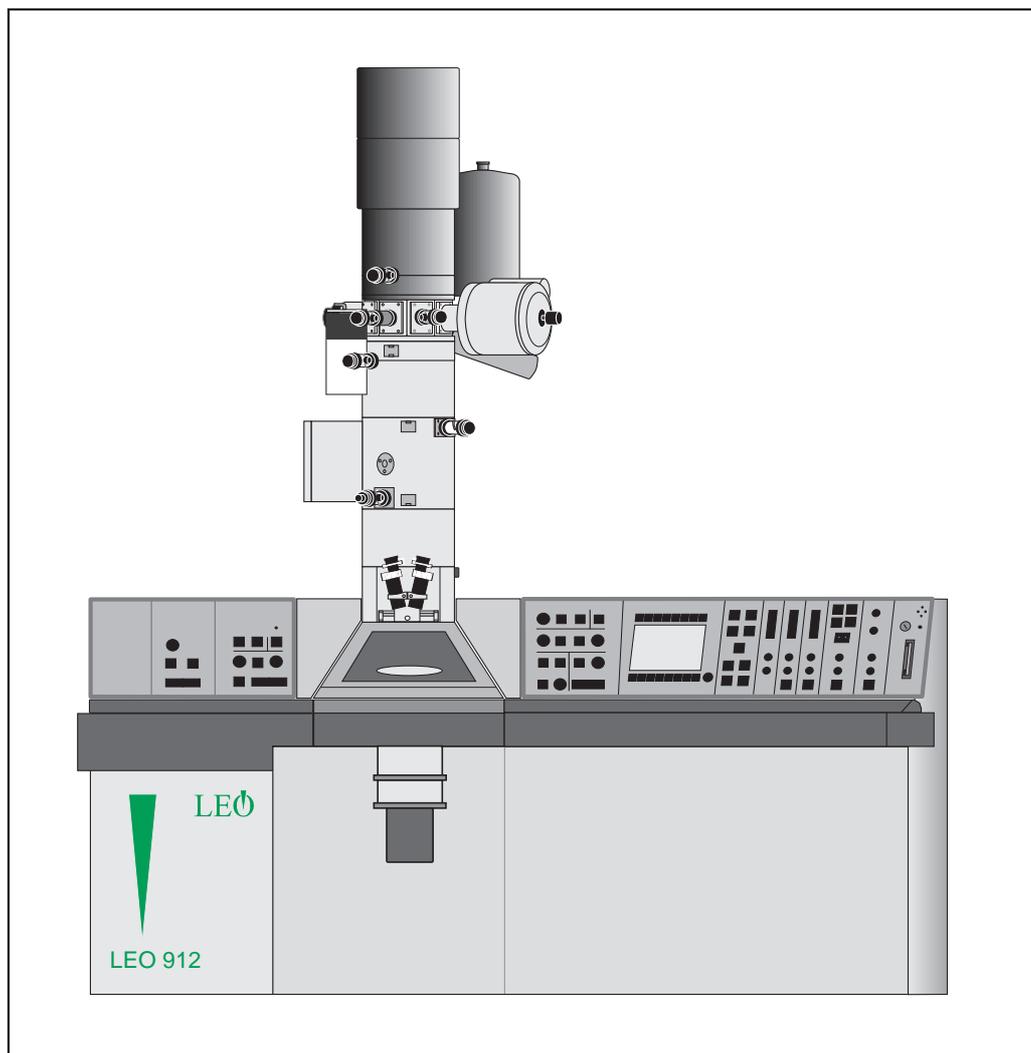


# Transmission Electron Microscope **LEO 912 OMEGA**

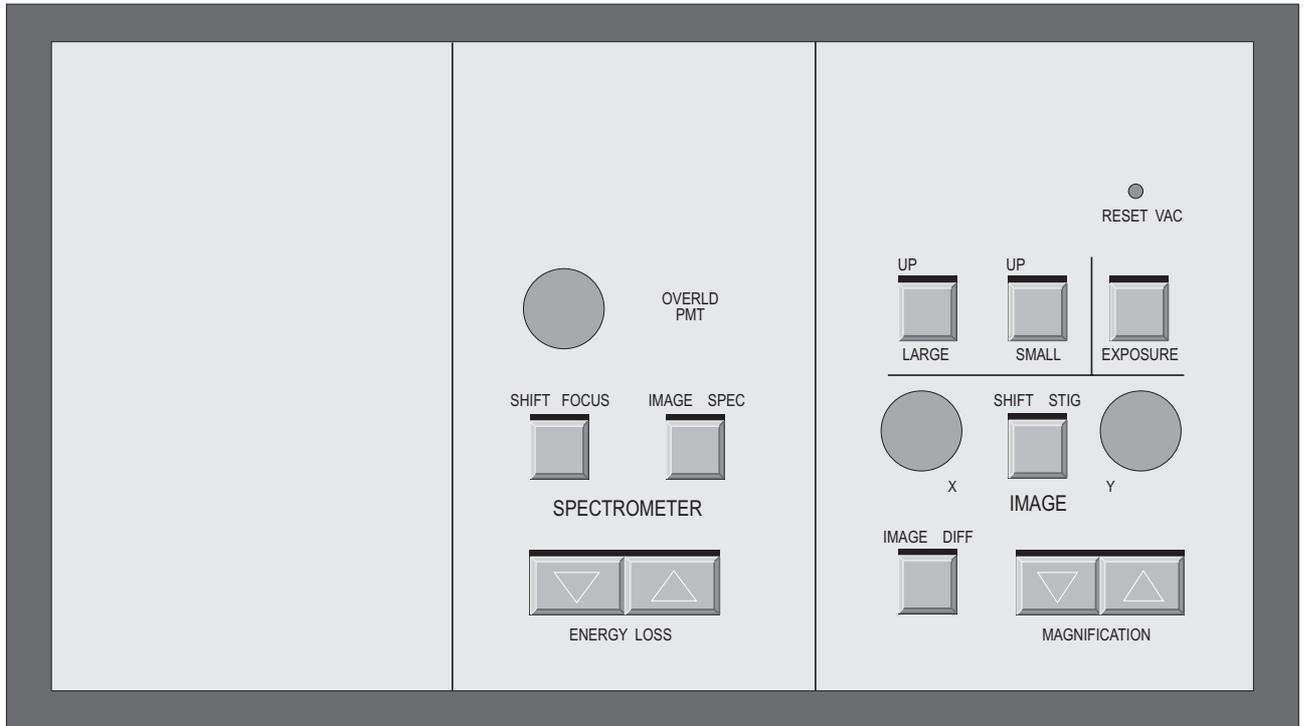


**Brief Manual  
G 34-655 e**

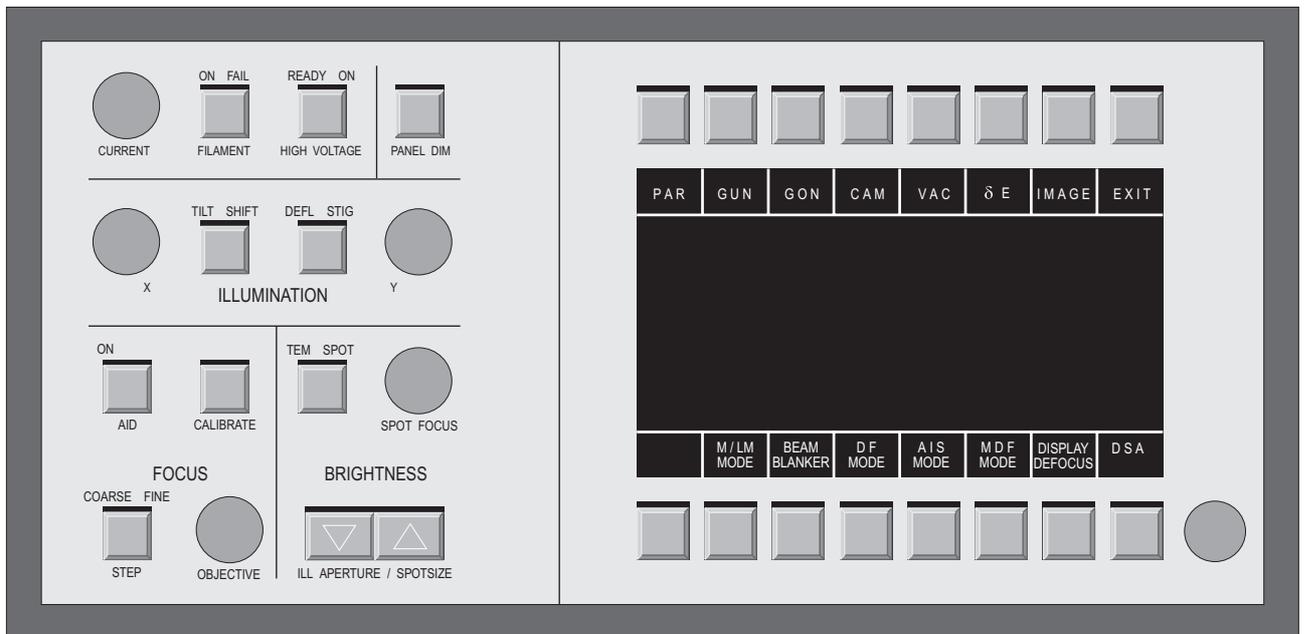
**LEO**

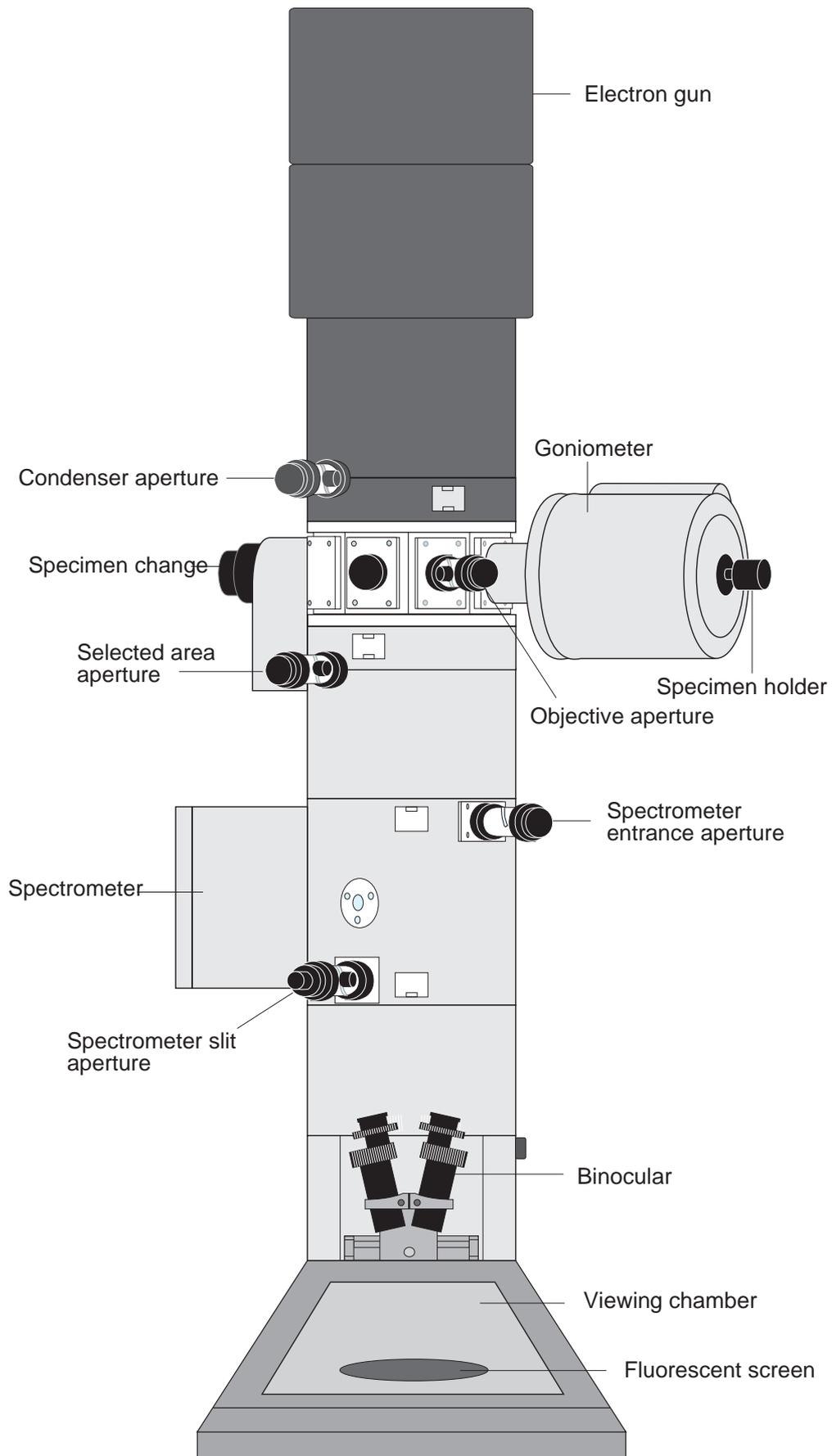
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### Left panel



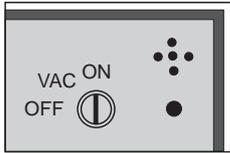
### Right panel





# 1 Microscope Operation

## 1.1 EM system turn-ON



### Getting the system ready

- **Turn key switch over VAC to ON.**
  - The vacuum system is activated for normal operation.
  - Rotary pump starts, Turbo pump starts.
  - DOS operating system, LEO 912 program and user data are loaded from the LEO 912 OMEGA hard disk.
  - LEO 912 OMEGA START menu is displayed.
