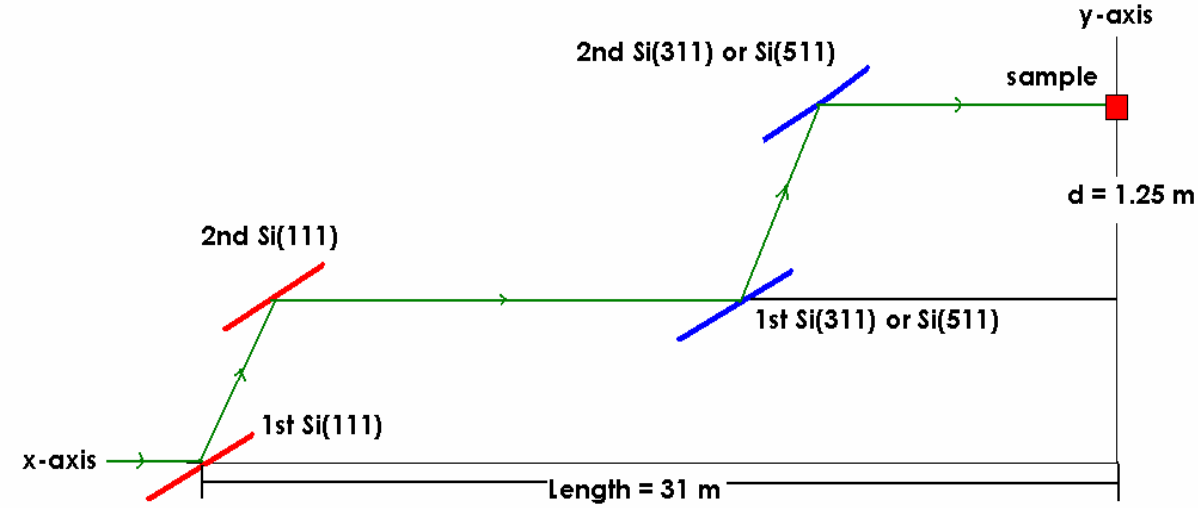

Figure 1: *a sketch of each monochromator position in this beamline*

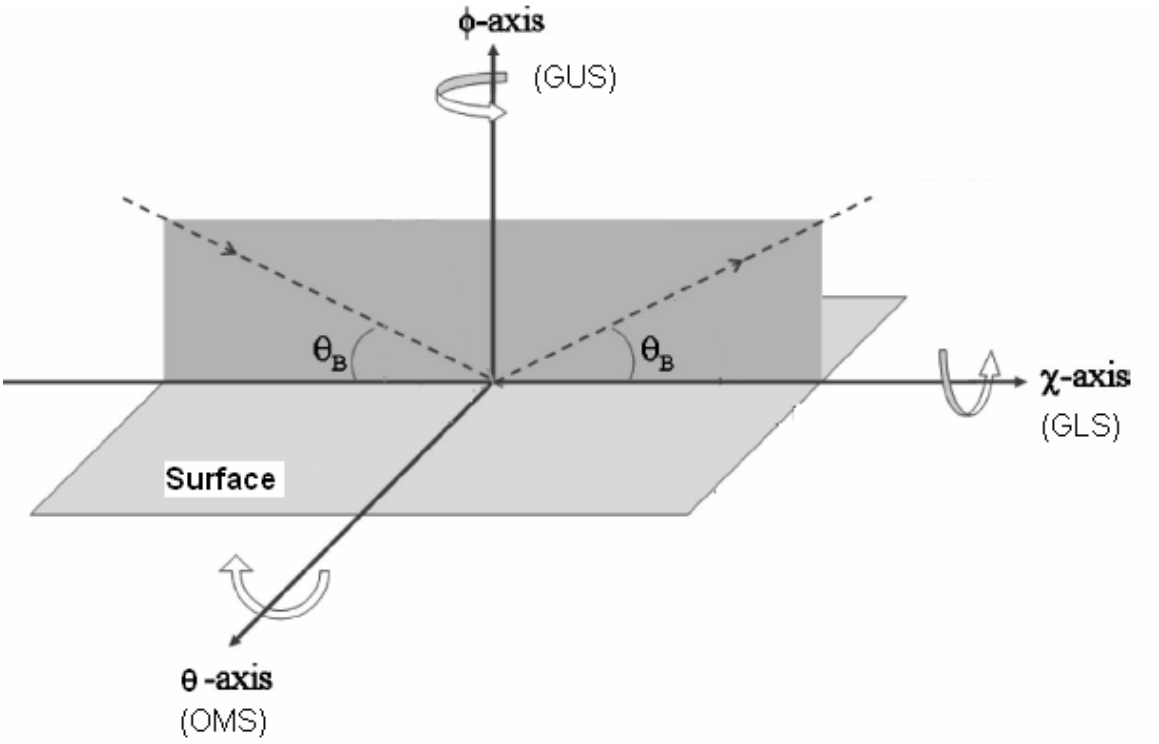

Figure 2: *Rotation axes (OMS, GLS, GUS are the names of the motors)*

For the first part, changing θ, each monochromator is considered separately by changing a small amount in θ in order to find the maximum angle that the beam will not hit at the sample over ±5 μm in y-direction. There are similar results for both the first crystal pair and the second one. The θ angles can be changed in only the order of 10^{-6} degree for Si(111) and Si(311) or Si(511) respectively.

Moving χ to change the x-position, the maximum calculated χ are only in the order of 10^{-5} degree for each monochromator. These are some examples from our calculation.

X-ray energy (keV)	***1st Si(111) (degree)***		***2nd Si(511) (degree)***	
	Delta θ_1	***Delta χ_1***	***Delta θ_4***	***Delta χ_4***
8.5	4.52908E-06	3.97473E-05	6.2367E-06	2.91669E-05
15.5	4.57380E-06	3.62015E-05	6.58578E-06	1.68084E-05

Table 1: *some examples from our calculation*

