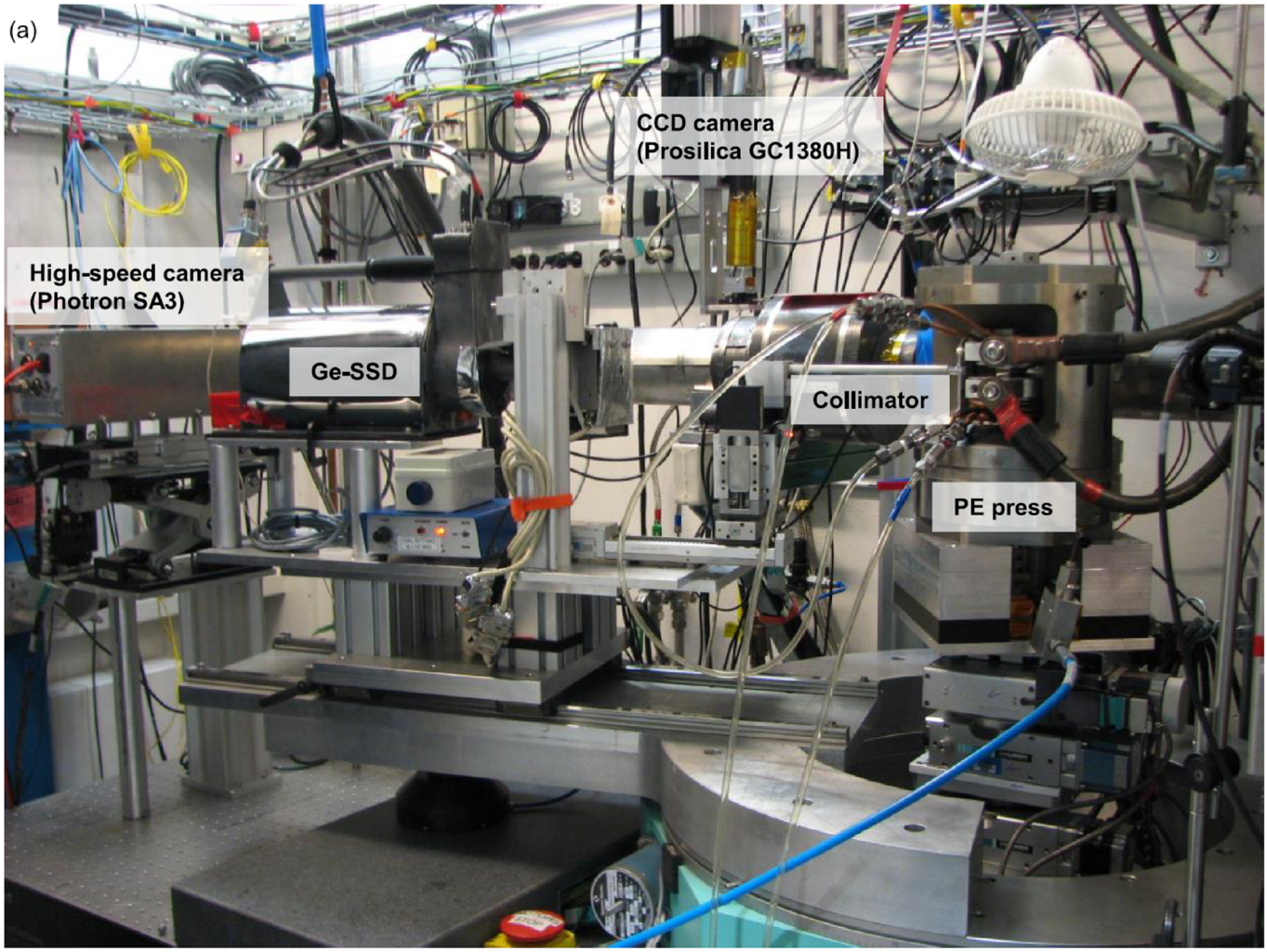
16 BMB PE Press Experiments User Manual



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Chapter 1

Beamline description

The HPCAT 16BM-B beamline is dedicated to white beam x-ray diffraction and radiography researches of matters under high pressure.

With the APS bending magnet white x-rays (5-120 keV), multi-angle energy dispersive x-ray diffraction (EDXD) technique is extensively utilized to obtain the structure factors up to Q=around 20 $Å^{-1}$. A Paris-Edinburgh Cell (PEC) (250 ton capability) is equipped for samples requiring a large scattering volume, especially, for the high-temperature melt and amorphous structure. The range of pressure and temperature is up to 7 GPa and 2500K. The PEC setup includes white beam radiography system, which allows the structure measurement with EDXD to be combined with radiographic volumetry, ultrasonic sound velocity measurement and/or falling sphere viscosity measurement.

Beamline Contact

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Table 1. Summary of 16BM-B features

Feature	Description
Source	bending magnet
Monochromator	N/A, white beam
Energy range	10-120 keV for parallel beam; 10-65 keV for focused beam
Beam size & focusing optics	50 μm x 50 μm to 2 mm x 2 mm for parallel beam (slit-limited) 10 μm (H) x 10 μm (V) FWHM with 200 mm Pt-coated Si KB-type mirror
Established techniques	Multi-Angle Energy Dispersive X-ray Diffraction White Beam Radiography Ultrasonic elastic wave velocity measurement Falling sphere liquid viscosity measurement
Detectors	Ge Solid State Detector, CCD camera (Prosilica GC1380H), High-speed camera (Photron FASTCAM SA3)
Support equipment	Paris-Edinburgh type large volume press with resistive heating capacity (PE anvils, boron-epoxy gaskets, cylindrical graphite heater, 8V-220A power supplier; temperature and pressure range up to 2500 K under and 7 GPa)

Chapter

2

Beamline operations for Paris-Edinburgh cell experiments

Login

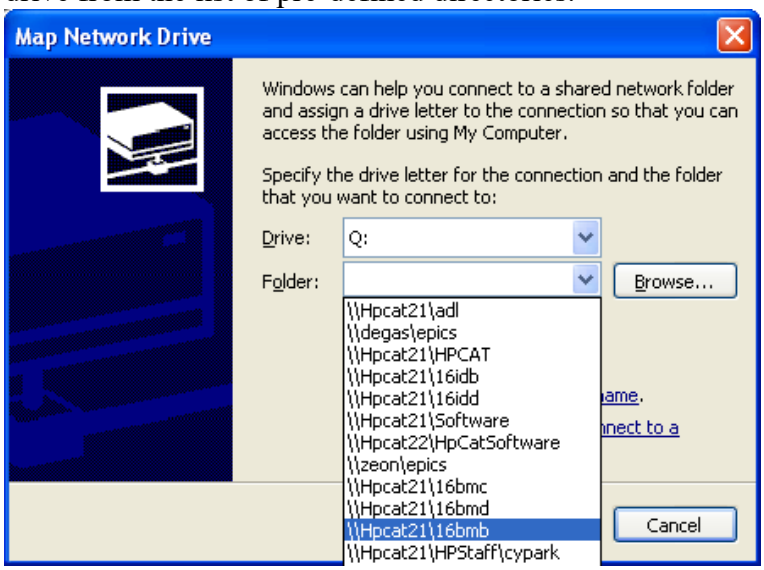
Both beamline control computer and data analysis computer (Windows XP Professional operating systems) has identical login id and password for users:

login: **16bmb_user**
password: **bmbuser@16**

This login id and password will be required when re-logging in the computers (e.g., after rebooting the operating system). The login name and password is valid to access the network folder \\hpcat21\16bmb.

Map the network drive

If you do not see the network drive named "16bmb on Hpcat21" in My Computer folder, please map the drive from the list of pre-defined directories.