  - Monitor display: LOADING SYSTEM DATA SET.

If the following message window appears, you may quit and follow the advice later.

!! IT IS TIME TO BACKUP ALL DATA !!  
ON A FLOPPY DISKETTE

!! CHECK THE AIR FILTER !!  
IN THE POWER SUPPLY

PRESS ANY KEY TO CONTINUE

- **Confirmation with any key on the keyboard.**
  - The START menu is displayed on the control monitor.
  - The host computer is ready by display of the START menu.
- **Push softkey START.**
  - LEO 912 MAIN menu is displayed.
  - Instrument readiness is indicated by a green LCD.

## 1.2 Anticontaminator



The use of liquid nitrogen requires special safety precautions for eyes and hands. See chapter 2 in the extended manual and contact your safety officer.

- **Remove lid from dewar.**
- **Fill in liquid nitrogen.**
  - Strong gas evaporation
- **Refill after 15 min. and close the dewar with lid.**

## 1.3 High-voltage selection

- **Call the GUN menu with softkey GUN.**
  - The selected high voltage is highlighted.
- **Push the corresponding softkey to change the high voltage.**
  - The new high voltage value is highlighted.
  - The objective lens is calibrated for this high voltage value (indicated by 2 sound signals).
- **Return to main menu with softkey RETURN.**

## 1.4 Operation of specimen holder

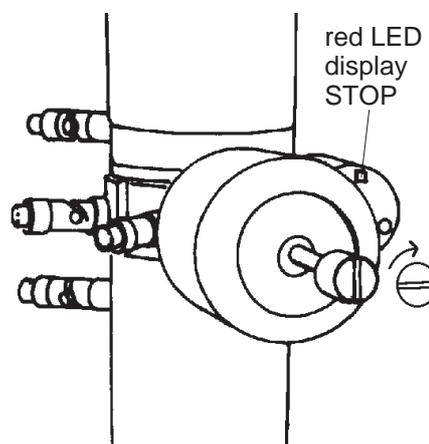


### SPECIAL NOTE:

Lock-in/out is only possible if the red LED display STOP on the goniometer is out. If not, air will penetrate into specimen and filament chambers and the ion getter pump will shut off.

