Standard Operating Procedure

Rigaku SmartLab XRD

I. Powder X-ray Diffraction (PXRD)

Yale West Campus
Materials Characterization Core
ywcmatsci.yale.edu

ESC II, Room E119C
810 West Campus Drive
West Haven, CT 06516

Version 1.0, May 2016

> *Please **FOLLOW the SOP strictly** to keep the facility in good condition. Any **explorations are strongly prohibited** unless permitted by lab manager*
> *__Only__ use your Core USB drive on the XRD computer.*
> *__NEVER__ surf the web on the SEM/EDS computer in order to minimize the risk of the computer being hacked*

> *Yale West Campus MCC facility users must acknowledge MCC in their publications that rely significantly on MCC resources. The general acknowledgement for XRD should read:*
> "The XRD data was taken using the Rigaku SmartLab X-ray at Yale West Campus Materials Characterization Core (MCC)."
> *The core reserves the right to use the data for core promotion*

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1 Introduction

a) Instrument features:
   - > Full automated alignment under computer control
   - > A high-efficiency 2D detector (Hypix 3000) with high-count rate
   - > Cross Beam Optics (CBO) permits easy switching between focusing (BB) and parallel beam (PB) geometries without reconfiguration
   - > In-plane diffraction arm for in-plane measurements without reconfiguration
   - > High temperature (~1500 C) measurements in air, vacuum and helium
   - > Air-Tight Sample Container for contamination free measurement

2) Location

Materials Characterization Core
Room E119
810 West Campus Drive
West Haven, CT 06516

b) Primary Staff Contact

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The Yale West Campus MCC Facilities are operated for the benefit of all researchers. If you encounter any problems with this facility, please contact the staff member listed above immediately. There is never a penalty for asking questions. If the equipment is not behaving exactly the way it should, contact a staff member.

Notice: Please **follow** strictly the **SOP** to keep the facility under good condition. We **DO NOT** recommend user explorations on program unless endorsed by core manager.

2 Sample Preparation

Note: always wear gloves for sample preparation! Clean the sample holder with alcohols (IPO/ methanol)

1) Powder samples

Large particle samples **should be milled to below 5 microns** to minimize measurement deviations due to particle size effects.

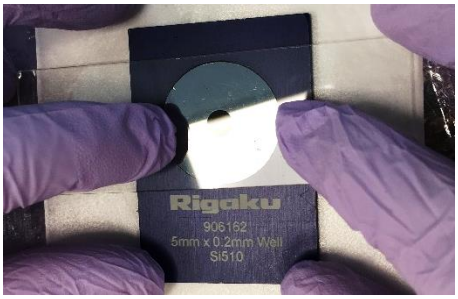
Note:

> It is highly recommended to use the **zero-background sample holder** so that the hump signal in the lower 2theta range (<20 degree) on regular glass holder can be avoided.

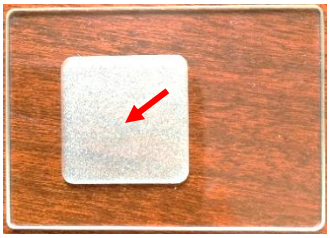
> If sample needs to be placed on SS plate, make sure use small size Length limiting Incidence Slit (**IS(L)**). A sharp diffraction peak can be seen on SS plate.

a) **Small amount powder samples** (~ **5 mg**)

> Choose the **zero background sample holder** and fill the 5 mm x 0.2 mm well with powder sample and press the powder surface with a glass slide so the sample surface flushes with the holder surface
Note: to avoid electrostatic discharge (ESD) on magnetic materials, cover the powder with a **weighing paper** first.



> Alternately, to increase sample exposure area to X-ray beam as diffraction signals are more sensitive on sample exposure area than sample depth, the regular glass holder with **20 x 20 x 0.2** mm square indent (the shallowest one) can be used. Spread sample at the indent center and flat the surface with an optical slide or flat spatula.
Note: an amorphous hump from glass may appear if sample area is smaller than the X-ray beam size.



