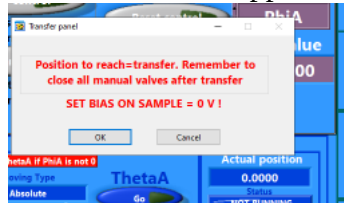


# PROCEDURE FOR STANDARD REFLECTIVITY MEASUREMENTS

## SAMPLE INSERTION:

<configure experimental chamber> click on TRANSFER button (The monitor valve will be automatically closed). Turn ON light. Select camera 1 in the camera control program (PC Bear-BL1-TOP VIEW) ;

The window will appear



Click on OK and in case remove SAMPLE BIAS. If sample is Grounded, just click Ok. The transfer positions are written:



Go to the transfer position by clicking on GO for each movement:

ThetaM=0 (+-3)  
ThetaA=0  
PhiA=0  
XM=4  
YM=19.5  
ZM=-5.5  
phiM=+92

Open valves between experimental chamber and garage and insert sample.  
Remove to put back the blank corks to all the windows of experimental chamber.  
Remind also to close all valves between exp. Chamber and the other ones.

## Available Detectors

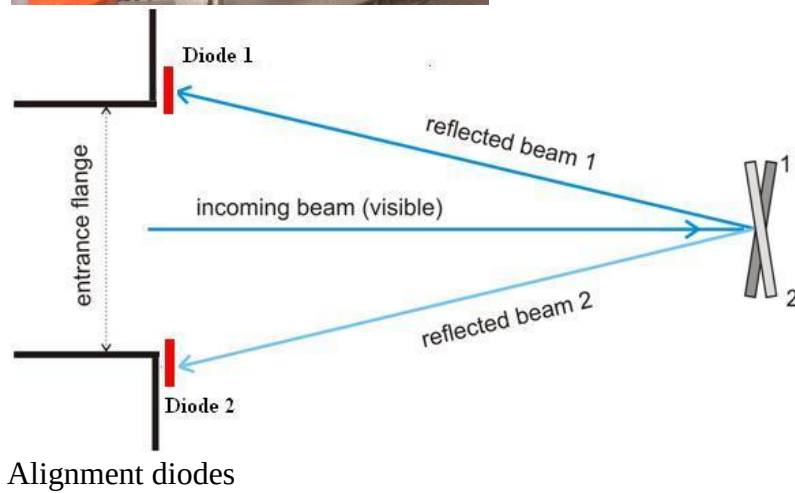
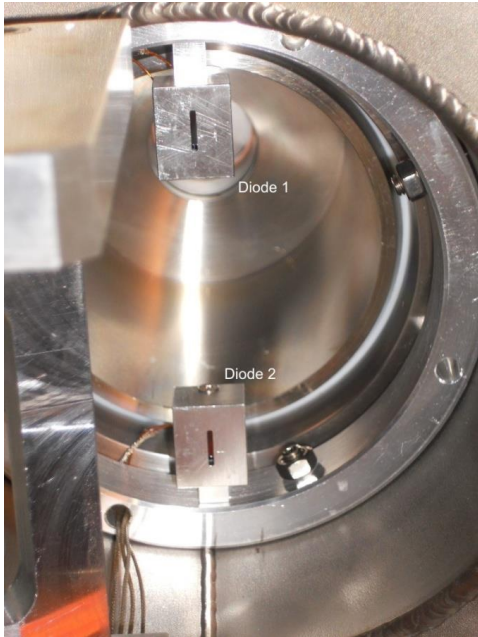
The available detectors for reflectivity are silicon diodes, whose rough positions and acceptances are given below:



## Procedure for alignment of the sample

### Sample alignment general description

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  $xz$ , and in determining the angular offset of  $\Theta_M$  respect to the real incidence angle  $\Theta$  and in minimizing the distance of the surface of the sample from the center of the chamber. This procedure is usually done looking at the visible light of the beam (zero order), and using a standard routine.



## Rough alignment of the sample with 0 order

This action consists in positioning the sample at 45° and centering the sample through movements x and y of manipulator respect to the beam)

Turn Light ON to control ThetaA movement by eye;

<Configure experimental chamber> the axes chamber in transfer position are (PhiA=0; ThetaA=0; XM=5, YM=20, ZM=-5.5, phiM=92, ThetaM=0).

