User Manual 1

for

BL-18 B (Indian Beam line) at Photon Factory

Instructions for the users:

a. General instructions.

b. Sample mounting procedures.

   i. Mounting of powder sample in capillary tube.

   ii. Mounting of the capillary tube on the rotation stage.

   iii. Mounting of crystal.

   iv. Mounting of thin films on substrate.

   v. Mounting of low temperature setup.

   vi. Mounting of high temperature setup

c. Procedure for transferring the collected data.

d. Preliminary analysis of the data.

e. Format of report to be submitted by the users (at the end of the experiment).

General Instructions

Before entering into the hutch check for the gas (N_2 for ion chambers, He for beam tubes) flow at the gas bottles.

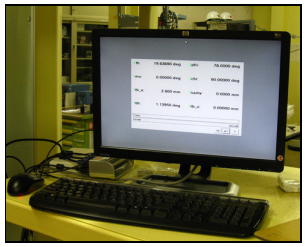
After entering into the hutch, check for the gas flow at the flow meters behind the four circle goniometer.

Controller – 1 is for motor control of the four circle goniometer.

Controller – 2 is for motor control of the eight circle goniometer.

NIM BIN for the SCA of the point detector, the pizo controller and preamplifier of the ion chambers are installed at top of the same rack used for controller – 2

2D detector (Pilatus) computer CPU was located at the top of the same rack used for controller – 1.



Same monitor, keyboard and mouse for display of both the controllers and the Pilatus PC, change the white USB extension card connected to this monitor kept under the staircase, depending on the requirement of display and select the corresponding display at the monitor.

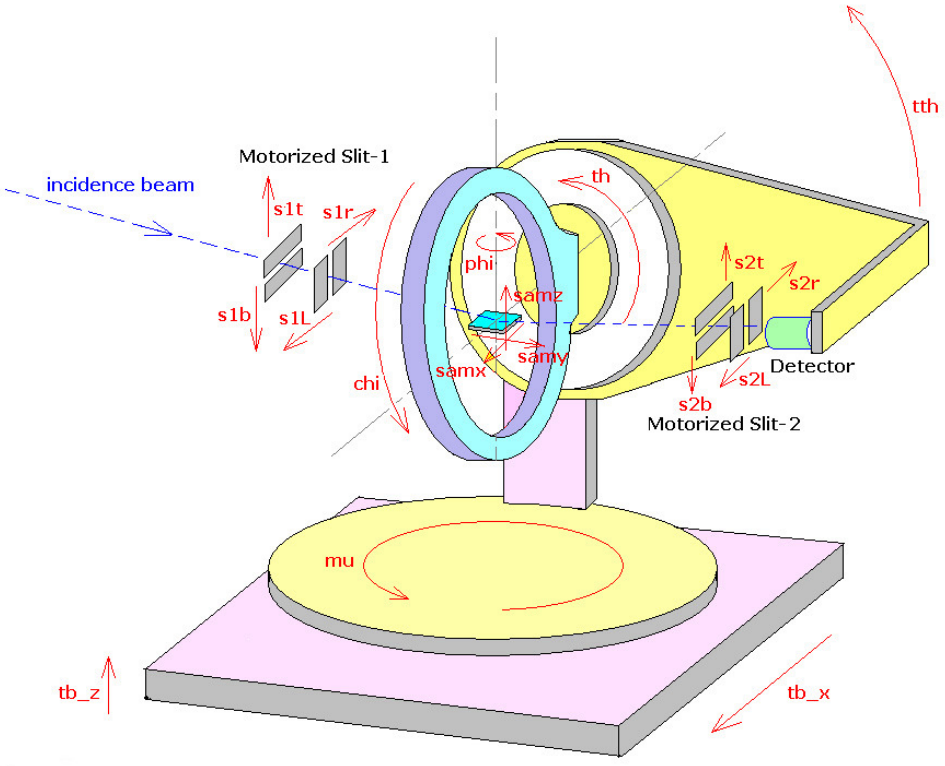
Fedora system (release 9) is used for the goniometer control and windows system for the beam control.

All the goniometers at BL-18B are configured according to the right handed Cartesian co-ordinate system, where + y-axis represents the incoming beam direction.

