KMC2 - diffractometry

(version 2006_09_10)


6-circle diffractometer (HUBER)

psi-geometry

4-circle diffractometer with omega/2theta-axis vertically, minimum step size: 2.5×10^{-4} degree.

Open Eulerian cradle (5 kg load) equiped with an xyz-stage.

Two additional axes, mu and nu (minimum step size: 2.5×10^{-3}) to allow for non-coplanar scattering.

A motorized x-y-z stage is mounted at the sample stage. Two sets of motorized slits and one manually adjustable slit set are used to shape the beam and as collimation path for the detector arm.

(beam propagates in y-direction)
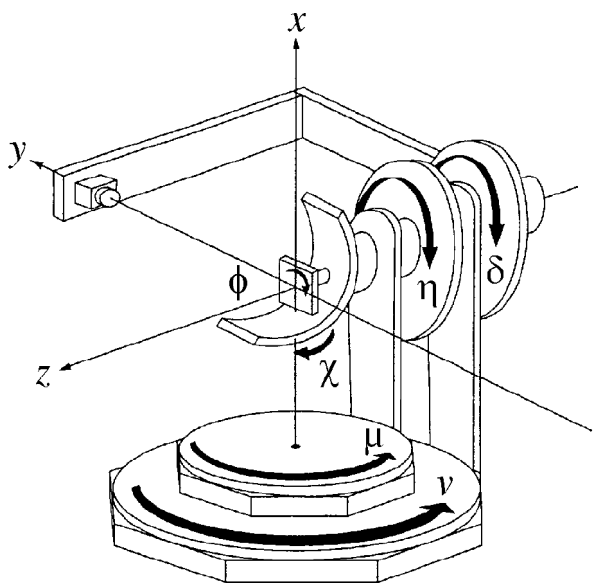
Scintilation detector:

Cyberstar X1000 by Oxford Danfysik is used for high-resolution measurements. Detector parameters can be changed from the computer.

2D-detector:

gas filled wire detector by BRUKER AXS

active area: 115 mm in diameter

energy range: 3 - 15 keV

spatial resolution: 150 μm

maximal overall intensity: 40000 c/s

maximal local intensity: 300 c/s/pixel in 512x512 mode

The total intensity can be read on the rate-o-meter mounted in the 19" rack.

Ionisation chamber:

IC Plus with 50 mm active length incl. "piggy back".

electronic module for minimization of noise

There are commands to change the voltage and the range of the ionisation chamber, but they are not necessary for simple measurements.

High-temperature vacuum chamber

A single-sample diffractomtry can be mounted on the goniometer instead of the x-y translation stage. Vertical translation and all goniometer axes are available, but, due to different outlets on the chamber, in limited range. Chamber is able to heat up to 1000°C.

Axes names

Huber:

Sample axes: *mu, eta, chi, phi*
Detector axes: *nu, del*

Slits: JJ X-ray slits move each knife independently. There are two slit systems, SD and SF, so the motor names are : *SF_t*, *SF_b*, *SF_l*, and *SF_r* for top,bottom, left, and right knife of the SF slits respectively, and *SD_t*, *SD_b*, *SD_l*, and *SD_r* for SD slits.

Depending on the detector and configuration used, SD and SF have different usages:
1) 1D detector: SF is the beam definition slit, SD is together with SH (the manual Huber slit) used as the detector collimation slit.
2) 2D detector: SF is the beam definition slit.
3) 2D detector, diffuse scattering at small angles: SD is the beam definition slit, SF is the guard slit.

Except for the "mv" commands to move each single edge, there are special commands for slits:
*SF_open X , SD_open X* – opens the slits horizontally and vertically X milimeter.
beam_find_det_SF, beam_find_mon_SF, beam_find_det_SD, beam_find_mon_SD performs the scans to find the beam position, moves all slits to the center of the beam, resets them to 0, and opens them 0.1x0.1 mm. The "det" and "mon" commands use the detector or monitor to estimate the center of the beam.
beam_profile_SD, beam_profile_SF perform two scans resulting in horizontal and vertical profile of the beam at the slit position.