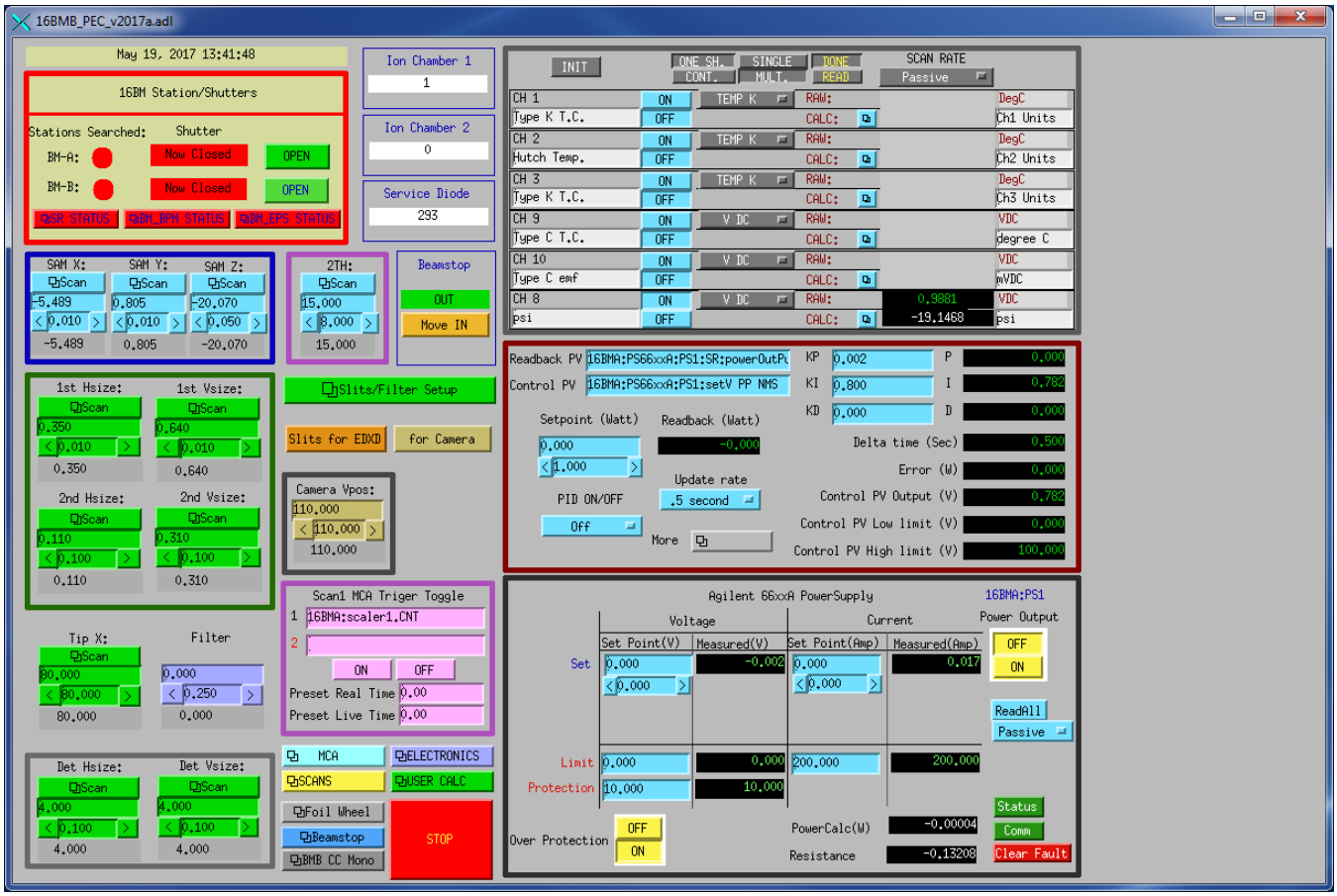


How to start EPICS user interface

On the Windows desktop of both computers, users will find a shortcut named “16BMB-PEC Interface”.

This shortcut executes “16BMB_PEC.adl” with necessary options and input parameters. Please keep the options as-written to correctly start the interface.

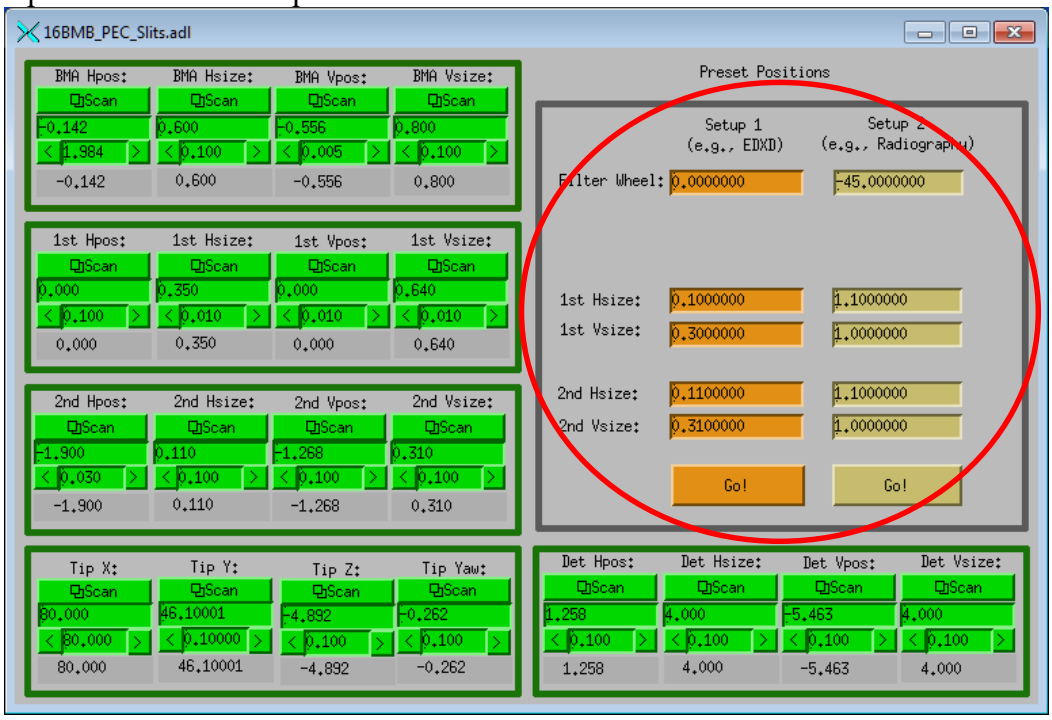
Attached below is a screen shot of the PEC User Interface.



It interfaces the control widgets with 16BM station shutters, SR status, EPS status, most demanded motors for user experiments, intensity monitors with ion chambers and diode, and power supply controller.

Setup of slits and filter for EDXD and radiography measurement, respectively

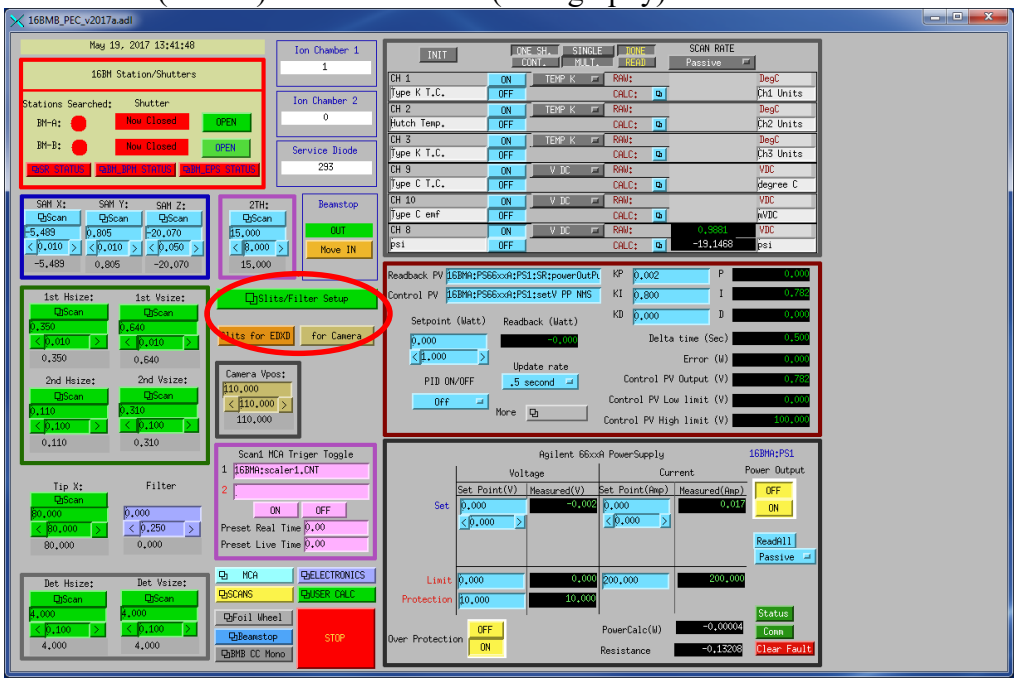
Open ‘Slits/Filter Setup’ on the PEC user interface.



Input slit sizes and filter setup values in the ‘Preset Position’ window. **Please do not change other parameters (e.g., Tip X, Y, Z, and so on).**

Setup 1 is for EDXD measurement and setup 2 is for radiography measurement. Filter value is typically 0 (no filter) for EDXD measurement and -45 (100 μm molybdenum) for radiography measurement. Please close the window after completion.

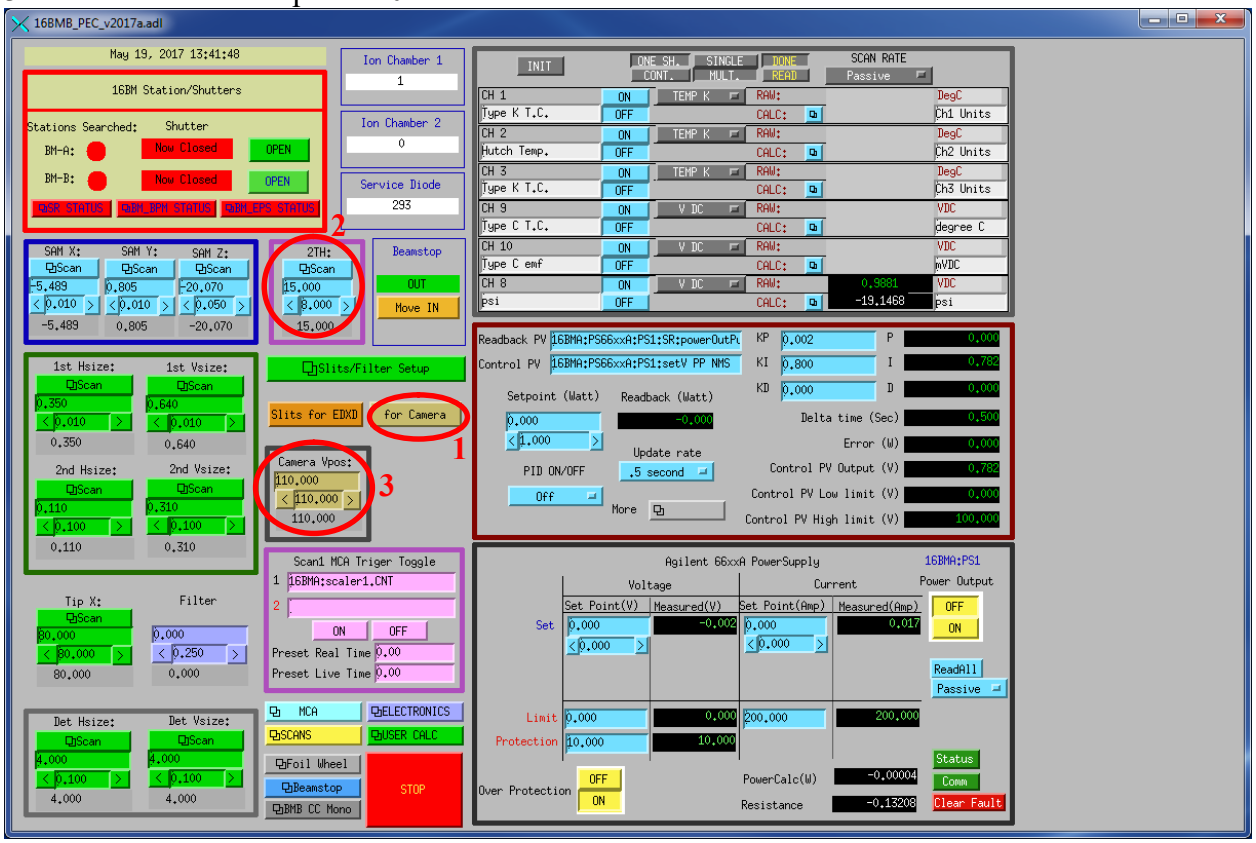
‘1st Hsize’, ‘1st Vsize’, ‘2nd Hsize’, ‘2nd Vsize’, ‘Filter’ setups change simultaneously by clicking ‘Slit for EDXD’ (EDXD) or ‘For Camera’ (radiography).



