

LIBRA® 120

Transmission Electron Microscope

User's manual



Enabling the Nano-Age World



LIBRA®120

Operating Manual
344200-0000-036

User's manual



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1 General

In the Transmission Electron Microscope (TEM) elastic and inelastic scattering processes are the most important interactions between primary electrons and the specimen.

Inelastically scattered electrons in particular contain a great amount of information about specimen structure, mass distribution and thickness, and about the distribution of elements and molecules. Such information cannot be utilized in conventional TEMs, where inelastically scattered electrons even reduce image contrast and image sharpness.

But the integration of an imaging energy spectrometer in the imaging beam path of the TEM permits information contained in inelastically scattered electrons to be fully utilized, and considerably increases the quality of imaging and diffraction. The Energy Filter Transmission Electron Microscope (EFTEM) offers many more operating modes than conventional TEMs, e.g.:

- Elastic brightfield imaging
- Electron Spectroscopic Imaging (ESI)
- Electron Spectroscopic Diffraction (ESD)
- Electron Energy Loss Spectroscopy (EELS)
- Element or structural contrast imaging
- Plasmon loss imaging
- Image EELS, etc.

Because of these advantages, filter electron microscopy has found worldwide acceptance within a very short time.

The LIBRA 120 is the successor of the EM 912 OMEGA which was the first EFTEM using a fully magnetic omega spectrometer. The easy and comfortable operation of the LIBRA 120 is fully maintained by integration of the energy filter in the electron optical system of the instrument.

One of the major characteristics of the LIBRA 120 is the design of the electron optics. The optically correct position of the essential elements of illumination and imaging systems and of the imaging spectrometer guarantees that objective lens and omega filter operate at fixed excitation in all illumination and imaging modes. This reduces adjustments by the user to a minimum.

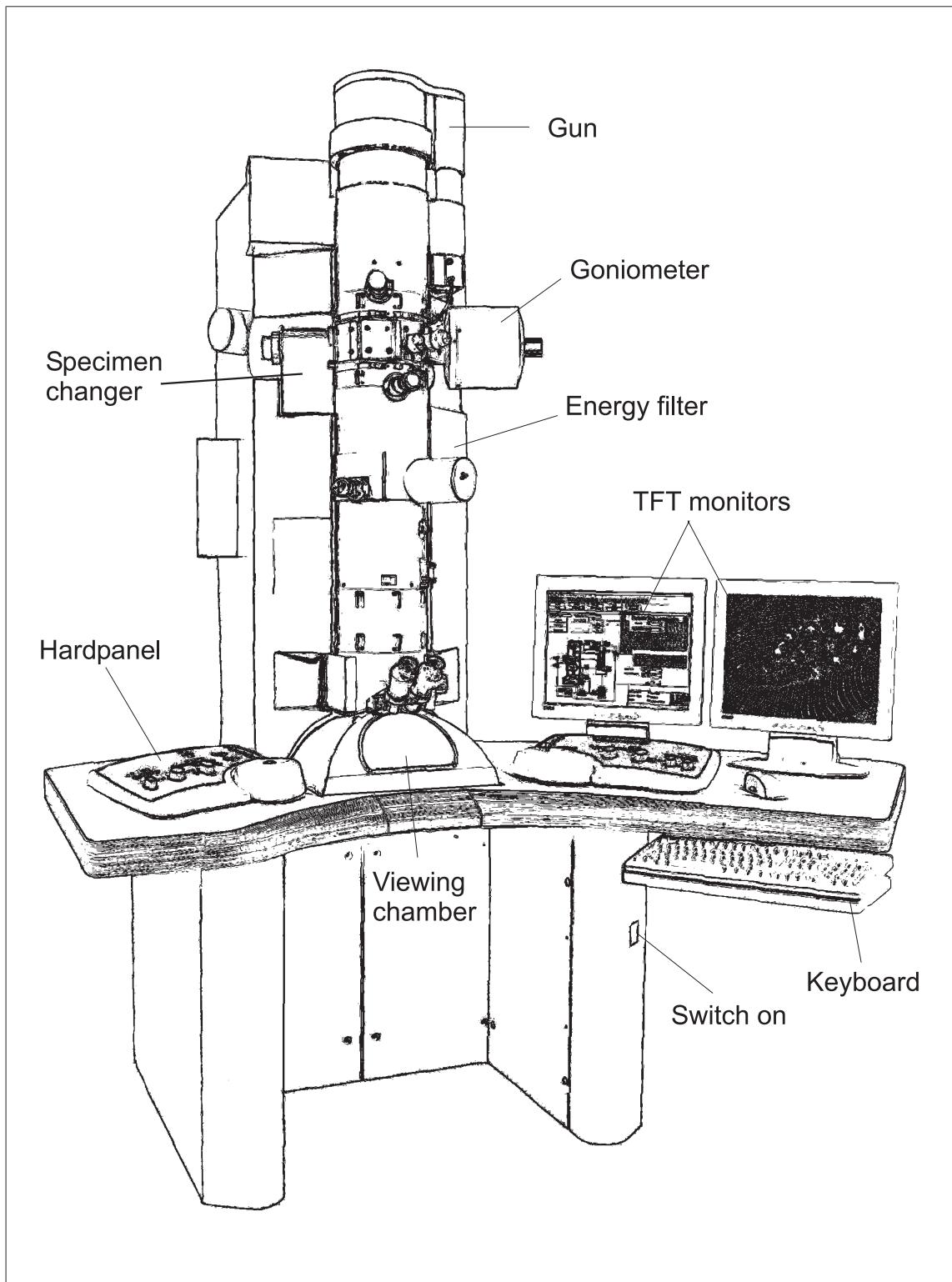
The LIBRA 120 features the Köhler illumination system which was realised in the EM 912 OMEGA for homogeneous and parallel specimen illumination. An automatic system assures that only the visible area on the fluorescent screen is illuminated at all magnifications, which provides the best specimen protection.

The modern “multiprocessor/multitasking” computer control of the instrument based on the Windows platform together with 2 modern Hard Panels permit easy and effective use even of extraordinary imaging and analysis modes.

The LIBRA 120 is of modular design. All functions necessary for standard examinations are incorporated in the base instrument. It can be upgraded for all presently known imaging and analysis modes by corresponding hardware and software supplements.

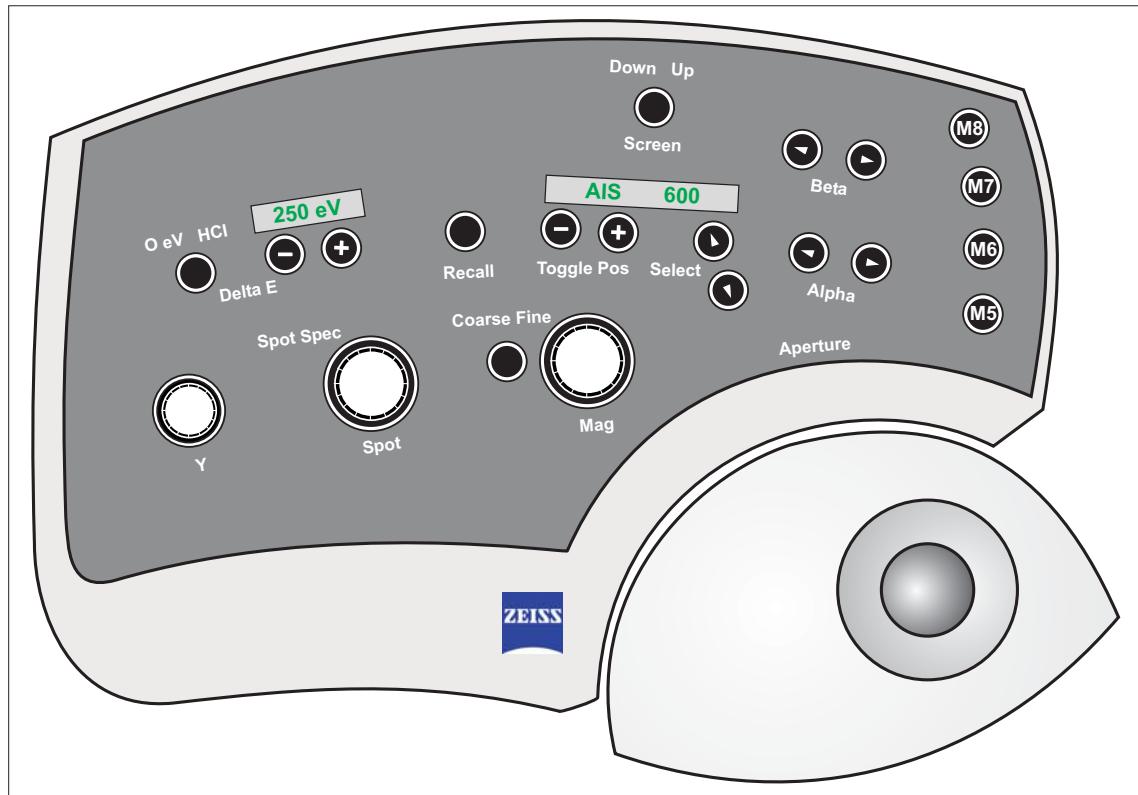
All important instrument functions are also remote-controllable by an external computer, and allow automation of all kind of functional operation.

Overview of the LIBRA 120

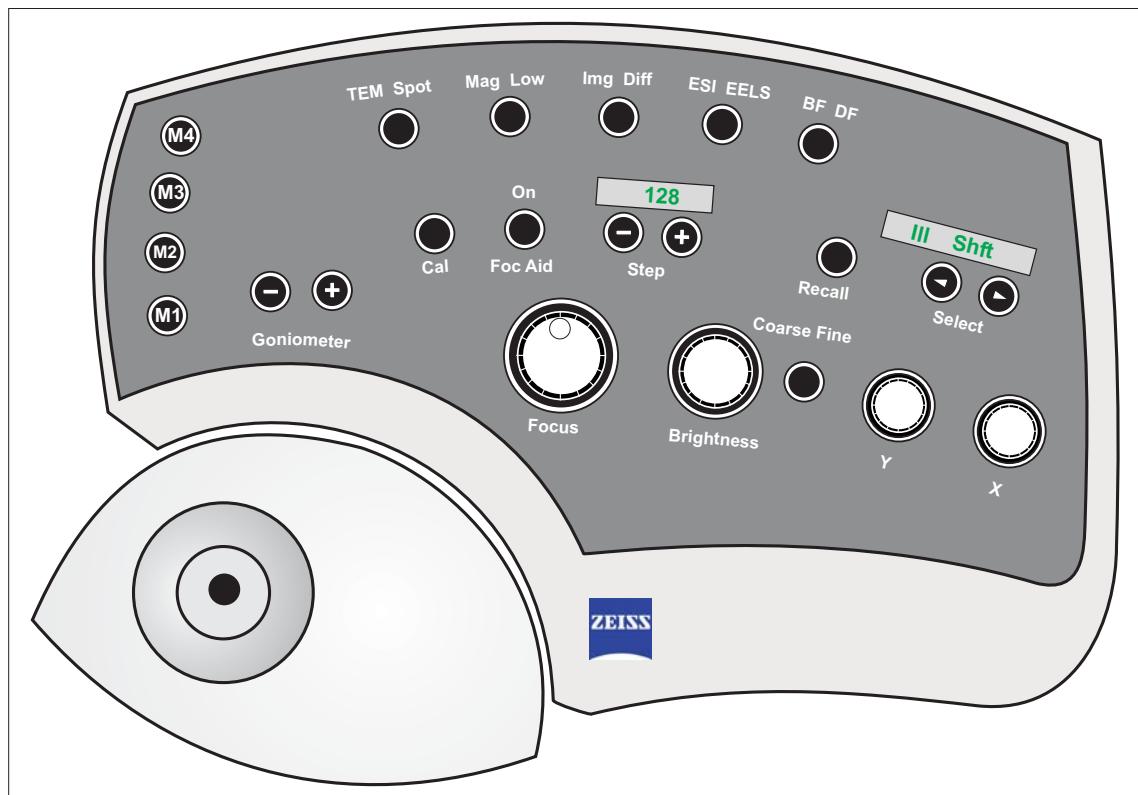


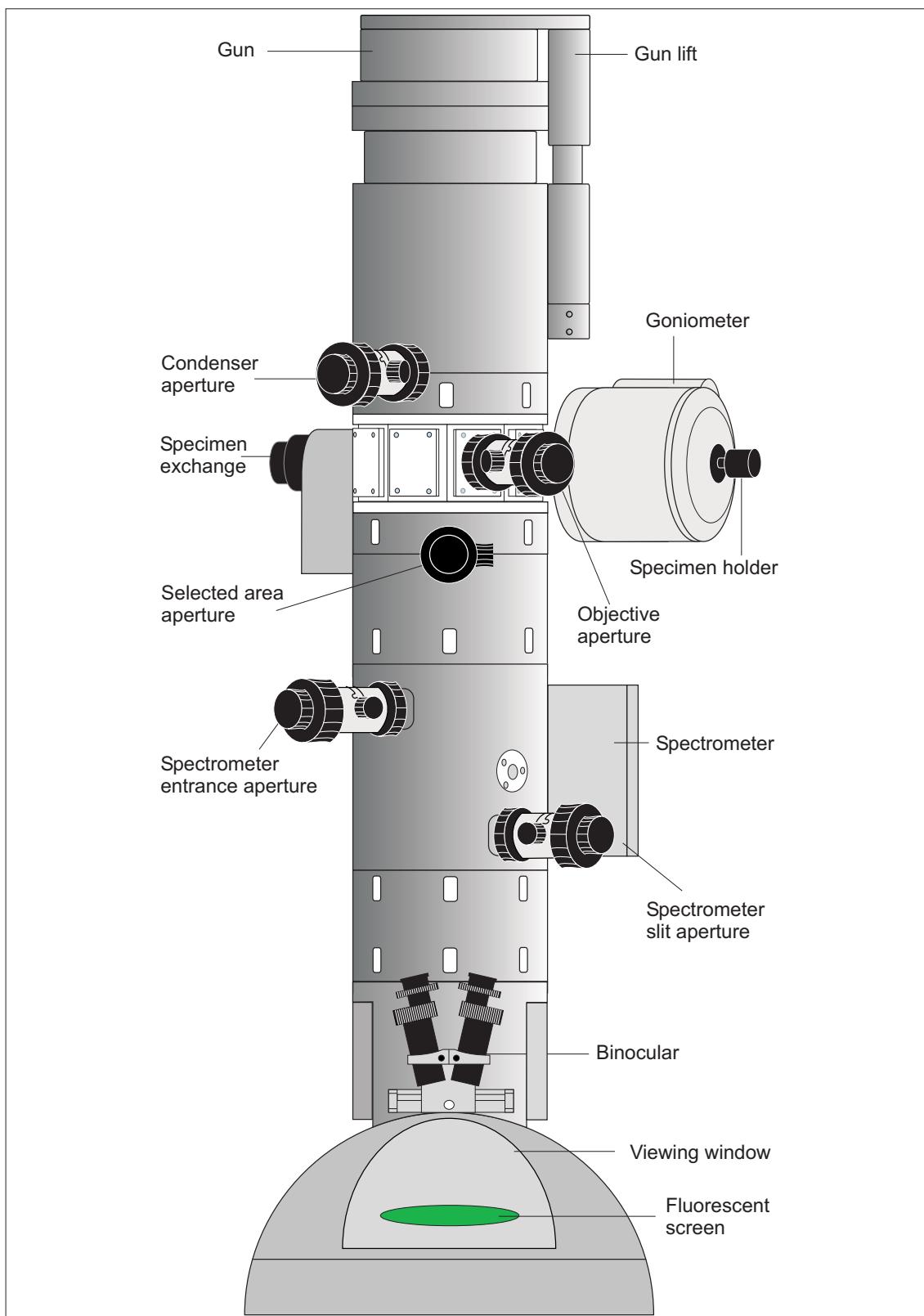
Hardpanels

Hardpanel on the left hand side



Hardpanel on the right hand side



Column LIBRA 120

2 Safety

This chapter comprises the most important safety rules for the daily work. For more information look at the separate manual.

2.1 Safety precautions for Beryllium

Beryllium is used in the LIBRA 120 for technical reasons, but limited to the absolutely necessary quantity.

Beryllium in the base instrument

The LIBRA 120 is prepared for the attachment of an EDX detector. Beryllium is required in the objective lens area to prevent bremsstrahlung.

With the objective lens closed the user is not directly exposed to Beryllium fine dust or vapor.

- Maintenance work in the objective lens area (exchange of objective lens apertures) and mounting of attachments on the objective lens should be carried out only by the ZEISS NTS Service.
- The following general notes on the use of Beryllium should be strictly observed.

EDS rods made of Beryllium

Specimen holder and holding plate of the rods are made of Beryllium. If EDX rods are correctly used, no dust or vapor is generated which would be harmful to your health.

When handling the rod, the following safety precautions should be strictly observed:

- Touching the front end of the rod briefly by mistake is harmless but should be avoided to prevent vacuum breakdown. Use only the prescribed tools for specimen exchange (tweezers, plate lifter).

It is recommended to wear gloves.

- Mechanical polishing, the use of etchants for cleaning, and baking (heating) of the specimen holder are strictly forbidden, because highly toxic dust and vapor may be generated.
- Stop working if abrasion traces are noticeable on the specimen holder.
- Only specialists who know the risks involved in the use of Beryllium are allowed to handle the EDX rods (accessibility).
- The rods must be stored in a fireproof, lockable place.
- Please observe the following general instructions for the use of Beryllium.

General notes on the use of Beryllium (excerpt from the corresponding DIN data sheet)



CAUTION

Beryllium is a highly toxic, easily oxidizable, hard metal. Animal experiments have proved it to be cancerogenic. Beryllium is especially dangerous, if it enters the body through the respiratory system in the form of dust or vapor. Serious allergic and chronic poisoning symptoms will appear, followed by severe organic lesions. The effects of the poisoning may appear even many years after exposition.

- Suitable safety measures must be taken for all kinds of processing likely to produce Beryllium dust or vapor (grinding, sawing, soldering) or soluble Beryllium compounds (mordant, etchant). No such processing is required in the LIBRA 120.
- Short-time, barehanded contact with clean Beryllium parts (surfaces) is harmless, but gloves should be worn for handling Be parts.
- Do not eat, drink, or smoke in the working area.
- Store Beryllium parts fireproof and well locked (e.g. in metal containers).

Waste disposal

Beryllium-containing parts must be specially disposed of as toxic waste, in compliance with the relevant legal and local provisions.

2.2 Safety precautions for Liquid Nitrogen

The anticontaminator, an attachment of the LIBRA 120, uses liquid Nitrogen. It must be refilled once a day at least . The following safety precautions must be observed for the use of liquid Nitrogen.

Preliminary remarks

Approx. 78% of our respiratory air consist of actually harmless Nitrogen.

The use of Nitrogen liquefied at a temperature of minus 196 °C may be dangerous.

The following risks are particularly important:



CAUTION
Scalding by liquid Nitrogen on the skin!

Because of the low temperature liquid Nitrogen may destroy the skin severely, similar to scalding by hot fluids or burns.

Splashes or small drops of liquid Nitrogen cause the generation of an insulating layer of gaseous Nitrogen between skin and nitrogen drops due to the body temperature (Leidenfrost phenomenon).

There will be no such protective effect due to Leidenfrost's phenomenon if more liquid Nitrogen gets in contact with the skin or clothes are soaked. The drastic thermosteresis of liquid Nitrogen on the skin will then bring about the aforementioned burn symptoms.



CAUTION
Implosion risk when pouring liquid Nitrogen into glass Dewar vessels!

Dewar vessels are double-walled, with a vacuum between the walls (thermos bottle principle).

Always wear a face mask when handling glass vessels, or, better, use metal Dewar vessels. Due to their higher stability metal Dewar vessels withstand the temperature shock when pouring in liquid Nitrogen.



CAUTION
Explosion risk if liquid Nitrogen penetrates between the walls of the Dewar vessel!

Liquid Nitrogen which penetrates the insulating space through pores or fissures of a defective Dewar vessel will evaporate due to heat absorption on the inner side of the outer wall. The vessel will explode because the gaseous Nitrogen cannot escape quickly enough.



CAUTION

Fire risk due to enrichment of liquid Nitrogen by liquid oxygen!

Oxygen liquifies already at minus 183°C, and the Oxygen in the air condenses on the surface of the liquid Nitrogen (minus 196 °C) and enriches it gradually by liquid oxygen. At concentrations of more than 5% liquid Oxygen, flammable material such as dust, paper, wood shavings or foam material will burn when falling into the Dewar vessel.

Therefore:

- Cover the vessels so that evaporating Nitrogen can escape, but the entrance of air is inhibited and no flammable material can fall in.
- Use vessels with narrow neck which are also more economic because of slower Nitrogen evaporation.



CAUTION

Oxygen deficiency risk due to Nitrogen enrichment of respiratory air!

The amount of evaporating Nitrogen in the workroom should not noticeably reduce the relative amount of oxygen in the respiratory air. The permissible lower limit of the Oxygen content is 20vol% (normally approx. 21vol%).

Standard values for maximum amount of liquid Nitrogen in surface rooms

- Enclosed room with windows and doors: 1.5l/cubic meter room volume
- Naturally aired room: 30l/cubic meter room volume
- Constantly air-conditioned room: 150l/cubic meter room volume

Liquid Nitrogen may be used or stored in subterranean rooms only if

- a controlled mechanical ventilation is available

or

- evaporated Nitrogen which because of its higher density accumulates at the ground or in depressions is able to drain off without damage.

2.3 X-ray safety

Electron beams which strike matter generate X-rays. Their intensity and hardness (wavelength) depend on the accelerating voltage, the beam current and the material they strike.

It is guaranteed for the LIBRA 120 by a number of active measures (beam current limitation, safety breakers) and passive protection provisions (shieldings) that the permissible maximum doses in compliance with the X-ray rules (Modified RöV) of July 2002 are not exceeded, even in case of irregular or faulty operation.

The earlier classification of 2 categories does not exist anymore. EMs with or without an X-ray detector belong to the same category (this was different in the past).

The radiation limit is: **1 µSv/h measured in 10 cm distance from the device.**

The X-ray measurement determines the equivalent dose in Sievert (Sv). The equivalent dose describes the effect of ionizing radiation on the human body.

In Germany owners of instruments have to inform the local authorities before start-up of the EM.

After the approval the instrument may be operated without the need for permanent X-ray safety control. After expiration of the approval a new one has to be applied for.

The approval applies only to the entire instrument, including all current column and chamber accessories. Any changes or replacements of accessories require a new approval.



WARNING

All shieldings especially at the column must not be removed.

Only the ZEISS service is authorized to remove the shielding for service purposes.

3 LIBRA 120 Components

3.1 Computer system

General

Mouse driven system operation by menu-oriented graphical user interface (GUI). Hard panel control for most common functions. Complete data record storage and personalised storage of user parameters on hard disk.

Expert Operating System

Driving electron optics and controlling the subsystems; interface and safety filter for remote operation; comprising a host computer with Windows® operating system.

Dedicated Autonomous Subsystems

Autonomous microprocessor subsystems for monitoring and control of vacuum system, goniometer, high voltage and system status. Immediate protection circuit response and host communication.

Multilayer Management System

Event-driven, bi-directional communications between host computer and subsystems through optical CAN-bus. Direct control of lens and deflection systems through multiplexing control system. Full remote capability through 2x RS232 and 1x TTL interface.

Overview

The LIBRA120 computer system comprises

- a WINDOWS operated PC System.
- EO boards controlling the Electron Optics and the subsystems like Camera, Vacuum, Gun etc. via CAN bus.
- a flexible data and program memory.

Inputs for the computer system are made:

- from two control panels to the left and right of the column.
- from the Graphic User Interface (GUI).
- from a keyboard

Output units are

- TFT display
- a printer

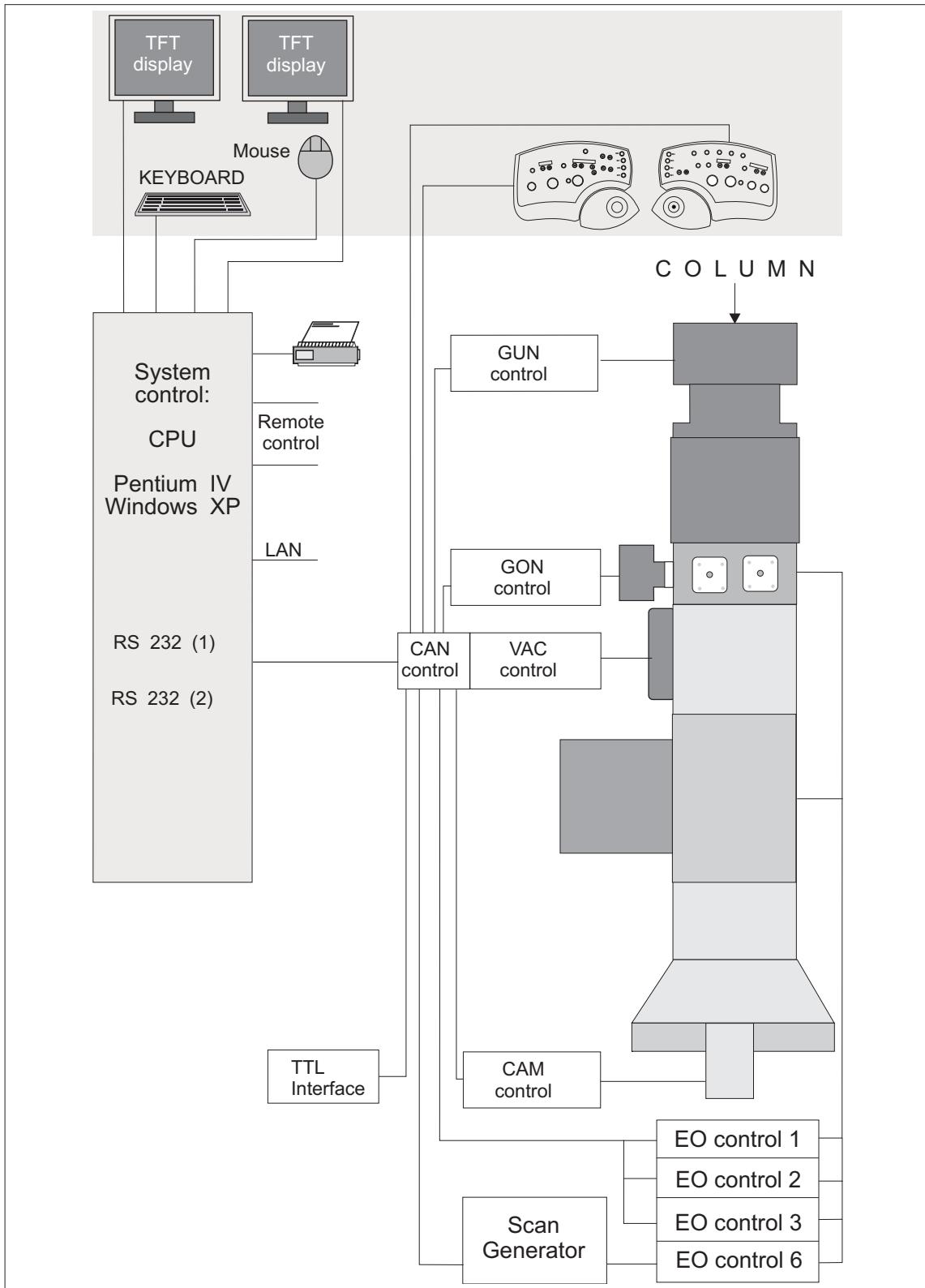


Fig.: 3 - 1 Block diagram of the LIBRA 120 Computer System

3.2 Electron optics

The electron-optical system of the LIBRA 120 consists of ten lenses in total, and an imaging omega spectrometer integrated in the system as a 1:1 imaging element. The total system comprises the following optical components:

Illumination system

A flexible condenser system for Köhler illumination in TEM mode (imaging, selected area diffraction) and for SPOT illumination (spot analysis, scan mode, convergent beam electron diffraction).

Objective lens

A Riecke/Ruska-type condenser/objective single field lens with constant excitation, which guarantees a fixed position of the first diffraction or intermediate image.

First projector lens system

A three-lens magnification system to form an image of the first intermediate or diffraction image in the fixed entrance image plane of the following energy spectrometer.

Energy spectrometer

An omega imaging energy spectrometer which supplies an achromatic 1:1 image of its entrance image plane, and at the same time forms a spectrally dispersed 1:1 image of its entrance pupil (energy loss spectrum) from which the analyzer slit selects the desired energy field.

Second projector lens system

A two-lens magnification system to image the filtered image or diffraction pattern or the electron energy loss spectrum on the fluorescent screen or the camera system of the microscope.

3.2.1 The Illumination System

A single-field condenser objective lens is the most suitable for the analytical Transmission Electron Microscope (TEM). The symmetrical position of the specimen in the center of the pole piece gap permits

- High-resolution TEM mode
- the generation of small electron probes for analysis (SPOT mode)
- Selected Area Diffraction (SAD)
- Scan mode
- optimized arrangement of the detectors for the scanning modes (SE, BSE, EDX)
- maximum specimen tilt

In former designs of this of objective lens type, either an auxiliary lens or a considerably changed excitation of the objective lens have been used for TEM illumination, whereas the LIBRA 120 applies the Köhler illumination principle known from light optics.

In the analytical TEM, optimized illumination must meet the following requirements:

- The illumination angle must be variable to match coherence of illumination, image brightness and specimen contrast.
- The size of the illuminated specimen area should not be much larger than the imaged specimen area, to prevent irradiation damage and contrast-reducing scattering electrons.
- The beam should be vertically incident on all spots of the specimen to prevent off-axis image distortion.
- The specimen should be homogeneously illuminated.
- Easy change between TEM and SPOT illumination.
- Tilt and shift of illumination should be independently adjustable.

Illumination modes

There are two possibilities of specimen illumination in the LIBRA 120 (Fig. 3-3):

TEM widefield illumination for

- TEM imaging and
- Selected Area Diffraction

SPOT illumination for

- Materials analysis (EDX)
- Diffraction examinations (Nanoprobe, Convergent Beam Electron Diffraction (CBED))
- Scanning Transmission Electron Microscopy (STEM))

3.2.1.1 TEM illumination in MAG mode

The illumination system consists of 3 condensers. The 1st and 2nd condensers form a zoom system which images the crossover of the gun variably reduced in the constant Zoom Image Plane (ZIP).

In *Magnification (MAG (M)) mode* for magnifications starting at 4,000x the 3rd condenser transmits the crossover image to the front focal point of the objective prefield lens. The illuminating cones for each object point are thus parallel to the optical axis and have the same aperture (σ).

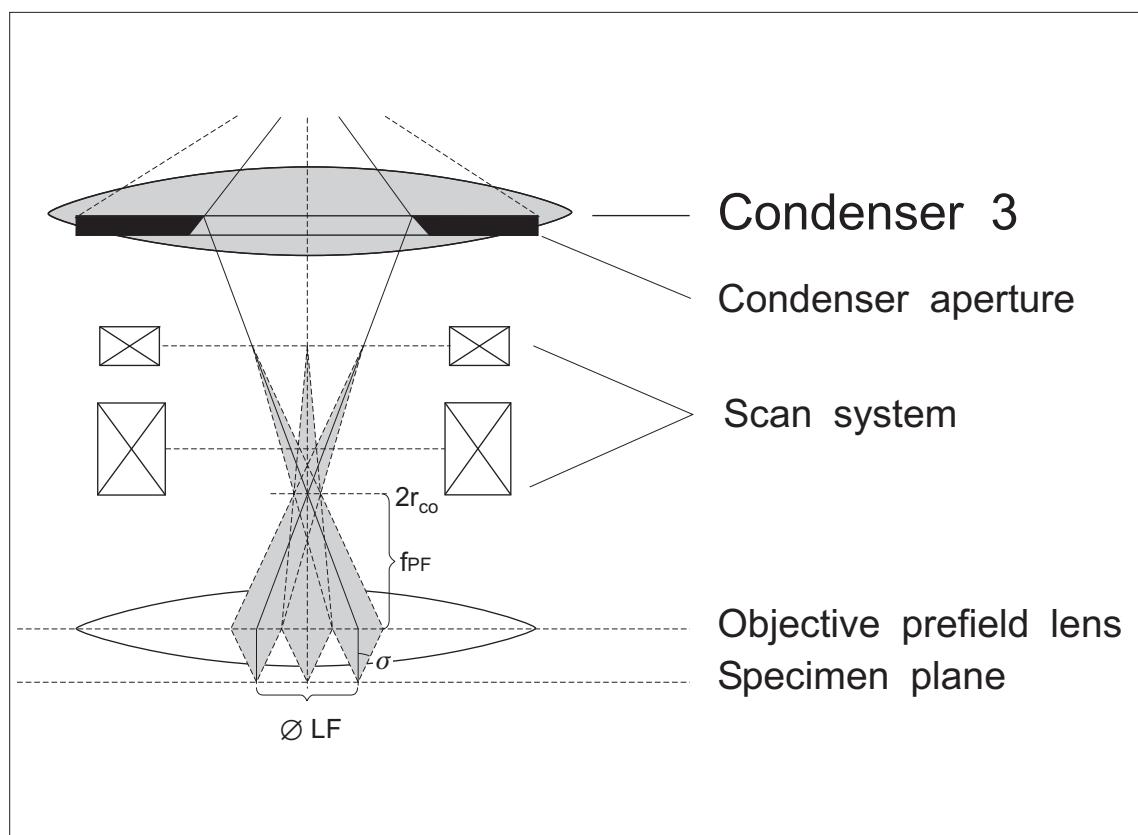


Fig.: 3 - 2 Illuminating cones for several object spots

The illuminating aperture σ is given by the radius of the crossover image r_{co} , divided by the focal length of the objective prefield lens f_{PF} , and can be varied by different excitation of the condenser zoom lenses C1 and C2.

The condenser aperture is placed in the principal plane of the 3rd condenser lens and limits the luminous field. The size of the luminous field LF is defined by the diameter of the condenser aperture and its demagnification by the objective prefield lens.

Luminous field and illuminating apertures are selected by separate independent variables and are thus also variable independently of each other.

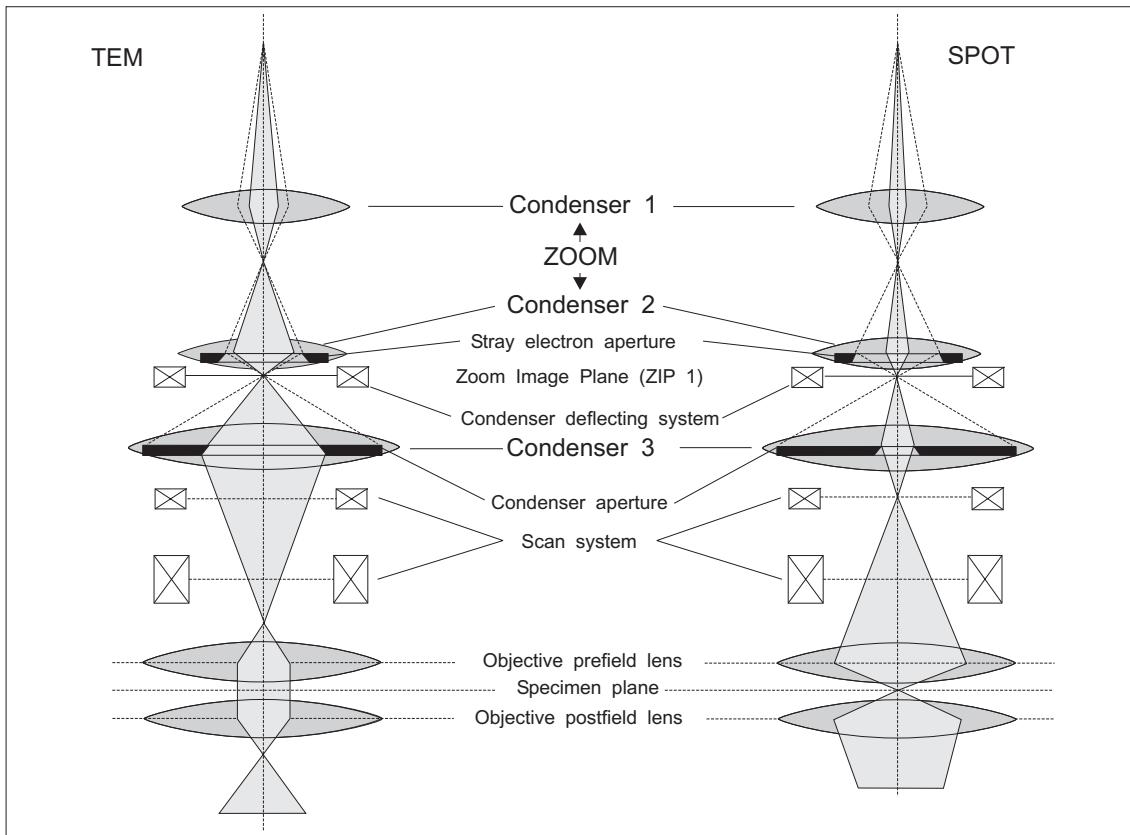


Fig.: 3 - 3 TEM and SPOT illumination in MAG mode

3.2.1.2 SPOT illumination in MAG mode

For SPOT illumination in MAG mode the 3rd condenser images the crossover in the entrance image plane of the objective prefield lens, which produces an approx. 20x reduced crossover image on the specimen. Here, the condenser aperture acts as a condenser aperture for the focused spot.

3.2.1.3 TEM illumination in LM mode

In Low Magnification (LOW MAG (LM)) mode for magnifications from 50x to 1,250x the objective lens operates with a long focal length. The focal point of the objective prefield lens lies in the medium plane of the upper deflecting elements of the Scan System. The 3rd condenser images the crossover image from the zoom image plane (ZIP) in this plane.

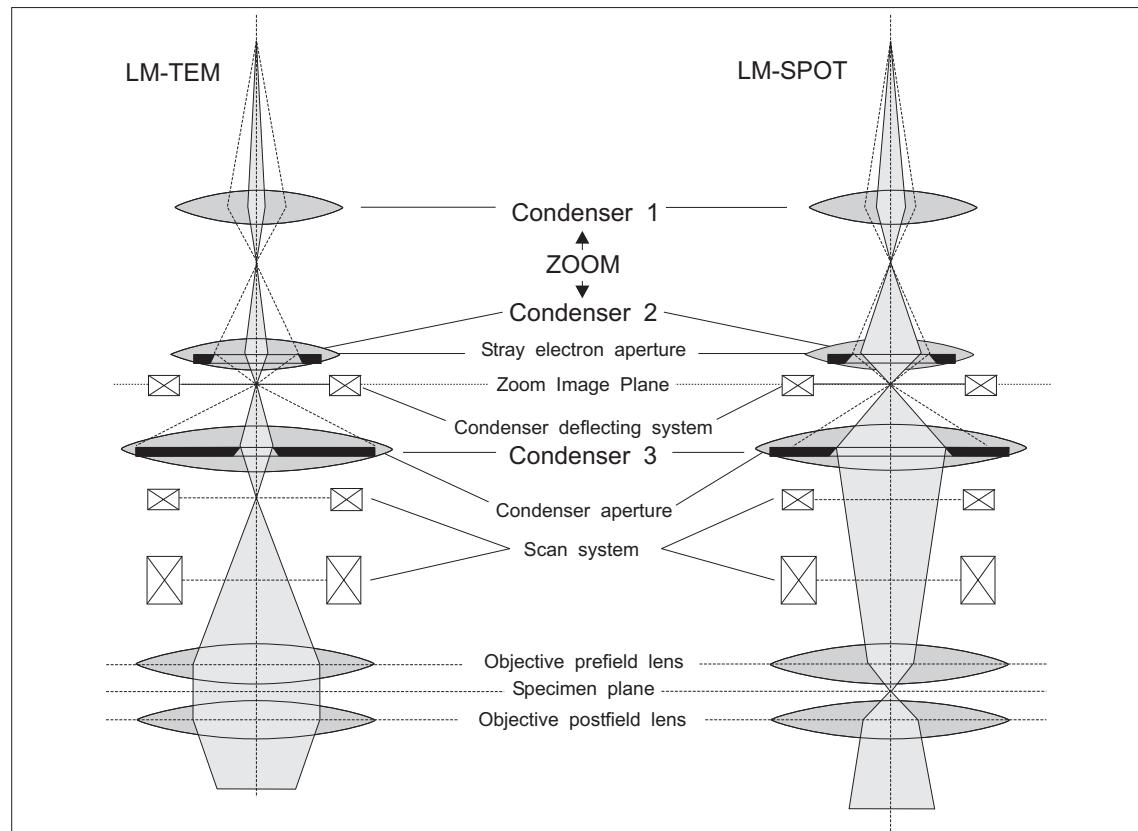


Fig.: 3 - 4 TEM and SPOT illumination in LM mode

3.2.1.4 Illumination direction

Illumination shift (ILLUMINATION SHIFT)

A double deflecting system (Scan System) above the objective lens has its transition point in the front focal plane of the objective prefield lens. It allows the illuminating electron beam to be shifted without tilting.

Together with TEM illumination it permits among others Minimum Dose Focusing (MDF) in the LIBRA 120. The method consists of focusing on a specimen spot adjacent to the selected specimen area, which prevents damage of the specimen by irradiation. With illumination shift the beam jumps only for the time of exposure to the area selected for photography. In SPOT mode the illumination shift can be used to direct the luminous spot with the scan shift of the Scan System to any specimen spot, thus realizing SCAN mode.

Illumination tilt (ILLUMINATION TILT)

The upper deflecting system of the Scan System has its tilt plane in a conjugate plane of the object plane (objective lens front focal plane). The illuminating electron beam thus passes at an angle through the object plane if only the upper deflecting element is activated.

With an objective lens aperture, the beam tilted this way can produce also darkfield images (DF mode) in TEM illumination. The direction of the incident beam is then adjusted so that the scattered electrons reach a path parallel to the optical axis and thus produce the image. The objective lens aperture will here mask out the non-scattered electrons.

Illumination tilt applied in SPOT illumination allows diffraction examinations using Convergent Beam Electron Diffraction (CBED).

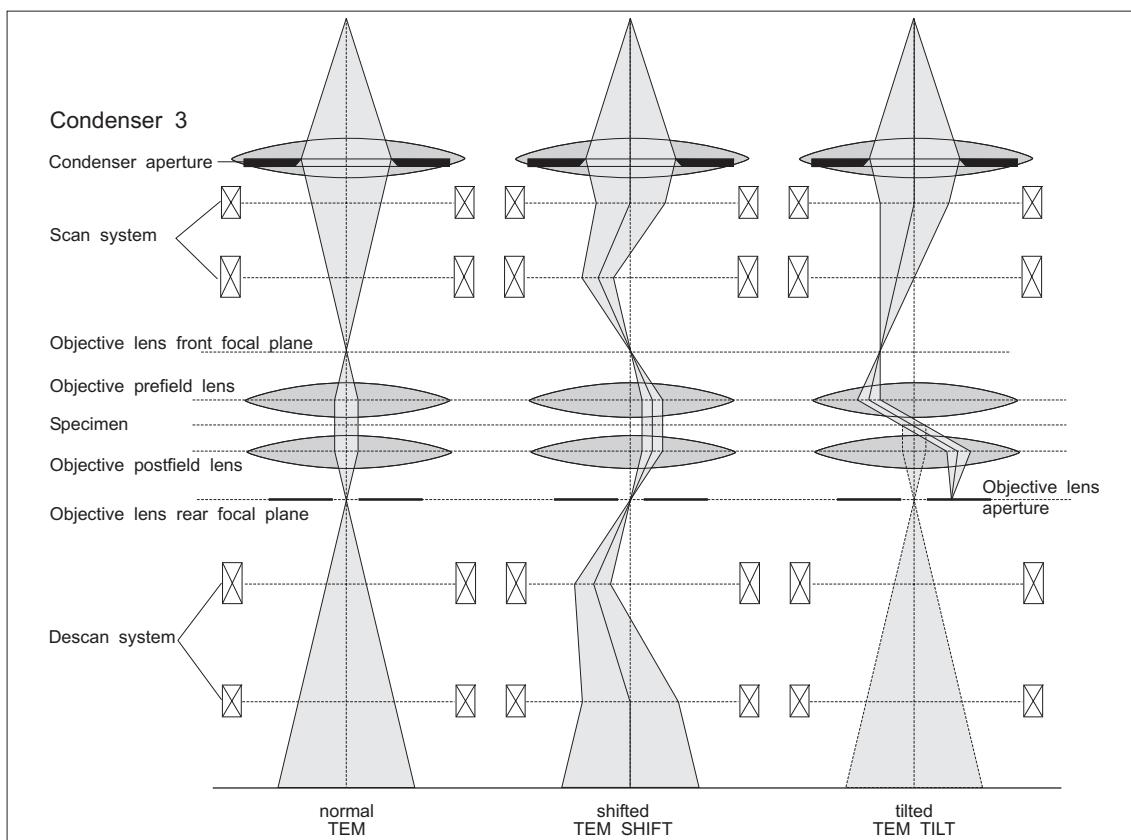


Fig.: 3 - 5 Normal, shifted, and tilted TEM illumination in MAG mode

The different illumination beam paths for normal, shifted, and tilted illumination are shown in Fig. 3-5 and 3-6.

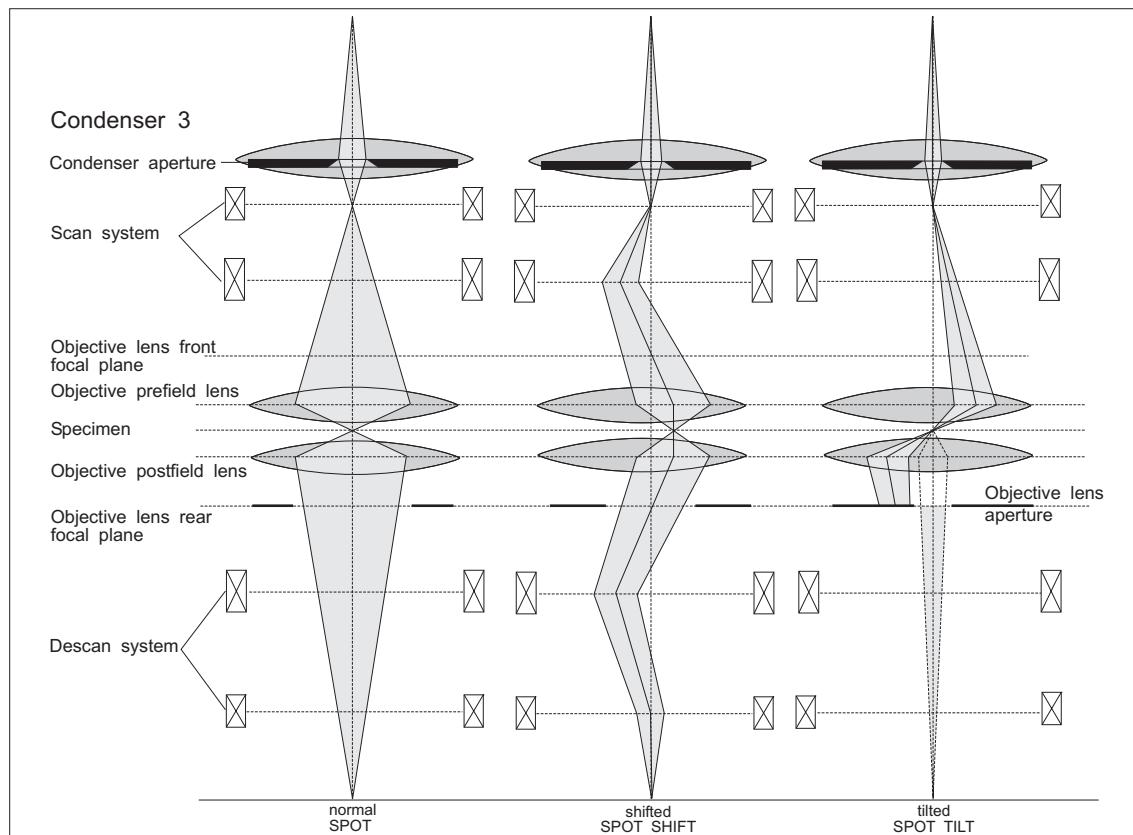


Fig.: 3 - 6 Normal, shifted, and tilted SPOT illumination in MAG mode

3.2.1.5 Brightness adjustment

The brightness (illuminating aperture or ILL. ANGLE) is adjustable by changing the crossover image with the condenser zoom system. The adjustment is made with the double key ILL. APERTURE / SPOT SIZE in the field BRIGHTNESS, which changes the lens currents of condensers 1 and 2 by the condenser zoom control.

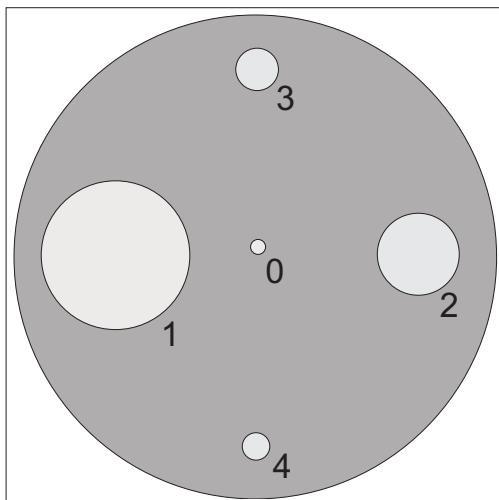
3.2.1.6 TEM illumination aperture selection

The LIBRA 120 offers 3 possibilities for illumination-aperture selection:

- Manual change of condenser aperture (= luminous field aperture).
- Electronic change of AIS aperture with automatic aperture selection as a function of the adjusted magnification (**Automatic Illumination-aperture Selection (AIS mode)**).
- Electronic change of the AIS aperture with aperture selection by the user (**Manual Illumination-aperture Selection (MIS mode)**).

In AIS or MIS mode the condenser deflecting system deflects the beam to either one of 5 different holes of the multi-hole aperture (Fig. 1-17). The 5-hole aperture is arranged in the main plane of the 3rd condenser lens. The 3rd condenser lens images the crossover in the front focal point of the objective prefield lens. The Scan System operates synchronously with the condenser deflecting system and deflects the beam after passing through the aperture back to the optical axis.

The assignment of magnification and AIS aperture is as follows:



| AIS position | Diameter (µm) | Magnification range |
|--------------|---------------|---------------------|
| 1 | 600 | 4k - 16k |
| 2 | 300 | 20k - 31k |
| 3 | 150 | 40k - 63k |
| 4 | 75 | 80k - 125k |
| 0 | 37 | $\geq 160k$ |
| 0 | 37 | SPOT mode |

The beam path for AIS and MIS mode is shown in Fig. 3-7 for on-axis and off-axis aperture positions.