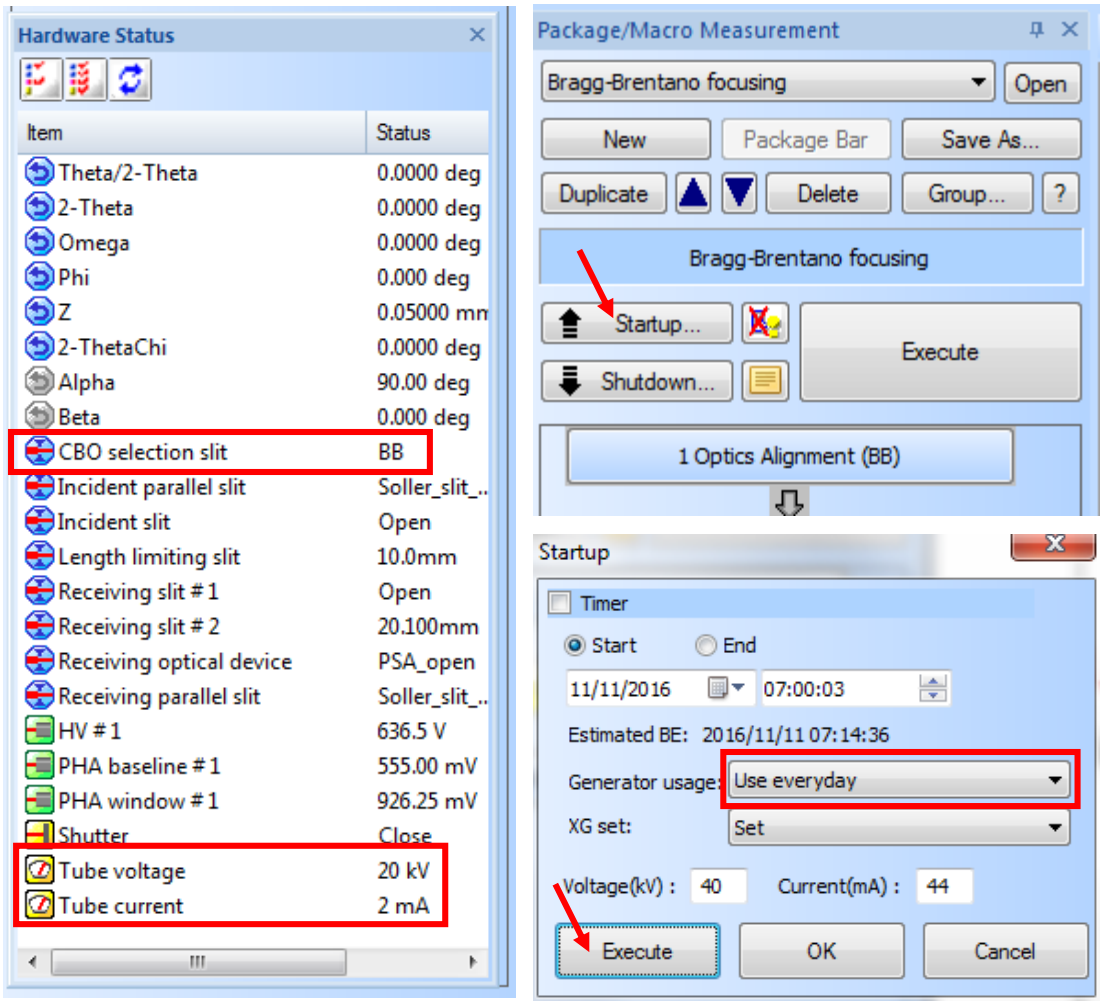
b) **Large amount powder samples**:
   > Fill the 20 mm x 20 mm square indent on the **glass holder** with the sample
   > Flat the sample surface to flush with indent edge using an optical slide.
   Note: For multiple samples, the sample height alignment is required only for the first sample if all samples are prepared in the same way.

