
The image shows the cover of a spiral-bound notebook. The cover is a light tan or beige color with a subtle, textured pattern. On the left side, there is a silver metal spiral binding. The notebook is set against a light yellow background. The text on the cover is centered and written in a dark blue, serif font.

***Bruker D8 Discover with GADDS
XRD Basics
Training Notebook***

***NanoTech User Facility (NTUF)
Center for Nanotechnology
University of Washington
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- I. Safe Operation
 - II. Component Overview
 - III. Software
 - IV. Bruker GADDS Operation (powder diffraction)

I. Safe operation

- **In apparent EMERGENCY ONLY press the big red button on the right or on the left for complete shutdown of the system.**
- **X-Ray shutter and cabinet access**

☞ The x-ray shutter can only open when the cabinet doors are closed and locked. Radiation safety is assured. To open the cabinet doors, you must press the green “Open Door” button on the right-hand or left-hand side of the cabinet. If the shutter is open when this button is pressed, it will close before unlocking the doors.

- **Area Detector Safety**

☞ **The GADDS area detector is the most expensive component of the system (\$100K). It can be permanently damaged if the following rules are not followed.**

- **Beryllium window**

☞ The active area of the detector is covered with Beryllium. This material is toxic and also very fragile. It is very important that it is never touched or bumped by anything.

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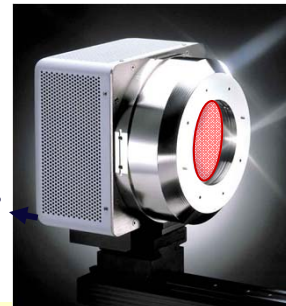
- **Detector Bias**

☞ Part of this detector is made up of a multi-wire grid that is under a very high voltage (detector bias ON). If the detector is moved or jostled by anything except the 2theta stepper motor, these wires can fuse together. Always turn the detector bias OFF before moving this detector (it requires a special training or ask the staff).

- **Direct Beam damage**

☞ If any part of this detector is exposed to the direct x-ray beam with no absorbers it will be immediately saturated and will quickly be permanently damaged. To avoid this, never position the detector at 2theta less than 20 degrees during a scan. If the detector is moved closer than the standard 15 cm, then this value will be even smaller.

Area detector



Continued...

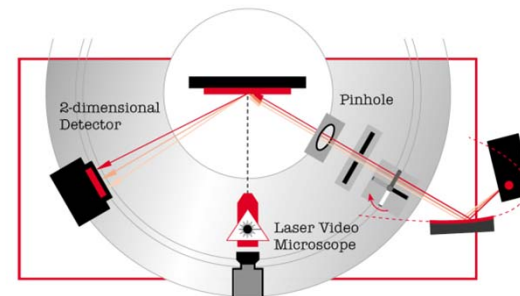
– Component collision

- ☞ This system is composed of many sensitive, fragile components, positioned close to one another, most of which are capable of movement. There is always a risk of collision. Although there are hardware and software collision limit switches designed to keep this from happening, they do not protect in every possible situation. The safest course is to manually move (see) all components through the most extreme range positions you wish to use in your scan and be sure that no collision occurs.
- ☞ Also, try to avoid collisions between instrument components and yourself when inserting or removing samples. In particular, try not to bump the laser/video system as it has been precisely aligned and any jostling may disturb this alignment.

II. Component Overview

- **X-Ray source and incident beam optics**

- Rotating Cu target
- Laterally graded, multilayer mirror (Gobel Mirror)



- Converts radiation into a 1.2 mm wide parallel beam of monochromatic radiation.

- Pinhole collimator (usually 0.5 mm diameter) which converts beam into a point.

- **Eulerian Cradle (goniometer)**

- Accurate positioning of sample. (see section)

- **Laser/Video alignment**

- A video microscope and a laser that allow precise positioning of sample X,Y, and Z coordinates. The cross-point of the laser beam and the optical axis of the zoom video are pre-aligned to the instrument center.



- **Area Detector**

- Multi-Wire proportional area detector.

III. Primary Software (see shortcuts on the monitor)


- **GADDS software**

-  GADDS software for control, data acquisition and 2-D diffraction data processing.