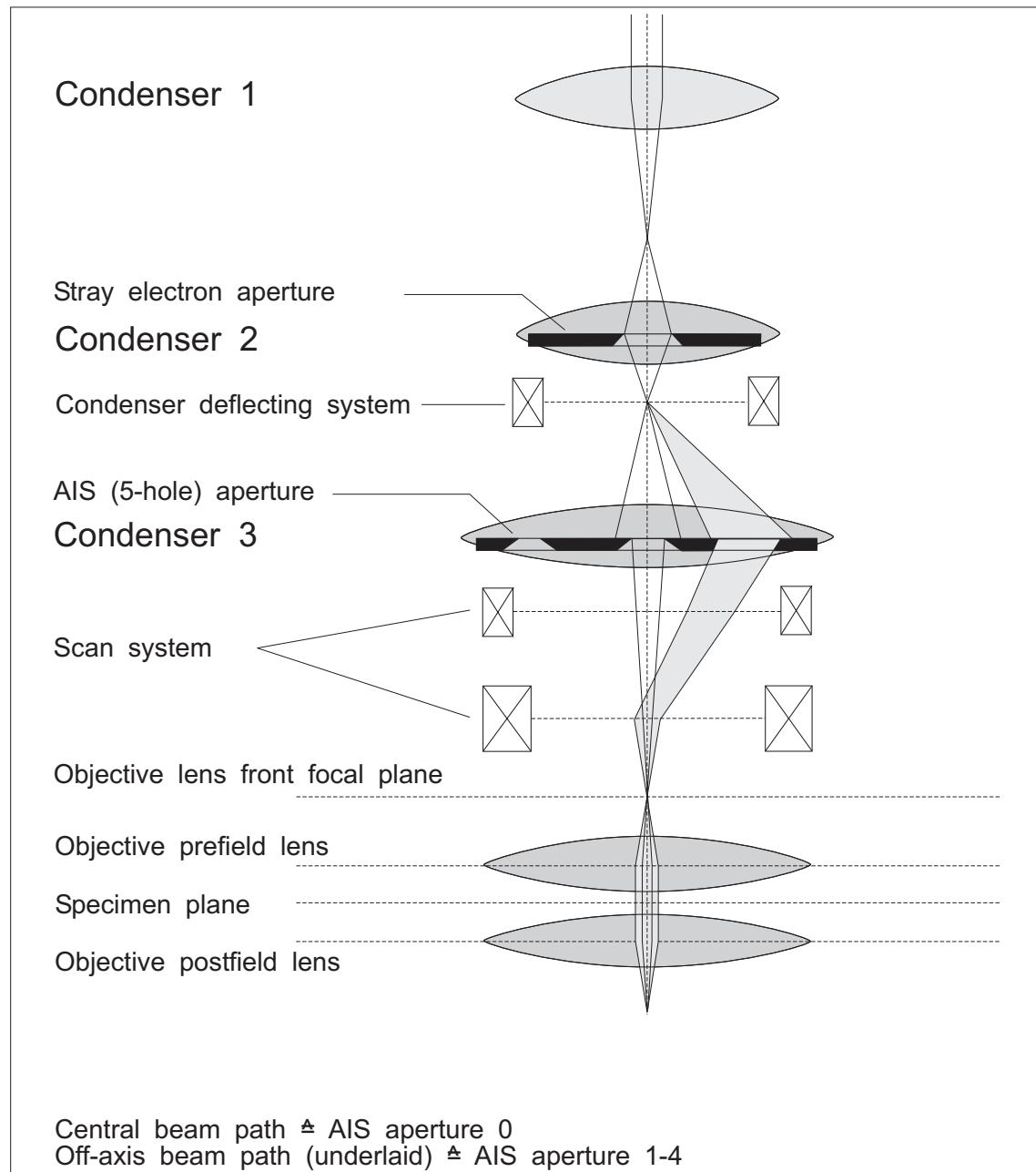


Fig.: 3 - 7 Beam path in AIS mode

3.2.1.7 AIS control

The AIS control unit serves to select the condenser deflecting system and synchronize it with the Scan System control. The latter joins the static values of illumination tilt and shift with the offset values of the selected AIS aperture. It also controls the power supplies for the upper and lower deflecting elements of the Scan System.

In AIS mode the beam passes obliquely through the 3rd condenser (C3). The excitation of the condenser stigmator is, therefore, adjusted depending on the AIS aperture. The condenser stigmator control is connected with the AIS control unit for this purpose. Separately for each AIS aperture a tilt correction of deflection and lens aberrations is required, which becomes effective through the Scan System Control.

The stigmator values and the values of illumination tilt and shift are entered in the menu AIS ADJUST by the encoders X and Y on the right hardpanel.

In AIS mode the C3 fine current value is set by turning the encoder SPOT FOCUS fully counterclockwise; the setting is invariable because the excitation of C3 influences the direction of irradiation.

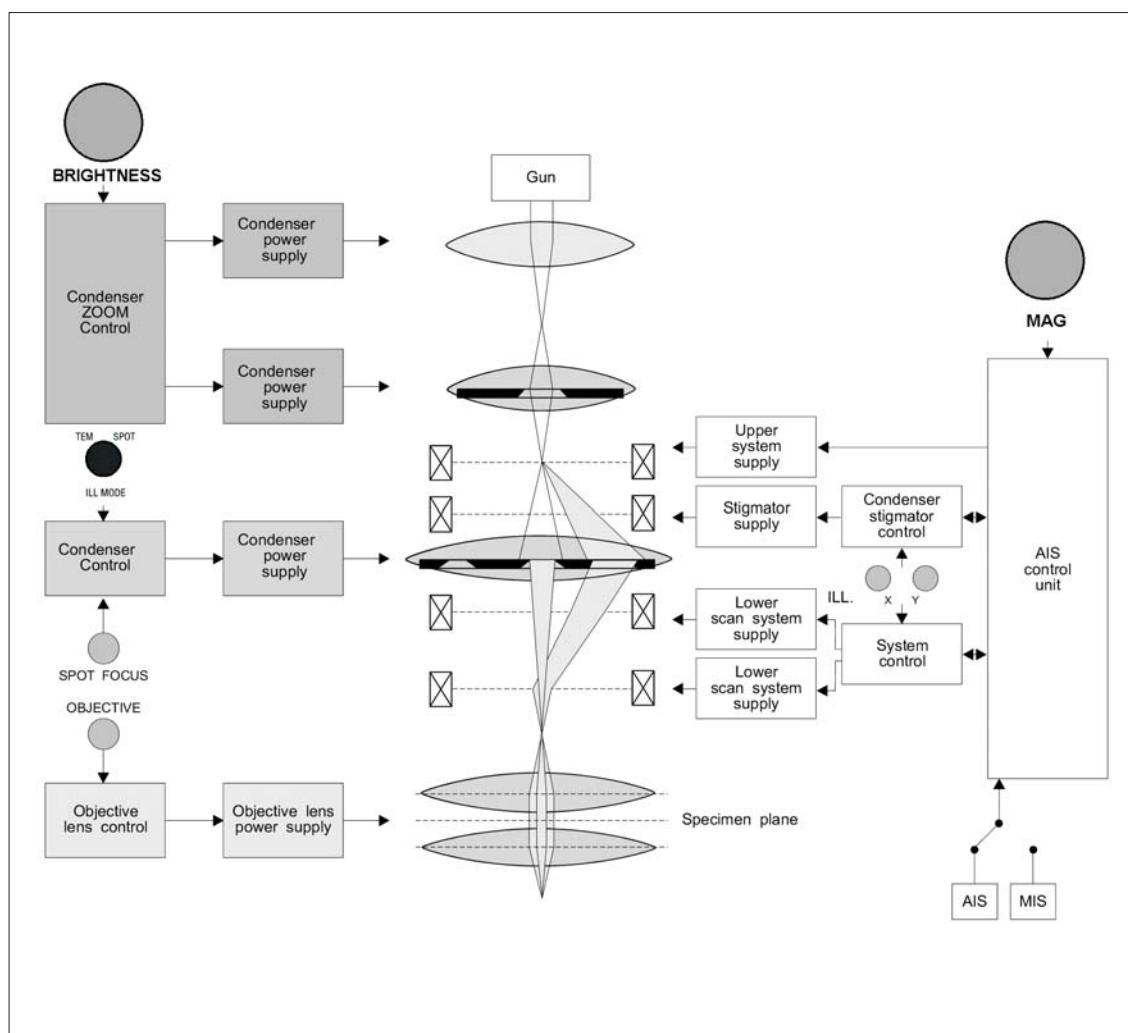


Fig.: 3 - 8 AIS beam path and AIS control

3.2.1.8 HR Mode (High Resolution Mode)

To minimize the energy spread in the illumination system (Boersch effect), the number of crossovers in the beam path should be reduced to a minimum.

The LIBRA 120 offers the HR mode with only one CO image in the front focal plane of the objective prefield lens (OPF) (Fig. 3-9). In this mode the CO of the gun is imaged for illuminating angles $\sigma \geq 1.6\text{mrad}$ in the front focal plane of the OPF lens by C1 and C3 only. The AIS mode is automatically deactivated, if the HR mode is activated and the illuminating angle is selected $\geq 1.6\text{mrad}$.

NOTICE:

Köhler illumination is also realized in HR mode, because the electron source (CO) is imaged in the focal plane of the last condenser lens (OPF), and the OPF lens images the condenser (luminous-field) aperture in the specimen plane at the same time.

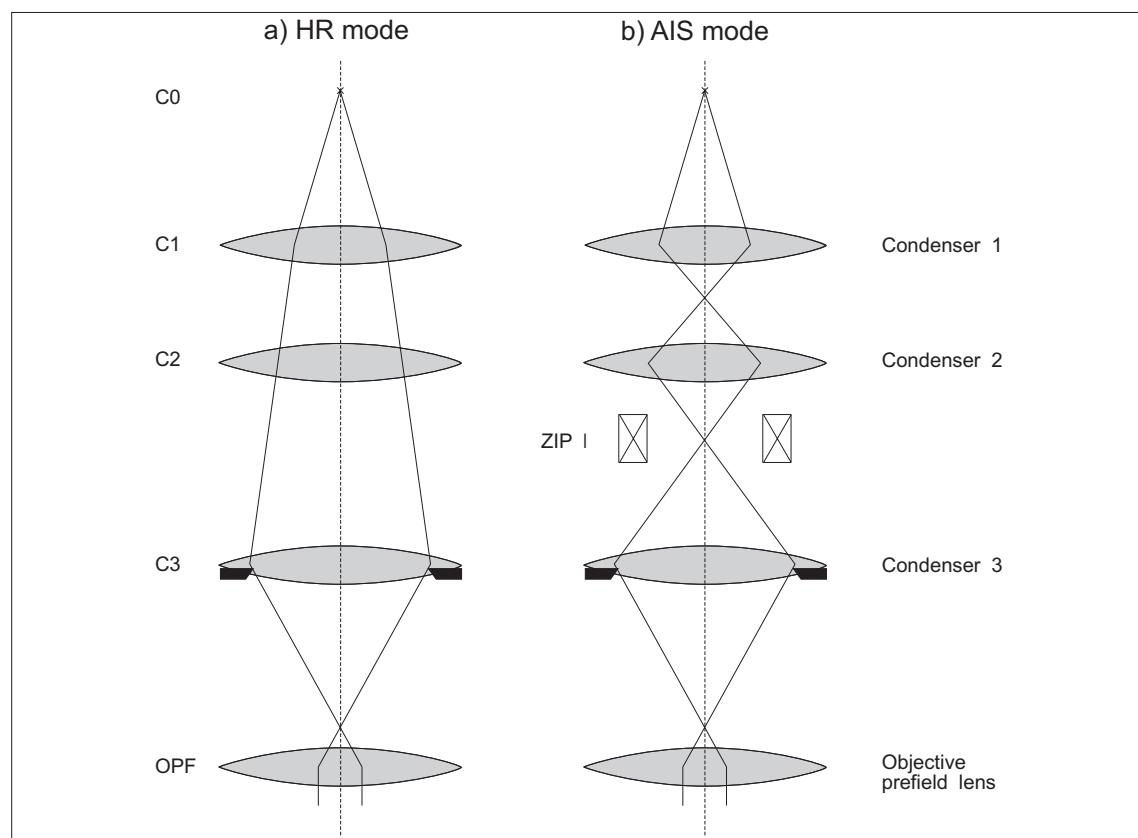


Fig.: 3 - 9 HR mode in the LIBRA 120

3.2.2 The imaging system

The imaging system includes these four principal components:

- Objective lens
- first projector lens system
- Omega energy filter
- second projector lens system

3.2.2.1 Objective lens

The objective lens according to Riecke/Ruska is a condenser/objective single-field lens with the specimen plane in its geometric center. The lens has two functions:

- The upper part, the objective prefield lens, acts as fourth condenser lens. According to the Köhler illumination principle it provides a parallel beam to illuminate the sample in TEM mode. In SPOT mode the beam is focussed onto the sample und a convergent angle.
- The lower part is the objective lens proper. It operates at fixed excitation so that the focus is maintained during magnification change (Parfocal Imaging System).

The symmetrical design of the objective lens and the Köhler illumination provide optimised conditions for imaging, diffraction, and analysis. The specimen always remains in the geometrical center of the pole piece gap, and this specimen position is the eucentric axis of the stage as well. The objective lens can be automatically calibrated to eucentric position at the touch of a button.

There are two operating modes of the objective lens:

Short focal-length mode

- 3.0 mm focal length (high excitation of objective lens)
- Diffraction image in plane of objective lens aperture diaphragm
- Real object image in selector aperture plane
- Total magnification range: 4.000x to 500,000x

Long focal-length mode

- 90mm focal length (low excitation of objective lens)
- No real object image produced by the objective lens only
- Total magnification range: 80x to 2,000x

3.2.2.2 First projector lens system

The first projector lens system consists of 3 lenses.

The correct transmission of the first intermediate image or the diffraction image into the imaging energy filter requires a projector lens system of three independent lenses.

Features of the system in imaging mode:

- Variable magnification in the range from 80x to 500,000x.
- Imaging the back focal plane of the objective lens into the entrance pupil plane of the spectrometer.
- Imaging the first intermediate image plane into the entrance image plane of the spectrometer.

Features of the system in diffraction mode:

- Variable camera length in the range from 300mm to 800mm.
- Imaging the first intermediate image plane into the entrance pupil plane of the spectrometer.
- Imaging the diffraction pattern in the back objective lens focal plane in the entrance image plane of the spectrometer.

The entrance pupil plane (plane of the entrance crossover) and the entrance image plane of the omega filter are fixed. The first projector lens system is therefore a zoom system.

3.2.2.3 OMEGA spectrometer

The OMEGA spectrometer generates an achromatic 1:1 image of its entrance image plane into its exit image plane. All electrons from one object (or diffraction) point are focused in one image point in the achromatic plane (spatial focusing).

At the same time the OMEGA filter produces a spectrally dispersed 1:1 image of its entrance pupil into the spectrum (energy-dispersive) plane . All electrons of the same energy but from different object or diffraction points are focused in one point in the spectrum plane (energy focusing). The orbits of electrons with different energy have different directions in the achromatic image plane (directional focusing). This yields in the energy-dispersive plane an electron energy loss spectrum (EELS) of the viewed object area or diffraction pattern. Defined energy electrons can be selected from the achromatic image by a slit aperture. The width of the analyzer slit defines the energy resolution of the image.

The slit aperture position is exactly on the optical axis of the microscope. To select a specific electron energy for specimen or diffraction spectrum imaging, the primary energy (high voltage) is changed. The slit aperture stays on the microscope axis. With this method the orbit of the selected energy loss electron is always exactly axial, with constant excitation of the imaging system and energy spectrometer, and constant position of the analyzer slit.

The energy range is selected either manually by pushing a button or automatically controlled by the computer, in digital increments of 0.2eV. The excitation of the illumination system is automatically adjusted so that all illumination parameters are maintained.

The dispersion of the omega spectrometer in the slit plane is approx. 1mm/eV at 120keV electron energy.

3.2.2.4 Second projector lens group

The second projector lens group consists of two lenses. At variable magnification it images either the achromatic image plane or the spectrum plane on the recording system of the LIBRA 120. This enhances considerably the imaging and analysis capability of the energy-filter TEM, compared with conventional TEMs.

3.2.2.5 Operating modes

The imaging and diffraction modes are displayed in Fig. 3-10. In LOW MAG mode the objective lens is weakly excited, in MAG and HIGH MAG modes it is highly excited.

Imaging or diffraction mode is adjusted by the first projector lens system, and the corresponding image or diffraction pattern transmitted to the omega filter. The second projector lens system sets either the filter imaging or spectrum mode, and transmits the energy-filtered image (or diffraction pattern) or its EEL spectrum to the recording system. The correlation is shown in Fig. 3-13.

3.2.2.6 Lens programs

There is a lens program for each step of the adjustable accelerating voltage (80 and 120kV). The user cannot change the optimized programs.

With the option FREE LENS CONTROL the user can set up max. four imaging programs, e.g. copy one of the given lens programs and change it to suit his own requirements.

| Imaging mode | | | |
|--|-----------------------------------|--|--|
| LM mode 80x - 2 000x | M mode 4000x 25 000x | M mode 31 500x - 250 000x | HM mode 315 000x - 500 000x |
| 15 steps | 10 steps | 10 steps | 3 steps |
| Diffraction mode | | | |
| M mode CL: 144 mm - 3600 mm | | LM mode CL: 10 m | |

Fig.: 3 - 10 Imaging and diffraction modes

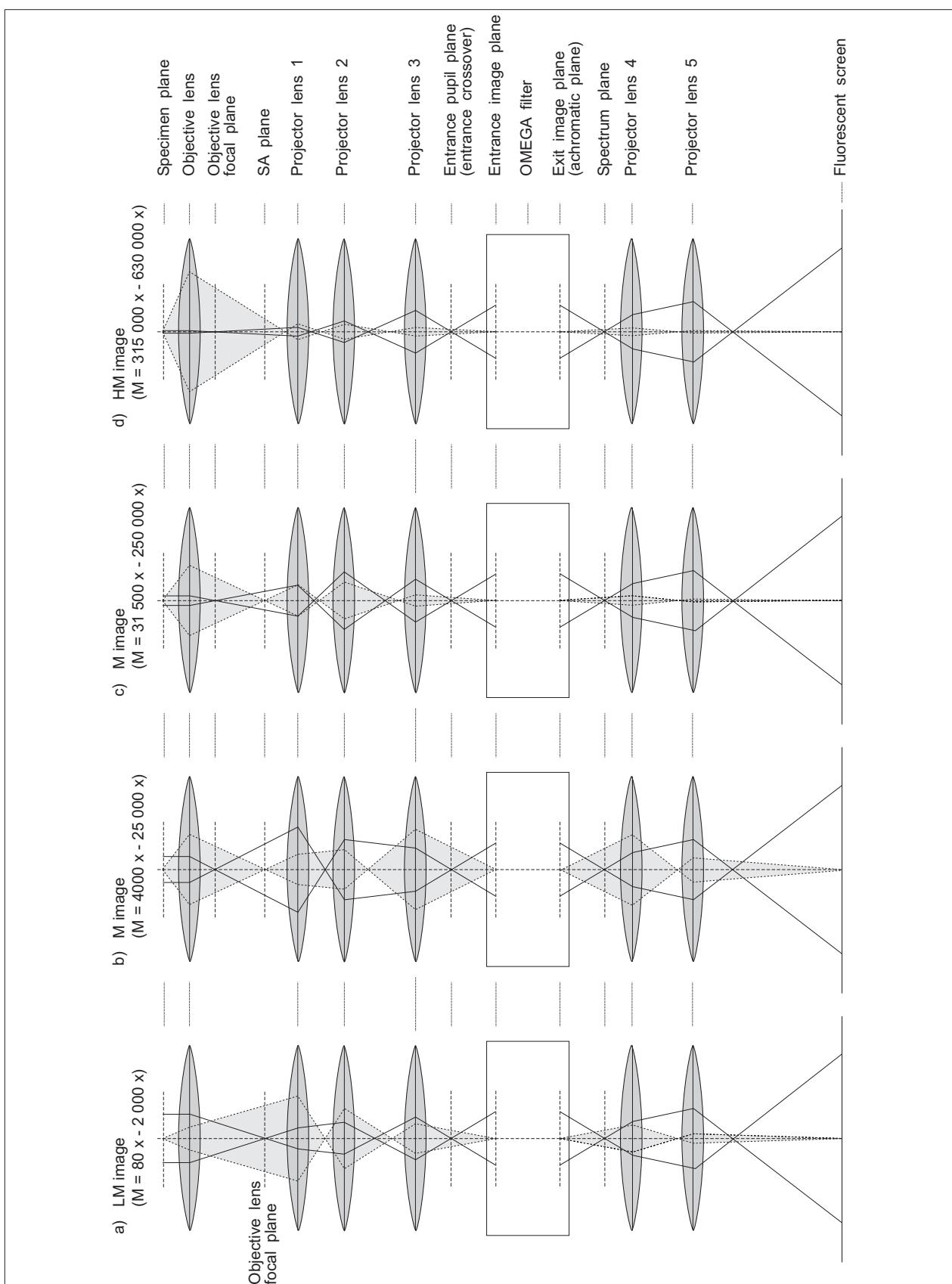


Fig.: 3 - 11 Beam paths of imaging systems in MAG mode

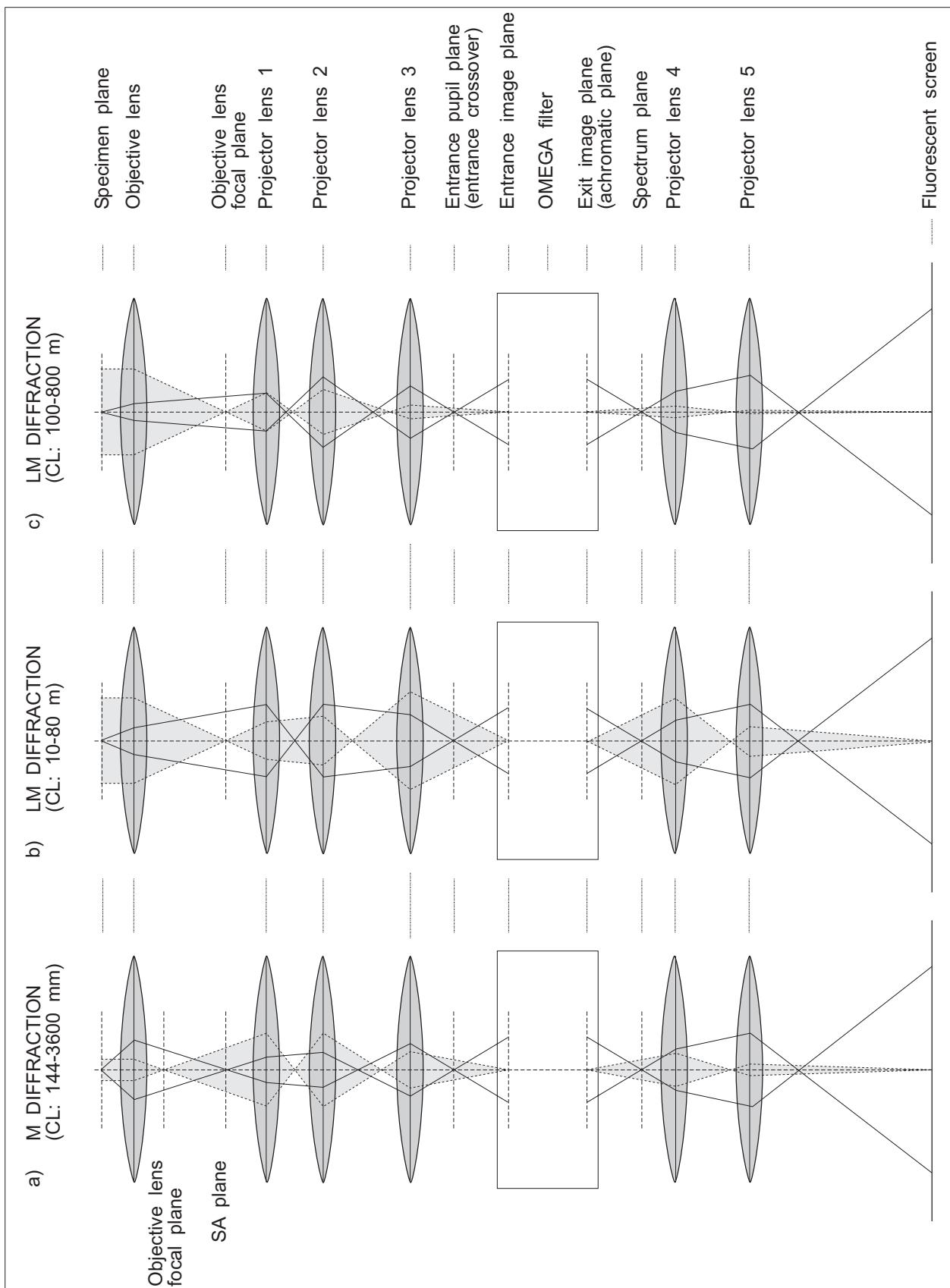


Fig.: 3 - 12 Beam paths of imaging systems in LOWMAG mode

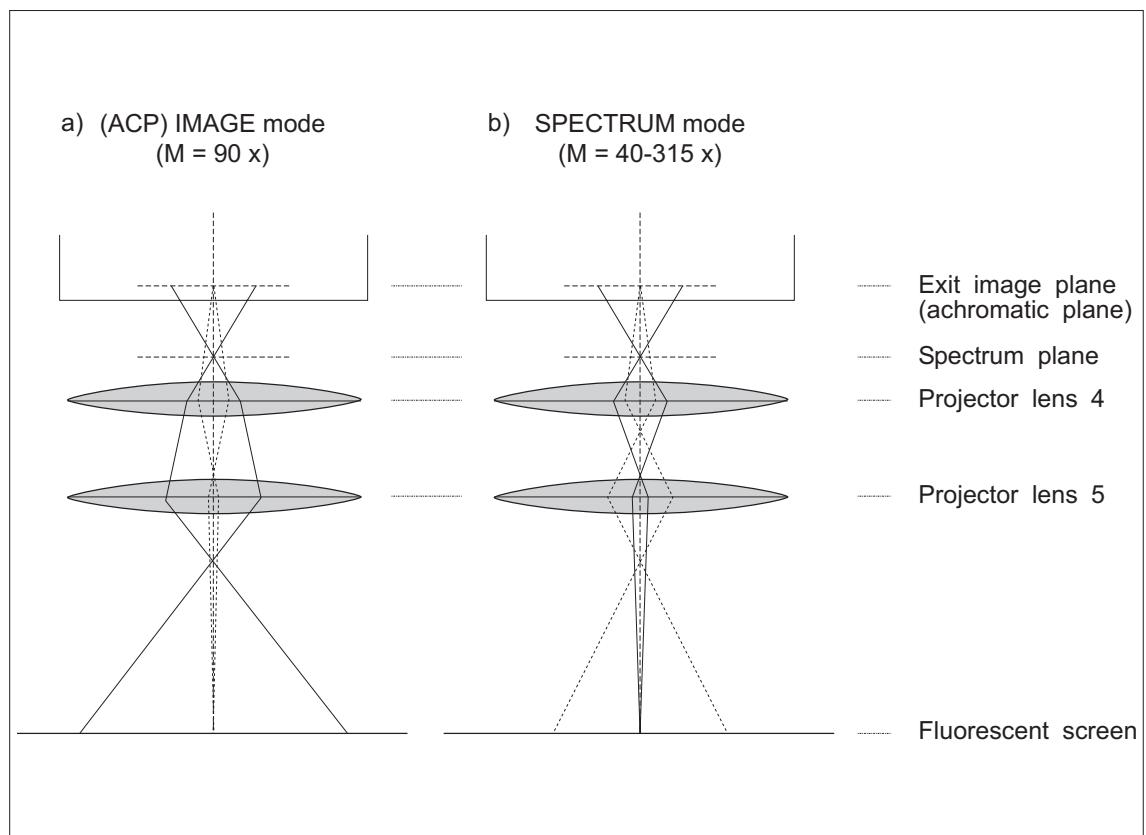


Fig.: 3 - 13 Beam paths in 2nd projector lens system

3.3 Vacuum system

The differential pump system of the LIBRA 120 provides an oil-free vacuum pumped by a scroll pump and a split-flow turbomolecular pump (TMP). An integrated Nitrogen cryotrap in the pumping column provides a vacuum in the 10^{-8} mbar range.

The column is divided in 2 vacuum chambers by valve V3. A differential pressure stage aperture maintains the different vacuum ranges ($10^{-8} / 10^{-5}$ mbar). The upper part of the column is evacuated by the high pressure stage of the TMP and the lower part (camera chamber) is pumped by the low pressure stage of the TMP. The valve V3 is closed, when the column or the camera chamber is ventilated.

In the optional double differential pump system an additional Ion-Getter-Pump (IGP) is evacuating the gun chamber (see next page). The gun is separated from the column by valve V9.

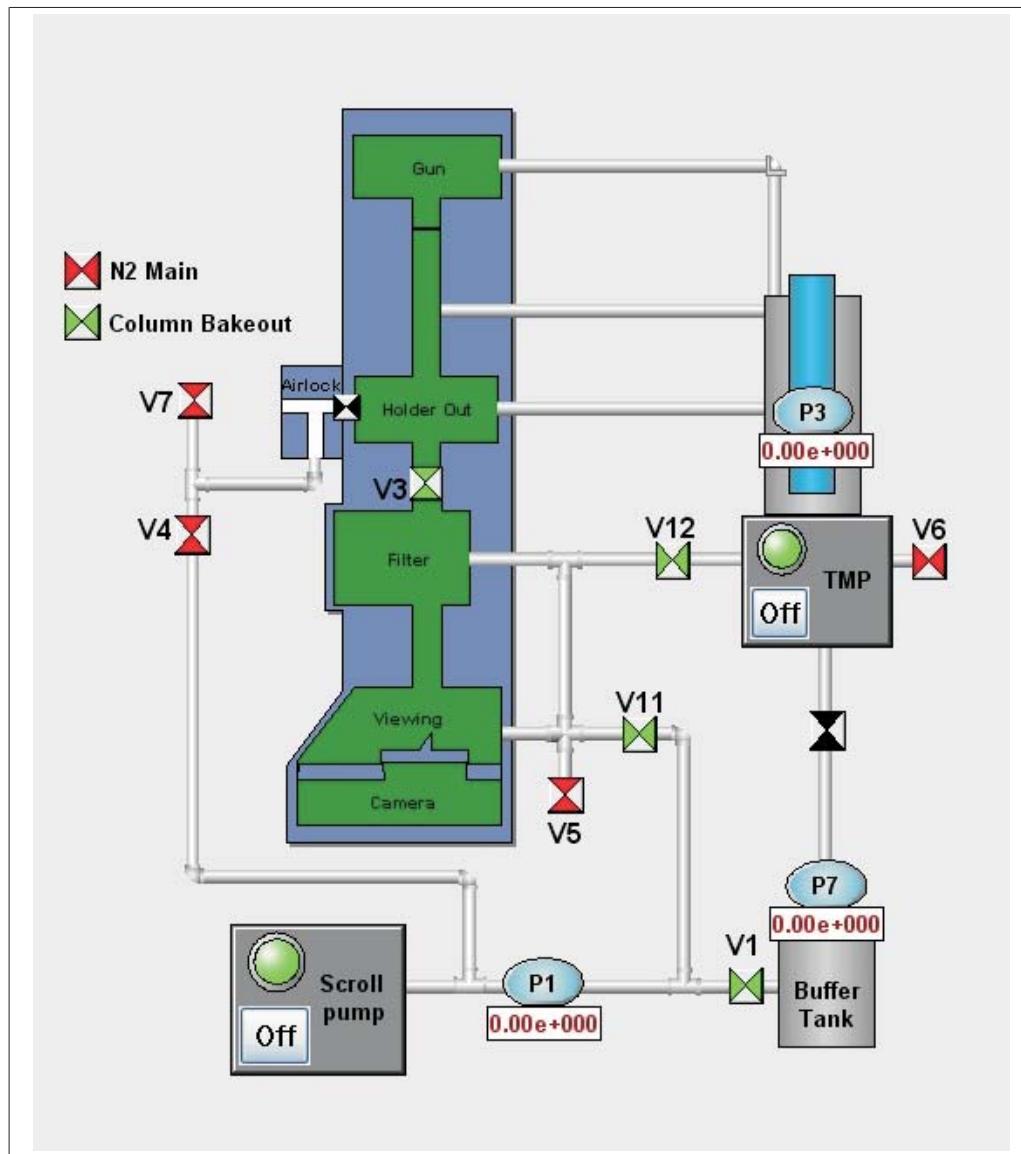


Fig.: 3 - 14 Standard Vacuum System LIBRA 120

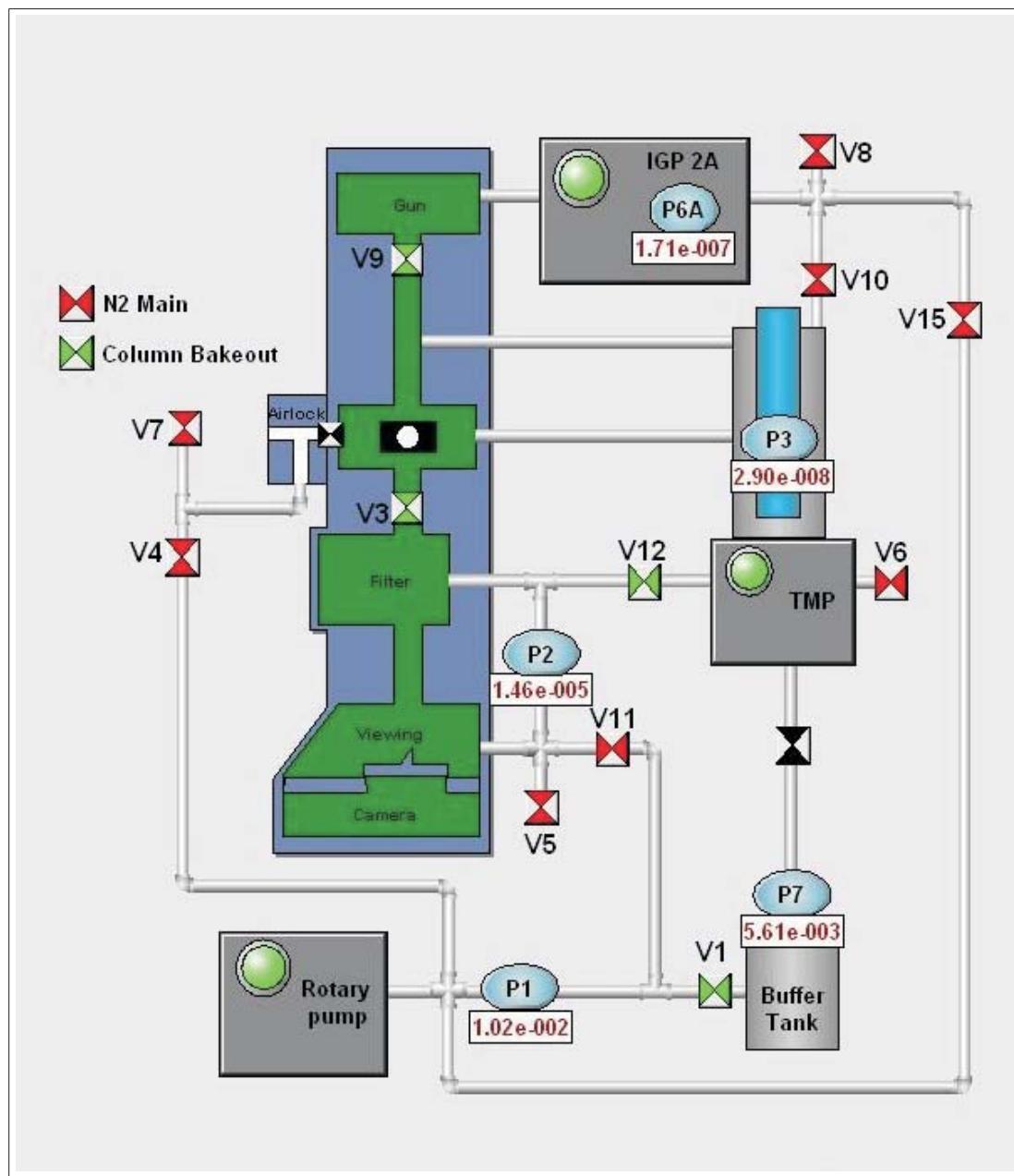


Fig.: 3 - 15 Extended Vacuum System LIBRA 120

Scroll pump

- produces the prevacuum for the Turbo-molecular-pump.
- evacuates the airlock to insert the specimen holder.

Buffer tank

- allows an intermittent pumping mode for the scroll pump.

CT Cryotrap

- cooling trap in the pump column.
- improves the vacuum for LaB6 operation.
- improves the vacuum conditions for the sample.

TMP Turbomolecular pump

- evacuates the column between V3 and V9 with its high compression stage.
- evacuates the the camera chamber below V3 with its low compression stage.
- provides the start vacuum for the IGP pump.

IGP Ion Getter Pump

- provides the High Vacuum for the Gun.
- Operating principle: Titanium evaporation.

V1 Angle valve between Scroll pump and Buffer tank

- Is opened to evacuate the buffer tank when the high pressure threshold is reached.

V2 Manual valve between Buffer tank and Turbo pump

- Is closed for transport of the EM.

V3 Valve between Column and Viewing chamber

- Is closed to ventilate the column or the camera chamber.
- Is opened to illuminate the viewing screen.

V4 Valve between Scroll pump and Airlock

- Is opened to evacuate the airlock to lock in the specimen holder.

V5 Ventilation valve for the Camera chamber

- Is opened to ventilate the camera chamber.
- Sheet films can be loaded.

V7 Valve to flush the airlock with Nitrogen gas

- Prevents the airlock from contamination.

V8 Ventilation valve for the Gun

- To lift the Gun head for filament exchange.

V9 Separation valve between Gun and Column

- Is closed to ventilate the Gun or the Column.
- Is opened to illuminate the viewing screen.

V10 Bypass valve between Turbo pump and IGP

- Is opened to support the Gun vacuum.

V11 Angle valve between Scroll pump and Camera chamber

- Is opened to evacuate the camera chamber after sheetfilm loading.

V12 Angle valve between Turbo pump and Camera chamber

- Is opened to improve the camera vacuum after evacuation by the scroll pump.

3.4 Goniometer

The goniometer integrated in the LIBRA 120 is of the eucentric side entry type with the following base components:

- Specimen stage with specimen shift
- Specimen tilt and
- Specimen holder

Four axes of the standard equipment are motorized for specimen manipulation:

- X-axis
- Y-axis
- Z-axis (height adjustment to position the specimen to the eucentric axis and for coarse focusing)
- Θ axis for specimen tilt about the eucentric axis

Optional specimen rods allow rotation or tilt of the specimen in an additional axis besides the aforementioned.

Two specimens can be loaded in the standard rod and one each brought in the beam path with the specimen changer. Special rods with specimen holders made of graphite or beryllium (see chapter 2, Safety precautions) reduce the background signals in EDX analysis. The goniometer movement in X and Y direction is controlled by a joystick. The tilt function is performed by push buttons on the left hardpanel.

Specimen positions for all axes can be stored and recalled in the GONIOMETER point list.

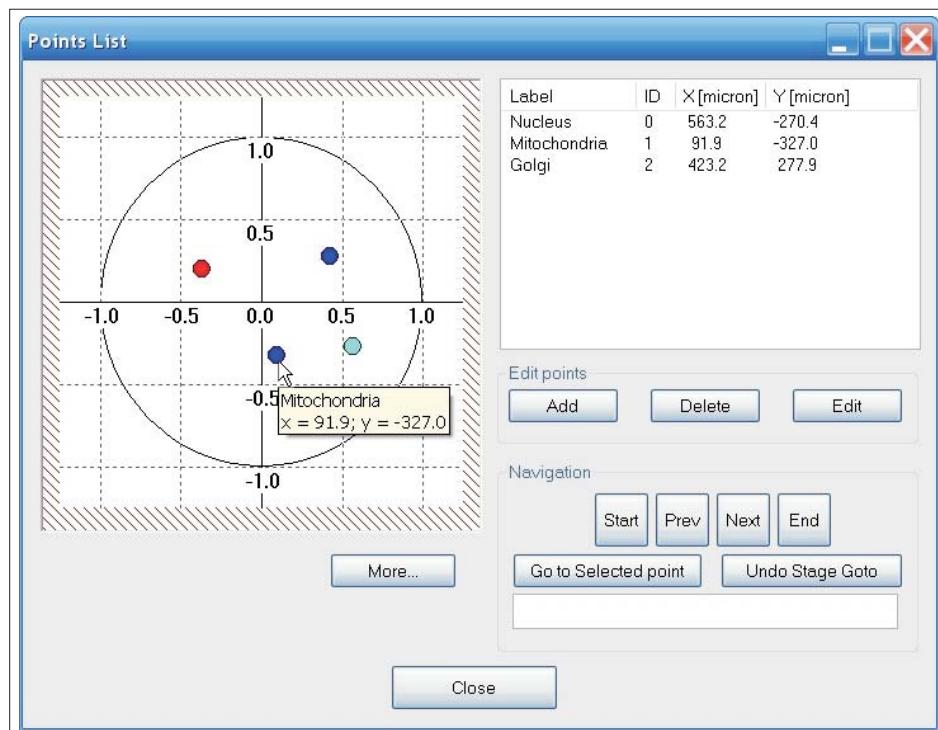


Fig: 3 - 16 Goniometer points list

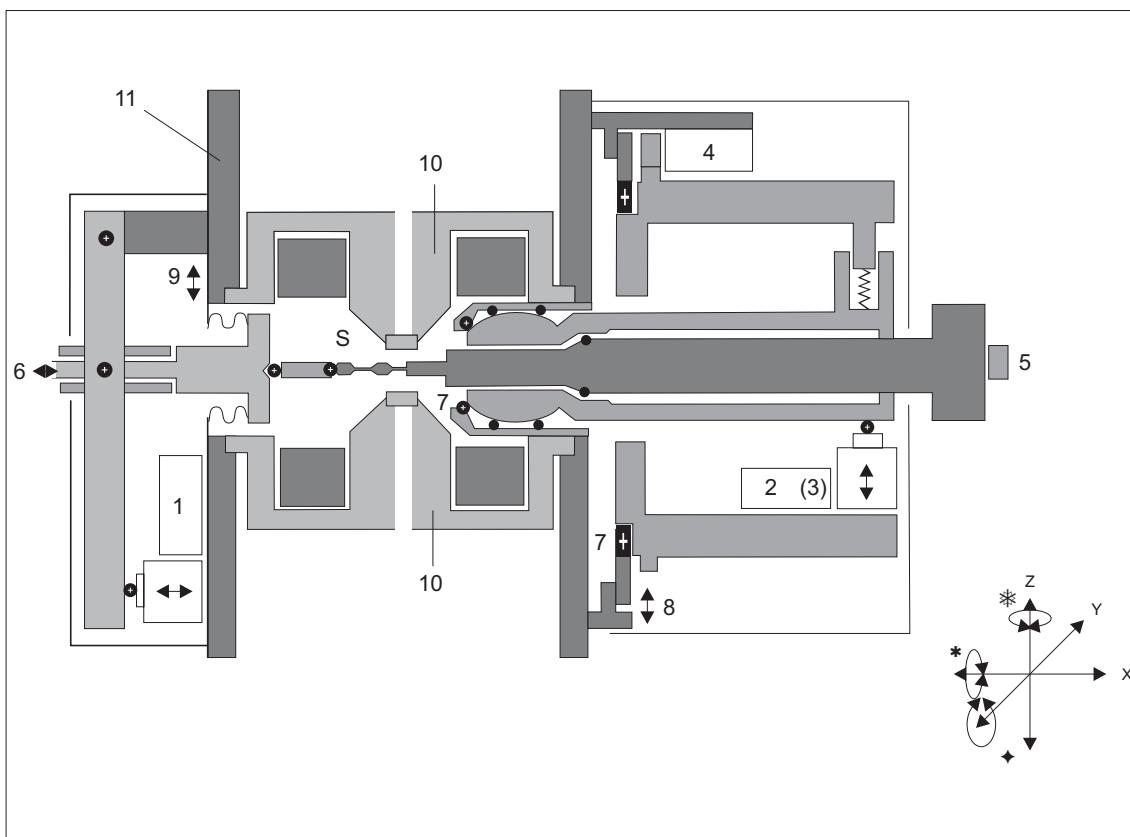


Fig.: 3 - 17

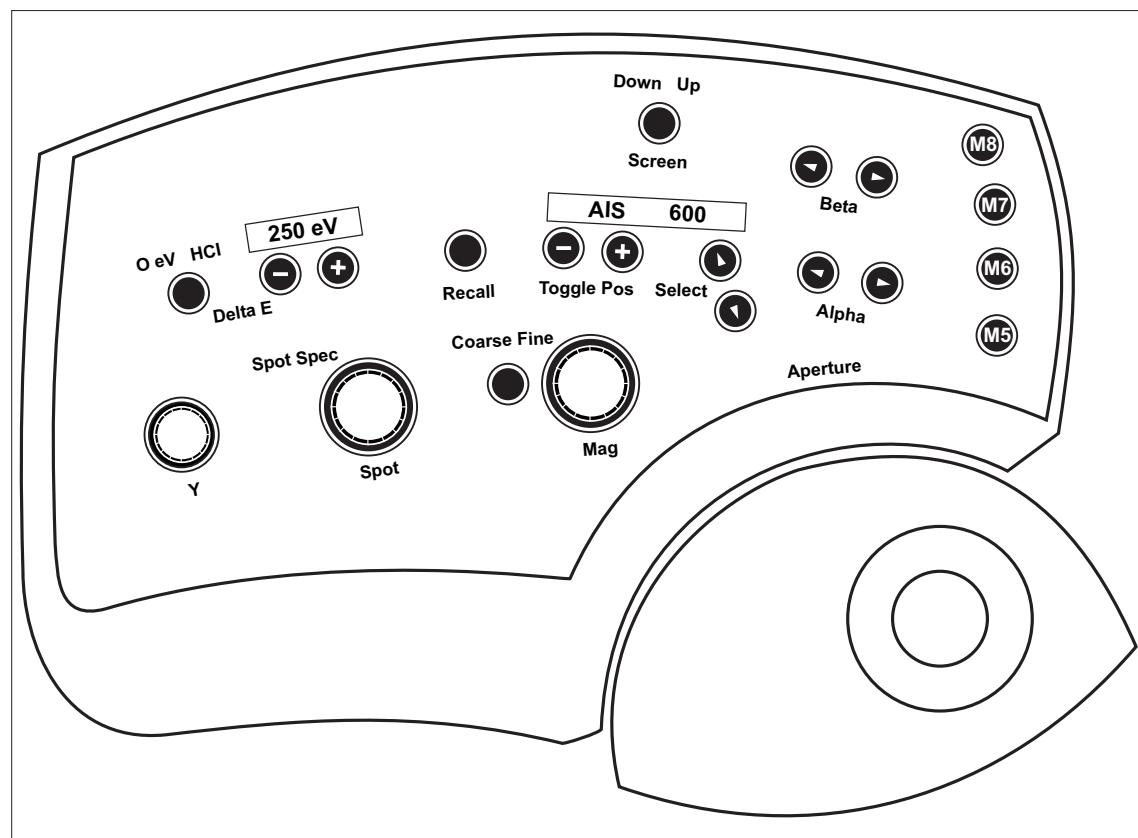
- S Specimen position (2 specimens)
- 1 Motor drive for X shift
- 2 Motor drive for Z shift
- 3 Motor drive for Y shift (not visible)
- 4 Motor drive for eucentric tilt Θ
- 5 Motor drive for optional rod (2nd tilt axis for specimen rotation ε)
- 6 Specimen changer for 2nd specimen position
- 7 Bearing for eucentric tilt
- 8 Mechanical (factory) alignment of eucentric axis
- 9 Mechanical (factory) alignment of counterbearing
- 10 Objective lens
- 11 Column

3.5 Control Panels

The hardpanels are the interface between the user and the microscope. They can be moved on the desk to provide the best ergonomic position. The user is able to turn knobs and to push buttons as in previous microscope generations. The difference is that this "analogous" operation is digitally controlled.

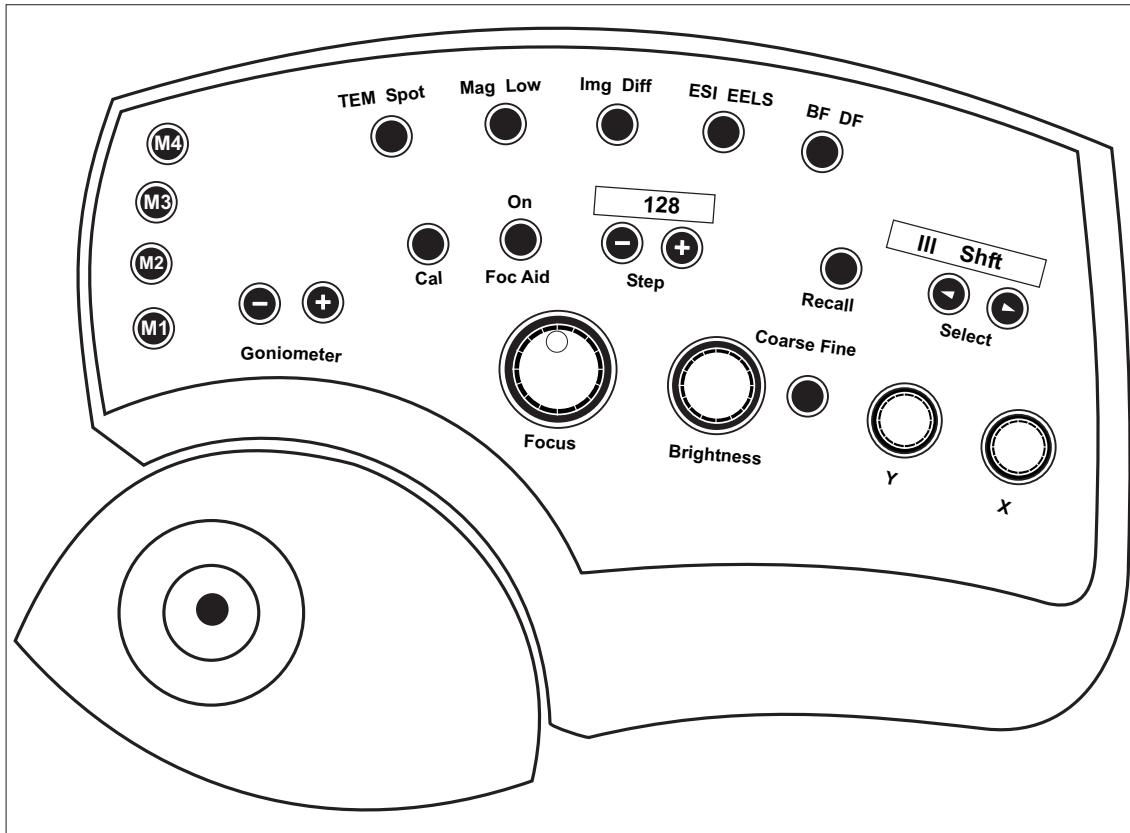
In addition push buttons as M1 to M8 can be used to start macro functions. The default assignment of the macro buttons can be modified by the user and adapted to his needs.

Left hand side hardpanel



The main button on the left Hardpanel is the **Mag** knob. Further buttons are used for goniometer operation, screen lift, spot focus, energy loss settings and MIS aperture selection. The macro buttons are assigned as followed:

- M 1: User Beam Blank On/Off**
- M 2: not assigned**
- M 3: not assigned**
- M 4: Image Shift on Trackerball**
- M 5: Image Shift on Trackerball**
- M 6: Mag Display on the Left Hardpanel**
- M 7: Overview Mode On/Off**
- M 8: Large Screen Up/Down**

Right hand side hardpanel

The right hardpanel provides all illumination and imaging mode knobs. The focus and brightness is controlled from this panel as well. Further on the goniometer with its specimen holder can be moved in X, Y and Z direction.