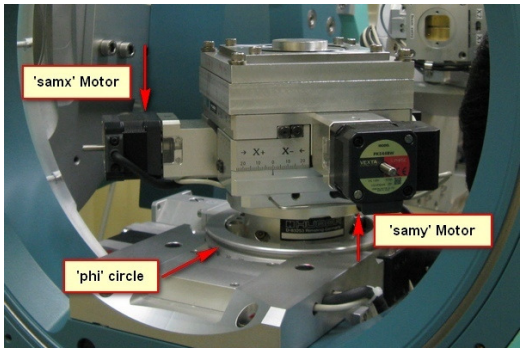
Motors used in *fourc* configuration:

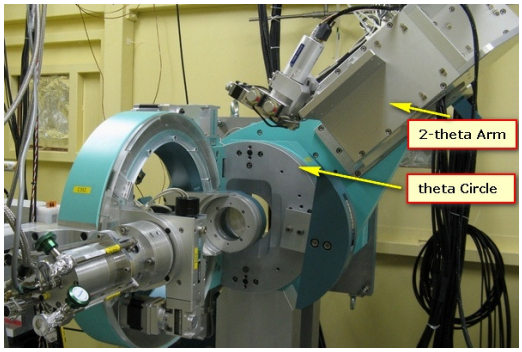
filt	Filter before the ion chamber.
th	Sample rotation around the x-axis
tth	Detector rotation around the x-axis
chi	Sample rotation around the beam direction, about y-axis,
phi	Sample in-plane rotation, rotation in the goniometer axis
mu	Whole goniometer rotation about the goniometer axis
s1slit	all the motors of slit-1 (primary beam slit, including the pseudo-motors for gap and center of the slits)
s2slit	all the motors of slit-2 (diffracted beam slit, including the pseudo-motors for gap and center of the slits).
tb_x	Goniometer table x-motion motor
tb_z	Goniometer table z-motion motor
samx	Sample x-motion motor
samy	Sample y-motion motor
samz	Sample z-motion motor
bm_h	Beam monitor horizontal
bm_v	Beam monitor vertical

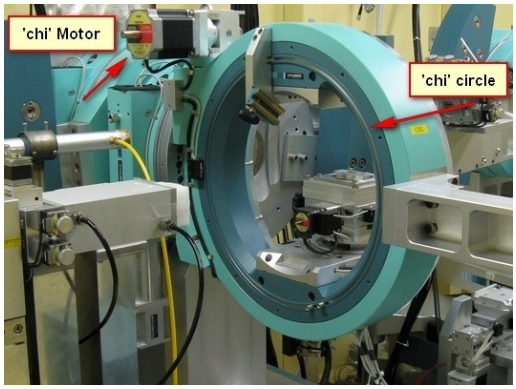
Line diagram of the four circle goniometer

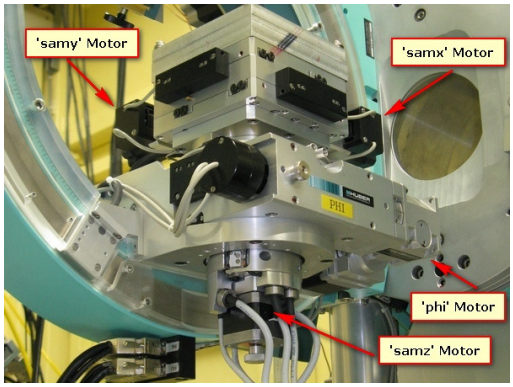


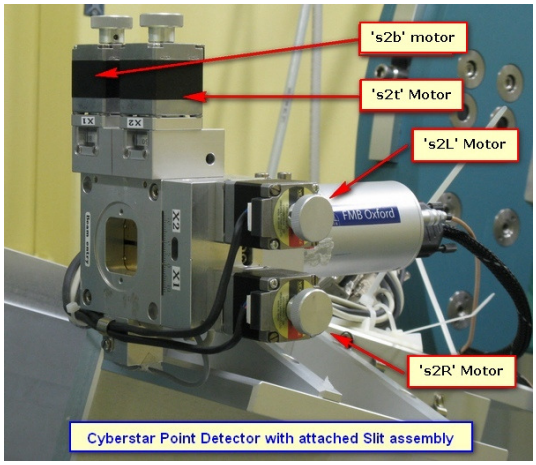
Various motors in the four circle Goniometer

