

EBSD

User Guide

EBSD Instructions

Please note that the EBSD detector is very sensitive and will be very close to your sample. You must be aware of your sample and detector geometry at all times. For this reason please follow the rules:

1. Be aware of the sample and detector geometry at all times.
2. Do not make large X, Y or Z movements of the stage while the detector is close to your sample.
 - a. To make large changes move the detector away, change stage and focus and then move the detector back in only as far as it will not hit your sample.
3. Do not rotate the sample!
4. Make sure that the detector is fully retracted before you unload the shuttle.
5. Make sure that the detector is fully retracted before you load the shuttle.
6. As always, make sure the sample fits under the height gauge.

Specimen Geometry Restrictions

Due to the possibility of damage to the detector, your sample should not be larger than a standard 12.5mm pin mount holder (Figure 1). If the sample or area of interest is small it should be placed at the bottom of the specimen holder. Only flat specimen holders (and TKD holders) are allowed. Thin samples are preferred. Maximum sample thickness is 5mm.



FIGURE 1 YOUR SAMPLE CAN BE NO LARGER THAN THIS.

The specimen should be placed in the pre-tilted end of the shuttle holder, as shown in Figure 2.



FIGURE 2. USE THE 45 DEGREE PRETILTED END OF THE SHUTTLE HOLDER

Do Not:

1. Rotate the stage!
 - a. Stage rotation should stay at -58.2
2. Move stage while EBSD detector is inserted!
3. Tilt sample while detector is inserted!
 - a. Stage tilt should always be 25 degrees when the detector is inserted or being inserted!
4. Switch to mode 2!

Sample Set Up

Insert sample in Verios with area of interest located on the bottom of the stub if the sample is smaller than the stub.

1. Area of interest should always be at the bottom (closest to the detector) to avoid hitting the detector.
2. Do not use a pre-tilted sample stub; ALWAYS use the pre-tilted location on the shuttle.
3. Ensure your specimen is not too tall using the standard Verios height gauge.



Instrument Set Up

Start-up and align the SEM Column as usual (the EBSD user should be familiar with the operation of the SEM prior to using the EBSD detector).

1. Select Voltage and make sure that you have chosen sufficient current.
 - a. Higher voltage usually results in better signal and sharper band resolution, lower voltage will result in a smaller interaction volume
 - b. 10-20kV is a good starting point.
 - c. Recommended currents are in the 1.6 – 26 nA range.
2. Focus on sample and then link the stage.
3. EBSD must be done in field free mode! (mode1) Do not switch to mode 2!
4. **Tilt the stage to 25 degrees of tilt – this is critical!**
5. Set sample height (Z). Shorter working distances will produce a finer beam. Working distances in the 8 mm to 12 mm are recommended.
 - a. Move in incremental steps of no more than 2 mm per step.
 - b. Focus and Link the stage at each step
6. Find and focus at area of interest. You will no longer be moving the stage. Ensure this is the correct position.
 - a. Check that it is the correct position again!
7. Go to the Navigation tab and select the Tilt correction sub-tab.
 - a. Set the Dynamic Focus to ON.
 - 1) Confirm that the total tilt angle is 70 degrees
 - 2) It is possible to either use a manual tilt of 70 or
 - 3) It is possible to use the automatic stage tilt (25) plus a pre-tilt of 45
 - b. DO NOT engage tilt correction. It is OK to use Tilt correction while finding the region of interest. Do not use tilt correction from the Verios while collecting EBSD data. The tilt correction will be done within the AZtec EBSD software.

Software Set Up

1. Run Tidyup (if desired)
2. Start Aztec
 - a. Load an EBSD profile – if you do not know which one, choose the EBSD Standard profile
 - b. The EBSD profile should have tilt correction already on and set to 70 degrees – it is smart to confirm this
 - c. **DO NOT CHANGE THE EBSD STANDARD PROFILE!**
3. Ensure your project is being saved to the SSD (not the data server).
 - a. This should be the D drive on the EDS/EBSD computer
 - b. You will move your data to the data server after you finish collecting data.
4. Make sure you choose EBSD (not EDS)
 - a. Most users will want to use the Map function.