- **Video**

-  Displays video microscope crosshairs and sample in a window in real time. Used to align sample and to take pictures of area probed.

- **EVA**

-  Short for Evaluation. Bruker-AXS analyses software for viewing/processing Intensity vs 2theta data, Search/Match using ICDD powder diffraction file database etc. (see EVA application notes for details).

- **PDFMaint**

-  Searches ICDD data.

- **Merge**

-  Merges frames in the .raw file.

- **File transfer**

-  Generates data file in text format.

A graphic of a spiral-bound notebook with a brown cover and a silver spiral binding on the left side. The notebook is open to a white page with a yellow border. A horizontal line is drawn across the page, just above the main title.

IV. BRUKER GADDS OPERATION

A. System Startup

☞ The X-ray source should be stabilized for 30 minutes before taking measurements. Therefore, the first thing to do is to ramp up from default standby power of 20 kV, 10 mA to the standard operating power of 40 kV, 120 mA.

– Ramping up Procedure

☞ Make sure the enclosure doors are closed and locked and the Alarm indicator (red LED) is not on.

☞ Open GADDS software.



☞ Click **“NO”** at “Set generator to user settings: 40 kV, 120 mA?”

☞ The current power setting is seen at the bottom right corner of the GADDS window: KV 20; MA 10

- Use the script **@ramp_up to ramp up power**

☞ Enter Command Mode: From the menu, Special -> commandmode

☞ Type @ramp_up and press Enter.

☞ View the voltage and current increase in the bottom right corner of the GADDS window.

☞ Wait 30 minutes before taking measurements. During this time you can mount your sample, do sample alignment and setup scan conditions.

B. Mounting the sample

- **To open access to the sample space:**

- ☞ From the GADDS menu, choose Collect -> Goniometer -> Drive
- ☞ In the window which will open, enter $2\theta=20$; $\omega=0$; $\chi=90$; press OK.
- ☞ **Monitor the instrument as drives move and quickly hit any key on the keyboard to stop movement if you think a collision may occur.**

- **To mount the sample:**

- ☞ If you use the standard disc-shaped sample holder with sticky tape, just attach your sample onto the tape. Have your second hand under the sample to prevent dropping the sample or its parts on the goniometer gears.
- ☞ If you would like to use a different holder (say the vacuum chuck) please contact the staff to be trained first.

- **Notice:**

- ☞ **Be extremely cautious not to touch or move any parts in the tool enclosure.**
- ☞ It is a good practice to always use gloves at this stage.

C. Moving the detector – ask staff

- 📄 If you need to move the detector along the rail, please contact the staff!
- 📄 The system requires re-calibration after every move (done by staff)


D. Align the sample

- In Collect -> Goniometer -> Drive window, enter $2\theta=60$; $\omega=60$; $\chi=67.5$

☞ Monitor the instrument as drives move and quickly hit any key on the keyboard to stop movement if you think a collision will occur.

- Start the VIDEO software , if it is not running, by double-clicking the icon



☞ You may have to click the “start the grab” button () to view the current image of your sample on the computer screen.

- Enter manual mode: Collect -> Goniometer -> Manual

☞ Using the hand-held remote control, you can move the sample in small increments. Use the video camera crosshair to align the sample in X and Y.

☞ You may need to use the lamp and the ZOOM drive on the camera and/or adjust the lamp to help you see the sample.

☞ When changing the ZOOM, do not go below 2 or above 6!

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- Turn on the laser (hit the L button)

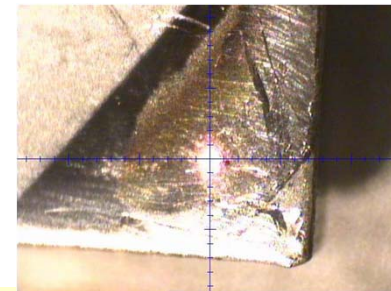
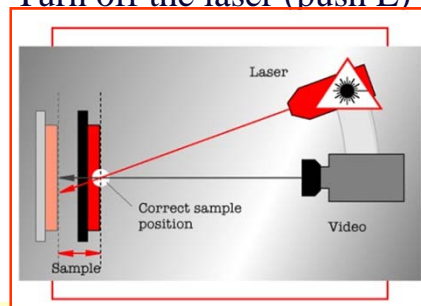
Using the hand-held control tool, adjust the depth position (Z) until the laser spot appears in the crosshair of the VIDEO (refer to figures below)

Notice: You may see two reflections if the sample was a (semi)transparent film or a loose powder on a substrate; in this case use the upper spot for Z alignment.

