

Procedure for sample alignment (with 0 order light)

The procedure for the alignment of the sample consists in minimizing the angular pitch, i.e. the angle between the normal of the surface and the plane xz , in determining the angular offset of θ_M respect to the real incidence angle θ and in minimizing the distance of the surface of the sample from the centre of the chamber. This procedure is usually done looking at the visible light of the beam, and following these steps:

Rough alignment of the sample (Positioning the sample at 45° and centering the sample through movements x and y of manipulator respect to the beam):

- 1) Open all the valves of the beamline, except REFO (to let go the visible light);
- 2) Remove filter (if any), open the slit full ($900 \times 450 \mu\text{m}^2$) and set monochromator in zero order (to get visible light);
- 3) Put the experimental chamber in «0 order» position ;
- 4) Adjust X_M et Y_M to have the beam at the desired location on the sample. This is done with θ_M at about -45 (Visualize with camera 1);

Adjustment of sample tilt (see Figure 1 a): rotation of the sample tilt until the beam reflected by the sample is brought in the plane xz . This is done by setting the reflected beam on the center of the slits of the upper diode placed at the entrance flange.

a)

b)

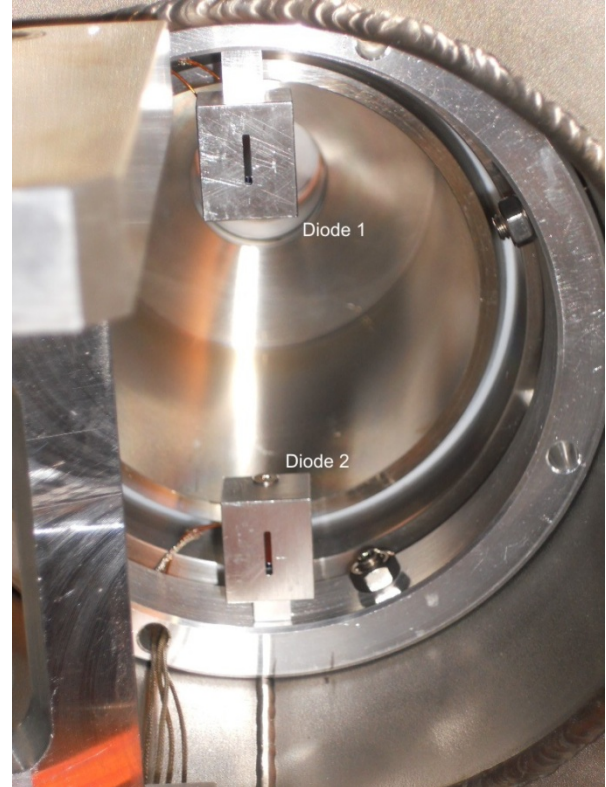
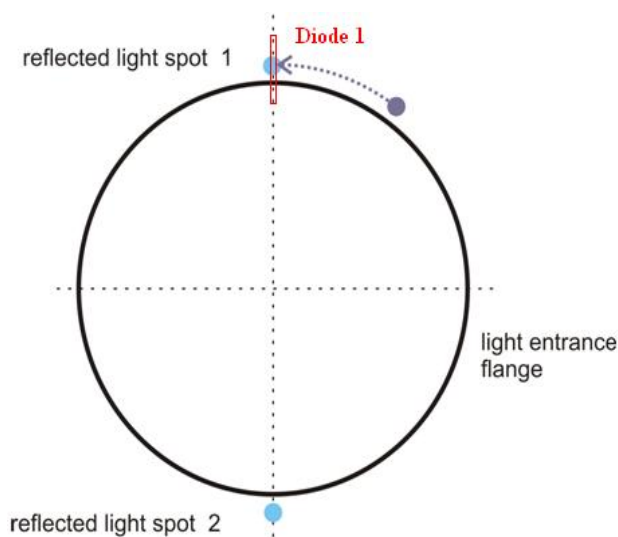


Figure 1a: Scheme for Tilt correction;

Figure 1b: image of the two vertical slits diodes used for alignment.