3.6 Electron optical column

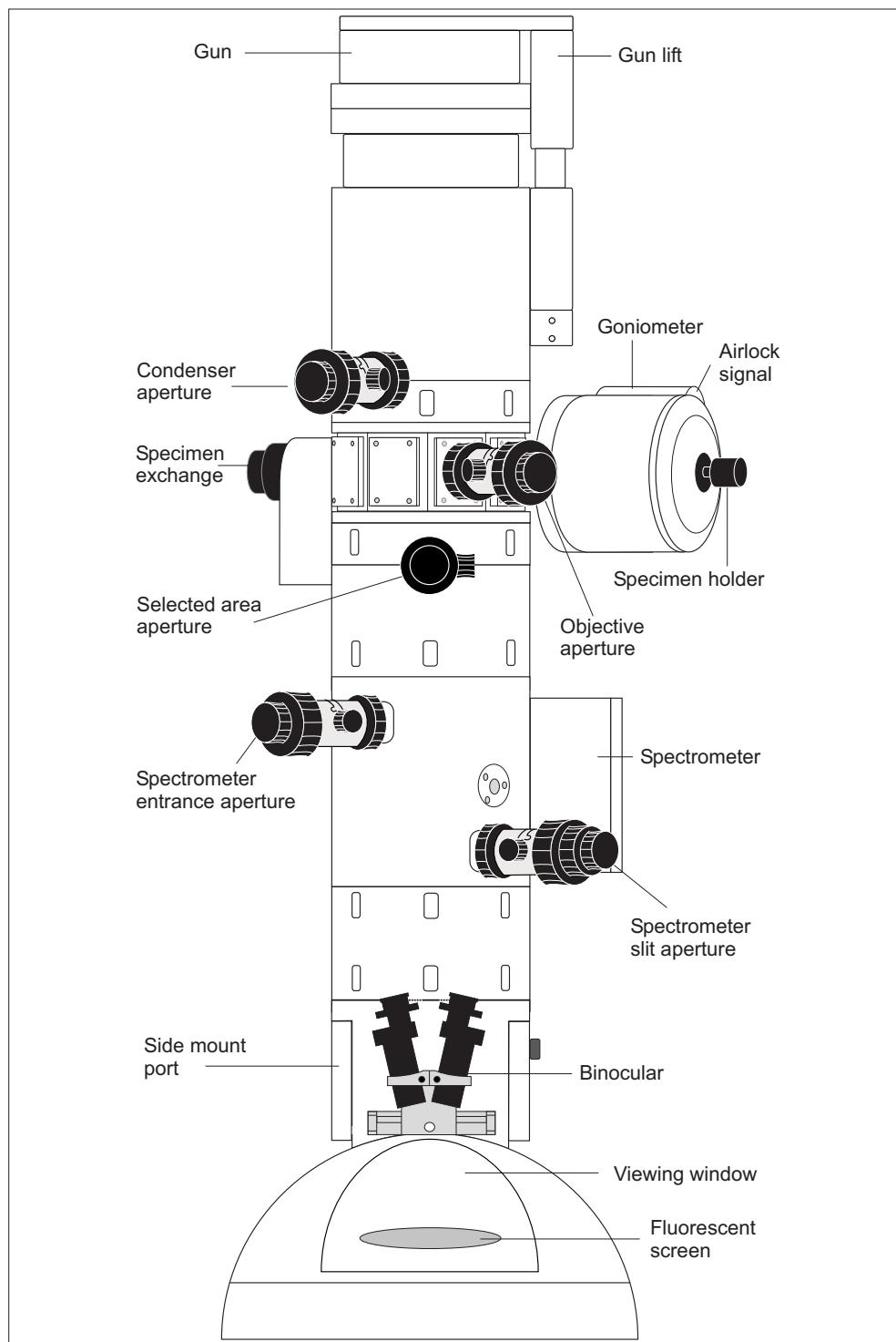


Fig.: 3 - 18 Electron - optical column

Airlock signal lamp

(not visible)

During specimen lock-in/out it displays non-attained prevacuum for lock-in.

| | |
|--------------------------------|--|
| Condenser aperture | (Luminous field aperture) Aperture holder: Position 1: empty Position 2: 400 µm Position 3: Central hole of the multi-hole aperture 37 µm aperture (for the 5 hole AIS aperture) or 800 µm aperture (for Bio AIS aperture) |
| Objective lens aperture | (Contrast aperture) Aperture holder: Position 1: 90 µm Position 2: 60 µm Position 3: 30 µm |
| Selector aperture | (also selected-area or intermediate-image aperture)(Option) Aperture holder: Position 1: 400 µm Position 2: 200 µm Position 3: 50 µm |
| Goniometer | to move the specimen. |
| Specimen rod | to accommodate max. 2 different specimens. |
| Specimen exchange | 2 positions Large knurled knob to coarsely shift the specimen holder to either of the two positions. Small knurled knob to secure in the corresponding position. |
| Gun | If the column is ventilated it can be swung out after pushing the key on the gun lift, e.g. for filament exchange. |
| Gun lift | (pneumatic) |
| Spectrometer | Omega electron-energy filter Elimination of inelastically scattered electrons by elastic filtering by the omega filter. The spectrometer consists of four magnetic prisms and one hexapole/quadrupole corrector in the symmetry plane. |
| Spectrometer entrance aperture | Assignment of the aperture holder: Position 1: 1000 µm Position 2: 650 µm Position 3: 100 µm |

Slit aperture for slit selection and adjustment

Positions: 6 positions in longitudinal direction, to use in case of contaminated edges by aperture shifting to an unused area of the slit)

Adjustment of slit in longitudinal direction and alignment to the energy axis are possible

Variation of slit width

Binocular

Magnification 9x

Diopter adjustment for ametropic users separately for each eyepiece.

Large fluorescent screen

can be raised for sheetfilm photography or SSCCD camera acquisition.

Small fluorescent screen

used for focusing with the binocular (lowered position).

Side mount port

to install a TV camera and/or a SSCCD camera.

3.6.1 Aperture drives

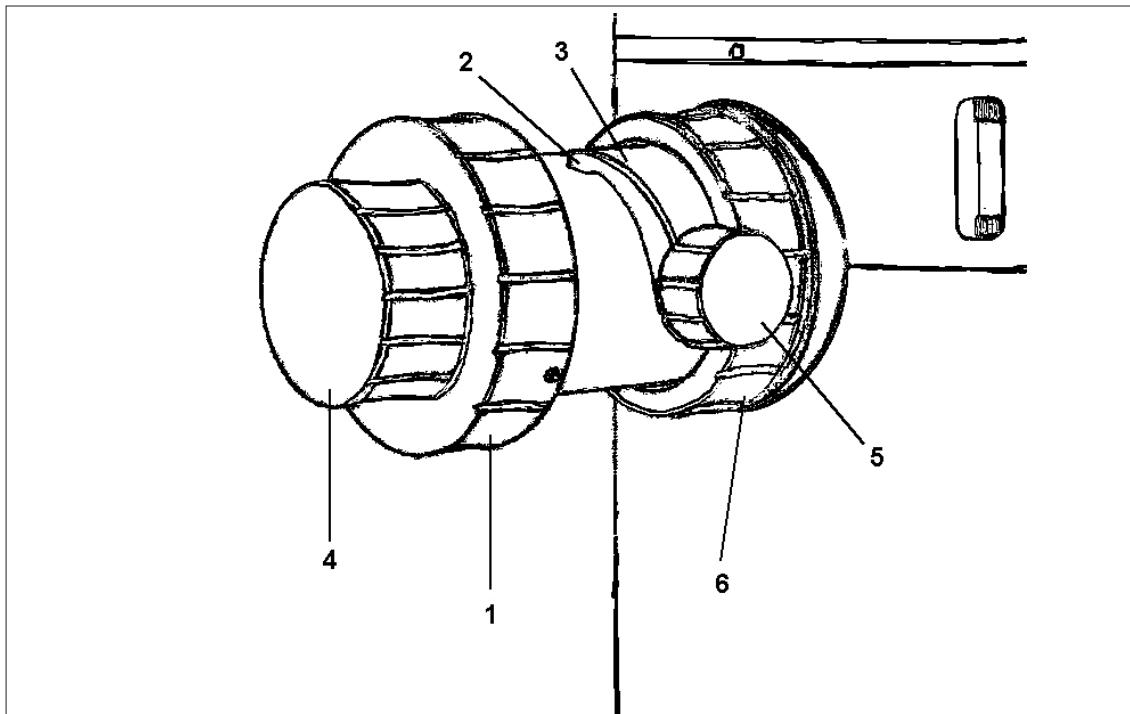


Fig.: 3 - 19 Aperture drive (except for slit aperture)

The aperture drives serve to select an aperture quickly and to adjust the aperture precisely:

- Condenser aperture
- Objective lens aperture
- Selector aperture
- Spectrometer entrance aperture

The 4 aperture drives are of the same mechanical design:

- With the large knurled knob (1, Fig. 3-19) on the face of the drive the aperture holder is axially shifted coarsely to the individual aperture positions. The drive engages several notches (3) on the inner edge of guide (2), which defines the different aperture positions.
Knurled knob turned fully counterclockwise: aperture not in beam path.
- The small knurled knobs on face (4) and side of drive (5) are used like micrometer screws for the precision shift of the aperture to the corresponding aperture positions (e.g. for centering).
Knurled knob (4) on the face shifts the aperture image on the fluorescent screen from left to right; knob (5) at the side shifts it up and down.
- The knurled ring on the column (6) acts as retaining ring which secures the aperture drive to the column.

3.6.2 Slit aperture drive

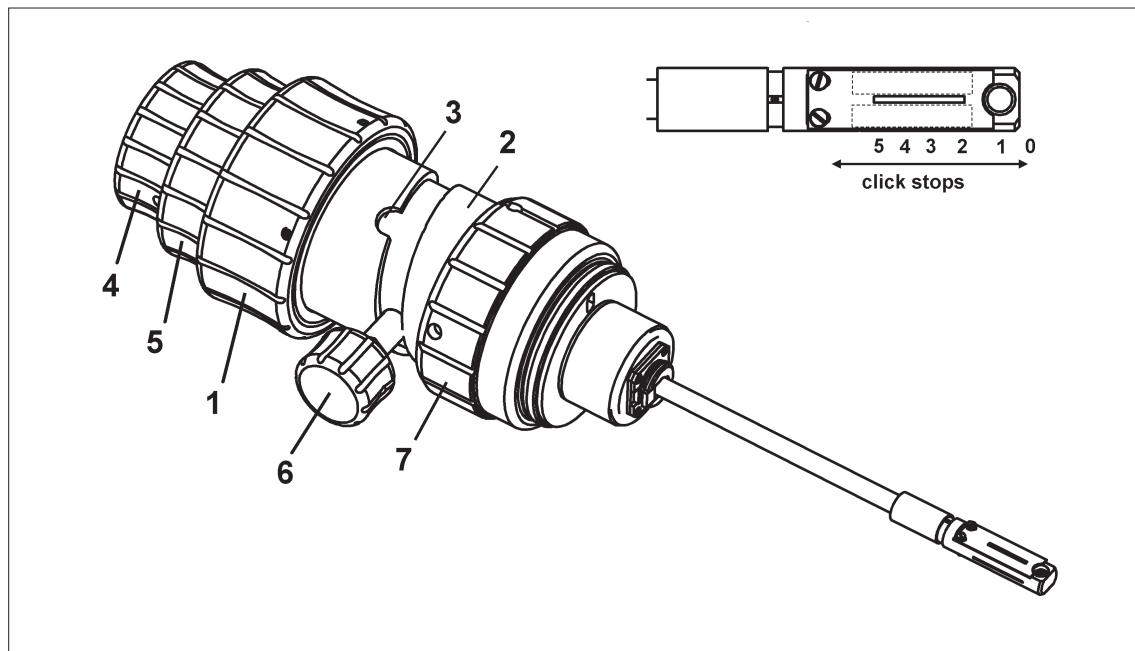


Fig.: 3 - 20 Variable slit aperture drive

With the slit aperture drive you select the slit area and adjust the slit aperture. The mechanical design is described below, the possible adjustments are shown in Fig. 3-20.

With the large knurled knob (1) on the front of the drive the aperture holder can be shifted longitudinally in 6 positions. Notches (3) inside guide (2) let the drive snap in at the individual position. See the drawing in the upper right corner.

With the small knurled knob (4) on the front the aperture is adjusted in longitudinal direction.

Knurled knob (5) in the middle serves to vary the slit width of the aperture.

The slit is adjusted in the energy axis with the small knurled knob (6) at the side of the drive.

Knurled ring (7) on the column is a retaining ring which secures the slit aperture drive to the column.

4 WinTEM Software

4.1 Introduction

The Transmission Electron Microscope LIBRA 120 is a fully computer controlled instrument. All operations, settings and functions necessary for the handling of the microscope are controlled by use of a keyboard and a mouse.

Additionally two hardpanels can be used for operation as well. The hardpanels provide the advantage that setting and adjustment of important parameters such as magnification and focus, stigmator or beam alignment is possible using knobs (encoders).

4.2 The EM Server

The Server is the central controller of the microscope. It co-ordinates requests for data and control actions from other applications which it routes to low level control modules and also notifies all relevant applications of changes in the microscope.

The server must therefore remain active whenever the microscope is in use.

The server is also responsible for performing the log on function when an interface application is started. On validation of the log on it establishes the operating environment for the specific user. In conjunction with this the Server also provides the User Preferences dialog.

The server maintains a window for the display of messages and a log file.

On request, or when all registered interface applications have closed, the server performs the log off function..

If you explicitly attempt to close the Server it will request permission from all the currently registered interface applications. Only if all applications grant permission will the server close, it may therefore be necessary to close all interface applications before closing the server.

4.2.1 Logging On

When the first User Interface application starts the **server** requests the User to Log On.

You **Must** enter a valid Username and password. Three attempts are permitted.

On Initial installation of SmartSEM two UserNames are provided which initially have null passwords, these are **SYSTEM** and **GUEST**.

The System Supervisor should use the Administrator to define passwords for these UserNames and create new UserNames as required.

4.2.2 Message Window

The message window for the displays operational messages.

Each message has a time stamp and the messages are listed in chronological order.

The last 100 messages (since the server was started) are displayed.

4.3 The Administrator

The Administrator provides for establishing different user directories, editing existing folders and user configurations. A user directory contains frequently modified configuration parameters of the WinTEM user interface and system software files for specific users. Each specific user has his own directory for configuration parameters, toolbar, menus, data zones, operation modes, etc. Each user interface will thus load with the user specific configuration settings.

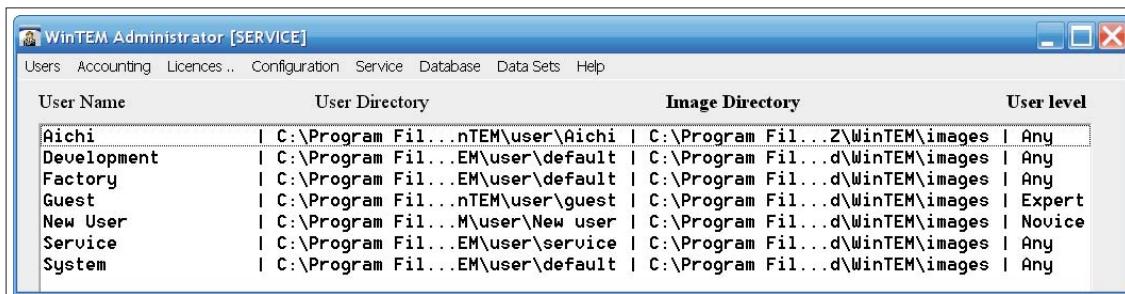
4.3.1 Installing a new user

After the installation, a new user can be installed. This should be set up by the person responsible for the system or the service engineer. The Administrator is opened using the Windows Overlay as follows:

- **START → PROGRAM → WinTEM → WinTEM ADMINISTRATOR.**

Once the program begins loading, you will be prompted to enter a name and a password. Log on is possible with the user name SERVICE and the current service password or with the user name SYSTEM. The user SYSTEM does not need any password.

- Click on **OK** to confirm.



The screenshot shows a Windows application window titled "WinTEM Administrator [SERVICE]". The menu bar includes "Users", "Accounting", "Licences ..", "Configuration", "Service", "Database", "Data Sets", and "Help". The main area is a table with four columns: "User Name", "User Directory", "Image Directory", and "User level". The data in the table is as follows:

| User Name | User Directory | Image Directory | User level |
|-------------|----------------------------------|----------------------------------|------------|
| Aichi | C:\Program Fil...nTEM\user\Aichi | C:\Program Fil...Z\WinTEM\images | Any |
| Development | C:\Program Fil...EM\user\default | C:\Program Fil...d\WinTEM\images | Any |
| Factory | C:\Program Fil...EM\user\default | C:\Program Fil...d\WinTEM\images | Any |
| Guest | C:\Program Fil...nTEM\user\guest | C:\Program Fil...d\WinTEM\images | Expert |
| New User | C:\Program Fil...M\user>New user | C:\Program Fil...d\WinTEM\images | Novice |
| Service | C:\Program Fil...EM\user\service | C:\Program Fil...d\WinTEM\images | Any |
| System | C:\Program Fil...EM\user\default | C:\Program Fil...d\WinTEM\images | Any |

Fig.: 4 - 1 List of the installed users

- Open PD menu *Users* and select *New*.
 - The window *Creating a New User Profile* is opened.
- Type in the new user name, e. g. *Microscopist*.
- Click in the input field of the *User Directory*.
 - A new window is opened to define the path for a new user directory.
- Replace the default user by e. g. *Microscopist* in the path line.
- Click on *Create Directory*, if the desired directory does not exist in the Windows Explorer.
- Click on **OK**.
 - The new path is displayed in the input line of the *User Directory*.

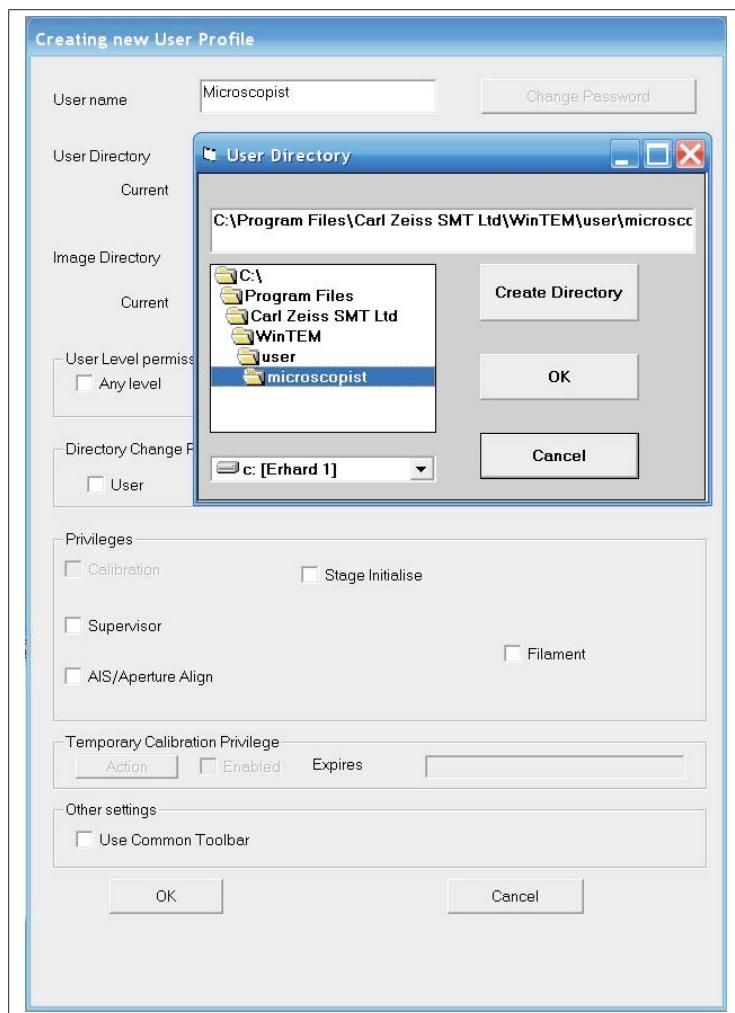


Fig.: 4 - 2 The User Profile Window

- Click in the input field of the *Image Directory* path and proceed in the same way as for the user path.
- Close the Window *Create a new User Profile* with *OK*.
 - The *Microscopist* user is listed.

| User Name | User Directory | Image Directory | User level |
|--------------|----------------------------------|----------------------------------|------------|
| Aichi | C:\Program Fil...nTEM\user\Aichi | C:\Program Fil...Z\WinTEM\images | Any |
| Development | C:\Program Fil...EM\user\default | C:\Program Fil...d\WinTEM\images | Any |
| Factory | C:\Program Fil...EM\user\default | C:\Program Fil...d\WinTEM\images | Any |
| Guest | C:\Program Fil...nTEM\user\guest | C:\Program Fil...d\WinTEM\images | Expert |
| Microscopist | C:\Program Fil...er\microscopist | C:\Program Fil...d\WinTEM\images | Expert |
| New User | C:\Program Fil...M\user\New user | C:\Program Fil...d\WinTEM\images | Novice |
| Service | C:\Program Fil...EM\user\service | C:\Program Fil...d\WinTEM\images | Any |
| System | C:\Program Fil...EM\user\default | C:\Program Fil...d\WinTEM\images | Any |

Fig.: 4 - 3 Updated list of the installed users

- Open the PD menu *User* again and *Edit* the new user.
 - The window *Editing User Profile* is displayed.

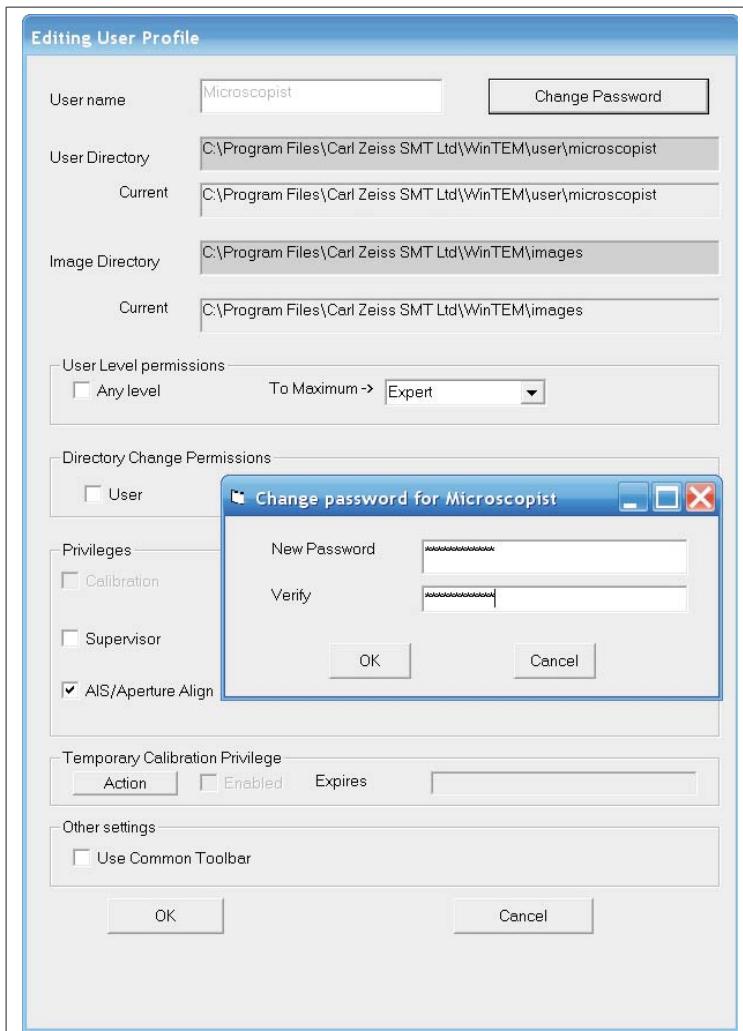


Fig.:4 - 4 Window Editing User Profile

- Click on *Change Password* and edit a password for this user.
- Quit with *OK* and close the window *Editing User Profile* with *OK*.

CAUTION:

After Installation of the first user a password should be created for the **SYSTEM** user. This should only be known to the Administrator. In case of a password loss it can be set up by the ZEISS service personal.



4.4 Customising the WinTEM GUI

4.4.1 Introduction

This chapter describes the new WinTEM GUI. The WinTEM is highly configurable to suit the needs of the individual user. This has introduced some extra complexity however so we need to address each of the elements that we can configure.

Which GUI elements can be configured?

- Toolbar
- Status Bar
- Dark room display
- Hard Panel
- Macros

There are a few requirements for configuration:

- Each user has his own personalised configuration. This configuration cannot be overwritten when the software is upgraded.
- There is a Default configuration that can be selected. The user can “upgrade” or “revert back” to the Default configuration.
- A new user will have the default configuration.
- Changing configuration settings between users is easy.

The implementation of these requirements relies on the use of “default” and “user” directories, and configuration files in these directories.

Default configuration files are for instance in the directory:

<Drive>:\ Program Files\Carl Zeiss SMT Ltd\WinTEM \DISTRIB, where the exact path can vary.

User configuration files are in the directories under: ...\\WinTEM\\user. For example, if your username is “Johnny”, then the configuration files are in the sub-directory: ...\\WinTEM\\user\\Johnny.

4.4.2 Toolbar

From the toolbar the user can execute commands, toggle parameter states and execute Macros. The default toolbar for WinTEM looks like this:



The buttons become visible when the mouse hovers over them. The toolbar also displays parameters in “checked state” to indicate they are activated.

4.4.3 Toolbar configuration

In WinTEM, click on the menu **Tools > Toolbar Editor...** This will open the Toolbar editor. Click **Add Button** to create a new toolbar button.

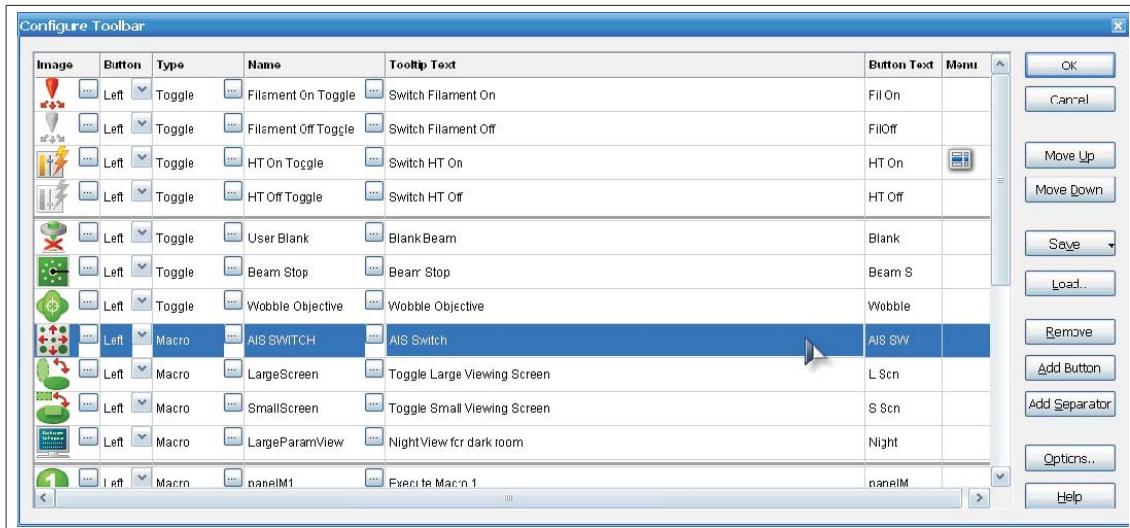


Fig.: 4 - 5 Toolbar editor in Windows XP

In the left **Image** column you can select an icon for the toolbar button. These icons are stored in the directory ...\\WinTEM\\Icons. The user can add his own icons to the toolbar too. These icons will have to be of size 32x32 and stored in the ...\\WinTEM\\Icons\\UserIcons directory.

Next step in the configuration is to click on the field in the **Type** or **Name** column. The Select Function dialog pops up. You can add types of functionality to the button. Commands, Dialogs, Macros, Parameters, Special Functions or Toggle. You will use commands, toggle parameters and Macros the most.

A command will simply execute a WinTEM command.

A toggle parameter toggles between two states: **On** and **Off**. When a state is **On** then there is an outline around the button. Macros will be discussed later. As from version 1.3 of WinTEM the user can have text displayed under the icon. The maximum number of characters is six.

4.4.4 Toolbar Files

The default toolbar set-up file for WinTEM is **defaultTEM.xml** (Distrib directory), and the current user toolbar file is **currentTEM.xml** (User directory). In the future we will have to make toolbars for different machine types, such as "FEG" or "Libra 120". These will have to be distributed and imported via the Configuration dialog.

The STEM application also has its own toolbar. The configuration files are called **current.xml** and **default.xml**. In the near future we will call them **currentSTEM.xml** and **defaultSTEM.xml**.

4.4.5 Common Toolbar

Certain users can be configured to use the “Common Toolbar”. This configuration is done in the “WinTEM Administrator” program.

Go to menu Users > Edit... (or: New...) and the “Edit (Create) User Profile” dialog box will pop up. At the bottom of this, under the heading “Other settings” you can tick the “Use Common Toolbar” checkbox. The user will now have to use a common toolbar without being able to modify her toolbar. The user will not use her own Toolbar set-up file, but a file **CommonTEM.xml** (and **CommonSTEM.xml** for STEM) in the top-level user directory, i.e. in the directory ...\\WinTEM\\user\\CommonTEM.xml.

There is no CommonTEM.xml file by default and the Toolbar Component will complain if it cannot find it. Therefore, the “System” Administrator has to provide the Common(S)TEM.xml files in the \\user directory. This is done from the Toolbar Editor button “Save > Save as Common Toolbar”.

If a user is confined to using the Common Toolbar then she cannot edit her toolbar any more. It is therefore good practice to leave at least one user (e.g. the “System” user, who is the Administrator of the microscope) not to use the Common Toolbar, so he can edit any toolbar, also the Common Toolbar.

4.4.6 Status Bar

The Status Bar in the WinTEM is displayed at the bottom of the screen. It is configured to display some important machine parameters and commands. Commands? – yes, the user can execute commands by clicking on them. Also parameters can be changed and toggle states can be set. This does not hold for read-only parameters, such as goniometer positions, which cannot be set. Disabled parameters or commands are greyed-out.



Fig.: 4 - 6 The Status Bar

The programmer can configure colours, font, font size, number of rows and number of columns. The user can configure which parameters are in the display.

4.4.7 Status Bar configuration

The user can open the configuration dialog box by right-clicking with the mouse on the status bar.

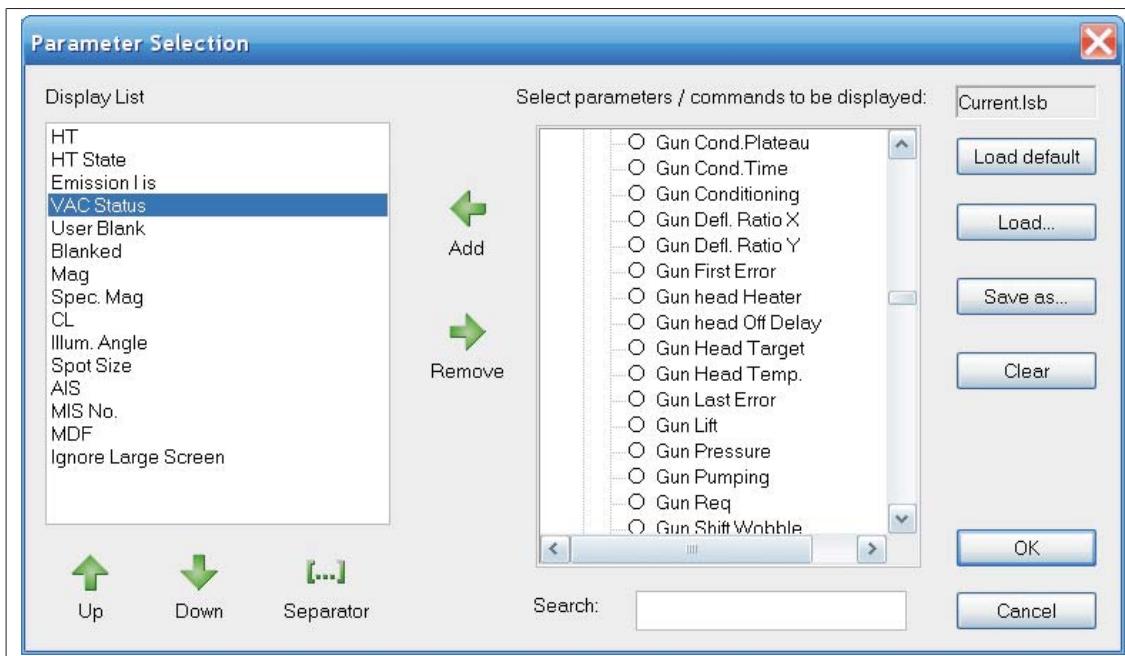


Fig.: 4 - 7 Status Bar configuration dialog box

The parameters that are going to be displayed are in the list on the left. You can list as many parameters as you like. If there are too many entries then the ones at the bottom will be ignored.

You can change the order of display by moving parameters up or down. You can also add separators, which is basically a blank space.

The list on the right displays all the parameters, states and commands you can chose from. If you know the name then you can type it in for an automatic search in the open list.

In the top-right corner of the dialog is the configuration filename displayed. You can also load the default or any other configuration file. Clicking OK will save all changes in your configuration file.

4.4.8 Status Bar files

The status bar will search for the Current.LSB file in the user directory. Alternatively it will search for it in the \Distrib directory, before using the Default.LSB from the the \Distrib directory.

4.4.9 Night View Display for the dark room



The night view display can be activated from the toolbar by clicking the button: . A black, screen-filling dialog will pop up, with some essential status information in dark red colour. If there is a second monitor this will be blacked-out completely. Note that the dark room button needs to be configured with the standard macro “LargeParamView” otherwise it will not work. Alternatively you can activate it from the menu “View > Night View” or by typing <Ctrl> + D.

Configuring the dark room display works the same as configuring the status bar. The same dialog box will appear; only the configuration data are stored in a different file.

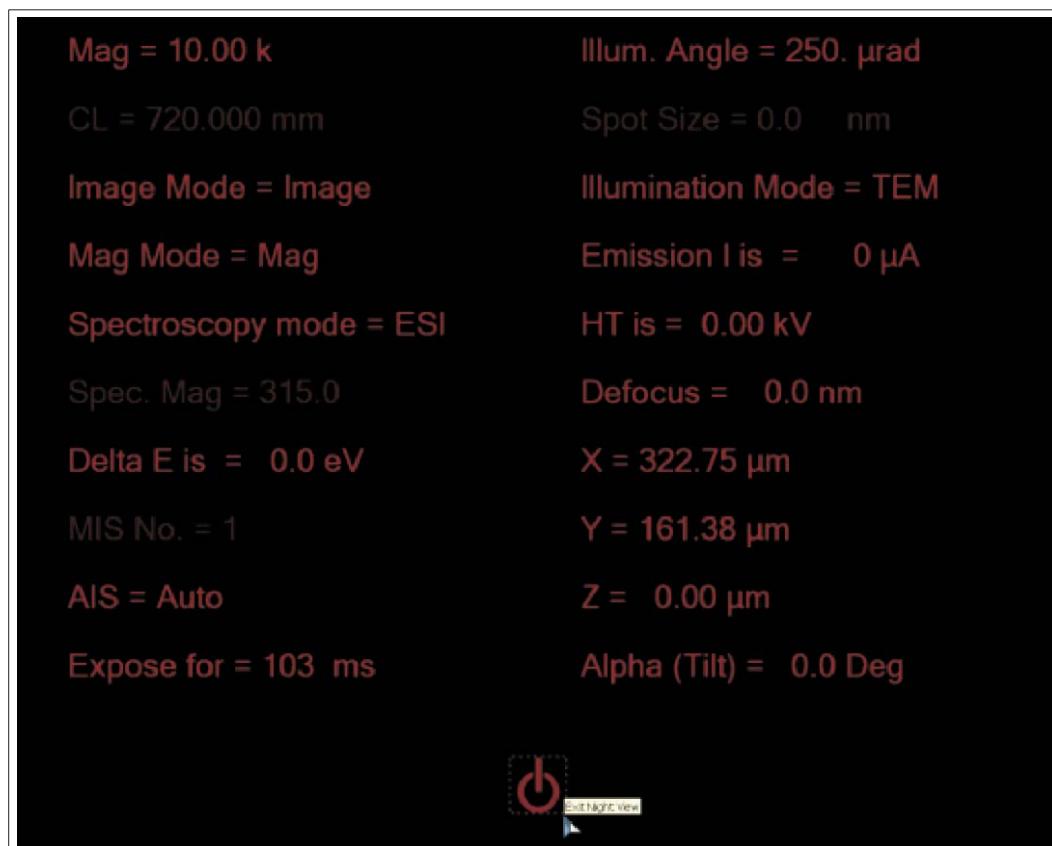


Fig.: 4 - 8 Night view display

4.4.10 Dark Room files

The configuration files for dark room display are also of the type *.LSB. The configuration file is DarkScreen.LSB. This file is in the user directory but the default one can be found in the \Distrib directory.

4.4.11 Macros

Using Macros is a very versatile way of operating the TEM. From a Macro you can execute a series of commands, use conditionals such as **if ... then ... else**, execute dialogs, set states, set parameter values.

4.4.12 Macro Editor

The Macro Editor is a stand-alone application that can be invoked from the menu **Tools > Macro Editor...**. When you open the Macro Editor it loads the Macro Library of the current user by default. In this Macro Library there are usually a number of macros already that you can use, or that are in use by the program. For instance, the LargeScreen macro is assigned to the Large Screen Up/Down button on the toolbar.

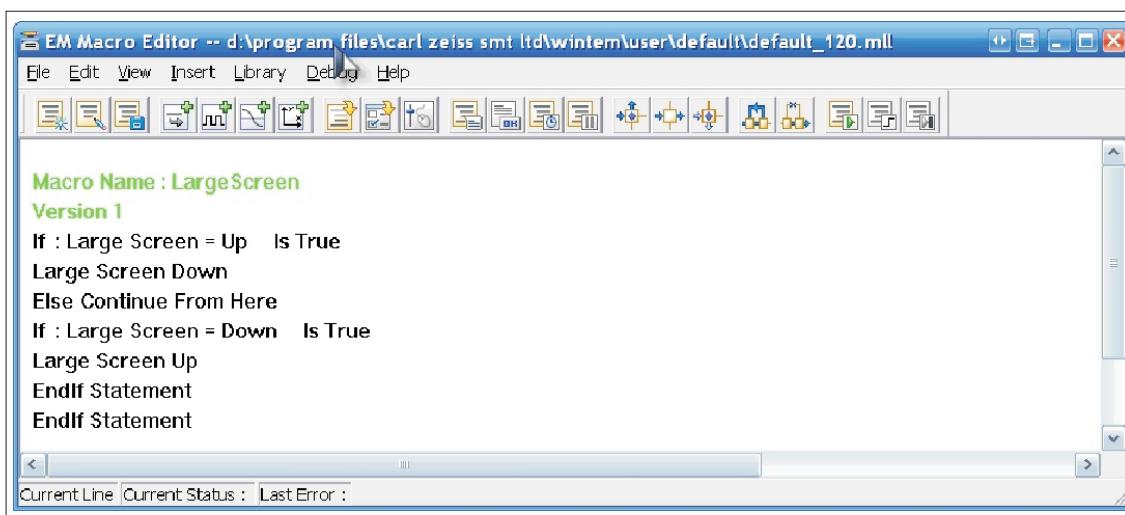


Fig.: 4 - 9 The Macro Editor Application. In the title bar is the name and path of the current macro library

Use the Insert menu to insert a line of text in the macro, be it a command, set a parameter value, invoke a decision structure or even execute a program. You cannot edit the macro text directly.

4.4.13 Macro files

The default library file that the macro editor will open is the ...\\WinTEM\\user\\current.mll. Note the *.MLL extension. New macros can be added to the library. You can also import macros from stand-alone macro files. Example LargeScreen.mlf. Note the *.MLF file extension. You can also export an existing macro from the editor to a *.MLF file.

The ...\\WinTEM\\Distrib directory contains the default macro library **default.mll**. If there are any new macros added or updated then they will be put in this file. The user who wants to have the latest version of the macro library has to go to the menu **Library > Load Standard Library**. This will replace the content of current.mll with that from default.mll. This will also erase any custom macros the user might have. To avoid erasing the custom macros you better chose the option **Library > Merge Standard Library** which adds new macros one by one to current.mll and asks if existing macros need to be replaced.

In the menu Generator > Save System Conditions... the user can save general system conditions into Macro Library Condition file (*.MLC). An MLC macro restores the system conditions into a working state. In the SEM this functionality is connected with the green traffic light button. At the moment this functionality does not work on the TEM because we need to define which system conditions actually need saving in such a macro.

4.4.14 Hardpanel

Control of the TEM via the hardpanels is an essential part of the user experience. The knobs, buttons, joystick and trackerball on the hardpanel provide certain quick-access functionality to the user. The location of these different knobs is fixed and the user will train “muscle memory” to find the right functionality.

4.4.15 X, Y knobs

In order to limit the number of knobs on the panel certain functionality is dynamically assigned to general X,Y knobs. The user can toggle between 6 often-used X,Y parameters from the Hardpanel itself. The parameter assignment works in tight collaboration with the software. If a parameter is assigned to a software navigation X,Y control (NavBox), then it is assigned to the Hardpanel as well. In the software there are a great number of these so-called NavBoxes and a great number of X,Y parameters associated with them (~35 in total) and all of these can be assigned to the Hardpanel.

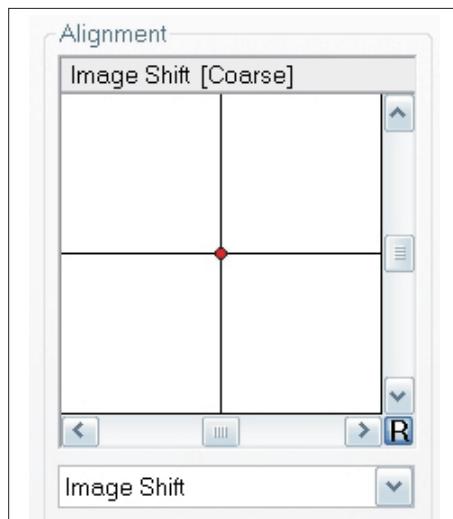


Fig.: 4 - 10 Navbox for X,Y adjust

4.4.16 Execute macros

On the Hardpanel there are 8 macro buttons, named “M1”,..., “M8”. Pressing a macro button will automatically execute a macro called **panelM1**,..., **panelM8** etc. These macros can be created or customised in the Macro editor.

4.4.17 Free configuration

It is possible to freely configure knobs, LEDs, joystick and tracker ball on the Hardpanel. This is done with macros. Normal users should not need to do this however and some assignments will be overwritten by software anyway. Free configuration goes against the principle that the user should expect the same functionality on the same location.

4.4.18 Panel Settings

The back light and sensitivity of some deflectors can be changed from the Panel Settings dialog box, accessible via menu Settings > Panel...

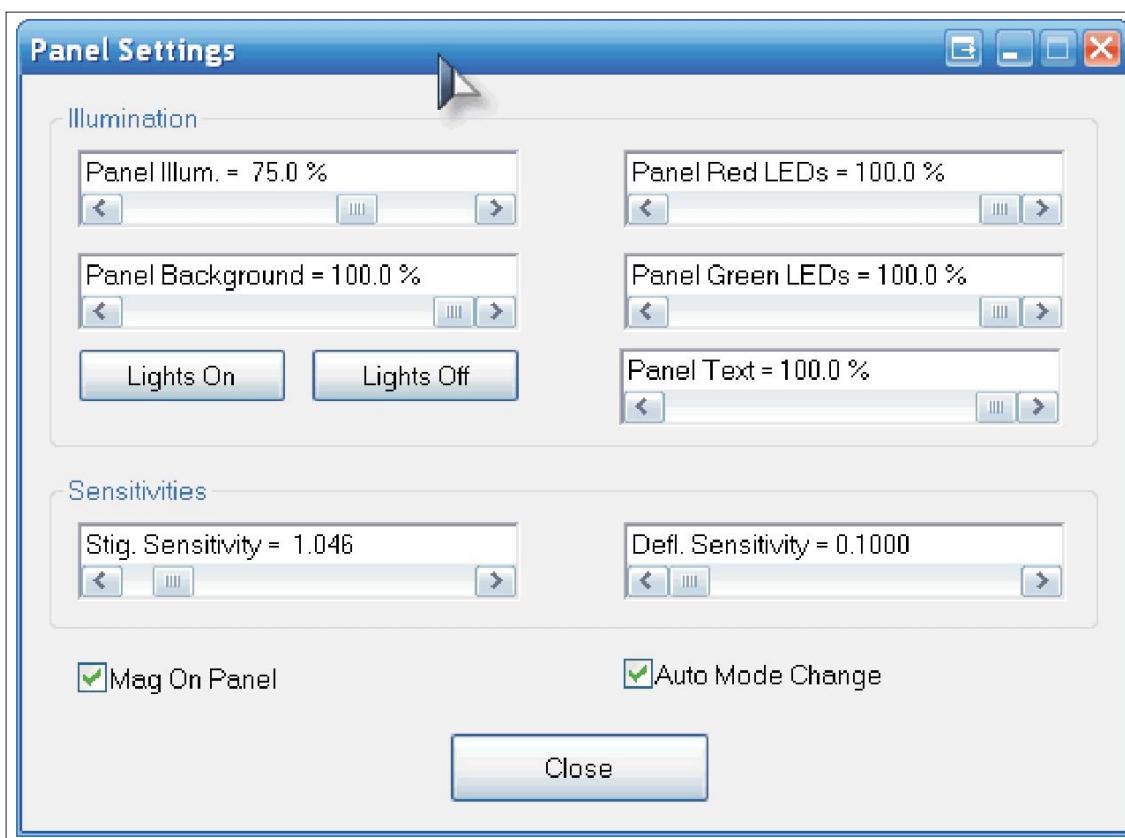


Fig.: 4 - 11 The Panel Settings dialog box

4.4.19 Parameter View

The TEM parameter view is a floating window that displays parameters that the user has selected. In style, functionality and configuration it is similar to the status bar but because of its ease of configuration the parameter view is actually more often changed and used.

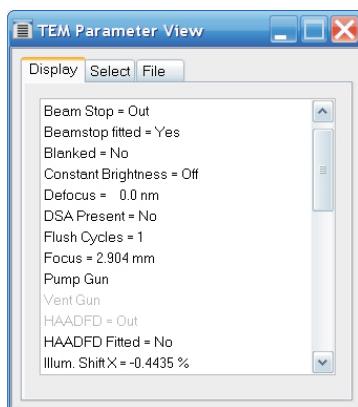


Fig.: 4 - 13 Display tab

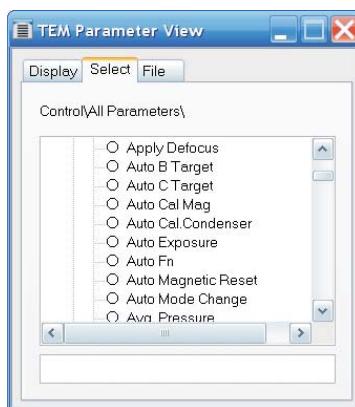


Fig.: 4 - 14 Select tab

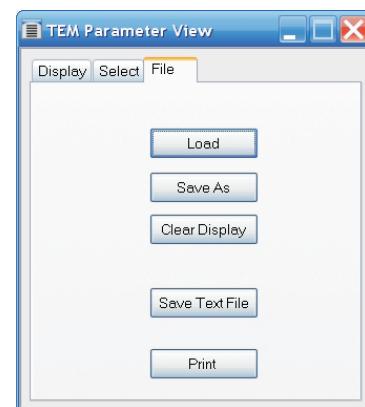


Fig.: 4 - 12 File load/Save tab

Parameter View files

The File tab allows exporting and loading configurations of parameters to display. One can think for instance of making a collection of parameters for “Vacuum”, one for “Goniometer” and load these when needed. Any default configuration is stored in the file **Current.STS** in the user directory. There is no default file in the \DISTRIB directory.

4.4.20 Other Files

In this chapter we look at some other configuration files in the user directory. These are not directly used to change the appearance of the UIF.

XYZ files

These files contain the goniometer points list. The file **global.XYZ** contains the so-called “global points list”, visible to all users. Points in the global list have a dollar ‘\$’ in front of their name. The file with the current points is **current_0.XYZ**. There are also other files with names **current_1.XYZ** - **current_9.XYZ**. These files are points lists in different co-ordinate systems. At the moment the multiple co-ordinate system functionality is not used so the XYZ files do not contain any points.

PRE files

They contain pre-set minimum and maximum values for certain parameters.

ANP files

These are Image Manager Annotation files, used by STEM

UTB files

These are the old user toolbar configuration files. Remove them when you see them because they can mess up the STEM toolbar initialisation.

5 Microscope Operation

Many microscope functions are accessible in the WinTEM window and on the hardpanels. Both options are described in this manual. The user can select the most convenient one.

5.1 EM system turn-ON



NOTICE:

To keep the LIBRA 120 in best working conditions the vacuum system should run all the time. Therefore the LIBRA 120 can only be turned off with the main switch behind the instrument.

If the LIBRA vacuum system and the electron optics power supply were in *READY* status (green light **ON**), nothing is to do at the console. Continue with *Start the PC*.

Was the yellow button pushed, only the vacuum system is in *READY* status. This might be a *STANDBY* position e. g. over the weekend.

In case of a restart of the entire system (main switch OFF or a start from STANDBY):

- Push the green button at the console.
 - The vacuum system is starting till the vacuum status *READY* is reached (displayed on the PC Monitor).
- Start the PC.
 - After booting the Windows Operating System is loaded.
- Click on the icon *WinTEM* on the desktop.
 - The WinTEM server is loaded.
 - The access window is displayed.
- Type in the user and the password.
 - The WinTEM program is loaded and the WinTEM main menu is displayed (next page).



Fig.: 5 - 1 Turn ON/OFF buttons

5.2 Anticontaminator

**NOTICE:**

The use of liquid Nitrogen requires special safety precautions for eyes and hands. See safety chapter in this manual.

Liquid Nitrogen helps to improve the vacuum in the specimen area, because it serves as a cryo pump. It is very recommended for long time irradiation on the same specimen area.
It is not cooling the sample!

