INCA EBSD help summery

1. Hardware setting

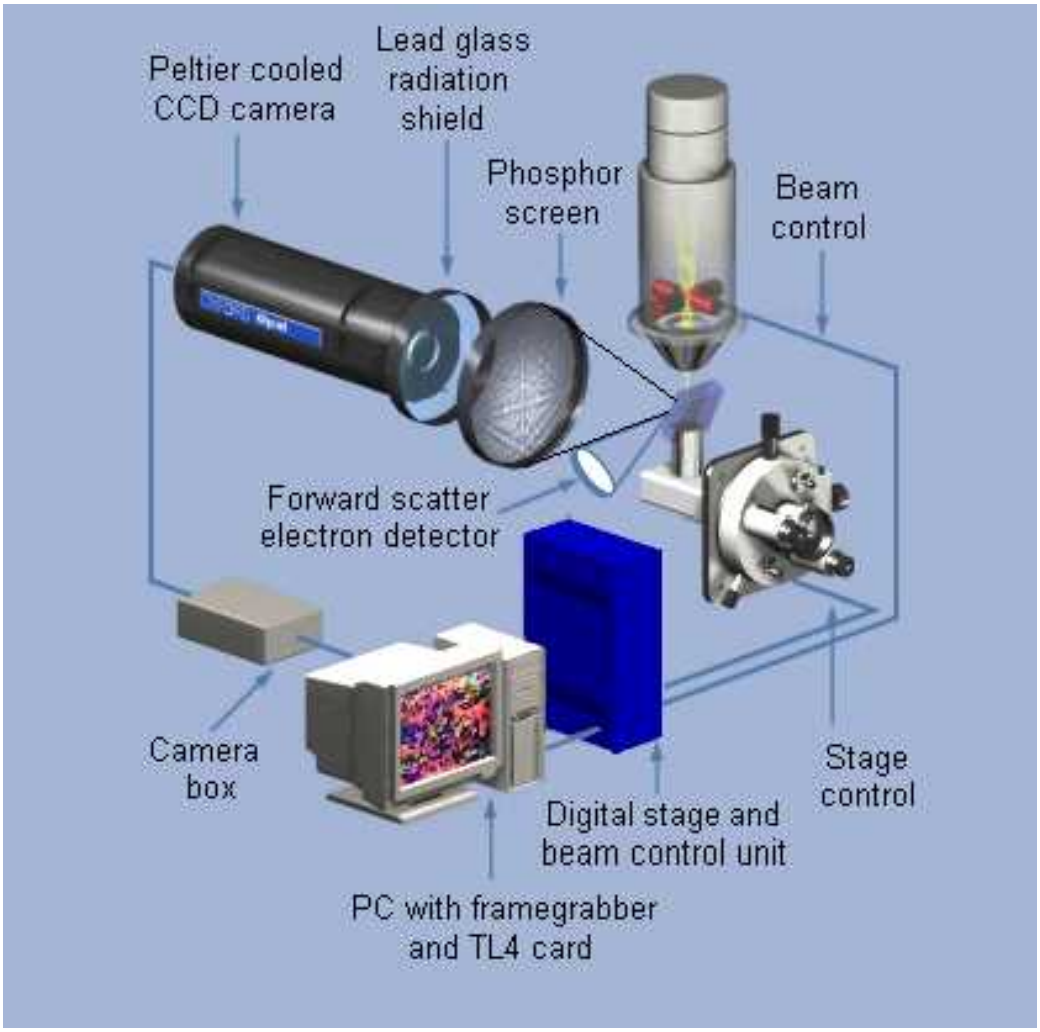
The EBSD hardware consists of a sensitive Peltier cooled CCD camera mounted on the SEM chamber, coupled with associated control and communication hardware, and a computer running the INCA crystal program. The camera views the phosphor screen, which collects the patterns.

The INCA Crystal software allows adjustment of the camera acquisition conditions such that electron backscatter diffraction patterns can be obtained for a wide range of microscope conditions, i.e., high to low beam current.

This is achieved by appropriately selecting from the software user interface:

- Adjustment of the camera gain or sensitivity.
- 'Binning' of pixels in the image (sometimes referred to as 'compression') which enhances camera sensitivity, but reduces pattern resolution.
- Selecting integration time which allows different intervals of on chip integration.
- The camera hardware can be inserted into the operating position which brings the phosphor screen into close proximity to the tilted sample. See ***Collection Geometry***. When not in use, the camera hardware can be retracted out of the way.