2) Film samples
   a) The film samples can be placed either on **zero background sample holder** or regular glass holder. Choose right Incidence Slit sizes (**IS1** and **IS(L)**). **N**ormally **IS(L)** should be chosen as **half** of the sample size across the X-ray beam).
   b) Alternately the stainless steel plate with the right height spacer can be used if the X-ray beam size is chosen smaller than the sample size.

3 Hardware Status Check

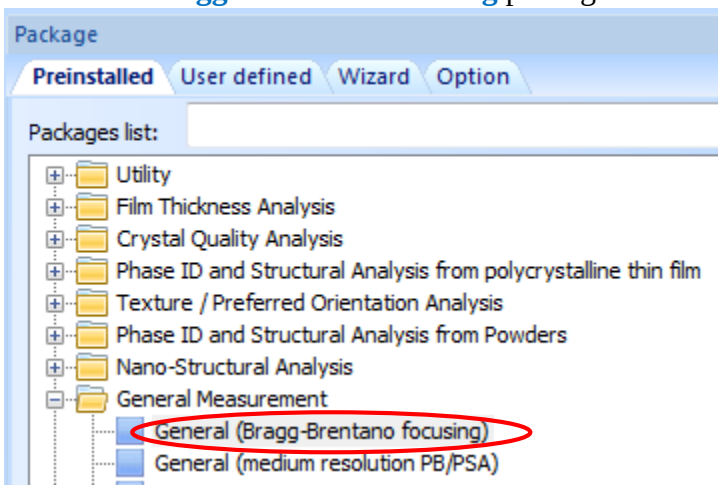
1) Open the **SmartLab Guildance** software if it was closed (login: administrator, password: rigaku)
2) Check the **Hardware Status** window on the bottom left of the software:
   a) Make sure that the "**CBO selection slit**" is "**BB** (Bragg-Brentano focusing mode)"
   Note: if it shows "**PB** (Parallel Beam mode), the **Optics Alignment** is required before measurement (see step 4)
   b) Check if the X-ray is at standby (Tube voltage: **20 kV** and Tube current: **2 mA**).
   If yes, click 

button in the **Package/Macro Measurement** window.
   In **Startup** window, choose "**Use everyday**" if the machine was used within 24 hours and hit "**Execute**" button.
   Note: it will take 15 minutes for the X-ray power to resume the operation power (**10 kV** and **2 mA**).

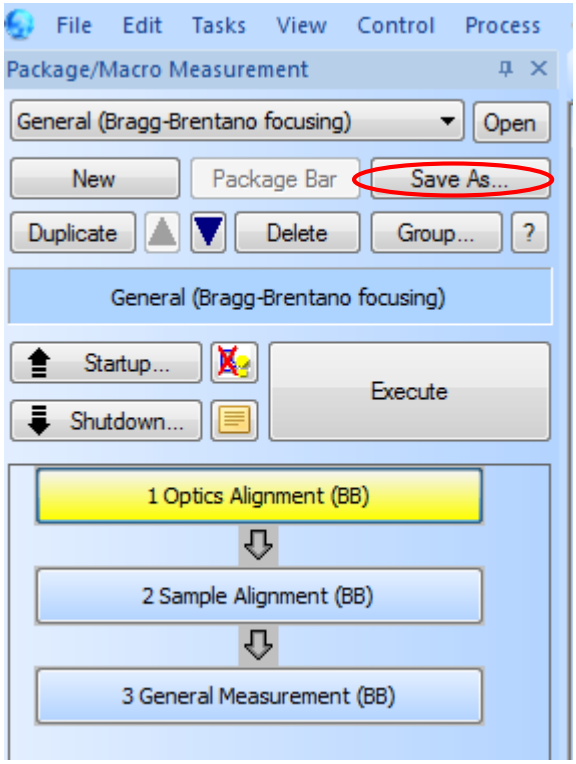


4 User Package Definition

1) On the **Package** window at the right side of software window, find **Preinstalled**> **General Measurement** > **Bragg-Brentano Focusing** package



2) **Double Click** on **General (Bragg-Brentano Focusing)** to open the package in the **Package/Macro Measurement** window:



3) Click on **Save As** button in above window to save the package as user defined package which will appear in the **User defined** tab on the right side of the Guidance window. On the bottom of **User defined** tab click  to create a user folder and move the package file into the folder by clicking  .

