Ma3921

From Id11

Thursday May 03rd

WL setup the experiment. energy 38 keV.

main data folder is

/data/visitor/ma3921/id11/

matlab licence check:

https://compweb.esrf.fr/public/nice/matlab_licence.php

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install DCT code within ma3921

downloaded the installation scripts from the sourceforge repository.

python dct setup.py -b master ~/idl1/dct -m 2013a -mp /sware/com/matlab 2013a

sample list

ammount of deformation is meant as total strain; ex situ indicates deformation performed at UCSB; in situ indicates deformation performed at ESRF

larger grain size (about 120 microns)

- T1 1.01 % strain ex situ EBSD and DIC
- T2 0.58 % strain ex situ EBSD and DIC
- T3 (strain) in situ EBSD and DIC
- T4 0.49% strain ex situ EBSD and DIC, DCT scan prior to arrival
- T5 (strain) in situ EBSD and DIC, labDCT scan by Nicolas, DCT scan prior to arrival
- T6 (strain) in situ, no EBSD
- T7 0.64 % strain ex situ EBSD and DIC

smaller grain size (about 80 microns)

- Ts1 0.44% strain ex situ EBSD and DIC
- Ts2 0.67% strain ex situ EBSD and DIC
- Ts3 0.83% strain ex situ EBSD and DIC
- Ts4 (strain) in situ, EBSD and step0 DIC
- Ts5 (strain) in situ, EBSD and step0 DIC

DCT analysis

sample T1

data folder for this sample is

```
/data/visitor/ma3921/id11/t1_/
```

install sample T1 (1% strain) in the beam, DIC face upstream, notch up. detector distance approx 5 mm. found Pt mark 562 microns from the center. Will do DCT scans 600 microns beam height with 100 mirons overlap in total and 50 microns off center to match DIC region.

• first scan 50 microns off center with starting at diffrz = 90, we can change this value by adjusting the FTOMO par["start pos"]

```
fasttomo360 t1_dct_top_
```

• second scan 550 microns off center with starting at diffrz = 90

```
fasttomo360 t1_dct_top2_
```

• first scan was indeed and reconstructed, distance fitted to 6.51 mm

```
gtMatchGUI
```

grain 10 showed both basal and prismatic slip in the DIC. trying topotomo alignment.

```
calc_poles_tilt_id11_index2(p, 10, [1 2 3 4 5 6 7 8])
```

found two reflection to align and tested the second one sucessfully

reconstruction of t1_dct_top2_

if not ma3921 user, use ssh (pw Vur67+95):

```
ssh -X ma3921@rnice8
```

reserve a gpu machine on rnice8

```
oarsub -I -l walltime=12:00 -qgpu
```

launch matlab dct session

```
~/idl1/dct/dct_launch.py
>> addpath ~/idl1/scripts
```

launch a batch job monitoring gui

```
~/idl1/dct/dct_launch.py batch gui
```

- gtSetup, change output path to /data/visitor/ma3921/id11/t1/t1_dct_top2_, define number of phases (1), and check camera x distance (6.51)
- interlace scan -> need to copy references for the end of the second turn: use copy refs('t1 dct top2 ') from 0 rawdata folder
- gtPreprocessing (from base directory + monitor jobs in the DCT Batch GUI and resubmit any job that did not finish)
- gtSegmentationGUI (accept default parameters, adjust threshold levels and 'Save and launch' segmentation)
- gtMATCHGUI (Pre-Match, remove families > 8 deg, thr_theta=0.1 deg, Match, Fit Fit Fit, Match, Store, Save Parameters, Reset, Match, Save (Top button))
- gtSetupIndexing (say no to 'use existing params for phase01' -> untick use input file)
- gtSetupForwardSimulation (use all the default choices)
- gtSetupReconstruction (do not reconstruct abs volume), launch reconstruction, then rerun to launch grain segmentation
- GtGrainsManager -> assemble phase01
- GtGrainsAnalyser -> idem to see with the IPF coulouring

sample Ts3

install sample Ts3 (0.83% strain) in the beam, DIC face upstream, notch up. found Pt mark. Will do DCT scans 600 microns beam height with 100 microns

overlap in total and 50 microns off center to match DIC region.

```
fasttomo360 ts3_dct_top_; mvr samtz 0.5; fasttomo360 ts3_dct_bot_
```

translations were samtx=0.6 samty=0.025 samtz=1.996 (top) samtz=2.496 (bot) tilt values were samrx=-1.2 samry=0.7

Check evolution of contrast for grain11 as a function of distance nfdtx

```
nfdtx 102.2 grain11_205_1_ nfdtx 104.2 grain11_205_2_ nfdtx 106.2 grain11_205_3_ ... nfdtx 136.2 grain11_205_18_
```

..then try reproducibility nfdtx 136.2 grain_205_18 -> 23 We like the trade-off between band-contrast and distortion for the distance corresponding scan grain11 205 6:

nfdtx 95.77 corresponds to 0 distance for the topotomo detector.