For a thick sample/substrate, or if the previous user had moved the disc sample holder to an uncommon position, you may need to adjust the holder position manually (use the tools in the enclosure) before finely adjusting Z with the manual control box.

If the lamp has been on during mounting, turn it off and move it out of the way of intended detector movement.

- Turn off the laser (push L) and exit the manual mode (Esc button)



E. Planning a measurement

- If you have an idea of what your sample is and want to know the best measurement range, check out the ICCD data from PDFMaint.

- 📄 Click on the PDFMaint icon
- 📄 Open C:\dprd\jcp2.2ca
- 📄 Click search and identify your search criteria.
- 📄 The more reliable PDF cards are of quality: I (indexed), * (high), and C (Calculated).
- 📄 For known compound, mark the required elements GREEN, possibly present elements GRAY, all others – RED in the periodic Table.

F. Create your project or working directory

- GADDS sets up a working directory in C:\Frames\'year\'\'month\'YOUR PROJECT
- ☞ Copy an existing project: On the menu bar, click Project->Copy
- ☞ Type your new project name in Title and your new working directory in Directory
- ☞ Click OK
- ☞ A new directory will be created and calibration files copied there. You will be able to find your directory in “Frames”
- ☞ **Notice:** Using USBs is forbidden on the tool’s computer. If you need to take your data home, they should be first copied to the shared drive after completing the measurements.

G. Taking measurements

- 📄 On GADDS, click Collect -> Scan -> Single run
- 📄 Make sure the OK button or Enter is pressed only when the setup is completed to avoid false start.
- 📄 To cancel a measurement after it has started, press Ctrl+C
- 📄 For a sample scan, to cover 2θ approximately from 15 to 95 degrees (in 3 frames), see next page.

Sample measurement with three frames

Options for Collect Scan SingleRun

Frames Seconds/frame

2-Theta deg Omega deg Phi deg Chi deg

X mm Y mm Z mm Aux mm

Scan Axis #

Mode Rotate sample Sample Osc Amplitude mm

Frame width

Frame header information

Title

Sample name

Sample number

Filename generation

Job name Run # Frame #









First filename

Max display counts Realtime display

Pre-clear Capture video image Auto Z Align N

Options for single run (suggested settings)

For any of the drives, simply enter “@” to use the value of it’s current position.

- | | |
|---|--|
|  # Frames | How many area detector exposures in this run. |
|  Seconds/frame | Amount of time area detector is actively counting during the frame exposure. |
|  2-Theta | Starting position of the center of the area detector. Coverage is about +/- 15 degrees from this point. (Note: minimum value is 20 degrees at 15 cm detector distance, and bigger at shorter distances!) |
|  Omega | generally should be ½ of 2-theta |
|  Phi | @ |
|  Chi | @ |
|  X,Y,Z | @ for all |
|  Aux | Zoom value – use @ |

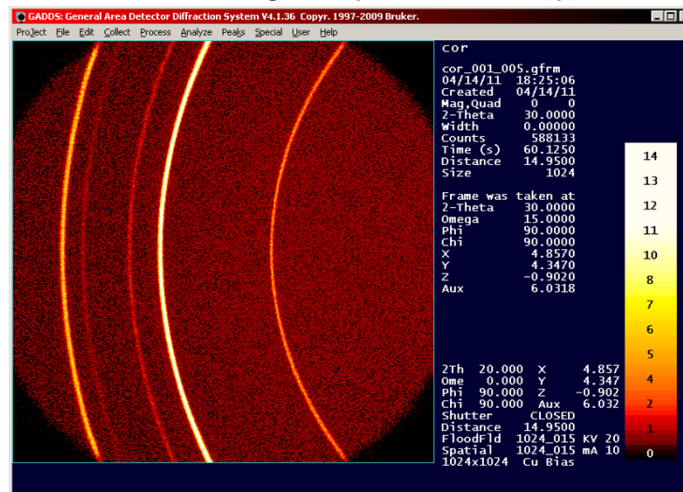
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- 📄 **Scan Axis #** drive number to change from frame to frame
(None if only 1 frame)
- 📄 **Frame width** Amount to change drive # between frames (can be positive or negative)
- 📄 **Mode step/scan** Set to STEP
- 📄 **Rotate Sample** If checked, rotate sample during scan to improve sampling statistics (check for collisions)
- 📄 **Sample Osc** Small oscillations in X,Y, and/or Z to improve sampling volume
- 📄 **Amplitude** Amplitude of oscillations
- 📄 **Frame header Info** All refers to information that will be displayed on each frame