5 Sample Loading

1) Press the Door Lock button on the Rigaku SmartLab instrument cabinet door and slide the door open

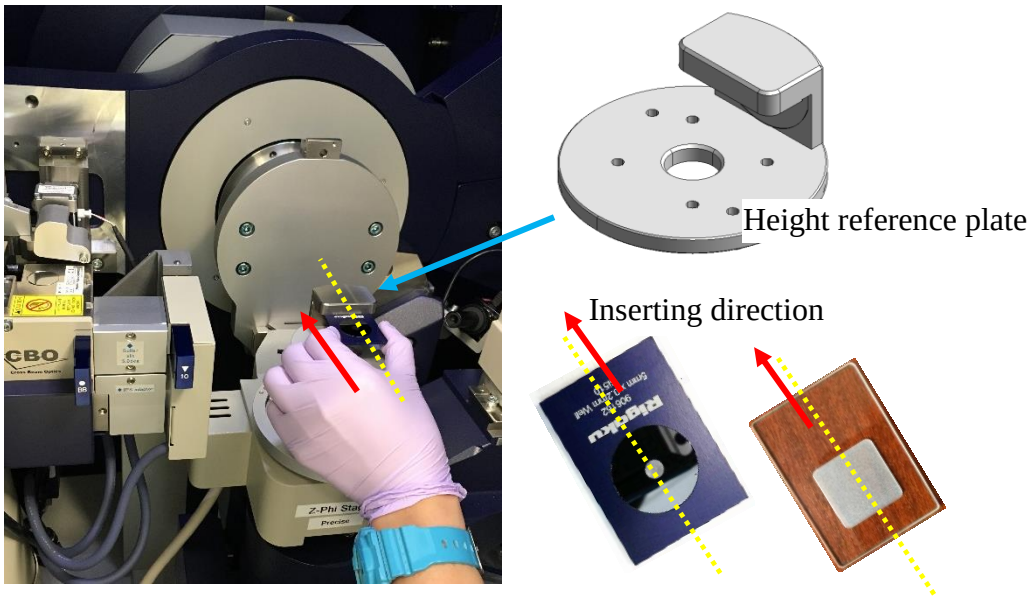
2) Push the glass sample holder or zero background holder into the **height reference sample plate**

Note:

> Make sure the sample holder aligns to the center of the **height reference plate** **Warning:** large misalignment will lead to X-beam incidence on sample holder)

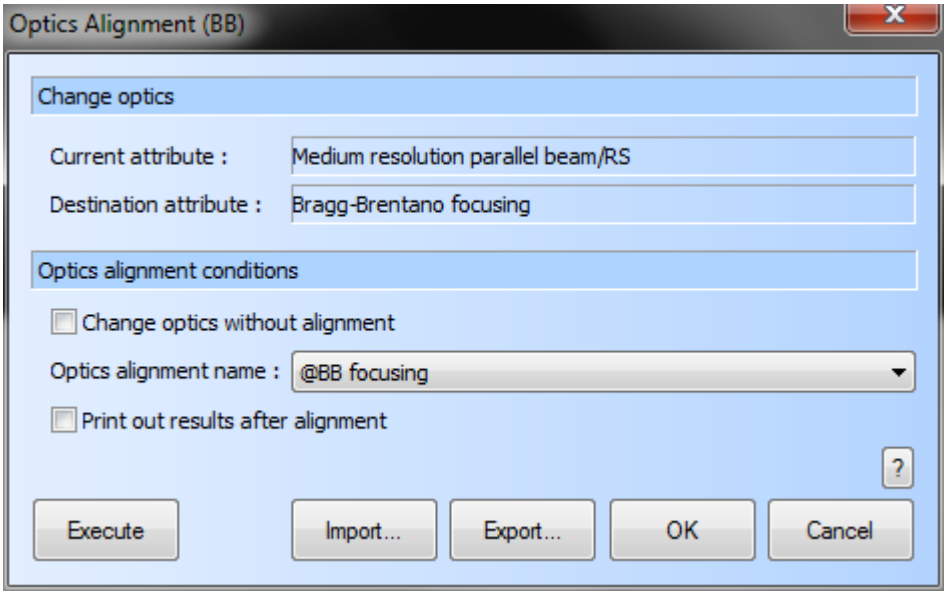
> If no enough sample to cover the entire square well on the glass holder, try to extend the sample area along the longer side of holder (dashed line)