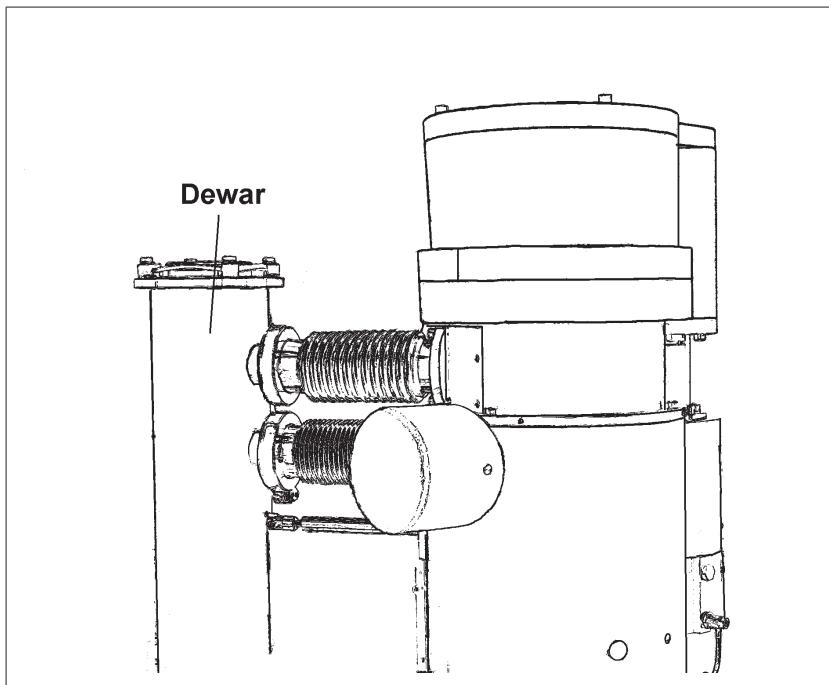


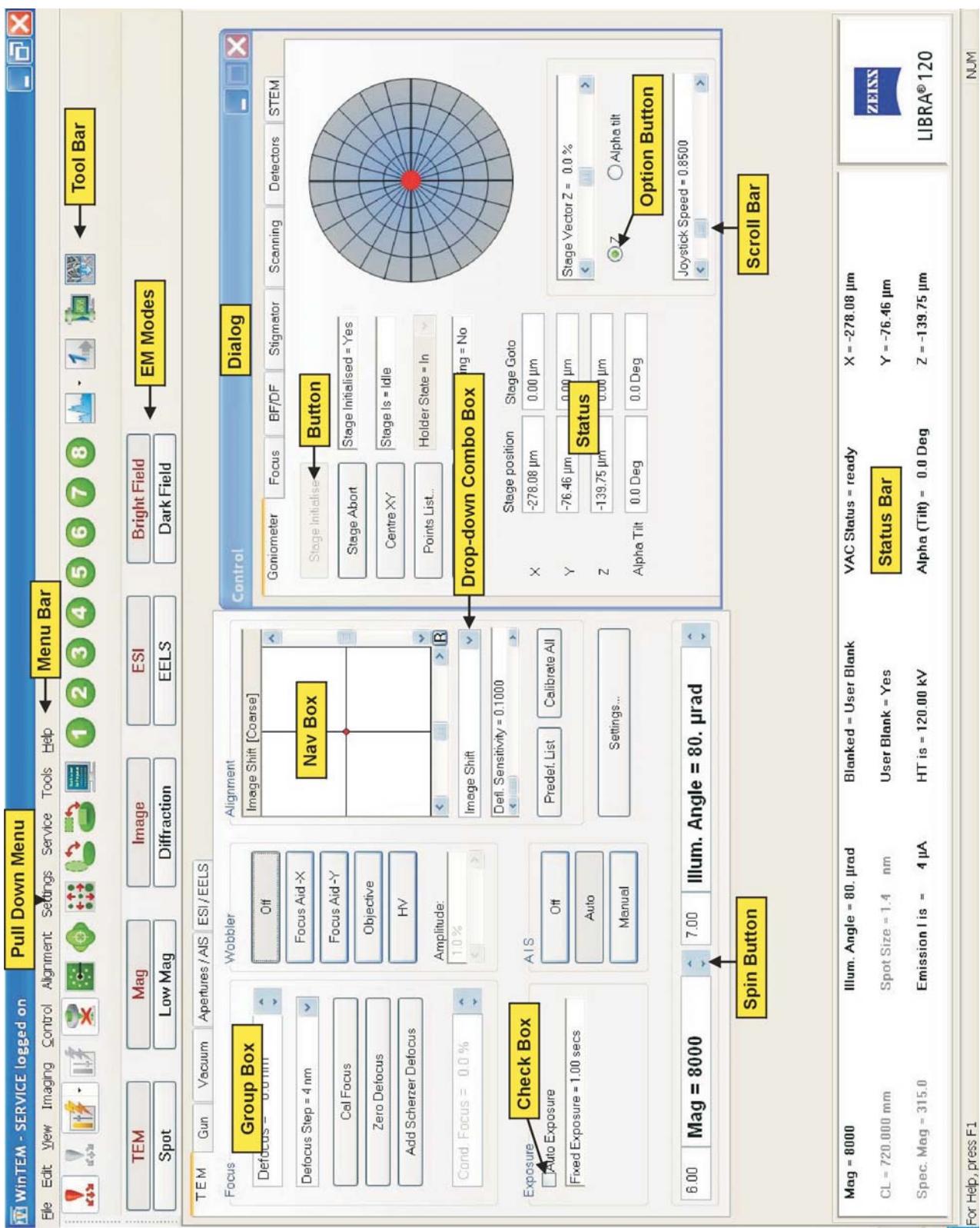
Fig.: 5 - 2 Liquid Nitrogen dewar of the LIBRA 120

- Remove cap from Nitrogen dewar and put in the funnel.
- Fill in liquid Nitrogen carefully.
 - Strong gas evaporation!!
- Refill after 15 min. and close the dewar with cap.

**NOTICE:**

The cooling time of a cold and filled up dewar is about 8 h. Refill Nitrogen in time before it is warming up. Thus you can avoid any turn off of filament and high voltage because of low vacuum.

WinTEM Main Menu



5.3 High Voltage

- Open the *Gun* tab.
 - The selected high voltage is displayed in the *High Voltage* group box.

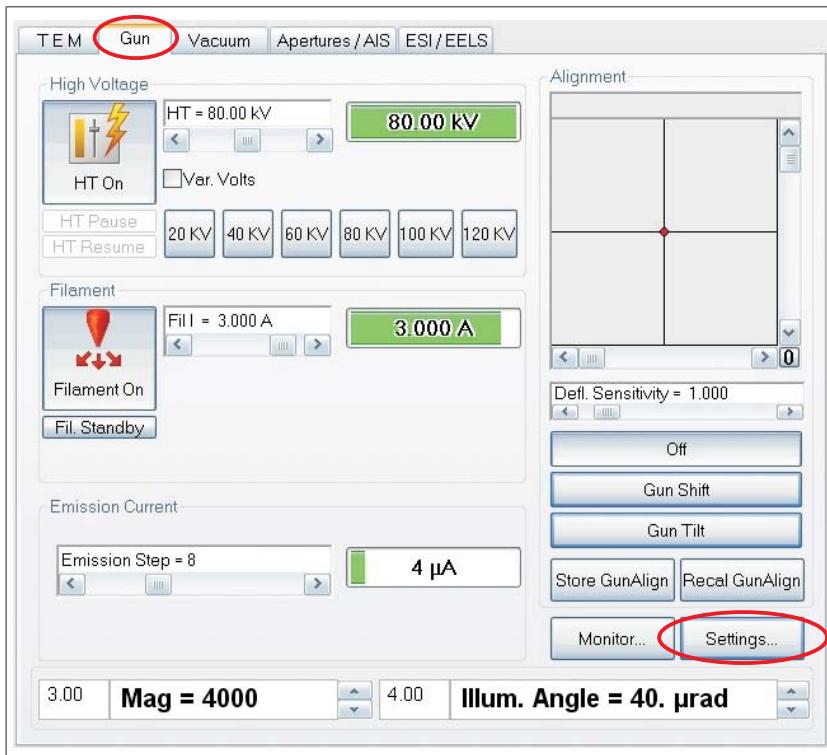


Fig.: 5 - 3 Gun tab

- To turn on the selected high voltage click on *HT On*.
 - The *HT On* Icon is changing to *Ramping*.
 - The green bar displays the progress of ramping.
- To turn on the filament click on *Filament On*.
 - The *Filament* Icon is changing to *Ramping*.
 - The green bar displays the progress of ramping.
 - After ramping there should be a display of the emission current ($> 0 \mu\text{A}$) in the control box *Emission Current*.
 - A proper brightness and mag setting should lead to fluorescence on the viewing screen.
- To change high voltage and filament parameters click on *Settings*.
 - Standard high voltage steps are 120 kV and 80 kV.
 - After selecting a new HT the LIBRA 120 is automatically calibrated for this HT.

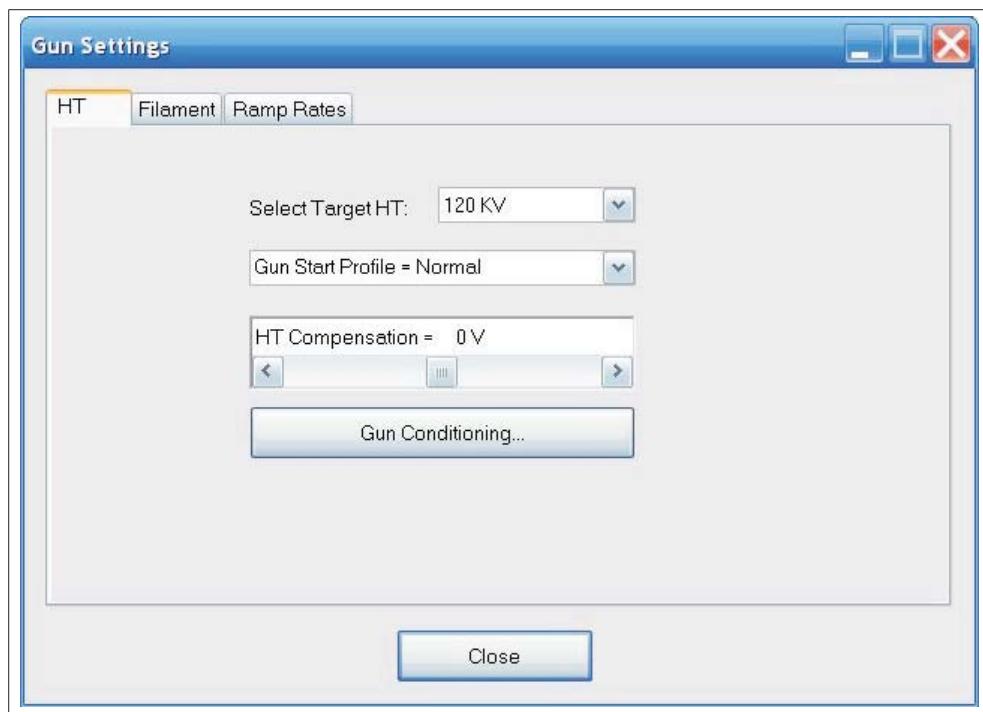


Fig.: 5 - 4 Gun settings for the high voltage

- Select the target HT in the tab sheet *High Tension*, if different from the display e. g. 80 kV.
 - The high voltage is ramping down to 80 kV.



NOTICE:

The function *Gun Conditioning* is highly recommended after filament exchange and after a longer Turn Off of the microscope.

It is used instead of turning on the high voltage the standard way.

More information in the chapter *Maintenance*.

- Click on the tab sheet *Filament* to change the filament type and the limits of the filament current.
- Reset the filament age after filament exchange.

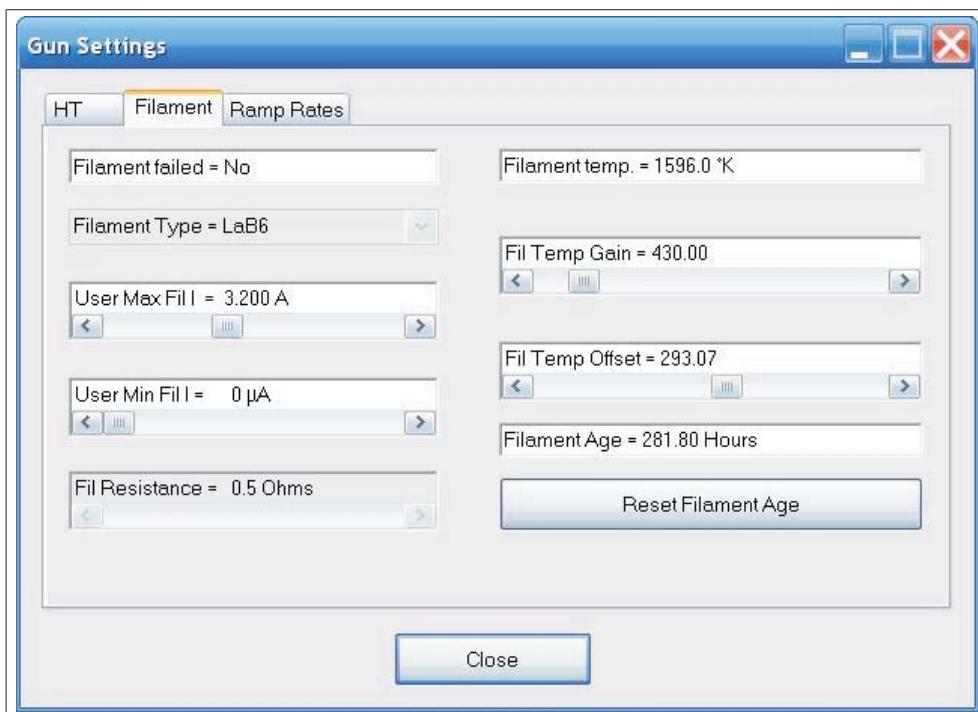


Fig.: 5 - 6 Filament parameters

- Click on the tab sheet *Ramp Rates* to change the ramping speeds for filament and high voltage.

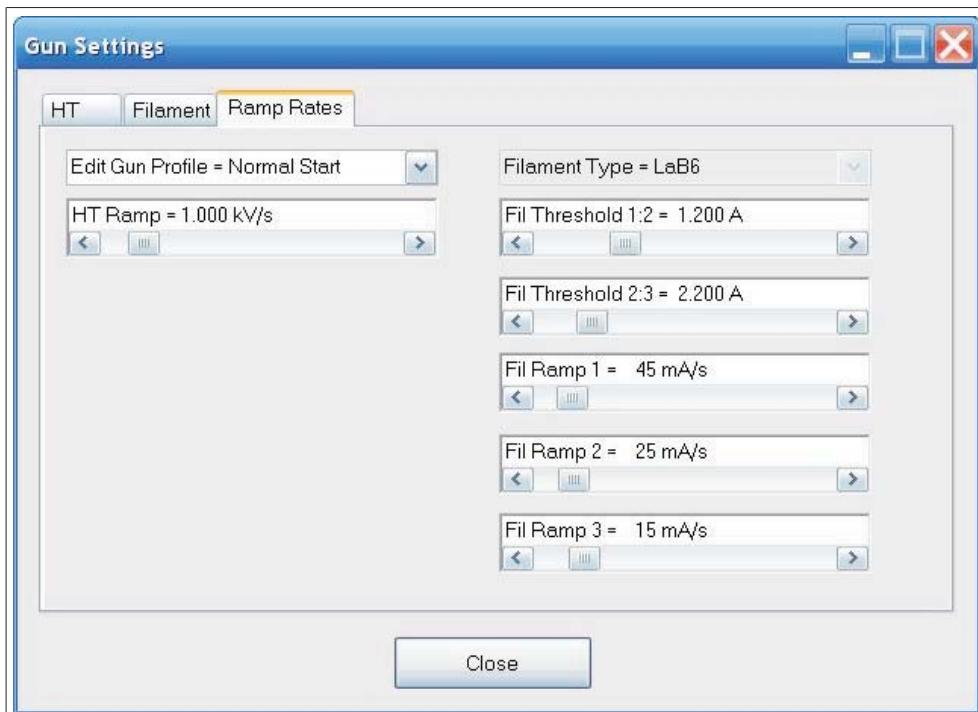


Fig.: 5 - 5 Ramp rates for filament and high voltage

- Check the beam blunker status in the status bar.
 - **User Blank** should be **No**. Changes are possible in the tool bar or status bar.
 - If there is no fluorescence on the screen, retract the slit aperture, retract the specimen holder, lower the mag (about 10k), increase the brightness (about 0.5 mrad).



- To turn off filament and high voltage click on *Filament On* and *HT On*.

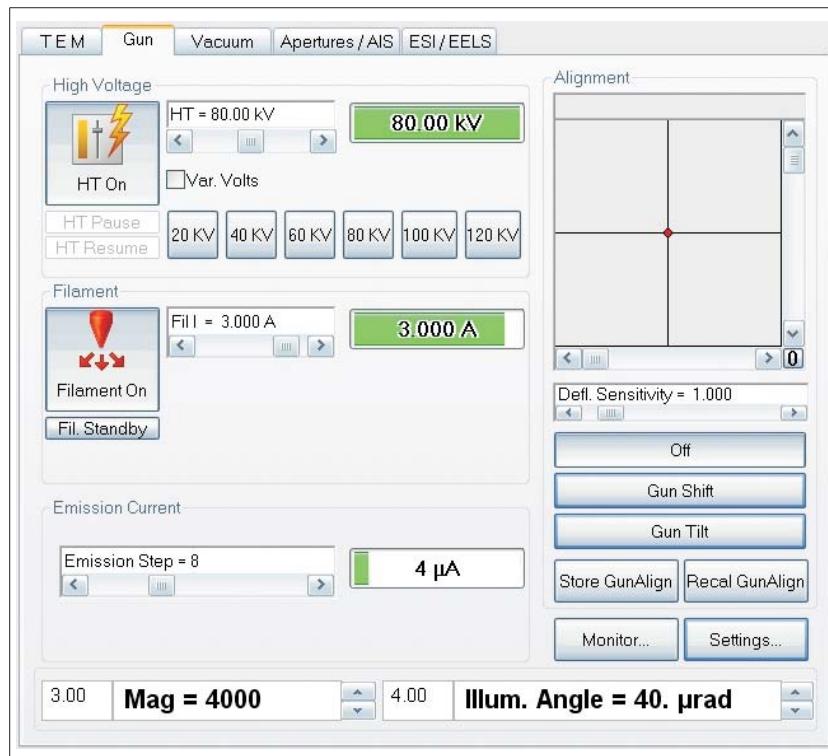


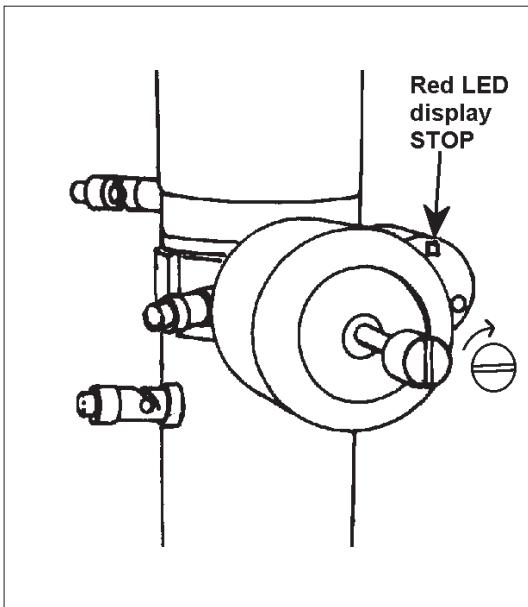
Fig.: 5 - 7 Gun tab



NOTICE:

The function *Fil Standby* is highly recommended for a longer break or instead of a shut down over night. The filament current is reduced by 40% ($3.000 \text{ A} \rightarrow 1.800 \text{ A}$). After another click on this function the filament current is reset to 100%.

5.4 Operation of the specimen holder

**WARNING:**

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

Rule:

No action, if LED display STOP is on.

5.4.1 Removing the specimen holder

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

5.4.2 Loading the specimen

See figure on the next page.

- Put specimen rod into the wooden container.
- Loosen screw with collet chuck and lift it off together with holding bow.
- Insert new grids with tweezer.
- Put the holding bow on the 2 samples and tighten screw with the clamping tool.

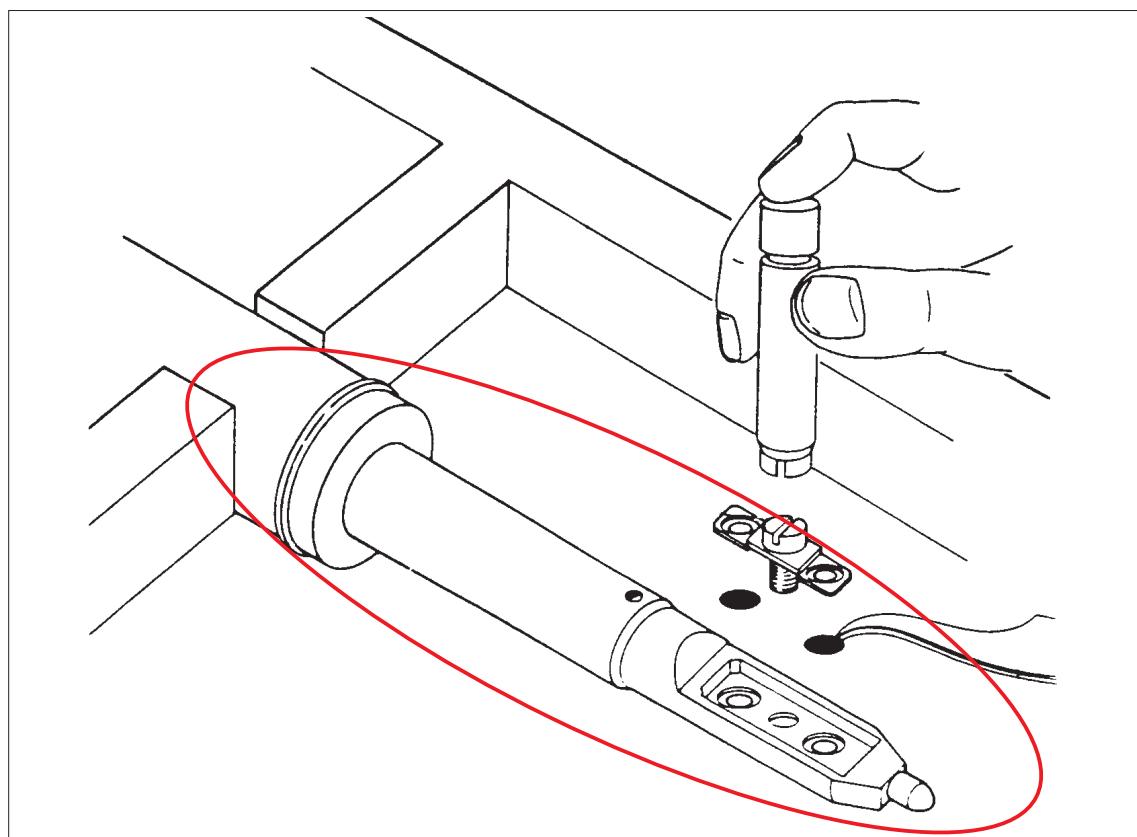


Fig.: 5 - 8 Loading of samples



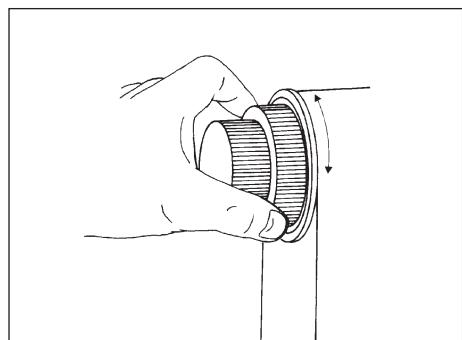
CAUTION:

It is very important to keep the red marked area of the holder very clean. This part goes into the high vacuum of the instrument. It must not be touched with bare fingers.

5.4.3 Changing the specimen position

How to insert the specimen holder see next paragraph first.

- Turn the large knurled ring to the left.
- Turn the small knurled ring to the left or to the right stop depending which position it was.
 - The sample holder moves further in or out.
- Tighten the large ring.
 - The selected specimen position is visible on the viewing screen.



5.4.4 Locking-in the specimen holder


NOTICE:

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

The LIBRA airlock features an N₂ flush which allows a very clean holder transfer into the high vacuum.



Fig.: 5 - 9 Vacuum menu with N₂ flush function

The N₂ flush box provides the function *Close N₂ Flush* or *Open N₂ Flush*; but the flush is only working, when the airlock is opened.

In addition the airlock can be flushed during the prepumping phase. The number of flush cycles can be selected. For cryotransfers it should be zero.

- Slide specimen rod carefully into the goniometer as far as it will go (Fig. 5-10).
- Turn specimen rod carefully clockwise through about 20° all the way to prevacuum position (Fig. 5-11).
 - Airlock flush stops, if activated before.
 - 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 the stop position (Fig. 5-12 (1)) and turn it carefully through further 90° (2); guide rod slowly (3) into the microscopy position (Fig. 5-13).

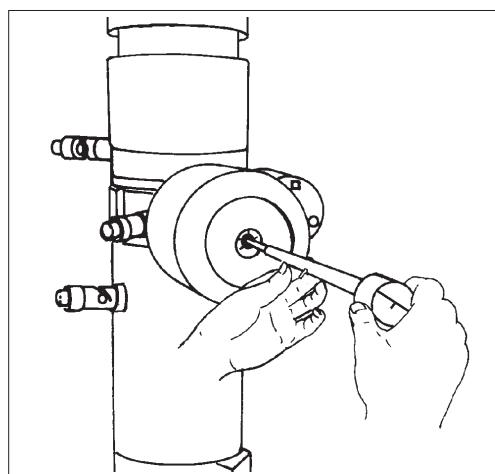


Fig.: 5 - 10 Insertion of specimen rod

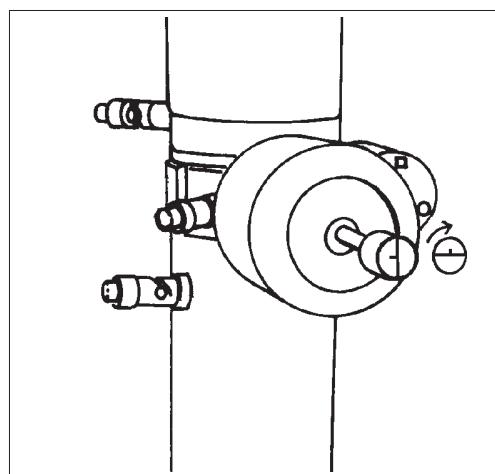


Fig.: 5 - 11 Pre-evacuation

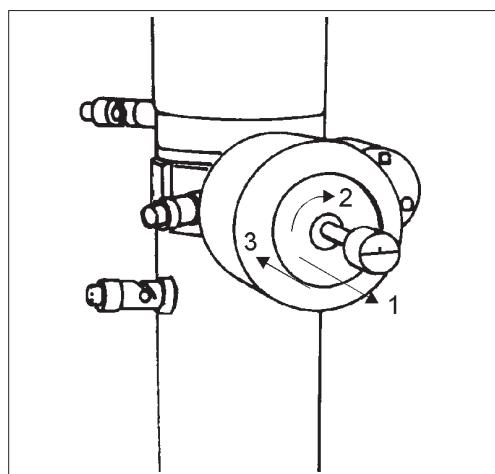


Fig.: 5 - 12 Locking in specimen rod

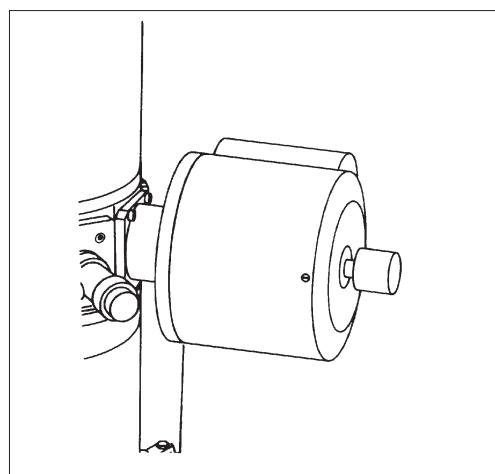


Fig.: 5 - 13 Microscopy position

5.5 Stage control

The stage can be operated by the joystick on the right hardpanel or by mouse in the control window of the main menu.

In addition to specimen movement sample positions can be stored and recalled.

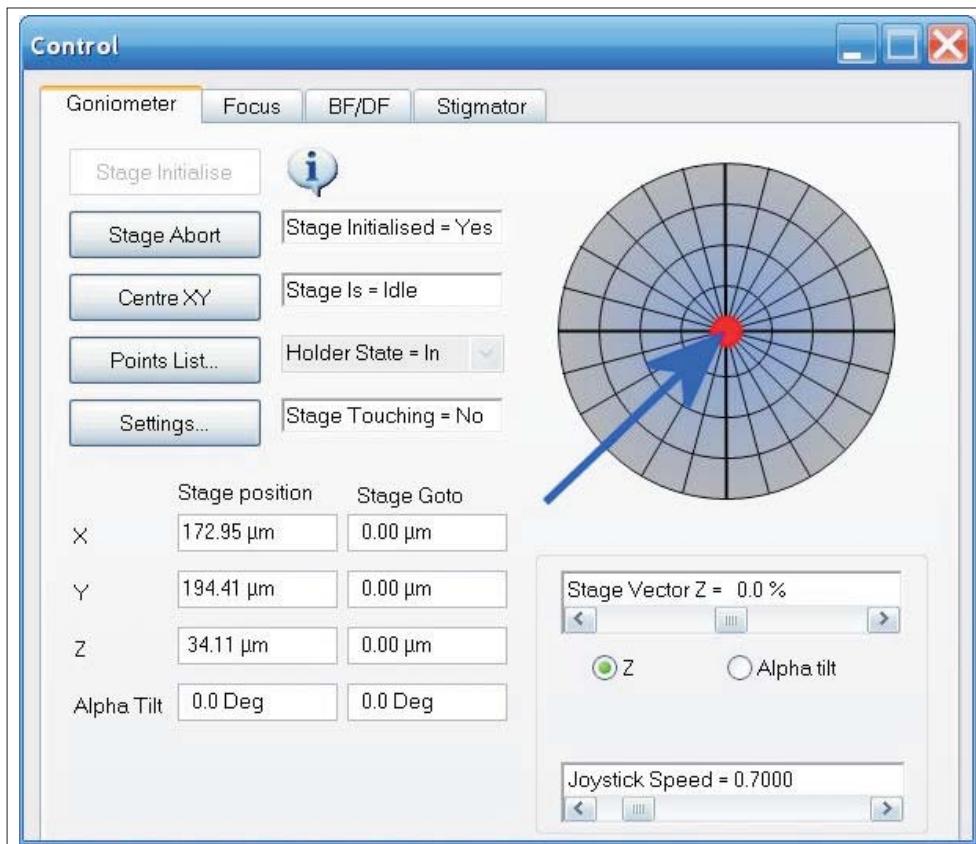


Fig.: 5 - 14 Stage operation with Windows mouse

- Grab the red central point of the visual joystick (keep left mouse click pushed) and move it to the desired direction.
 - The further the red point is moved off center the faster the sample is moving.
 - When releasing the mouse click, the red point jumps back to the center and the goniometer stops moving.
- Click on *Points List* to store and recall sample positions.
 - The points list is displayed on the next page.

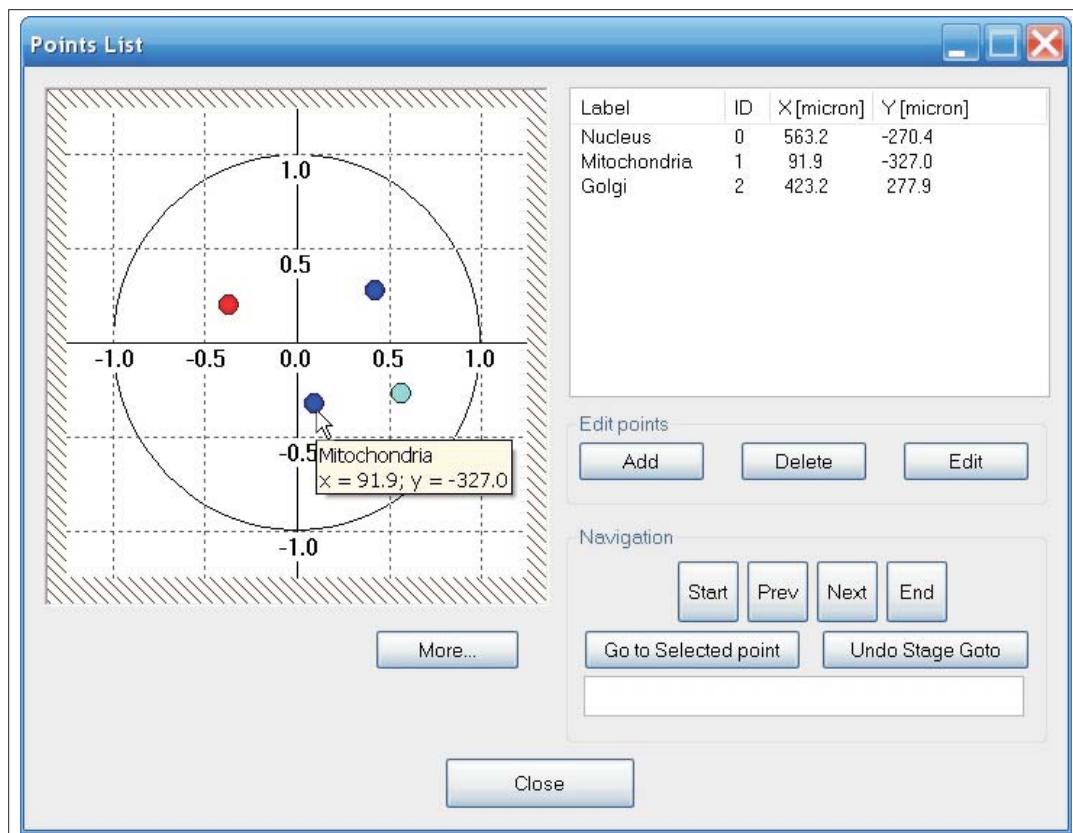


Fig.: 5 - 15 Points list for display of sample positions



Display of the current position (stored position or not stored yet).



Display of the last stored position.



Display of all previous postions.

Edit points window:

Add: The current position is added to the point list.

Delete: The selected position of the list is deleted.

Edit: Name and coordinates of the selected position can be edited.

Navigation window:

Start: moves to the first position of the list.

Prev: moves from the current position to the previous position.

Next: moves from the current list position to the next one.

End: moves to the last position of the list.

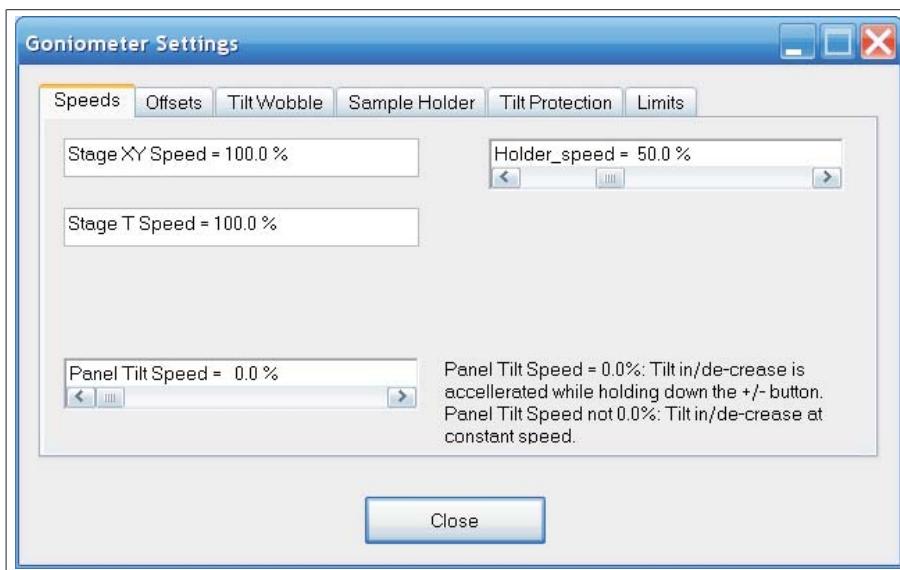


Fig.: 5 - 16 Goniometer speeds

- Click on *Goniometer Settings* in the *Control* menu.
 - The tab sheet *Speeds* is opened.
 - The speed for the different goniometer axes can be adjusted.
- Click on e. g. *Stage XY Speed* and type in a new speed value.



NOTICE:

For tomography applications the tilt speed should not be higher than 25%.

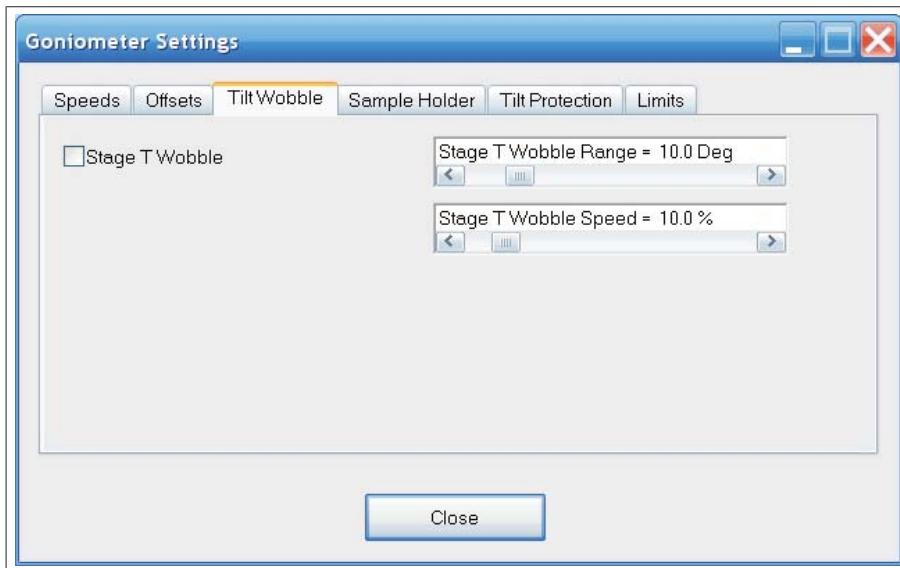


Fig.: 5 - 17 Goniometer Tilt Wobble

- Click on tab sheet *Tilt Wobble*.
 - The wobble range and wobble speed can be adjusted.

**NOTICE:**

The tab sheet **Sample Holder**, **Tilt Protection**, and **Limits** are settings to be modified by the Administrator or a Zeiss service engineer.

The tab sheet **Offsets** contains the factory settings.

5.6 Basic alignment



NOTICE:

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

5.6.1 Preparation for the basic alignment

- Retract rod as far as it will go and turn it carefully somewhat counter-clockwise to keep it in this position.
- Insert condenser aperture (in general 3rd clickstop) and activate the AIS mode *Auto* in the *TEM* tab sheet of the main menu.
 - It is provided that the mechanical and electrical AIS adjustment was checked at the installation of the LIBRA 120.

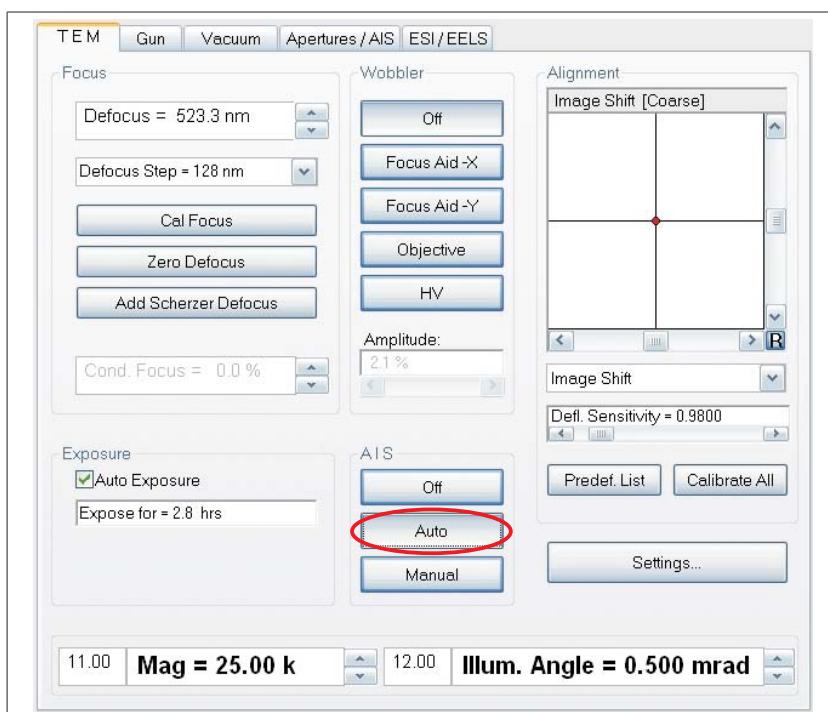


Fig.: 5 - 18 TEM Tab Sheet

- Remove objective aperture.
- Remove spectrometer entrance aperture and spectrometer slit aperture.
- Turn on HT and filament as described in §§ 5.3.
- Select mag of 10.000x with knob *Mag* on the left hardpanel.

5.6.2 Calibration to factory settings

Either

- Calibrate objective lens current with *Cal Focus* in the group box *Focus*.

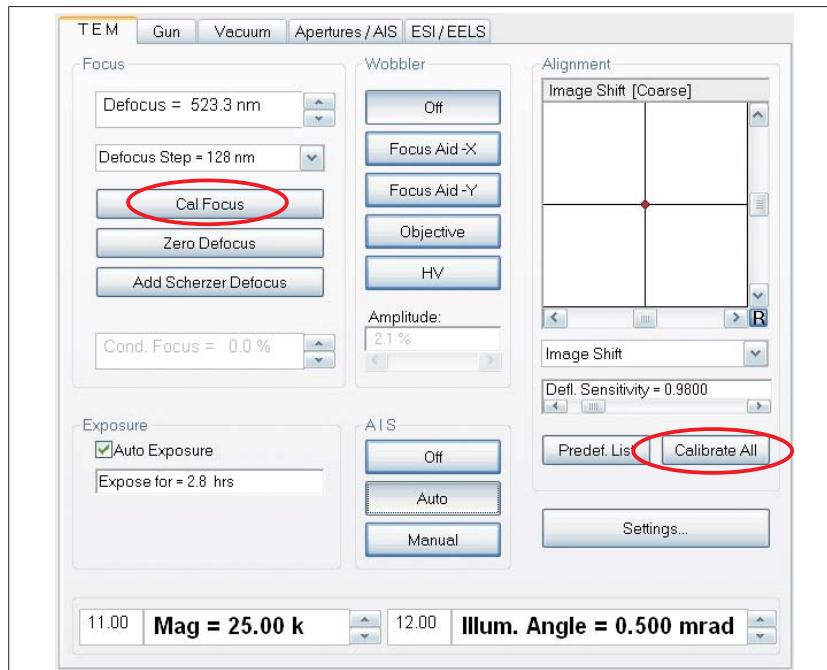
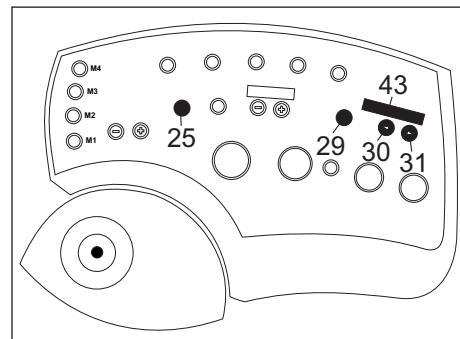


Fig.: 5 - 19 Adjust tab sheet in the WinTEM main menu

Or

- Push button *Cal* (25) on the right hardpanel.
 - Both actions lead to the same result.
 - Further deflecting systems can be selected and calibrated.
- Select *Img Shft* with button (30 or 31).
 - The function is shown in the display (43).
- Calibrate *Img Shft* with button *Cal* (29).



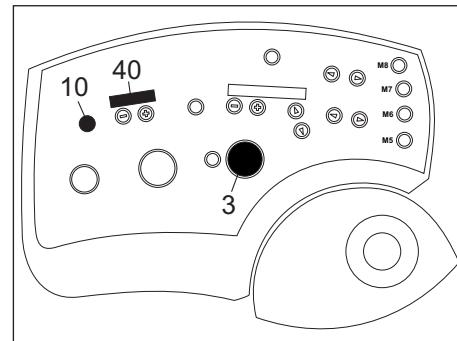
NOTICE:

This is the minimum calibration to be done. If there is no fluorescence on the screen, and all other options were excluded calibrate all deflecting systems.

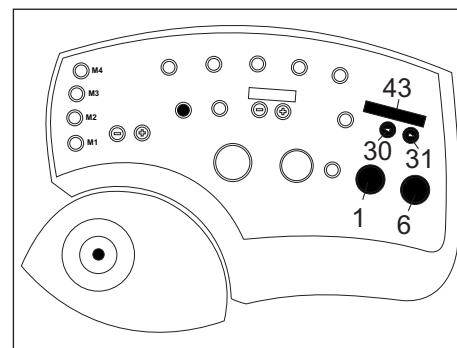
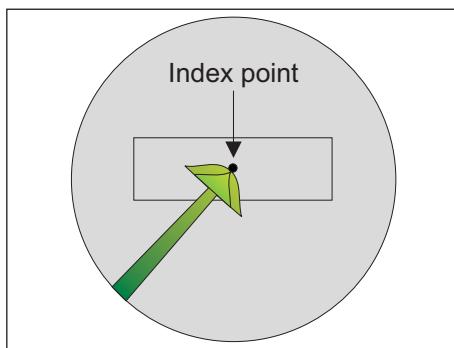
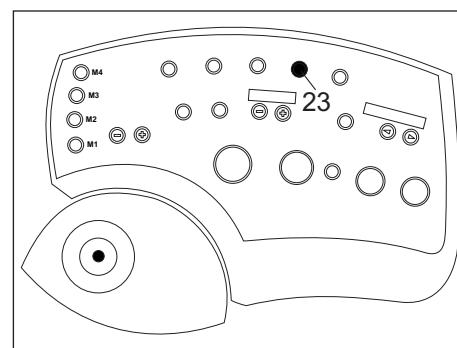
- Click on *Calibrate All* in the group box *Alignment*.
 - This function is not available on the hardpanels.

5.6.3 Spectrometer alignment

- Click on 0 eV in the *Delta E* group box in the *ESI/EELS* tab sheet to reset the energy loss setting to zero.
- or
- Push button (10) on the left hardpanel till the display (40) shows 0 eV.
 - Button (10) allows toggling of 3 energy loss modes:
0 eV → 250 eV (HCl) → user defined.
- Set mag to 12.500x with mag knob (3).



- Switch to spectrum mode by pushing button (23) on the right hardpanel.
 - *EELS* is highlighted on the hardpanel.
 - The spectrum caustic is visible on the viewing screen.
 - *Spec Shft* is shown on the display (43).



- Shift the tip of the spectrum caustic to the index point of the small viewing screen with knobs (1) and (6).
 - The graphics on the right shows the adjusted position.

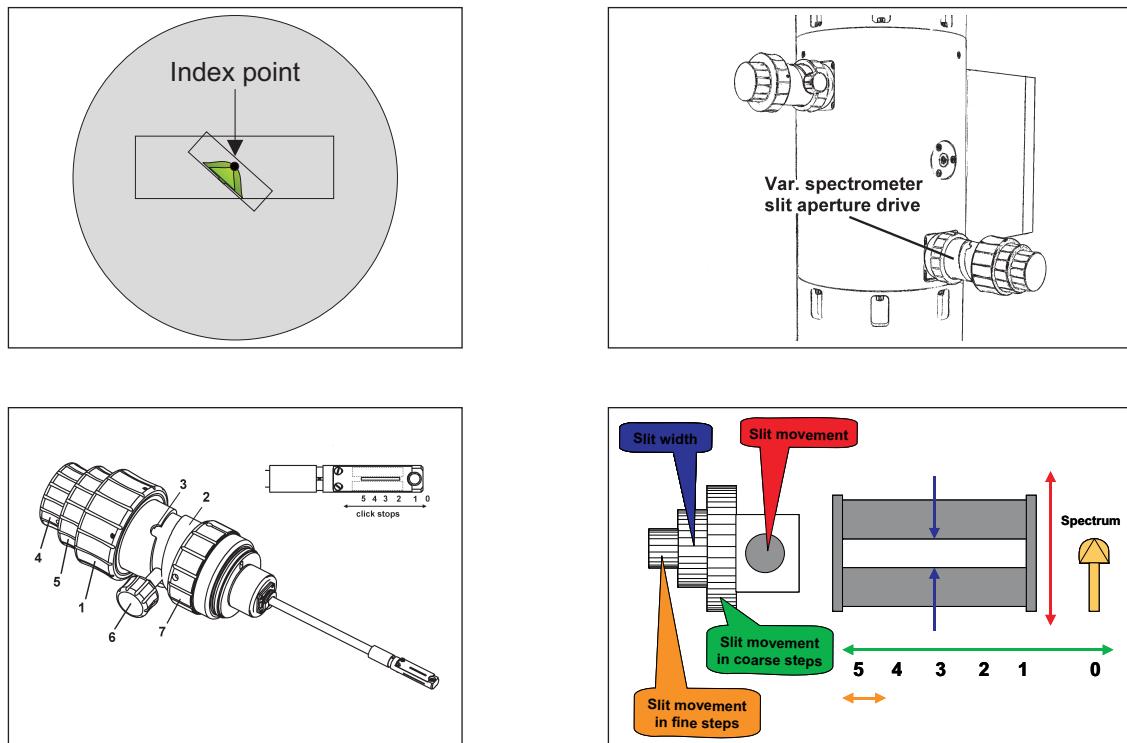
- Switch back to IMAGE mode with button (23).
 - *ESI* is highlighted.
 - *Img Shft* is shown on the display (43).
- or
- Stay in spectrum mode to adjust the spectrometer slit aperture.

5.6.3.1 Variable slit aperture drive installed

NOTICE:

The slit aperture allows to select a range of the energy loss spectrum for imaging. The adjustment below represents the so called “zero loss” imaging mode. All energy loss electrons are cut off by the slit.

To take advantage of energy filtering the slit should be used **at all times** except for acquiring EELS spectra.

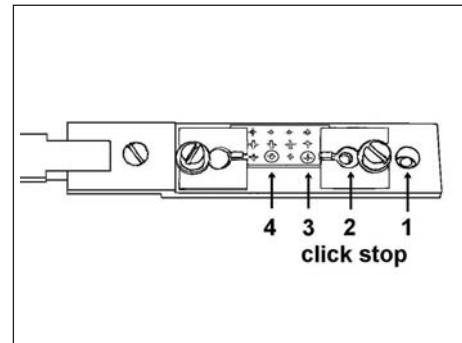
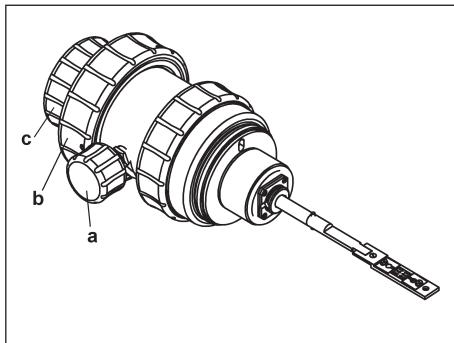
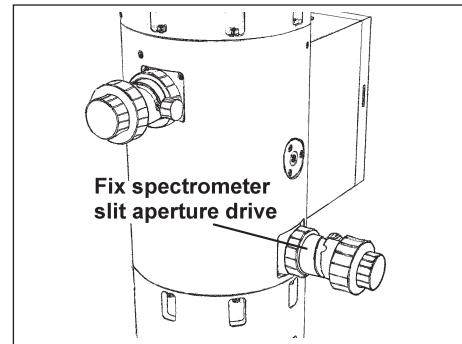
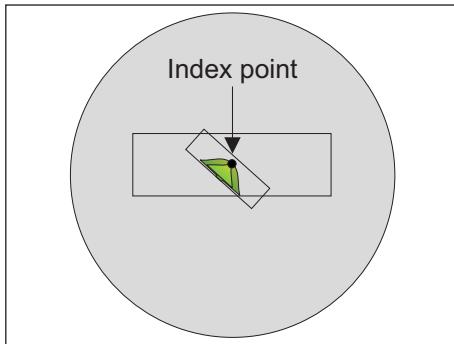


- Turn the knurled ring (1) from left stop to the e. g. 2nd click stop position.
 - This position provides the beginning of the variable slit widths.
 - The other click stops just provide a different mechanical slit position.
 - A centered spectrometer entrance aperture as described in 5.9.3 is provided.

- Move the slit position with drive (6) and adjust the slit width with drive (5) that the spectrum caustic is located within the slit as shown in the upper left graphics.
 - Make sure that the other edge of the slit is not too close to the spectrum tip.
 - If the spectrum tip hits the slit edge it appears as a darkfield effect in the center of the image.

- Switch to image mode with button (23) on the right hardpanel.
 - *ESI* is highlighted.