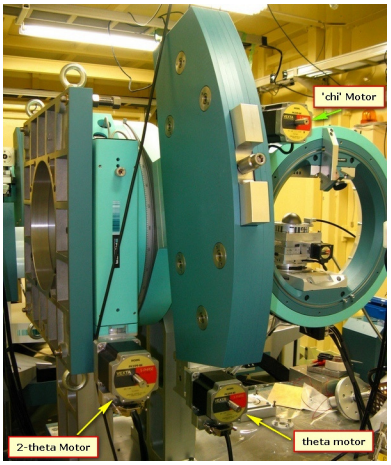


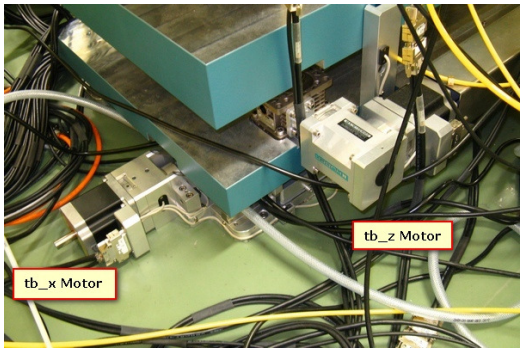


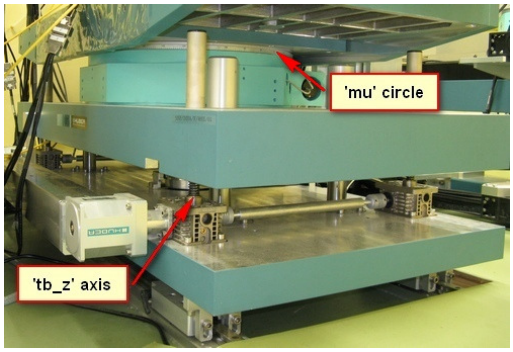






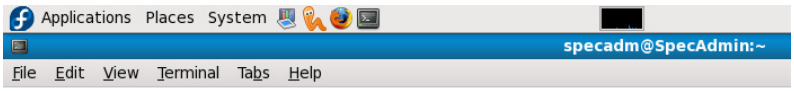






Check position of 4-circle Goniometer motors on SPEC

fourc command '*wa*' will list both the user and dial positions of all the configured motors.



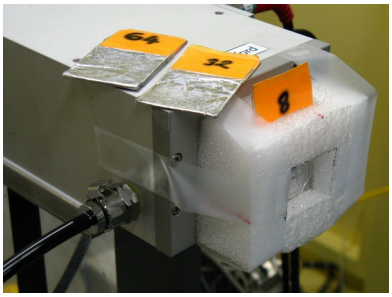

```
603.FOURC> wa

Current Positions  (user, dial)
   2Theta     Theta       Chi       Phi    S1_left  S1_right S1_bottom    S1_top
      tth        th       chi       phi       s1l       s1r       s1b       s1t
   0.0000    0.0000  -90.0000   10.6720    0.4374    1.5626   -1.3586    1.5086
 -39.1794   19.8969    0.0000   10.6720    0.0000    0.0000    0.0000    0.0000
S2_bottom   S2_left  S2_right    S2_top   S1v_gap   S1v_cen   S1h_gap   S1h_cen
      s2b       s2l       s2r       s2t    s1vgap    s1vcen    s1hgap    s1hcen
   0.1000    1.7000    0.3000    0.1000    0.1500    1.4336    2.0000   -0.5626
   0.0000    0.0000    0.0000    0.0000    0.1500    1.4336    2.0000   -0.5626
  S2v_gap   S2v_cen   S2h_gap   S2h_cen   Table_X   Table_Z        Mu  Sample_X
   s2vgap    s2vcen    s2hgap    s2hcen      tb_x      tb_z        mu      samx
   0.2000    0.0000    2.0000    0.7000   -0.0006   -1.2000    0.0000    0.0000
   0.2000    0.0000    2.0000    0.7000    0.0000    0.0000    0.0000    0.0000
 Sample_Y  Sample_Z Beam_mon_ Beam_mon_    Filter   Anal_th  Anal_tth  Motor 23
     samy      samz      bm_h      bm_v      filt      a_th     a_tth      free
   0.0000    0.0000    0.0000    0.0000    0.0000    0.0000    0.0000    0.0000
   0.0000    0.0000    0.0000    0.0000    0.0000    0.0000    0.0000    0.0000
```

Command '*wm*' followed by motor name shows position of that motor.

The arms of the goniometer can be varied to a required position by using the '*umv*' followed by the motor name command.

Never allow the direct beam without proper absorber to fall on any type of detector.



Absorbers currently in use

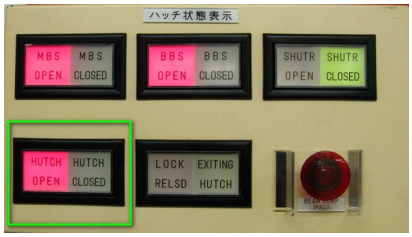
64 - (64 x 20 micron Al foil)
32 - (32 x 20 micron Al foil)
16 - (16 x 20 micron Al foil)
8 - (8 x 20 micron Al foil)

The experimental area is prone to high radiation, thus it is surrounded by a hutch protection. Before starting the any measurement, this hutch should be closed properly (otherwise you are not allowed to do measurements).