Schematic layout of components:



2. Pattern Calibration

INCACrystal must be calibrated with the germanium sample supplied. Germanium is used because it has a repeatable pattern and easy to recognize zone axes.

INCACrystal is calibrated at two working distances. This has the advantage of being able to use INCACrystal at any working distance.

There are two different methods of calibrating the system
1. Using two camera positions and calculating the point in both patterns which do not change.
2. Using a single pattern, and automatically search for pattern center.

1. Using the two camera position approach to calibrate the system

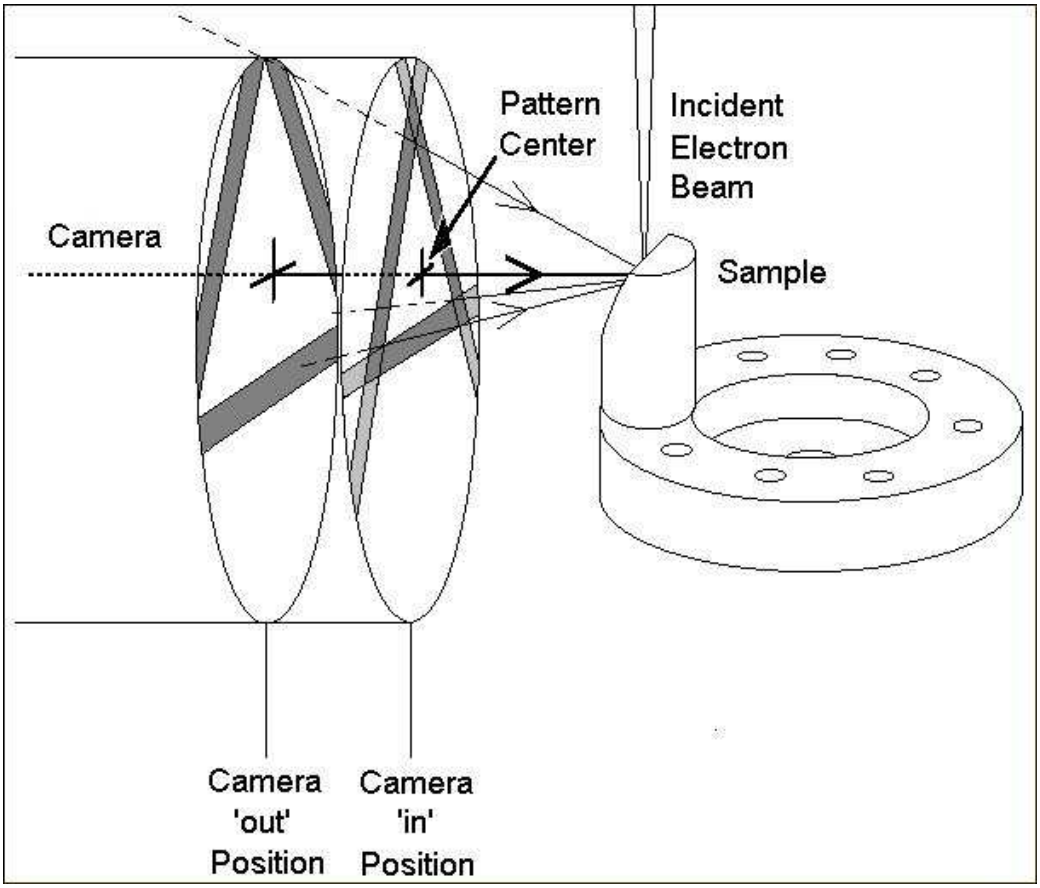
Pattern calibration has two stages:

- Finding the Pattern Center at the two working distances.
- Determining the camera to sample distance.

Pattern Center

A pattern center is found at two working distances. These two working distances should be separated by at least 5 - 10 mm, e.g. 15 mm and 25 mm. These are called pattern center (A) and pattern center (B). Finding these two pattern centers allows Crystal be operated at any working distance. However the practical working distances depend on sample size and position of the sample relative to the phosphor screen.

The Pattern Center is found by using two patterns acquired at different camera positions.



