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Standard Operational Procedure for High-Resolution Thin-Film X-Ray Diffractometer

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Modification Statement

Revised Date	Version	Details of Revision	Revised by	Approved by	Approval Status
2020.03.23	V1.0	Trial	Xiaohe MIAO	Xingyu LU	

Content

1. Purpose

This Standard Operational Procedure (SOP) is to standardize the operation procedure of Bruker D8 Discover High-Resolution Thin-Film X-ray Diffractometer, to ensure of a proper and regulating use.

2. Scope

This SOP is applicable to all users operating Bruker D8 Discover High-Resolution Thin-Film X-ray Diffractometer.

3. Responsibilities

3.1. Users: strictly follow the instructions and immediately report to staff if any troubleshoot.

3.2. Staff: authorizing users who pass the training and qualification and standardizing their operations.

4. Safety regulations in X-Ray laboratory

4.1. All personnel entering the lab should be aware of potential X-ray radiation hazard. It's mandatory to take the HSE lab safety and radiation trainings.

4.2. All personnel entering the lab should be familiar with chemical safety knowledge, water/gas knowledge.

4.3. Strictly adhere to the safety regulations in XRD lab and notice the alerts. Lab coat is required in XRD lab.

4.4. Independent operation is permitted only after operational training and qualification and following SOP. It's illegal to use the facility without training. Operations beyond SOP are prohibited. It's not allowed to use the machine under the circumstance of modifying hardware or removing safety circuit.

4.5. It's strictly forbidden to transfer data with USB and get network connected.

4.6. Users should operate in strict accordance with SOP; staff should make frequent inspection to correct violations in time and avoid safety risks. Tidy up the working bench and properly make record after experiment.

4.7. Gloves are mandatory during sample preparation and transferring. No bare hands for sample holders. No gloves for keyboard, mouse and doorknob.

4.8. All users are personally responsible for maintaining working bench neat and organized. Tools should be returned to original containers. Smoking, eating, drinking, and storage of food are not permitted in the lab.

4.9. Be strictly subjected to University policies. All lab waste must be segregated and disposed into the appropriate waste container accordingly, such as toxic trash, reagents, utensils and sharps.

4.10. Please inspect electricity prior to leaving the lab and close the door.

4.11. Please report to the staff if you find anything wrong with the system, do not try to repair by yourself.

4.12. For accidents caused by operations beyond SOP, corresponding users will be responsible for violations and penalties will be applied.

5. Usage policies of X-Ray facilities

5.1. Usage policies of Bruker D8 Discover High-Resolution Thin-Film XRD (HR TF-XRD)

This equipment is obedient to university policies for management of large-scale facilities with the principle of “concentrated investment, unified management, public open, sharing resource”, to support academic research and teaching. It’s proposed appropriate charge based on machine usage. The equipment will be open to the whole society when it meets the needs from Westlakers’ research.

Training and service of Bruker D8 Discover HR-TF XRD is classified into 6 ranks：

(1) Training: requests from users and scheduled by staff. The training involves sample preparation, introduction of XRD lab regulations, basic theory of HR-TF XRD, equipment configuration, sample mount procedure, data collection and processing.

(2) Independent operation (Primary): sample mount and independent operation for data collection for out-of-the-plane symmetric scan and rocking curve for film samples, as well as data processing and uploading.

(3) Independent operation (Intermediate): sample mount and independent operation for sophisticated applications, for instance, out-of-the-plane asymmetric scan, skew scan and Rocking curve, rapid reciprocal space mapping as well as data process and upload.

(4) Independent operation (Advanced): sample mount and independent operation for advanced applications of Phi scan, high-resolution reciprocal space mapping, X-Ray Reflectivity (XRR), in-plane Grazing Incident Diffraction (GID), high-resolution in-plane diffraction.

(5) Service: staff analyze samples submitted by users with sufficient information and measurement requirements.

This equipment can be reserved on “LIMS” via the link below and necessary information should be provided:

http://share.westlake.edu.cn/lims/!equipments/equipment/index.551.reserv

5.2. Reservation policy

To sufficiently use the machine to serve Westlake researchers, XRD lab has proposed 24/7 reservation policy. Herein, reservation can be made at least 1 day in advance, even during weekends, while at least 2 days in advance during public holidays, such as National Day, Chinese New Year.

It’s advised to adhere to reserved slot strictly, avoid of wasting machine hours. Communicate with the next user when overtime with the support of staff. Free cancellation can be made 8 hours in advance. In case of absence without no notice, his/her independent operation access will be suspended for 1 month.

Reservation hours		Reservation slot per person	Measurement content
Working hours (Monday~Friday)	09:00 am ~17:30 pm	Minimum 30 min each time	Independent operation, service
Non-working hours	18:30 pm ~ 8:00 am	Minimum 30 min each time	Independent operation

(Monday~Friday)			
Non-working hours (weekends & public holidays)	09:00 am ~ 8:00 am	Minimum 30 min each time	Independent operation

Notes:

1) Internal users are permitted to operate the machine after qualification;
2) Please keep accurate records for experiments as well as machine status after experiment;
3) It's inhibited to modify, disassemble or adjust any parts of the equipment.
4) Please immediately report to the staff if find anything wrong with the equipment is found, requesting for a prompt repair.
5) User's research group should afford the repair charge for instrument damage due to his/her individual mis-operation.
6) Please contact the staff for operations beyond SOP.
7) Do Not delete raw data on the control computer/workstation, otherwise contact the staff.
8) Uploading raw data via NAS drive and downloading to personal computer for further data process instead of copying data by USB. Raw data will be remained on control computer/workstation for 2 months (temporarily; can be extended further if there is sufficient capacity of hard drive to keep it).
9) All users are personally responsible for maintaining working bench neat and well organized. Samples should be taken away from XRD lab, otherwise discarded without notice.