- 5) Adjustment of the sample tilt, to do with camera 4. The middle of the beam should fall in the slit.
 - o Go to around $\theta_M = -90$ and $\theta_A = -20$ (else shadow);
 - o With «incremental» motion, move tilt by ± 2 mm to engage it (+2 mm to move the beam towards Aloisa, -2 mm to move the beam towards Lilit);
 - o Move with 0.2 mm steps (then 0.1 and 0.05 mm steps) to put the beam into the diode slits;
 - o If adjustment going up, then go beyond the position by 0,05 mm (to take into account the latch);
 - o Go back to 0 mm with «absolute» motion;

Determination of θ_M offset (see Figure 2): determination of the positions θ_{M1} and θ_{M2} at which the reflected beam is on the center of the upper and lower diodes placed at the entrance flange. The average of these two positions refers to the normal incidence condition $\theta = 0^\circ$, and then the offset is given by

$$\text{offset} = \left| \frac{\theta_{M,1} + \theta_{M,2}}{2} \right| - 90$$

With this procedure, the angular precision is about 0.1 degrees.

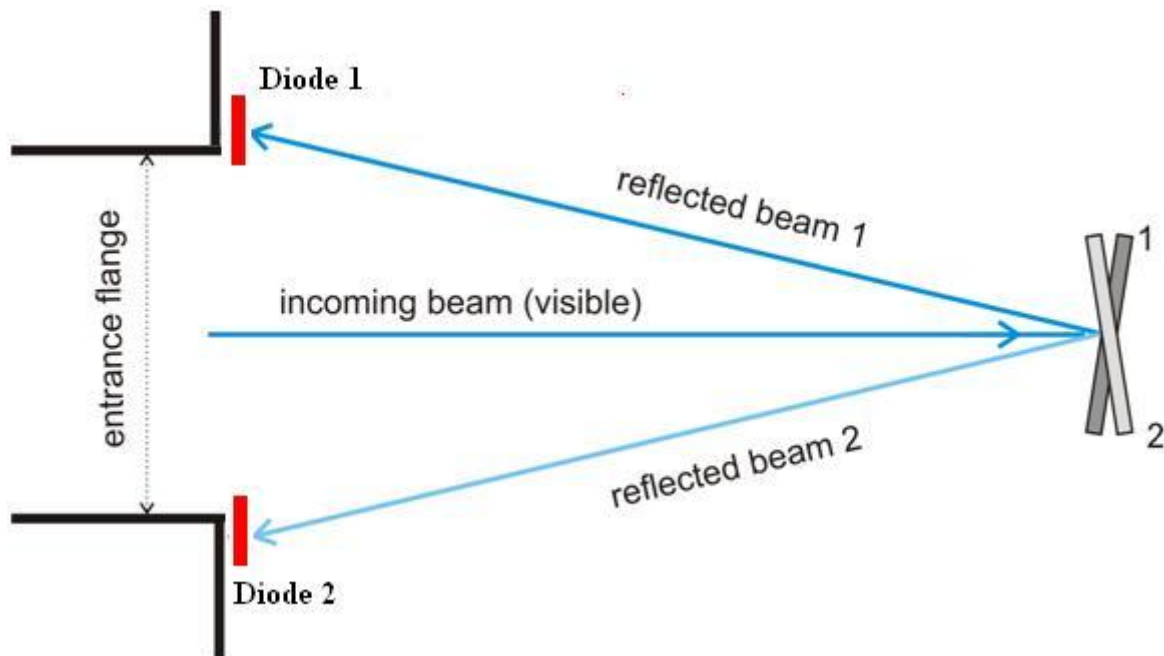


Figure 2: Scheme for θ_M offset correction in the plane xz.