Rule:

**No action if LED display STOP is on.**



### 1.4.1 Removing specimen rod

- **Retract rod as far as it will go and turn it carefully counter-clockwise until it stops.**
  - Airlock valve closes (click).
- **Again turn rod counter clockwise until stop and pull it carefully out of the goniometer.**

### 1.4.2 Load specimen

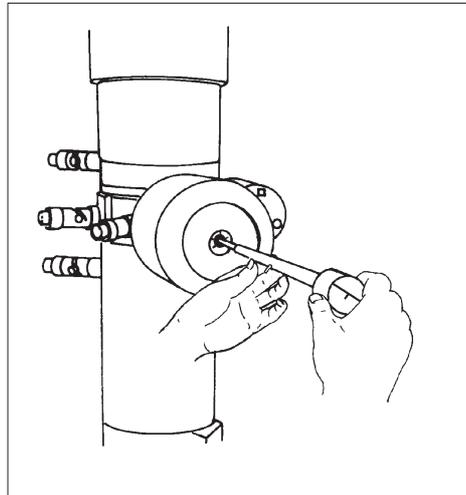
- **Place specimen rod in container.**
- **Loosen screw with collet chuck and lift it together with holding bow off.**
- **Insert new grids with tweezers.**
- **Replace holding bow and tighten screw.**

## 1.4.3

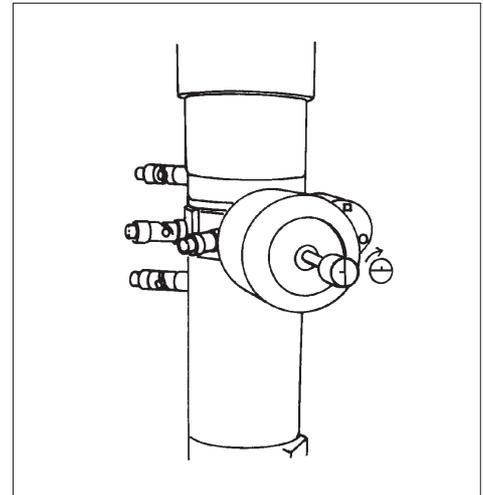
**Lock-in specimen rod****SPECIAL NOTE:**

The normal position of the specimen rod is in the high vacuum.  
Take it only out for specimen exchange.

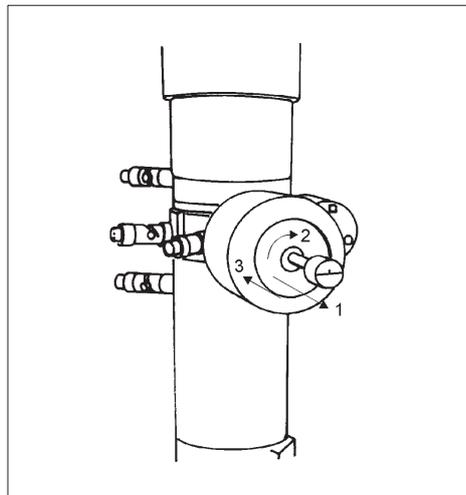
- **Slide specimen rod carefully into goniometer as far as it will go.**
- **Turn specimen rod carefully clockwise through 90° all the way to prevacuum position.**
  - Airlock valve opens (click).
  - Red LED STOP is on. Specimen rod pre-pumped in approx. 20 sec. LED STOP goes out.
- **Wait until LED STOP is out.**
- **Pull specimen rod back to stop and turn it carefully through further 90°; push rod slowly into the microscopy position**



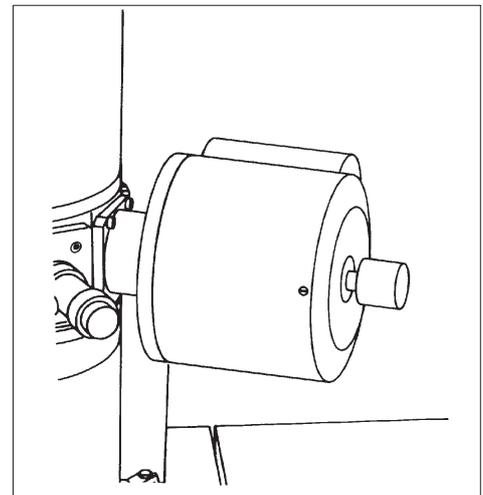
Insertion of specimen rod



Prevacuation



Prevacuation concluded



Microscopy

## 1.5 Basic alignment



### SPECIAL NOTE:

Use a test specimen (e. g. carbon foil) for the entire alignment of the microscope. Beam sensitive specimens are not useful for the alignment.

