

Beamtime mg25682-2

(09/03/2021 – 12/03/2021)

Energy Calibration:

CeO₂ (in a 0.5 mm thick flat shim)

103785.nxs >> scan t3.m2z 850 1250 100 pilatus_eh1_sw_cbf 30

Beam size = 0.5 mm x 0.5 mm

Dawn calibration (Automatic routine, None fixed, images used from 850 to 1250, 8 peaks used):

➤ **E = 99.801 keV, lambda = 0.12423 Å**

Checking different exposure times:

t3.m2z = 927

beam size 0.5 mm x 0.5 mm

CeO₂: 103764.nxs

Dawn calibration (Automatic routine, fixed energy at E = 99.801 keV, 12 peaks):

- calibration file "calibration_nxs=103764_t3m2z=927_checkExpTimes.nxs"
- detector position at t3.m2z = 927 corresponds to the sample-to-detector distance of 908.54

nxS	Ti64 50% reduction sample	expTime [s]
103789	6	1
103790	6	0.5
103791	6	0.25
103792	6	0.1
103793	6	0.05
103794	6	0.025
103795	6	0.015
103796	6	0.01
103797	6	0.008
103798	6	0.006
103799	6	0.005
103800	6	0.004

Data integration:

- using the pipeline "pipeline_CakeRemapping_nxs=103764_t3m2z=927_checkExpTimes.nxs".
- data integrated: files from 103789.nxs to 103800.nxs

Stacking of samples from 87.5% deformed samples

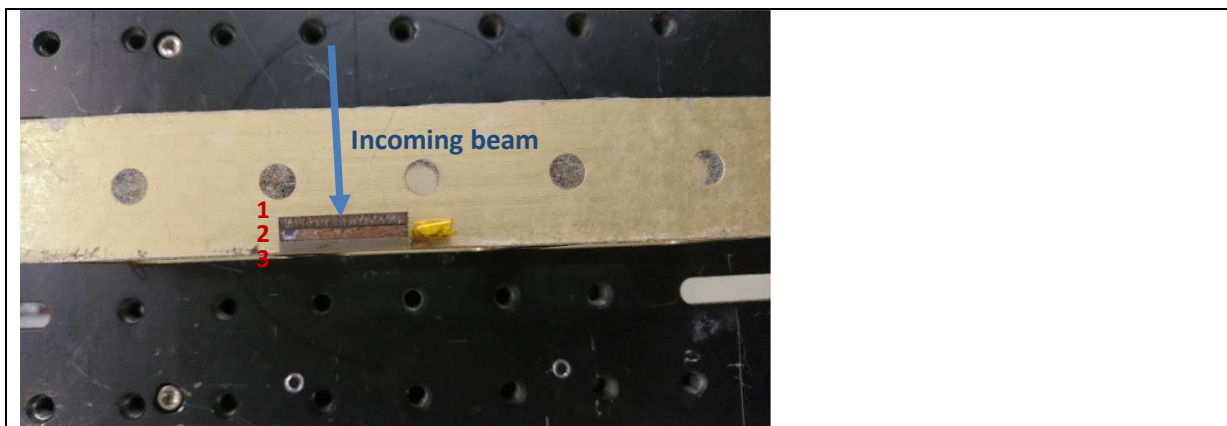


Fig. 1. The example of the experimental mounting of stacked samples (in concrete 1&2&3) and the CeO₂ sample positioned next to the stacked sample on a sample stage. The CeO₂ sample is used for the sample-to-detector distance calibration.

CeO₂ (in the nut): 103801.nxs, exp time 30s; t3.m2z = 757 (“short sample-to-detector distance”)

Dawn calibration (Automatic routine, fixed energy at E = 99.801 keV, 12 peaks):

- calibration file “calibration_nxs=103801_t3m2z=757_StackedSamples.nxs”
- the detector position at t3.m2z = 757 corresponds to the sample-to-detector distance of 740.6

nxs	sample	x_start	y_start	x_end	y_end	t3.m2z	D
103802	“1&2&3”	11.9	38.9	32.7	43.3	757	740.6
103803	“2&1&3”	11.9	38.9	32.7	43.3	757	740.6
103804	“2&3&1”	11.9	38.9	32.7	43.3	757	740.6

Data integration:

- using the pipeline
“pipeline_CakeRemapping_nxs=103801_t3m2z=757_StackedSamples.nxs”.
- data integrated: files from 103789.nxs to 103800.nxs

Ti64 powdered sample in the capillary

- Capillaries are mounted in the goniometer (and aligned using the EH1end camera) and are rotated around the vertical axis “z” during data collection. The speed of rotation was 90 deg / second and the theta stage moved from -1 deg to 180 deg. The beam size was 0.5 mm x 0.5 mm.
- LaB6 standard (in a capillary with 1 mm diameter) and Ti64 powder is in a capillary with 0.8 mm diameter).

nxs	sample	t3.m2z	D [mm]	
103814	LaB6	744	735.9	calibration_nxs=103814_t3m2z=744_PowderedSample.nxs
103815	LaB6	914	906.0	calibration_nxs=103815_t3m2z=914_PowderedSample.nxs
103817	Ti64 powder	914	906.0	-
103818	Ti64 powder	744	735.9	-

Data integration:

- Using the pipeline for cakeRemapping with the corresponding calibration file.