> Close the cabinet door and hit **Door Lock** button

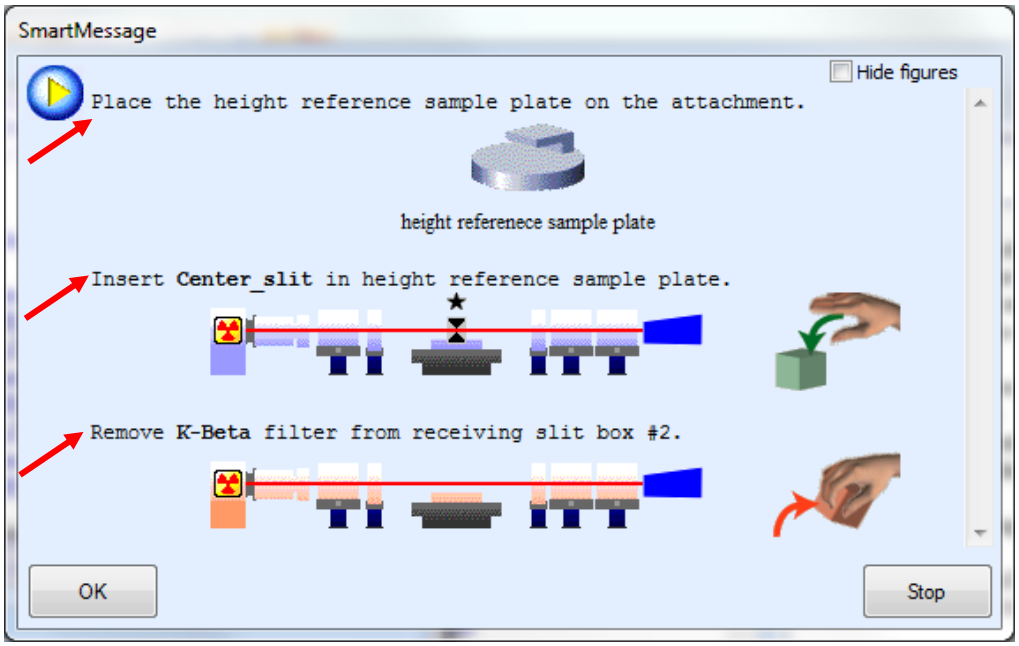


6 Optics Alignment

1) Put the "**BB**" slit into the **CBO** adapter and click on 1 Optics Alignment (BB) in the **Package/Macro Measurement** window, hit "**Execute**" and follow the instruction on the popped up **SmartMessage** window:



2) Press **OK** on the **SmartMessage** window after finish required steps.
Note: this step may take about 5 minutes. Once finishes, the **Optics Alignment (BB)** window will be active again.



