

# Scanning electron microscope

## Instruction manual

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# 1. General remarks

When operating the scanning electron microscope (SEM) of Physics Teaching Labs regard the following precautions:

1. Read the instruction manual carefully and ask the teaching assistant or lab instructor in case of questions before switching on the instrument.
2. Never run the instrument without water cooling.
3. Do not mix the blue button Operate with the red switch OPERATION when following the instruction manual. The blue button Operate at the head of the electron column sets the vents of column and sample chamber to evacuate them. The red switch OPERATION switches on the high voltage power supply of cathode that generates the electron beam.
4. After switching-on of the instrument or flooding of sample chamber, do not switch on the OPERATION button before vacuum is established. Appropriate vacuum is indicated by the green light (V.L.) on the control panel. In addition, wait 1-2 minutes after the lamp lights up before pressing the OPERATION button. The cathode will burn if switched on while column is not under vacuum.
5. Avoid high emission currents. For this regard:
  - Check the control panel and take care that the settings given in Tab. 2-1 are chosen before you switch on the instrument.
  - Always set the emission control knob to the lowest value before:
    - (a) pressing OPERATION button,
    - (b) changing the high voltage range (wait also 10-20 sec before increase of emission in this case).
6. At the end of the day, remove samples and sample holder from sample chamber.
7. The following instructions are an extract of the full instruction manual of SEM. If you need further information please refer to the full instruction manual available in Physics Labs.



## 2. Switching-on

Procedure to switch on SEM is given in following steps.

1. Turn on water cooling. The flow meter should run smoothly, i.e.  $\approx 2$  rounds/sec.
2. Check whether sample chamber is closed.
3. Switch on main power supply (next to working bench of lab).
4. Switch on POWER of instrument.
5. Set the vent of column to **Shut** (yellow). Button should be kept pressed in this case and lights up when the vent is in the right position.
6. Press RP to switch on **Rotary Pump**.
7. Press DP to switch on **Diffusion Pump**. The diffusion pump has to run for at least 15 min before the column and sample chamber can be evacuated.

Area	Operating element	Position
Control panel (right side)	EMISSION Meter select switch HIGH VOLTAGE control DYNAMIC FOCUS control STIGMATOR X and Y ALIGNMENT X and Y SPOT SIZE control	straight to the left EMISSION on request 0° 5.0 on scale 12.00 o'clock position 12.00 o'clock position (pressed)
Control panel (left side)	MAGNIFICATION setting DUAL MAG: SCAN MODE select sw. IMAGE selector switch button CONTRAST control BRIGHTNESS control	100x or lower OFF RAPID SE 12.00 o'clock position 9.00 - 12.00 o'clock position

**Table 2-1:** Operational settings of SEM before switching-on

8. In the meanwhile, check the control panel and set all operating elements according to Table 2-1.
9. After 15 minutes, evacuate column and sample chamber by pressing **Operate** (blue) at the column head. Set the meter select switch on the control panel to VAC to check the vacuum. If needle is in the green area of the scale, the signal lamp V.L. shines green.
10. If vacuum does not appear please check the following:
  - Cooling water circulation o.k.?
  - Do the control lamps in the switches POWER, RP and DP shine?

- Control if O-ring at the top of column is dirty (dust, fibres). If needs be wipe with fibre-free Kleenex® tissue. The same is valid for the sealing at the sample chamber door. Do not wipe O-rings with solvents!
  - Check whether the thermo safety switch of the diffusion pump has reacted and the heating of the diffusion pump is switched off (pump is cold then).
11. 1-2 minutes after the green signal lamp (V.L.) shines up, the high voltage supply of cathode may be switched on. Set the meter select switch to EMIS to check the emission current and check whether the EMISSION control knob is turned to the left. Switch on the instrument by pressing the red button OPERATION. Proceed with optimizing of settings.