2.2 Angular Darwin width

To consider the Bragg reflectivity of the Si-crystal in a perfectly monochromatic beam as the incident angle (Bragg-angle) is varied, angular Darwin widths (ω_D^{total}) are determined from this equation

$$\omega_D^{total} = \zeta_D^{total} \tan\theta$$

where ζ_D^{total} is a material depending number which we calculated for Si and θ is the Bragg-angle [1,2]. Subsequently, the angular Darwin width varies with energy through the variation of tanθ. Moreover, from this calculation, as the Bragg-angle is reduced the angular Darwin width becomes smaller. The movement of the angles, corresponding to 5μm deviation at the sample, are well within the Darwin width.

2.3 Miscut Angles of Si-crystals

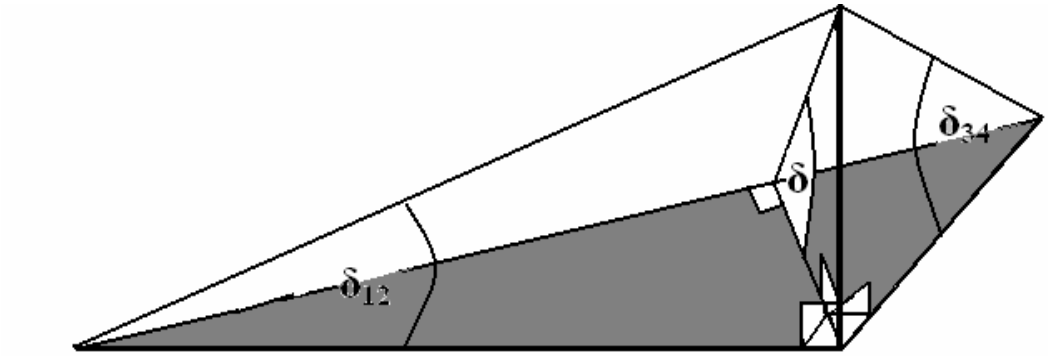

Figure 3: *schematic used to derive miscut angle* (δ) *in this work*

In this work the miscut angles (δ) of Si-crystals are needed to be figured out, thus the method of calculation for these angles are introduced in this part. Consider Fig.3, the miscut angle can be calculated from the following equation [3]:

$$\tan^2(\delta_{12}) + \tan^2(\delta_{34}) = \tan^2(\delta)$$

3. Experimental part

3.1 Description of Beamline D4 at DORIS III

Figure 4 shows the top and the side view of the beamline D4 at DORIS III. The beam enters the experimental hutch from the left through a slit with four separately moveable blades (see Fig 4). Next, in a lead shielded box a 200mm long Rhodium coated mirror reflects the beam downwards. Leaving the box the beam hits a fluorescent diamond screen. With a camera the visible light from the diamond, and thus the beam can be made visible. In the following, a monochromator can be chosen between four different crystals (Si(111), Si(220), Ge(111), Ge(220)). The diffracted beam goes on through the flight tube with the second slit at the end and passes a Kapton foil. A scintillation detector measures the intensity of the X-ray beam by counting the scattered photons from the Kapton foil. Then the beam hits the sample on the sample table. The following aperture system in front of the detector has a 4-blade slit at each end. The detector is a Cyberstar Scintillation counter [4, 5].

3.2 Beam Alignment

The beamline is used by many different users and has no responsible beamline scientist. Therefore, we had to align and calibrate nearly every single motor before we could start to make our measurements there. The made the alignment is in the following order:

1. Choose a beam size (4mm x 0.05mm) by changing position of the four blades of 1st slit
2. Determine the critical angle α_c of the mirror and move the mirror to a position where only the reflected beam leaves the box. ($\alpha_c = 0{,}14°$ for a photon energy of 18keV)
3. Make sure that the beam really hits the wanted Si(111) monochromator
4. Calibrate the Bragg angle (OMM) by scanning the K-absorption edge of a Zr-foil at various OMM. The K-edge of Zr lies at 17.998 keV which corresponds to a theoretical Bragg angle of 6.306°.
5. Determine the rotation axis of the sample. At different rotated positions of the table view the position of a pin on the sample table with binoculars. Move the table that way, that the pin stays at the same position while rotating the table.