- 6) Choose Ammeter CH1 for Instrument1 and Ammeter CH2 for Instrument2, and Configure both instruments (6.4 μA range);
- 7) Do a θ_M scan with two regions (scanning of the second zone around the present value of θ_M , -7° for the first zone with respect to the second zone, each zone scanned over 2°). The scan can be done after setting Dark OFF and Waiting time zero;
 - o Put the cursors at the half-maxima of Instrument 1 and 2;
 - o Read the mean value of the θ_M angle, which is the angle corresponding to normal incidence;
 - o Copy the value of the θ_M offset;

*Determination of correct Z_M : it consists in the minimization of the distance of the surface of the sample from the centre of the chamber (see **Figure 3**): after the determination of the offset, the sample could not be positioned in the axis θ_M . This can be done rotating the sample till grazing incidence and checking if the spotlight remains on the centre of the sample. Finally, the last check is done by verifying that the reflected beam from the sample set at $\Theta_G=10^\circ$ is completely inside the electron analyzer hole set at $2\Theta=20^\circ$.*

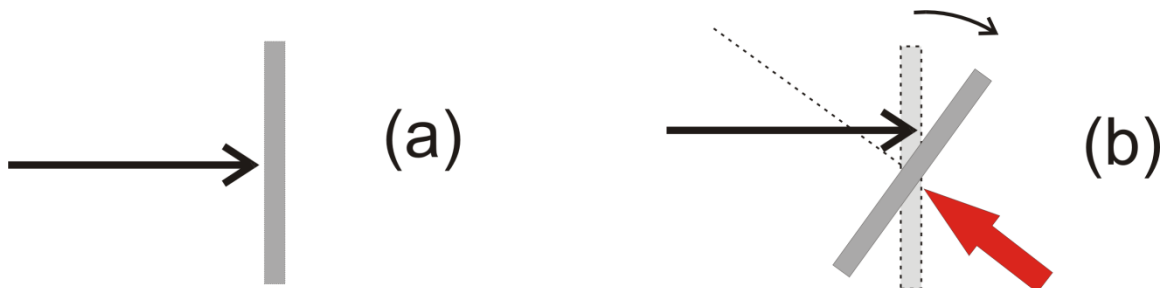


Figure 3: Scheme for minimization of h , the distance of the sample from chamber center.

- 8) Verify the sample height at 10° grazing ($\theta_M = -10$ -offset), (to see with camera 3):
 - Go to θ_M to the position -10-offset;
 - Go to θ_A to the position -20 (if it is not yet the case);
 - Go to ϕ_A to position 90 (electron analyzer in the incidence plane);
 - Move Z_M so that the beam is centered in the hole of the electron analyzer;
 - Go back to camera 1;
- 9) Go to ϕ_A to 0 position;

Procedure for standard reflectivity measurements using detectors on the frame of electron analyzer

- 10) Choose a photon energy to continue alignment (generally, energy at which the first measurements will be made). Choose the required inclusion angle of the grating. Choose a filter if necessary. Close the entrance slit to the desired spot size (typically $900 \times 100 \mu\text{m}^2$);
- 11) Open REFO valve;
- 12) Choose Keithley 1 as Instrument 1 and Keithley 2 as Instrument 2 and configure both instruments ;
- 13) Go to the desired θ_M angle then turn off the gauge (else saturation of diode current);
- 14) Put θ_A at the approximate value corresponding to twice the grazing angle and taking into account the offset determined from the value obtained with the direct beam;
- 15) Go to ϕ_A to about 45 for the two diodes to set the diode in the incidence plane. Fix ϕ_A to the position of the maximum;

- 16) Do a Z_M scan with a 6 mm width around its present value to check the sample uniformity and to refine the positioning of the sample;
- 17) Do a θ_A scan, 10° wide around the present value (typical 0.2° step) and copy the θ_A offset. The shift is about 30° between the large and small diodes;
- 18) Fix θ_A to the position of the maximum determined in 17;
- 19) Do a ϕ_A scan of 10° around 45° for the large and small slits diodes (typical 0.2° step);
- 20) Fix ϕ_A to the position of the maximum determined in 19.