Attenuator: There is an attenuator wheel in the primary beam. With the command:
att X
one can insert X µm thick Cu foil in the primary beam. Available foils are: 0(no foil), 25, 50, 100, 150, 200, 250, 300, 500µm. 50µm Cu in the beam has 10% transmission for 8keV beam.
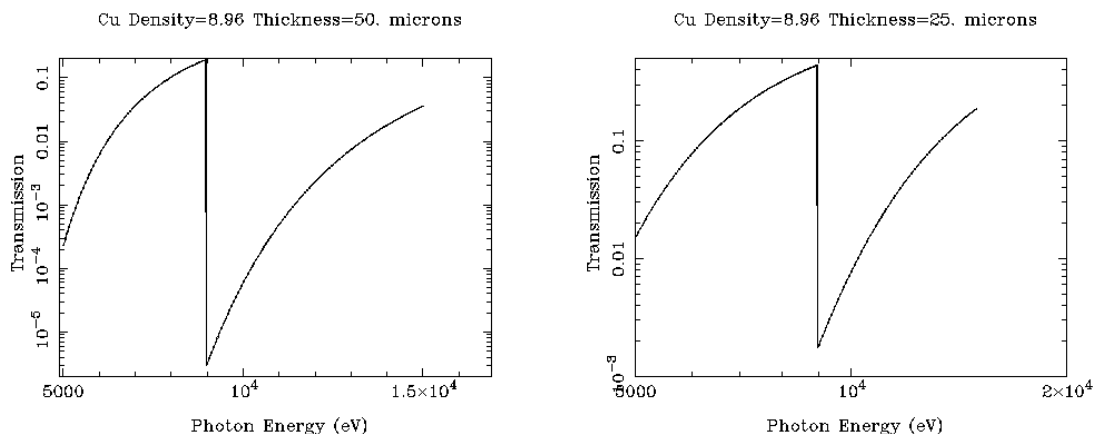
To ask which filter is currently in the beam use the command:
att_get
The motor moving the filter is named "*att_w*", and is written in spec files for every measurement.
The attenuator motor has no limit- or home-swithes. At the beginning of the measurement (with the main shutter closed) do *att 0* and than manually turn the wheel until the filter 8 (0 mu thickness) is in the beam.

Spec commands

Starting the measurement:

newfile YYYY_MM_DD_samplename.spec
opens the new file and starts writing the results in it. Do it for every new sample. The format of the filename is free, but the proposed format helps to find the files and sort them without opening.

Moving motors

The basic command for moving the motors is "*mv*". It tells spec to move the specified axis to specified position.
mv motor position – moves the motor to absolute position
mvr motor distance – moves the motor specified distance relative to current position
umv, umvr – the same but showc the updated coordinate on the screen while moving

home motor – starts the home-searching procedure.

To ask for the position of motors, on can type
wa
and the positions of all motors are printed to the command interface.

Counting

to perform counting for 10 seconds type:
ct 10
to count 1 second do:
ct

Scans

Simple scan with one motor use ascan macro:
ascan motor start end steps time
e.g.:
ascan del 20 30 10 1
moves motor *del* to positions 20, 21... 29, 30 (10 steps, 11 measurements) and counts 1 second at each position. Motor stays at the end position after scan.
To move two motors synchronously do
a2scan motor1 start1 end1 motor2 start2 end2 steps time
Instead of the absolute coordinates for scan, it is possible to give the positions relative to current position of axes. Instead of *ascan* use *dscan*. To perform a theta-2theta scan around a selected peak, optimize the intensity by moving all motors and then do:
d2scan del -1 1 eta -0.5 0.5 1000 1

Mesh

Mesh is a special form of scan where two motors are moved independently to scan a region in two dimensions...