Finding the pattern center is broken down into four steps

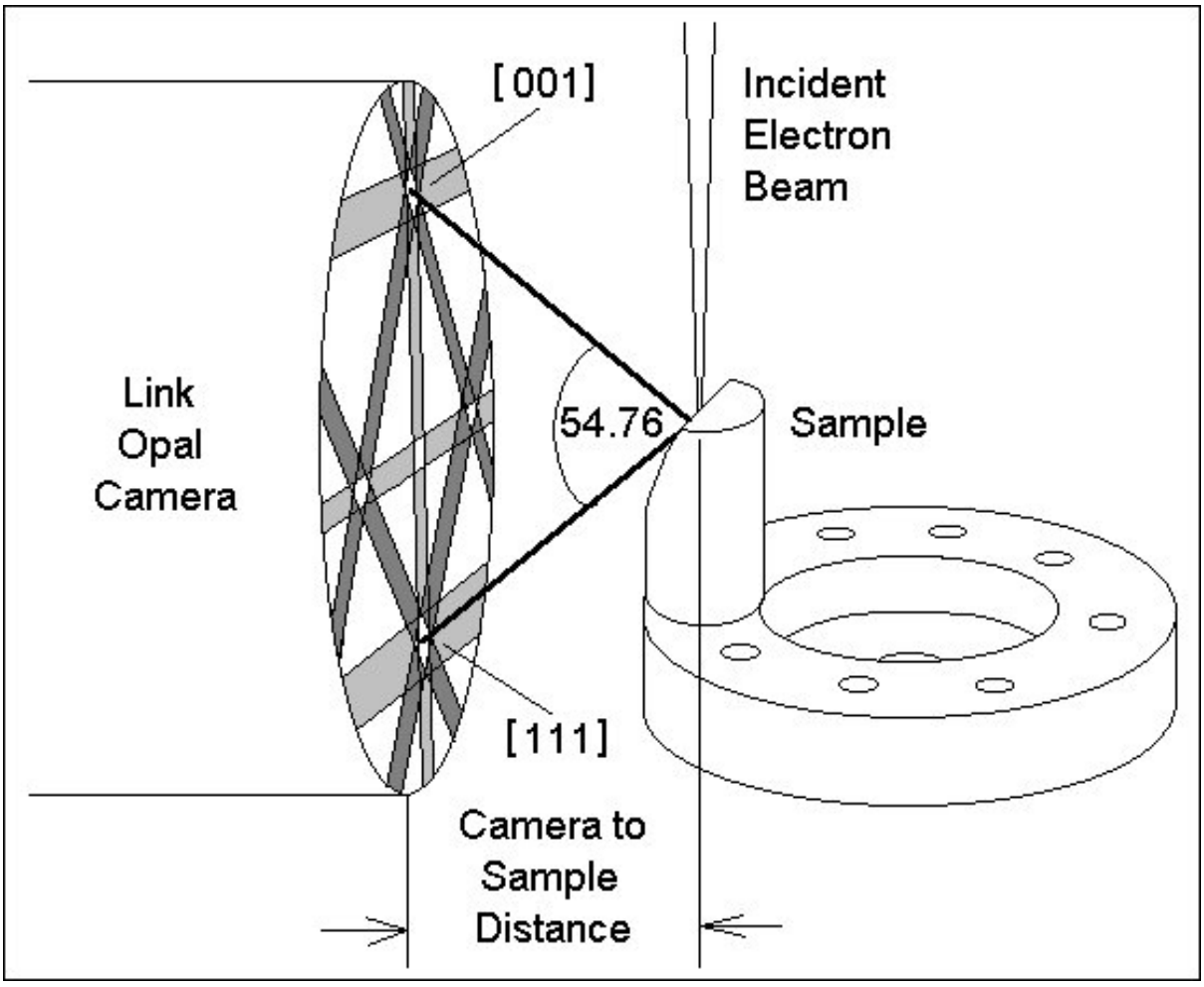
- **Pattern Calibration** - This step selects the calibration parameters, i.e. working distance and which pattern center you are finding, Pattern Center (A) or Pattern Center (B).
- **Collect out pattern** - This step adjusts the camera settings and collects the wound out pattern for the selected working distance.
- **Collect in pattern** - This step adjusts the camera settings and collects the wound in pattern for the selected working distance.
- **Determine the pattern center** - This step uses the stored wound in and wound out pattern to calculate the pattern center for this particular working distance.