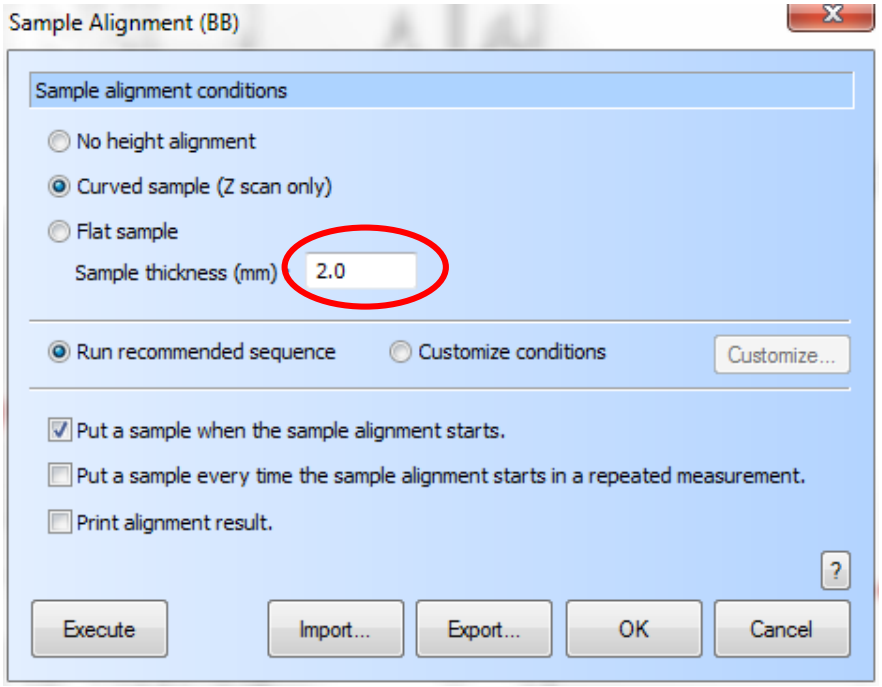
3) Click **OK** on the **Optics Alignment (BB)** window after the optics alignment finishes

7 Sample Alignment

1) Click on 2 Sample Alignment (BB) in the **Package/Macro Measurement** window
2) On the popped up **Sample alignment conditions** window:
   a) Choose **Curved sample (Z scan only)** for particle samples, or choose **Flat sample** for flat samples such as thin films, single crystals.

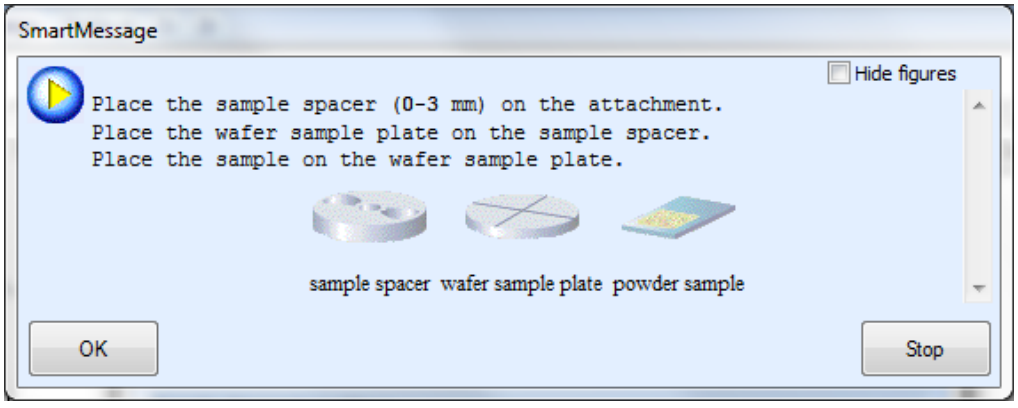
b) Select **Sample thickness (mm): 2.0** (if powder sample surface flushes with glass holder, or higher numbers (2.0 + solid sample thickness)
Tips: for multiple samples with similar thickness or powders flushing with the holder, **NO NEED** to repeat sample alignment.

c) Check the box next to "**Put a sample when the sample alignment starts"** and click **Execute**



d) Follow the instructions in the popped up **SmartMessage** window

Note: just hit **OK** if the **step 4)** Optics Alignment is skipped.