Insert EBSD Detector

Take great care when inserting the detector. If the detector touches anything, it will be ruined.

1. Preparing to insert the detector
 - a. Open the EBSD Detector Control Menu in the lower right corner of the Aztec software.
 - b. Under “Proximity,” verify that the proximity sensors are enabled.



- c. Under “Elevation,” verify that the elevation is **2.0 mm** (max when fully retracted). If not, press the “Up” button to move the detector to the maximum elevation. Note that the elevation will change as the detector is inserted with 2.0 mm as the maximum when the detector is fully retracted.
2. Go through the following steps to insert the detector
 - a. **DO NOT INSERT THE DETECTOR IF YOU HAVE NOT TILTED THE STAGE!**
 - b. Ensure that the CCD camera is live.
 - c. Under the “Insertion” menu, press the “In” button ONCE. The detector will move quickly to the soft stop at 70 mm.
!DO NOT CHANGE THE SOFT STOP!
 - d. Ensure the CCD chamber cam is live!
 - e. Press “In” a second time to slowly move the detector inward. Keep the cursor over the stop button so that the detector can be stopped quickly if needed.
 - f. The detector will first become visible at approximately 142 mm of insertion with the detector obviously visible at 146 mm.

The SEM image should become noisy as the detector is inserted, especially after 140 mm. This is because of the proximity detectors.

- g. Unless stopped by the operator, the detector will move until it is fully inserted at 176.5 mm or the proximity sensor is tripped. The detector will be very close to the stage and sample!
- h. A good starting point is 174mm.



DANGER! DO NOT use the “Move To” function when inserting or retracting the detector. The detector will move very quickly to the position that is selected, which risks damaging the detector. The minimum cost to repair a damaged detector is \$20k!