5.3. Evaluation policy

Researchers like professors, graduates, postdoctoral fellows are eligible for training, scheduled by the staff, which contains three parts.

Part I, introduction of XRD lab safety regulations, equipment configuration and basic theory.

Part II: operational training, including sample preparation, the hand-on practical operation according to SOP and basic data process.

Part III: trainees passed the operational evaluation should practice at least twice within a week with inspection of XRD staff.

The staff consider that the trainee's operational skills achieves at relevant level, should authorize trainee with qualified access. Mis-operation by users who should afford the repair charge, also be disqualified and be charged double for repetitive training in future.

Notes: Users should close sessions of Part I and Part II of the training within 1 week, or else apply for another training. Qualified users guarantee about independent operation at least once per month. And it will be failed to reserve the machine if the usage frequency of the equipment is lower than once per month.

Requirements for trainees:

(1) Understanding the theory, configuration and function of High-Resolution Thin Film XRD, strictly obey regulations, checking the status and reporting problems.

(2) Proficient in operating HR TF-XRD and data format conversion; strictly follow SOP to avoid troubleshoots. Make proper records for HR TF-XRD after measurement.

5.4 **Troubleshoot report**

1) Users are requested to report any problems related to the instrument or the facility (power, water, AC …) in the logbook AND contact the staff.

2) For reporting the troubleshoot, taking the screenshot and save in Error Report folder named as "PI name-user-sample identity-troubleshoot time". Also specify your observation briefly in comment.

6. Standard operation procedure for High-Resolution Thin Film X-Ray Diffractometer (Bruker D8 Discover)

6.1. Introduction

Model：Bruker D8 Discover

Origin：Germany

6.2. Basic information

This High-Resolution Thin Film X-Ray Diffractometer, is designed for analyzing epitaxial thin films and also for polycrystalline thin films, which contains the core components (such as the rotational Cu anode, optics and duo detectors) and a chiller, which is equipped with Kα1 monochromator to filter off Kα2 X-Ray as well as improve its resolution. Furthermore, great gain in intensity and resolution benefits from duo detectors of LynxEye and Scintillation counter, facilitating applications of offset coupled 2Theta-Omega scan, Rocking curve, reciprocal mapping, Grazing Incident Diffraction (GID) and mirco-area analysis. Moreover, this equipment is also equipped with temperature-dependent in-situ chamber, TC-DOME, whose temperature accommodation varies from -150 °C to 450 °C, and RT to 1100 °C.

6.3. Specifications

Bruker D8 Discover HR TF-XRD has been equipped:

6.3.1 6KWTXS-HE high-brilliance rotational Cu anode;

6.3.2 High intensity 2-bounce monochromator-CCC2（Ge022）with resolution of 0.012°;

6.3.3 High intensity 2-bounce monochromator-ACC2（Ge004）with resolution of 0.004°;

6.3.4 7-axis high-accuracy centric Eulerian cradle with vacuumed sample stage and laser-video microscope;