5.6.3.2 Fix slit aperture drive installed



- Turn the knurled ring (b) from left stop to the 3rd click stop position where the large slits are located.
 - This position provides slit widths which can be used for zero loss imaging over the negative format as shown in the left upper image.
 - A centered spectrometer entrance aperture as described in 5.7.3 is provided.

- Select and adjust a slit with knurled ring (c) and move it with drive (a) that spectrum caustic is visible within the slit.
 - Make sure that the other edge of the slit is not too close to the spectrum tip.
 - If the spectrum tip hits the slit edge it appears as a darkfield effect in the center of the image.
- Switch to image mode with button (23) on the right hardpanel.
 - *ESI* is highlighted.

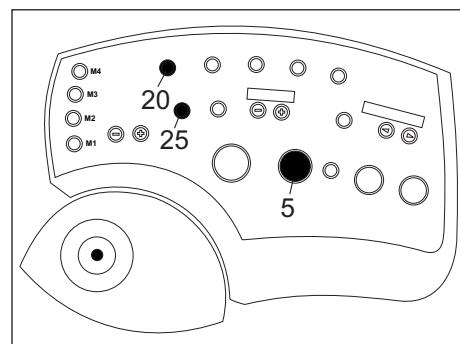
5.6.4 Alignment of TEM- and SPOT-illumination



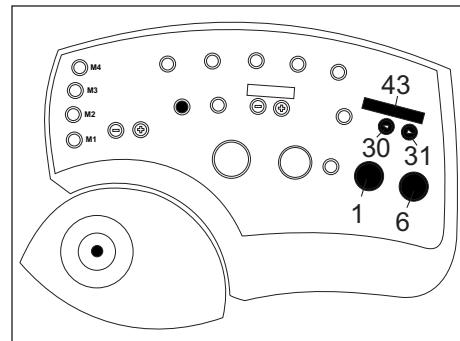
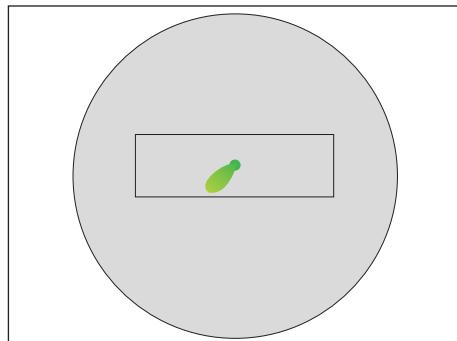
NOTICE:

Depending on the type of condenser aperture (5-hole or BIO AIS) the described phenomena can be different. More details can be found in chapter 5.7 or 5.8.

- Switch to spot illumination with button (20).
 - *Spot* is highlighted.
 - *III Shift* is automatically selected on the display (43).
- Push button *Cal* (25) to calibrate the objective lens and condenser C 3.
 - The spot should be imaged and focussed on the viewing screen.



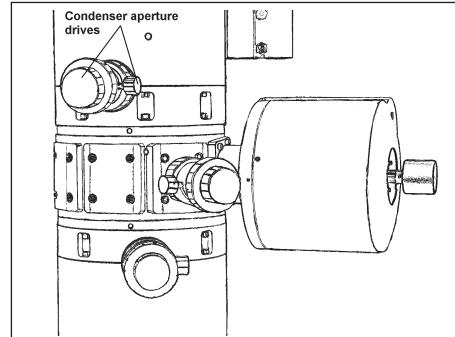
- Increase spot up to 50 nm with knob (5).
 - The spot size is displayed in the WinTEM main menu.



- Move spot to index point with knobs (1) and (6).
 - The asymmetric spot is only visible by using the 5-hole AIS aperture. Therefore the following alignment step is only valid for the 5-hole aperture.

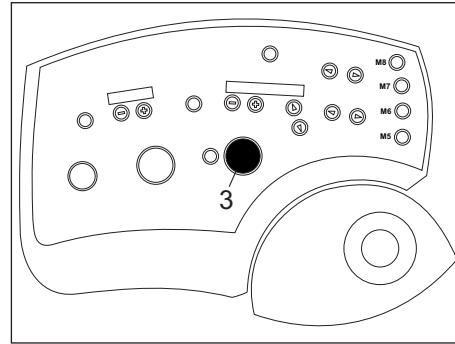
- Center the condenser aperture, if the spot looks like on the above image.
 - Only visible and necessary to adjust with the 5 hole AIS aperture.

- Use the condenser aperture drives to adjust a symmetric spot.
 - The asymmetric halo is gone.

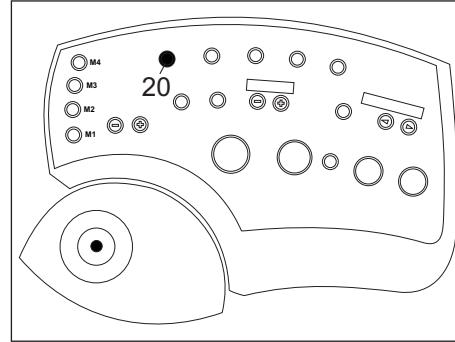


- Increase the magnification to 31.500x with knob (3) and readjust spot to the index point as described above.

- Increase magnification to 160.000x with knob (3) and readjust spot to the index point as described above.



- Switch to TEM mode with button (20) and center condenser aperture to the large screen (see aperture drive on the previous page).
 - *TEM* is highlighted.
 - The central AIS aperture (only 5-hole AIS aperture) is mechanically centered.



5.6.5 Beam alignment (Gun tilt)



NOTICE:

There are good results at **all** illumination angles, if the beam alignment is optimised.

- Select *Diff* mode with button (22).
 - *Diff* is highlighted. The diffraction spot is visible on the viewing screen.
- Set illumination angle (brightness) to 0.06 mrad (60 μ rads) with knob (5).
 - Display of the illumination angle in the lower right corner of the tab sheet *Gun*.
- Focus the diffraction spot with knob (4).
- Shift diffraction spot to the index point with buttons (1) and (6).
 - *Img Shft* mode is automatically selected on the display (43)
- Click on *Gun Tilt* in the *Gun* tab sheet.
 - The current position of *Gun tilt* is displayed in the navigation box.

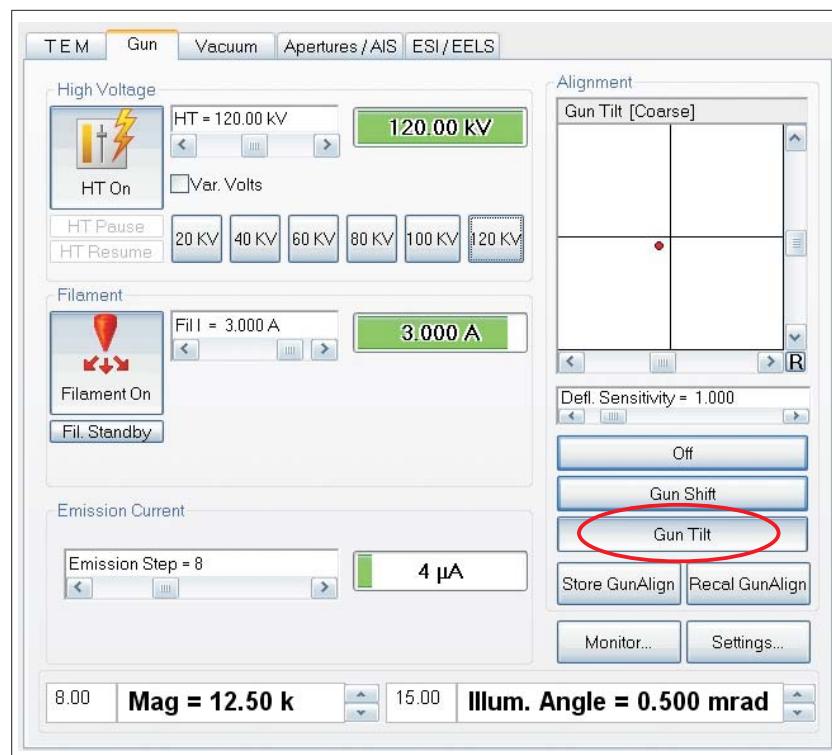
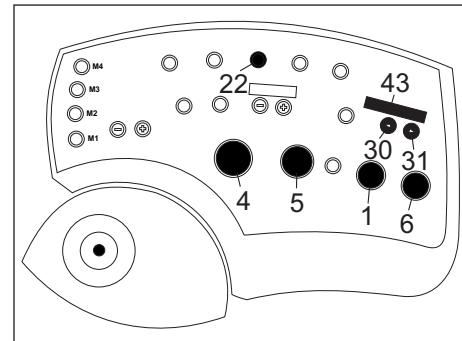


Fig.: 5 - 20 The Gun menu

- Increase the illumination angle to 1.6 mrad with brightness knob (5) on the right hardpanel.

**CAUTION:**

Before increasing the brightness read the next steps and make familiar with them. The excessive brightness can damage the focussing screen. So, keep the irradiation time as short as possible.

It is also helpful to use a thicker sample and to reduce the field of illumination by applying the MIS mode.

Either

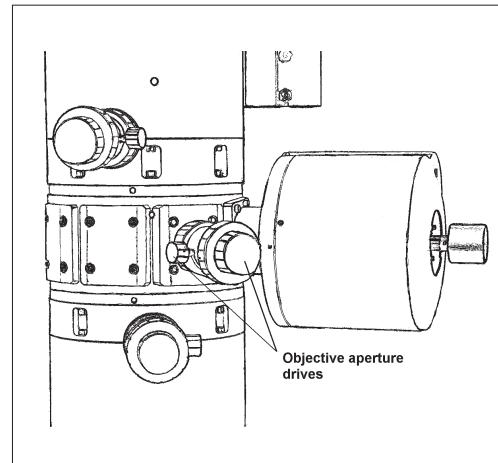
- Shift the diffraction spot to the index point with the sliders in the navigation box.

Or

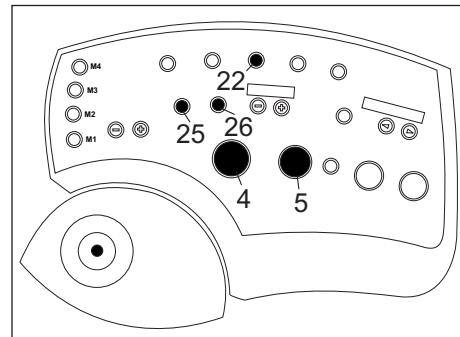
- Click on *Set to panel X Y knobs* and use knobs (1) and (6) for the alignment.
- Store *Gun Tilt* with *Store Gun Alignment*.
 - Repeat this procedure till the diffraction spot stays on the index point at 0.06 and 1.6 mrad.
- Push button (22) on the right hardpanel to return to image mode.
 - *Img* is highlighted.

5.6.6 Adjustment of eucentric plane

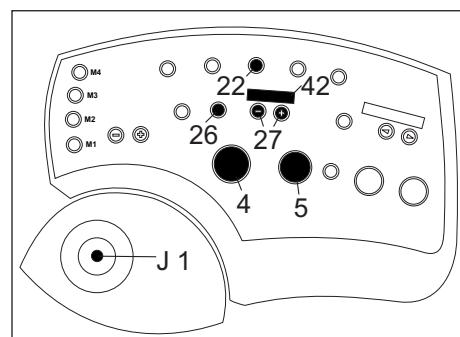
- Move specimen rod all the way into the goniometer.
- Insert the desired objective aperture (1st, 2nd, or 3rd clickstop).
- Switch to *Diff* mode with button (22) on the right hardpanel.
- Center the objective aperture with the marked aperture drives so that the diff. spot is in the center of the aperture.
- Switch back to *Img* mode with button (22).



- Select magnification of 25.000x with button *Mag* on the left hardpanel and look for a specimen detail with sufficient contrast.
- Adjust brightness with button (5).
- Calibrate the objective lens with button (25).
- Turn on *Foc Aid* with button (26).
 - *ON* is highlighted and the sample detail is oscillating on the viewing screen.
 - *Mech* is automatically displayed (42).



- Focus the specimen mechanically (Z-adjust) with button *Foc* (4) until it stands still.
 - The specimen is now in the eucentric plane of the goniometer.
- Turn off *Foc Aid* with button (26).
 - *ON* is off.
 - *Auto* is automatically displayed (42).



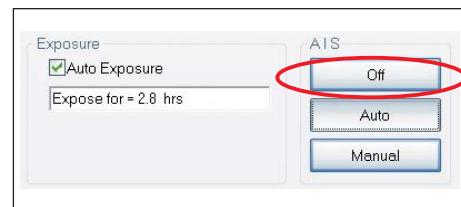
5.6.7 Adjustment of beam tilt



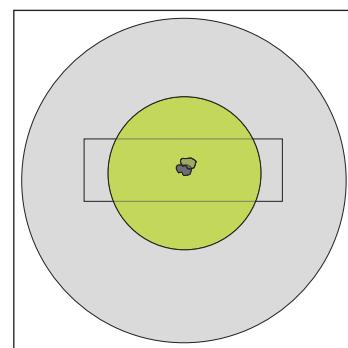
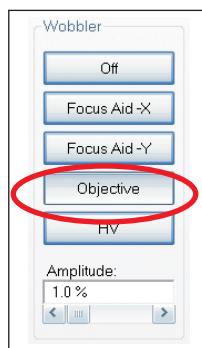
NOTICE:

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

- Turn off the AIS mode in the group box *AIS*.
- Select magnification of 50k with knob *Mag* on the left hardpanel.
 - The central AIS aperture is imaged on the viewing screen.

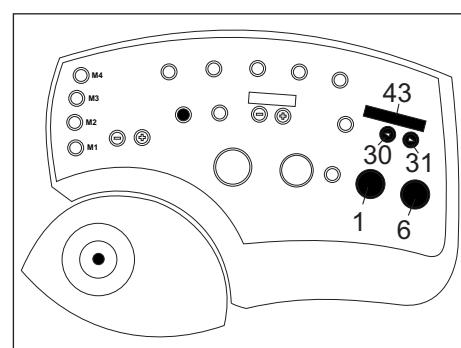


- Find a specimen detail with sufficient contrast and move it to the index point.
- Activate the function *Objective* in the group box *Wobbler*.
 - The specimen detail is oscillating, if there is a beam tilt.



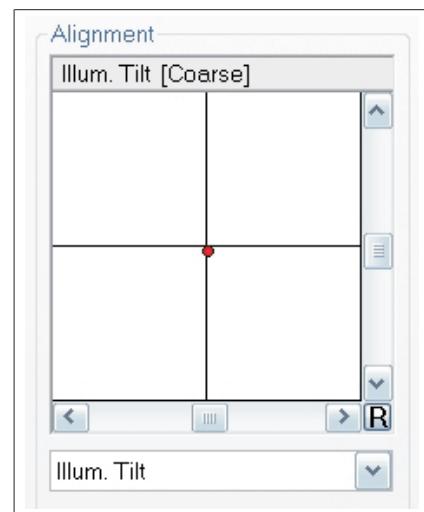
Either

- Minimise the specimen movement with knobs (1) and (6).
- Deactivate the wobbler by pushing again on the function *Objective*.
- Turn on the *A/S* mode in the group box *A/S* again (*AIS* = auto).
 - The viewing screen is fully illuminated.



Or

- Move the horizontal and the vertical slider with the cursor until the sample detail is not oscillating anymore.
- Click on *Objective* one more time to deactivate it.
 - The function *Image Shift* is displayed in the group box *Alignment* and on the hardpanel display.



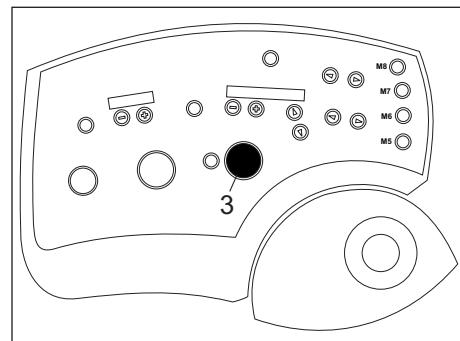
5.7 5 hole AIS aperture adjustment

NOTICE:

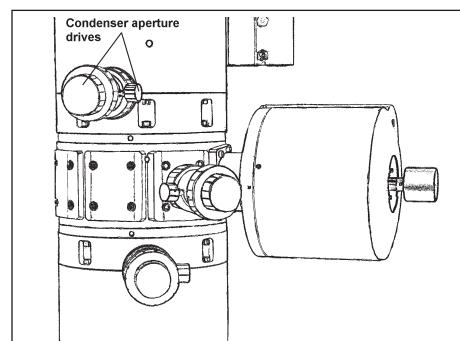
The AIS adjustment requires a basic alignment of the TEM. Two different AIS apertures are available. **5 hole AIS** or **7 hole BIO AIS**.



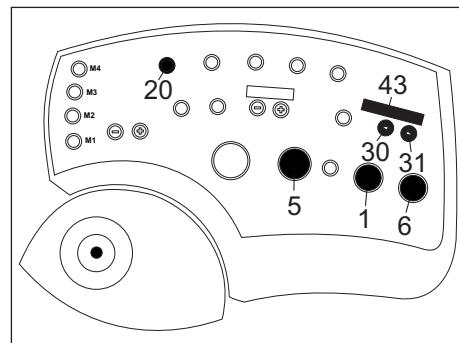
- Set magnification to 4.000x and brightness to 0.5 mrad.
- Select *AIS Off* in the tab sheet *TEM* of the WinTEM main menu.
 - The central AIS aperture is imaged on the viewing screen.



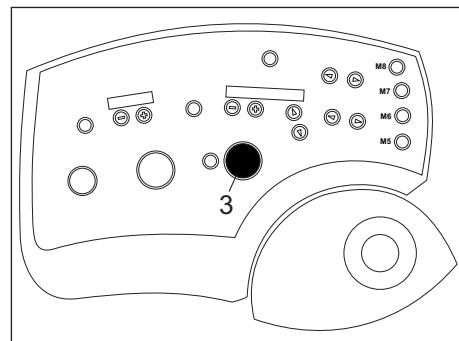
- Center the central AIS aperture (37.5 µm) to the index point.
 - It is provided that the alignment point *5.6.4 Alignment of TEM and SPOT-illumination* was done.



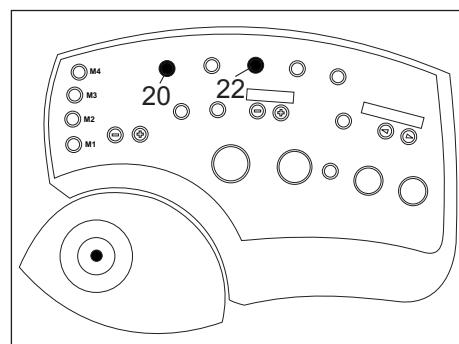
- Switch to SPOT mode with button (20).
 - *Spot* is highlighted.
 - *Img Shft* is automatically displayed (43).
- Increase spot size to 50 nm with knob (5).
 - If the brightness is too high, increase the mag by a few steps first.



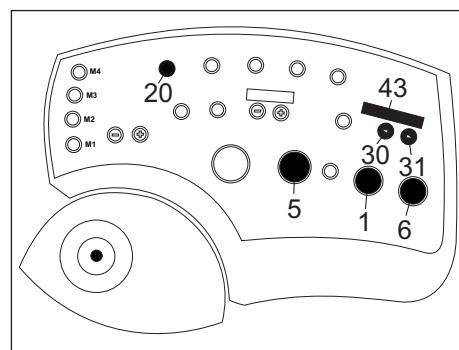
- Increase mag to 160.000x with knob (3).
 - Make a stop at 50.000x to adjust the spot to the index point (described in the next step).
- Adjust the spot to the index point with knobs (1) and (6).



- Switch to TEM mode with button (20).
 - *Tem* is highlighted.
- Center the AIS aperture to the large viewing screen with condenser aperture drives.
- Switch to DIFF mode with button (22).
- Select CL=580 mm with *Mag* button (3).



- Select *Img Shft* on the display (43) with buttons (30) or (31).
- Shift the diffraction spot to the index point with buttons (1) and (6).
- Switch back to IMG mode with button (22).

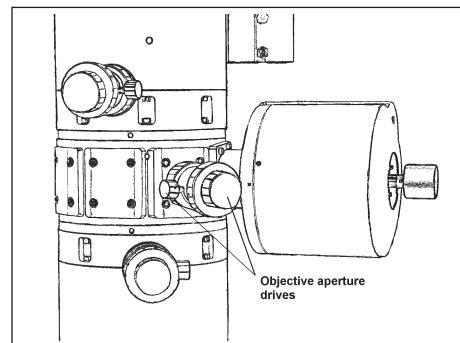


CAUTION:

Irradiation in DIFF or SPEC mode can damage the screen.



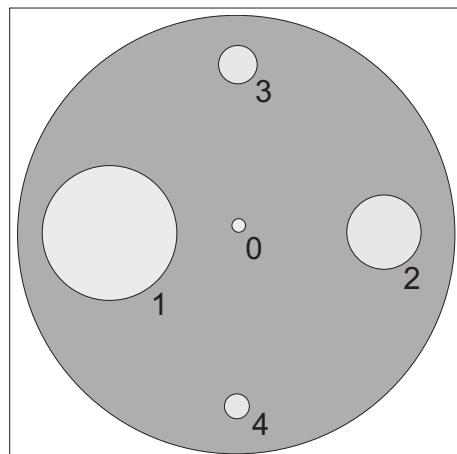
- Insert the 90 µm objective aperture (1st clickstop).
- Center it to the diffraction spot with the aperture drives.
- Switch to *Img* mode with button (22) on the right hardpanel.



NOTICE:

At this point the preparation for the AIS alignment is finished and the real AIS alignment starts.

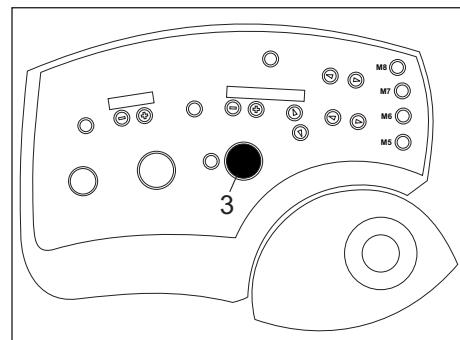
The AIS illumination is using different condenser aperture assigned to different magnification ranges. The selection of the aperture is defined by the magnification. Since the off-center apertures are selected by deflecting systems the user has to do the alignment. The settings are stored and can be recalled at any time.



| AIS position | Diameter (µm) | Magnification range |
|--------------|---------------|---------------------|
| 1 | 600 | 4k - 16k |
| 2 | 300 | 20k - 31k |
| 3 | 150 | 40k - 63k |
| 4 | 75 | 80k - 125k |
| 0 | 37.5 | ≥ 160k |

Fig.: 5 - 22 AIS 5 hole aperture

- Decrease magnification by 3 steps → 80k with knob (3).



- Open the PD Menu *Alignment* and click on *AIS*.
 - ***Auto Mode Change*** has to be selected.
- Select *AIS = Auto*.
- Select *Lower* in the group box *Parameter Adjust*.
- Select mag 80k with the slider or with the knob on the left hardpanel.
 - AIS # 4 is selected automatically.

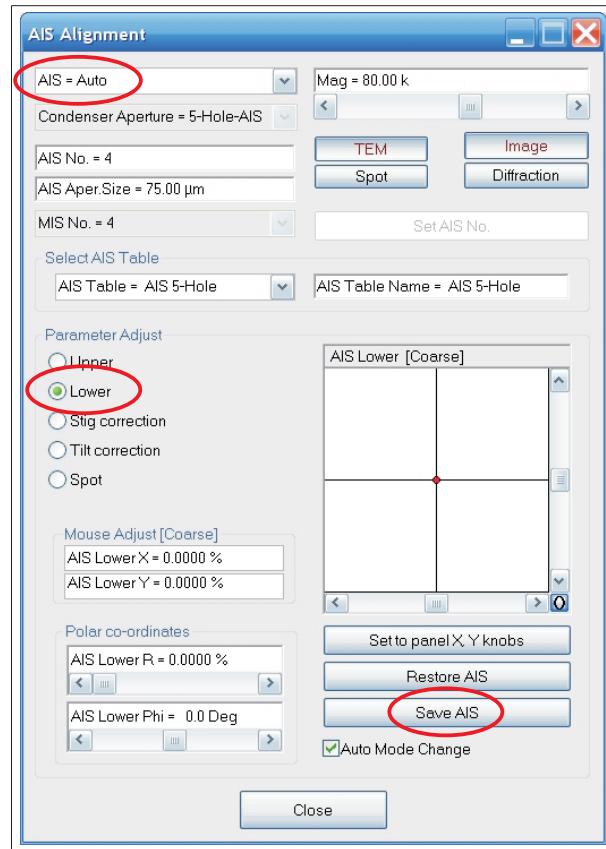
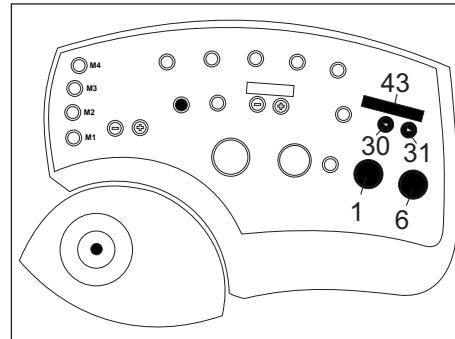
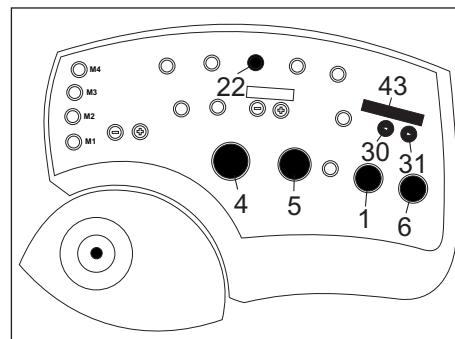


Fig.: 5 - 23 AIS alignment window

- Center AIS aperture # 4 with knobs (1) and (6) on the right hardpanel.
 - The deflecting system which is selected in the *Parameter Adjust* box is automatically shown on the display (43).
- Save settings with *Save AIS*.



- Click on *Stig correction* in the *AIS Alignment* dialogue box.
 - *Diff* mode is selected automatically.
- Focus diffraction spot with knob (4).
- Compensate astigmatism of the diffraction spot with buttons (1) and (6).



- Select *Tilt correction* in the group box *Parameter Adjust*.
- Center diffraction spot to the index point with knobs (1) and (6) on the right hardpanel.
- Save settings with *Save AIS*.

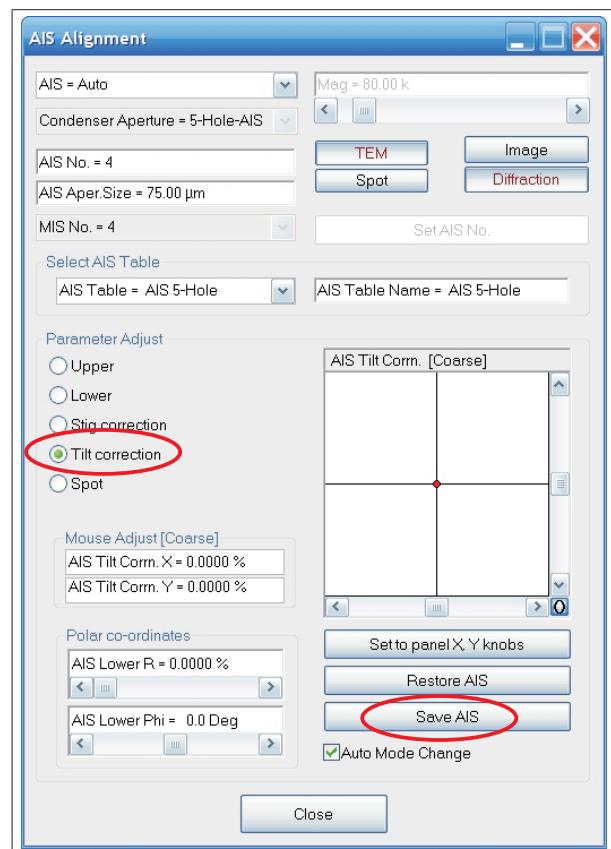
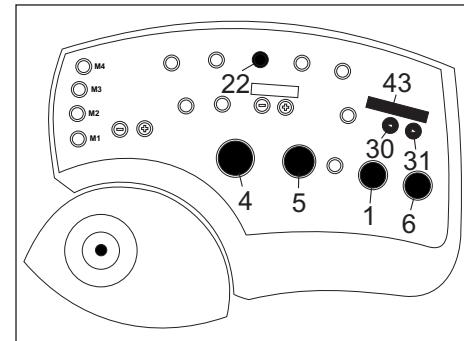


Fig.: 5 - 24 AIS alignment window

- Switch to *Img* mode with button (22).
 - *Img Shft* is shown on the display (43) as the default setting.

**NOTICE:**

AIS aperture 3, 2, and 1 are centered in accordance. # 3 at 40k, # 2 at 20k and # 1 at 10k.

5.8 BIO AIS aperture adjustment

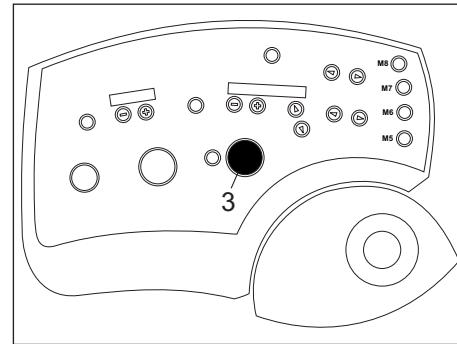


NOTICE::

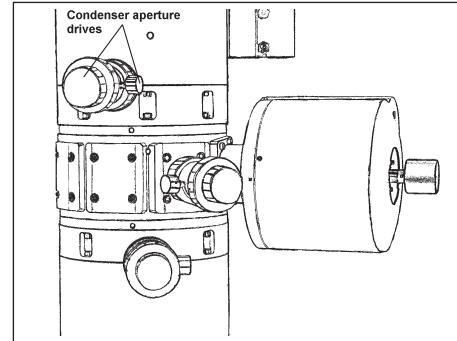
The BIO AIS aperture has its largest hole in the centre. Therefor this aperture can remain inserted in all imaging modes. For a defined spot size a single hole 50 µm aperture in one of the other clickstop positions is used.

The assignment of the apertures to the proper Mag is already done in the factory. So, the AIS alignment can be done in automatic mode.

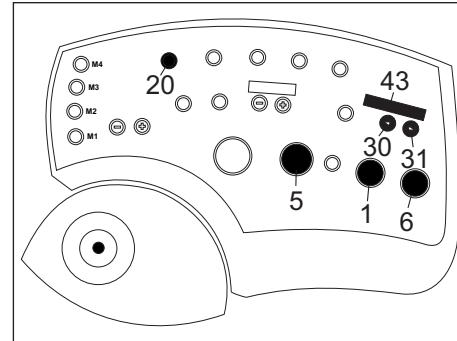
- Set magnification (3) to 5.000x and brightness (5) to 0.5 mrad.
- Select A/S Off in the TEM tab sheet of the WinTEM main menu.



- Insert the central AIS aperture by turning the condenser aperture drive to the 3rd clickstop.
 - The 800 µm AIS aperture is visible on the screen.
- Adapt the mag that the aperture is just a bit smaller than the large viewing screen.

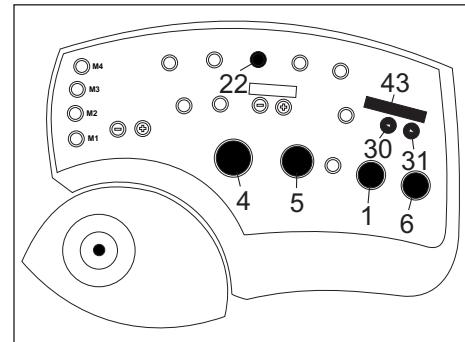


- Switch to SPOT mode with button (20) and center SPOT to the index point with knobs (1) and (6).
- Switch to TEM mode with button (20) and center the 800 µm aperture to the large screen.



- Go up with the magnification to 125 k with knob (3) on the left hardpanel.
- Select SPOT mode (20) and center spot to the index point with knobs (1) and (6).
- Select TEM mode (20).

- Select DIFF mode with button (22) and center diffraction spot to the index point with knobs (1) and (6).
 - Display (43) shows *Img Shft.*
- Select IMG mode (22).
 - If the brightness is not sufficient, increase by 1 or 2 steps



- Open the PD Menu *Alignment* and click on *AIS*.
- Select *AIS = Auto*.
 - *AIS No. = 6* is displayed.
 - *AIS Aperture = 50 μm* is displayed.
- Select *Lower* in the *Parameter Adjust* box.
- Center the 50 μm aperture with knobs (1) and (6) on the right hardpanel.
 - If the aperture is larger than the viewing screen, average the centering.
- Select *Tilt correction* and center the Diff. Spot to the index point with knobs (1) and (6).
 - If the Diff Spot is astigmatic, select *Stig correction* first and compensate the astigmatism.

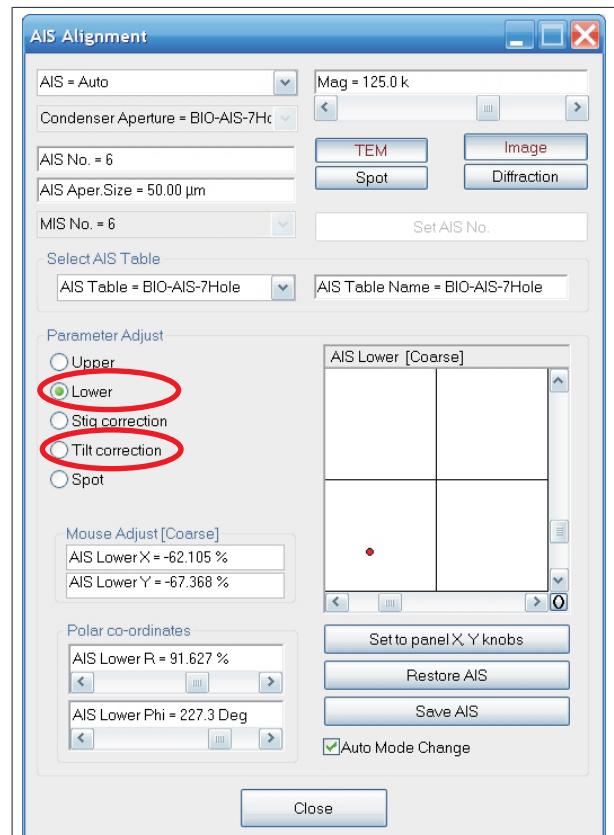


Fig.: 5 - 25 AIS alignment window

- After *Tilt* and *Stig* correction click on *Lower* again and select the magnification step for the next larger AIS aperture according to the list below.

**NOTICE:**

After finishing the alignment at **one** mag step do not forget to store the new settings with “**Save AIS**” before switching to the next mag step!!

The next aperture is # 5 which is automatically selected at 80k followed by # 4 at 50k etc.

| AIS position | Diameter (μm) | Magnification range |
|--------------|----------------------------|---------------------|
| 0 | 800 | $\leq 10\text{k}$ |
| 1 | 500 | 12,5k - 16k |
| 2 | 320 | 20k - 25k |
| 3 | 200 | 31,5k - 40k |
| 4 | 125 | 50k - 63k |
| 5 | 80 | 80k - 100k |
| 6 | 50 | $\geq 125\text{k}$ |

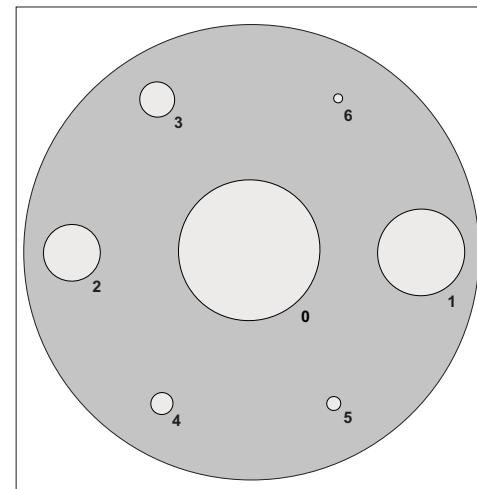


Fig.: 5 - 26 AIS 7 hole aperture

The “AIS upper” setting is done separately, because it might cause more aperture drift due to major changes of brightness.

- Select mag of 125k and click on *Upper*.
- Increase brightness (5) to maximum and optimize it with knobs (1) and (6).
- Save settings with *Save A/S*.
- Select the next lower Mag step (80k) and proceed as above.

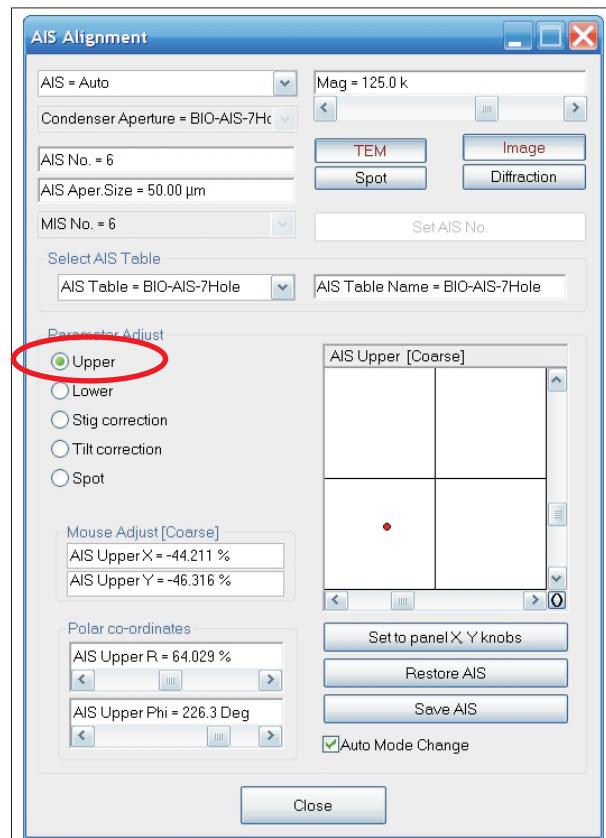


Fig.: 5 - 27 AIS alignment window

5.9 Centering of apertures



NOTICE:

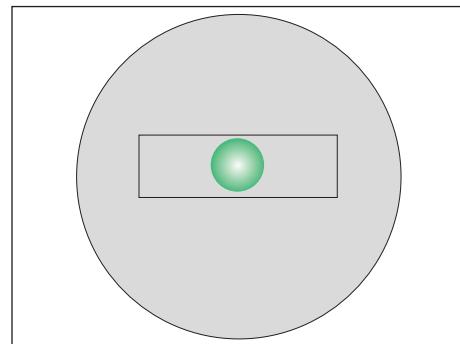
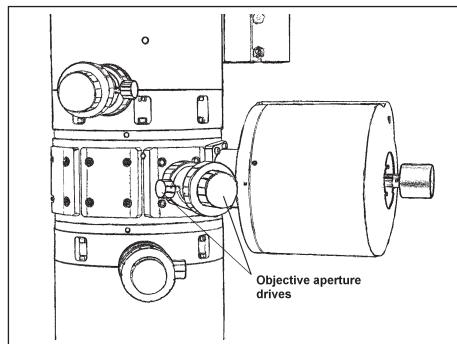
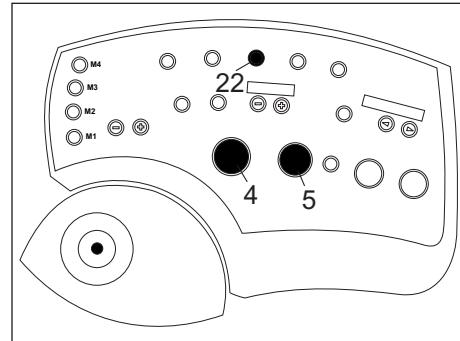
This paragraph comprises the centering of the objective aperture, the intermediate aperture and the spectrometer entrance aperture.

The objective aperture is the most important one, because a non centered aperture results in astigmatic images.

5.9.1 Centering the objective aperture

- Insert a sample into the airlock of the LIBRA 120 (e. g. Carbon film).
 - See paragraph 5.4.2 *Loading the specimen*.

- Push button (22) to turn on DIFF mode.
- Adapt brightness of the diffraction spot with knob (5).
 - Excessive brightness might damage the screen.
- Focus diffraction spot with knob (4).



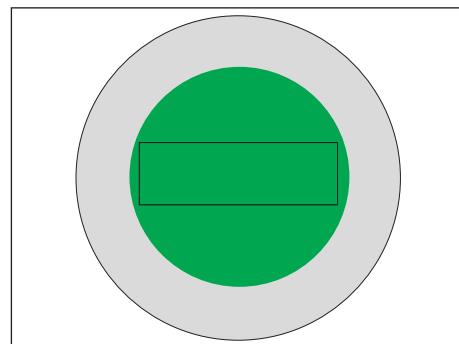
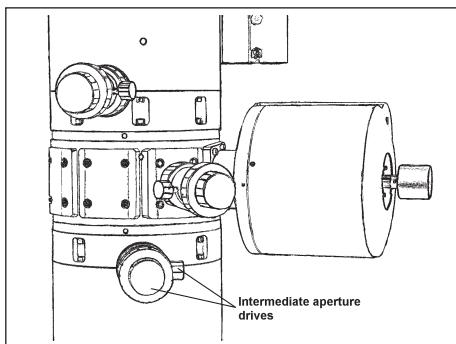
- Insert the desired objective aperture (1st, 2nd, or 3rd click stop) and center it with the aperture drives.
- Push button (22) again to come back to *Img* mode.
- Adjust the appropriate brightness with knob (5).

5.9.2 Centering the intermediate aperture

NOTICE:

The intermediate aperture is used to define the specimen area for diffraction. In LOW MAG mode it serves as a contrast aperture.

The default setting of apertures is 100 µm, 50 µm, and 20 µm which corresponds to the 1st, 2nd, and 3rd clickstop at the aperture holder.

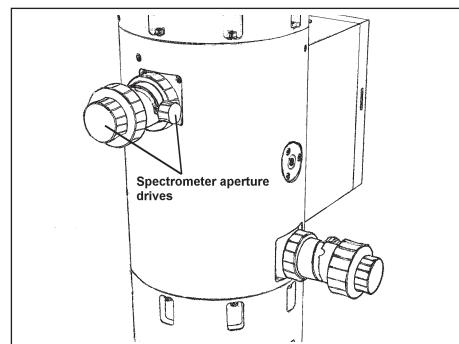


- Insert the desired aperture by turning the big knurled ring and center it with the aperture drives to the viewing screen.
 - The aperture size depends on the specimen area to be diffracted.
 - Using it in LOWMAG range the size depends on the desired contrast.
(Thumb rule: the smaller the more contrast but the more critical, if off-center).

5.9.3 Centering the spectrometer entrance aperture

Centering the spectrometer entrance aperture is identical the intermediate aperture procedure.

- Insert the desired aperture by turning the big knurled ring and center it with the aperture drives to the viewing screen.
 - Even in the left stop position of the big knurled ring the largest aperture is inserted.



5.10 Compensating astigmatism

5.10.1 Compensating the image astigmatism



NOTICE:

Compensating the image astigmatism is one of the most important steps for a perfect image. It has to be carried out on the viewing screen unless there is a digital camera which provides the FFT function.

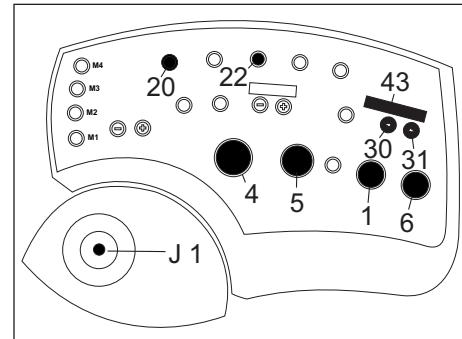
- Increase mag to 160.000x or higher with knob (3) on the left hardpanel.
- Adjust the intensity by changing the emission current step in the *Gun* tab sheet.
 - The group box *Emission current* displays the actual current and step.
- or
- Adjust brightness with knob (5) on the right hardpanel.



NOTICE:

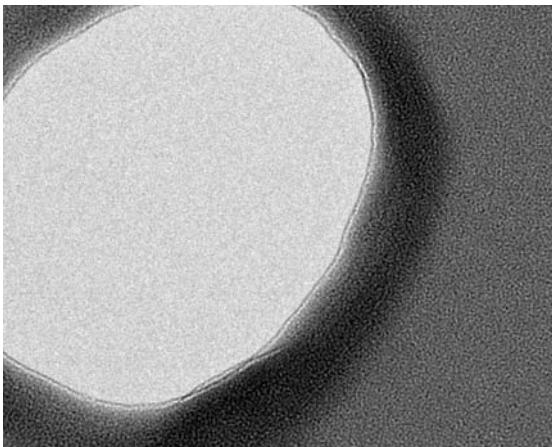
The illumination angle should not be larger than 1.6 mrad. Larger illumination angles decrease the image quality.

- Through-focus specimen with knob (4).
(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 *Obj Stig* (43) with button (30) or (31).
- Compensate astigmatism with the knobs (1) and (6).
 - The astigmatism is compensated, if there is no privileged direction of the grain image in under- and overfocus.
- Switch back to *Img Shift* with button (30) or (31).
- Adjust brightness und magnification to the following application.

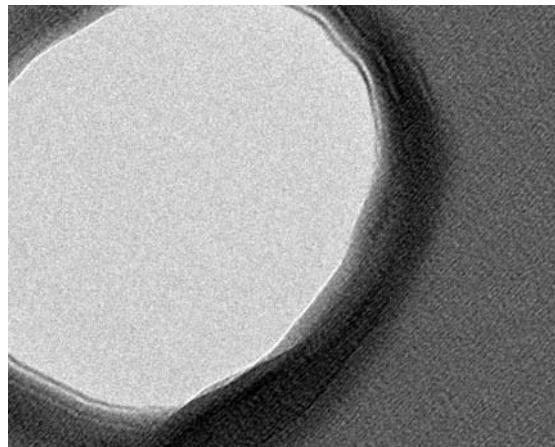


Astigmatism examples on the next page.

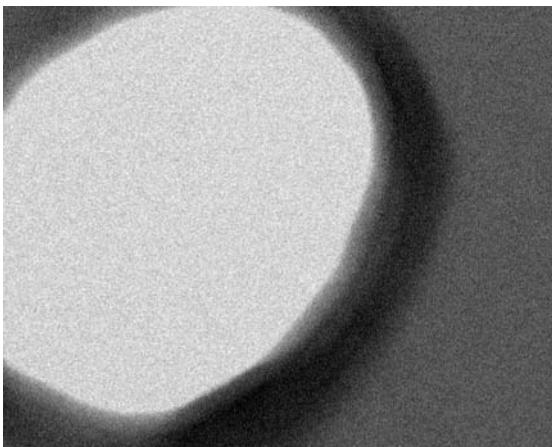
Astigmatism compensated



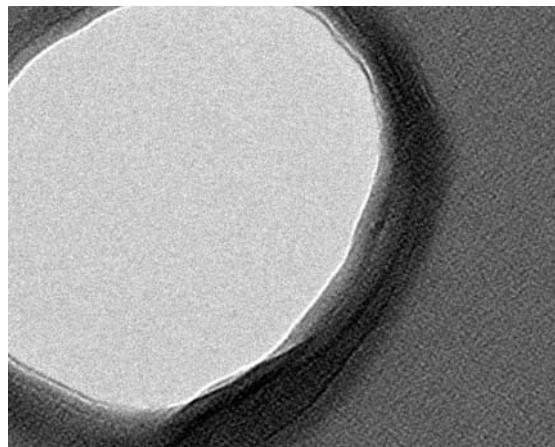
Astigmatism existent



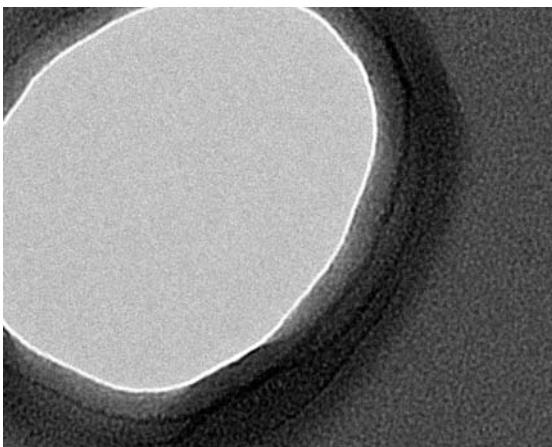
Overfocus



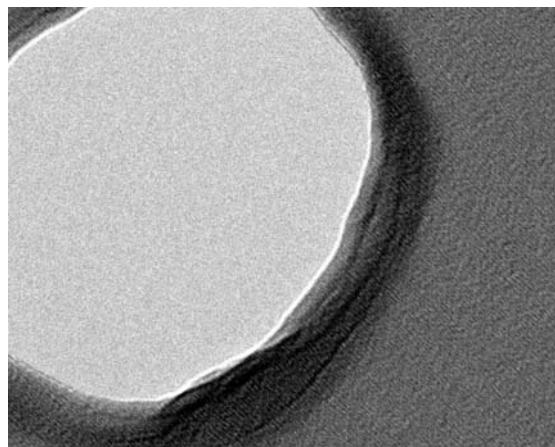
Overfocus



Near Focus (Grain almost disappeared)



Near Focus (Grain still visible)



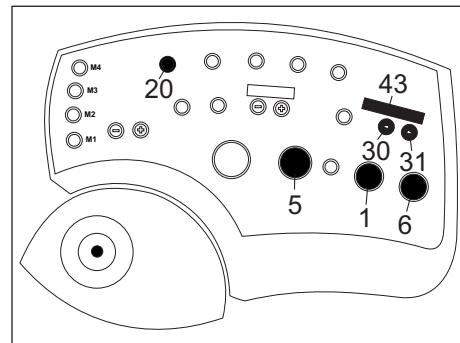
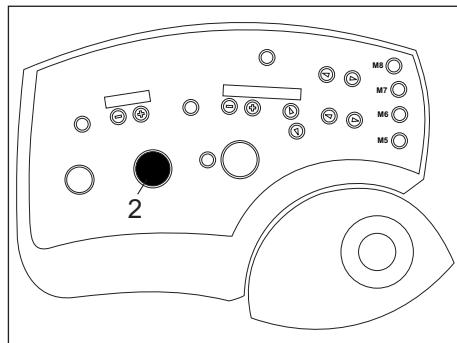
Underfocus

Underfocus

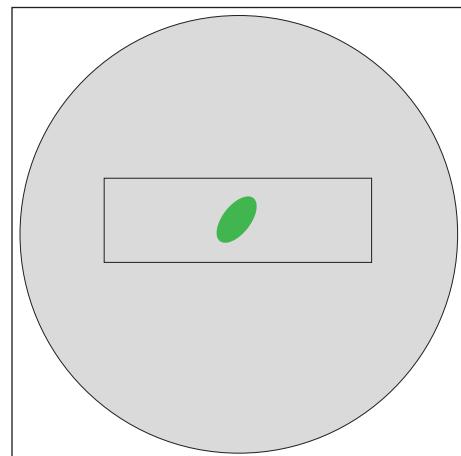
5.10.2 Compensating the illumination astigmatism

NOTICE:

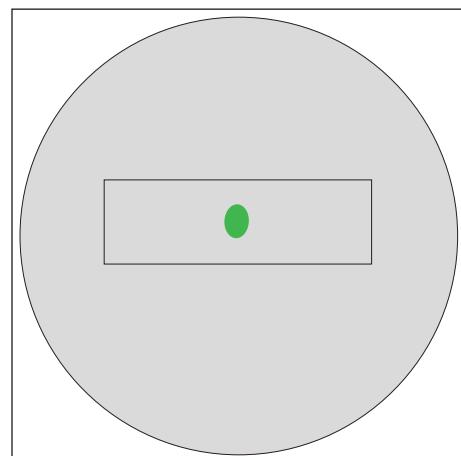
The illumination astigmatism is compensated in Spot mode.