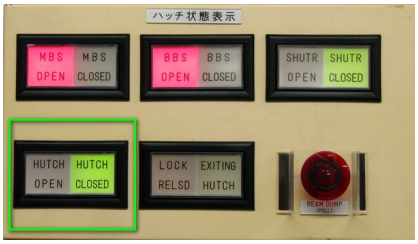
While closing the hutch for beam exposure, press the **out button**, you will get a warning sound with yellow light,

Then, come out of the hutch and close the door lock as soon as the warning sound goes off

Check for the hutch close indicator whether the hutch was closed properly or not

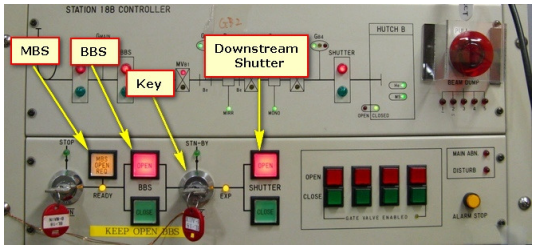


Hutch door is in opened condition

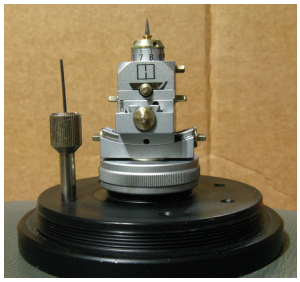


Hutch door is in closed condition

Once the hutch was closed use the same key of the hutch door at the shutter control panel for opening the downstream shutter (SHUTTER) for getting the x-ray beam to carry out an experiment (make sure that the *main beam shutter* (MBS), and the *branch beam shutter* (BBS) are open, before closing the hutch. if MBS and BBS are closed contact the in-charge of experiment).



Aligning the centre of goniometer to the beam centre.



The direct X-ray beam should pass through the center of rotation of the goniometer. The center of rotation should be found with a pin and a telescope.

1 Make the X-ray beam to be perpendicular to the 2θ axis. This is verified by comparing X-ray burns made on X-ray sensitive paper with 2θ near the direct beam direction and with 2θ offset by 180°.

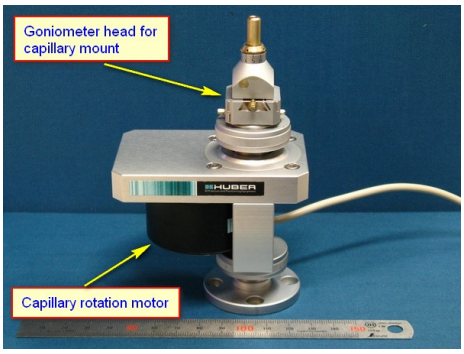
2 Zero of the 2θ must coincide with the direct x-ray beam.

3 The χ rotation axis should be perpendicular with the direct beam direction which defines the zero of θ.

4 The φ rotation axis should coincide with the θ rotation axis to set the zero of χ.

Mounting of powder sample in capillary tube.

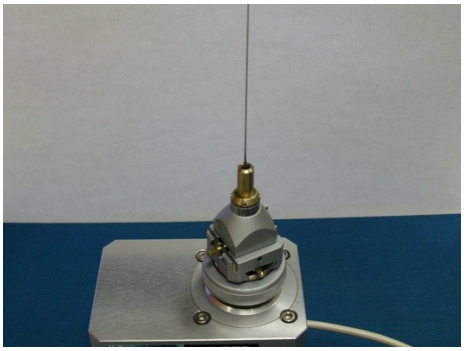
Capillary tubes of 0.5 mm or less (preferably 0.3 mm best choice) diameter should be used in case of powder samples (capillary tubes larger than 0.5 mm should not be used). Capillary should be made of special glass (low x-ray absorption glass) for measurements at ambient conditions.



As most of the modern diffraction instruments are highly accurate, total result of a diffraction experiment depends on how well the sample was prepared. The accuracy of the powder diffraction data is influenced by grain size distribution of powder sample. The larger grain results in inhomogeneity of intensity distribution between different Bragg reflections. On the other hand, the smaller grain yields broadening of diffraction profiles. It is well known that the optimal particle size is a few micrometers, optimal particle size should be maintained for obtaining good data (best particle size ~2μm). In order to achieve particle size of this range, the sample powder should be finely and uniformly ground and separated from the larger particles. For separating particles of uniform size from random distribution of sizes there exist several way such as liquid separation, sieving etc. Normally liquid separation is the best for x-ray measurements, this can be done by dispersing the fine particles in non-soluble liquid under sonication and separating different layers of the liquid containing particles of different size and dry them out.