- \sim 19h: check rocking curve with lrock1 0.01; optimize focusing (mvr tfz 0.04) (NB: we translated the topotomo detector into the direct beam (umv d3tz 0) for this alignment)
- -> Topotomoscan grain11 tt1

We

Align grain #64 samrx = 15.866 samry = 7.879 samtx = 0.475 samty = -0.275 samtz = 2.035

-> Topotomoscan grain64 tt1

Align grain #31 samrx = -6.3068 samry = -11.071 samtx = 0.665 samty = -0.2555 samtz = 1.912

-> Topotomoscan grain31 tt1

Align grain #5 samrx = 11.341 samry = -4.449 samtx = 0.685 samty = -0.09 samtz = 1.860

-> Topotomoscan series a tdifferent distances:

```
* grain5_tt0_ nftdx=107.2
* grain5_tt1_ nftdx=112.2 -> our regular distance
* grain5_tt2_ nftdx=117.2
* grain5_tt3_ nftdx=122.2
```

Align 1st reflection for grain #54 samtx = 0.665 samty = -0.209 samtz = 2.187 samrx = -18.67 samry = 14.167 diffry = -7.553

-> Toposcans at nfdtx 107.2 & 122.2 f grain54_r1_tt0_ grain54_r1_tt1_

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Align 2nd reflection samtx = 0.677 samty = -0.16 samtz = 2.057 samrx = 9.112 samry = -2.261 diffry = -6.4

-> Toposcans grain54_r2_tt0_ grain54_r2_tt1_

Align grain 39 samtx = 0.65 samty = -0.248 samtz = 1.805 samrx = 4.379 samry = -4.268 diffry = -6.4

sample Ts2

install sample Ts2 (0.67% strain) in the beam, DIC face upstream, notch up. Centered with respect to both heads. 2 DCT scans 600 microns beam height.

```
fasttomo360 ts2_dct_top_; mvr samtz 0.5; fasttomo360 ts2_dct_bot_
```

translations were samtx=0.252 samty=0.135 samtz=1.907 (top) samtz=2.407 (bot) tilt values were samrx=-1.2 samry=2.7

Topotomo setup

back to sample T1, sample moved during positionning (was not glued). WL found the rotation offsets so we can still use the DCT data to do TT alignment. tilt values were samrx=0.6 samry=0.8 diffrz=83 (here we find the same diffraction spots than before).

installing topotomo camera (cMOS PcoEdge 6.5 microns pixels) tilted by 13.5 degrees.

put sample ts3 back in to record a scan to calibrate detector distance and tilts. d3tx = 100.2 d3tz=0, d3ty=14.0

in situ setup

Nanox machine

sample T5

The goal is to locate a region of interest and map it with topotomo at 0.2%, 0.4%, 0.6%, 0.8% and 1.0% strain.

platinum marks located at pixels 461 and 1525 (about 1.5 mm away). zone of interest located in the EBSD region, about 250 microns below the middle section. DCT camera set at 7 mm to reduce overlap.

3 DCT scans in the initial state:

```
fasttomo360 t5_dct_bot_
fasttomo360 t5_dct_cen_
fasttomo360 t5_dct_top_
```

DCT cen is the zone of interest, DCT bot is 0.5 mm below, DCT top is 0.4 mm above (reached limit on samtz). A list of 10 grain was selected for TT inspection:

```
52 16 13 7 73 24 36 62 2 3 4
```

first round of tt scans were done.

```
t5_dct_cen_grain0052_tt_1
t5_dct_cen_grain0016_tt_1
t5_dct_cen_grain0013_tt_1
...
```

All tt scan stored in folder

```
t5 tt 1
```

measured section from PCT: 0.388 mm x mm

loading with piezo actuator until 80 N (stress motor value is 75). Load values have been reported to file loadramp_1_.txt (also saved by spec). approx 0.2% strain (Young's modulus is 104 GPa). sample moved, motion was corrected by 151x1.4 microns along samtz and 50x1.4 microns along samtx. new samtx, samty positions have been reset to zero. Now samtz = -4.718

Corrected rotation by -0.4 deg on samrx and -0.3 deg on samry. new values are: samrx=-0.4, samry=1.5.

```
fasttomo360 t5_dct_cen_1_
```

re-indexed new DCT acquisition, found new grain ids with function gt

```
[match, dev, dc, drot, conflicts, tol] = gtINDEXMatchGrains(grain1.grain, grain2.grain, 1, dc, drot, 52 16 13 7 73 24 36 62 2 3 4 85 17 12 9 79 24 32 66 1 16 4
```

Modification of scripts

loading of the specimen

```
Load_1 : voltage: 75V; Force: 80.8N; image 112
Load_2 : voltage: 120V; Force: 160.9N; image 118
```

No evidence of slip in load_1. Evidence of slip in load_2 in most of the TT grains.

Maximum range of piedzo. Unloading to 30N and manual mechanical loading in the chamber up to 150N Alignement of specimen

```
Load_3 : voltage: 77V; Force: 214.4N; image 49
```

Macroscopic strain between 0.5% to 0.8%: no macroscopic yielding. grains are cropped on TT.

```
Load_4 : voltage: 86V; Force: 228.1N; image 08
```

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