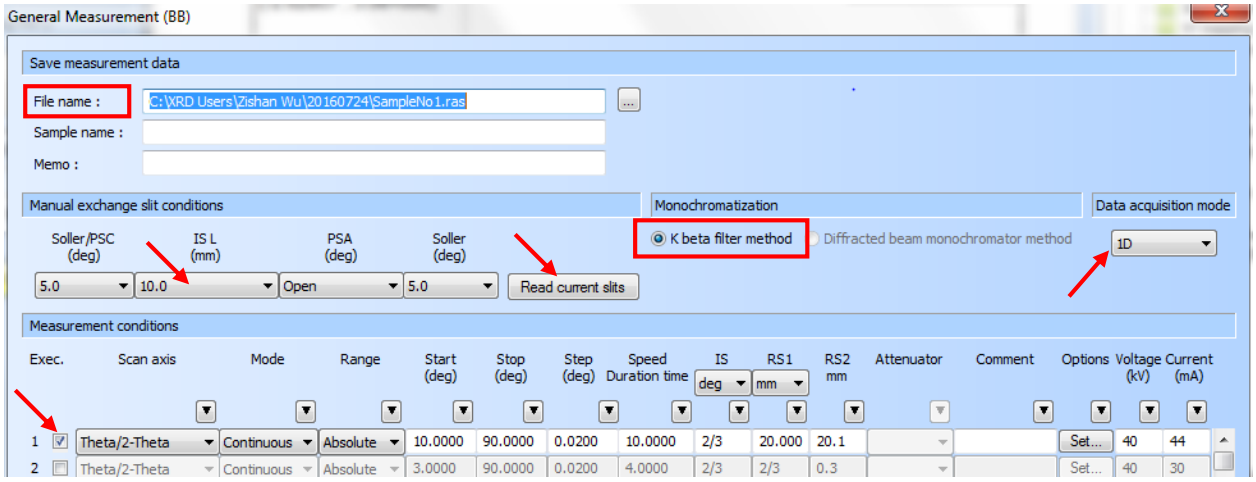


Note: the sample alignment takes about 2 minutes after which the **Sample Alignment (BB)** window will become active.

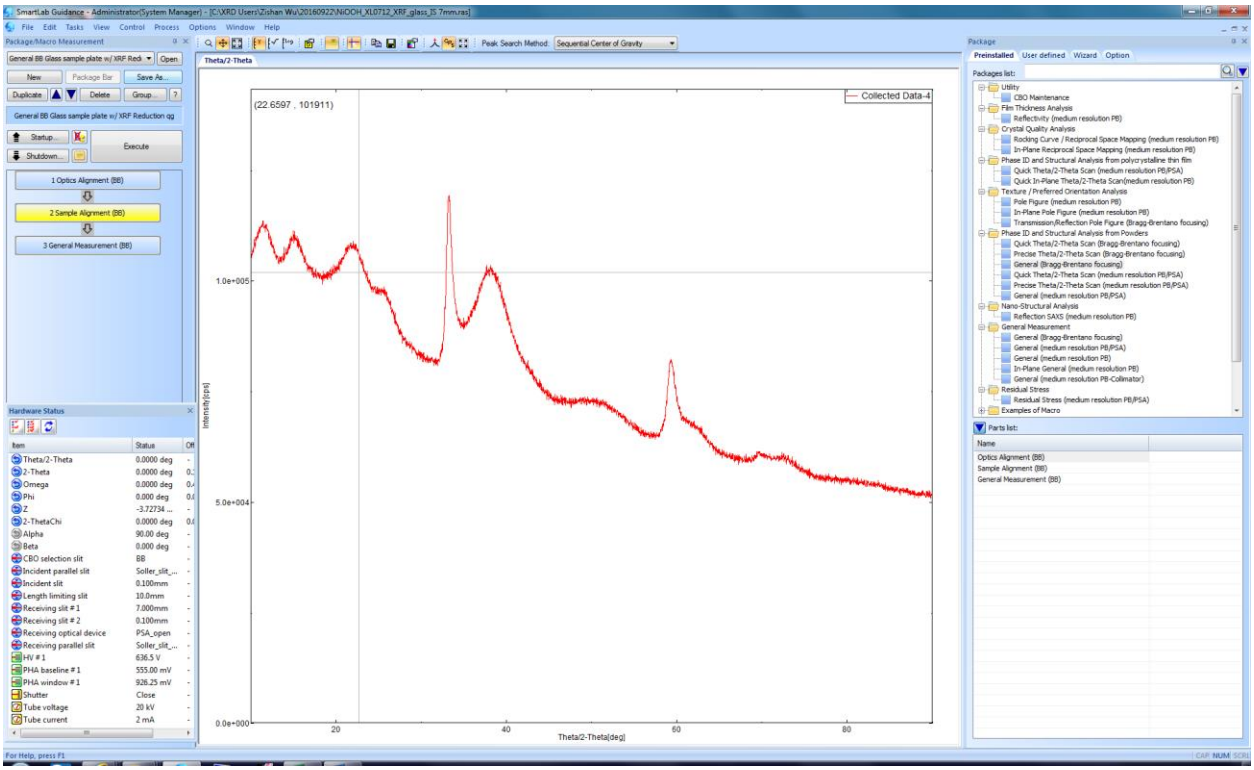
e) Hit **OK** on the **Sample Alignment (BB)** window