|                                                                                                                                         |  |                                                                                              |  |                                                                                                         |  |
|-----------------------------------------------------------------------------------------------------------------------------------------|--|----------------------------------------------------------------------------------------------|--|---------------------------------------------------------------------------------------------------------|--|
| shut down the control<br><input type="button" value="Restart control"/><br>Press this after shut down the control                       |  | Press this in case of problems with control<br><input type="button" value="Reset position"/> |  | <b>Axis value</b><br>0.0000                                                                             |  |
| <b>Analyzer</b>                                                                                                                         |  |                                                                                              |  |                                                                                                         |  |
| Do not move ThetaA if PhiA is not 0<br>Moving Type<br>Incremental<br>Increment Angle (°)<br>0.0000<br><input type="button" value="Go"/> |  | Actual position<br>0.0000<br>Status<br>NOT RUNNING<br>Error<br>No error                      |  | Moving Type ThetaM<br>Incremental<br>Increment Angle (°)<br>0.0000<br><input type="button" value="Go"/> |  |
| Moving Type<br>Incremental<br>Increment Angle (°)<br>1.0000<br><input type="button" value="Go"/>                                        |  | Actual position<br>-0.0019<br>Status<br>NOT RUNNING<br>Error<br>No error                     |  | Moving Type PhiM<br>Incremental<br>Increment Angle (°)<br>0.0000<br><input type="button" value="Go"/>   |  |
| <b>Chamber</b>                                                                                                                          |  |                                                                                              |  |                                                                                                         |  |
| Moving Type PsiC<br>Incremental<br>Increment Angle (°)<br>0.0000<br><input type="button" value="Go"/>                                   |  | Actual position<br>-0.0000<br>Status<br>NOT RUNNING<br>Error<br>No error                     |  | Moving Type XM<br>Incremental<br>Increment (mm)<br>0.0000<br><input type="button" value="Go"/>          |  |
|                                                                                                                                         |  |                                                                                              |  | Actual position (mm)<br>-0.0000<br>Status<br>NOT RUNNING<br>Error<br>Undefined                          |  |
|                                                                                                                                         |  |                                                                                              |  | Moving Type YM<br>Incremental<br>Increment (mm)<br>0.0000<br><input type="button" value="Go"/>          |  |
|                                                                                                                                         |  |                                                                                              |  | Actual position (mm)<br>19.5269<br>Status<br>NOT RUNNING<br>Error<br>Undefined                          |  |
|                                                                                                                                         |  |                                                                                              |  | Moving Type ZM<br>Incremental<br>Increment (mm)<br>0.0000<br><input type="button" value="Go"/>          |  |
|                                                                                                                                         |  |                                                                                              |  | Actual position (mm)<br>-5.5049<br>Status<br>NOT RUNNING<br>Error<br>Undefined                          |  |
|                                                                                                                                         |  |                                                                                              |  | Moving Type TiltM<br>Incremental<br>Increment (mm)<br>0.0000<br><input type="button" value="Go"/>       |  |
|                                                                                                                                         |  |                                                                                              |  | Actual position (mm)<br>0.0007<br>Status<br>NOT RUNNING<br>Error<br>Undefined                           |  |
| monitor pressure<br>7.153e-10<br><input type="button" value="Turn gauge on"/>                                                           |  |                                                                                              |  |                                                                                                         |  |

Put the axes chamber in «0 order» position looking at camera 1 and by clicking on the buttons Go:

phiM=-3

ThetaM=-45

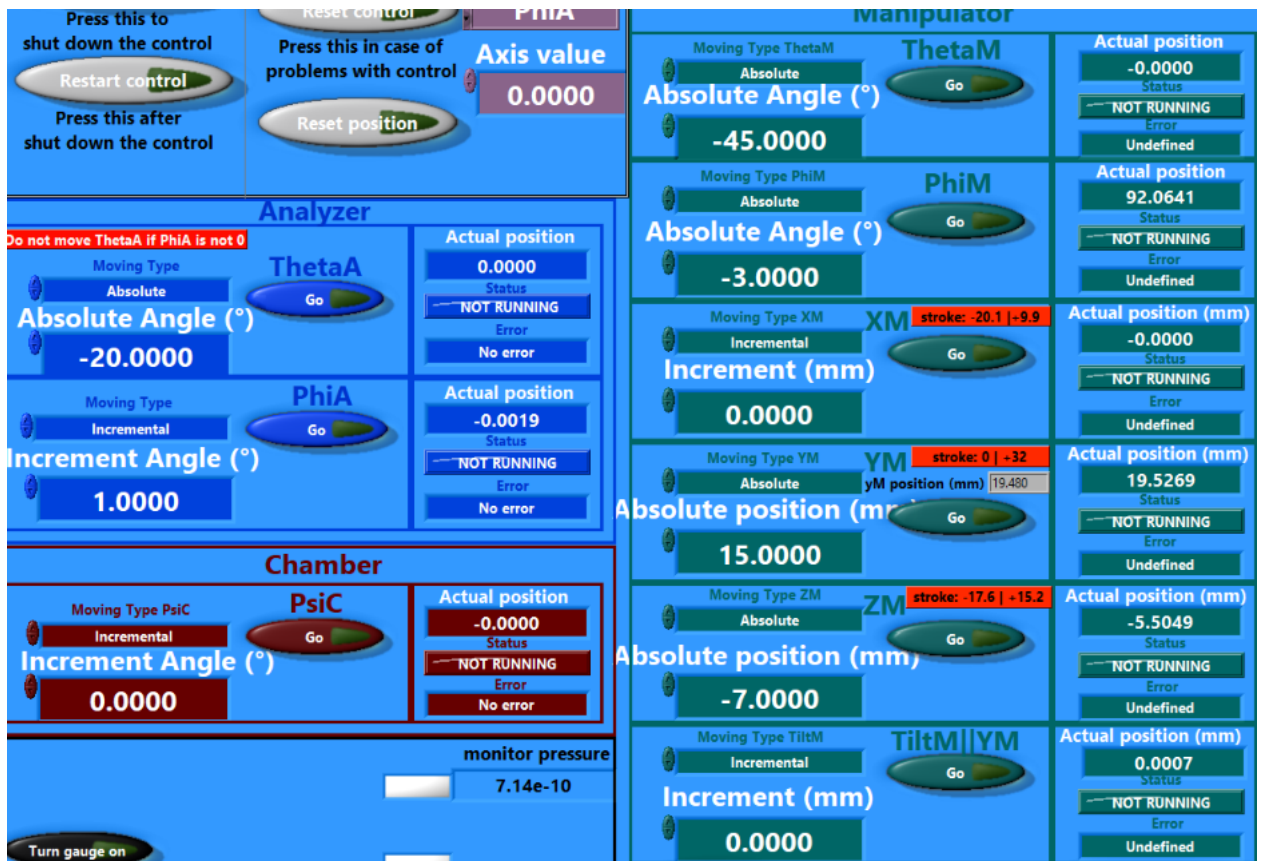
PhiA=0

ThetaA=-20

XM=0

YM=15

ZM=-7



<Configure monochromator> 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). In the case that the 0 order is not visible, this is due to a slight shift of the angular position of the monochromator. In this case, this is the iteration:

- Click on the button offset G1200 on the right button of the window;
- Increase the value Delta 0 order position of a quantity of the order +100;
- Click again on the Set 0 order button;

If the 0 order is still not visible, check the monitor intensity on the main program. The 0 order is really centered on the exit slits if the current is of the order of  $\mu\text{Amps}$ . If the monitor current increases, go back to step b) increasing the parameter Delta, otherwise decrease the parameter Delta, until the 0 order becomes visible.

Check that all the valves of the beamline are open, except REFO (to let go the visible light);



Roughly align the sample checking that the beam 0 order is on the center of the sample, and by rotating the sample around -45 check that the reflected beam dazzles the camera 1;

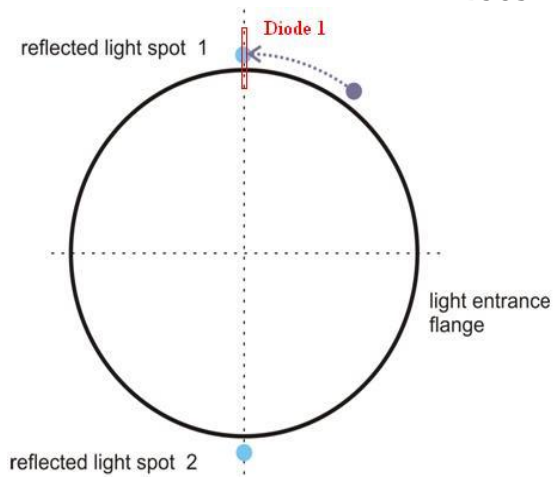
move ThetaM=-80 for fine alignment of XM;

Switch on camera 2 (Front View); <Configure experimental chamber> Adjust XM to have the beam at the desired location on the sample. If zero order spot on the sample is shadowed by wires use ThetaM=-80

Adjust TILTM roughly with camera 1, so that scattering plane corresponds to the X-Z plane of the laboratory reference system;

Switch on the camera 3 (Diode view); <Configure experimental chamber> Move ThetaM in the range -80 to -95 (experimental chamber window) searching for a reflected beam on monitor CH 3. If the reflected beam is visible somewhere around the diode 1 push stop motor and adjust ThetaM to keep the beam as close to diode slit as possible;

## ADJUSTMENT OF SAMPLE TILT



### Scheme for Tilt correction

Move the sample TiltM until the beam reflected by the sample is brought in the vertical plane so it can get inside of the diode 1 and 2 slits. This is done by setting the reflected beam on the center of the slits of the upper diode placed at the entrance flange.