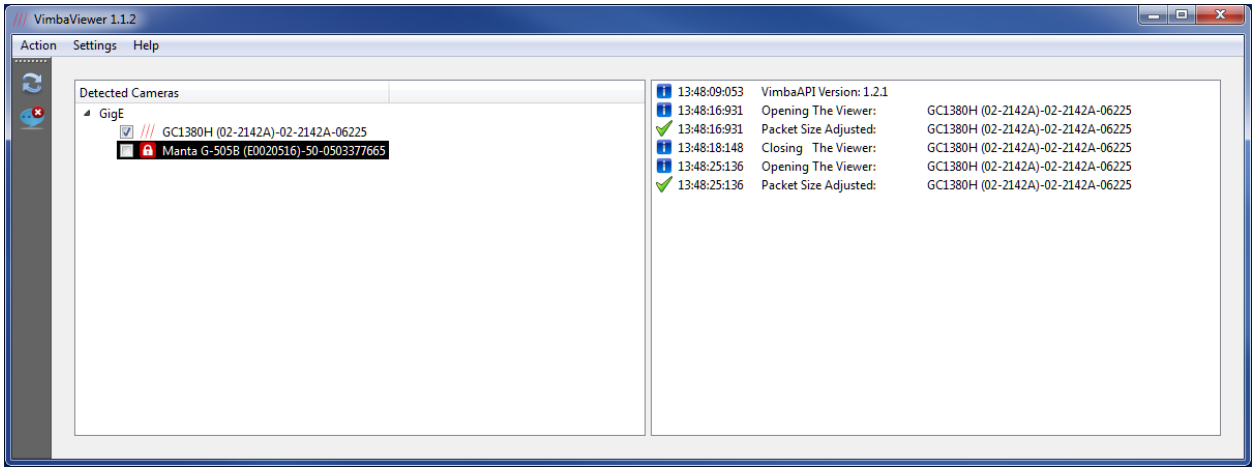
Radiography measurement

On the PEC user interface,

1. Click ‘for Camera’ to open slits and to put filter.
2. Confirm and/or move ‘2TH’ to >15°.
3. Move in ‘Camera Vpos’ to 0.



Open ‘AVT Vimba Viwer’ on desktop.
Check ‘GC1380H’.



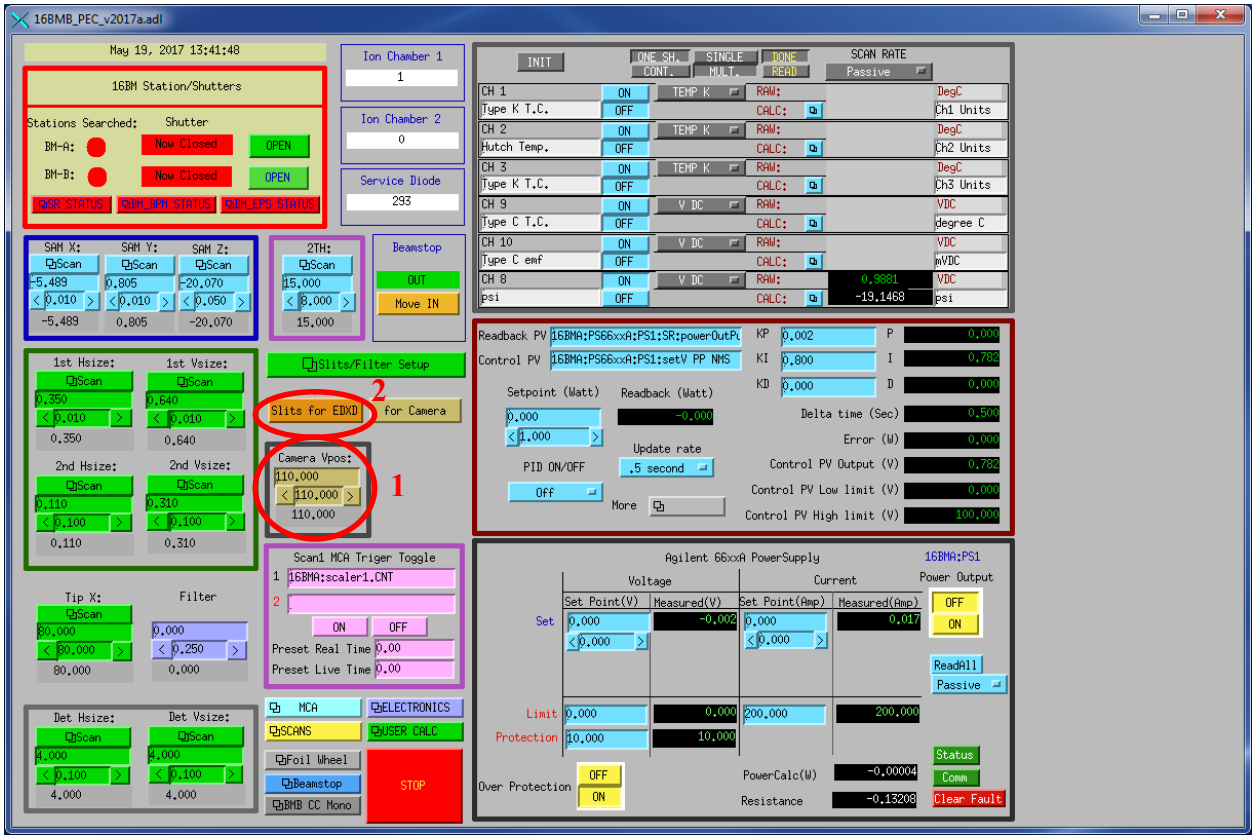
Click start to start camera.

To save image, stop camera.
File-Save image as.

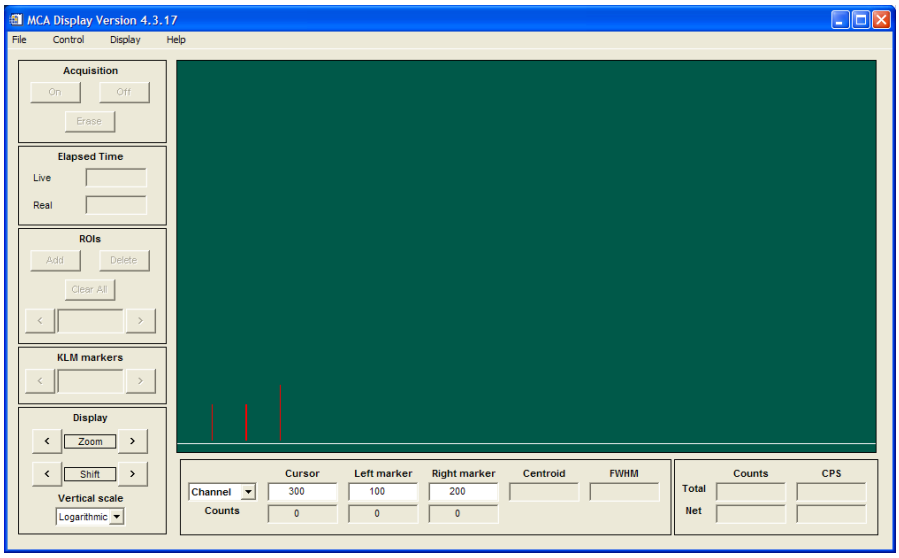
Energy-dispersive x-ray diffraction (EDXD) measurement

On the PEC user interface,

1. Move out ‘Camera Vpos’ to 110.
2. Click ‘Slits for EDXD’ to narrow slits and to remove filter.



-Open ‘mca’ from shortcut on desktop



-File\foreground\open detector.
-Click ‘OK’ without any change.

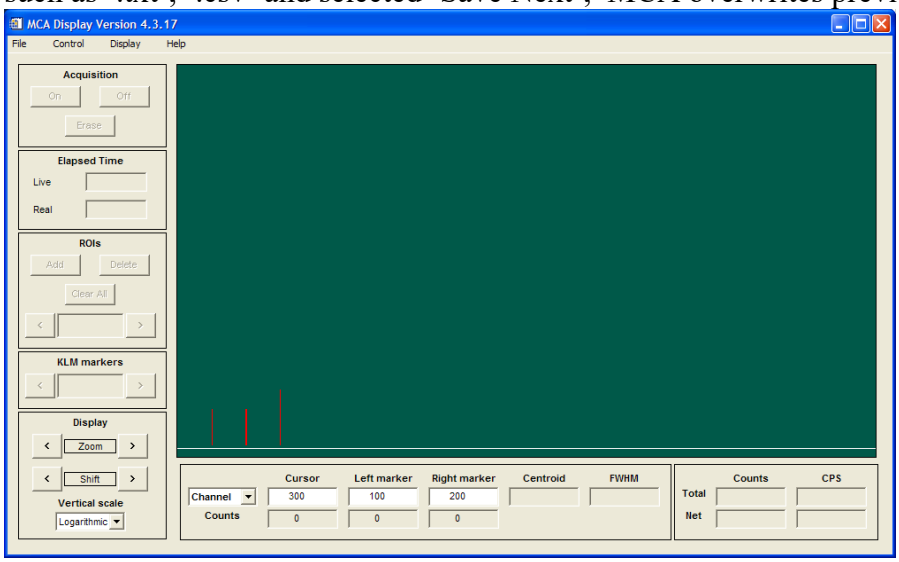
-Find sample Y, Z, and X positions before starting EDXD data collection (cf. page 20-21).