These steps are then reiterated to find the pattern center for the second working distance.

Note: Ensure that the appropriate radio button is selected for either Pattern (A) or Pattern (B) when calibrating at their respective working distances. After collecting Pattern (A), the second radio button must be selected. Failure to select the second button will result in the Calibration for Pattern (A) being overwritten by the calibration for Pattern (B).

Camera Distance

The camera distance is calculated by identifying two zone axes within the 'wound in' pattern.



Determining the camera distance comprises of just one step.

- **Determine Camera Distance** - During this step the pattern from the wound in position is displayed. The two known zones are identified by marking them with a line connecting their centers and entering the angle between them.

2. Using a single pattern

As this method is an automatic method of calculating the pattern center information the best possible pattern is required, and the process is again repeated at two different working distances, the sample and camera geometry should be set before attempting to calibrate the pattern center.

Ensure the camera is moved to the standard acquisition position and the microscope is focused with a high magnification. If possible set the microscope to 30KeV to enhance the pattern quality.

Now acquire a number of good quality patterns to perform the auto calibration on. The incorrectly identified bands can be removed or modified, and bands which were not found can be added.

After pressing the calibration button each pattern can be used to calculate the pattern center. The detected bands of each pattern are used to find a solution with a low residual error, searching for the pattern center which gives the best solution.
The pattern center information is averaged and stored by default, but the user can double click on a pattern center in the Confirm calibration list, to store an alternative pattern center. At this point please ensure that the Solution overlay matches the pattern. Now repeat the process at another working distance.

3. EBSD mapping

The Acquire map step, allows the collection of ***TopMap*** data from the currently selected Site of Interest, which may have been acquired in the Analyze or Mapping navigator. Any number of TopMaps may be selected from the Site of Interest.

Region of interest

Acquire Map allows the collection of TopMap data. Following selection of the site of interest in the previous step, ensure that the electron image is displayed by clicking on button  . Then select the regions of interest using following buttons:

 Whole Site of Interest

 Rectangular region

 Line

 Freehand drawn region

The freehand tool is particularly useful for collecting TopMap data from irregular shaped regions of interest such as microstructural grains or contacts on a silicon chip. Any combination or number of rectangles, lines and freehand regions may be selected within the Site of Interest.

Mapping Resolution

To select the resolution of the map, select the number of pixels per image width, from the drop down menu described as Crystal map size.

For example, selecting 512:-

Selecting 512 from the menu for a whole Site of Interest map, will result in a map 512 pixels wide. If a rectangle, line or freehand region is selected at 512, then the pixel size selected is the same as that for a full 512 wide area.

The mapping status box will display the total number of pixels to be collected and the estimated time to completion.

Start Acquisition

Press start to commence data acquisition. Mapping status will show will update to show the number of pixels remaining and the estimated time to completion.

Maximizing pattern acquisition speed

The camera should have previously been optimized to ensure good pattern solves, within a minimum integration time, during the Sample Camera Setup stage. This will ensure an optimum pattern collection rate. To further maximize acquisition speed, the electron image should be displayed and the display pattern option switched off.

4. Sample Preparation for EBSD

Because diffracted electrons only escape from the uppermost layer of the specimen surface (of the order of a few 10's of nanometers deep), specimen preparation for EBSD is consequently critical to achieve good results. If the specimen surface retains surface damage on a crystallographic scale, or has any surface contaminant/oxide/reaction product layers present, then EBSD pattern formation may be suppressed.

Standard preparation methods may be employed successfully with care and perhaps in conjunction with extra steps to achieve an adequate surface finish. As a general rule standard preparation methods can be progressed to the final polishing stage without any deviation from the normal route employed. Thereafter, an additional polishing stage using colloidal silica is all that is required to achieve a finish suitable for EBSD.

However, different materials respond differently to common preparation methods. Therefore the material under investigation should be considered on an individual basis and prepared appropriately. The manufacturers of preparation equipment should be consulted for the applicability of a given approach for a given material.

The following steps are critical for good specimen preparation:

Cutting

Mounting Samples

Grinding

Polishing

Etching

Electrolytic Polishing/etching

Alternative Preparation Methods Includes information on: Plasma Etching, Ion Milling, Attack Etching

yingday@ntnu.no
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