8 Sample Measurement

1) Click on the **General Measurement (BB)** tab on the **Package/Macro Measurement** window and perform following steps:
   a) Change **File name** and folder
   b) Check **K beta filter method**
   c) Select **1D mode**
   d) Read Current Slits
   e) Choose the right **IS L** size for samples (slit size: sample size = 1:2), **typically 5.0**
   Warning: do not change other parameters on the same line.
   f) Set **typical Measurement conditions**:
      > Check **Exec 1** box → **Continuous** (Mode) → **Absolute** (Range) → **10.0000** (Start) → **90.0000** (Stop) → **0.0500** (Step) →**10.0000** (Speed) → **2/3** (IS) →**20.000** (RS1) → **20.0** (RS2) →…→**40** (Voltage) → **44** (Current)
      Warning: the **maximum power** for X-ray source is **40 kV** and **44 mA**. Any higher numbers will cause **severe damage to X-ray tube**. **Violation** will lead to user account suspension and cost charges on user PI's account.
      Tips: to improve signal/noise ratio, users can choose smaller steps, slower speed, smaller IS, RS slit sizes or smaller Current.
   g) Check "**Drive the 4 axes to the current zero positions after measurement completed**" on the bottom the **General Measurement (BB)** window.
   h) Click on "**Execute"** to start measurement
   i) Follow the instruction on SmartMessage windows if appear and hit **OK**



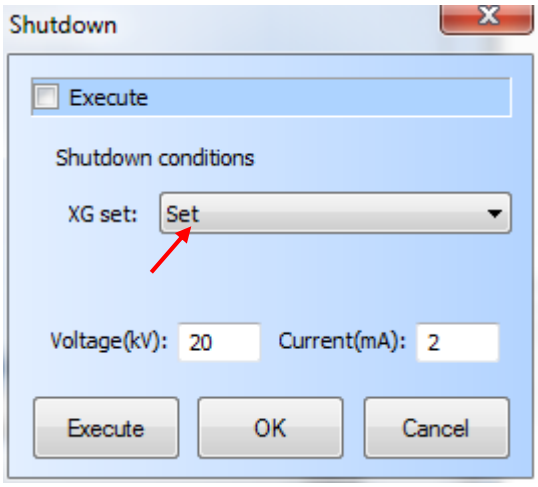
2) XRD spectra will be automatically saved after scan



3) For multiple samples, change sample and start from **section 3.6)**

9 Checklist after Experiment

1) **For the last user of the day**, click Shutdown... in Package/Macro Measurement window to set X-ray tube power to minimum (**20 kV** and **2 mA**)



2) **Remove** sample from the stage
3) **Clean** the sample holders and glass slides with clean wipes using Methanol/IPA.
4) **Store** the samples holders in assigned organizer box.

5) **Use ONLY** the core USB flash drive to transfer data from XRD computer to the workstation in the core, and then use either your own USB flash drive or internet to retrieve data.
6) **Sign out** on the **logbook** and report any problems.
7) **Keep the SmartLab Guidance software on!**