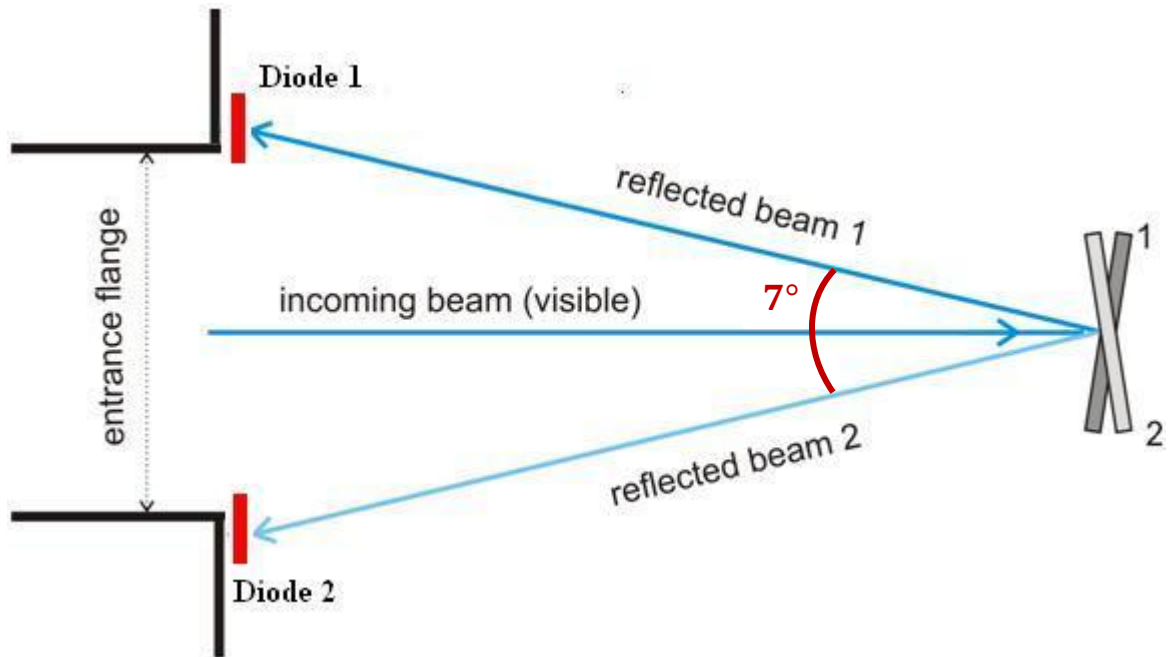
With «incremental» motion, move tilt by  $\pm 4$  mm to engage it (+2 mm to move the beam towards Lilith (right), -2 mm to move the beam towards Aloisa (left)); Move with 0.2 mm steps (then 0.1 and 0.05 mm steps) to put the beam into the diode slits;

The procedure is finished when the 0 order is centered on the slits of diode 1 (FIGURE BELOW)



image of the two vertical slits diodes used for alignment.

## DETERMINATION OF THETAM OFFSET



*Scheme for ThetaM correction in the plane xz.*

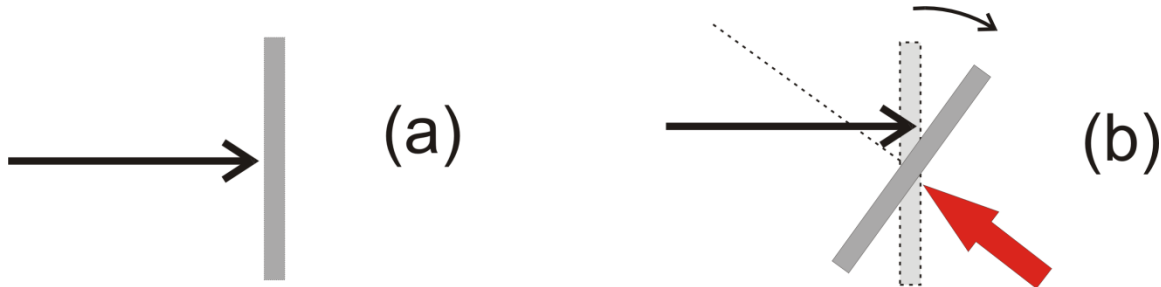
Determination of the positions THETAM1 and THETAM2 at which the reflected beam is on the center of the upper and lower diodes placed at the entrance flange. The average of these two positions refers to the normal incidence condition  $\text{ThetaG}=90^\circ$ , and then the angular ThetaM corresponding to normal incidence is given by  $\text{ThetaM}_{90}=(\text{ThetaM1}+\text{ThetaM2})/2$ . With this procedure, the angular precision is about 0.1 degrees.

0 order light on the center of diode 1;

<Main program>: Click on Align Sample green button: this starts an automatic procedure that gives the angular value of  $\text{ThetaM}_{90}$  corresponding to normal incidence ( $\text{ThetaG}=90^\circ$ ). This angular value is given in the acquisition panel (below scan type menu). ( $\text{ThetaM}_{90}=-93$  is a typical value);

### ROUGH CHECK OF $Z_M$ USING 0 ORDER

Determination of correct  $Z_M$ : it consists of distance minimization of the sample surface from the center of the chamber (see Figure below): after the determination of the offset, the sample could be positioned in the axis  $\theta_M$ . This can be done rotating the sample till grazing incidence and checking if the spotlight remains on the center of the sample.