- Switch to Spot mode with button (20).
 - Spot is visible on the screen.
- Through-focus Spot with knob (2).
 - The spot is astigmatic, if it is distorted as shown in the left image.
 - It changes the direction of distortion, when going to the other focus position.
- Select *III Stig* with buttons (30) or (31) on the display (43).



- Compensate illumination astigmatism with knobs (1) and (6).
 - The astigmatism is compensated, when the spot shape is identical in over- and underfocus.
 - **Notice** that the spot is not a circle, when watching it on the small screen.
- Switch to *TEM* mode with button (20).
 - *Img Shft* is shown on the display (43) automatically.



5.11 Focusing the specimen

- Adjust binocular to your eye sight and focus it on the index point of the small screen.
- Move the binocular horizontally until you find a specimen detail for focusing.
- Or
- Move a specimen detail to the index point.

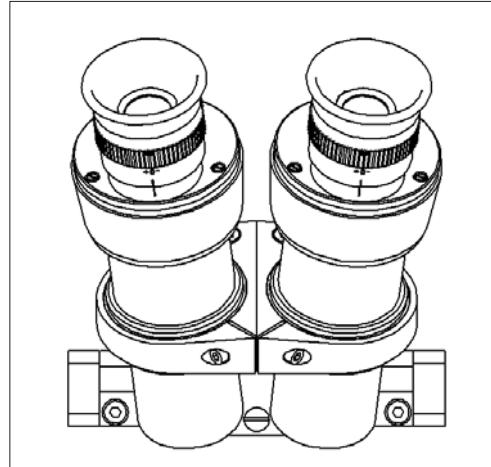
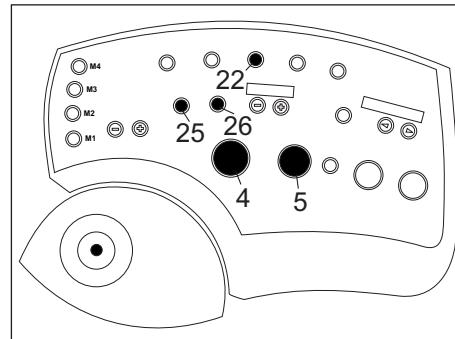


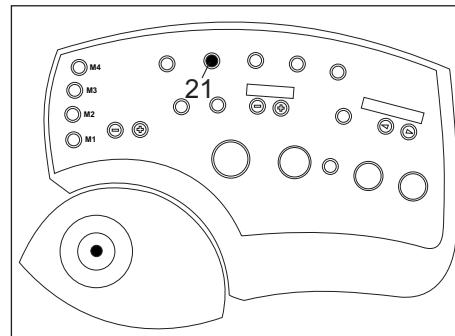
Fig.: 5 - 28 Binocular of the LIBRA 120

- Calibrate objective lens with button (25).
- Turn on *Foc Aid* with button (26).
 - If specimen detail is moving, adjust the eucentric plane (§§ 5.6.6).
- Turn off *Foc Aid* with button (26).
- Focus the sample with knob (4).



5.12 Low Magnifications

- Select LM mode with button (21) on the right hardpanel.
- Retract slit aperture, objective aperture, and condenser aperture (if 5 hole).
- Select magnification between 80x and 2000x with knob (3) on the left hardpanel.
- Adjust brightness with button (5) on the right hardpanel.
- To return to Mag mode carry out the previous steps the other way round.



5.13 ESI imaging modes

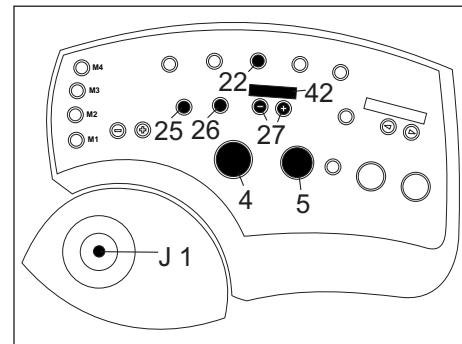
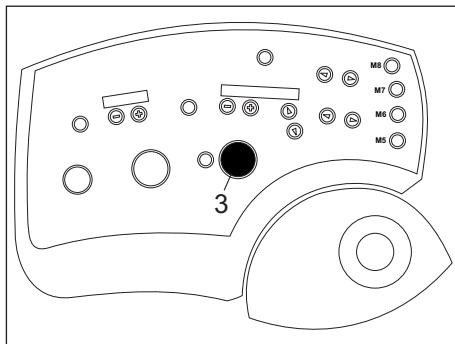
**NOTICE:**

ESI (Electron Spectroscopic Imaging) takes the advantage of the energy filter by using the slit aperture. The name of imaging mode depends on the setting of the energy loss.

5.13.1 Elastic imaging

**NOTICE:**

This mode is working with zero loss electrons only by cutting off the energy loss electrons. It provides the best image contrast in brightfield mode and can be applied on thin and thick samples in brightfield, darkfield, and diffraction mode. As far as biological are concerned they are mostly stained with heavy metals.



- Load a sample into the holder and insert the holder into the goniometer (see 5.4.2) of the LIBRA 120.
- Find the area of interest with joystick (J1).
- Select the desired magnification with knob (3) on the left hardpanel.
- Adjust eucentric plane according to 5.6.6 and spectrometer according to 5.6.3.
- Adjust the brightness with knob (5) and focus the sample with knob (4).
 - Make sure that *Mag Dep* is selected on the display (42).
 - For focusing in zero loss turn to the left (underfocus)
- Start the acquisition with the digital camera or start the exposure with the sheetfilm camera.

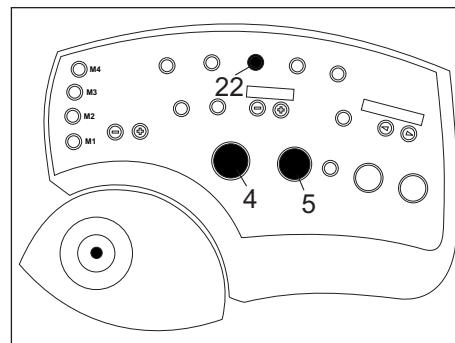
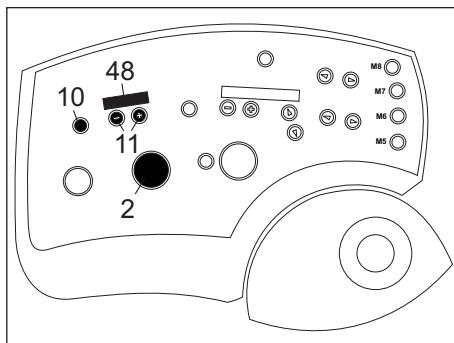
5.13.2 Inelastic imaging


NOTICE:

This mode uses inelastically scattered electrons. In general the samples are very thin (<50nm) and unstained. The energy loss is set to 250 eV (below the Carbon edge) to get the highest contrast by element and mass thickness contrast.

Since the brightness of inelastically scattered electrons is some magnitudes lower than in zero loss, the bombardement with electrons is very high. Therefor the sample should be much more stable than usually. A very small mesh size (700 mesh) provides the stability.

- Load a sample as for elastic imaging 5.4.2 and select Mag with knob (3).
- Find the area of interest with joystick (J1) and move it to screen center.
- Adjust eucentric plane according to 5.6.6 and spectrometer according to 5.6.3.



- Increase the energy loss to 30 eV by pushing button (11+).
 - The brightfield image turns to darkfield.
 - The contrast is much better due to mass thickness differences.
- Adjust the brightness with knob (5) and focus the sample with knob (4).
 - In energy loss only the Gaussian focus provides a sharp image.
- Push button (10) until 250 eV is shown on display (48).
- Adjust brightness (5) and focus the sample (4) by using a Slow Scan CCD camera.
 - At this energy loss the brightness on the viewing screen is too low for focusing.
- Acquire the final image with a reasonable signal.
 - The exposure time is mostly several seconds because the brightness in energy loss is very low.
 - For a sheetfilm exposure the Kodak film SO 163 only features sufficient sensitivity.

5.13.3 Contrast tuning of thick specimens


NOTICE:

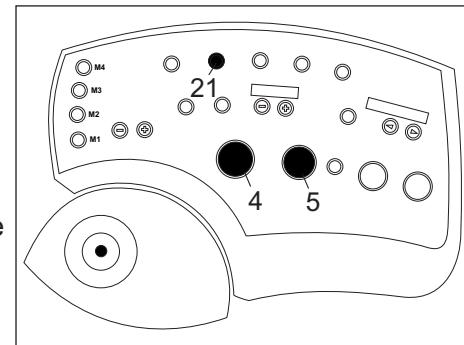
Thick samples often show great differences in mass thickness. Therefor Imaging at the most probable energy loss is recommended (> 0 eV). Here the mass differences are much smaller without losing the resolution.

Because of the strong heat absorption the 30 μm objective aperture should be used to minimise the beam damage.

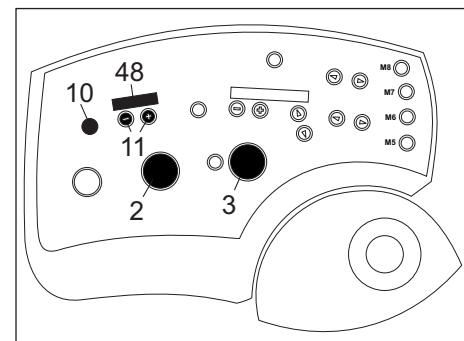
In addition pre-radiation in LowMag, a small mesh size grid, a sandwich grid, or supporting film on the grid will contribute to stabilisation.

This procedure is only necessary for biological specimens which were embedded in resin.

- Insert the sample and switch to LowMag with button (21).
- Adjust medium brightness with knob (5) to get the stabilisation effect.
- After 5 to 10 minutes switch back to Mag mode with button (21).
- Set up the microscope as for elastic imaging 5.13.1.



- Increase ΔE with button (11+) to the most probable energy loss (50 to 200 eV).
 - The most probable loss is easy to find with a digital camera providing the online histogram function.
 - Without a digital camera the shortest exposure time can be measured on the small screen.



- Select magnification with knob (3).
- Focus the specimen with knob (4).
- Adjust brightness with knob (5).
- Acquire the image either with the digital camera or with the sheetfilm camera.

5.14 Diffraction modes

5.14.1 Selected Area Diffraction (SAD)

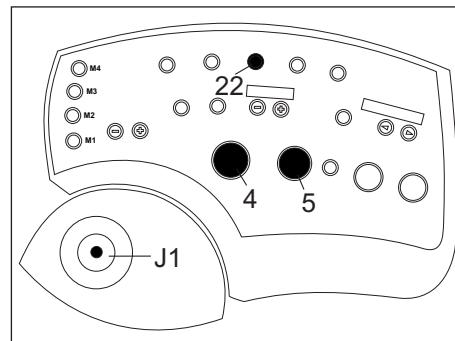
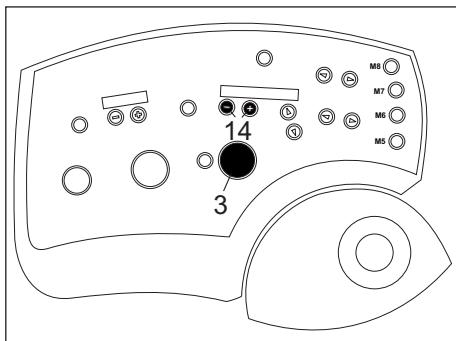
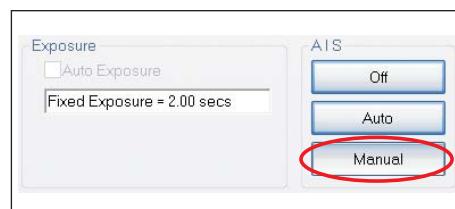


NOTICE:

In general the selected area (SA) for diffraction is defined by the size of the intermediate aperture.

But Köhler illumination can use the condenser aperture to define the field of illumination and therefore the field of diffraction. This is very much recommended since the number of apertures is higher and the precision of the selected area is so much better.

- Select A/S = *Manual* in the group box A/S which is part of the *TEM* tab sheet.



- Select a reasonable magnification with knob (3) and move the area of interest to the center of the screen with J1.
- Select a condenser aperture with buttons (14) to define the field for diffraction in the specimen plane.
- Insert the spectrometer slit aperture as described in 5.6.3.
- Switch to DIFF mode with button (22).
 - Diff* mode is highlighted.
- Focus the diffraction pattern (zero beam) with knob (4).
- Retract objective aperture, if inserted.
 - The diffraction pattern of the selected area is displayed on the viewing screen.
 - The filtered diffraction pattern provides more information because of the blocked inelastically scattered electrons.

**CAUTION:**

The zero order diffraction spot is very bright. Therefor the viewing screen can be damaged. Decrease brightness or insert the beam stop.

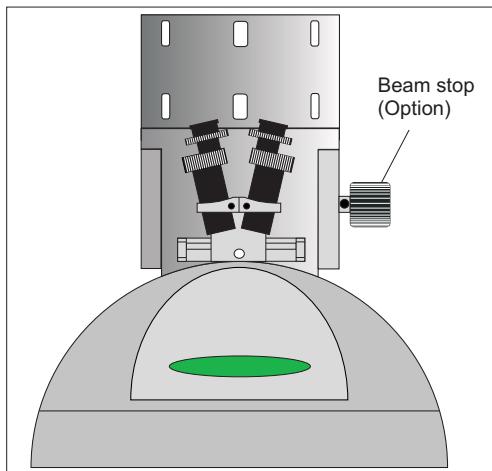


Fig.: 5 - 30 Mechanical beam stop

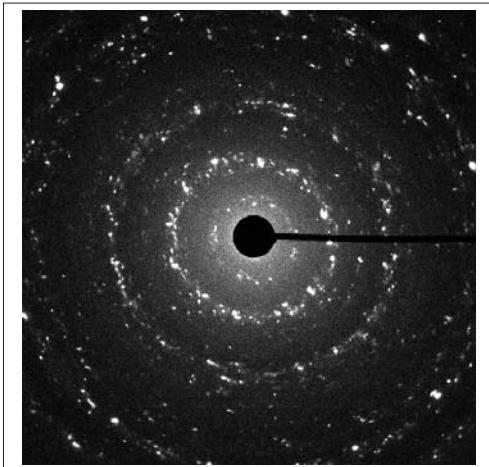


Fig.: 5 - 29 Diff pattern with beam stop

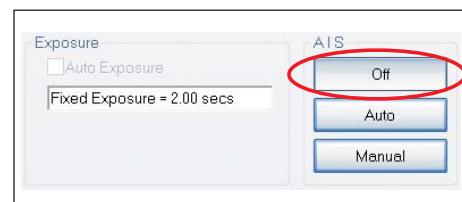
- Select the camera length CL to adapt the diffraction pattern to the camera format.
- Adjust the beam stop that the zero order spot is stopped (see Fig.: 5 - 29).
 - Stopping the zero beam also equalises the dynamic range of the diffraction pattern.

5.14.2 Convergent Beam Electron Diffraction

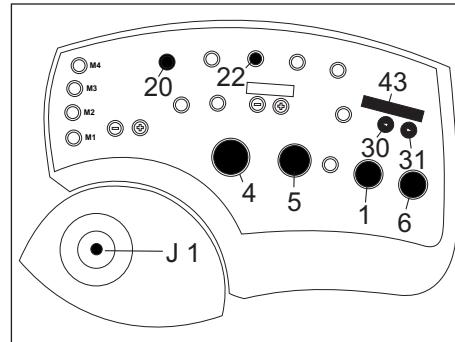
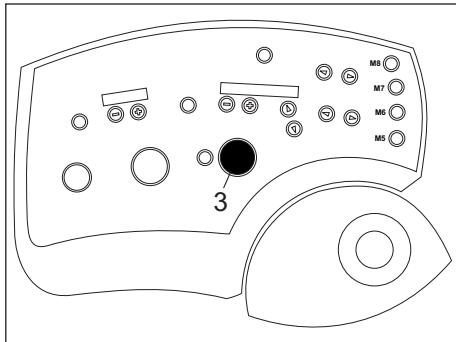
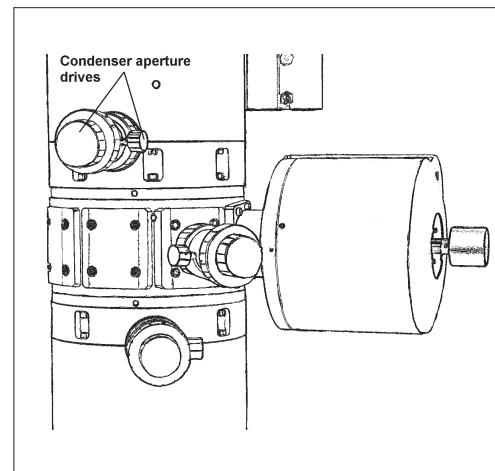
**NOTICE:**

CBED over all other diffraction techniques generates diffraction patterns from very very small regions. The convergent beam is formed in spot mode down to 1,6 nm spot size.

- Select *A/S = Off* in the group box *A/S* which is part of the *TEM* tab sheet.



- Take out the condenser aperture by turning the large ring to the left clickstop.
- Move the sample to the center of the viewing screen with joystick J1.
 - Basic alignment of the LIBRA 120 is provided.
- Insert a condenser aperture $\leq 37\mu\text{m}$ and center it.
 - The aperture size defines the size of the diffraction disks.



- Switch to SPOT mode with button (20) and move spot to the index point with knobs (1) and (6).
 - Make sure that */// Shft* is selected on display (43) before moving the spot.
- Switch to TEM mode (20) and center the selected condenser aperture mechanically.
- Insert the spectrometer slit aperture as described in 5.6.3.
- Take out the objective aperture, if inserted.
- Switch to SPOT mode with button (20) again and push the *Cal* button on the right hardpanel.
 - The spot is focused on the sample.
- Check with knobs (1) and (6) that the sample region is illuminated by the spot.
 - To prove this the TEM illumination can help.
- Adjust the spot size to the desired setting with knob (5).
- Switch to *Diff* mode with button (22).
 - The convergent beam diffraction pattern is displayed on the viewing screen.

5.14.3 Low Angle Diffraction


NOTICE:

This diffraction mode can be applied for spacings from 100 nm to several microns.

- Insert the sample into the goniometer.
- Select the LM range and center e. g. the central AIS aperture (37,5 µm).
 - In LM mode AIS is off. Only condenser apertures on the axis can be used.
 - About 1 mesh of the grid type 200 is illuminated.
 - The objective aperture is out.
- Select Mag 1000x (for this sample).
- Focus the sample with Focus knob (4) on the right hardpanel.
- Reduce brightness with knob (5).
- Insert the slit aperture.
 - Contrast enhancement by eliminating inelastic scattering.
- Switch to DIFF mode with button (22).

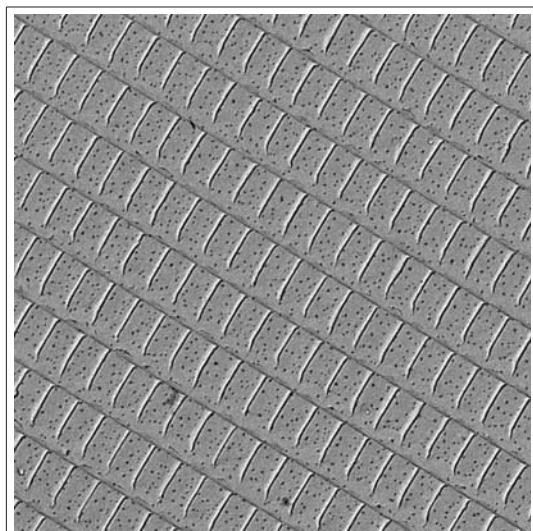


Fig.: 5 - 31 Replica with 0.82/1.64 µm

- Adapt the brightness that the pattern is not overexposed.
- Focus diffraction pattern with knob (4).
- Insert the beam stop (Option) to blank the zero order beam.
- Acquire the diffraction pattern e. g. with SSCCD camera.

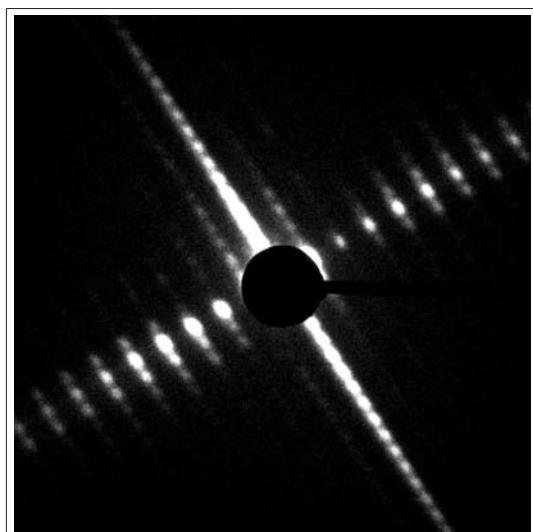


Fig.: 5 - 32 LA Diff pattern from the sample

5.15 Darkfield modes

5.15.1 Darkfield with carthesian coordinates

**CAUTION:**

Since the zero order diffraction spot is hitting the objective aperture, be careful with the brightness in darkfield imaging. The beam can melt the aperture, if staying too long in darkfield mode at very high brightness.

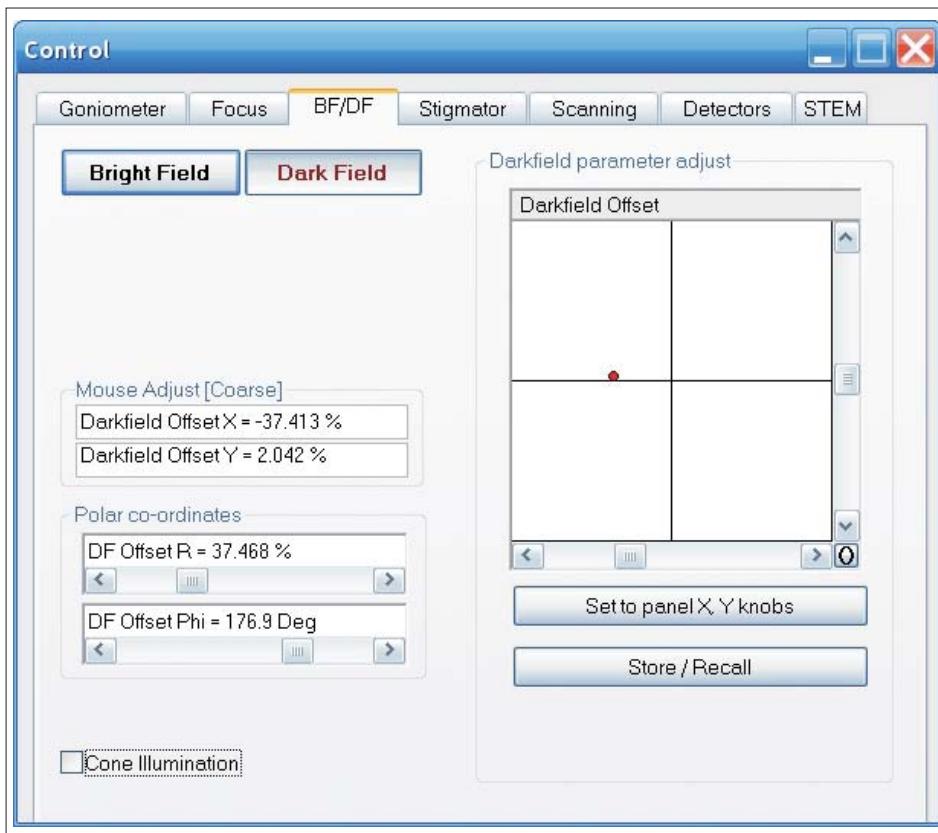
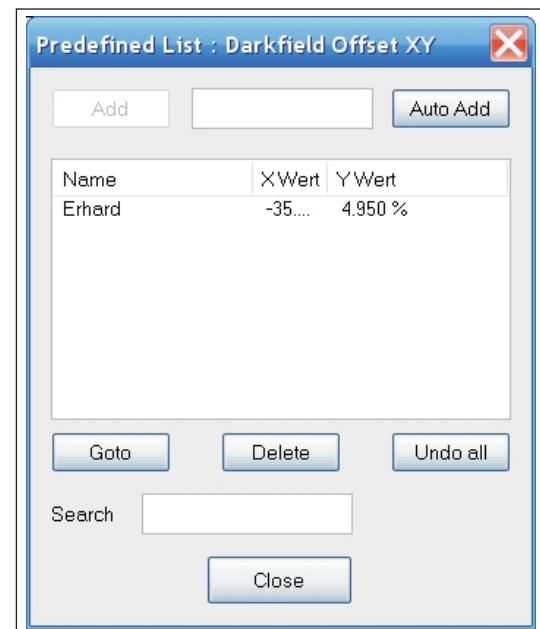


Fig.: 5 - 33 Control menu with opened Tab sheet *BF/DF*

- Open tab sheet *BF/DF* and select *Dark Field* mode.
- Switch to *Diff* mode and move the zero order beam with *Darkfield Offset* out of the objective aperture.
 - That is the basic condition to get a darkfield at all.

- Shift the desired diffraction spots or rings into the center (index point) of the objective aperture.
 - The setting can be edited and stored with *Store/Recall*.
 - Further settings can be stored and recalled from the *Predefined List*.
- Switch back to *Img* mode and adjust the brightness carefully.

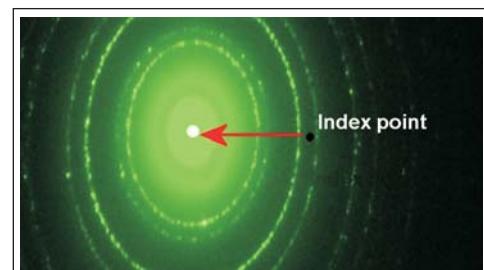
- Focus the image with the *Focus* button on the right hardpanel.
 - The only possible focus in darkfield is the Gaussian focus.
 - The image can also be filtered as every other image.
- Click on one of the presettings in the list to recall different coordinates.



5.15.2 Darkfield with cone illumination

- Open tab sheet *BF/DF* and select the *Dark Field* mode.
- Click on *Cone Illumination* in the tab sheet *BF/DF*.
- Switch to *Diff* mode.

- Increase the diameter of the diffraction circle with DF Offset that it is blocked by the objective aperture.
 - Thus the brightfield image is blocked by the aperture and a rotating darkfield appears.
- Switch back to *TEM* mode and adapt the brightness carefully.



- Insert objective aperture and center it to the index point.
 - The objective aperture should be small ($60 \mu\text{m}$ or $30 \mu\text{m}$) to use the desired spots or rings for the dynamic darkfield image only.

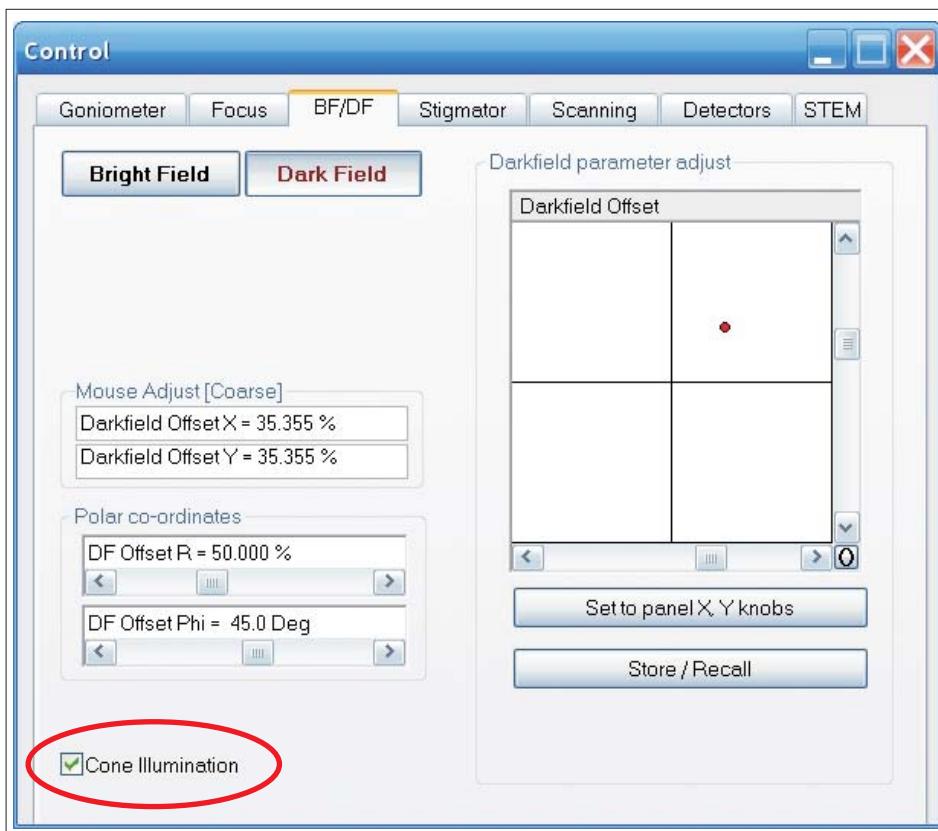
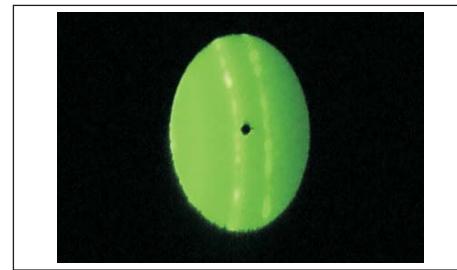


Fig.: 5 - 34 Tab sheet *BF/DF* in the Control menu

- Activate *Cone Illumination*.
- Switch back from *Diff* mode to *TEM* mode and adjust the brightness.
- Focus the darkfield image with knob *Focus*.
 - Since the darkfield image is not a static one, it seems to flicker on the viewing screen.
 - The focus is optimised, when the movement (flickering) is minimised.
 - The flickering can be integrated with the SSCCD camera by applying longer exposure times.

5.16 Low Dose Imaging



NOTICE:

Low Dose Imaging is using deflecting systems above and below the sample to minimise the electron dose for the acquisition. Before using this mode the MDF (Minimum Dose Focusing) system has to be set up.

5.16.1 MDF setup

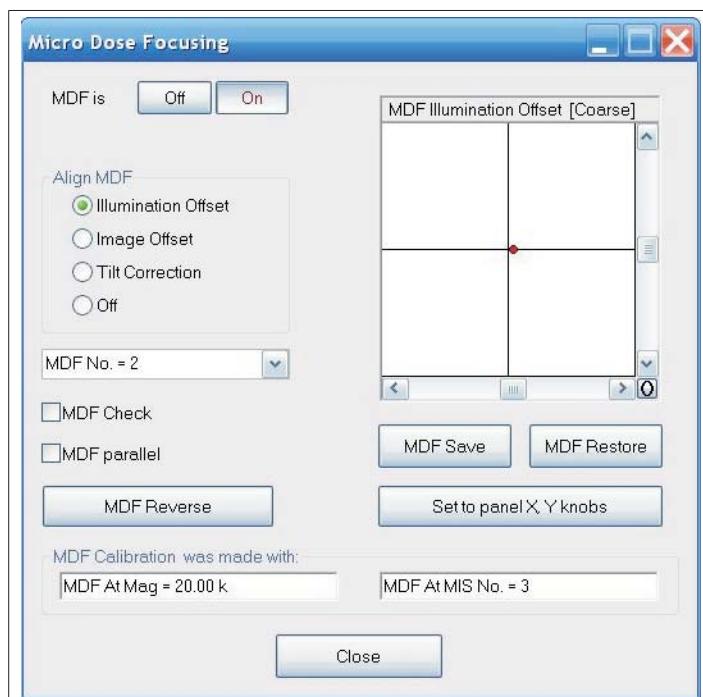


Fig.: 5 - 35 MDF setup menu

- Move cursor to PD menu *Control* in the main menu and open the *MDF* menu by a double click.
- Turn the MDF system to *On* and select an MDF No. 1 - 5.
- Select *Illumination Offset* and move the illuminated field **off center** with the X, Y sliders or with the navigation point (1st step).
 - It is provided that the AIS aperture is inserted and the AIS mode is *Auto* or *Manual*.
- Click on *MDF Save* to store the illumination offset.



NOTICE:

Using the AIS system in manual mode the field of illumination can be adapted to the size of the camera detector. Thus the MDF offsets are smaller which provide a better focus precision.

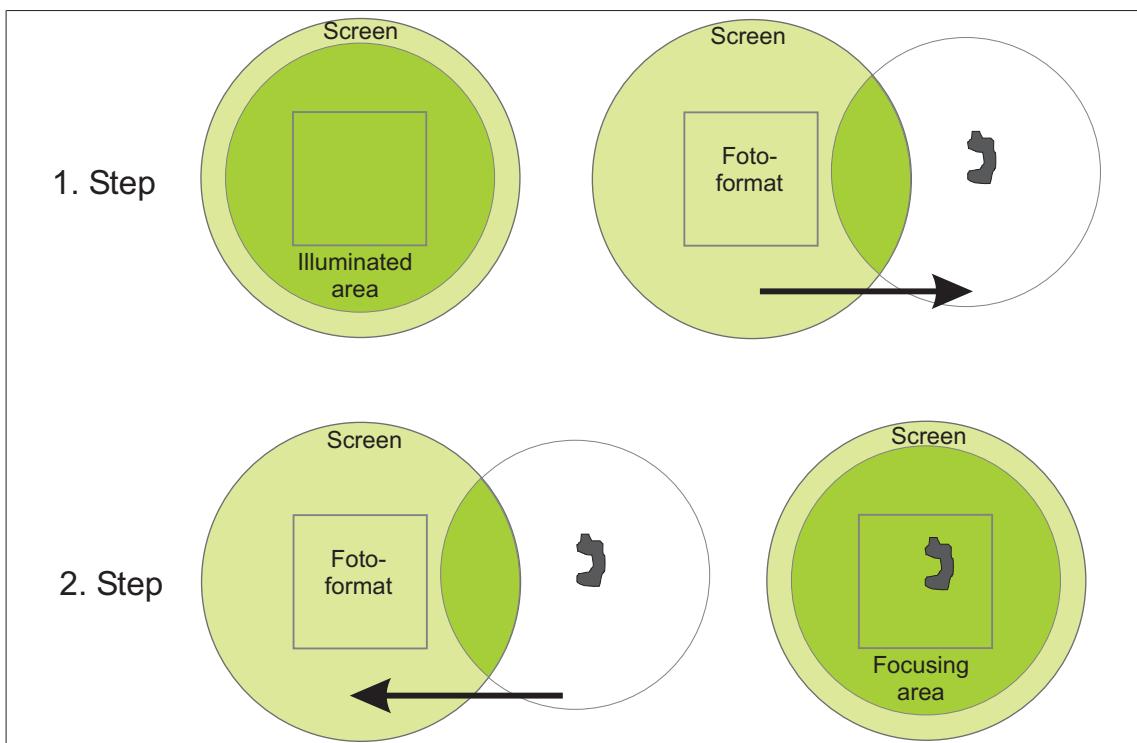


Fig.: 5 - 36 MDF setting

- Click on Image Offset in the MDF menu and move the illuminated off center area back to the center with X, Y sliders or with the navigation point (2. Step).
 - The X, Y knobs on the right hardpanel can also be used to adjust the offsets.
 - Before use click on *Set to panel X, Y knobs*.
- Click on *Save MDF* to store the image offset.
- Select *Tilt Correction* in the MDF menu and switch to *Diff* mode either on the right hardpanel or in the EM mode bar in the main menu.
- Move diffraction spot to the center of the objective aperture with X, Y sliders or navigation point in the MDF dialog box.
 - It is provided that the diffraction spot was centered before MDF was activated.
- Click on *Save MDF* to store the tilt offset.

**NOTICE:**

When MDF is ON, the focusing position is shown on the screen. For image acquisition the MDF mode is turned off by the sheetfilm camera or by the SSCCD camera.

5.17 High Resolution Mode (HR)



NOTICE:

The HR mode is a special illumination mode with excellent coherence and sufficient brightness. That will be achieved by minimising the number of illumination crossovers.

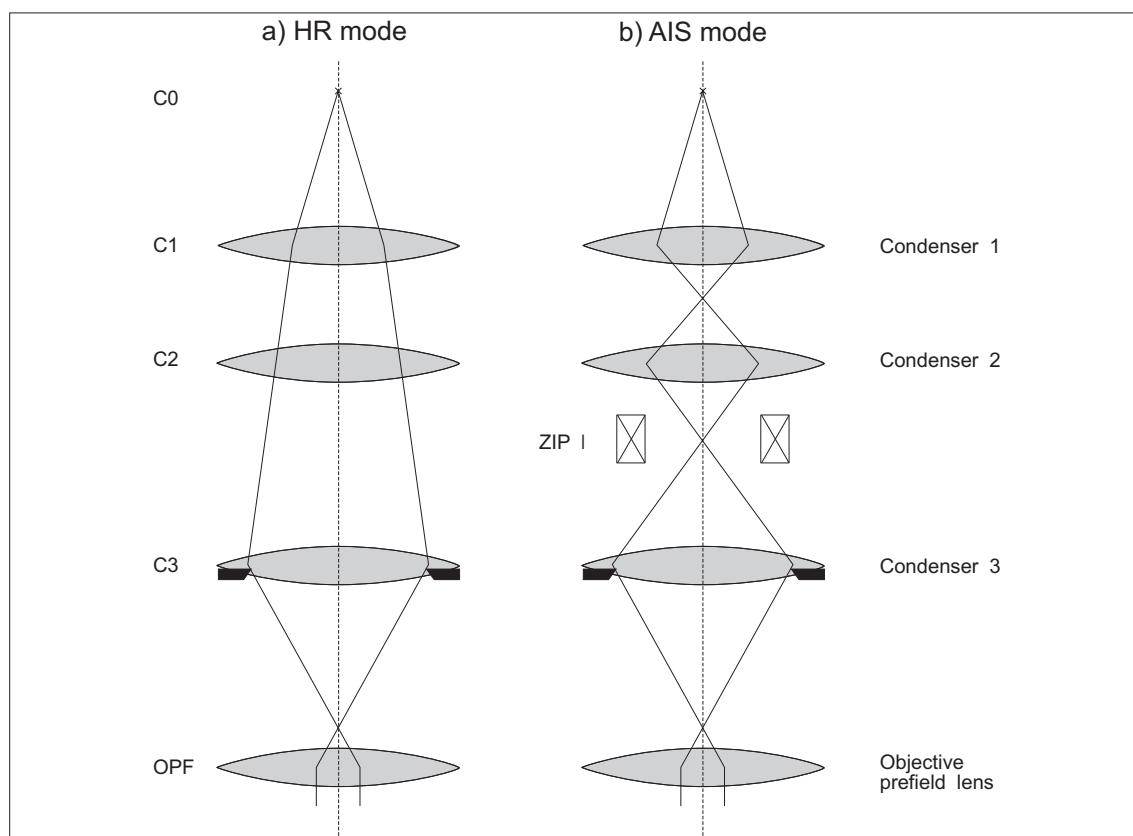


Fig.: 5 - 37 HR mode in the LIBRA 120

The HR mode is only active if the illumination angle is set to 3,2 mrad or higher. As soon as the illumination angle is lower than 3,2 mrad the HR mode is not active.

The Koehler illumination principle is also realised in the HR mode. As shown in the above scheme there is only one crossover in the front focal plane of the prefield lens.

The AIS system is deactivated automatically. So the sample is illuminated through the central AIS aperture.

5.17.1 Turning on the HR mode

- Open PD menu *Alignment* \Rightarrow *Illumination* and activate *High Resolution* mode.

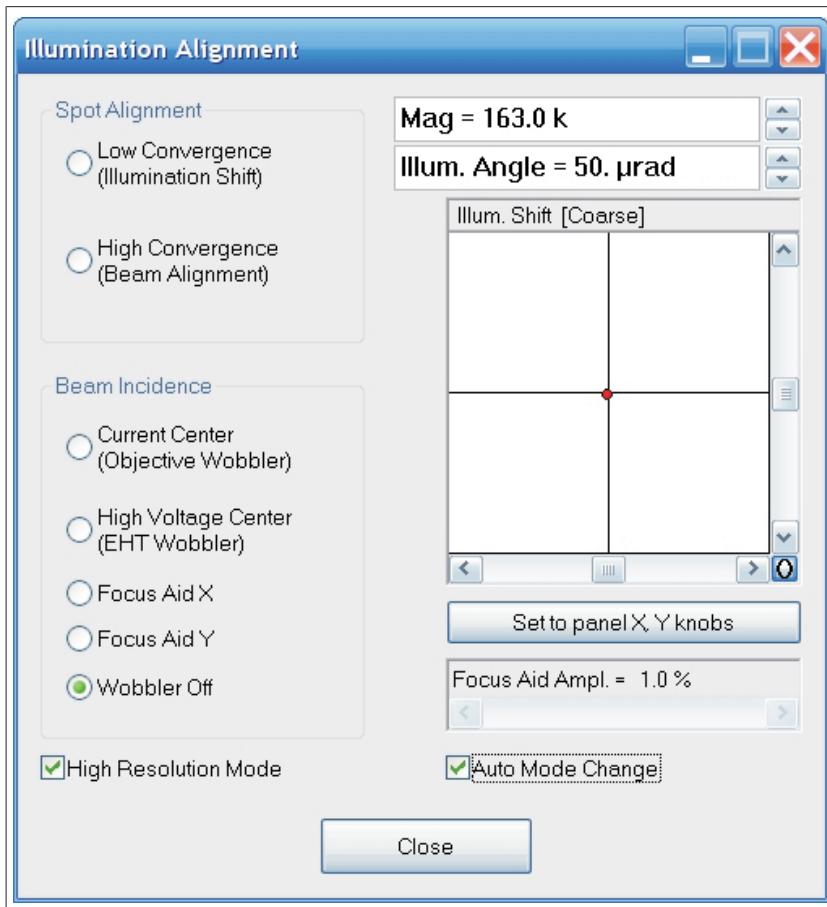


Fig.: 5 - 38 Activated HR mode



NOTICE:

If the HR mode is used more frequently it is recommended to install the function in the status bar. Then the window Illumination Alignment needn't to be opened anymore.
The HR mode can be activated just by clicking on the function in the status bar.

| | | | | |
|----------------------|------------------------|------------------|---------------------------|-----------------------|
| HT = 120.00 kV | VAC Status = not ready | Mag = 163.0 k | Illum. Angle = 1.000 mrad | MIS No. = 6 |
| HT State = Off | User Blank = No | Spec. Mag = 0.00 | Spot Size = 0.0 nm | MDF = Off |
| Emission I is = 4 pA | Blanked = No | CL = 0.000 mm | AIS = Auto | Resolution = High Res |

The function is inserted in the window *Parameter Selection*.

- Make a right click in the status bar.
- The window *Parameter Selection* is opened.

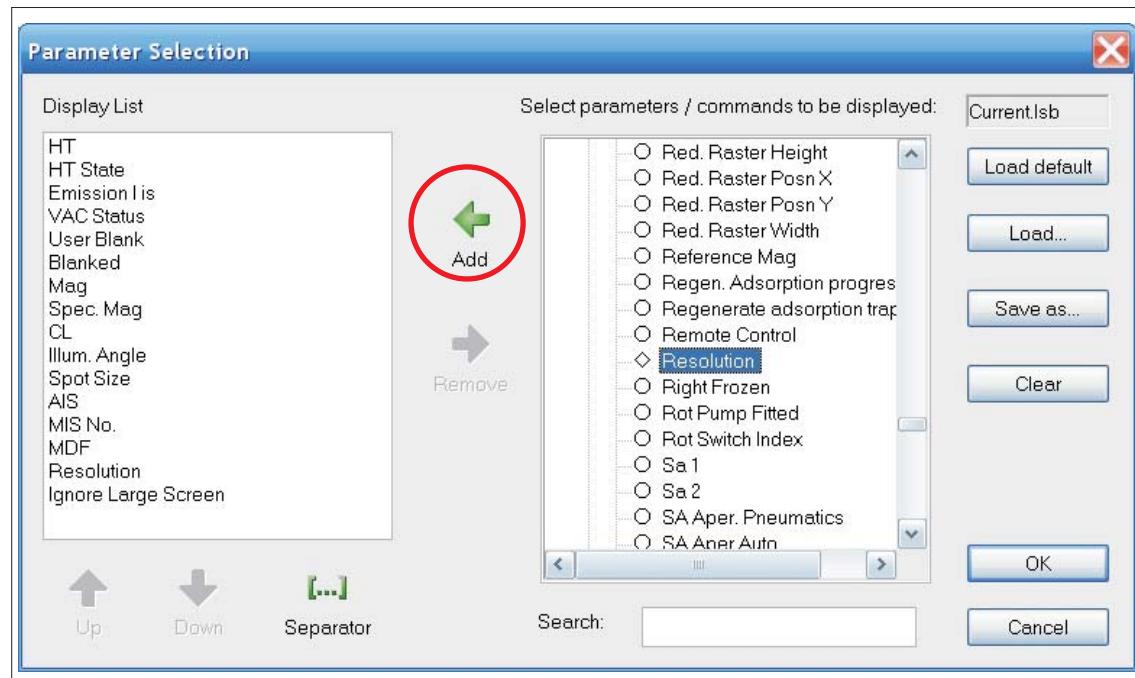


Fig.: 5 - 39 The window *Parameter Selection*

- Search for the function *Resolution* and use the button *Add* to insert it.
- The position in the status bar can be changed by using *Up* or *Down*.

5.17.2 Aligning the HR mode

- Select magnification and increase the illumination angle up to 3,2 mrad.
- Activate the HR mode in the status bar or in the window *Illumination Alignment*.
- Switch to *Diff* mode.
 - The diffraction spot is mostly a little off the center of the objective aperture.
 - It is provided that the diffraction spot is centered in the normal mode.
- Open the tab sheet *Gun* in the WinTEM main menu.
- Activate the function *Gun Tilt*.
 - The *Gun Tilt* values are displayed in the nav box.

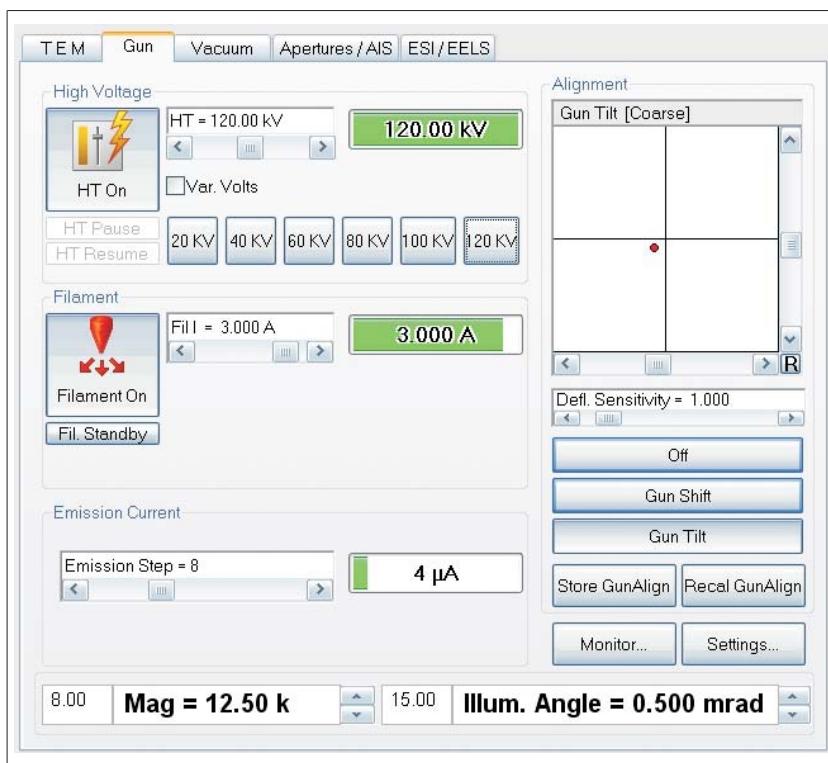


Fig.: 5 - 40 Tab sheet Gun

- Shift diffraction spot to the center of the objective aperture with knobs X, Y on the right hardpanel.
- Store the *Gun Tilt* values with *Store Gun Alignment*.
 - There are different values for the HR mode.
 - The Gun Tilt values for the normal mode are not overwritten.

5.18 EM system Turn-Off

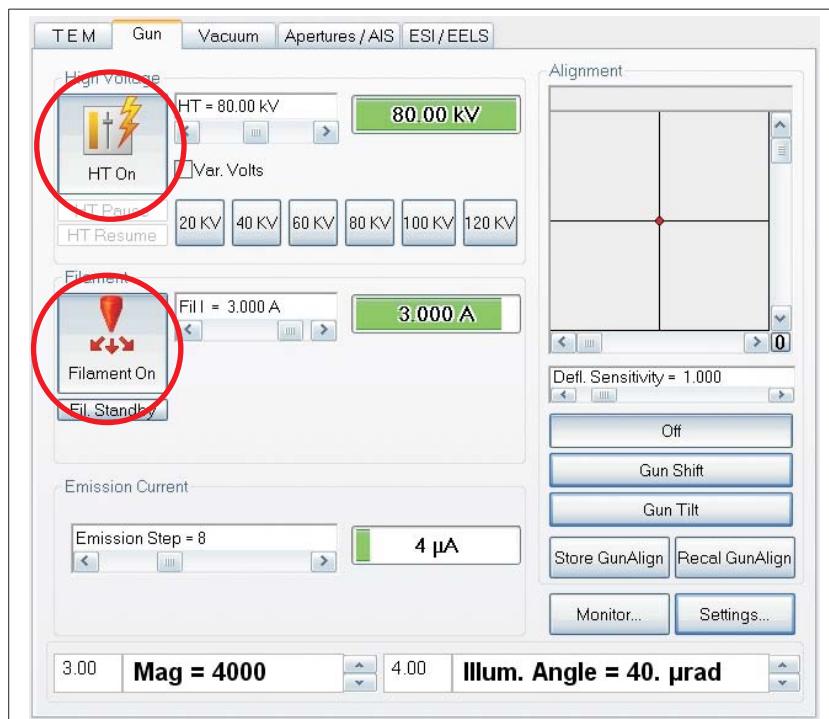


Fig.: 5 - 41 Filament and High Voltage in the Gun Tab Sheet

- Turn off the filament and HT in the tool bar or in the *Gun* tab sheet.
 - The filament and HT is ramping down displayed by the green progress bar.
- Close the WinTEM program and then the WinTEM server.
- Push the yellow Standby button at the console.
 - The power supply for the lenses and electronics is turned off.
 - The vacuum system is kept running.



6 Sheetfilm Camera Operation

6.1 Description

The sheetfilm camera is located below the viewing screens. The exposure procedure is fully automated. The camera is operated and monitored by the WinTEM software.

Loading of films or taking out exposed films is done in red light.



6.2 Loading the film cassette

- Load the film into the cassette as shown on the left side.
 - The film size is 3 1/4 x 4".
 - Watch the mark at the film edge.
 - The film emulsion is facing up with the marking on the upper right position.