-Start EDXD data acquisition (cf. following pages for the usage of MCA software).

MCA

Data acquisition

-To start data collection, press ‘Erase’ and then ‘On’ in ‘Acquisition’.
-To stop data collection and to save, press ‘Off’ in ‘Acquisition’. Then, File\Save As. **Note:** Please use extension ‘.000’ to avoid mis-overwriting by a command ‘File\Save Next’. If you use other extension such as ‘.txt’, ‘.csv’ and selected ‘Save Next’, MCA overwrites previously saved file.



Other features on the window

Left column (from top to bottom):
-ROIs (see following pages for the usage).
-KLM markers=By selecting element, MCA shows K and/or L shell emission lines positions.
-Display (Zoom, shift, and vertical scale).

Bottom:
By selecting a position on EDXD data by ‘Cursor’, ‘Left marker’ or ‘Right marker’, you can get Energy and Counts information.

Cursor: Left click
Left marker: Wheel click
Right marker: Right click

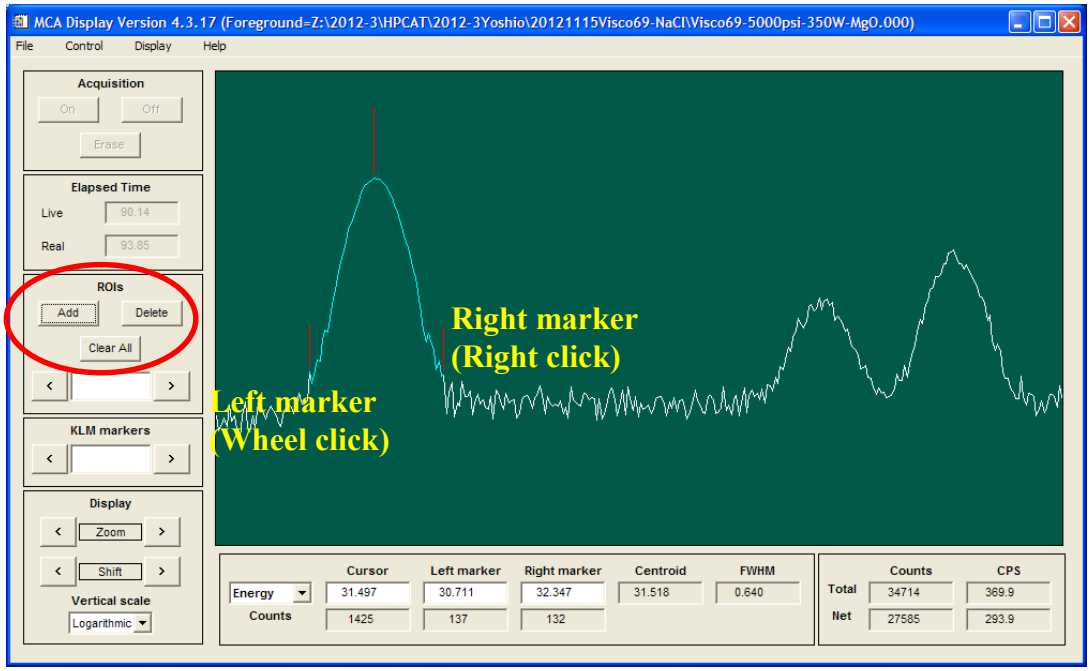
Region of Interest (ROI)

There is two methods to add ROI.
(1) Manual selection of ROI.
(2) Make ROI on all peaks for a crystal by using the JCPDS data.

(1) Manual selection of ROI

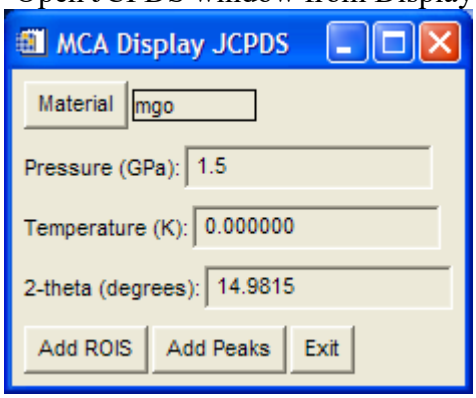
-Select region of interest by ‘Left marker’ and ‘Right marker’.
-Then, click ‘Add’ button in ‘ROIs’ window.
-Selected area will be blue line.

-To erase a ROI, please click ‘Delete’ after selection of the ROI.
-To erase all ROIs, please click ‘Clear All’.

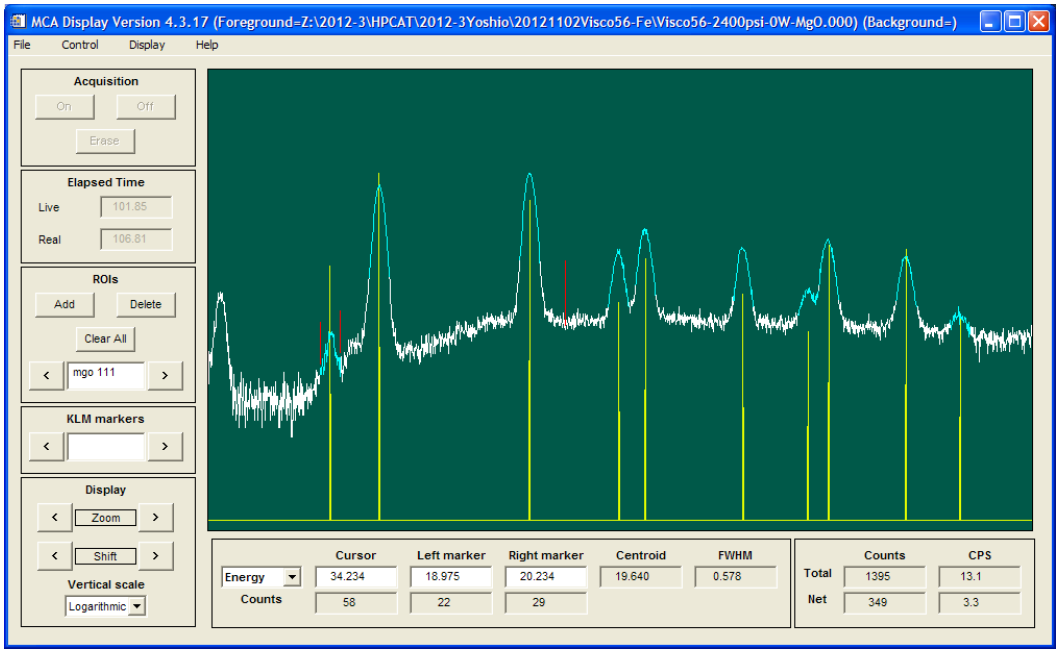


(2) Make ROIs on all peaks for a crystal by using JCPDS data

-Open JCPDS window from Display\jcpds.



-Select material by opening a jcpds file (JCPDS data is at C:\\MCA_BMB).
-Input 2-theta angle.
-Yellow lines, which indicate positions of the peaks of the material, appear below EDXD data.

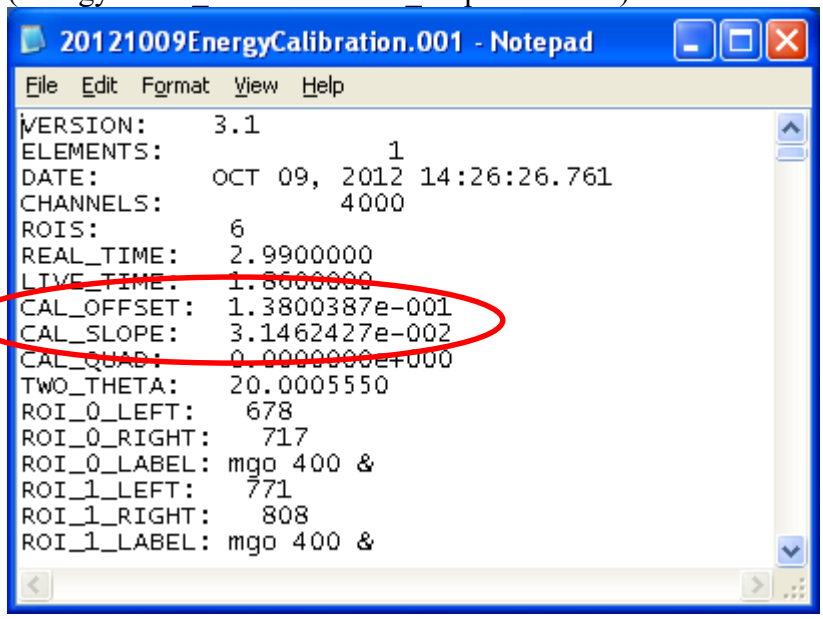


-The positions of yellow peaks can shift by changing ‘Pressure (GPa)’.
Note: Temperature (K) is currently not working well. If you want to shift peaks for high temperature data, please type negative value of pressure.

-Then, click ‘Add ROIS’ in JCPDS window to add ROIs for all peaks.