- 📄 **Filename generation** Filename is generated from combination of all 3. Frame # automatically increments by one from frame to frame.

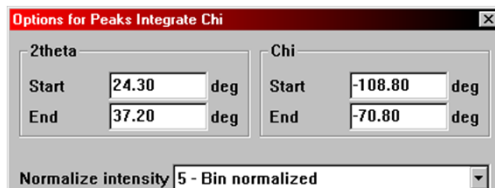
Starting measurement

- Double check all of your entered values and click *OK*
- Watch the instrument as it moves to be sure no collisions will occur.
- Ctrl – C stops movement or scan if you wish to end early.
- Frames look like this for a good powder sample:

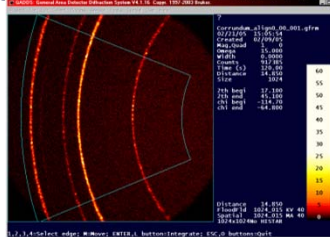


Analyzing a scan

- Bring up the frame you wish to analyze using Ctrl – left or right arrow.
- After you display the frame, click *Peaks ... Integrate ... Chi ...* and click *OK*. Make sure **Normalize intensity** is set to “5 – Bin normalized” (refer to figure below)



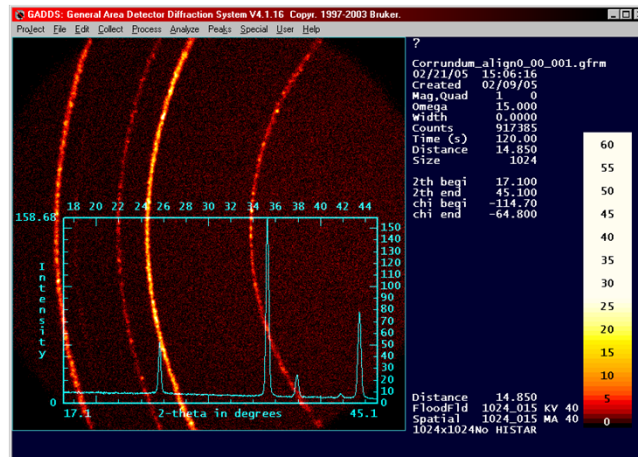
- You can now graphically choose the area over which you would like to integrate (blue line indicates the area). The 1,2,3, and 4 keys allow you to change the 4 edges. (refer to figure below)**



- ** It is recommended that you not integrate over the very edges of the detector (especially high and low 2theta)

Continued...

- Click *Enter* when finished and the Intensity vs 2theta scan will appear (refer to figure below)



Continued... Saving data

- a) Save your data.
- i) Choose a Title for this data file.
- ii) The File name defaults to the name of the frame minus the frame number. Change this if you like.
- iii) In the Format box choose:
 - DIFFRACplus* Bruker-AXS binary file (if you wish to use other Bruker software with this data)
 - PLOTSO* 2Theta vs I values saved in text format
- iv) Selecting the **Append Y/N** checkbox means that if this file already exists, it will add the current data to the existing file. This can be useful if you have more than one frame from the same sample.
- v) Due to the sensitivity of this area detector, background and peak intensities can be much lower than traditional point detectors. To ensure that meaningful intensity data is not “rounded” or “truncated” (e.g. 2.345 counts rounded to 2) by other software, it is recommended that you use a **Scale factor** of 100 or 1000.