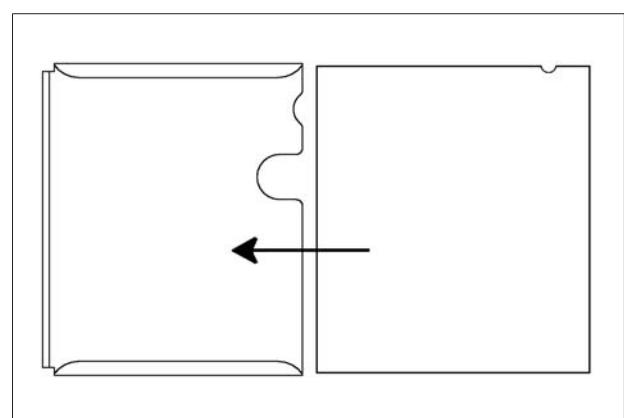


Fig.: 6 - 1 Loading film

6.3 Loading the magazine

- Hold the cassette at side 2 in an oblique position above the magazine.
- Put down side 1 to the bottom of the magazine and then drop side 2.

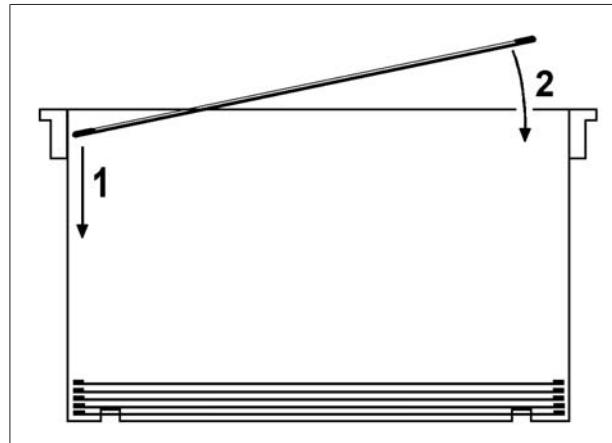


Fig.: 6 - 2 Magazine with cassettes

- Fill up the magazine as shown on the right.
 - **IMPORTANT!**
Watch the orientation of the cassette in the magazine.
 - The magazine can take up to 45 cassettes.

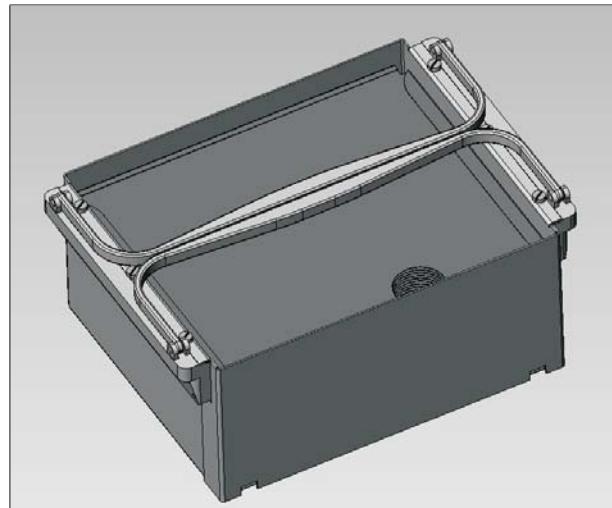


Fig.: 6 - 3 Magazine loaded with cassettes



CAUTION:

A wrong position of the cassettes in the magazine can lead to malfunctions of the sheetfilm camera.

- Put the filled magazine in a desiccator and let it pump over night.
 - For transport use the black, light tight container.

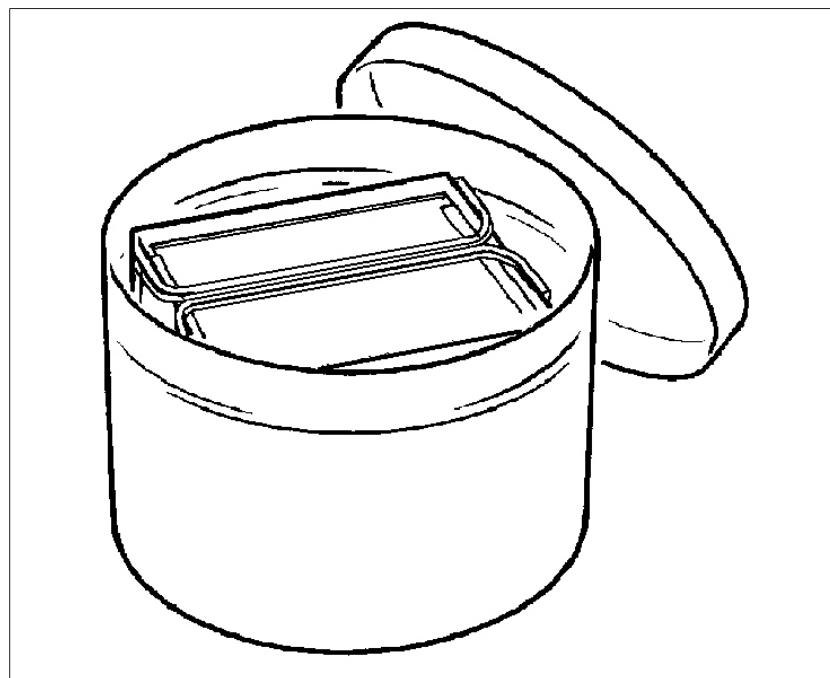


Fig.: 6 - 4 Transport container with magazine

6.4 Ventilating the camera chamber

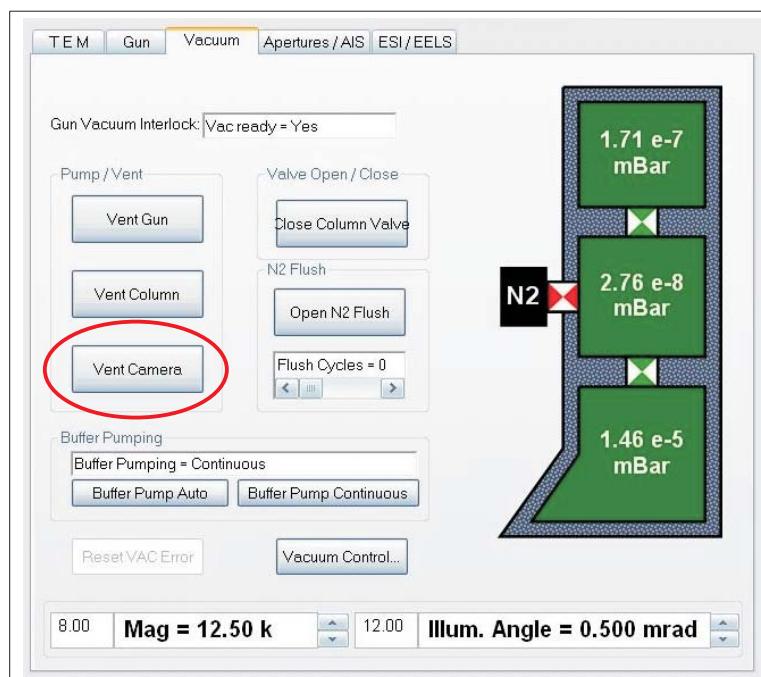


Fig.: 6 - 5 Vac menu

- Click on *Camera Vent* to ventilate the camera chamber for loading the magazine.
 - The camera chamber is ventilated automatically.

- Lift the cover of the sheetfilm camera to load the magazine.



Fig.: 6 - 6 Sheetfilm camera opened

- Take magazine from the desiccator and put it into the sheetfilm camera.
 - Watch the orientation of the magazine !



CAUTION:

As long as the magazine is not in a closed box every action must be done in safety light.

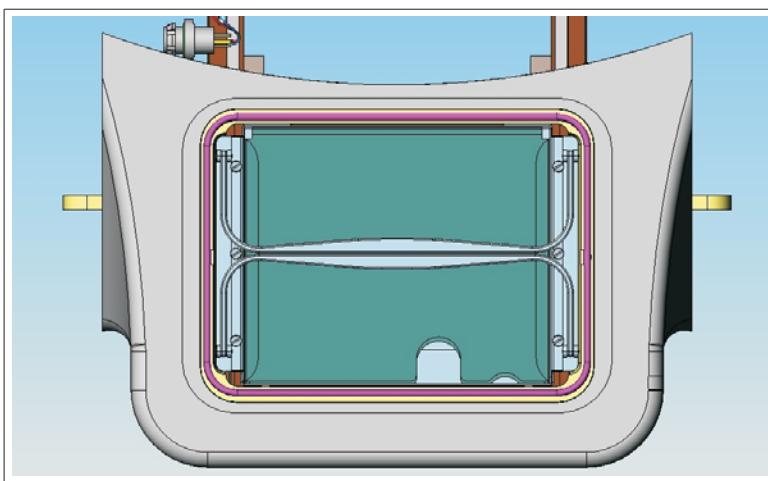


Fig.: 6 - 7 Sheetfilm camera with magazine inserted

- Close the camera with lid and click on *Camera Pump* in the Vac menu.

6.5 Exposing negatives

After the vacuum is *READY* the sheetfilm camera can be used for exposures.

- Open the PD menu *Settings* and click on Camera.
 - The sheetfilm camera status should be *Ready*.

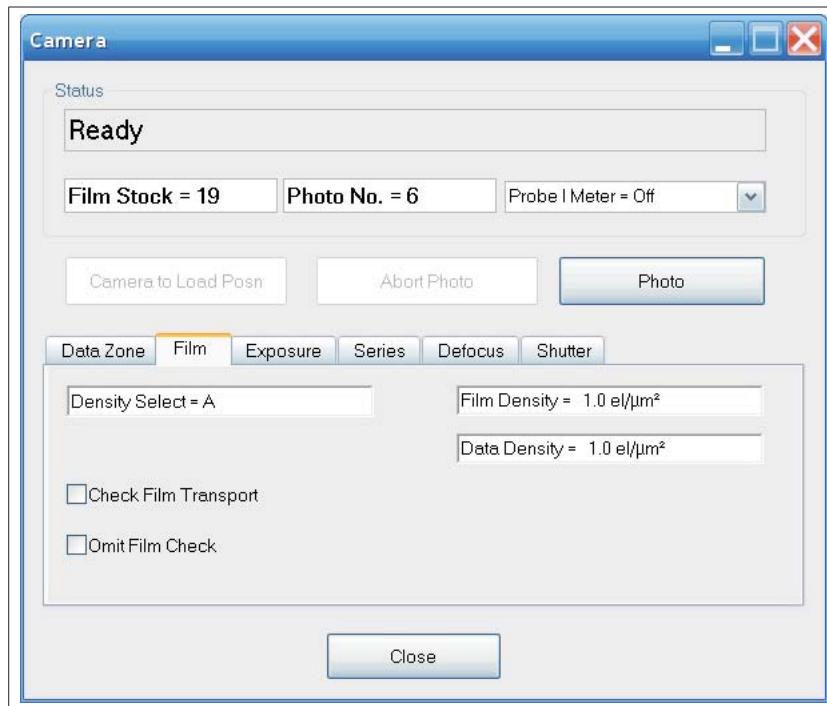


Fig.: 6 - 8 Sheetfilm camera menu with tab sheet *Film*

6.5.1 Preparing the exposure

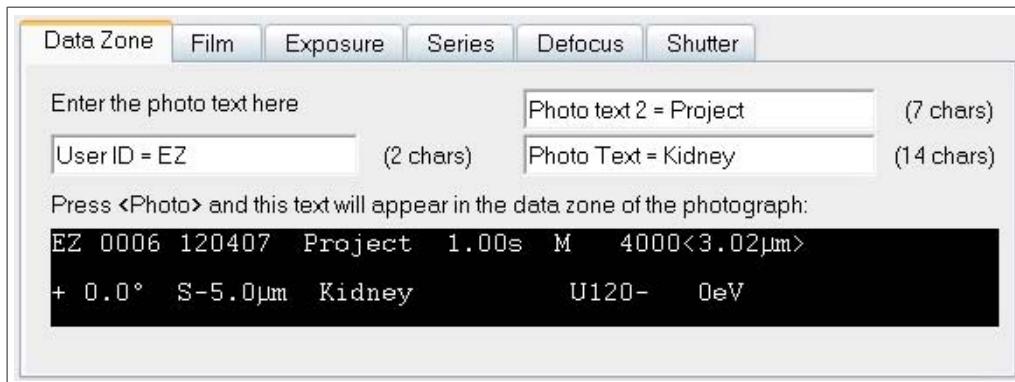


NOTICE:

There are a couple of tab sheets for different settings and selections. The most important ones are *Data Zone*, *Film*, and *Defocus* which are explained in detail in the following.

- Click on the tab sheet *Film* to set film and data density.
 - The above setting can be used for Kodak Sheetfilm SO 163.
 - Settings for other film types have to be figured out by an exposure series.
 - Two different settings can be stored under *A* and *B*.
- Click on the tab sheet *Data Zone* to make inputs for user and sample identification.
- Click in the field *User ID* = and put in a user ID up to 3 char.
- Click in the field *Photo Text* = and put in up to 16 char.

- Click in the field *Photo Text2 =* and put in up to 8 char.

Fig.: 6 - 9 Tab sheet *Data Zone*

| Film # | Text | | Mag |
|------------|---------|------------|--------------------------------|
| User ID | Date | Expos time | Scale |
| EZ | 0006 | 300907 | Project 1.00s M 20000 < 604nm> |
| α 0.0° | L 0.0nm | Kidney | U120- 0eV |
| Tilt angle | Text | | Delta E |
| Defocus | | | HV |

Fig.: 6 - 10 Data Zone of the negative

- Click on the tab sheet *Exposure* to select the exposure mode.

Fig.: 6 - 11 Register *Exposure*

**NOTICE:**

For most of the applications the *Auto Exposure* mode is very much recommended. For diffraction exposures the *Fixed Exposure* mode is being applied.

- Click on the tab sheet *Defocus* to define a defocus mode.
 - If no additional defocus is being applied, select the *Fixed Defocus* mode and set the defocus to zero.
 - If the Objective Wobbler is used for focussing, select an automatic defocus LOW, MEDIUM or HIGH.
 - The calculated defocus is displayed in *Photo Defocus* =.
 - The function *Apply Defocus* shows the automatic defocus on the screen for a short moment. This defocus value is applied for the exposure later.



Fig.: 6 - 12 Tab Sheet "Defocus"

The tab sheet *Series* provides the settings for different series modes.

The following types of series are available:

- Focus
- Exposure
- Delta E
- Tilt

The tab sheet *Shutter* provides the different shutters for the exposure. In general the Gun blanker (Pre Specimen Blanker) is used.

6.5.2 Exposure

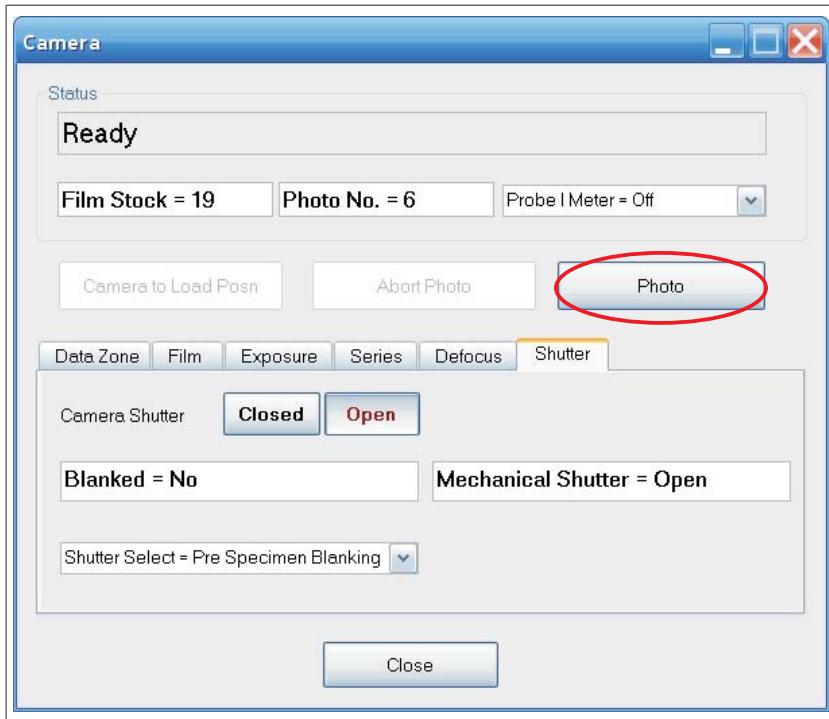


Fig.: 6 - 13 Camera menu for exposure start

- After adjusting the focus and brightness click on the button *Photo*.
 - The negative is moved from the magazine to the exposure position.
 - There are different intermediate stops for the data exposure before the image is exposed.
 - The actual status of the exposure is displayed in the status line.



NOTICE:

The camera can be ventilated at any time to take out the exposed negatives. This procedure takes place under red light again.

6.6 Removing negatives

- Ventilate the camera chamber as described in § 6.4.
- Check ventilation status by lifting the camera lid.
 - When ventilated continue to take out the receiving container.

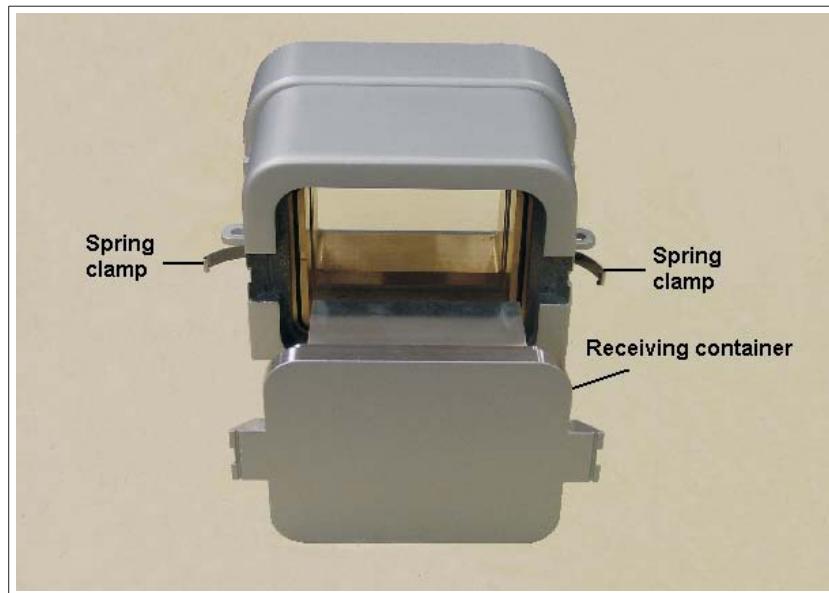


Fig.: 6 - 14 Sheetfilm camera from underneath



CAUTION:

Hold the receiving container before opening the spring clamps! It might drop.

- Open the clamps with your thumbs and take out the container.

- Put the black transport box over the receiving container and turn the whole thing upside down.
 - The sheetfilm holders fall into the transport box.
 - **Or**
- Put your fingers over the holders and turn the container upside down. Then put the negatives into the transport box and close it.

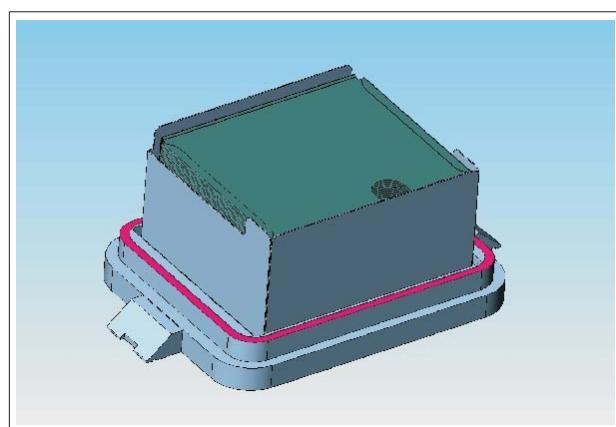
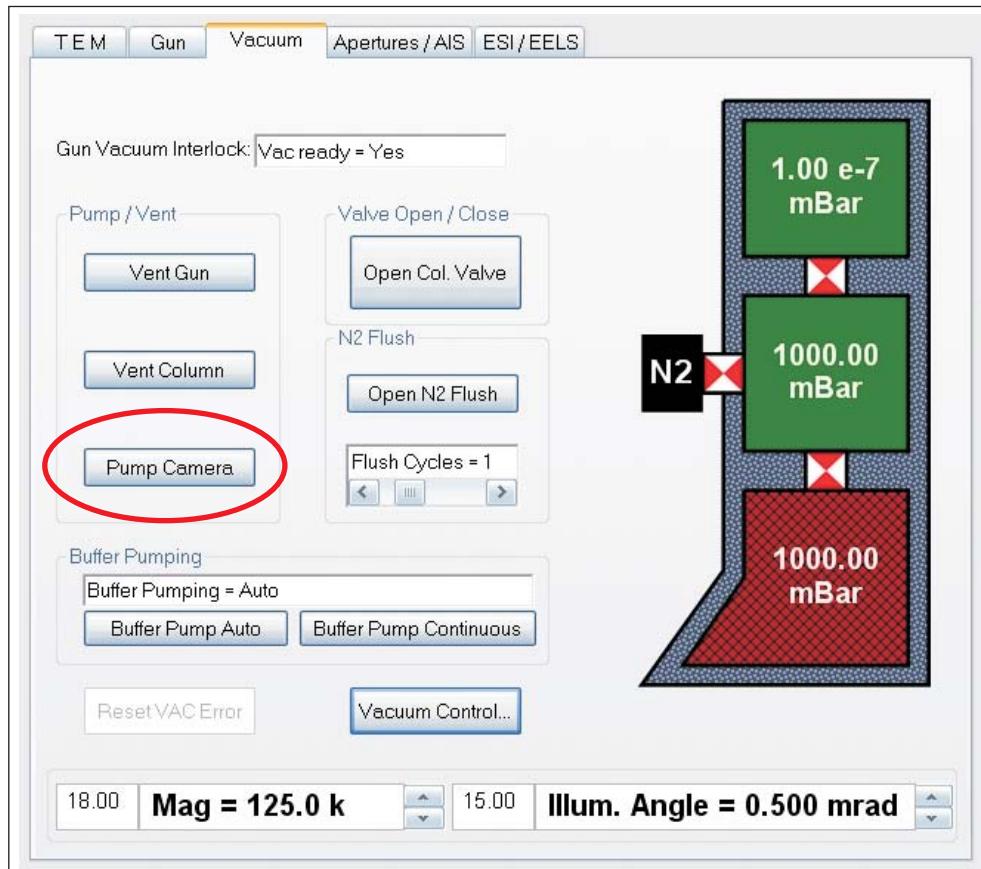


Fig.: 6 - 15 Receiving container with negatives

6.7 Restarting the SF camera

- Clean the O-ring of the receiving container to get rid of any dust particles.
- Insert the receiving container into the camera and lock the spring clamps.
- Open the Vac menu and click on *Pump Camera*.
 - After a few minutes the vacuum is ready



7 Maintenance



NOTICE:

For troublefree continuous operation of the instrument parts like filament and apertures should be regularly exchanged, parts cleaned and conditions controlled.

One of the most important service steps the user can do is described in this chapter. Any other manipulations on the instrument are carried out by experienced and specifically trained service engineers. A service contract is highly recommended.

7.1 Exchange of filament and anode

Conditions:

- Microscope in full operation, HV and filament turned off.
- VAC status = *ready* in the Status bar of the WINTEM main menu.

7.1.1 Ventilation of filament chamber (GUN)



Fig.: 7 - 1 VAC menu of the WinTEM main menu

- Click on *Vent Gun* in the group box *Pump/Vent*.
 - The column valves are closed.
 - The ventilation valve is opened and the gun is ventilated.

7.1.2 Filament exchange

- Push the button (gun lift) to lift and to swing away the gun.

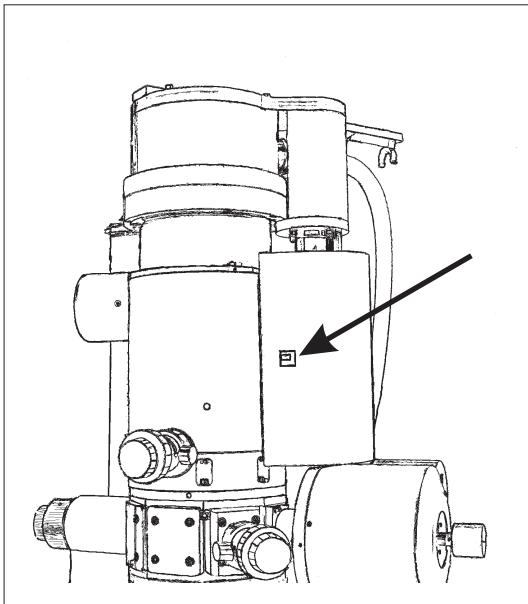


Fig.: 7 - 2 Lifting Gun

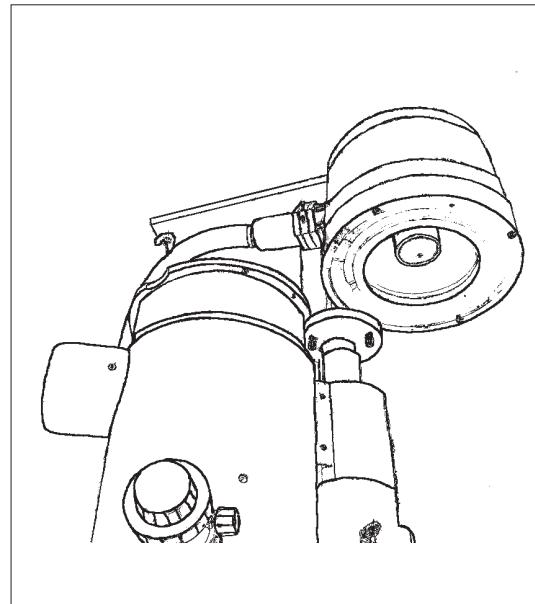


Fig.: 7 - 3 Swinging Gun away

- Grab the retaining ring (2) with clean gloves and unscrew the ring.
- Pull filament holder (1) out of the plug contact.
- Insert a precentered cleaned spare filament holder into the plug contact.
 - Notice the orientation of the pins and the Wehnelt cylinder.
- Secure filament holder by lightly tightening Wehnelt retaining ring manually.

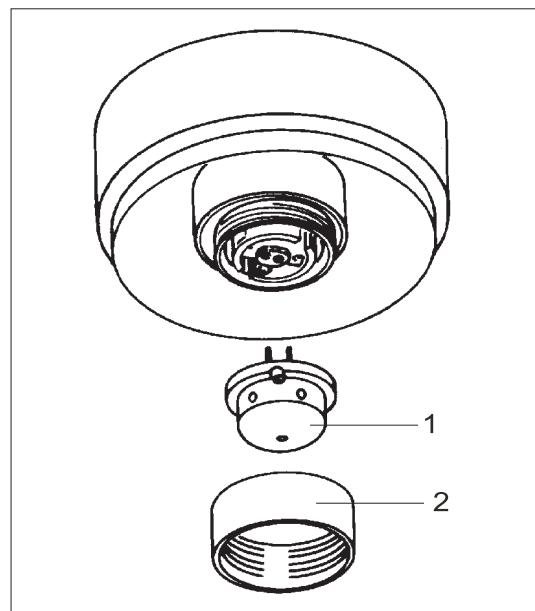


Fig.: 7 - 4 Wehnelt cylinder

7.1.3 Anode exchange

- Unscrew the anode with Latex or cotton gloves.
- Insert a cleaned anode and make sure that it is tight.
- Blow off possible dust on the anode and in the anode housing using a rubber blower.
- Wipe carefully over the GUN O-ring with your bare finger to get rid of any dirt.
- Swing back the GUN to the stop, hold it and push the button “gun lift” as shown in Fig.: 7 - 3.
- Click on *Gun Pump* in the *Vac* menu to evacuate the Gun.

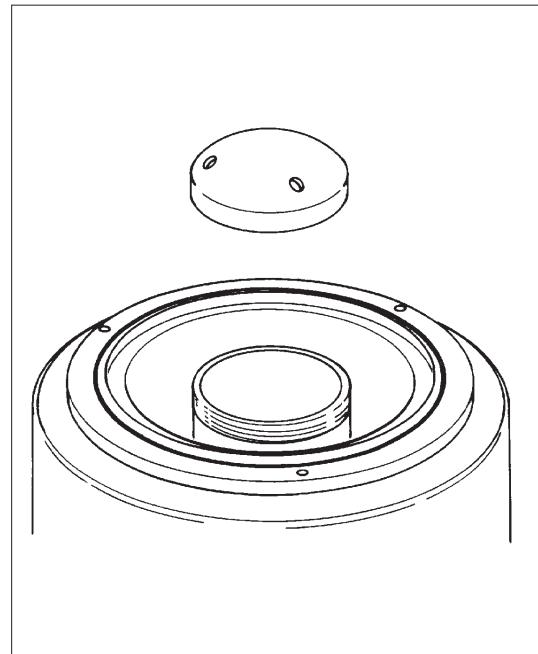


Fig.: 7 - 5 Anode of LIBRA 120

7.2 Restart after filament exchange


NOTICE:

For a safer restart it is highly recommended to use the function *Gun Conditioning* in *Settings* to ramp up the HT by monitoring.

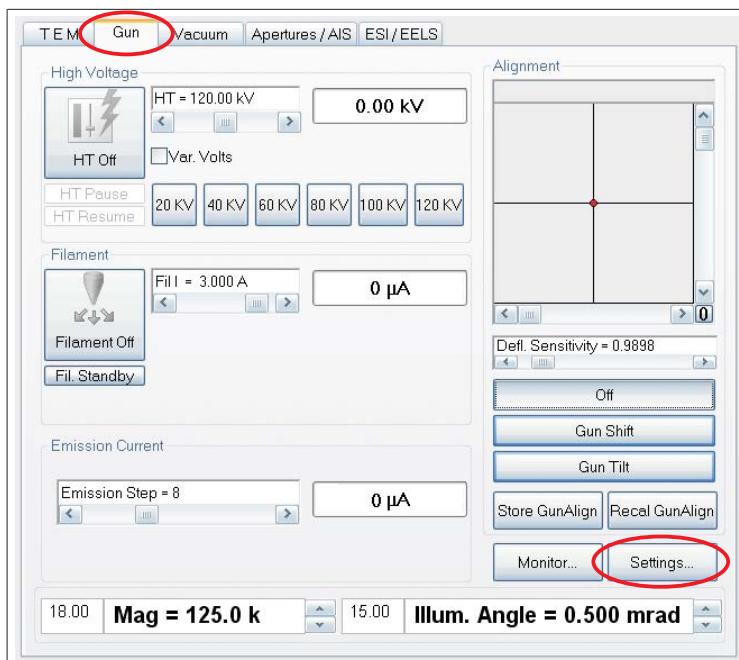
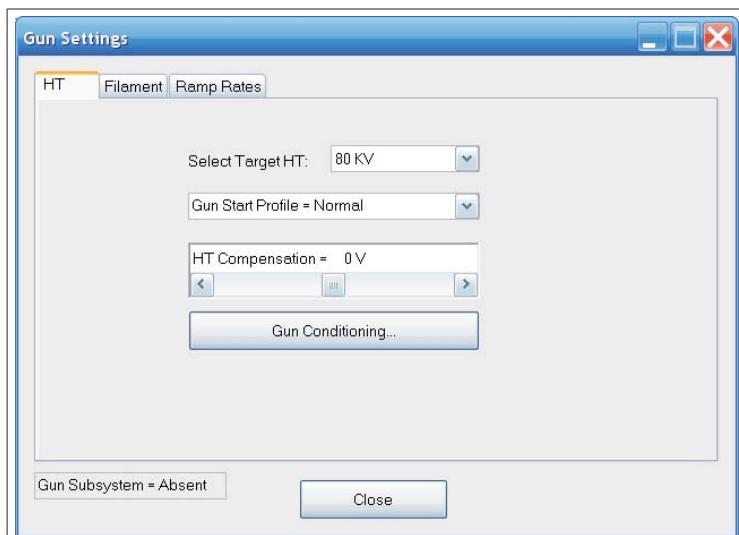


Fig.: 7 - 6 Gun tab sheet

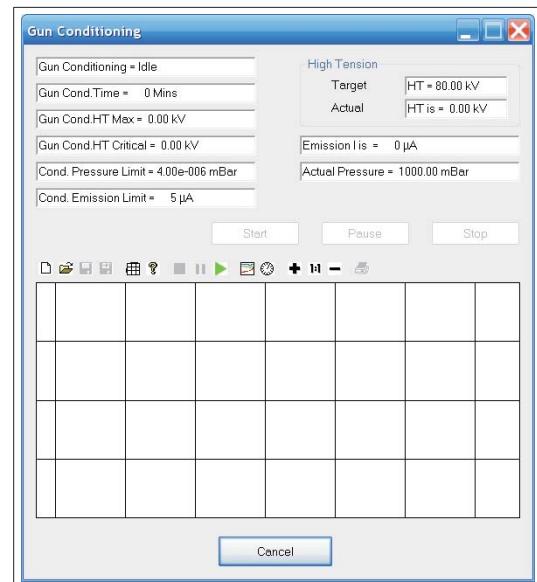
- Open the tab sheet *Gun* in the WinTEM main menu and click on *Settings*.



- Select Target HT (before using 120 kV it is recommended to condition 80 kV first.)

- Click on *Gun Conditioning*.

- Click on button *Start*.
 - The HT is ramped up controlled by the vacuum and the leakage current.
 - The conditioning is stopped when the HT is reached.



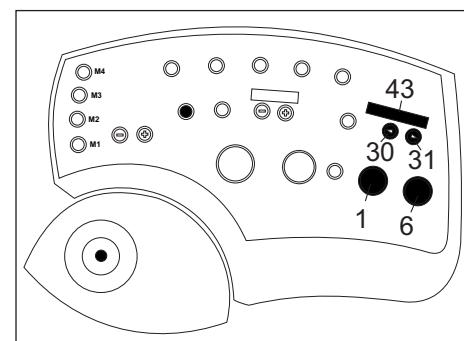
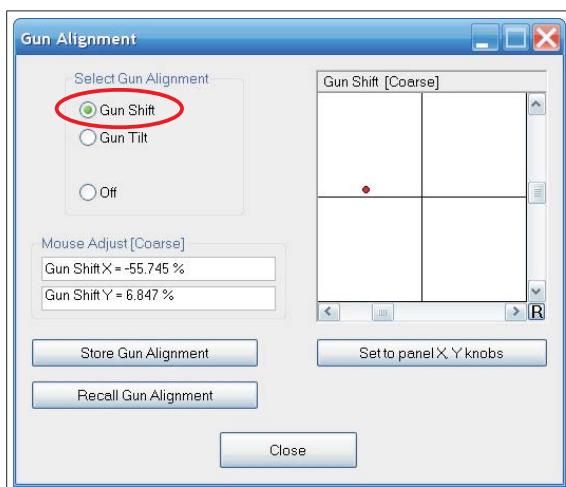
- Close the window *Gun Conditioning* and click on *Filament On* in the *Gun* tab sheet.
 - The filament current is ramped up.

- After the ramping is finished check the emission current and provide sufficient emission (5 - 10 μ A).

- Retract the specimen holder and all apertures.
 - Nothing should be inserted which could block the beam to be searched.

- Select a reasonable Mag to search the beam (10.000x).

- Select a reasonable brightness (0.5 mrad).

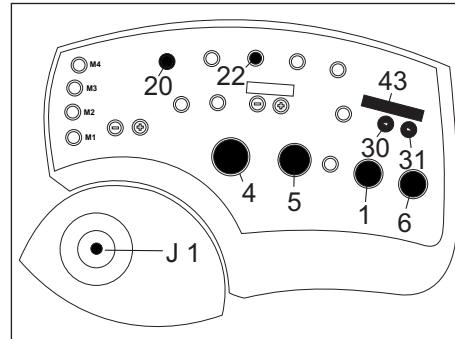
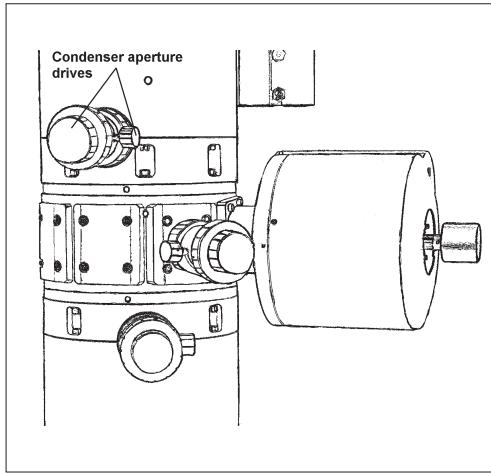


- Move the cursor on the PD menu *Alignment* and click on *Gun Alignment*.
 - The *Gun Alignment* box is opened.
- Click on the *Gun Shift* function.
- Grab the red navigation spot and move it slowly around the center in smaller and larger circles until any brightness is visible on the screen.
- Continue optimising the brightness with knobs (1) and (6) on the right hardpanel.
- Click on *Store Gun Alignment* to save the settings and close the *Gun Alignment* box.

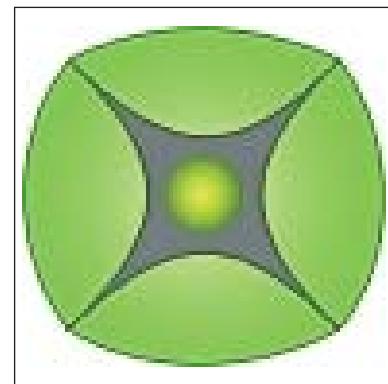
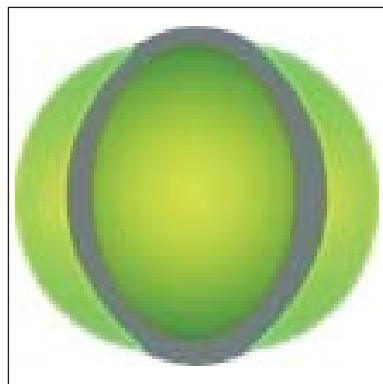


NOTICE:

This is the coarse Gun Alignment to get brightness on the screen at all. The final alignment is continued.



- Switch to Spot mode with button (20) and move the spot to the center with knob (1) and (6).
- Switch back to TEM mode with button (20).
- Insert the central AIS aperture (3rd clickstop) and center it mechanically to the screen.
 - Make sure that AIS mode is off, otherwise the central AIS aperture is not visible.
- Switch to SPOT mode again with button (20).
- Increase Mag (> 50.000x) on the left hardpanel and the Spot (>50nm) with knob (5).
- Increase the emission step until you get the undersaturated image of the filament.



- Adjust the filament image with the sliders in the *Gun Alignment* window or use the knobs (1) and (6) on the right hardpanel.
 - The filament image of a hairpin filament looks as shown in the left image.
 - The filament image of a LaB6 filament looks as shown in the right image.
- Click on *Store Gun Alignment*.
- Decrease the emission step or increase the filament current in the *Gun* tab sheet until the spot is homogenous.
 - This step is valid for both types of filament.

**NOTICE:**

After finishing the Gun Shift (Filament Precentering) check the Gun Tilt (Beam Alignment). The procedure is described in chap. 5.6.5.

7.3 Electrode cleaning

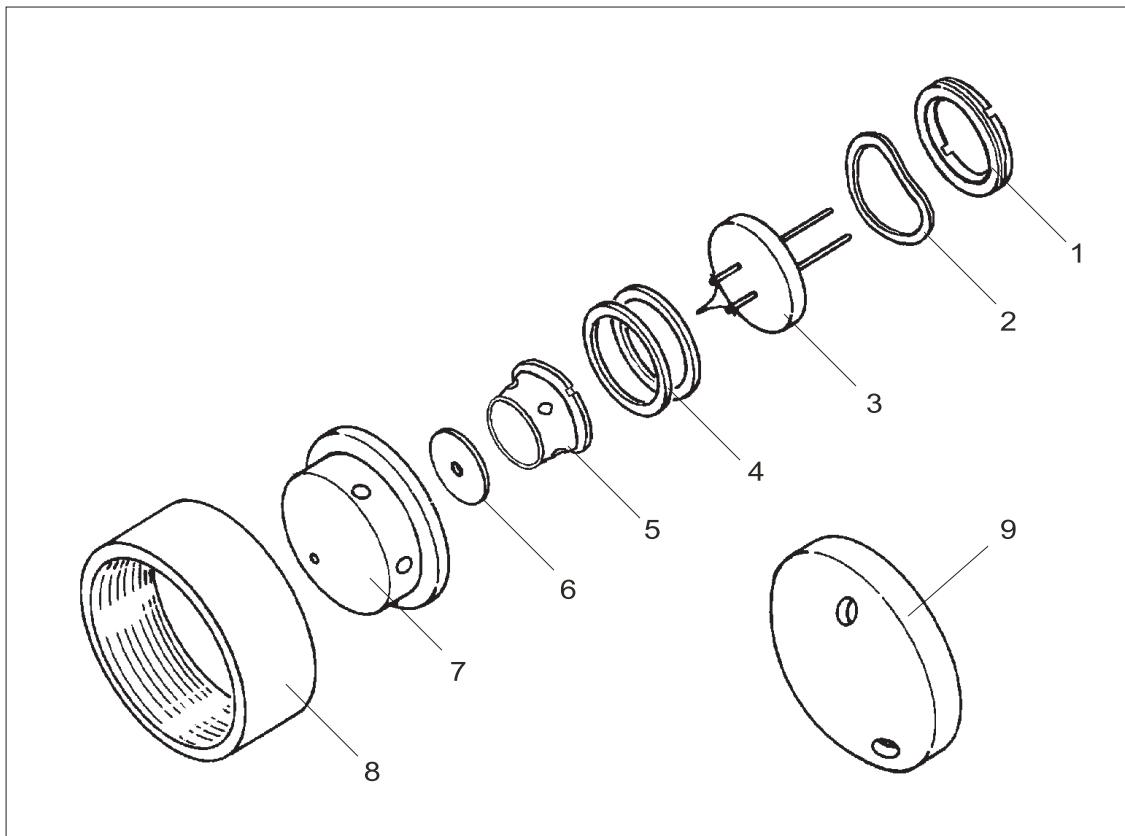


Fig.: 7 - 7 Parts requiring cleaning

The following parts (Fig. 7-7) need cleaning:

- Filament holder (7) with parts 5 and 6.
- Retaining ring (8)
- Anode (9)

The filament holder must be disassembled before cleaning; it consists of the following items:

- Locking ring (1)
- Washer (2)
- Filament (3)
- Spacer rings of different thickness(4)
- Wehnelt insert (5)
- Wehnelt aperture (6)
- Wehnelt cylinder (7)

The following cleaning agents are required:

- Alcohol and acetone as solvents
- Sidol Stahlglanz for cleaning

Tools required for cleaning:

- Clean cotton
- Lint-free cloth (e.g. Linen)
- Wooden sticks of different diameters
- Polishing paper or linen (grain 800 or higher)
- Padded tweezers
- Ultrasonic bath with heating. If the bath has no heating, infrared lamp or hot-air cabinet for drying is recommended.
- Stereomicroscope (if available)

7.3.1 Mechanical precleaning

Wehnelt cylinder

- Clean surfaces with Sidol; no contamination and scratches should be visible.

**NOTICE:**

The outer sphere surface and the Wehnelt hole must be treated with special care to prevent leakage current and micro discharges.

- Rinse parts thoroughly in water.
- Remove Sidol residues with cotton soaked in acetone.

Wehnelt insert

- Remove evaporated layers especially on inner surfaces with Sidol. High polish not necessary.

Wehnelt aperture

- Remove evaporated layers with Sidol or polishing paper.
- Polish surfaces and aperture hole with Sidol; no scratches should be visible.
- Clean aperture hole with pointed wooden stick and cotton.

Anode and retaining ring

- Remove evaporated layers, traces of impact and scratches with Sidol and polishing paper.
- Polish surfaces and hole with Sidol; no scratches should be visible.

Spacer rings and locking ring

must be cleaned in ultrasonic bath.

Ultrasonic bath for all parts**NOTICE:**

Arrange parts in ultrasonic bath so that they are not scratched. This applies in particular to the sphere surface and the hole of the Wehnelt cylinder.

- Take the electrode (Wehnelt or anode) with the tool and put into the glass filled with acetone.
- Rinse parts with acetone in ultrasonic bath for 5 to 10 minutes, which removes any Sidol residues.
- Repeat the procedure in alcohol.
- Take out parts with the tool and dry in hot-air cabinet at max. 100°C or under infrared lamp.
- For assembly place cleaned parts with clean cotton gloves on clean base next to stereomicroscope; polished surfaces facing up!

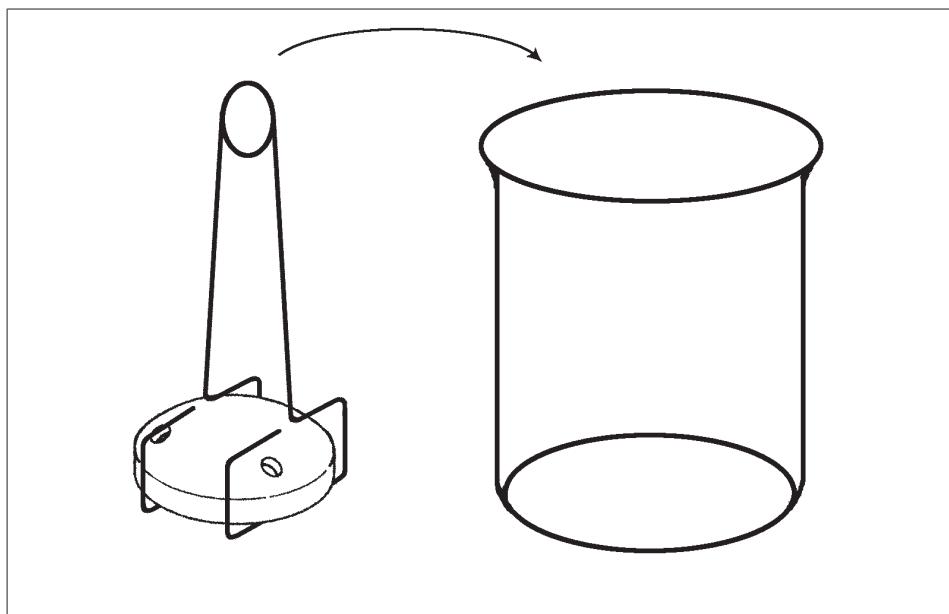


Fig.: 7 - 8 Chemical cleaning of the electrodes

7.4 Assembly of filament holder

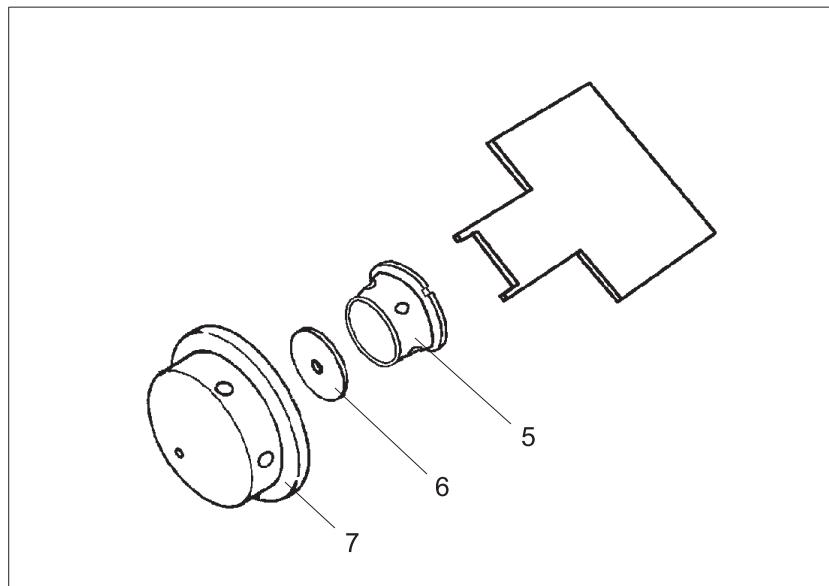


Fig.: 7 - 9 Assembly of the filament holder

The assembly of the Wehnelt cylinder is done in 2 steps:

- Assemble parts 5 to 7 according to Fig. 7-10.
- Insert the spacer rings (4) and finish the assembling with parts 1 to 3 (Fig. 7-10). As long as the same Wehnelt cylinder is used the new hairpin filament needn't to be adjusted in height. The same spacer rings can be used.

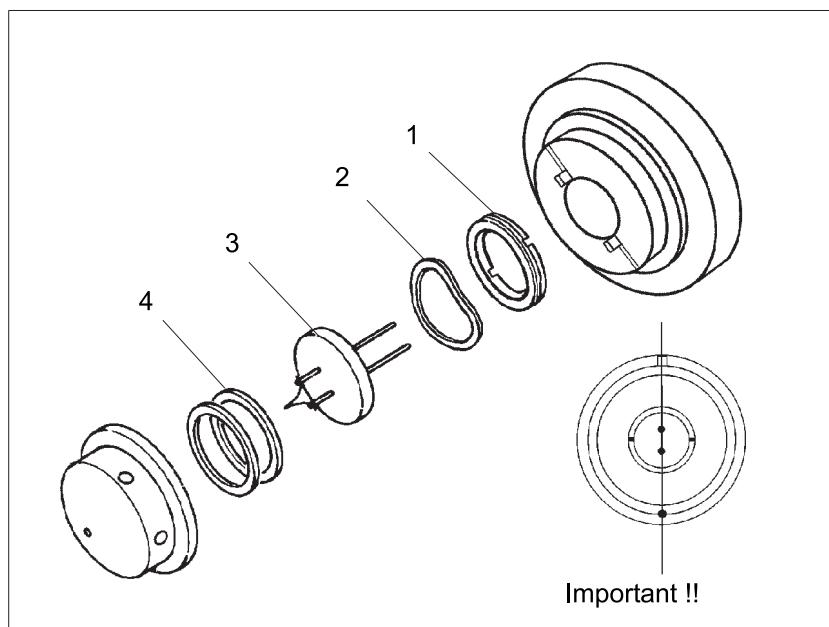


Fig.: 7 - 10 Assembly of filament holder

7.5 Mechanical adjustment of the filament

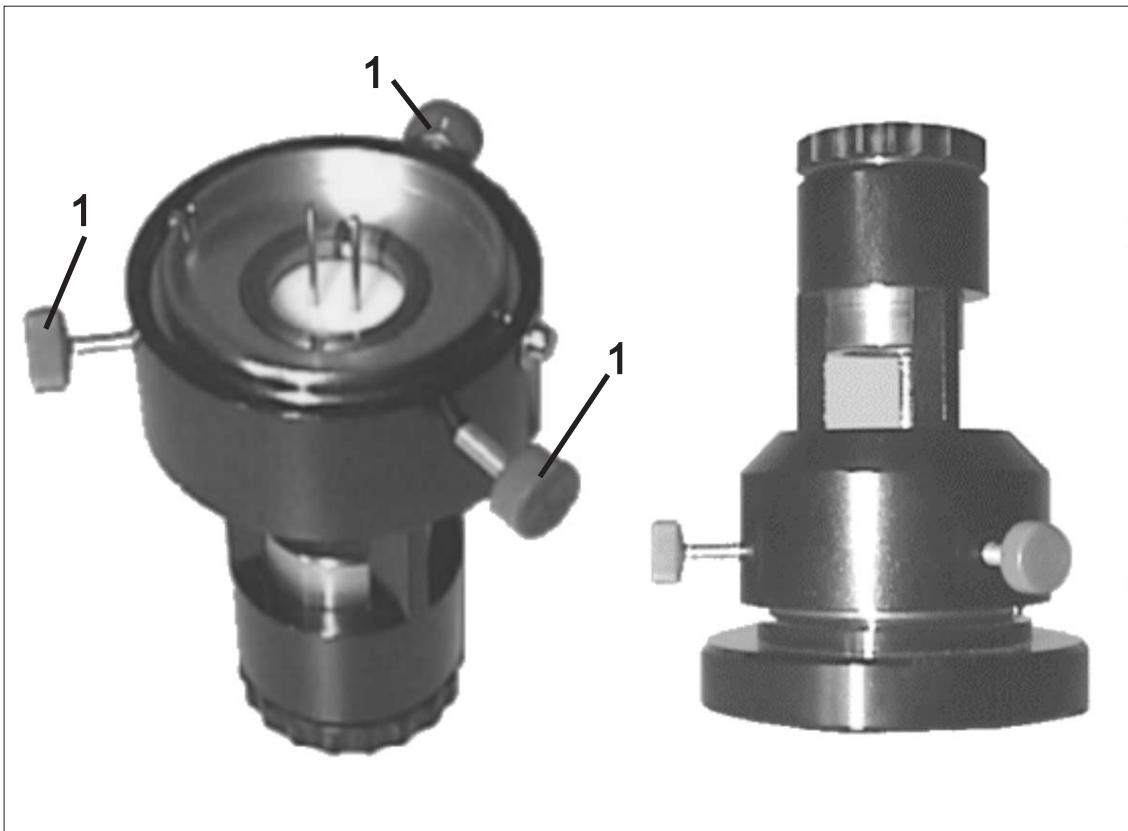


Fig.: 7 - 11 Filament adjustment device

- Insert the Wehnelt cylinder into the adjustment device. Take care of the orientation.
- Screw in the adjustment screws (1) until they are in contact with the ceramic part of the filament (left).
- Turn the adjustment device upside down and put it on the other part of the adjustment tool (right).
- Adjust the filament tip with the 3 screws (1) that it is in center of the Wehnelt aperture. Use the magnifier for observation.
- When the filament is centered, keep the lower part und turn the upper part until the final ring is tightened. Check again the filament position.
- Take off the upper part and turn it upside down again (left).
- Loosen the screws (1) to take the Wehnelt cylinder out of the adjustment device.