Energy calibration

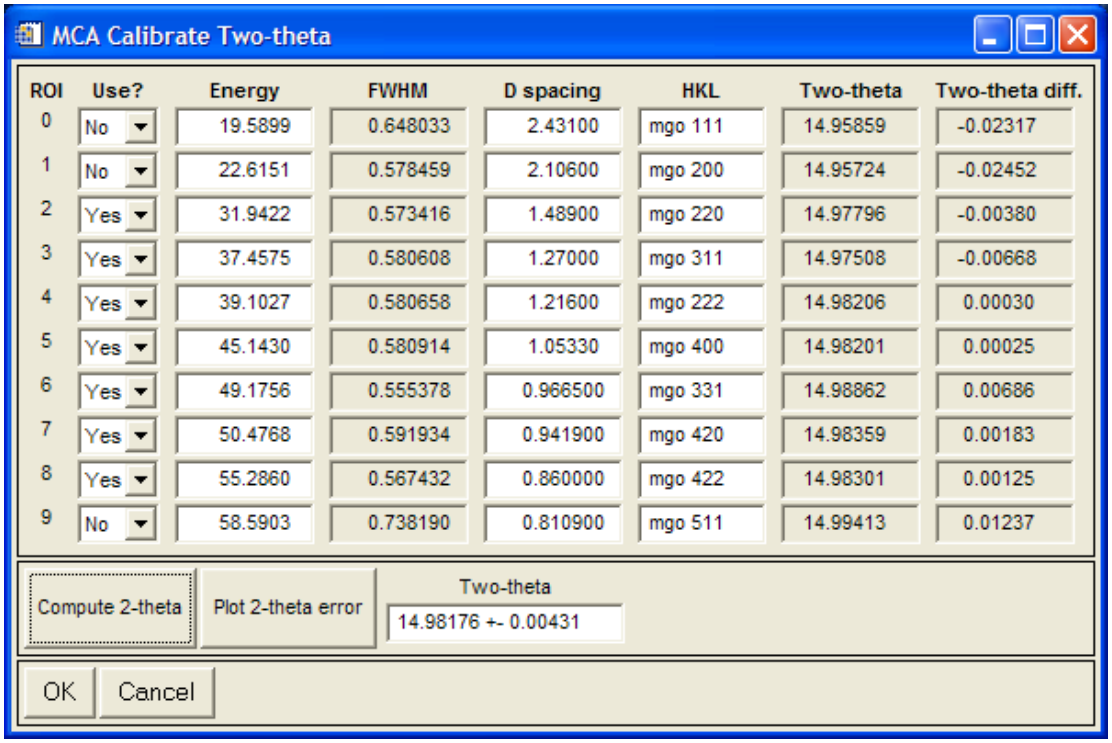
Beamline scientist does energy calibration of the germanium solid state detector by using Fluorescence lines of silver at 22.104 keV (Kα) and 24.942 keV (Kβ1), and gammas from ^{109}Cd (88.04 keV) and ^{57}Co (122.10 keV) at the beginning of each beamtime cycle. Parameters of energy calibration (Energy=CAL_OFFSET+CAL_Slope×Channel) can be found in the header of the EDXD data file.



2theta angle calibration

Beamline scientist does 2theta angle calibration at 7°, 15°, 23°, and 31° using unit-cell volume of Au, and make linear equation to calculate 2theta angle.
2theta angle calibration for MgO ring in PE cell at ambient pressure may provide better pressure calculation with internally consistent ambient condition calibration. Followings are procedure of 2theta angle calibration:

-Collect MgO EDXD pattern.
-Make ROIs for all MgO peaks using JCPDS data at 0 GPa (cf. page 13).
-Select Control\Calibrate 2theta on Menu bar.
-Please remove weak or overlapping peaks by selecting 'No' in the second column 'Use?'.
Note: Because the MCA does not have background subtraction feature, background slope at low energy (<~25 keV) probably due to absorption by cell assembly influences on determining peak position. It is better not to use low energy data for 2theta angle calibration. Typically, at 2theta of ~15 °, the first and second peaks show marked deviation from other peaks.
-Click 'Compute 2-theta'.
-2theta value appears in the 'Two-theta' box.
-Then, please click OK to apply the 2theta calibration.
The 2theta calibration result is also saved in the header of the data file.

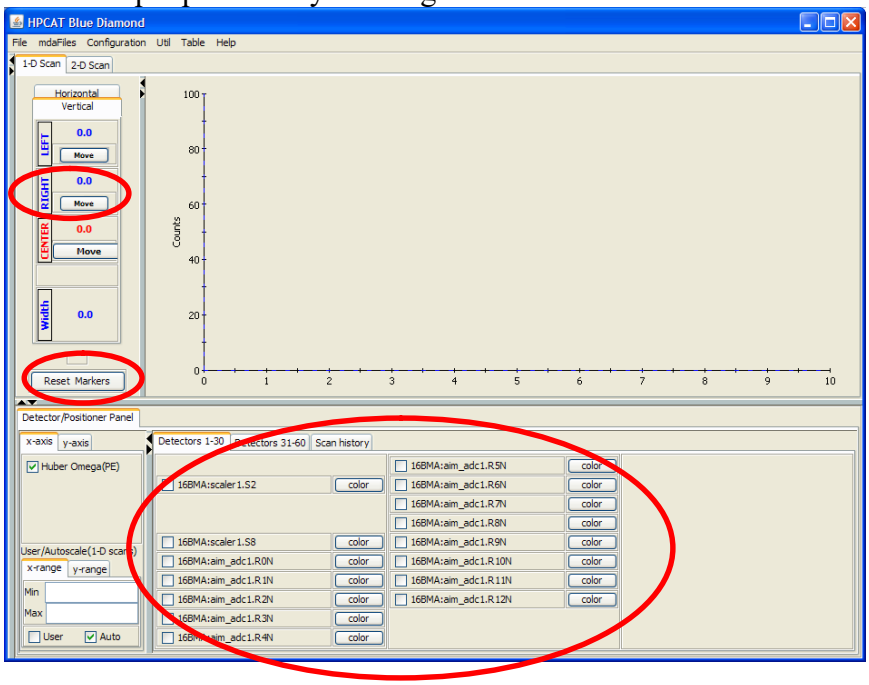


Bluediamond

The Java-based **HPCAT Bluediamond** software is a real-time scan viewer program. The user shortcut can be found on the Windows desktop, which executes "java –jar C:\HPCATSoftware\bluediamond.jar". If the software is started fresh, go to "Configuration"-> "open" to open the input configuration file named "16BMA.txt" in the "C:\HPCAT Software" directory. Note that this directory is local, but can be any directory in the network. The software is straightforward to use and most of the menu items are self-instructing.

Detector
-16BMA:scaler1.S2=Beam intensity monitor by 'Ion Chamber 1' placed at the entrance of BMB hutch.
-16BMA:scaler1.S4=Beam intensity monitor by 'Ion Chamber 2' (used only in liquid density measurement).
-16BMA:scaler1.S8=Beam intensity monitor by 'Service Diode' placed at the downstream of sample. This is mainly used for scanning sample Y and/or Z position by absorption contrast.
-16BMA:scaler1.R0N-R12N=Intensity of ROI in MCA software. This is mainly used for scanning sample X position in EDXD measurement (page 21).

To use line cursors (two vertical and two horizontal), click 'Reset Marker'.
The cursor feature is useful for graphical determination of the FWHM and peak center position. You can move sample position by clicking 'Move' button at the 'Center' in the left column.



Sample Y and/or Z positions search

There are 2 ways to search sample Y and/or Z positions:

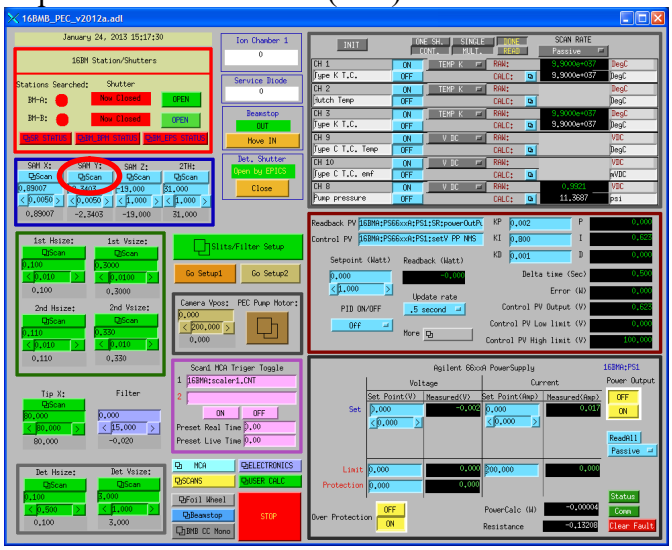
(1) Search by radiography image
(2) Scan absorption profile

(1) By radiography image

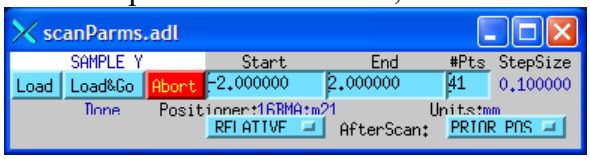
-Move to radiography measurement setup (cf. page 8).
-Narrow slit size to those of EDXD measurement.
-Mark the narrow slit size and position by tape or something on monitor.
-Open slit (click 'for Camera') for radiography measurement.
-Move sample position to x-ray beam position shown by a mark on monitor.

(2) By scan

-Move to EXDX measurement setup (cf. page 9).
-Confirm 2TH is >10°.
-Open 'Bluediamond' (cf. page 16 for Bluediamond software).
-Select '16BMA:scaler1.S8' in Detector in Bluediamond.
-Open 'scan' on SAM Y (or Z)



-Set scan parameters of 'Start', 'End' and '#Pts' (#Pts has to be odd number) (confirm 'Relative').