### 3. Switching-off

#### Stand-by mode (e.g. for change of samples)

1. Turn emission control knob to left stop.
2. Switch off Operation
3. Press Shut

#### Switching-off with longer interruption (end of day)

1. Turn EMISSION control knob to the left stop.
2. Switch off high voltage supply by pressing red switch OPERATION.
3. Press knob Shut (yellow) at column head.
4. Switch off diffusion pump (red switch DP).
5. Wait at least 20 min until diffusion pump has cooled down.
6. Switch off POWER of SEM.
7. Turn off cooling-water for diffusion pump.
8. Switch off main power supply (next to working bench of lab).

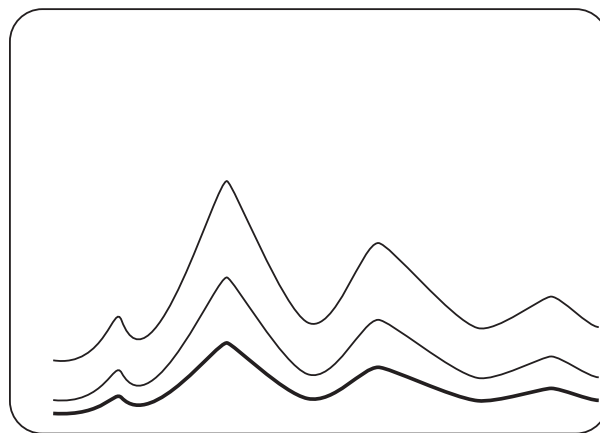




At the beginning, the emission of the cathode has to be set to operating point. The emission should not be changed after the first setting. Afterwards the micrograph is optimized by adjustment of focus, contrast and brightness.

### Optimal setting of the emission current (operating point)

1. Meter select switch on control panel should be set to EMIS.
2. Set MAGNIFICATION dial on small enlargement (30 - 100 x)
3. Set scan mode to RAPID.
4. Set image selector switch to SE.
5. Check choice of acceleration voltage.
6. Turn emission regulator to the right until coarse figure appears.
7. Change scan mode to NORMAL and set image selector to WFM (**w**ave **f**orm **m**onitor). The monitor shows a function with some minima and maxima depending on the brightness and contrast of the picture along horizontal axis (see Fig. 4-1).



**Figure 4-1:** Optimization of emission current.

Turn emission regulator to the right as long as the brightness given on y-axes of display increases (line is shifted upwards). Then lower the brightness by approx. 1 line width by turning the regulator to the left. Emission should now be optimal.

Possible problems:

- If the sample is not in focus you might see just a straight line instead of any minima and maxima. However, you can adjust the emission as given above.
- You might not see any line on the monitor. Increase brightness to lift up the line.

8. Press the button SE and change scan mode to RAPID. Picture must appear.

### Focus control

- Set image select button to SE and scan mode to RAPID and REDUCED AREA
- Set brightness and contrast subjectively with regulator
- Possibly higher enlargement (Detail) e.g. 200 - 500 x
- Use button FOCUS COARSE AND FINE
- Squeeze out button REDUCED AREA
- Picture must now be sharp.

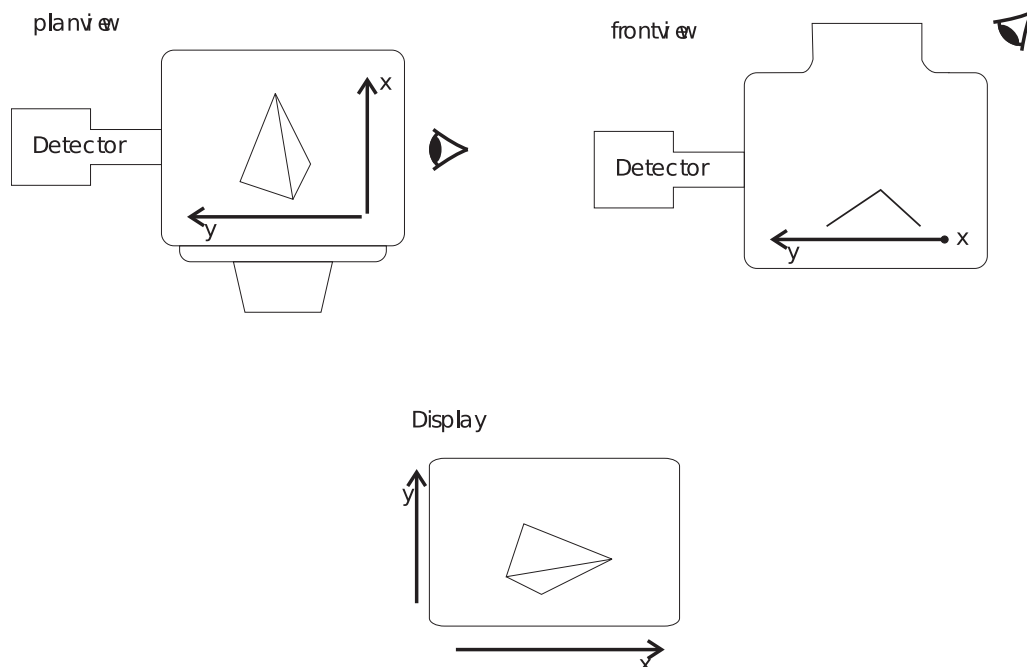
Possible problems:

- It is not possible to sharpen the picture within the range of the focus control. Check whether the working distance factor is set to the approximately distance of sample from last aperture of column. When adjusting the height (Z-drive), this must be corrected with the WORKING DISTANCE button too.
- After moving the object by X, Y or rotation drive, new focusing may be needed.
- When tilting the object and for small enlargements the tilting angle must be indicated with the rotary button DYNAMIC FOCUSING.

## 5. Sample exchange

If you start working with the SEM, the sample is inserted into the chamber before the sample chamber is evacuated. If samples have to be replaced during operation start as follows:

1. Switch off OPERATION and turn emission control knob to the left, respectively 0.
2. Ventilate column and sample chamber by pressing **Shut** (yellow) and **Air** (red) afterwards.
3. Control sample tilting angle to 0°!
4. Center sample with X and Y drive at the sample chamber door to  $X = 304$  and  $Y = 220$ .
5. Open door of sample chamber.
6. Put sample with holder in the specimen stage. Fix grub screw at the edge of the specimen stage disk (electrical contact!).
7. Estimate the distance of lower aperture to object surface and set WORKING DISTANCE accordingly.
8. Prepare a small sketch of the sample and its position in the chamber. This will make observation easier.  
\* The observer observes the sample on the screen as if he stood right next to the column and looked from the top on the sample.



**Figure 5-1:** Relation between orientation of sample and view on display.

9. Control: Are seal surfaces of sample chamber and door o.k.? Close door.
10. Press **Operate** (blue) to evacuate chamber and column.

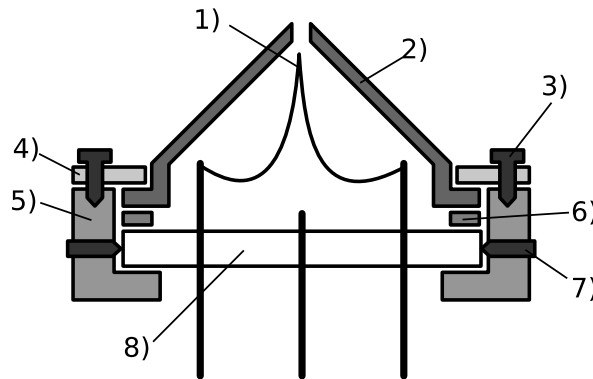
11. Control vacuum on the display instrument. If needle is in the green area of the scale the signal lamp V.L. shines green. Now wait 1-2 minutes and press red switch OPERATION.
12. Then optimize settings as described in Section: **Settings Optimization**.

## 6. Filament replacement

### Installation of a new filament

Before installing a new filament, refer also to the full version of SEM instruction manual.