Ti64 bulky samples



Fig. 2. Ti64 bulky samples mounted on the holder together with CeO2 flat shim samples placed at the edges of the holder.

Theta stage (ss1.theta) was optimized to get the sample-to-detector distance to be same for both flat shim CeO2 specimens mounted together with Ti64 bulky samples.

ss1.theta = -0.25 CeO2_right: 103825.nxs → D = 905.84 mm (detector stage t3.m2z at 927)

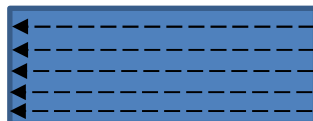
CeO2_right: 103826.nxs → D = 905.86 mm (detector stage t3.m2z at 927)

CeO2 (in the nut) at the sample position (see Fig. 2)

Dawn calibration (Automatic routine, fixed energy at E = 99.801 keV, 12 peaks and 10 peaks)

nxs	sample	t3.m2z	D [mm]	
103852	CeO2	757	737.14	calibration_nxs=103852_t3m2z=757_BulkySingleSamples.nxs
103853	CeO2	927	907.29	calibration_nxs=103853_t3m2z=927_BulkySingleSamples.nxs

Samples are scanned line by line from the top right corner (x_start, y_start) to the left bottom corner (x_end, y_end) with the same step size of 0.5 mm in both horizontal and vertical directions.



```
>> scan ss1.y1 y_start y_end step ss1.x x_start x_end step pilatus_eh1_sw_cbf expTime bm
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The master script for data collection was scanSamples_AllSamplesAgain.py. It is saved in the directory /dls/i12/data/2021/mg25682-2/scripts/

nx	sample	x_start	y_start	x_end	y_end	t3.m2z	D
103828	87.5% S6	-122.6	39.1	-101.6	43.1	927	907.29
103829	87.5% S5	-100.7	39.1	-79.7	43.1	927	907.29
103830	87.5% S4	-78.8	39.1	-57.8	43.1	927	907.29
103831	87.5% S3	-56.8	39.1	-35.8	43.1	927	907.29
103832	87.5% S2	-35	39.1	-14	43.1	927	907.29
103833	87.5% S1	-13.2	39.1	7.8	43.1	927	907.29
103834	50.0% S2	12.4	34.1	22.4	43.1	927	907.29
103835	50.0% S1	23.2	34.1	33.2	43.1	927	907.29
103836	50.0% S6	42.7	34.1	52.7	43.1	927	907.29
103837	50.0% S5	53.8	34.1	63.8	43.1	927	907.29
103838	50.0% S4	65	34.1	75	43.1	927	907.29
103839	50.0% S3	76.2	34.1	86.2	43.1	927	907.29
103840	87.5% S6	-122.6	39.1	-101.6	43.1	757	737.14
103841	87.5% S5	-100.7	39.1	-79.7	43.1	757	737.14
103842	87.5% S4	-78.8	39.1	-57.8	43.1	757	737.14
103843	87.5% S3	-56.8	39.1	-35.8	43.1	757	737.14
103844	87.5% S2	-35	39.1	-14	43.1	757	737.14
103845	87.5% S1	-13.2	39.1	7.8	43.1	757	737.14
103846	50.0% S2	12.4	34.1	22.4	43.1	757	737.14
103847	50.0% S1	23.2	34.1	33.2	43.1	757	737.14
103848	50.0% S6	42.7	34.1	52.7	43.1	757	737.14
103849	50.0% S5	53.8	34.1	63.8	43.1	757	737.14
103850	50.0% S4	65	34.1	75	43.1	757	737.14
103851	50.0% S3	76.2	34.1	86.2	43.1	757	737.14

Data integration:

- the pipeline “pipeline_CakeRemapping_nxs=103853_t3m2z=927_BulkySingleSamples.nxs” was used to process files from 103828.nxs to 103839.
- the pipeline “pipeline_CakeRemapping_nxs=103852_t3m2z=757_BulkySingleSamples.nxs” was used to process files from 103840.nxs to 103851.nxs.

General comments about data storage.

All required information can be found on <https://www.diamond.ac.uk/Users/Experiment-at-Diamond/IT-User-Guide.html>. Data are stored and accessible on the Diamond file system for up to 40 days from the date of you visit. See [here](#) for how to access data. After 40 days, your data will be available from the tape archive. See [here](#) for accessing your data from the archive.

For the beamtime mg25682-2, the main directory is “/dls/i12/2021/mg25682-2/”.

- Raw data are saved in subdirectory “.../rawdata/”.
- Processed (integrated) data are saved in subdirectory “.../processing/integrated/”.
- Dawn calibration and pipeline files are saved in subdirectory “.../processing/DAWN/”.
- Jython scripts used for data collection are saved in subdirectory “.../processing/scripts/”.