-Click 'Load&Go' to start scan.
-Scan results will appear in 'Bluediamond' window.

Sample X position scan

Sample X position can be adjusted by using intensity of sample or diffraction pattern. However, it is difficult to scan sample X with diffraction intensity of amorphous material. We recommend scanning sample X by using diffraction intensity of MgO ring. Followings are procedures:

-Move Y -1.5 mm from sample Y center to see diffraction patterns of MgO.

-Add ROIs for MgO peaks.

-Then, move Y -1.3 mm position from sample Y center (+0.2 mm Y from -1.5 mm position or move back to the sample Y center and move -1.3 mm Y).

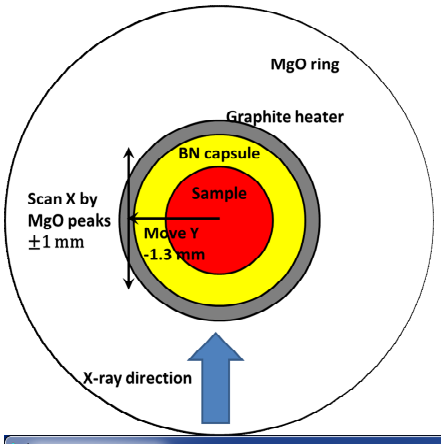
-In order to connect EPICS motor control and MCA software, please click 'ON' in 'Scan1 MCA Trigger Toggle', and then input data acquisition time for each step in 'Preset Real Time' (typically, 5-10 second).

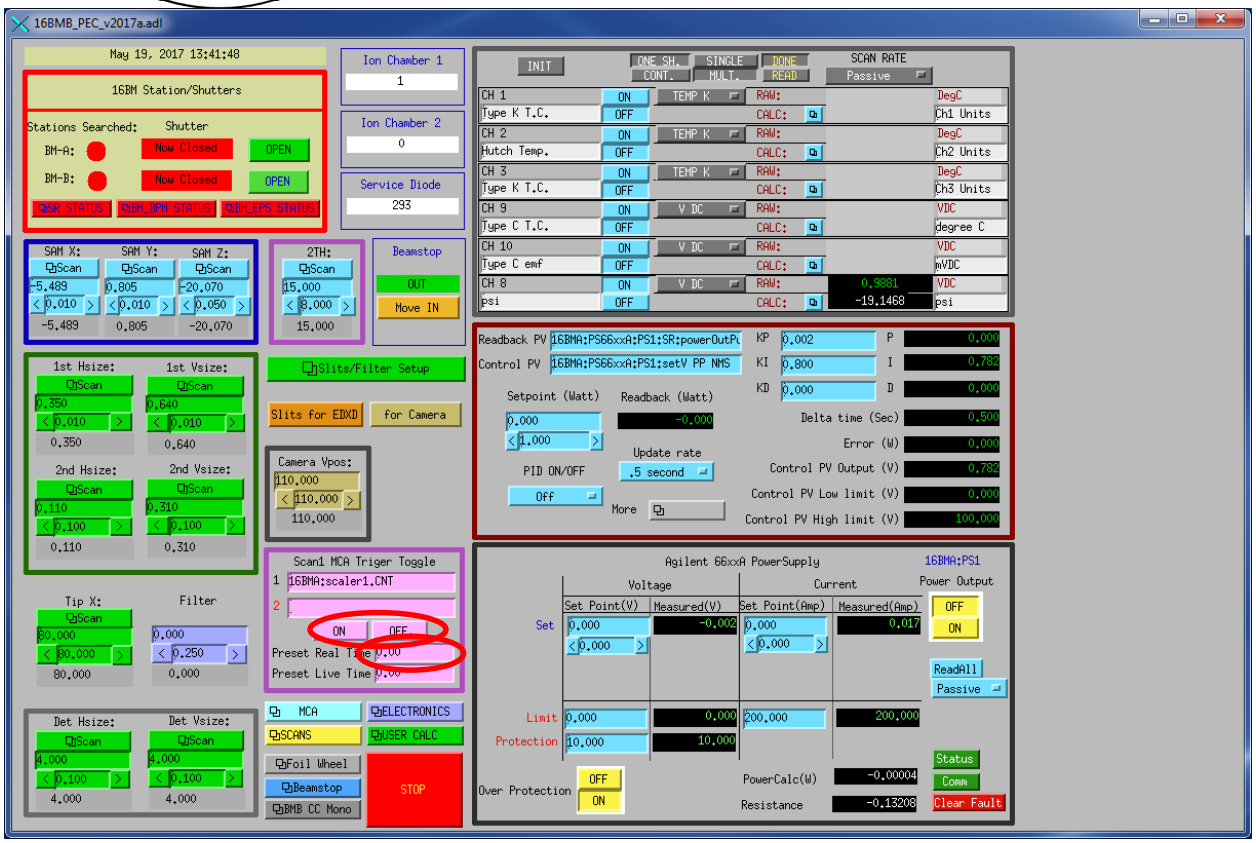
-Open 'Scan' in 'SAM X', and input parameters (typically, Start=-1, End=1, #Pts=21).

-Then, click 'Load&Go' to start scan.

-Sample X center is the location where MgO diffraction intensity is the minimum.

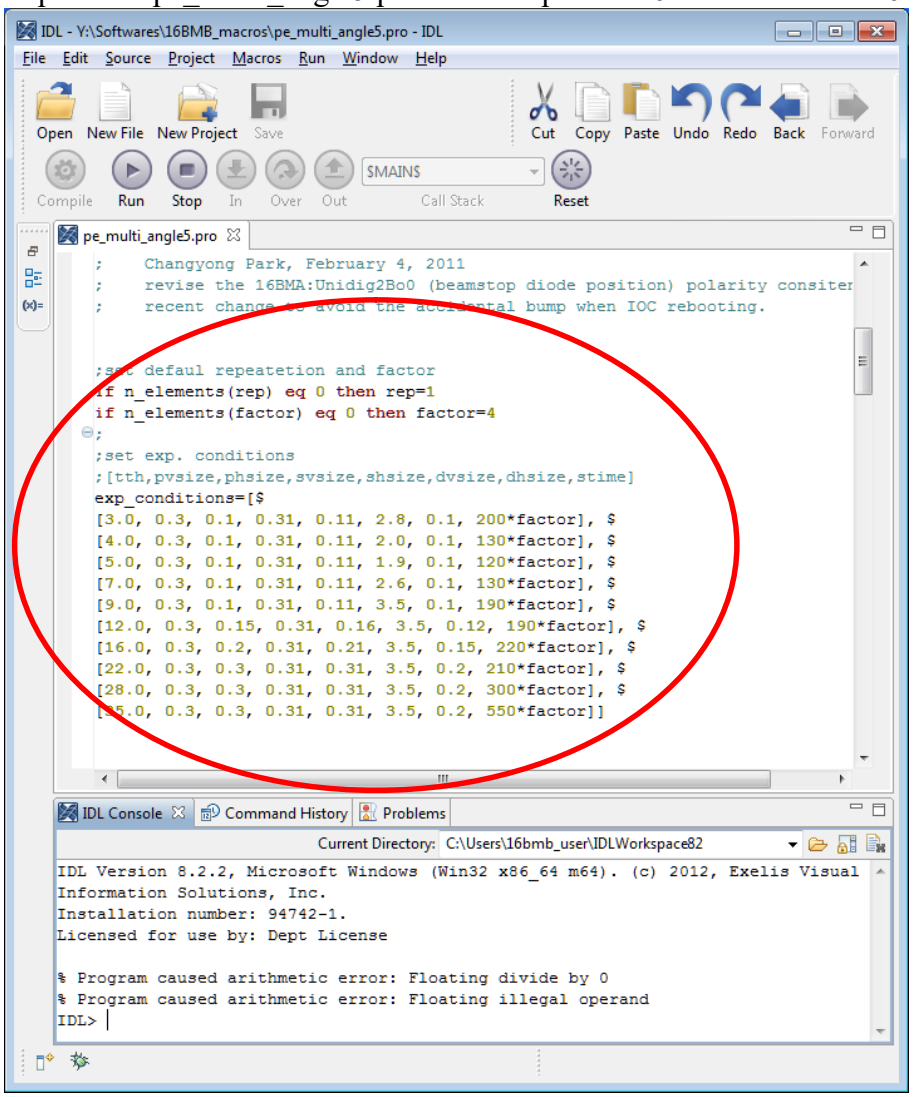
Note: After the scan, please do not forget to 'OFF' 'Scan1 MCA Trigger Toggle', and input 0 in 'Preset Real Time'.





IDL macro for liquid/glass structure measurement

An IDL macro is available for automatic data acquisition of EDXD pattern with varying 2theta angle.
-Open 'IDL' from the desktop shortcut.
Note: please open new IDL window even if IDL is running for MCA, and so on.
-Open file 'pe_multi_angle5.pro' from \\hpcat21\16bmb\Softwares\16BMB_macros.



-Scroll down until this table.
-Input parameters
1. tth=2theta angle
2. pvsize=1st slit Vertical size
3. phsize=1st slit Horizontal size
4. svsize=2nd slit Vertical size
5. shsize=2nd slit Horizontal size
6. dvsize=Detector slit Vertical size
7. dhsize=Detector slit Horizontal size
8. stime=Data collection time in 'Live time' (i.e. Actual data acquisition time is Live time + Dead time)

'factor' multiplies acquisition time.
If you want to repeat measurement, you can set 'rep'=2 or higher.

-Confirm followings:
‘Camera Vpos’=110
‘Beamstop’=OUT
‘Tip X’=0
‘Scan1 MCA Trigger Toggle’=OFF (nothing in line 2)
Both ‘Preset Real Time’ and ‘Preset Live Time’=0
Slit and Filter setup is ‘EDXD’ condition (‘Filter’=0, slit size is small)
‘position of sample is correct’.