The sample powder with sufficient particle size and distribution will be loaded in to a capillary tube. There exist modern equipment for loading capillary tubes, however, sample can be loaded in to the capillary with the help of ink filler or by vibrations using a small fine file. The capillary should be uniformly packed with the sample, without gaps and closed with wax. If the samples are air sensitive the above process should be done in a glove box and the capillary should be sealed. It can happen some times that the sample may be sticky and the filling of the capillary becomes difficult, in such a cases sample can be adhere to the outer walls of a thin capillary <0.3mm coated with vacuum grease or Vaseline.

Mounting of the capillary tube on the rotation stage



The capillary should be mounted on the rotation stage of the goniometer using wax (if there exists no special arrangement for mounting) such that its length should coincide with the φ axis of the goniometer (which will be at the center of the direct beam). If the capillary is not aligned with the φ axis, it will wobble about the φ axis when the rotation motor is on, to check for this a laser mounted in line with the direct beam or a microscope should be used and adjusted accordingly.

Mounting of crystal

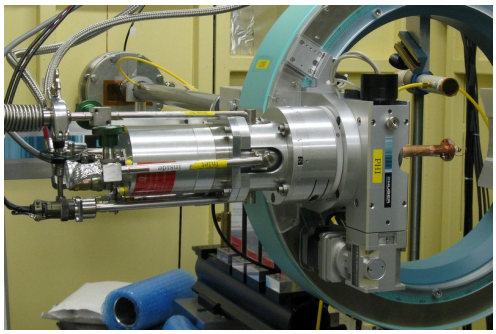
Single crystal can be fixed to the small goniometer head with wax, which will be mounted on the φ circle of the four circle goniometer. However align the sample co-ordinated axis to the goniometer axis one should calculate the orientation matrix, this can be done with the help of work by W. R. Busing. et. al. (Acta Cryst. 22 (1967) 457) or D. P. Shoemaker. et. al. (Acta Cryst. A26 (1970) 97).

Mounting of thin/thick films on substrate

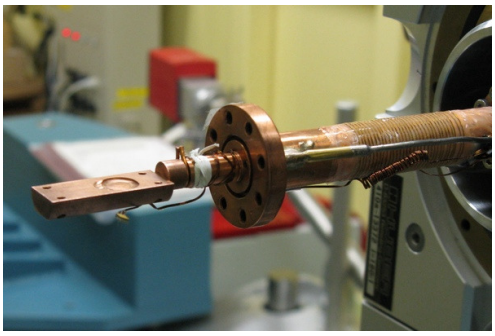
Thin/thick films on substrate can be attached to the sample stage with X-, Y- and Z-motion with the help of double side tape. The film should be aligned to the direct beam before doing any measurement.

Mounting samples on Low Temperature Setup

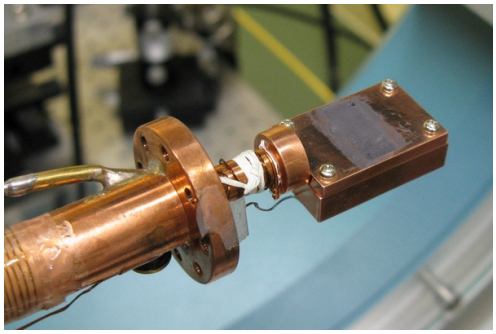
CCR cryo-head should be loaded on to the *phi*-stage keeping *chi* at -180°. The slot on the cryo-head should match with the notch on the *phi* circle. The cryo-head should be attached at this position with the larger circular ring nut from the down side. After fixing the cryo-head to the *phi*-stage bring back the *chi* to -90° to carryout the experiments.



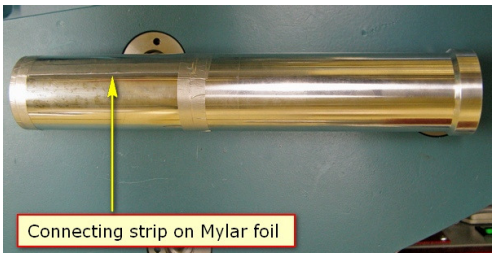
Low temperature setup loaded on the four circle goniometer.



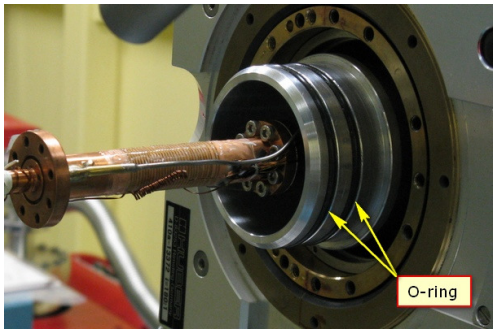
Small sample mount for low temperature setup