### 1.5.1 Preparation for basic alignment

- **Retract rod as far as it will go and turn it carefully somewhat counter-clockwise to keep it in this position.**
- **Insert AIS aperture and activate the AIS mode in the main menu.**
- **Remove objective aperture.**
- **Remove spectrometer entrance aperture and slit aperture.**
- **Turn on high voltage and filament.**
  - Red LED on top of key FILAMENT is on. Red LED ON is blinking on HIGH VOLTAGE key.
  - Red LED ON on top of key HIGH VOLTAGE is on; High voltage has reached it's rated value; Emission current generally  $> 1\mu\text{A}$ ; if not increase with encoder CURRENT.
  - Fluorescent screen is illuminated; if not move specimen, decrease magnification, set  $\delta E$  to zero, and adjust brightness.
- **Select magnification between 8.000x and 10.000x with buttons MAGNIFICATION  $\Delta \nabla$**

### 1.5.2 Calibrate to preset values

- **Calibrate objective lens current with button CALIBRATE in field FOCUS.**
  - while the objective lens is calibrated (indicated by 2 sound signals), the illumination is off.
- **Calibrate IM. SHIFT in the menu CAL.**

### 1.5.3 Alignment of the spectrometer

- **Reset energy loss to zero with button HCl/0eV in the menu  $\delta E$ .**
- **Switch to SPEC with button IMAGE/SPEC in the field SPECTROMETER.**
  - The spectrum caustic is visible on the screen.
- **Activate menu PARAMETER, push button CAL and then CALIBR. IM. SHIFT and CALIBR. ILL. DEFL.**
  - The illuminating and imaging system are calibrated.

- Select **SHIFT** function with button **SHIFT/FOCUS** in the field **SPECTROMETER**.
- Shift tip of the spectrum caustic to the index point of the screen with the affiliated encoder.
- Correct vertical deviation of the spectrum with the **IMAGE SHIFT** encoder **Y**.
- Switch to **IMAGE** mode with button **IMAGE/SPEC**.

#### 1.5.4 Alignment of TEM- and SPOT-illumination

- Switch to spot illumination with button **ILL Mode** and set spot size to 100 nm with button **ILL. APERTURE/SPOT SIZE**  $\Delta \nabla$ .
- Calibrate **C 3** with button **CALIBRATE** in the field **FOCUS**.
  - Spot should be imaged on the viewing screen.
- Move spot to index point on the small screen with illumination encoders **X, Y** in **SHIFT/DEFL** mode.
- Switch to **TEM** mode and deactivate **AIS** mode.
- Center condenser aperture to the index point of the small screen.
- Switch back to spot mode.
- Increase magnification to 31.500x and readjust spot to the center as described above.
- Increase magnification to 125.000x and readjust spot to the center.
- Switch to **TEM** with button **ILL MODE** and center **AIS** aperture to the large screen.

#### 1.5.5 Beam alignment

- Select 20.000x with buttons **MAGNIFICATION**  $\Delta \nabla$ .
- Select **SPOT** mode with button **ILL. MODE** in the field **BRIGHTNESS** and set spot size to 5 nm with buttons **SPOT SIZE**  $\Delta \nabla$ .
- Calibrate objective lens with button **CALIBRATE** in the field **FOCUS**. Focus spot with encoder **SPOT FOCUS**.
- Move spot to index point with encoders **ILL. SHIFT**.
- Activate the function **BEAM ALIGNM.** in the menu **GUN**.
- Set spot size to 100 nm with buttons **SPOT SIZE**  $\Delta \nabla$ .

- Shift spot to the index point of the small screen with the encoders X,Y in the field ILLUMINATION.
- Repeat the beam alignment till the spot position is identical at 5 nm and 100 nm spot size.
- Push button STORE GUN ALIGNM. and answer PROCEED ? with Y(es).
- Return to main menu with button RETURN and push button ILL. MODE to select TEM illumination.
  - The BEAM ALIGNMENT function is automatically deactivated, when leaving the GUN menu.

### 1.5.6 Adjustment of eucentric plane

- Move specimen rod all the way in.
- Activate AIS mode in the main menu.
- Insert an objective aperture and center it (see chapter “Center objective aperture”).
- Select magnification 25.000x and move specimen detail to the center of the small screen.
- Calibrate objective lens with button CALIBRATE in the field FOCUS.
- Switch on focus aid with button AID.
  - Illuminated ON indicates FOCUS AID is active; specimen detail is moving.
- Focus specimen mechanically using Z-CONTROL buttons, until specimen detail does not move any more.
  - Specimen is now in the eucentric plane of the goniometer.
- Switch off FOCUS AID with button AID.

### 1.5.7 Adjustment of beam incidence



#### SPECIAL NOTE:

This adjustment step is very important for a single field condenser/objective lens. Asymmetric diffraction fringes are created by a tilted illumination.

- Deactivate AIS mode in the main menu.
- Select magnification 63.000x.

- **Select FOCUS PAR in the PARAMETER menu and activate function WOBBLE OBJECTIVE.**
  - Specimen detail is moving in the center.
- **Switch to TILT/DEFL in the field ILLUMINATION.**
- **Minimize specimen movement with the encoders X and Y in the field ILLUMINATION**
  - The beam incidence is optimized.
  - The diffraction fringes are symmetric.
- **Switch to SHIFT/DEFL (normal position) in the field ILLUMINATION.**
- **Turn off function WOBBLE OBJECTIVE and return to the main menu.**
- **Activate AIS mode in the main menu.**

## 1.6 AIS adjustment



The AIS adjustment requires a basic alignment of the TEM. Do not change the illumination intensity during the AIS adjustment, since temperature changes of the aperture blade might create some drift of the AIS aperture. That influences the further procedure.