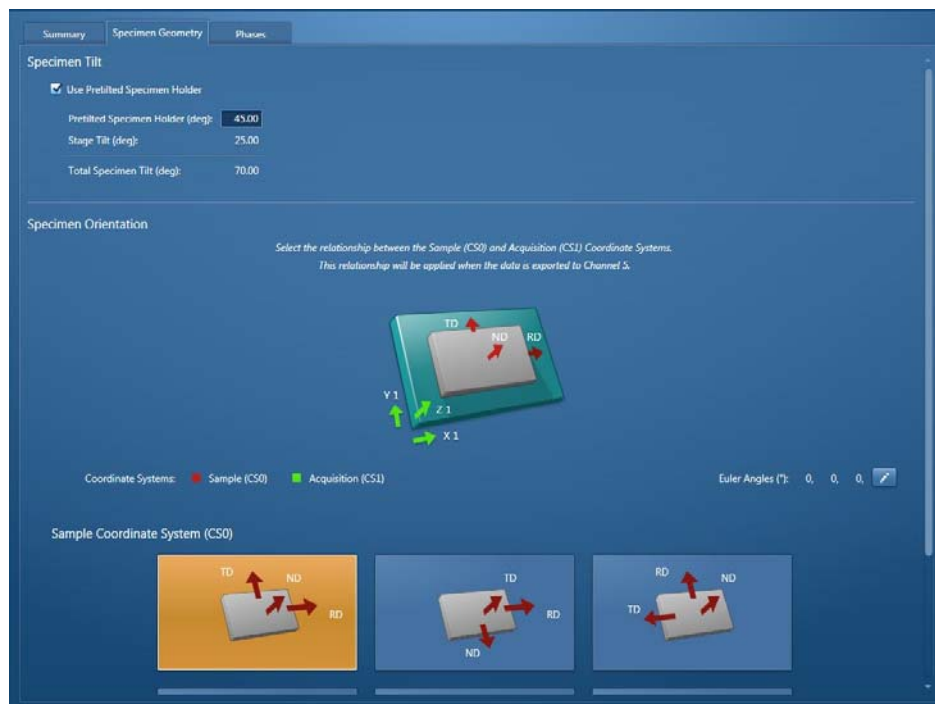
Describe Specimen

1. Summary

- a. Enter any pertinent information that you wish about your sample

2. Specimen Geometry

- a. Choose pre-tilted sample holder
 - i. Pre-tilt = 45 degrees
 - ii. Stage tilt will be 25 degrees
 - iii. Total tilt = 70 degrees
- b. Select the orientation geometry that is correct for your sample.
 - i. If you are not sure, pick the first one for the analysis
- c. Confirm the specimen tilt is correct.



3. Phases

- a. Select the phases present in the sample.
 - i. Select all possible phases
 - ii. The EBSD user will need to know what phases are to be identified prior to data collection
 - iii. If you don't know or the phase you need is not present, you will need to determine the structure via XRD
- b. Phases must be present otherwise the software will not be able to determine orientation.

The screenshot shows a software interface with four main panels:

- Phases in Databases:** A list of materials with their crystal structures. The 'Iron fcc' phase is selected.
- 3D Phase View:** A 3D visualization of the selected phase, showing a sphere with red and blue facets and axes.
- Phases for Acquisition:** A table showing the selected phases and their properties.
- Phase Details:** A panel showing the specific details for the selected phase.

Name	Structure
Alumina (alpha)	Trigonal, SG=167
Aluminium	Cubic, SG=225
Andalusite	Orthorhombic, Laue=3
B/SrCaCuO	Orthorhombic, Laue=3
Bronzite	Orthorhombic, Laue=3
Calcite	Trigonal, Laue=7
Cd	Hexagonal, Laue=9
Chromite	Cubic, Laue=11
Coesite	Monoclinic, SG=14
Copper	Cubic, SG=225
Corundum	Trigonal, Laue=7
Diamond	Cubic, Laue=11
Diopside CaMgSi2O6	Monoclinic, Laue=2
Dolomite	Trigonal, Laue=6
Enstatite Opx AV77	Orthorhombic, Laue=3

Include	Phase	Reflectors	Color
<input checked="" type="checkbox"/>	Iron fcc	44 reflectors	0
<input checked="" type="checkbox"/>	Iron bcc (old)	45 reflectors	240

Phase Details

Description

Name: Iron fcc
Reference: Z. Angew. Phys. [ZAPHA], vol. 23, pages 243-249

Structure

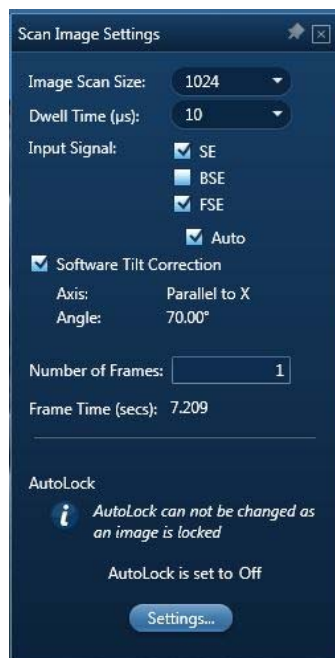
Crystal System: Cubic-High (m3m)
Laue Group: 11
Space Group: 225