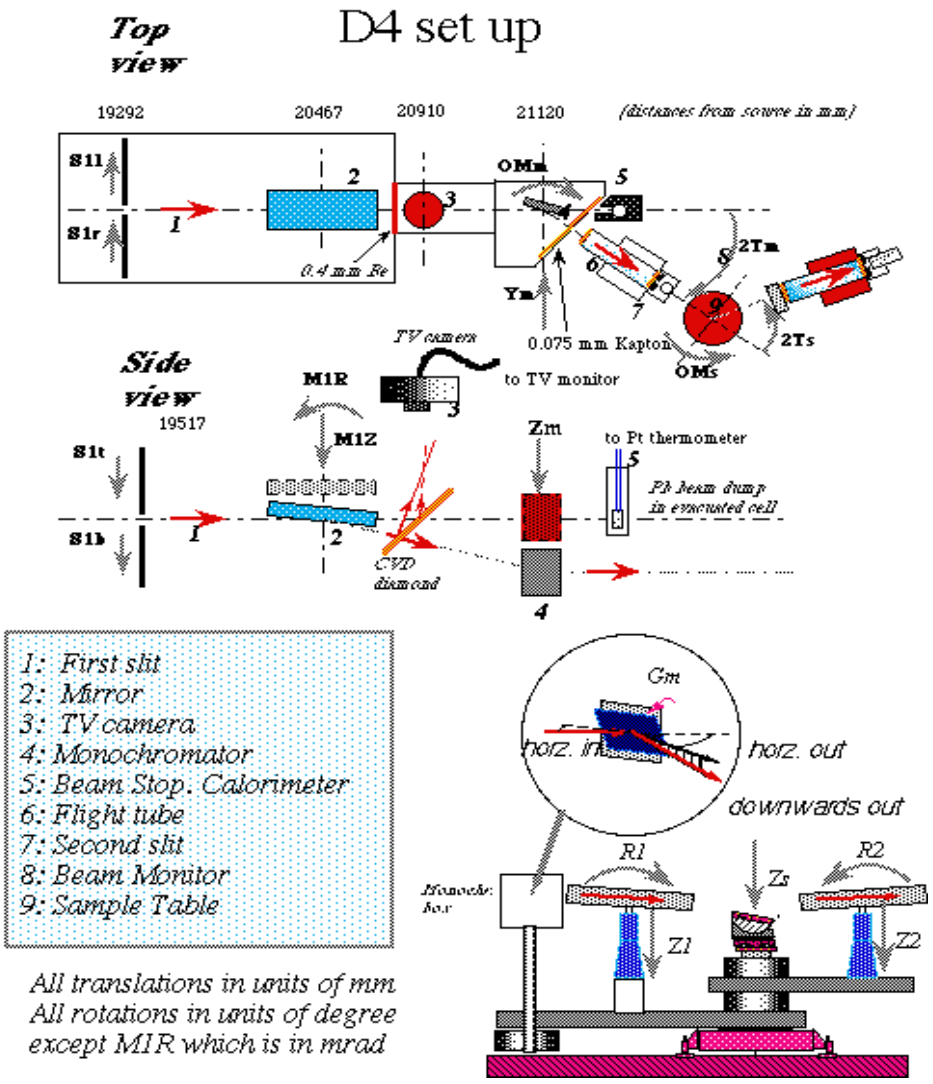

Figure 4: *The D4 Beamline at DESY. Top and side view*

6. Calibrate the diffractometer angle (2TM). Changing 2TM so that the beam is exactly in the centre of the calibrated pin. Set this angle to 2 times the Bragg angle (OMM).

7. Set each blade position of 2nd slit by scanning the position of it and measure the intensity. At half of the maximum intensity set the position to zero.

8. Put in small pieces of Fe-absorber with different thickness as attenuators in order to make the detector able to detect the photon intensity (detector cannot detect photon intensity exceeding 10^5 counts/minute)

9. Set each blade position of 3rd and 4th slit by using the same 2nd slit method

3.3 How to make the samples parallel to the beam?

The samples are two pieces of polished Si(100)-wafer (see Fig. 5.). One wafer is used as a mirror and the other one is used as a crystal. Before starting the experiment, one sample has to be made parallel to the beam and perpendicular to the table ground by moving two motors which are GLS and GUS (see Fig 3).

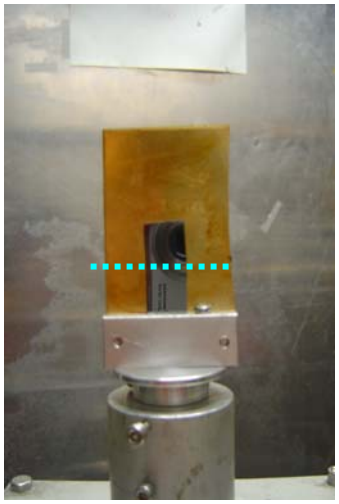

Figure 5: *Two pieces of Si(100)-wafers used as samples*

3.4 Measurement of the surface reflectivity

As it is mentioned above one of the samples is used as a mirror. Thus various small angles (OMS, TTS) are used to measure the reflectivity of this surface. The result from this experiment is illustrated in Fig. 6 (the black line is the observed data, the blue line is the fitted data and the red line is the difference of both data.)