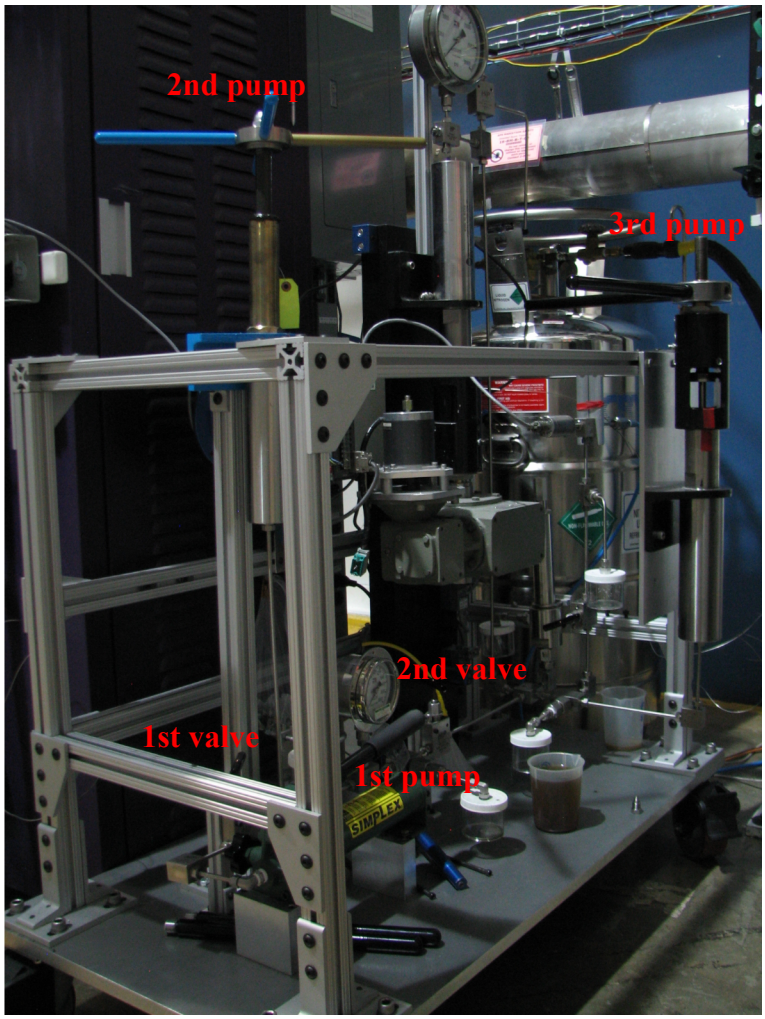
Then, please make dummy save file in MCA.
-File\Save As (please make a dummy file with specific name and extension ‘.000’)
-Open File\Preferences
-In preferences, please check ‘yes’ for ‘autosave when acquisition stopped’. (MCA will save file for each angle data with the name and numbered extension of ‘.001’, ‘.002’...).

Then, to start IDL macro,
On IDL window,
-Compile
-Run

Note: after finishing the IDL macro, please do not forget to check ‘no’ for ‘autosave when acquisition stopped’.
If you want to stop the macro, click Stop.

Increase pressure

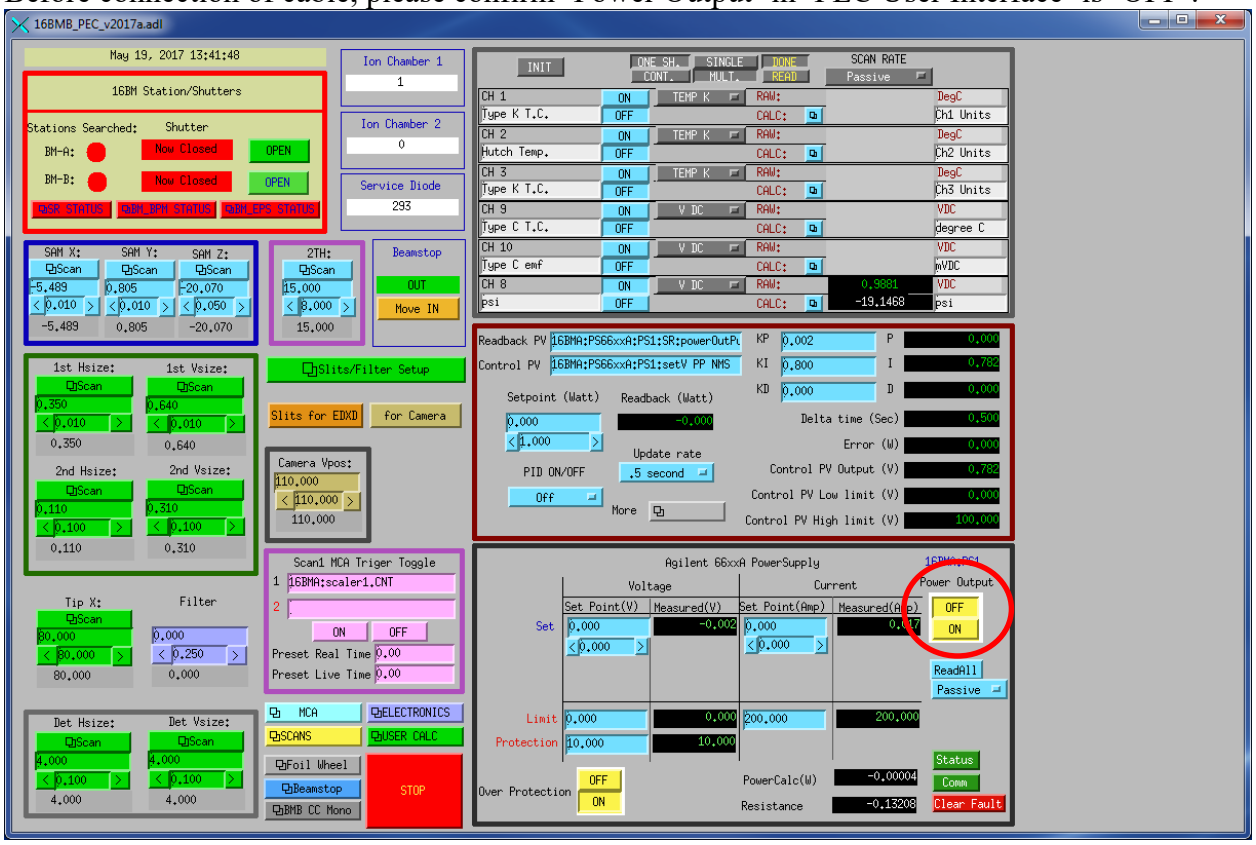
The PEC compression system consists of 3 pumps:
-1st jack pump up to 2,000-3,000 psi.
-2nd manual pump up to 8,000-9,000 psi (pump limitation is 10,000 psi).
-3rd motorized pump at higher pressures (pump limitation is 15,000 psi).



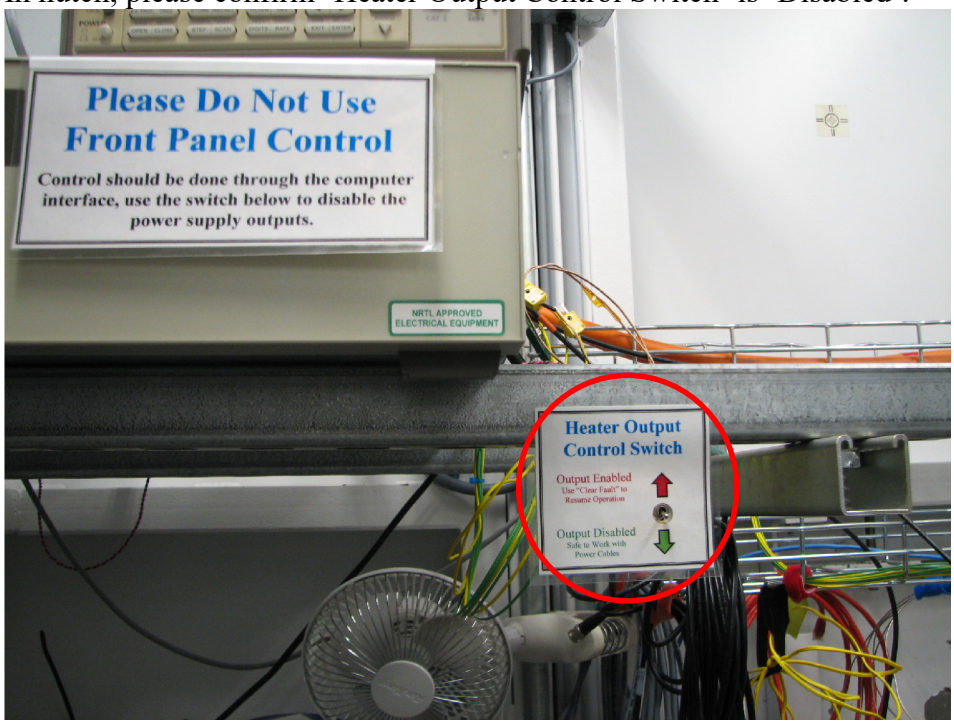
Procedures:
-Close valve on the 1st pump.
-Compress by the '1st pump' up to 2,000-3,000 psi.
-Close the '1st valve'.
-Rotate handle on the '2nd pump' clockwise to increase pressure.
-If you reach 8,000-9,000 psi, please close the '2nd valve'.
-Rotate handle on the '3rd pump'.