6.3.5 Auto switchable optics manipulated by control software;

6.3.6 Auto switchable Duo detectors: Scintillation counter and LynxEye detector (enabling RSM analysis);

6.3.7 Temperature chamber for thin films with two segments of temperature range: -150 ~ 450°C, and RT to 1100 °C

The configuration of Bruker D8 Discover HR TF-XRD is exhibited as below：

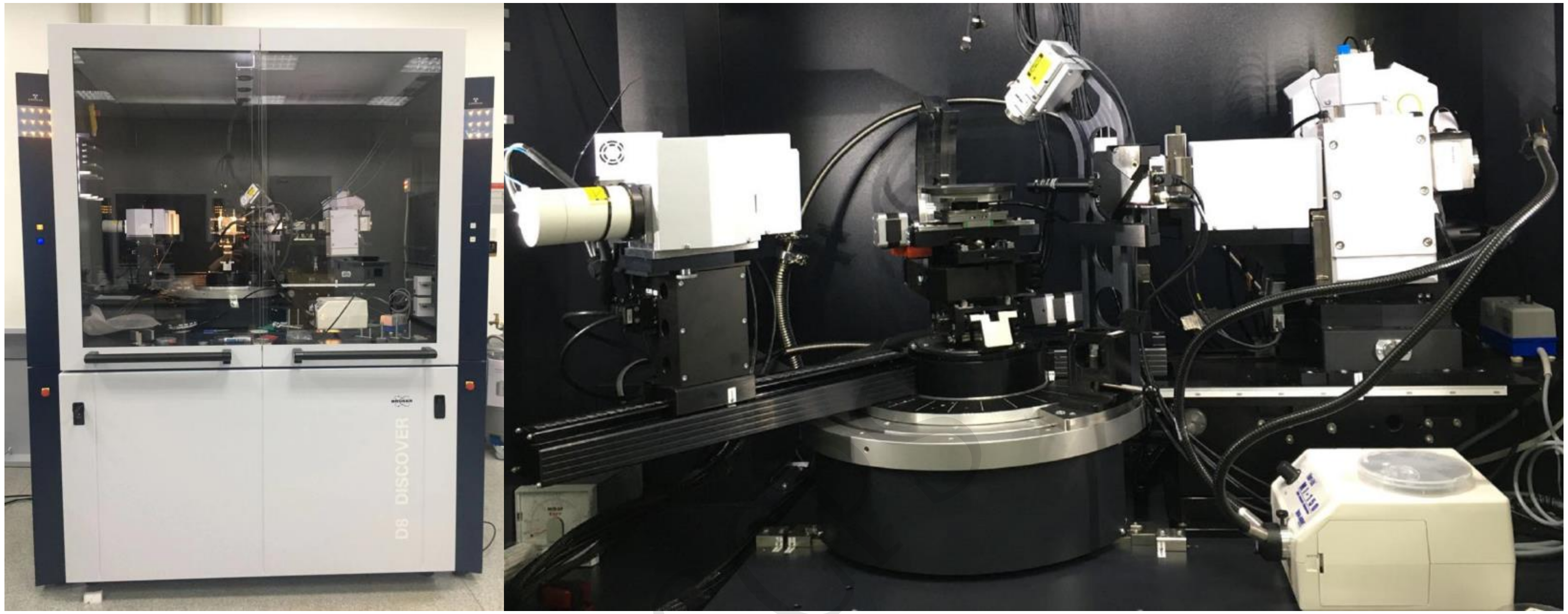

Fig 6-1 The configuration of Bruker D8 Discover HR-TF XRD

Location: Room 112, Level 1 Building 4, Yunqi Campus

Contact：Dr. Xiaohe MIAO, Tel: 0571-87310229, Email: miaoxiaohe@westlake.edu.cn

6.4. Sample requirements:

1) Non-toxic, incorrosive samples;

2) Flat and neat sample surface, block, pallet or fiber-like samples. And films deposited on substrates with thickness ≤ 1 mm, diameter ≤ 2 cm;

3) For XRR analysis, sample size should be less than 10 mm * 10 mm and thickness ≤ 100 nm;

4) Thin film samples are suggested to be screened by Powder XRD to understand the crystallinity;

5) Specify the analysis requirements, for instance, 2θ range;

6) Specify the storage condition of samples, like recycling, freezing, dry, lucifuge, etc.

7) Special applications, such as variable temperature experiments, are not arranged at nights

6.5. Power-on procedure for HR-TF XRD

6.5.1 Power on the chiller system, set the temperature at 22 ℃ with deviation of ± 2 ℃, and water pressure stabilize around 0.28 MPa (Fig 6-2); Please report to staff if values exceed the corresponding ones.

6.5.2 Turn the handle from "O" to "I" at left side of the machine (Fig 6-3), and turn on the green button, so that triggers for initialization; subsequentially, the indicator on upper-left control panel turns as white light to "I"; press the top button to power on high voltage, followed with the change of "I" to filament © (indicating of triggering the primary pump). Conditioning the tube is required for long-time shut down and the top button presents as  after conditioning. So the machine is ready for use.

Fig 6-2 Temperature and pressure shown on the chiller

Fig 6-3 Power buttons and indicators

6.5.3 Launch DFFRAC.SUITE and choose the role of “Lab Manager” without password and enter the software (Fig 6-4).

Fig 6-4 Launch DIFFRAC.SUITE

6.5.4 On Diffrac.Commander tab, check all drives and click “Initialize” to initialize all checked drives in Fig 6-5 (“! ” alerts remind of initialization when launch the machine and disappear when the machine is ready for use) .

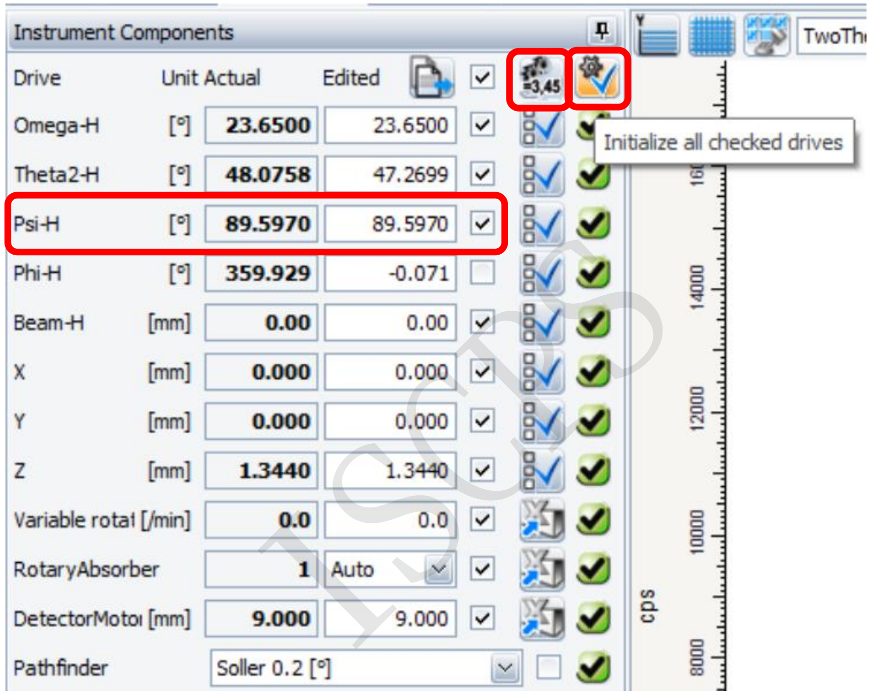

Fig 6-5 Initialization of drives

6.6 Mount thin film samples

The standard sample Si 004 is employed in this SOP for demonstration.

Set Psi = 90°, check it and click  to gently place the sample stage at horizon. The sample is mounted to the center of vacuum sample stage and set aside for 30 s (allowing the sample fixed by vacuum on the stage). Then set Psi = 0°, check it and click  to gently raise the stage to verticle (Fig 6-5 and Fig 6-6).

Note: It’s strictly prohibited to use 20 μm collimator during sample mount and alignment.