- **Select magnification 3.150x and set illumination angle to 0.5 mrad.**
- **Center central AIS aperture (37.5  $\mu\text{m}$ , 3rd clickstop) to the index point.**
- **Switch to SPOT mode and increase spot size to 100 nm.**
  - If the brightness is too high, increase magnification first.
- **Increase magnification to 125.000x; keep spot in the center with encoders X, Y in SHIFT/DEFL mode in the field ILLUMINATION.**
- **Switch to TEM illumination and center AIS aperture to the large screen as described above.**
- **Switch to DIFF mode, select CL = 580 mm and center diffraction spot to the index point with encoders X, Y in the field IMAGE.**
- **Insert desired objective lens aperture and center it to the diffraction spot (see chapter “Objective lens aperture”).**
- **Switch to IMAGE mode and activate AIS ADJ menu in the PARAMETER menu.**
- **Decrease magnification by 3 steps (63.000x)**

- **Activate function ADJUST AIS LOWER and center AIS aperture with encoders X, Y in the field ILLUMINATION.**
- **Switch to DIFF mode and focus the diffraction spot with encoder OBJECTIVE in the field FOCUS.**
- **Activate function ADJUST STIG CORR and correct astigmatism with encoders X, Y in the field ILLUMINATION.**
  - The diffraction spot may move by the astigmatism correction. If the spot is outside of the objective aperture, continue with the next step first and then repeat this one.
- **Activate function ADJUST TILT CORR and shift diffraction spot to the index point (Center of the objective lens aperture) with encoders X and Y in the field ILLUMINATION.**
- **Switch to IMAGE mode.**
- **Adjust the AIS system at 31.500x, 16.000x and 8.000x in the same way.**
- **Check the complete adjustment and optimize it.**

## 1.7 Centration of objective lens aperture

- **Select diffraction mode with button IMAGE/DIFF in the field IMAGE.**
- **Adjust the brightness of the diffraction image with the buttons ILL APERTURE  $\Delta$   $\nabla$ .**
- **Select a camera length of CL = 580 mm with the buttons MAGNIFICATION  $\Delta$   $\nabla$ .**
- **Center the diffraction spot to the index point with X and Y in the field IMAGE.**
- **Focus the diffraction image with encoder OBJECTIVE in the field FOCUS.**
- **Insert the desired objective aperture and center it to the diffraction spot.**
- **Switch back to IMAGE mode with button IMAGE/DIFF in the field IMAGE.**

## 1.8 Centration of spectrometer aperture

- **Insert entrance aperture (1st clickstop) and center it with the aperture drives to the large screen.**

## 1.9 Centration of spectrometer slit aperture

- **Switch to SPEC with button IMAGE/SPEC in the field SPECTROMETER.**
  - Spectrum caustic close to the center of the screen.
- **Center tip of the caustic to the index point with encoder in the field SPECTROMETER.**
- **Introduce slit aperture with the aperture drive and adjust the slit width that the spectrum caustic is within the slit.**
  - The tip of the caustic should not be too close to the corresponding side of the slit.



### SPECIAL NOTE:

If elastic brightfield is used only, the slit width might be increased so that the caustic tip is further off the slit edge.

- **Switch to IMAGE mode with button IMAGE/SPEC.**

## 1.10 Correction of imaging astigmatism

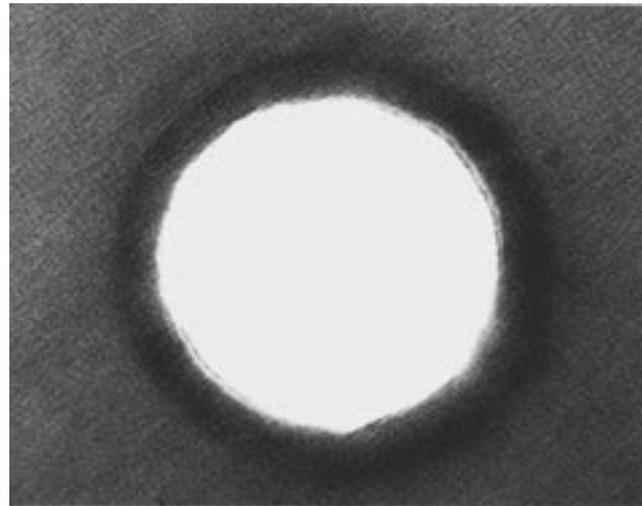
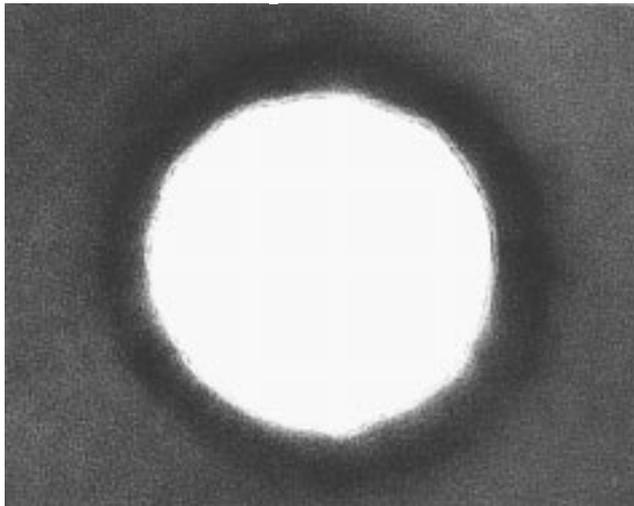
- **Select a magnification between 125.000x and 250.000x with buttons MAGNIFICATION  $\Delta$   $\nabla$ .**
- **Adjust brightness by changing the emission current with encoder CURRENT or the illumination aperture angle with the buttons ILL APERTURE  $\Delta$   $\nabla$ .**

Note, the illumination angle should not be larger than 1.6 mrad. Larger illumination angles decrease the image quality.