Unit Cell

a [Å]: 3.6599
b [Å]: 3.6599
c [Å]: 3.6599
Alpha [°]: 90.0

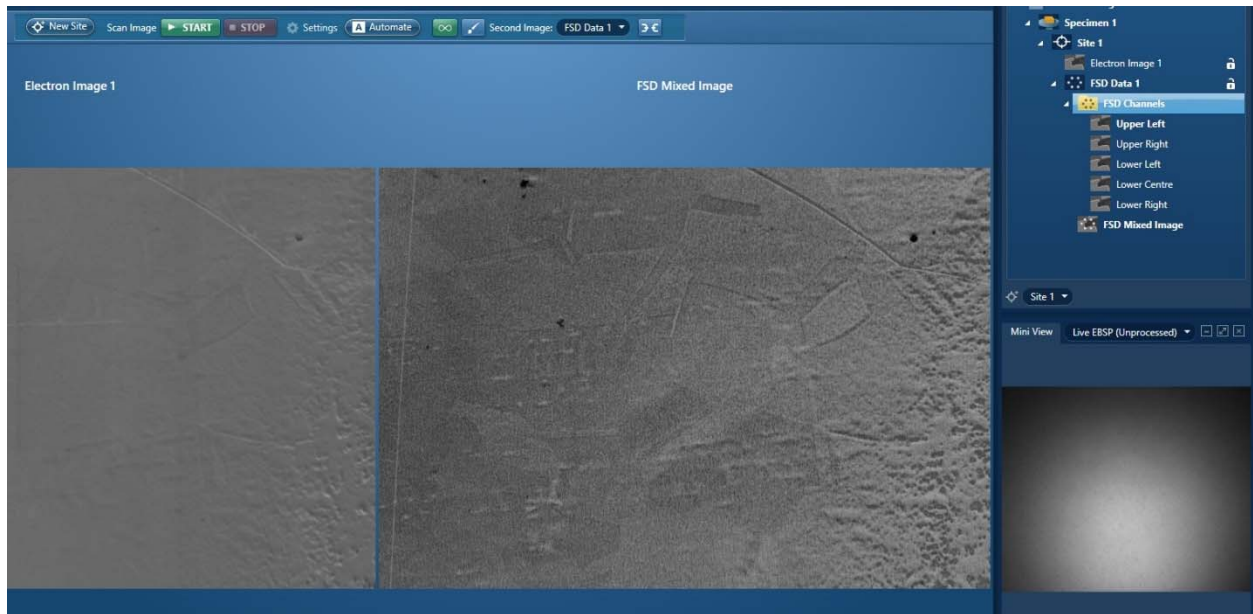
Scan Image

1. Turn off the proximity sensor in the EBSD Detector Control menu. After the proximity sensors are turned off, the bands of noise in the SEM image should go away.
2. Go to settings and make sure that you have at least 1024 and selected SE and FSE
 - a. SE is the image in the first quadrant of the Verios
 - b. FSE are the forward scattered electron detectors arrayed around the EBSD detector.
 - c. The Verios only has one output, so the BSE signal is the same as the SE signal.
3. In settings: Select software tilt correction to apply the tilt correction chosen earlier to the image and data.



4. Acquire the image data.
5. Choose the second image to be Electron Image1.
 - a. The mixed FSE or individual FSE images can be selected by clicking on the desired image in the data tree (or current site).
 - b. Often, one of the FSE images will give the best results

- c. The mixed FSE image may look fuzzy due to geometric effects
(individual FSE detectors are arrayed around the EBSD detector)



Signal Strength

If the Live Unprocessed EBSD image does not look like a bright sphere. You will need to Optimize first. Bands may be visible if a particular grain has been chosen.

Mini View Live EBSP (Unprocessed) [min] [max] [close]