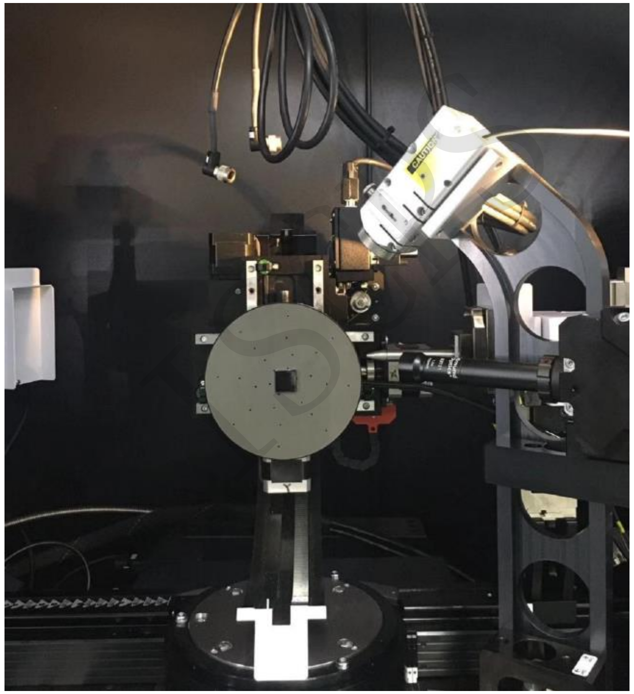

Fig 6-6 Sample mount to the vacuum sample stage

6.7 Symmetric scan for thin films

6.7.1 Check the intensity of 2-bounce monochromator CCC2（Ge 022）

During evaluating the intensity of incident beam, set the values as shown below in Fig 6-7: it reaches ~22000 Kcps with 0.5 mm collimator and ~53000 Kcps without colllimator correspondingly.

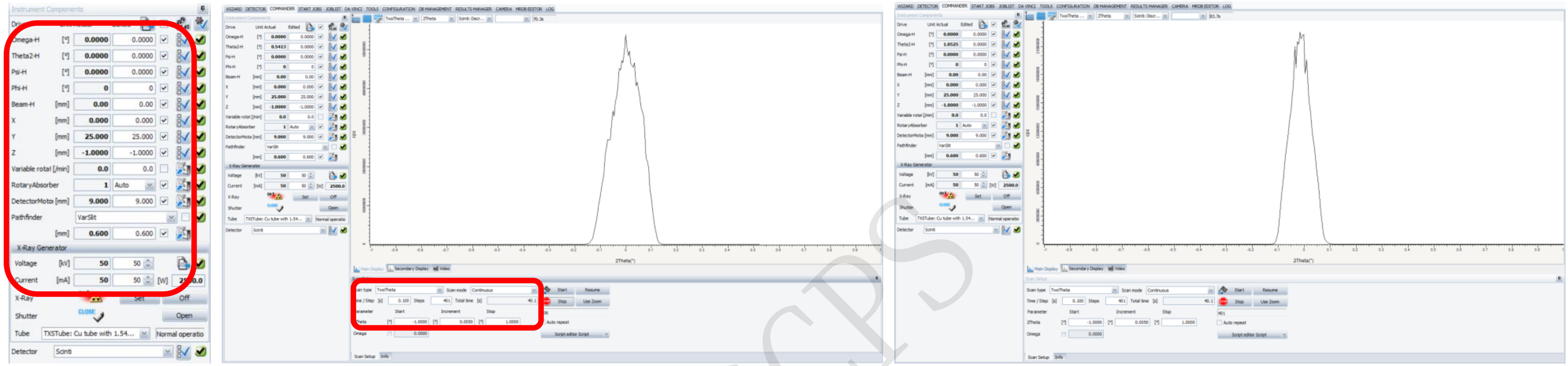

Fig 6-7 Intensity check for incident beam

6.7.2 Configuration and settings

6.7.2.1 Configuration：Ge 022 monochromator，0.5 mm collimator

6.7.2.2 Settings： Voltage/Current KV/mA (50/50), Z-axis = -1.2, Omega-H = 0, Theta2-H = 0, Psi = 0, Phi = 0, Beam-H = 0, X = 0, Y=25, Z-axis = -1.0, Rotary Absorber = Auto, DetectorMotor=9, Pathfinder -VarSlit = opening 0.6 mm, Detector = Scintillation;

6.7.2.3 Scan parameters: Scan type: TwoTheta, 2Theta range: -1 ~ 1°, Increment: 0.01 or 0.005°/step, time/step: 0.1 s/step，the peak locates around 0° (Fig 6-7);

6.7.3 Sample alignment

Set the sample at the center of X-ray beam (Fig 6-8):

The purpose of this step is to align the sample at the center of the incident X-ray Beam, two requirements detailed as below:

1) Sample top align to the beam center

2) Sample surface parallel to the incident X-ray beam

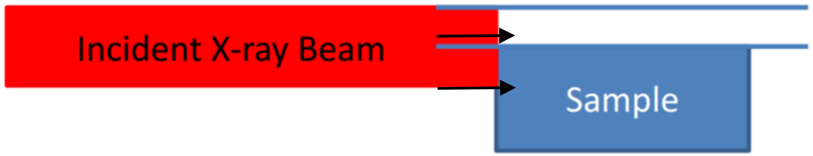

Fig 6-8 Sample alignment

6.7.4 Z-scan: maximum Z-scan range -1.2 ~ 1.9 mm

6.7.4.1 Set optics: Voltage/Current KV/mA (50/50), Z-axis = -1.2, Omega-H = 0, Theta2-H = 0, Psi = 0, Phi = 0, Beam-H = 0, X = 0, Y = 25,Z-axis = -1.0, Rotary Absorber = Auto, DetectorMotor = 9, Pathfinder-VarSlit = opening 0.6 mm, Detector = Scintillation;

6.7.4.2 Scan parameters: Scan type: Z, Scan range: -1.2 ~ 1.9, Increment: 0.01 or 0.005°/step, time/step: 0.1 s/step, find the Z height at half maximum intensity (~11000 Kcps), double click send the value to Z-axis box (Fig 6-9).