- **Through-focus specimen with encoder OBJECTIVE (Grain of a carbon film or embedding resin).**
  - The image is astigmatic, if the grain of the carbon film has a privileged direction in underfocus and is turned by 90° in overfocus.
- **Switch to STIG with button SHIFT/STIG in the field IMAGE.**
- **Compensate astigmatism with the encoders X and Y in the field IMAGE.**
  - The astigmatism is compensated, if there is no privileged direction of the grain in under- and overfocus.
- **Switch back to SHIFT with button SHIFT/STIG in the field IMAGE.**
- **Adjust brightness and magnification to the desired values.**

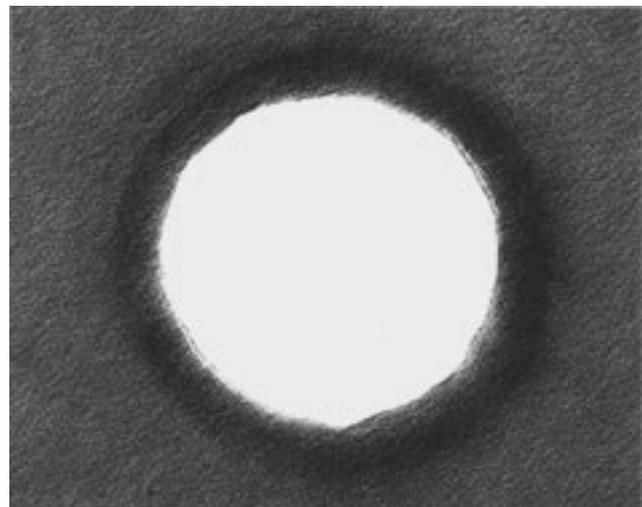
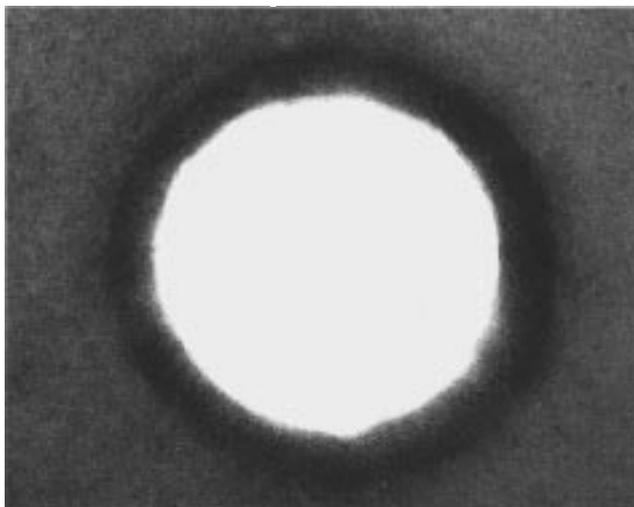
**Astigmatism compensated**

**Astigmatism existent**



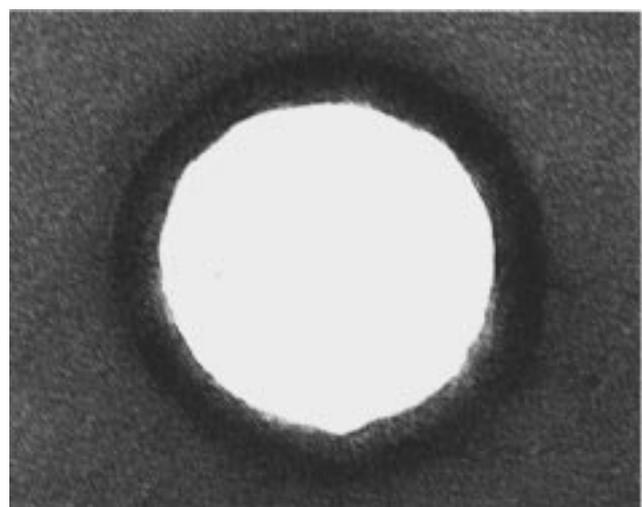
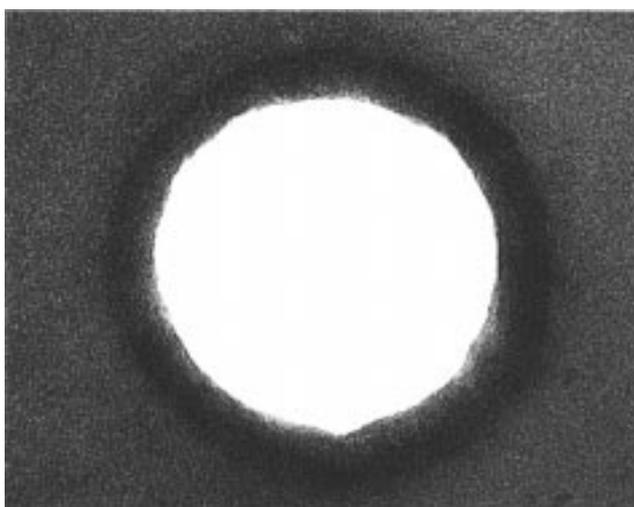
Overfocus

Overfocus



Near focus (grain almost disappears)

Near focus (grain remains visible)



Underfocus

Underfocus

## 1.11 Low magnifications (LM)

- Select LM mode with softkey M/LM in the main menu.
- Retract slit aperture.
  - The field of view might be limited by the objective lens aperture.
- Retract objective lens aperture and condenser aperture.
- Select magnification between 80x and 2000x with button MAGNIFICATION ▽ Δ in the field IMAGE.
- Adjust brightness with buttons ILL APERTURE ▽ Δ in the field BRIGHTNESS.
- Store desired specimen positions in the GON menu.
- Insert objective lens aperture and center it to the index point before switching to magnification range M (Specimen protection).