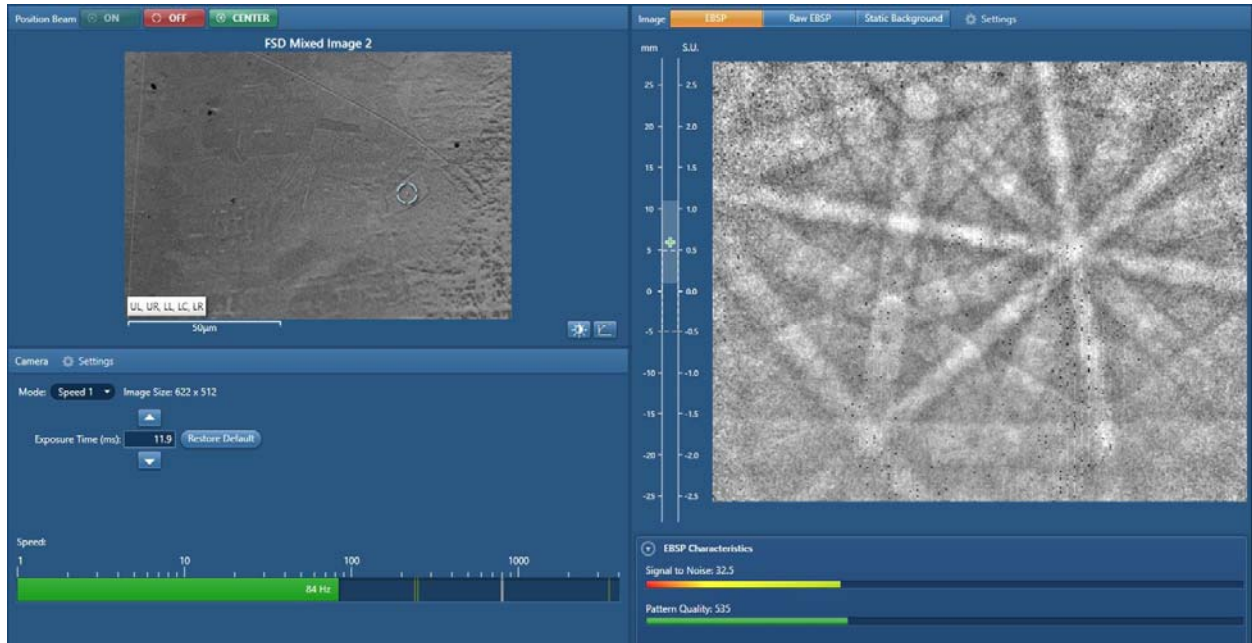


Signal To Noise: 4.0

Time per Frame 131.58 ms / 7.60 Hz

Optimize Pattern

Typical scans will use the “Speed 1” preset with auto-detected exposure. Well-annealed metals can use “Speed 2,” while poorly detecting materials may want to use “Resolution” or “Sensitivity”