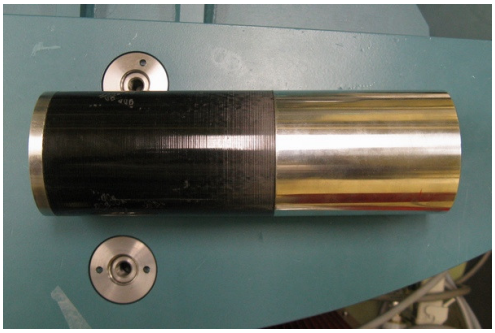
Larger sample holder for low temperature setup



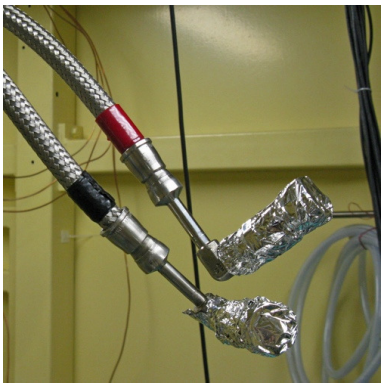
Heat radiation shield (Mylar foil coated with Al)

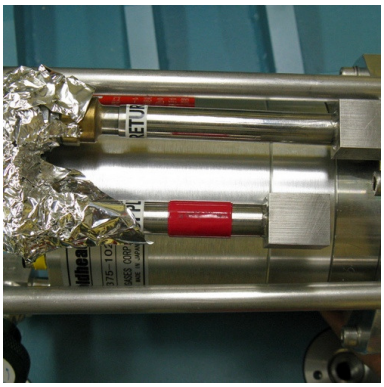


O-rings

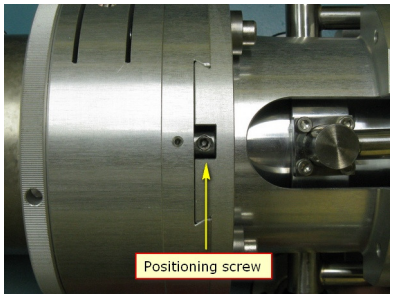


Graphite vacuum shield





CCR outlet and inlet using the red markers on the tubing to the cryo-head



Positioning screws with the locks on four sides of the cryo-head align the sample

CCR compressor

The cryogenic temperature controller used with the low temperature setup is Cryo Con Model 32,

Please refer to the quick start guide for the same, which is available along this manual and at http://www.cryocon.com/M32ProdFolder.asp.

Load the sample on the sample mount with proper packing, so that it will not fall out during attaching the sample mount to the cryo-head, evacuating the cryo-head and operation of the CCR. Attach the radiation shield (Mylar foil coated with Al) over the sample such that the connecting strip on the Mylar foil stay out of the beam path during the experiment. Load the graphite vacuum shield over the O-rings on the cryo-head

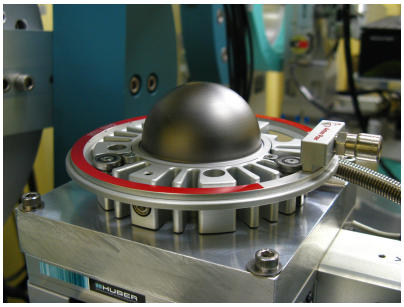
(care should be taken to avoid physical damage to the radiation shield and the vacuum shield while handling). Attach the CCR outlet and inlet using the red markers on the tubing to the cryo-head. Attaching the signal cable, the power cable and vacuum hose to the cryo-head. Evacuate the cryo-head using the turbo-pump by slowly opening the vacuum valve between the pump and the cryo-head (care should be take to prevent sample falling while evacuation). After attaining good vacuum, turn on the CCR compressor kept on top of the hutch. Use the positioning screws with the locks on four sides of the cryo-head align the sample w. r. t the direct beam keeping sufficient absorbers.

Temperature Range:

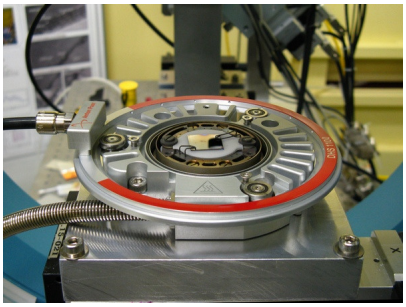
10 °K CCR
4 °K CCR with exchange gas

Mounting samples on High Temperature Setup

Replace the top Al plates generally used for thin film mounting on top of the sample stage with X-, Y- and Z- motion with the one with four holes suitable for mounting the high temperature cell. Mount the high temperature cell on top of the Al plate.