- The new filament and filament mounting are installed into the cleaned metal parts (clamping and holding rings, Wehnelt cylinder) as shown in the following sketch:



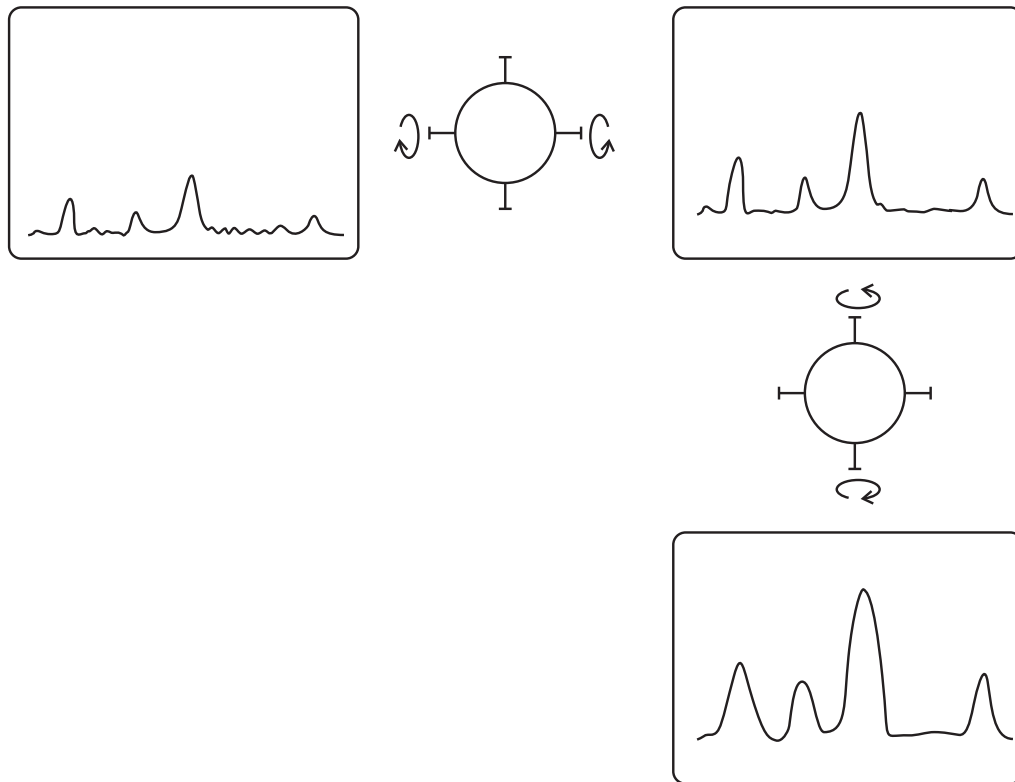
**Figure 6-1:** Sketch of a filament holder: 1) filament tip, 2) Wehnelt cylinder, 3) clamping screw, 4) clamping ring, 5) holding ring, 6) washer, 7) screws for centering, and 8) filament mounting with contacts.

- The clamping screws are only loosely fixed so that the filament mounting (ceramic) can still be moved by turning the screws for centering (set opposite in pairs) until the tip of the filament is positioned at the center of the Wehnelt cylinder opening. This should be done under a microscope. After centering, the clamping screws are fixed tightly.
- Insert the cathode into the holder of the head of column.
- Close sample chamber and column head and evacuate them by pressing the button Operate (blue).
- Check control panel according to Tab. 2-1.
- If green light (V.L.) is indicating for 1-2 minutes sufficient vacuum, switch on OPERATION button.
- Optimize settings for emission current as given in Subsection: **Optimal setting of the emission current (operating point)**.
- Proceed with optimizing of position of cathode given in Subsection: **Centering of cathode and electron beam** below.

### Centering of cathode and electron beam

- Use the Wave Form Monitor (image selector switch to WFM) option to optimize the position of cathode.

- Set scan mode to normal. A jagged line should appear. If no, please turn on button BRIGHTNESS until line appears at the lower edge.
- The cathode is mechanically centered with the four turning buttons set opposite in pairs at the upper part of the column until a maximum intensity (= max. moving of the curve into the Y direction) is shown on the screen. Example:



**Figure 6-2:** Alignment of filament holder

- The maximum value of brightness, i.e. intensity into Y direction, and thus the correct centering of the cathode and the electron beam can be controlled with the X and Y alignment regulators. If they are turned out of the 12 o'clock position, the intensity display must reduce. Otherwise, centering of electron beam has to be repeated.