Heating

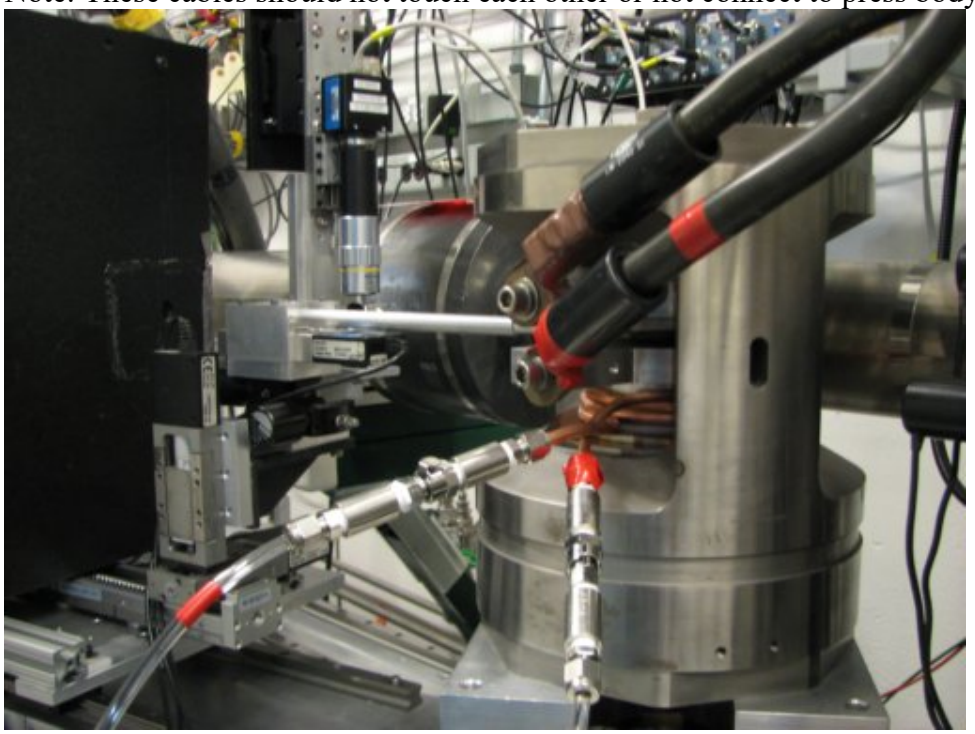
Before connection of cable, please confirm ‘Power Output’ in ‘PEC User Interface’ is ‘OFF’.



In hutch, please confirm ‘Heater Output Control Switch’ is ‘Disabled’.



-Connect cable on PEC
Brown cable (-) = Top ring.
Red cable (+) = Bottom ring.
Note: These cables should not touch each other or not connect to press body.

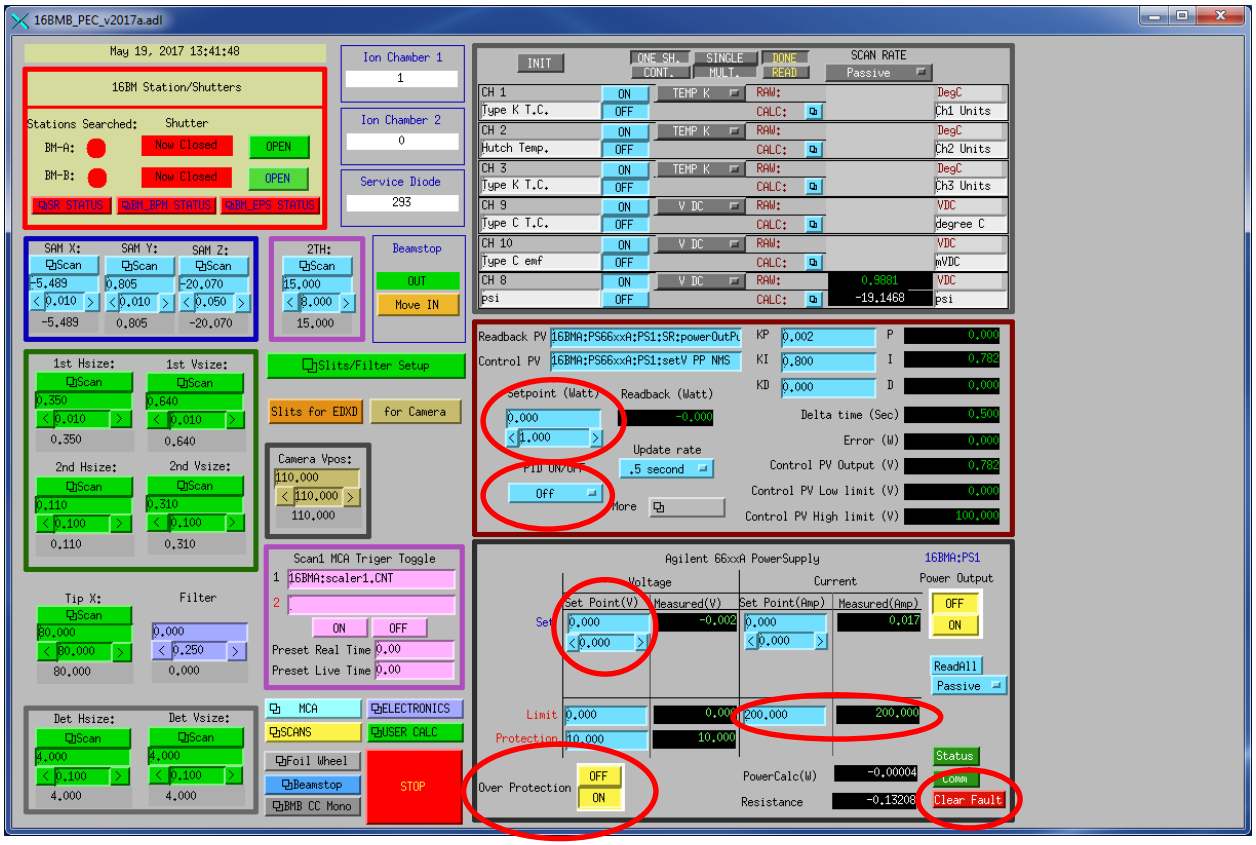


-Turn On a fun on PE press for cooling of press body.

-‘Enable’ on the ‘Heater Output Control Switch’.

-Before starting heating, it is recommended to start ‘Stripchart’ to save log of heating (cf. page 26 about Stripchart).

On ‘PEC User Interface’,
1. At first, please confirm ‘Voltage’ ‘Set Point (V)’=0, ‘Setpoint (Watt) on PID control = 0, and ‘Over Protection’ is ON.
2. ‘Power Output’ ON
3. Input 200 in ‘Limit’ under ‘Current’. Please input again even if the value is 200.
4. Click ‘Clear fault’.
5. ‘PID ON/OFF’ ON
6. Tweak ‘Setpoint (Watt)’ by 1 W to 3 W.
6. Check ‘Readback (Watt)’ is responding, and ‘Resistance’ is lower than 0.1 (typically, ~0.04-0.05 at ~1 W).
Note1: Response of heater is slow particularly at <10W. Please wait a while.
Note2: Increase of ‘Readback (Watt)’ may stop at <3W. If so, please check ‘Measured (Amp)’ under ‘Current’. If ‘Measured (Amp)’ value is 2.65, it is likely to forgot the procedure 3 (Input of 200 in ‘Limit’ of ‘Current’). In this case, please lower ‘Setpoint (Watt)’ to 0, turn OFF the ‘PID ON/OFF’, input 0 in Set Point (V), and turn Off the ‘Power Output’. Then, please restart the procedures.
7. If heater response and resistance is okay, increase ‘Setpoint (Watt)’ slowly (**it is better to keep <5 difference between ‘Readback (Watt)’ and ‘Setpoint (Watt).**).



Cooling can be done (1) slow cooling by gradually decreasing ‘Setup (Watt)’ to 0, or (2) Turn OFF ‘Power Output’ to quench sample.
In both case, after cooling,
-Input 0 in ‘Setup (Watt)’.
-‘PID On/OFF’ OFF
-‘Power Output’ OFF
-Input 0 in ‘Set Point (V) under ‘Voltage’.

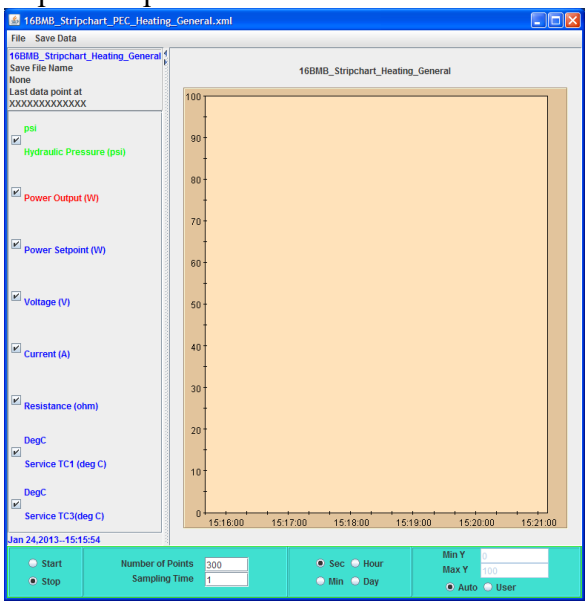
-‘Disable’ on the ‘Heater Output Control Switch’.

Note: Do not touch on press at least until turning off the power of heater power supply. Even after the power off, please take care. If you heated more than 1000 °C for more than several hours, press body may be hot. Please wait a while to cool down press body.

After cooling of press body, please remove heating cables.

Stripchart

Stripchart saves compression and heating records with time.
-Open ‘Stripchart’ from desktop shortcut.
-File\Open
-Open setup file.



This setup saves:
-PEC pump load (psi)
-Power Output (W) (Readback)
-Power Setpoint (W)
-Voltage (V)
-Current (A)
-Resistance (ohm)

To start recording,
-Click ‘Save Data’ and make file.
-Click ‘Start’ at the bottom left corner to start graphical view.

-View items can be selected by ‘check box’ on each parameter in left column.
-Graph scale, data point duration can be changed by using the bottom column features.