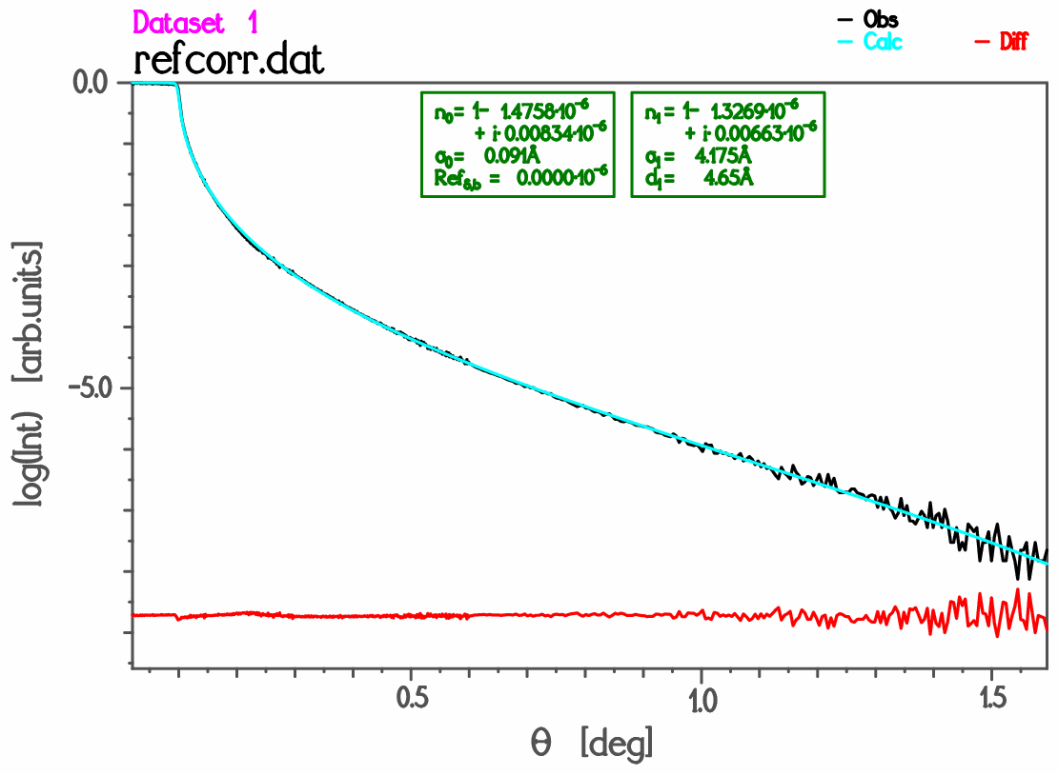

Figure 6: *Surface Reflectivity of Si at 18 keV measured from this experiment*

From this figure results that the surface of this sample used as a mirror is quite smooth (roughness about 4.2 Å). One very thin oxid layer (4.7Å) covers the Si substrate.
Moreover, the critical angle of this sample is measured with approximately 0.1 degree which corresponds to the critical angle of Si at 18 keV calculated by the theory.

3.5 Determine the position of the Si(1 0 0) lattice plane

In the next step we wanted to find the exact position of the (1 0 0) lattice plane relative to the surface. For that we calibrated all angles to the coordinate system of the surface. That means, if the angles are set to zero the center of the beam hits the edge of the wafer and the beam is exactly parallel to the surface of the wafer. In this coordinate system we searched for the (4 0 0), (8 0 0) and (12 0 0) Bragg Peaks, which are the allowed reflections in the diamond lattice type of Silicon. We could pinpoint these peaks very accurately. As it can be seen in Table 2, the measured angles in the coordinate system of the surface did not correspond exactly to the calculated Bragg angles. This difference is due to the miscut and should be the same for all all three measured Bragg angles, because

they belong to the same lattice plane. However, these differences δ_{12} (see Fig 3) were not the same. The reason for that was probably that the beam did not hit exactly the centre of rotation of the sample. This leads to different δ_{12} while rotating OMS. However, we made three measurements and we know the used rotation angles OMS. So in principle it should be possible to calculate the real angle δ_{12} from these measurements.