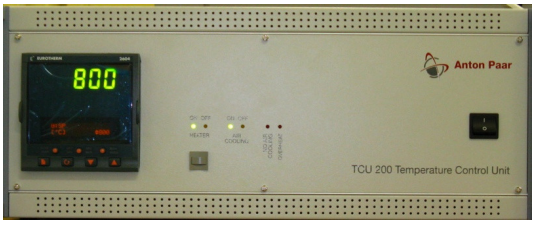
High temperature setup mounted on the four circle goniometer



High temperature setup loaded with the thin film without the graphite dome

Connect the power cable and the sensor cable from the temperature controller to the vacuum/gas hose of the cell. Connect the black tube between the temperature controller and the cell for compressed air cooling. Attach the compressed air hose to the back of the temperature controller. Switch on the compressor along with the cooler on top of the hutch and then switch on the heater which controls the compressed air flow.

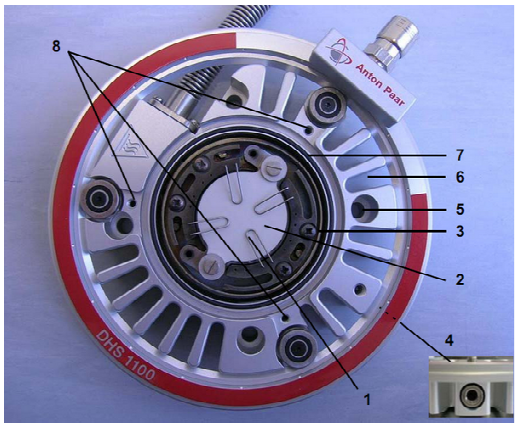
Air compressor for high temperature setup from Anest Iwata



High temperature controller

The dome is made of graphite, it should be handled carefully to prevent from mechanical damage. Aluminium base of the graphite dome should be used to carry the dome with a pair of tweezers. The sample should be fixed to the heater by the Inconel clamps.

In case of powder samples, load the sample on the quartz grow covered with cover plate, which in turn clamped with Inconel clamps to the heater.



Different internal parts

1. Springs for fixing the sample
2. Sample plate
3. Temperature shieldings
4. Pressure relief valve
5. Bore holes for mounting DHS-1100
6. Cooling fins
7. O-ring for the dome
8. Fixing screws for the dome

Bore hole for different connectors (showed on right side picture)

1. Blind plug
2. Feed through adapter
3. Pressure relief valve
4. Hose connector

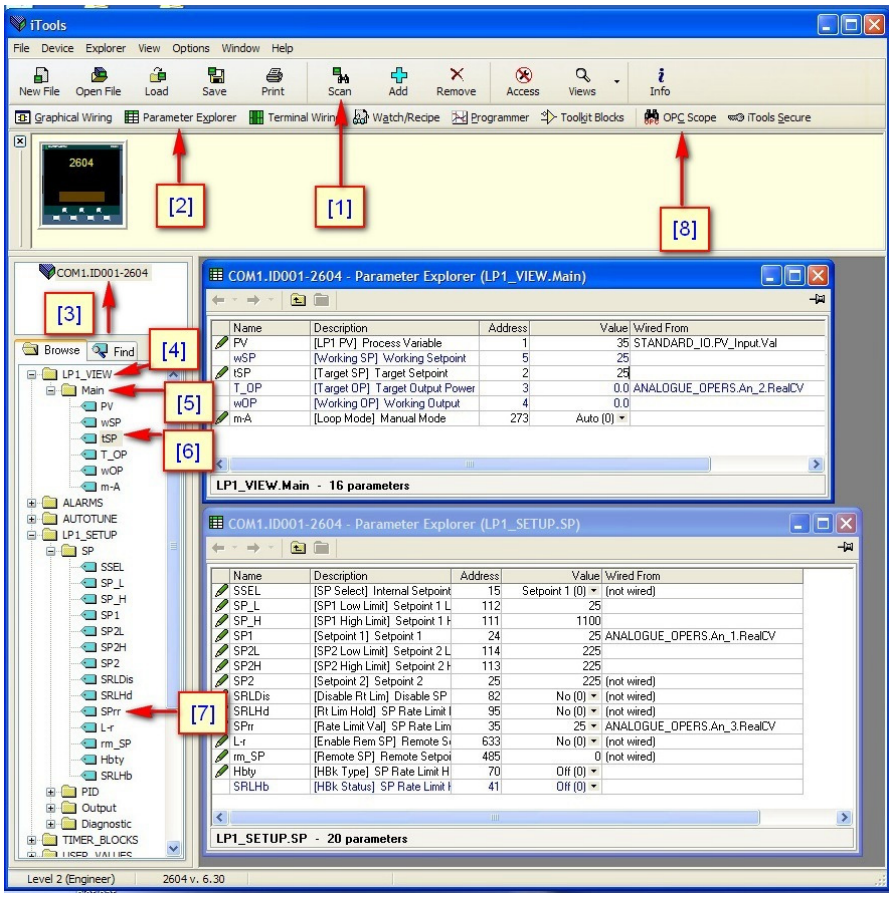
Attach the graphite dome to the heater after loading the sample. If you want to evacuate the sample chamber, use blind plug in the side grow and connect the vacuum/gas hose to the vacuum pump. If want to use the any protective gas or air use the pressure relief valve in the side grow with the help of a suitable Allen key and connect the vacuum/gas hose to the gas supply.