*Scheme for minimization of  $h$ , the distance of the sample from chamber center.*

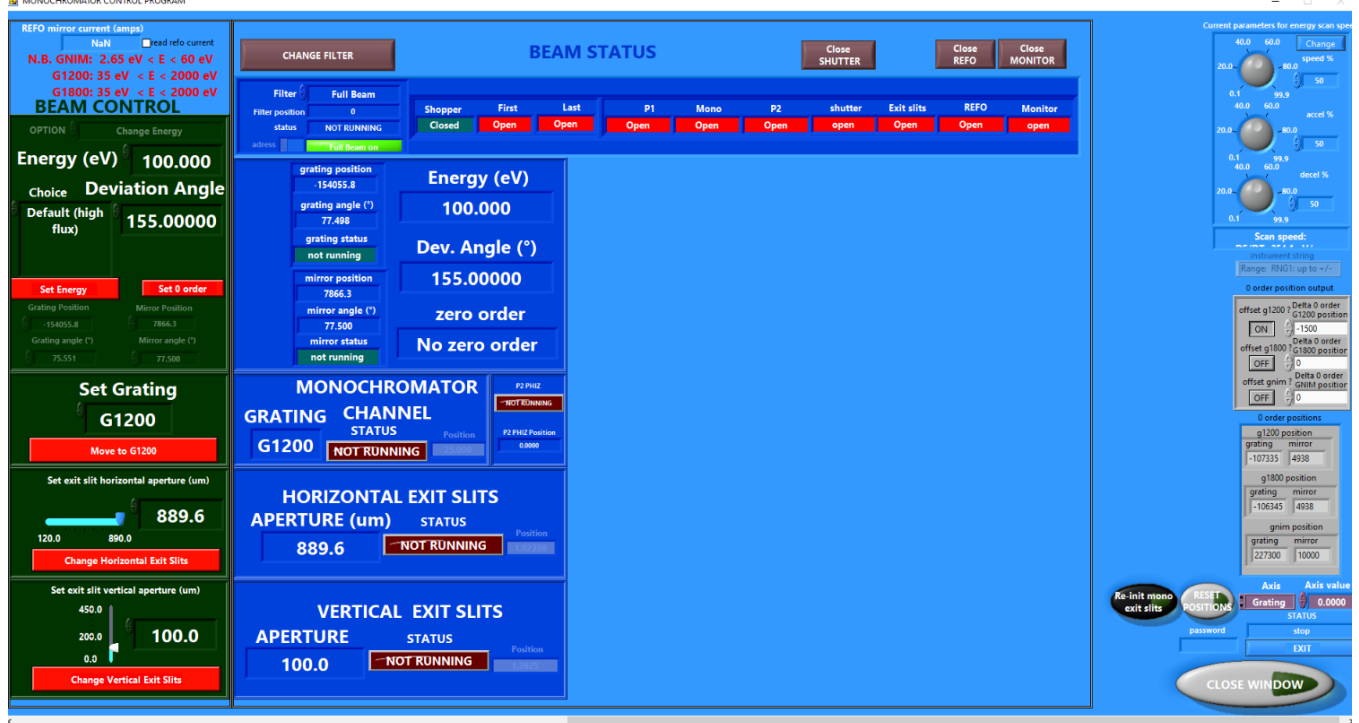
<Configure experimental chamber>: Verify the sample height at  $10^\circ$ - $20^\circ$  grazing ( $\theta_M = -10^\circ$  offset), (to see with camera 1):

- Go to  $\theta_M$  to the position  $-10^\circ$  offset;
- Move  $Z_M$  so that the beam is centered on the sample;

## FINE alignment using monochromatic light

Switch Gauge and Light **OFF**

<Configure Monochromator>; 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 X 100  $\mu\text{m}^2$ ); Open REFO valve.



<Main program>: Choose Keithley C (diode) as Instrument 3. Choose the diode for measurements. In case of standard reflectivity measurements use A2.

Set integration time (1 second for precise measurements, 0.4 seconds for quick measurements).

Choose Keithley A (mirror) as Instrument 2. Use the same integration time of Instrument 3

Choose Keithley B (sample TEY) as instrument 1. Use the same integration time of Instrument 1. Give a BIAS to the sample to optimize TEY, if required, otherwise set connection GROUND. In this case, after this launch a long time scan until sample current remains stable.

# SAMPLE/EMISSION CURRENT

Diagram 1: GROUND

range value  
**20 nA**

Integration time (sec)  
**0.4**

N.B. The default is 0.4 sec.  
For details, click the HELP button

Connection  
**Ground**

HELP

Measure Type Keithley B  
Current

error in

error out total

error out autozero

waiting time (msec) **0**

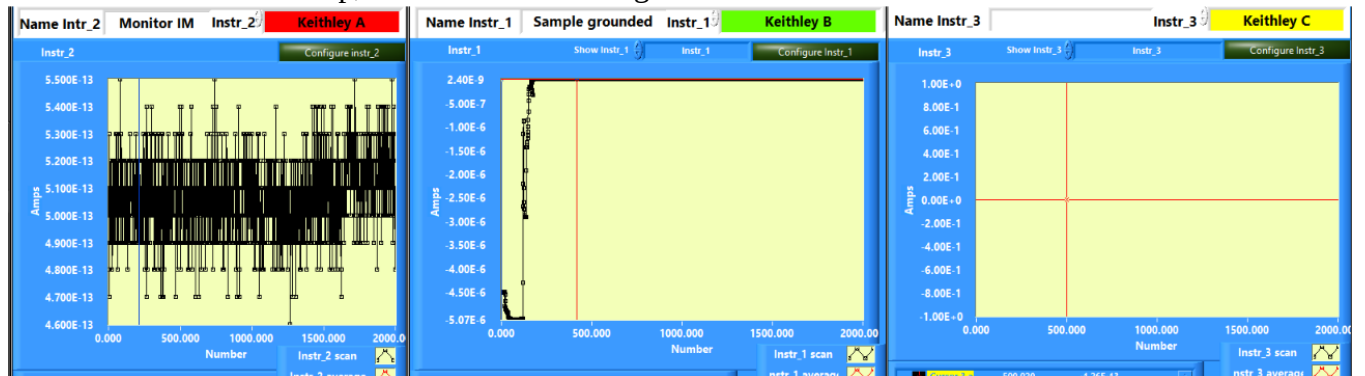
N.B. The correct waiting time is 20 msec

