Oxford EBSD operation manual for Quanta 200 FE-ESEM

Sample set-up

1. Use 45 pre-tilt holder to mount your sample. The optimum tilt angle of the sample surface is 70.
2. Find the area of interest and focus the image following routine SEM operation.
3. For EBSD, the optimum working distance is 13mm.
4. Typical imaging voltage for EBSD is 20 KeV, probe current is 1-5 nA. Try to use a small aperture.
5. Insert the EBSD detector. The detector should stop at 155 mm. Then you need to press the in button to move the detector in 0.1 mm a time until it's close to the sample. (typically 160-165mm, but depends on the sample configuration)

EBSD set-up

1. Open Flamenco and load the standard profile to proceed.
2. Press auto to continuously read the parameters of the SEM microscope (e.g. beam voltage, beam current, magnification etc.)
3. Under imaging mode, choose SE as electron source and acquire an SE image.
4. Make sure "Tilt correction" and "dynamic focus" boxes are checked in SEM column.
5. Under setup mode and "frozen SEM image" tab, choose a spot on the image and switch to "live EBSP" tab, the kikuchi pattern of the chosen spot position is shown lively here.
6. adjust parameters (e.g. spot size, camera gain, time per frame, binning etc., note that everytime you change binning, you should redo the background correction as shown in section c.) in camera controls to get a pattern with appropriate brightness and contrast.
7. Stop spot mode.

EBSD background correction

1. If your sample is polycrystalline, set the raster scanning speed to be really quick (e.g. 50 ns), the live EBSP should have no kikuchi lines. Press the

"acquire new background" button, and the background will be automatically saved.

2. If your sample is single crystalline, then you need to move a reference area (e.g. stub surface) to your view, then set the raster scanning speed to be really quick (e.g. 50 ns), the live EBSP should have no kikuchi lines. Press the "acquire new background" button, and the background will be automatically saved. Move back to your sample.
3. Now under spot mode, choose a spot on the image and switch to "live EBSP" tab, the Kikuchi pattern of the chosen spot position should be clean without the influence of the background.

EBSD pattern acquisition and indexing

1. Under setup mode and "EBSP Geometry" tab, press "adjust field of view" and "Maximum AOI with fixed field of view" buttons. You can also exam and adjust the field of view (rectangle) and AOI (circle) by pressing these two buttons.
2. Load the calibration file (e.g. WD 13mm 165mm) for your sample. Note that the calibration file is dependent on the physical configuration of the WD and the detector position.
3. Press "match units" button to choose the standard elements and composition for your sample, you can also add the database by loading the database in settings.
4. Choose the position under spot mode, then snap EBSP and detect bands.
5. Choose the maximum number of bands for detection (6 to 9 bands, the less symmetric your sample is, the higher the number), and then index the bands.
6. Press "active refine box", refine your index, check "V/H ratio" and then refine again.

Orientation map and phase map

1. Under imaging mode, choose electron source as BSE.
2. Use backscatter control to choose the detectors and adjust brightness and contrast. Detectors 2 and 3 are for orientation contrast imaging, and detectors 1 and 4 are for phase contrast imaging.
3. Under automatic mode, create a new job by choosing new mapping for beam

jobs. Adjust the points of the map and add this job to the job list. The Hough space resolution(typically 60-90) and number of reflectors (40-50, the smaller the faster) etc. can be adjusted to modify the mapping time. Then click run.

4. Wait until the map scan is finished. Enjoy the Coffee time.

Post-processing of the orientation map

1. Open manager data in the Channel 5 folder.
2. Open the obtained map project, drag it to "tango" to process the map, "manbo" to process pole figure, and "salsa" for orientation distribution function.
3. For map processing, BC means Band contrast map, right click it to add a new map, and set up the map properties of the new map. Drag the desired features (e.g. grain boundaries, phase interface, etc.) in the right column to the map in the left column.
4. You can reduce noise of the map by extrapolating zero solution and wild spikes.
5. Open "record browser" in project manager to see your raw data.
6. Open "view" and then "virtual chamber" to see and modify the geometry of your sample.
7. In Channel 5 folder, online help may help explain questions.

Emergency Information:

Medical Emergencies: Contact 911 and Public Safety (609) 258-1000
Room / facility emergencies: Contact Public Safety (609) 258-1000
Issues related to the instrument:

1. Contact IAC Staff.
2. Leave system as is, Do Not disable vacuum system.
3. Try to shut off the High Tension.

Audible/Siren Emergency Alerts:
Follow previous steps 2 & 3 and leave the building.

Emergency Contact Information:

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