Temperature range:

With Air, N_2, vacuum or inert gas	25 to 1100 °C
With Helium	25 to 1000 °C

RS - 232 cable of the temperature controller should be connected to the windows system, meant for the beam control. From this computer the temperature controller can be accessed using iTools software. Following commands are useful for operation of the controller from the computer.

Open the iTools … software



[1] Press the scan tool in the toolbar for the accessing the temperature controller address.
[2] Go to parameter explore.
[3] Select the device ID001-2604.
[4] Go to LP1_VIEW.
[5] Go to main, you will get the PV, SV, etc. display on the right panel.
[6] To change the set point go to LP1_VIEW then to Main then to tsp (double click), enter the desired value.
[7] To change the set point rate limit (SPrr) go to LP1_SETUP then to SP then to SPrr, enter the desired ramp rate of heating.
[8] For online display of the PV, go to OPC Scope, select the device (ID001-2604) on the left panel then go to LP1_VIEW then to main then to PV, select chart for the parameter

To auto adjust the sample to the beam center in case of using XYZ-sample stage

`umvr samz -4`	to reduce the sample height by 4 mm from the current position
`ct`	to check the counts, whether the sample is stopping the beam (if sample stops the beam move it until it is out of the beam path)
`lup tth -0.15 +0.15 50 1`	To measures the photon count in the 2θ range of ±0.15 degrees about the current position, in 50 steps for a dwell time of 1 sec. [You can find the shape of the beam and find the center of the beam.]
`p CEN`	Gives you the center of the peak
`umv tth CEN`	aligns 2θ at center position of the direct beam
`wm tth`	gives the current position of the 2θ
`set tth 0`	makes the current position as zero of 2θ
`umvr samz 4`	bring back the sample into the previous position
`ct`	Gives monitor and detector count for 1 sec.

`lup th -0.5 0.5 25 1`
`or`
`thcen_coarse`
	To find the maximum count w.r.t θ movement (to align the θ w. r. t direct beam.

`p CEN`	Gives you center of the above scan
`umv th CEN`	move to the center
`set th 0`	To set the zero of the θ
`ct`	get the count
`zcen_coarse`	center the sample
`wm samz th tth`	get the positions of z of the sample, θ and 2θ

To start a fresh measurement

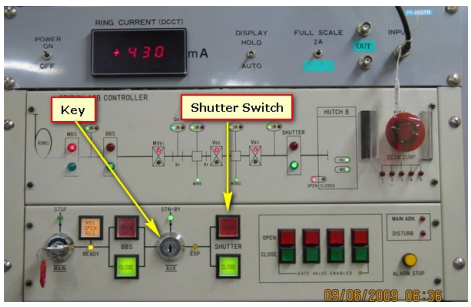
`newfile`	type file name
`a2scan th 5 30 tth 10 60 2500 1`	to give a scan of θ: 5°-30° and 2θ:10°-60° in 2500 steps with a dwell time of 1 sec

After measurement bring back θ and 2θ back to a safe position by (θ = 15° and 2θ = 30°) for the next measurement or keep the detector away from the direct beam, if there is no further measurement.

After completion of the experiment

Close the downstream shutter,

Take out the key from the shutter control panel and open the hutch.



Procedure for transferring the collected data.

Kindly collect your data before leaving form the beamline with help of beamline scientist/in-charge. Normally the data will be provided in standard ASCII format, however data can be provided in user required format depending on the availability of the software for conversion to that format. Users are advised to bring CD/DVD for collecting data (as USB flash drives, etc. may have chances of failure and transferring virus threats).

Preliminary analysis of the data.

After completion of the data collection compares the data with the standard reports (like JCPDS cards in the case of powder diffraction), look for any discrepancies and report to the beam scientist/in-charge. Check for the intensity distribution between the Bragg peaks and FWHM.

You can find some of the useful analysis softwares in the computer above the hutch, where you can do preliminary analysis. Powder diffraction users can use PCW23 for this purpose or any other software of their choice (us there are many software's for this purpose).

Note: users can find open source software's of their choice and other licensed ones in http://www.ccp14.ac.uk/ or in the book:

Format of report to be submitted by the users at the end of the experiment

Form can be found in the KEK website. Please submit the filled in form to the in-charge of your experiment.