	Si (4 0 0)	Si (8 0 0)	Si (12 0 0)
Measured Bragg (2TS/2)	14,725°	30,544°	49,630°
Calculated Bragg angle	14,696°	30,490°	49,559°
Miscut in OMS (δ_{12})	0,029°	0,054°	0,071°
Miscut in GLS (δ_{34})	-0.230°	-0,238°	-0,251°

Table 2: *Bragg angles and the miscut*

Scanning the angle GLS and looking for the maximum intensity we could measure the miscut angle δ_{34}. This angle did not differ very much for the three Bragg angles.

At last, with the equation in chapter 2.2 the actual miscut angle between the (1 0 0) lattice plane and the surface can be calculated.

3.6 Aligning the two wafers parallel

On the last day of our experiment we tried to find out how complicated it will be to align two planes exactly parallel with simple methods. We took the surface of the lower wafer (see Fig 5) as the reference. Then we moved the sample down and measured the position of the surface of the upper wafer. We did not measure the position of the (1 0 0) lattice plane because the surface was easier to measure and the miscut of the lower wafer was known.

The two wafers were fixed by grease on the same metal plate. For the two surfaces we measured a slightly difference of 0.075° in OMS. The length of the wafers was 10mm (see Fig 7). So we knew that we have to lift or lower one side of one wafer by just around 10µm.

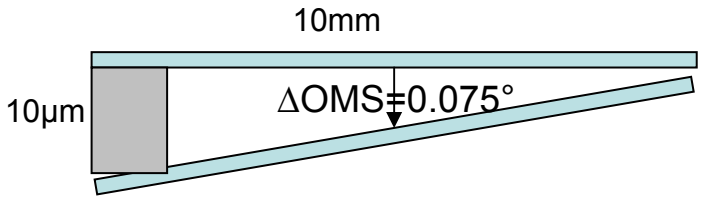

Figure 7: *The two wafers are not exactly parallel*

First we tried to achieve that by pushing on one side of the upper wafer. The pressure should remove a little bit of the grease underneath the wafer. This should lead to the wanted tiny tilt in OMS. Unfortunately this did not work out in the way we hoped. Even so the wanted tilt was so small we could not reach it with this procedure.
Next we underlaid aluminium foil under one side of the upper wafer. However we knew, that the aluminium foil has a thickness of 30µm and it was not a real surprise that we had no success to correct the tilt that way. Unfortunately, there was no time left for more experiments.

4. Conclusions

From the theoretical calculations and the experiment we made, one can conclude that the wanted alignment of the parallel laser will not be easy to achieve. Even so, it is not a real problem to measure the miscut angle, it is a real challenge to align the mirror perfectly. A very well adjustable sample holder would be needed. For example with the help of Piezo motors one could align the mirror very precisely on the sample holder. However, more motors in the beamline are not welcomed. It might also be possible to correct the laser beam via optical devices, like e.g. prisms. But this has to be tried and found out by others.

Acknowledgements

This work was supported by DESY summer student program. We are very thankful to our supervisor, Dr. Oliver H. Seeck, for his kindness, teaching and the opportunity to do this work. In addition, we would like to thank staffs in HASYLAB group for the facilities and their helpfulness.

References

[1] B.W. Batterman and H. Cole, *Rev.Mod.Phys.* **36**, 681 (1964).

[2] J. Als-Nielsen and D. McMorrow, *Elements of Modern X-ray Physics,* John Wiley & Sons, Ltd (2001).

[3] L.D. Doucette, M.Pereira da Cunha and R. J. Lad, *Rev.Sci.Ins.* **76**, 36106 (2005)

[4] http://www.fys.ku.dk/~als/d4html/beamlt.htm, retrieved September 3, 2007.

[5] http://hasylab.desy.de/e78/e672/e5111/e5204/index_eng.html, retrieved September 3,2007.