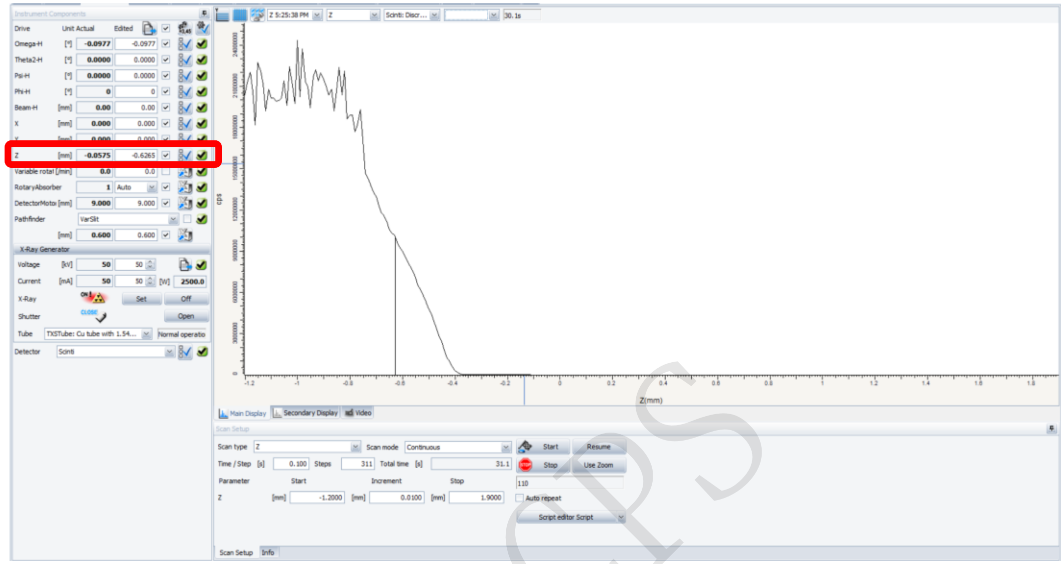

Fig 6-9 Z scan

6.7.5 Rocking curve

Scan parameters: Scan type: Rocking curve, Scan range: -1.0 ~ 1.0, Increment: 0.01 or 0.005°/step, time/step: 0.1 s/step, double click at the peak to send the value to Omega-H drive (Fig 6-10).

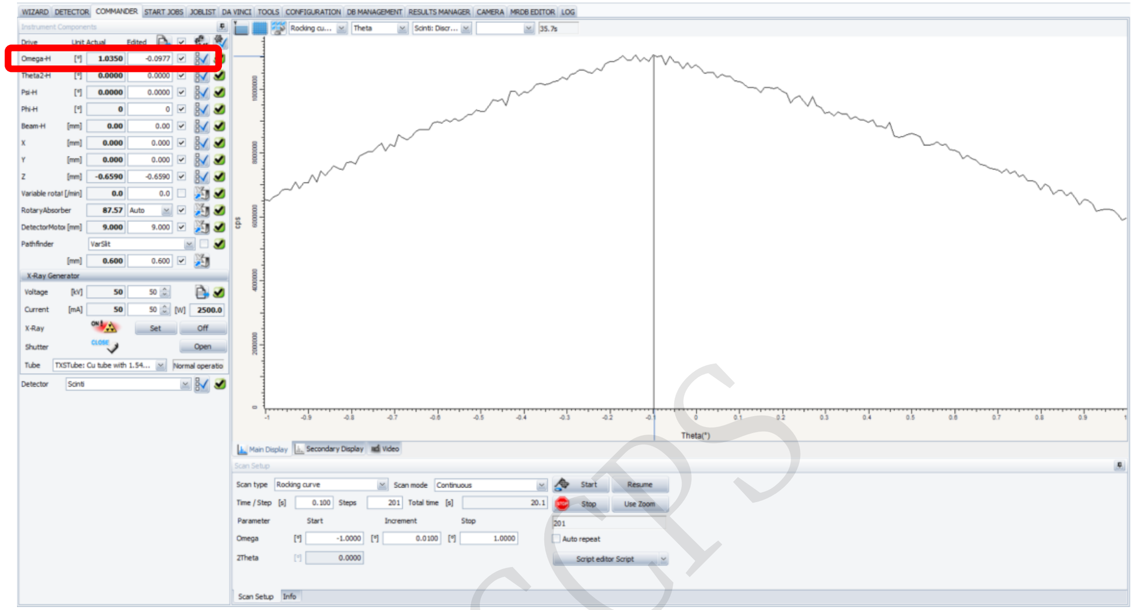

Fig 6-10 Rocking curve

6.7.6 Repeat 6.5.4 and 6.5.5 for Z-scan and Rocking curve two times, and update Z or Omega values to the corresponding drives until minimum deviation for Z or Omega. It ensures that the sample surface parallels to the incident beam.

6.7.7 Substrate measurement

6.7.7.1 Identified substrate

Regular substrates and their fingerprints: Si 004: 69.13°; Si 111: 28.5°; Sapphire 0006: 41.6°;

In this case, set Theta2-H for Si 004 = 69.13° with Omega-H (offset) of -0.0977° checked and perform Rocking curve (Fig 6-11):

Scan type: Rocking curve; Scan range of 34 ~36°, Increment 0.01°/step, time/step: 0.1 s/step, and double click at the peak to obtain the omega value and send it to Omega-H drive (Fig 6-11).

Note: Quick scan for large range with larger step size, then “Use Zoom” the range for fine scan with small step size.