2D detector

To setup spec for the use of the 2D detector, start the 'slave to spec' program at the windows computer (if it is not running) and type to the spec command prompt:
GADDS_on
You will be prompted for the wavelength and the distance between the sample and the detector. These values are used later as the start values for the calibration, they don't have to be accurate. (but the more they are accurate, the simpler the calibration)
To stop the acquisition of the area frames write:
GADDS_off
When in GADDS modus, all scans automatically acquire a 2D image for every single count command in the scan. The command "*ct*" is not changed, i.e. It only writes the total number of photons in the detector to the terminal line. No images are saved. To take only one frame use the command:
acquire X
This is going to start a scan with only one point and "X" seconds acquisition time and save an image to the hard disk.
The complete procedure of taking the correction and background files for the area detector is described elsewhere (Bruker AXS manual).

GID measurement

GID geometry is realized in the way that the scattering defracts the beam in vertical direction. This geometry does not require the polarization correction for the scattered beam. Sample is positioned vertically (surface normal is horizontal) at the goniometer by moving chi to 0. Axes *nu* and *mu* are used to set the α_i and α_f, the grazing incident and exit photon angle. Since phi and eta are colinear in this geometry, there is a possibility to rotate *phi* for large angles, but scanning and measurements should be performed using *eta* due to it's precision. For easier evaluation and understanding of data, *eta* should be *del/2*.

Sample is mounted on the goniometer head using Aluminum plates. Sample can be fixed to the plate using wax or double-sided adhesive tape.
Move motors to following positions:

mu	*nu*	*chi*	*eta*	*del*	*phi*
0	0	8	0	0	0

Switch the laser on at the control panel outside of the hutch. By moving the laser assure yourself theat the laser is hitting the sample. Close the left door of the hutch and mark the position of the laser spot on a peace of paper. in spec type *mv phi 180*. This would produce the second spot. Mark it, draw the line between two points, and find the middle. Repeat this procedure for *phi* values of 90 and 270. Correct the position of the laser spot into the middle of the cross by adjusting the goniometer head. Do *mv phi 0,* and if the laser spot does not move, the sample surface normal is paralel to the axis of phi rotation. Switch the laser off.
WARNING: Try not to look at the sample when the laser is switched on. Although the laser intensity is really low looking directly in the laser (or in the beam reflected by the sample) can seriously damage your eyes. If you keep all axes at values from the table, and left hutch door is closed at all times when the laser is switched on, you're probably safe.

mu, nu, α_i, and α_f

It is not straight-forward to use a psic goniometer for GID scattering. psic angles are different than the 'six-circle surface diffractometer'. Although all degrees of freedom are allowed, The detector arm coordinates *nu* and *del* are not equivalent to α_f and 2Θ.
Incident angles are simple: $\mu = \alpha_i$.
Exit side is much more complicated. nu=α_i+α_f is OK only in reflectometry, or in approximation for small delta angle.
To calculate the geometry from motor positions use:
$$\sin(\alpha_f) = \cos(\delta)\sin(\nu - \mu)$$
$$\tan(2\Theta) = \frac{\tan(\delta)}{\cos(\nu - \mu)}$$
And to calculate the motor positions from known geometry:
$$\sin(\delta) = \sin(2\Theta)\cos(\alpha_f)$$
$$\tan(\nu) = \frac{\cos(2\Theta)\cos(\alpha_f)\sin(\alpha_i) + \cos(\alpha_i)\sin(\alpha_f)}{\cos(2\Theta)\cos(\alpha_i)\cos(\alpha_f) - \sin(\alpha_i)\sin(\alpha_f)}$$

2Θ is the projection of the scattering angle to the surface, α_i and α_f are incident and exit grazing incidence angles. Be careful for *del* angles higher than 90°!!

Adjusting the sample surface in the beam.

There are different procedures depending on the orientation of the sample and on the surface properties of the sample, but since they all include the same elements, they'll all be described here.

