Introduction to the TESCAN MIRA3 LMH Schottky FE-SEM

- Global supplier of scientific instruments
- Designs and manufactures scanning electron microscopes in Brno, Czech Republic
- About 2000 instruments in more than 77 countries
- TESCAN USA is based in Warrendale, PA
Instrumental Capabilities

- **Secondary electron imaging** (Topography contrast)
- **Backscatter electron imaging** (Phase contrast)
- **Energy-dispersive X-ray spectroscopy** (Compositional analysis)

Topography Contrast

- SE are ejected from the k-shell of the specimen atoms by inelastic scattering interactions with beam electrons.
- SE originate within a few nanometers from the sample surface.
- Brightness of the SE signal depends on the number of secondary electrons reaching the detector, providing a topography contrast.
- Resolution is about 1-2 nm at normal operating conditions.
Phase Contrast

- BSE are back-scattered out of the specimen interaction volume by elastic scattering interactions with specimen atoms.
- BSE are used to detect contrast between areas with different chemical compositions since elements with high atomic numbers backscatter electrons more strongly than elements with low atomic numbers and, therefore, appear brighter in the image.

Compositional Analysis

- Analytical technique used for the elemental analysis or chemical characterization of a sample.
- Interaction of electrons with sample produces characteristic X-rays (elements emit X-rays that have a characteristic energy).
- You can do spot analyses to calculate mineral formula; map element abundances; measure element abundances along traverses etc.
Electron gun and column
EDS detector
Sample chamber
Electronics
Control unit
Dry scroll vacuum pump
Uninterruptible power supply
FE-SEM computer
EDS computer
Chamber View
BSE detector
Pole piece
Thin section
Automated stage
EDS detector
Sample chamber
Electronics
Control unit
Dry scroll vacuum pump
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EDS computer
Chamber View
BSE detector
Pole piece
Thin section
Automated stage
Getting Ready

- Samples need to have a conductive surface
- Unless you work with metals, you need to coat your samples with carbon (or gold)
- Prepare your samples prior to scheduled FE-SEM time
- Do not touch the surfaces of your coated samples with your bare hands
- If available, store coated samples in a desiccator or desiccator cabinet

Booking Instrument Time

- Book instrument time (at the moment use calendar in Katharina Pfaff's lab)
- Book a minimum of one hour instrument time ($35/hr for internal users plus 8.5% overhead)
- Leave 30 min time between end of previous session and start of your session
- Come ca. 10-15 min before start of your session
- Reserved time will be forfeited if you do not begin within 10 min of the scheduled start time, but you will still be charged a minimum of one hour instrument time
Laboratory Rules – All Users

- Do not put objects on the instrument as the electron optics is sensitive to vibration
- Do not modify the set-up of the instrument in any form (the FE-SEM operates at high voltage)

Start of Session

- Sign in log book and provide ISSV number
- Sample exchange will be performed by John DeDecker, Katharina Pfaff (or Thomas Monecke)
- We will bring the sample in focus for you at a working distance of 10 mm (SE, BSE and EDS are optimized for this WD)
- We will set the operating voltage to 20 kV for you
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<th>Name</th>
<th>User ID</th>
<th>Holder Type</th>
<th>Sample Name</th>
<th>SE</th>
<th>BSE</th>
<th>EDS</th>
<th>Beam on Time</th>
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<th>Total Beam Time</th>
<th>Beam Intensity</th>
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**Laboratory Rules – Easy Mode Training Level**

- You are not allowed to vent the chamber and change samples
- You are not allowed (and do not need) to change z-axis due to the risk of hitting one of the detectors with the sample or the stage
- You are not allowed to change beam voltage of the FE-SEM
- Use chamber view when you are uncertain where you are on the sample and during significant x- and y-translations
- You **must** switch off the chamber view if you use the EDS system as prolonged use will damage the detector

Graphical User Interface – Easy Mode Training Level

- Magnification
- WD (Focus)
- Brightness/Contrast
- Beam Intensity
- Scan Speed
- x- and y-control of stage (not commonly used because it easier to use control panel)
Graphical User Interface – Easy Mode Training Level

- Change SE/BSE
- z-axis control (do not use)

- Imaging mode
- Scan speed
- Magnification
- Focus
- Astigmatism
- Brightness/contrast
- Auto-brightness/contrast
- Beam Intensity
- Wobbling
- Picture taking
Brightness and Contrast

Contrast

Auto

Brightness

Focusing of the Image

Out of focus (SE image)

In focus (SE image)
Choose small object and increase magnification (yellow box)

Adjust focus (WD) until image is in focus

Magnification

Focus (WD)
Wobbling

- Centering of the aperture is conducted by wobbling (i.e., the FE-SEM automatically swings the focus back and forth)
- If the aperture is centered, an object should not move in x and y during wobbling, but only go in and out of focus
Find a small, bright object, increase magnification so that object fills about 1/3-1/2 of the yellow box

Adjust x- and y-position of the aperture until object does not move

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Remove Astigmatism

- Astigmatism is caused by non-circularity in the electron beam, which causes stretching of the image in x- or y-direction
- Adjust the x- and y-stigmators, which control the current in the scanning coils of the FE-SEM

- Very important for imaging at high resolution
- Astigmatism needs to be corrected frequently when working at high magnification as contamination in the column causes astigmatism
- If working at low magnification, astigmatism needs to be corrected every time a change in the setting occurs (i.e., beam intensity in easy mode training level)
Remove Astigmatism

1. Increase magnification of image significantly
2. Focus until you cannot improve the focus anymore
3. Use x- and y-stigmators to remove stretching of the image
4. Adjust focus as you optimize the x- and y-stigmators

Switching Between SE and BSE

- Insert and retract BSE detector only when chamber view is switched on (no real need to do that if BSE detector is in position)
Improve BSE Images

- Initially use auto-brightness and auto-contrast
- Make sure you are in focus
- Find grain you want to study
- Decrease brightness and increase contrast at the same time in a way that minerals having lower average atomic number go black while those with higher atomic number go white
- Choose a slow scan speed
- Need high-quality thin sections and good carbon coat

Use of the EDS System

- Blue light means EDS is ready to be used (red light = stand-by mode)
- Chamber view must be switched off when using the EDS

Open Quantax Esprit software on EDS computer and login
Switch on EDS system
End of Session

- Ask John DeDecker, Katharina Pfaff (or Thomas Monecke) to vent the sample chamber and return your sample
- If you are the last user of the day, ensure that the instrument and the EDS system are turned into stand-by mode
- Ensure that you sign out in the log book
Laboratory Rules – Easy Mode Training Level

- Ask if you are uncertain what to do
- This is not a machine to play with – you need to think about every step before you do it and you have to understand the consequences of every action before you execute it
- The instrument is not easy to break in the Easy User Training Level (it is clearly your fault if you do)
- You are not allowed to give access to other users and you are not allowed to train other users

Have fun imaging!