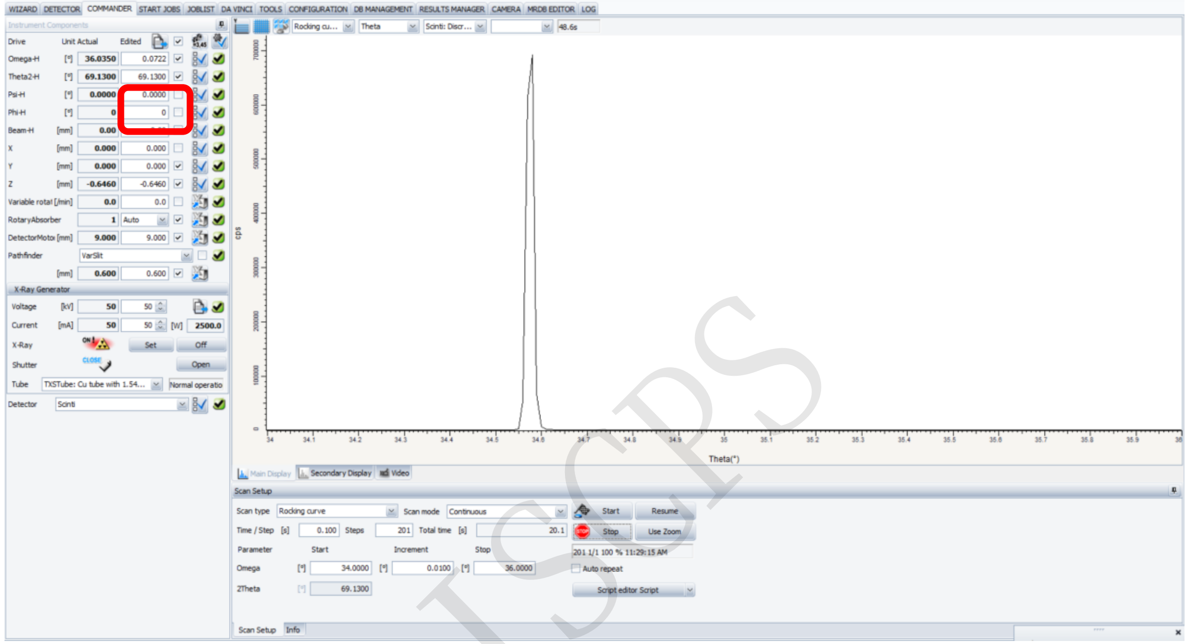

Fig 6-11 Rocking curve for substrate

6.7.7.2 Find the reference peak for unknown substrate (if not sure)

Under this circumstance, it’s essential to apply for 2Theta-Omega scan to determine the substrate and settings detailed as below:

Scan type: TwoTheta-Omega; Scan range of 68 ~ 69°, Increment 0.01°/step, time/step：0.1 s/step, double click to get 2Theta value for substrate and send it to Theta2-H drive (Fig 6-12);

Example: 2Theta-Omega scan with Omega offset checked

Quick scan for large range with large step size, then “Use Zoom” for small range with small step size

-Omega

Quick scan: 68~69°, step size: 0.01

Use zoom: 68.8~69.3°, step size: 0.005

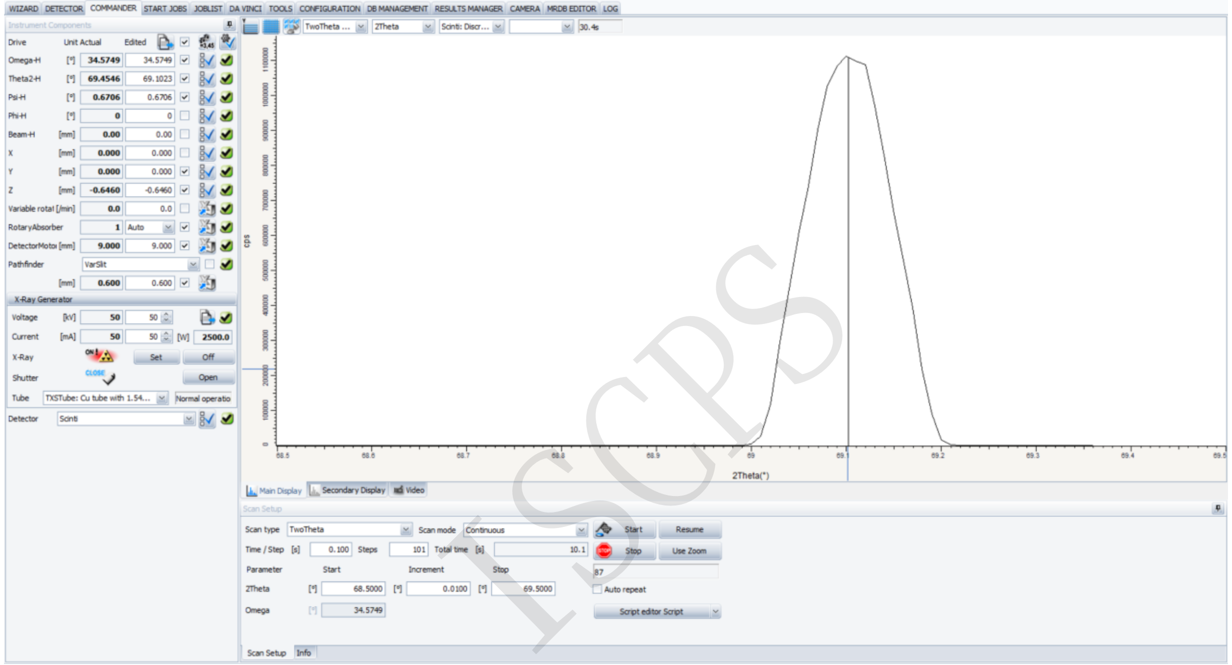

Fig 6-12 2Theta-Omega scan for substrate

Followed by rocking curve specified below:

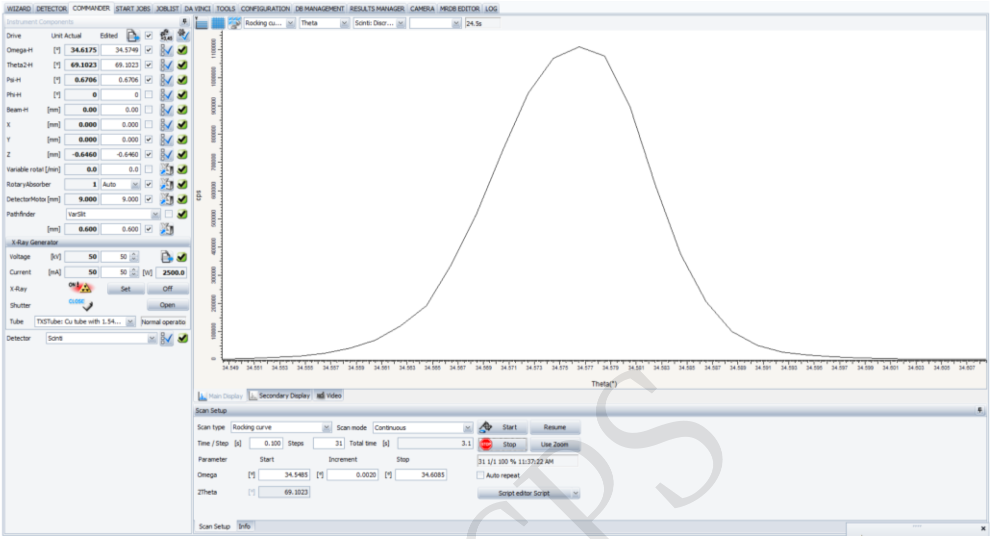

Fig 6-13 Rocking curve for substrate

Set parameters：Scan type: Rocking curve; Scan range of 34.5 ~ 34.7°, Increment 0.005°/step, time/step：0.1 s/step, double click at the peak and send the value to Omega-H drive.

6.7.8 Optimization of substrate peak

6.7.8.1 Optimization of Psi

Perform Rocking curve when Psi = 0: Omega range: 34~36°, Increment 0.005°/step, time/step 0.1 s/step, record the intensity from rocking curve and then manually input Psi value as 0.2, repeat Rocking curve, and mark the peak intensity; repeat Rocking curve for Psi = 0.4, 0.6, 0.8, 1.0, -0.2, -0.4, -0.6, -0.8, -1.0 and so forth on, take note for peak intensities to figure out the best one and then send corresponding values to Psi and Omega-H drives (Fig 6-13).

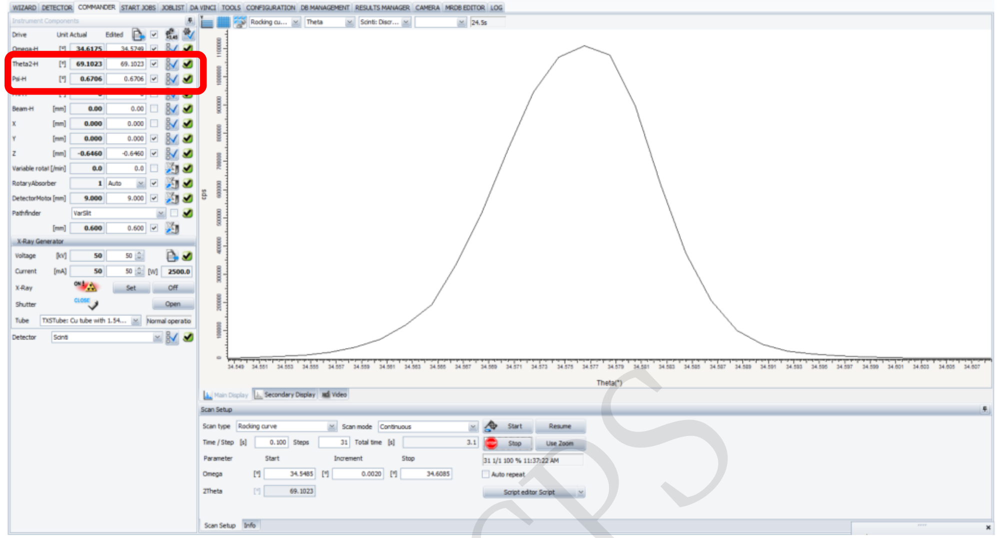

Fig 6-13 Psi and Omega optimization via Rocking curve

Example: Change Psi manually, and rocking curve to check the maximum intensity

Psi = 0.0, rocking curve counts max = 164,300

Psi = 0.2, rocking curve counts max = 190,270

Psi = 0.5, rocking curve counts max = 347,380 (compare to chi = 0, max = 163,310)

Psi = 0.65, rocking curve counts max = 373,100

Conclusion: Psi = 0.5 is best for diffraction.