CLOSE WINDOW

Diagram 2: BIAS

**Connection ON**

For the measurement setup, this is the final configuration:



## FINE ALIGNMENT

### CASE A) ULTRA GRAZING INCIDENCE ANGLE MEASUREMENTS ( $\Theta_G < 5^\circ$ )

<Configure experimental chamber> Go to the  $\Theta_M$  corresponding to the grazing angle 0. example  $\Theta_{M90} = -93$ .  $\Theta_{M0} = -93 + 90 = -3$ .

Move down the sample with  $z_M$  to  $z_M = -10$  mm;

Connect to the Keithley C the diode with small window B2;

Set the diode B2 in the I0 position ( $\Theta_A = -59.5$ ;  $\Phi_A$ );

Go with the to  $\Phi_A$  corresponding to the I0 position to set the diode in the incidence plane ( $\Phi_A = +2.5$  for detector A2).

Do a  $Z_M$  scan with a 6 mm width (step 0.1, and refine it to  $z_M = .025$ ) to find the position  $z_M$  until the detector is completely darkened: the Half Maximum corresponds to the  $z_M$  at which positioning sample (for instance  $z_M = -7$ ).

Position sample  $z_M$  at right  $z_M$  and scan  $\Theta_M$  around the  $\Theta_G$  angle 0 with step 0.05 (for instance, if  $\Theta_{M90} = -93$ , scan  $\Theta_M$  from  $\Theta_M -4$  to  $-2$ ) and find the maximum of intensity. The maximum corresponds to the grazing incidence angle  $\Theta_{M0}$ ;

Connect to the Keithley C the diode with large window A2;

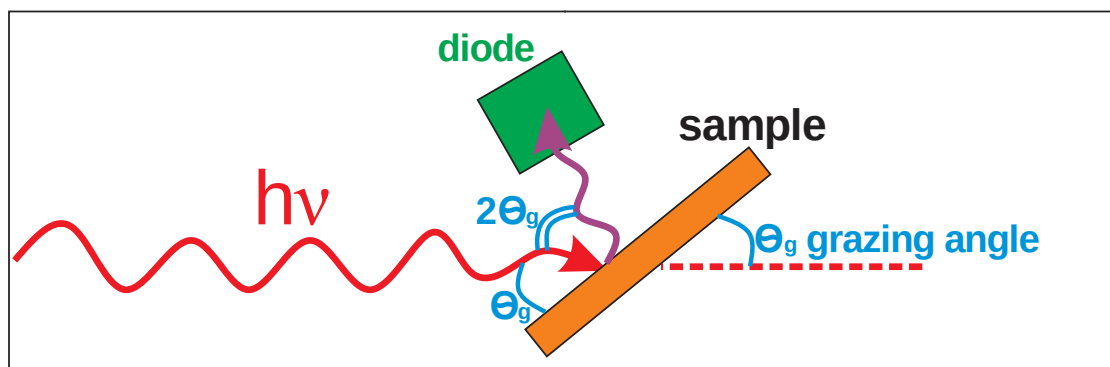
Measurements in grazing incidence will be done using as a reference this  $\Theta_{M0}$ ;

For instance,  $\Theta_G = 1.5$  will be acquired at:

$\Theta_M = \Theta_{M0} - 1.5$ ,  $\Theta_A = \Theta_{A0} - 3$ ;  $\phi_A = \phi_{A0}$

### CASE B) NORMAL INCIDENCE MEASUREMENTS ( $\Theta_G > 5^\circ$ )

<Configure experimental chamber> Go to the  $\Theta_M$  corresponding to the lowest grazing angle. example  $\Theta_G = 20^\circ$  and  $\Theta_{M90} = -93$ .  $\Theta_{M20} = -93 + 90 - 20 = -23$ .



Geometry of reflectivity measurement. **Example of** reflectivity measurements with diode A2.

Go to  $\Phi_A$  corresponding to the I0 position to set the diode in the incidence plane ( $\Phi_A = +2.5$  for detector A2).

<Main program>: Do a time scan to check if the reflected beam is on diode

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.