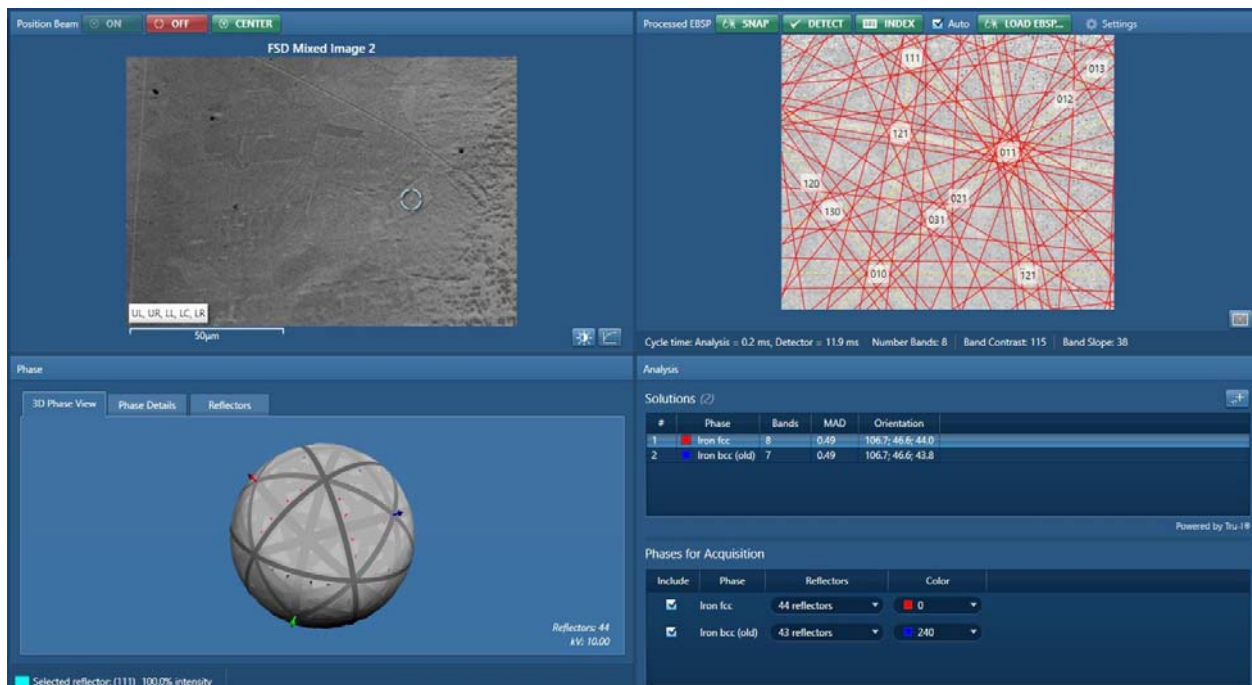


Optimize pattern showing a point selected in an FSE image and the corresponding EBSP

1. Press the Settings button in the pattern area and select Auto Background and then the Setup button. Collect static background at least 50 frames (400 is the default).
 - a. Wait a few seconds until finished
 - b. The background should be totally random without visible patterns.
 - c. If one chooses to change modes, the static background will need to be recollected.
2. Check EBSP by clicking around the image to confirm that EBS patterns are observed.
3. If the static background is dark or very dim, increase the exposure time. If the detector saturates, decrease the exposure time.

Optimize Solver

1. Turn on Position Beam and use the mouse to drag the point to several grains. If you are scanning a large area, ensure these grains are near the middle of the scan.
2. Make sure that the patterns can be solved as shown.
3. Go to settings
4. Indexing mode set to Refined Accuracy or Optimized TKD
 - a. It has been found that for most EBSD, Optimized TKD is the best indexing mode.
5. Number of bands should be 8.
 - a. This can be at lowest 6 and with a maximum of 12
6. Hough resolution between 60 and 120
7. Click in image and check the mean angular deviation (MAD) value. MAD should be less than 1. The lower the better.
 - a. If the MAD is above 1, adjust either the Hough resolution or the number of bands. The Hough resolution affects the MAD more than the number of band.
8. Click + in upper right of Solutions. This applies a refinement to lower MAD value.



Acquire Map Data

Now set-up the acquisition area and determine parameters.

Check the settings. The resolution can be adjusted as desired. More pixels = more time. If all information is desired, go to settings, select store all patterns. This generates a massive amount of data. You will need to remove this from the computer onto a hard drive immediately after your session is finished.

- 1) Press Start
- 2) Monitor the Progress in the Construct Maps Page
- 3) After doing a test run, set up area of interest and time desired.

Save Data

Report results

Site report (current site). Do site report for each site.

Move data from SSD to the data server.

Shutting Down EBSD

1. Make sure imaging is paused
2. Unpause the CCD camera
3. Re-enable the proximity detector
 - a. There is a delay between when the button is clicked and when it shows the proximity detector as being on
4. Check that the CCD camera is unpaused!
5. Retract the EBSD detector
 - a. Press the OUT button. The detector will quickly fully retract.

Wait until the detector is fully retracted before proceeding!

Ensure it is fully retracted and not at the soft stop!

6. Turn off dynamic focus (Verios)
7. Reload a standard EDS profile in AZtec

The following is to finalize shut down the Verios

8. Turn off the beam
9. Zero the stage tilt and scan rotation
10. Click on unload from the Verios navigation tab
11. After transfer, click release button
12. Remove your sample (one turn only!)
13. Reload the empty shuttle in the Verios
14. If you went over the time you reserved, please check out using Mendix