6.7.8.2 2Theta optimization

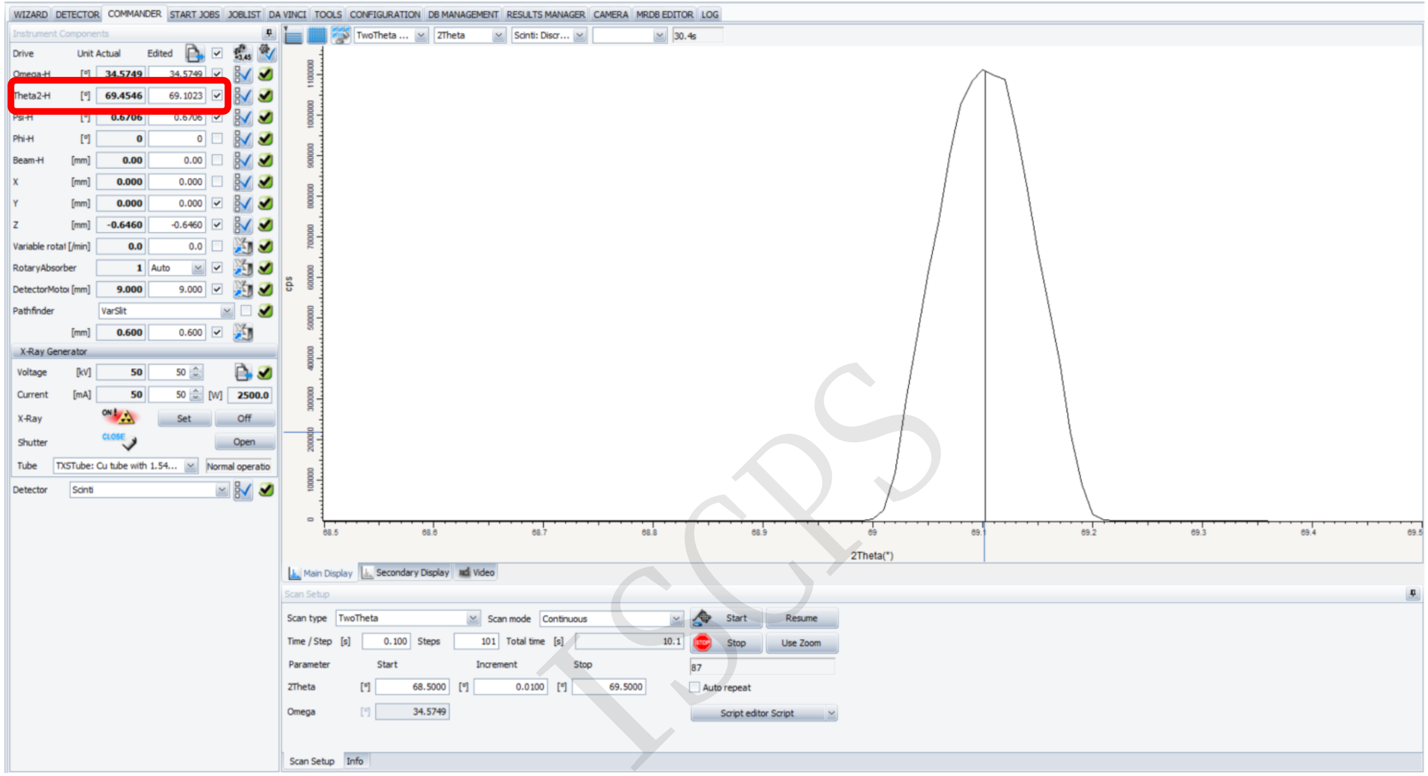

Fig 6-14 2Theta optimization

Apply 2Theta optimization after updating Omega:

Scan type: 2Theta, 2Theta range: 68~69°, Increment 0.01°/step, time/step 0.1 s/step, double click at the peak and send the value to Theta2-H drive.

6.7.8.3 2Theta-Omega scan for films

Parameter settings: Scan type: scan type: TwoTheta-Omega, 2Theta range: 65~70° (the 2theta range should be large enough to include peaks of the substrate and films).

6.7.8.4 Rocking curve for thin film

With 2Theta/Omega values checked (write down these two values in case needed later), perform Rocking curve for the film, save data as “.brml”.

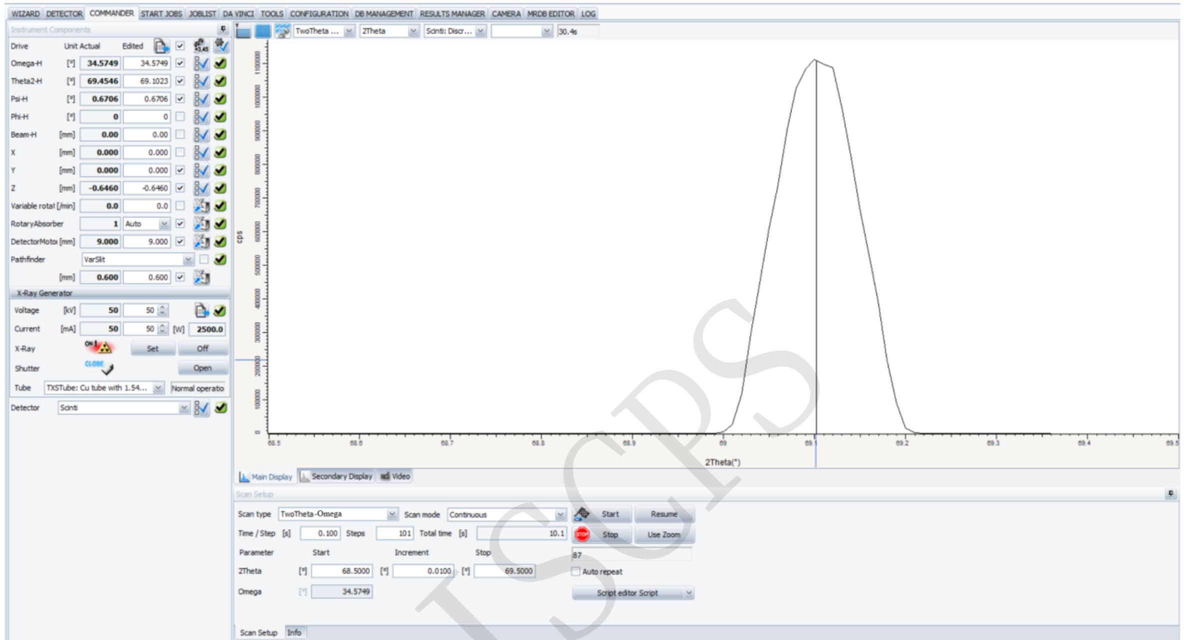

Fig 6-15 2Theta-Omega scan for thin film

7. Relevant supporting documents

Q/WU FLHR001 format file

8. Logbook

Logbook V1.0 for Bruker D8 Discover High-Resolution Thin-Film XRD.

Logbook										
Date	**User**	**PI**	**Sample ID**	**Sample details: composition and number**	**Test type**		**Sample recycle**	**Sample status**	**User contact**	**Comment**
					Service	**Independent operation**				

Attention: 1) Launch the experiment only when the instrument is under good condition. Once launched, it's regarded as good condition; 2) In case of any problems, contact staff.