## FINE ALIGNMENT OF THE DETECTOR

<Configure experimental chamber>\_Go to the highest grazing angle position, for instance  $\text{ThetaG}=80^\circ$ ,  $\text{ThetaM}=-80$ );

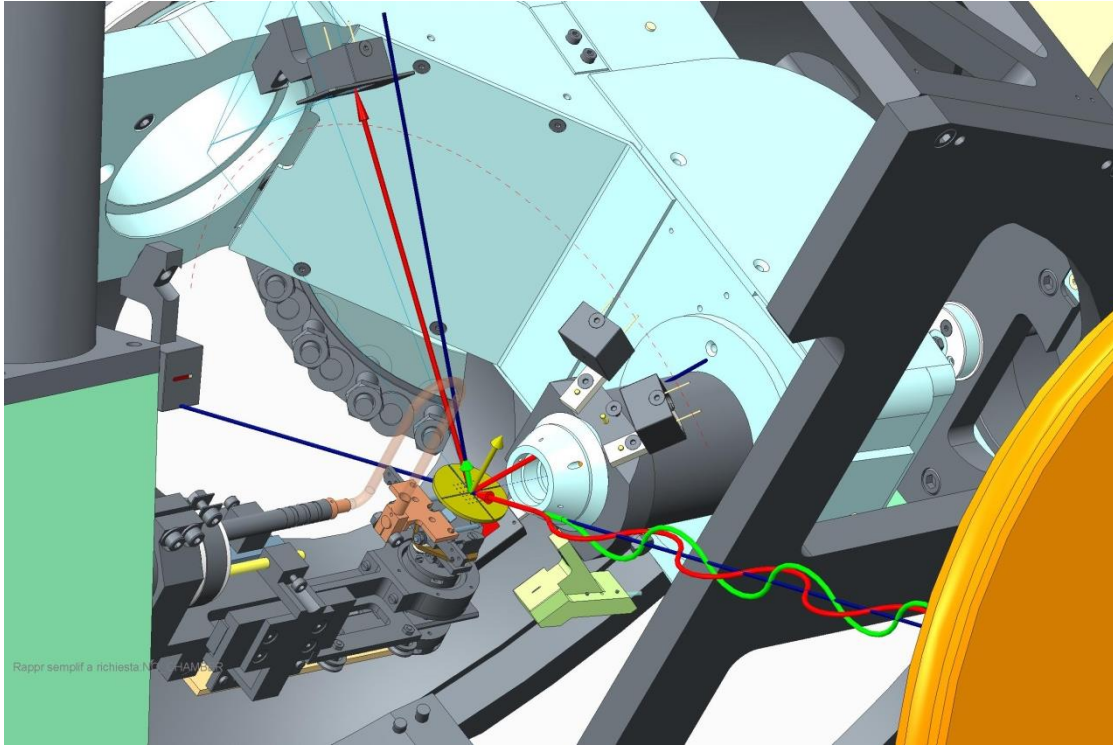
Go to the desired ThetaA angle: example For  $\text{ThetaG}=80^\circ$  and diode A2,  $\text{ThetaA}_{20}=-49.5-2\times\text{ThetaG}=-209.5$ .

Go to the desired PhiA position, i.e. the position of PhiA of I0 (PhiA about +2 for diode A2).

Alignment of diode: scan ThetaA around reflectivity position (ThetaA from -215 to -205). Put cursor 1 and 2 on the Half Maxima of the I3 curve and use the average of this value as detector position. Move detector in this ThetaA position (configure experimental chamber). (Example:  $\text{ThetaA}_{20}=-209.5$ )

Alignment of diode: scan PhiA around reflectivity position (PhiA from -3 to +7). Put cursor 1 and 2 on the Half Maxima of the I3 curve and use the average of this value as detector position. (Example:  $\text{PhiA}_{20}=+2$ ). Move detector in this PhiA position.

## Reflectivity Measurements



*Reflectivity measurement with B2 diode illustration*

<Main program>Measurement as a function of Energy: set Photon Energy vs Var\_1 vs Var\_2 and click on configure macro

Measurement as a function of incidence angle: set Theta-2Theta scan using the angular offset previously determined. Example: ThetaG=from 20 to 40°.

ThetaM,in=-43 (assuming ThetaM<sub>90</sub>=-93 we have that ThetaM<sub>40</sub>=-93+90-40=-43);

ThetaM,fin=-23 (assuming ThetaM<sub>90</sub>=-93 we have that ThetaM<sub>20</sub>=-93+90-20=-23);

ThetaA,in=-140.5 (assuming ThetaA<sub>20</sub>=-49.5 we have that ThetaA<sub>40</sub>=-49.5-40-40=-129.5).