1) Out enough attenuator so the detector can survive the primary beam. It is good to set the higher value and then gradually remove filter if necessary.
2) remove the sample from the beam by typing *umvr z -2*. This is going to move the sample 2mm out of the beam.
3) do relative scans in nu and del and move nu and del into the position where maximal intensity was measured. Detector is set now. This step is not necessary if you're using the area detector. The equivalent action is performed by saving a picture of the primay beam, which is later used to estimate the position of the detector center.
4) move the sample into the beam until only half of the intensity is measured. Sample shadows half of the beam now. Do this by typing *mvr z 0.5* until the beam vanishes (look at the rateometer or perform a *ct* after every movement). When near the position do *dscan z -1 1 20 1* and move motor z to the position with ½ maximal intensity.
5) To be sure that the sample is parallel to the beam do a rocking-curve scan and move the axis in maximum. If the sample normal is pointed to the roof (standard symmetrical diffractometry) then you have to scan *eta*, if in GID geometry, you'll have to scan *mu*.
6) repeat 4 and 5 if necessary.
7) If the samples are highly reflecting, this procedure may lead to an error. If the samples do not reflect, than the procedure is over, and the sample is aligned. Set the corresponding angles to 0. Points 8 to 10 are only for reflecting samples.
8) move detector to an angle which is larger than the double angle of total external reflection, e.g. to 1 deg. If you did rocking optimization with *eta*, than you'll have to move *del* now, if you optimized *mu* in 5) than you'll have to use *nu*.
9) Move the sample to the half of this angle. Perform the rocking curve and move the sample angle to the position where the maximum was detected. Set the sample angle to the half of the detector angle.
10) Steps 8-9 are somewhat complicated when using the area detector. One has to estimate angle resolution of the detector and monitor the position of the reflected beam. Leave this to experts...

Writing in Logbook

Although most measurement parameters are automatically saved by the controller program, there are some things which need to be written in the logbook. The preferred way to write the measurement diary is to write in the beamline logbook, users can get a photo-copy of the diary.

Things which are normally written in the logbook:
When starting the experiment or when changing the setup it is necessary to describe the experiment. The beamline can be used to perform several different types of experiment, so it is necessary to declare which experiment are we performing (XRD, powder diffractometry, GID, GISAXS, constant incident angle, constant exit angle, are we using capillaries or goniometer head, is the area detector going to be used, which slits are used for collimation...). A nice drawing or printed photo is good, especially if the experiment should be repeated after few months.
A short description of the system which is going to be measured is also helpful. A short note is enough.
Examples:
– Si-(001)-wafer, GID on 220 reflection, scanning for satelite peaks.
– TiH powder at temperatures up to 700°C in air and in vacuum, expected patterns: TiH_2, α-Ti, β-Ti, different oxides

At every *newfile* command, or when replacing the sample, a short description of the sample should be written in the book. This is the only way to correlate the experiment with the sample. If all samples are equivalent, a sample number would be enough. If there are ambiguities in the way the sample can be mounted, the mounting should be also described. This helps a lot when we are repeating the experiment.

Since during the measurement many unnecessary scans are performed, for every significant scan a scan number and the description should be noted. It is not wrong to write the actual command, if we are going to repeat this scan for next sample.
Example:
Scan 2 – sample height adjustment, sample height z=1.43
scan 25 – *d2scan del -1 1 eta -0.5 0.5 100 1* - theta-2theta scan around 002
scan 26 – background for scan 25

There are parameters which are NOT saved. These parameters are different for different experiments. Here only few examples:

– High precision diffractometry: There are three pairs of slits in the beamline, two of them are motorized, one is not. If the manual slit is used, it is necessary to write every single change in the log book.
– Area Detector: when using the area detector, the detector distance is very important parameter. When using the point detector, distance is important only if we are interested in the device resolution or in absolute scattered intensity. All distances between sample and slits and between sample and detector should be written in the logbook.
– High temperature chamber: Only one thermoelement is used by the controller and written in the file. All other parameters are not monitored by the computer. The most important of these values is the second thermoelement. During a heating experiment users should compare these two values and decide if both thermoelements are still connected to the stage. In case of large differences the heating should be interrupted. Other process values can also point to malfunction of the device, or to explain why an experiment didn't work (low vacuum would explain the oxide peaks in the scattering curve, high heater resistance can point to malfunctioning heater...). The set of values which should be written down at few points during the measurement include: time, temperatures of all thermoelements, pressure, voltage and current at the power supply.
– Wavelength or energy of the primary beam.