## 1.12 Medium and high magnifications

- Insert objective lens aperture (if not done yet after finishing LM).
- Insert condenser aperture (400 μm or AIS).
- Select M mode with softkey LM/M in the main menu.
- Select magnification between 3.150x and 500.000x with buttons MAGNIFICATION ▽ Δ .
- Retract condenser aperture for magnifications below 4.000x.
- Adjust brightness with the buttons ILL APERTURE ▽ Δ in the field BRIGHTNESS.
- Insert slit aperture and adjust the desired slit width.

## 1.13 Focusing the specimen

- Adjust binocular to your eye sight and focus it on the index point of the small screen.
- Move specimen detail to index point.
- Switch on focus AID and push CALIBRATE in the field FOCUS.

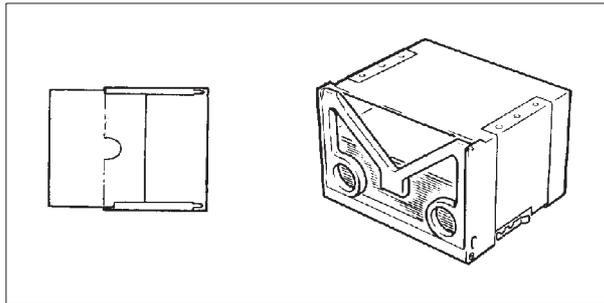
- Specimen detail is moving in the center.
- Minimize the specimen movement with Z-Control and then focus specimen with encoder OBJECTIVE, until the detail stops moving.
  - The specimen is in Gaussian focus.
  - The Gaussian focus is the start position for photography with automatic defocus.
- Switch off focus AID in the field FOCUS.

## 1.14 Photography

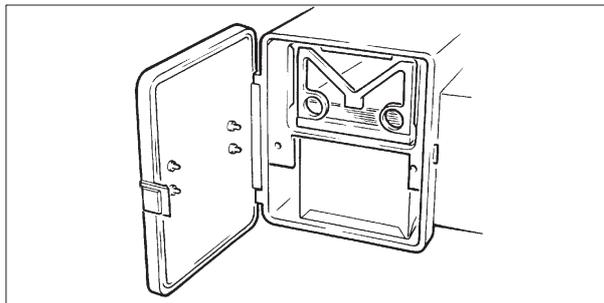
### 1.14.1 Loading and removing film

There are different types of cassettes available for the different types of fotomaterial. Here we deal with sheet film which is loaded in 1.5 mm thick metal cassettes. The size of film is 3.25 x 4 inch.

- Load film in the cassettes (1) and put them into the magazine.

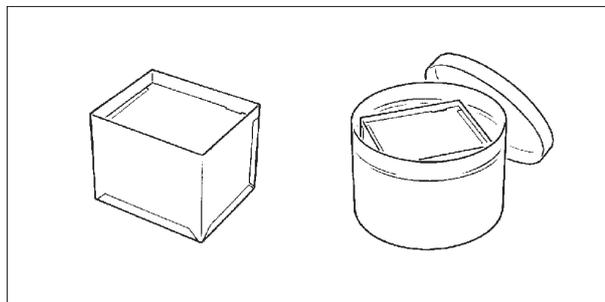


- Dry the films in a desiccator for several hours or overnight.



- Ventilate CAM in the VACUUM menu and insert new magazine in the upper part and the film collector in the lower part of the camera chamber.
- Close camera and EVAC CAM in the VACUUM menu.

- For removing films VENT CAM again and put films in the light-tight container.



## 1.14.2 Exposure

- Focus the specimen (see point 1.13).
- Activate submenu FOCUS PAR in the menu PARAMETER.
- Select defocus mode e. g. MAG.-DEP. DEFOCUS.
- Toggle defocus with button SELECT DEFOCUS.
  - The defocus value of the mode LOW, MEDIUM or HIGH is set automatically for the next photograph.
- Check visually the defocus with softkey CHECK DEFOCUS.
  - The image is shown on the screen with the desired defocus. It is indicated by a sound signal.
  - Judgment of the automatic defocus is possible before the photograph is taken.
- Start the exposure with the button EXPOSURE.
  - Large screen raises; film cassette moves to the exposure position, exposure is taken and cassette drops in receiving container.
  - Large screen is lowered. Camera is ready for the next exposure.

## 1.15 Turning - OFF

- Switch off FILAMENT and HIGH VOLTAGE.
- Return to main menu with softkey RETURN.
- Push softkey EXIT and answer PROCEED? with Y.
  - All instrument parameters are stored on harddisk.
  - Start menu is shown.
- Turn key switch to OFF
  - Turbo pump is partly ventilated with nitrogen gas.
  - Rotary pump is switched off.



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