See macro below:

|                       |                       |
|-----------------------|-----------------------|
| Energy (eV)           | Energy (eV)           |
| 45.0000               | 53.0000               |
| Final Energy (eV)     | Final Energy (eV)     |
| 55.0000               | 103.0000              |
| Delta Energy (eV)     | Delta Energy (eV)     |
| 0.5000                | 0.5000                |
| Choice angle          | Choice angle          |
| Degrees specification | Degrees specification |
| Deviation Angle       | Deviation Angle       |
| 155.00000             | 155.00000             |
| Selected Filter       | Selected Filter       |
| Al filter             | Si filter             |
| Channel               | Channel               |
| G1200                 | G1200                 |
| range Keithley B      | range Keithley B      |
| 20 nA                 | 20 nA                 |
| range Keithley C      | range Keithley C      |
| 200 pA                | 200 pA                |
| range Keithley A      | range Keithley A      |
| 20 nA                 | 20 nA                 |
| Var_1 value           | Var_1 value           |
| -70.800               | -70.800               |
| Var_2 value           | Var_2 value           |
| -199.500              | -199.500              |
| Bias CEM (V)          | Bias CEM (V)          |
| 0.000                 | 0.000                 |
| Bias Keithley B (V)   | Bias Keithley B (V)   |
| 0.000                 | 0.000                 |
| Name region           | Name region           |

|                          |              |              |
|--------------------------|--------------|--------------|
| <b>Number of regions</b> | <b>Var_1</b> | <b>Var_2</b> |
| 2                        | ThetaM       | ThetaA       |

Compile the macro using the appropriate values and click on CONFIGURE MACRO  
Start Scan

Once finished the scan, for all the used ranges, take Time scans:

Close Shopper, select Time scan, set Number of points 30-50, select the appropriate range of the Keithley, and Start scan. Each dark scan has to be associated with the corresponding measurement.

### ***S polarization Reflectivity Measurements***

<configure experimental chamber> Turn light ON.  
Set Absolute position  $\Psi_C=90$ , and click on GO.  
Check that all the cables are OK.

## I0 MEASUREMENT:

<configure experimental chamber> Put the sample away from the beam and detector:  
 $ZM_0 = -17$ ;  $\Theta M = 0$ .

Put detector in I0 position. For diode A2, for instance:  $\Phi A_0 = +2$ ;  $\Theta A_0 = -49.5$ .

For the refinement of the I0 position, this is the procedure:

<Main program>

Alignment of diode: scan  $\Theta A$  around I0 position (select  $\Theta A$  scan as type of scan and scan  $\Theta A$  from -65 to -55 step 0.25 for diode A2). Put cursor 1 and 2 on the Half Maxima of the I3 curve and use the average of this value as detector position.

<configure experimental chamber> Move detector in this  $\Theta A$  position. (Example:  $\Theta A = -59.5$ )

<Main program> Alignment of diode: scan  $\Phi A$  around reflectivity position (select  $\Phi A$  scan as type of scan  $\Phi A$  from -5 to +5 step 0.1). Put cursor 1 and 2 on the Half Maxima of the I3 curve and use the average of this value as detector position. (Example:  $\Phi A = +2$ ).

<configure experimental chamber> Move detector in this  $\Phi A$  position.

<Main program> Do the same photon energy scan in the range of measurements already performed. Select Scan Type Photon Energy vs Var\_1 vs Var\_2 and click on configure macro

See macro below, for example:

The image shows a macro configuration window with a blue background. It contains two columns of sliders for various parameters. The left column has sliders for Energy (eV) at 45.0000, Final Energy (eV) at 55.0000, Delta Energy (eV) at 0.5000, Choice angle, Degrees specification, Deviation Angle at 155.0000, Selected Filter (Al filter), Channel (G1200), range Keithley B (20 nA), range Keithley C (200 nA), range Keithley A (20 nA), Var\_1 value (0.000), Var\_2 value (-49.500), Bias CEM (V) (0.000), Bias Keithley B (V) (0.000), and Name region. The right column has sliders for Energy (eV) at 53.0000, Final Energy (eV) at 103.0000, Delta Energy (eV) at 0.5000, Choice angle, Degrees specification, Deviation Angle at 155.0000, Selected Filter (Si filter), Channel (G1200), range Keithley B (20 nA), range Keithley C (200 nA), range Keithley A (20 nA), Var\_1 value (0.000), Var\_2 value (-49.500), Bias CEM (V) (0.000), Bias Keithley B (V) (0.000), and Name region. At the bottom, there are three sliders: 'Number of regions' set to 2, 'Var\_1' set to ThetaM, and 'Var\_2' set to ThetaA.

Compile the macro using the appropriate values and click on CONFIGURE MACRO

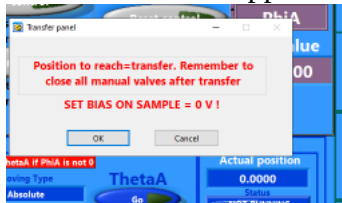
Start Scan

Once finished the scan, for all the used ranges, take Time scans:

Close Shopper, select Time scan, set Number of points 30-50, select the appropriate range of the Keithley, and Start scan. Each dark scan has to be associated with the corresponding measurement.

## SAMPLE REMOVAL

<configure experimental chamber> click on TRANSFER button (The monitor valve will be automatically closed). Turn ON light. The window will appear



Click on OK and in case remove SAMPLE BIAS. If sample is Grounded, just click Ok. The transfer positions are written in the controls of the window.



Go to the transfer position by clicking on GO for each movement:

ThetaM=0  
 ThetaA=0  
 PhiA=0  
 XM=4  
 YM=19.5  
 ZM=-5.5  
 phiM=+92

Open valves between experimental chamber and garage and extract sample.