7.6 Components of the tool kit



Fig.: 7 - 12 Tool kit parts

The tool kit box contains the following parts:

- 1 Holder for electrodes
- 2 Tool for Wehnelt insert
- 3 Aperture key
- 4 Screwdriver for condenser aperture
- 5 Tool for filament centering
- 6 Tool for filament centering
- 7 Fixation screwdriver
- 8 Tweezers
- 9 Socket screw wrench
- 10 Hexagon screwdrivers
- 11 Wehnelt cylinder in box
- 12 Filaments
- 13 Hard wood sticks
- 14 Different o-rings, spacer rings, and aperture rings

7.7 Cleaning of specimen holders



NOTICE:

The very sensitive specimen holder is an important component of the electron microscope and should be handled with care. The front part of the holder from the sapphire bearing to the O-ring seal is in high vacuum. Any contamination will impair the high vacuum, contaminate the specimen and, possibly, cause instability of image or illumination.

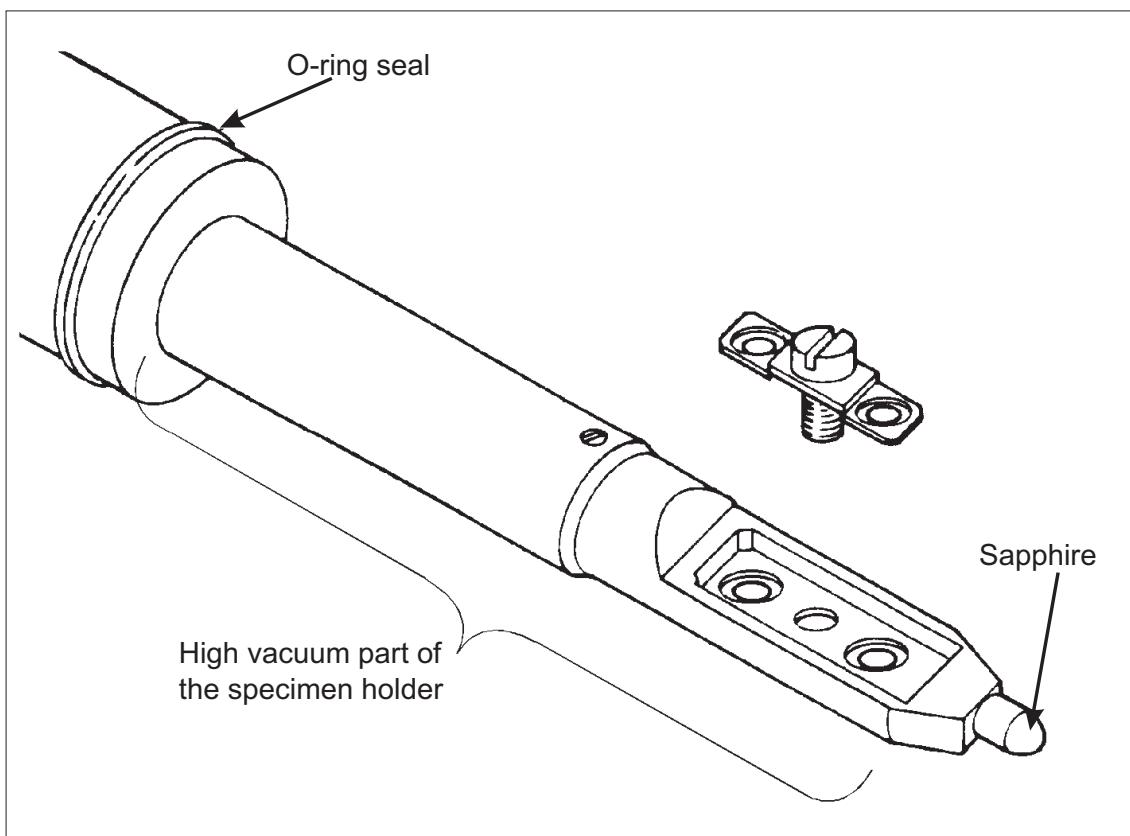


Fig.: 7 - 13 Specimen holder

The O-ring seal is the interface between atmospheric pressure and vacuum. The sealing ring must not be damaged or contaminated. It should be lightly greased (vacuum grease TF 50 HV, CZ ident. no. 101.311). If the ring is too dry, air may penetrate with lock-in. Too much grease causes contamination.

The sealing ring should be cleaned and greased from time to time by rubbing it between fingertips.

The area between sealing ring and sapphire must be absolutely clean. Any contact impairs the vacuum, lengthens the pump down time, contaminates the specimen, and, in element analysis, brings about wrong analytical results.

7.7.1 Cleaning procedure

The specimen holders are generally not or only slightly contaminated and can be cleaned with solvents.

- Clean clamping bridge in ultrasonic bath.
 - As far as O-ring seal clean specimen holder with cotton soaked in acetone. Do not place rod in ultrasonic bath; if necessary, immerse it for just a moment!
 - Dry rod by blowing on it with a rubber ball.
-

**CAUTION:**

The sapphire is glued on the rod tip. Avoid extended treatment with solvents, heating or cleaning in ultrasonic bath because the glue might be dissolved.

**NOTICE:**

For cleaning the special holders (rotary or double tilt rod) see the cleaning instructions provided by the manufacturers.

The most effective and most convenient way of holder cleaning is to use a plasma cleaner.

7.8 Apertures

7.8.1 Aperture holder removal

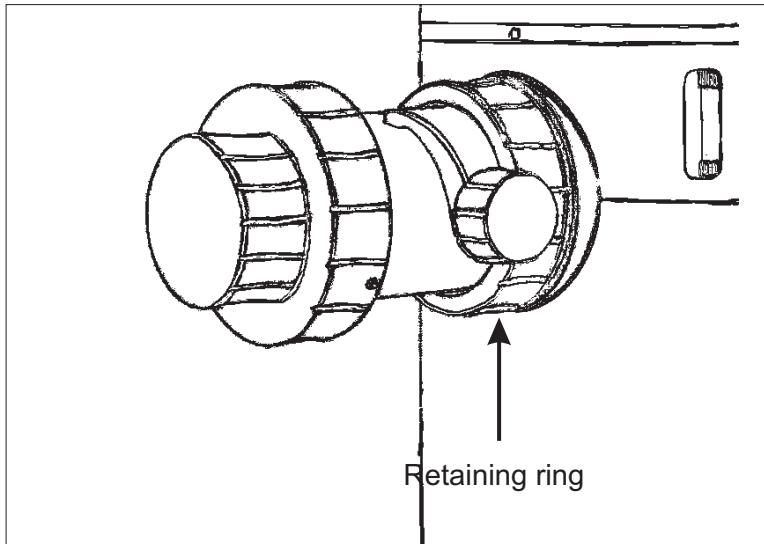


Fig.: 7 - 14 Loosening the retaining ring

- Push key *VENT / PUMP COL* in tab sheet *Vacuum*.
- Loosen retaining ring of the aperture drive (Fig. 7-14).
- Pull aperture drive carefully out of the column.
- Assemble in reverse order!

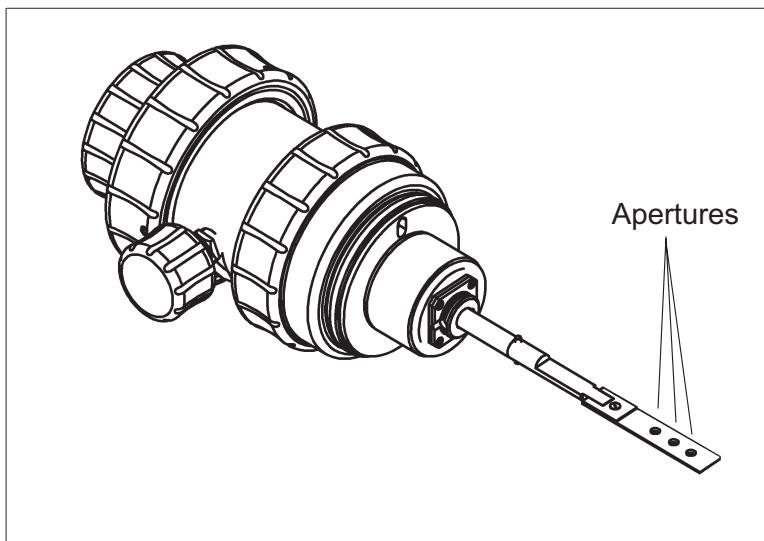


Fig.: 7 - 15 Aperture holder with apertures

7.8.2 Aperture exchange

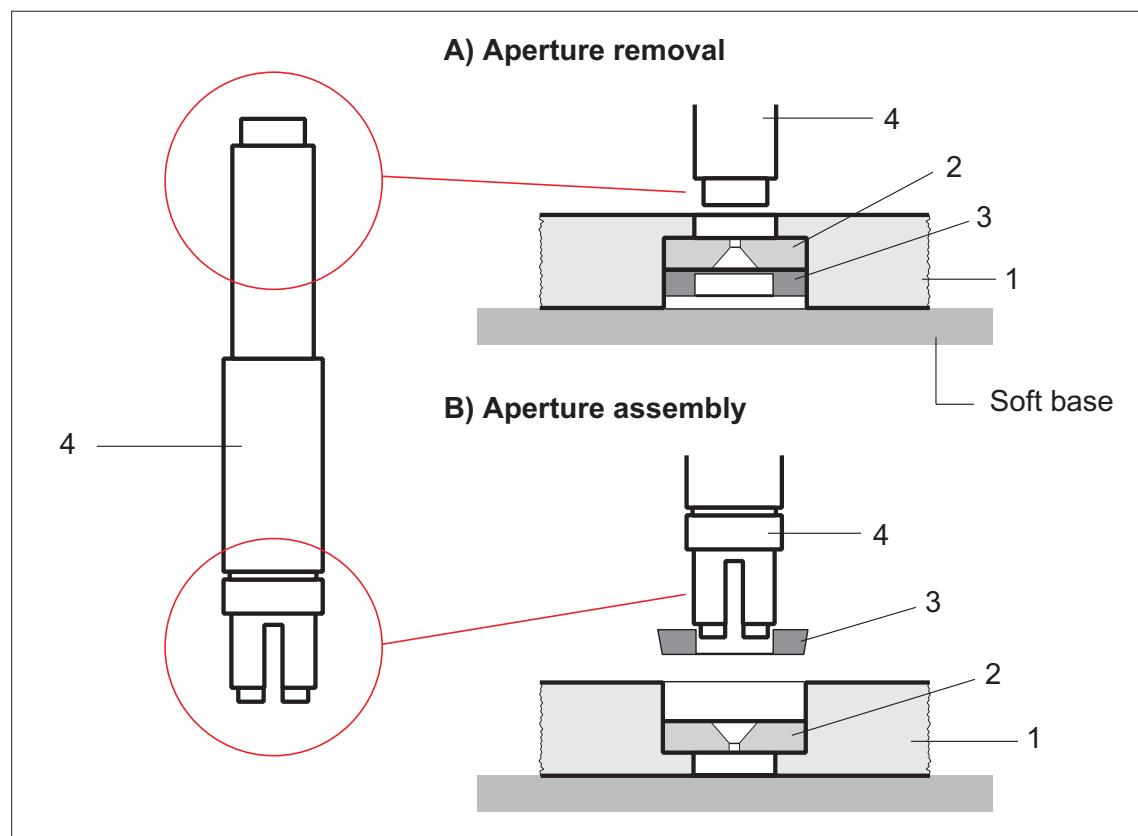


Fig.: 7 - 16 Aperture exchange

- Place aperture holder tip (1) on clean surface.
- Push aperture (2) out of the holder tip with tool (4) (Fig. 7-16 A).
- Turn aperture holder (1) through 180° and place it on the clean surface again.
- Insert new aperture (2) (Fig. 7-16 B).
- Attach spring ring (3) and press down with tool (4) (Fig. 7-16 B).



CAUTION:

If changing objective apertures the utmost cleanliness is necessary. Use Latex gloves for this procedure.

Any contamination in this area causes charging or image astigmatism.

For exchange of condenser or slit aperture call the ZEISS Service for assistance !!

8 Attachments

8.1 Digital Scanning Attachment

8.1.1 General

The Digital Scanning Attachment (DSA) opens a wide field of applications in a TEM. It provides additional results which cannot be obtained in a conventional TEM.

Depending on the different detectors (STEM DF/BF, SE, BSE) there is more detailed information about the sample. This information can be structure information or analytical information, if an EDS or EELS system is attached.

In most cases the TEM sample holder can be used for the different scanning modes.

Once the STEM unit is installed a few operation steps are needed to display an image on the screen.

The STEM detector is mounted at the left side mount flange. It is inserted pneumatically, when the STEM mode is started.

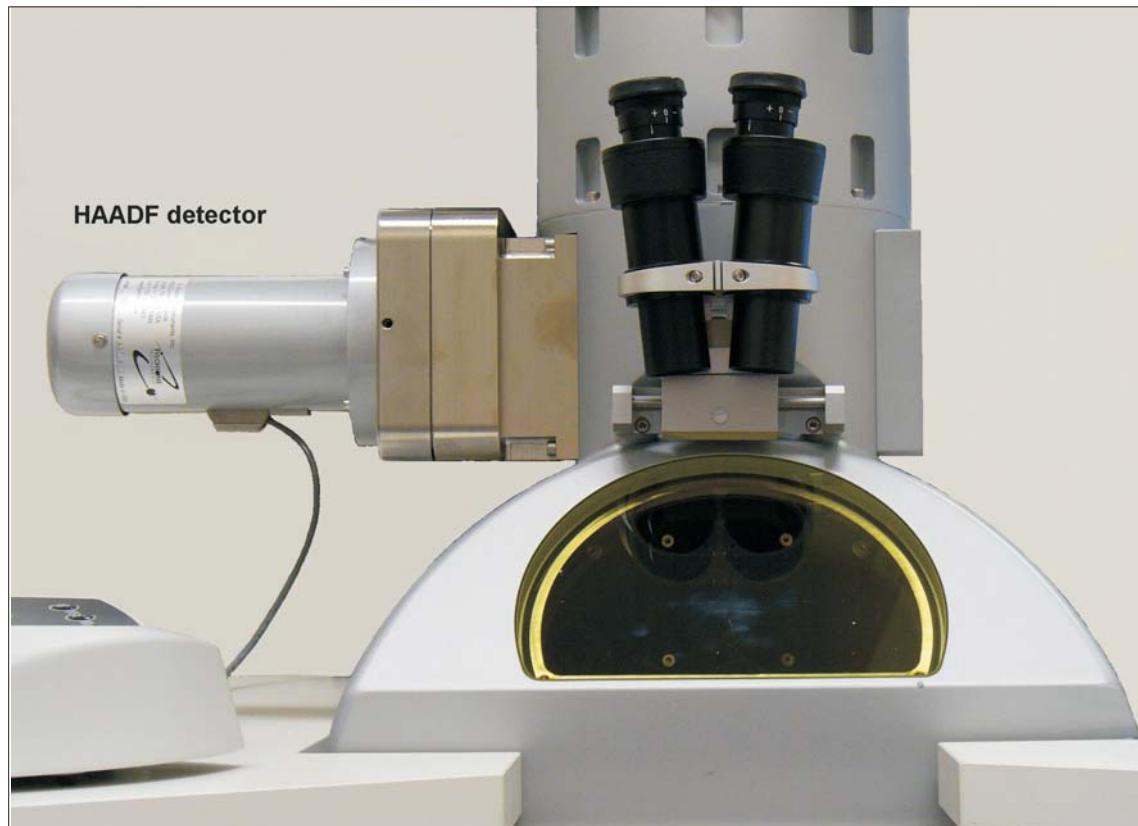


Fig.: 8 - 1 STEM detector at the side mount port

8.1.2 Detectors

When a focussed electron beam interacts with a sample there are different secondary effects. The main categories are transmitted electrons and reflected electrons. According to these effects detectors were developed to process these signals.

Fig. 8-2 shows the different detectors and their position related to the sample.

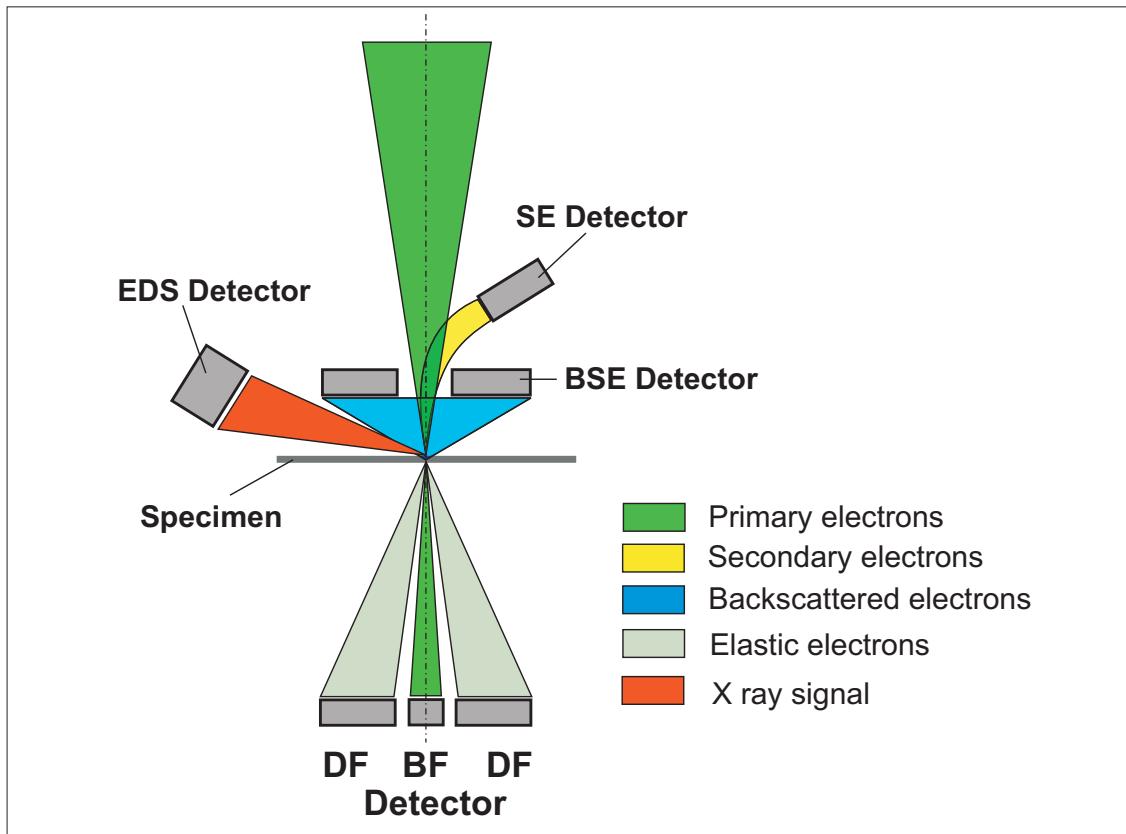


Fig.: 8 - 2 Positions and signals of the different detectors

SE Detector

SE electrons are generated at the sample surface by inelastic scattering. They are of low energy (< 50 eV). and provide surface information. The primary beam should be at the lowest HV step of the TEM.

BSE Detector

Backscattered electron are of higher energy. They are generated in the sample much deeper than SE electrons. The signal depends on the backscattered coefficient which is correlating with the Z-number. This contrast is also called Z-contrast.

STEM DF Detector

The scattered transmitted electrons are collected to form a darkfield image. The detector is of ring shape with a central hole. There are large area detectors to detect the electrons scattered under high angles (HAADF detector)

STEM BF Detector

The unscattered transmitted electrons are collected by the detector which is a Faraday cage and is located in the center of the DF detector.

8.1.3 STEM ray path and signal control

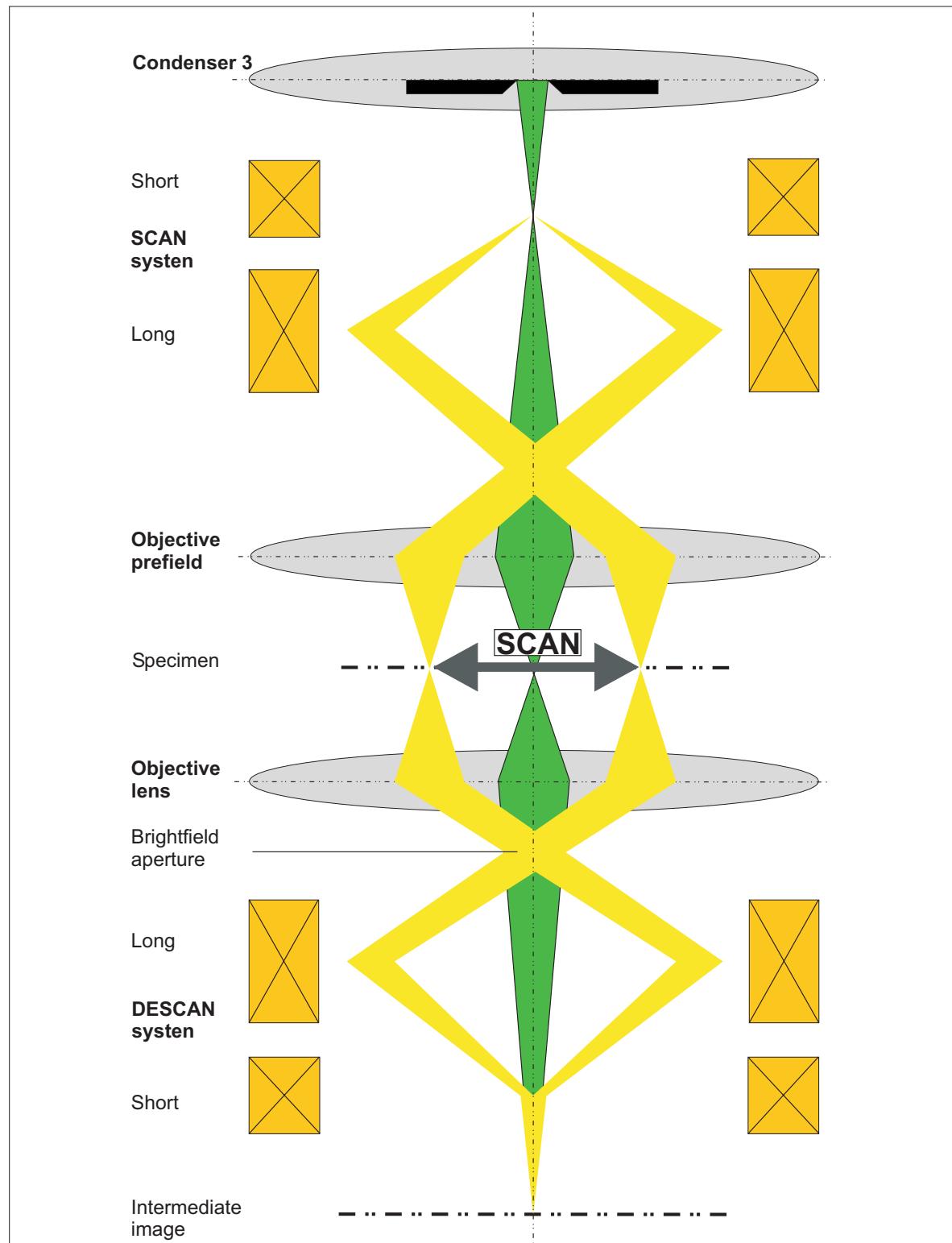


Fig.: 8 - 3 STEM ray path

When the STEM mode is activated the static deflecting systems above and below the sample become dynamic deflecting systems.

At the same time the SCAN system (above the sample) and the DESCAN system (below the sample) are synchronised to compensate the beam shift in the scanning mode.

The scan speed and the image size is realised by a scan generator (see Fig. 8-5).

Finally the diffraction plane is imaged to the detector plane. In case of a darkfield detector the zero order beam travels through the central hole. Only the scattered electrons hit the detector area. To optimise the darkfield signal the CL (camera length) can be changed. When the zero order beam is just filling the hole, the darkfield signal is the best. This is not valid for HAADF where the high angle scattering is collected.

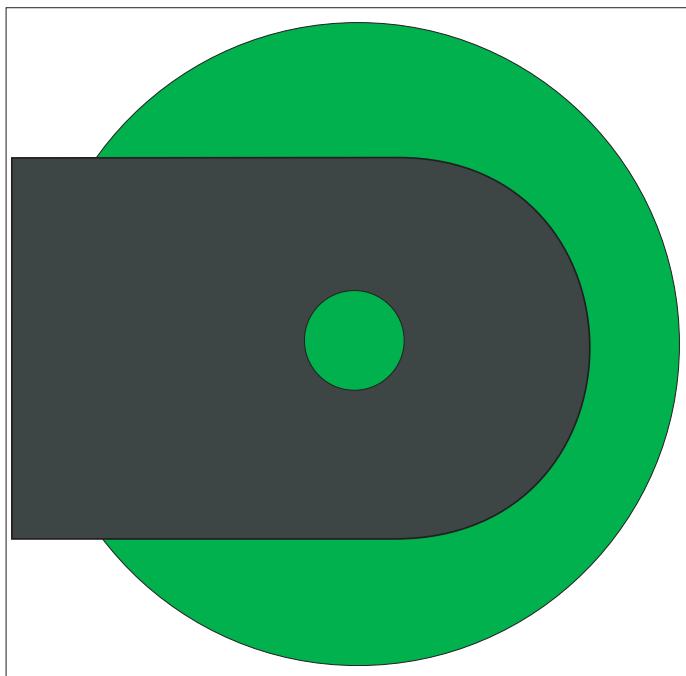


Fig.: 8 - 4 STEM detector inserted

Fig. 8-4 shows a Fiscione HAADF detector still with TEM illumination on the viewing screen. As mentioned above the diffraction disc of the zero order is adapted to the size of the central hole.

When the diffraction disc is shifted from the hole onto the detector, a brightfield STEM image can be obtained. But it always contains parts of the darkfield signal.

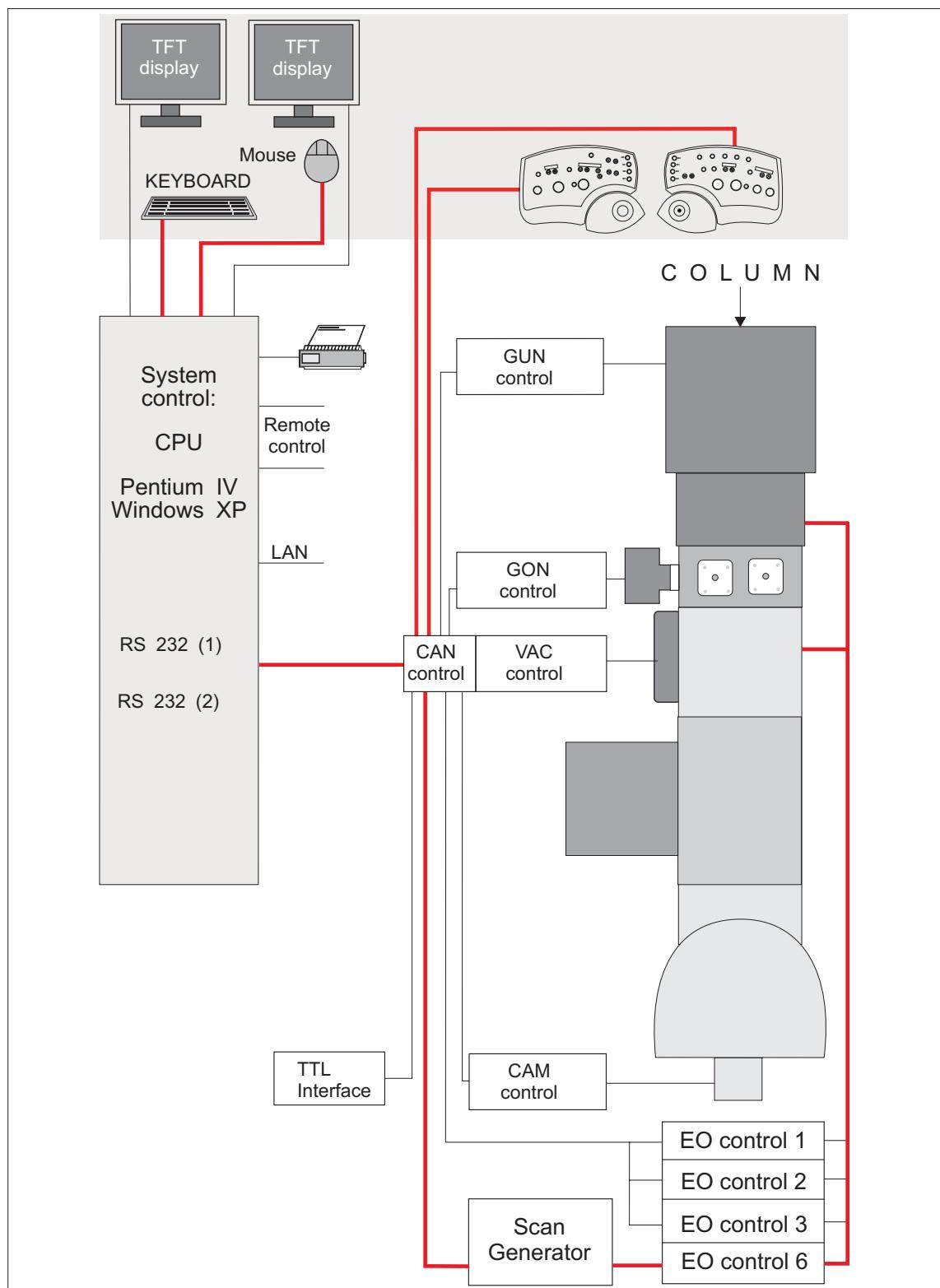


Fig.: 8 - 5 Diagram of the STEM signal

8.1.4 Operation

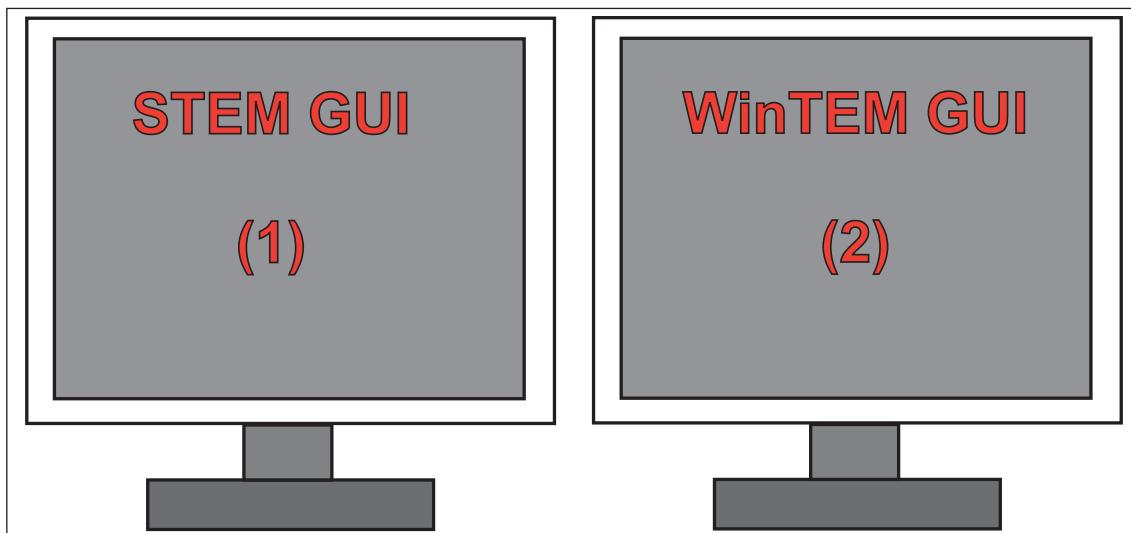


NOTICE:

When a STEM unit is installed, a second TFT display is part of the delivery. Make sure that the second monitor is configured in the WINDOWS XP operating system.

8.1.4.1 Configuration of the user interface (GUI)

- Start the WinTEM software and move the GUI window to the monitor assigned as # 2 in the operating system.
 - Be aware that the monitor # 2 is not automatically the right hand monitor.



- Start the STEM application in the toolbar with the marked icon.
 - The STEM GUI is opened.
 - The LIBRA® 120 is switched to SPOT and DIFF mode.
 - The hardpanels are configured for STEM operation.
 - The STEM mag is set to 4000x automatically.
- Move the STEM window to the monitor assigned as # 1 in the operating system.
 - Since the STEM life images are displayed in the overlay, they are only visible on the monitor # 1.

8.1.4.2 Illumination alignment


NOTICE:

It is provided that the TEM illumination especially in spot mode was checked.

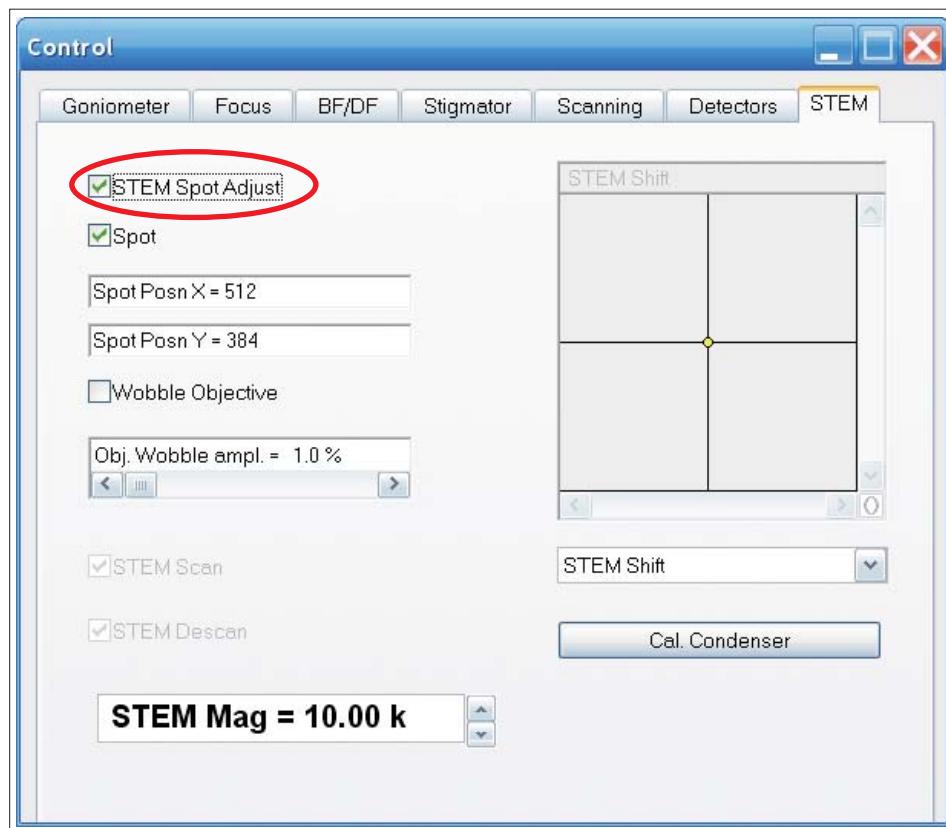
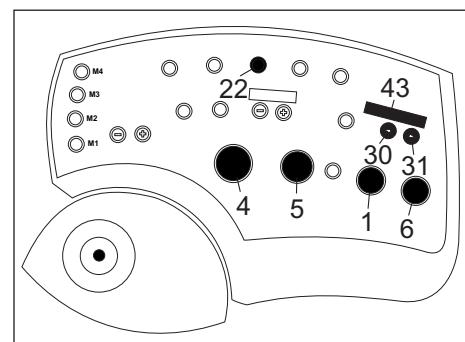


Fig.: 8 - 6 STEM tab sheet

- Activate the *STEM Spot Adjust* function in the tab sheet *STEM*.
 - The scan mode is stopped.
 - The hardpanels are switched back to TEM functionality.
 - The illumination can be alignment.

- Switch to *Img* mode with button (22).
- Set spot size to the desired value with knob brightness (5), e.g. 10 nm.
 - Spot should be in the center without any halo.
- Switch back to *Diff* with button (22).



- Set camera length to ≤ 720 mm with Mag knob (3).
 - The diffraction disc is visible on the viewing screen.
- Activate the HAADF detector in the Control box (Fig. 8-7).
 - The detector is inserted pneumatically

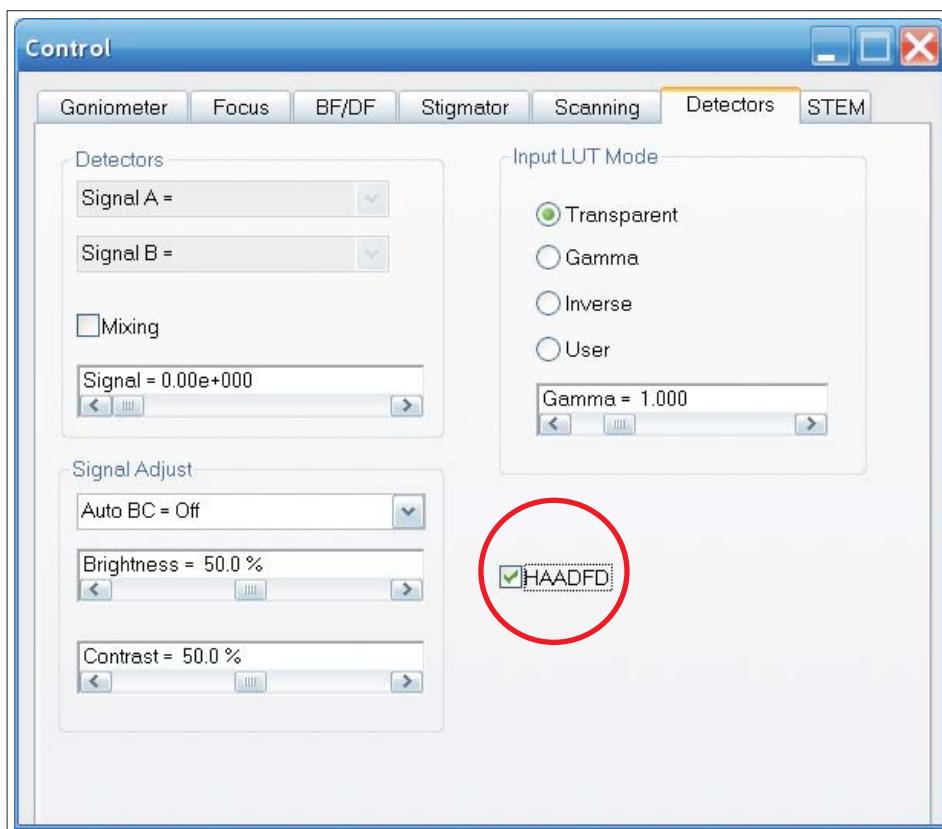
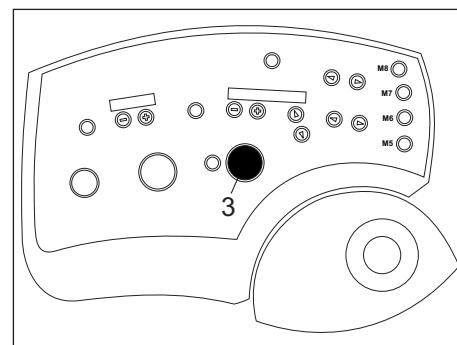
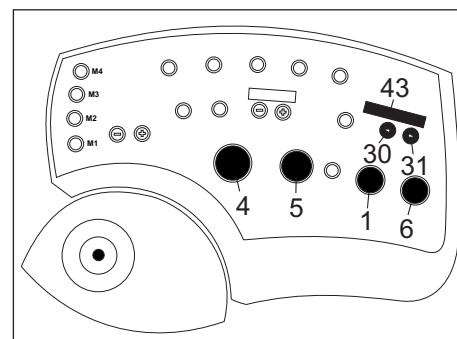


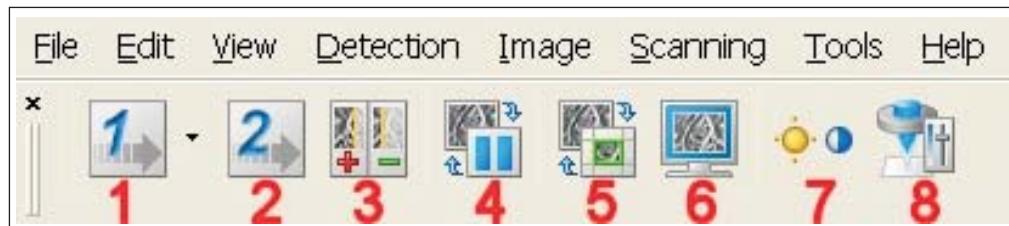
Fig.: 8 - 7 Detector Tab Sheet

- Select *BF Shift* on the display (43) with buttons (30 or 31).
 - If not available, select *BF Shift* in the Tab Sheet *STEM*.
- Adjust the diffraction disc to the center of the detector with knobs (1 and 6).
 - If the diffraction disc is too big, reduce the camera length by one step with Mag knob (3).



8.1.4.3 Acquiring and saving an image

- Insert a sample into the goniometer to go through the acquisition procedure.
 - The sample should have good contrast (e. g. a grating replica)
- Deactivate the function *STEM Spot Adj* (Fig. 8-6).
 - The scan mode is started.



- Icon ① defines the scan speed 1/3 with left/middle mouse click.
- Icon ② defines the scan speed 6/10 with left/middle mouse click.
- A click on the arrow between the 2 icons opens a menu with all scan speeds.
- Icon ③ changes the scan speed by 1 step up/down with left/middle mouse click.
- Icon ④ toggles between "Freeze" and "Freeze At End" which is displayed in the lower right image corner by a red spot.
- Icon ⑤ opens a reduced scan window with the left mouse click and a split screen with the middle mouse button.
- Icon ⑥ toggles between *Normal Scan* and *Reduced Scan* with the left mouse click, and opens the STEM Control Dialog menu with the middle mouse button..
- Icon ⑦ activates the mouse control for Contrast (left click) and Brightness (middle click).
- Icon ⑧ activates the mouse control for STEM Mag (left click) and Condenser Focus (middle click).
- Select the scan speed with Icon ① or ② and adjust contrast and brightness in the Control menu *Detectors* or with the mouse after clicking on Icon ⑦ .
- Freeze the image with a left mouse click on icon ④ in the toolbar.
- Open the File PD menu and click on *Save Image*.
 - The *Export TIFF* window is opened.
- Put in an image name and if desired a user text.
- Click on *Save*.
 - The image is saved in the defined image directory.
 - The image directory can be changed with button *Change Directory*.

8.1.4.4 Focusing in STEM mode

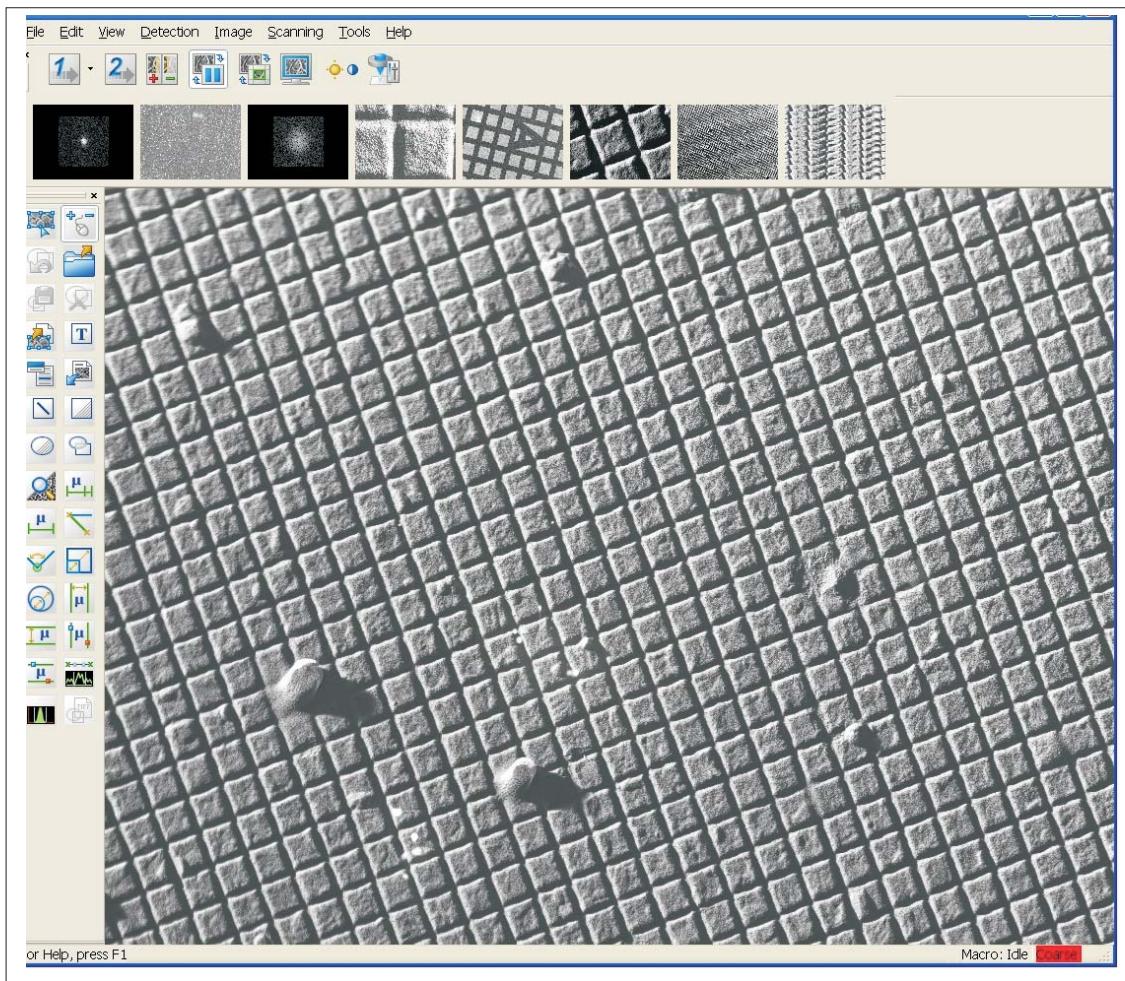


Fig.: 8 - 8 STEM image in the STEM user interface

- Adjust the contrast and brightness in the *Detectors* tab sheet (Fig. 8-7) with the corresponding sliders.
 - Make sure that Auto BC is in Off mode.
 - The image looks like in the above window.
 - If the image is out of focus, calibrate the objective lens (focus) and adjust Z height.
- Focus the sample with Focus knob (4).
 - Make sure that the focus mode is not any more in mechanical mode.
 - STEM focusing might be easier, if the reduced scan window is applied.

8.1.4.5 STEM tilt adjustment

- Select the mode *Reduced window* with the marked icon in Fig. 8-10.
 - The scan is only working in the reduced window (Fig. 8-10).
- Increase the STEM mag to e. g. 80k with the Mag button on the left hardpanel.
 - The mag can also be increased in the tab sheet *STEM*.
- Focus the sample with focus knob (4) on the right hardpanel.
- Activate the function *Wobble Objective* in the *STEM* tab sheet.
 - If the III tilt (STEM tilt) is not corrected the sample is moving laterally.
 - The Wobble Obj. amplitude can be adjusted in the *STEM* tab sheet (Fig. 8-9).

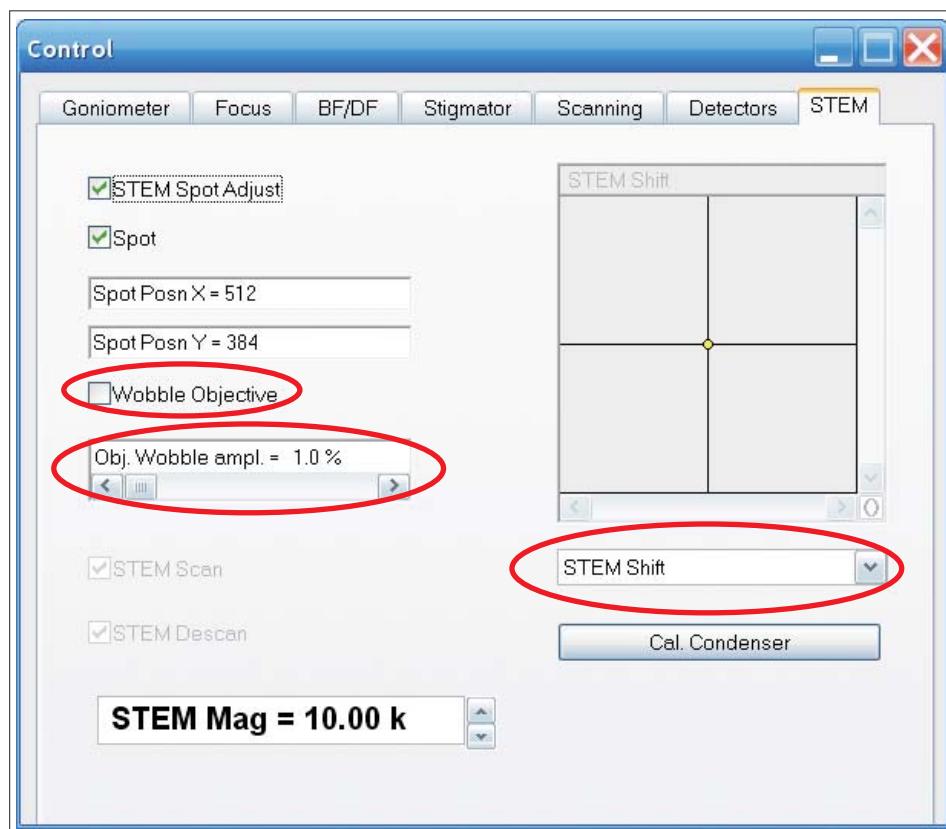


Fig.: 8 - 9 STEM Tab Sheet

- Select the *STEM tilt* deflecting system in the *STEM* tab sheet.
 - *STEM tilt* is also displayed on the right hardpanel.
- Minimise the lateral movement of the sample with knobs (1 and 6) on the right hardpanel.
- Turn off the function *Wobble Objective*.

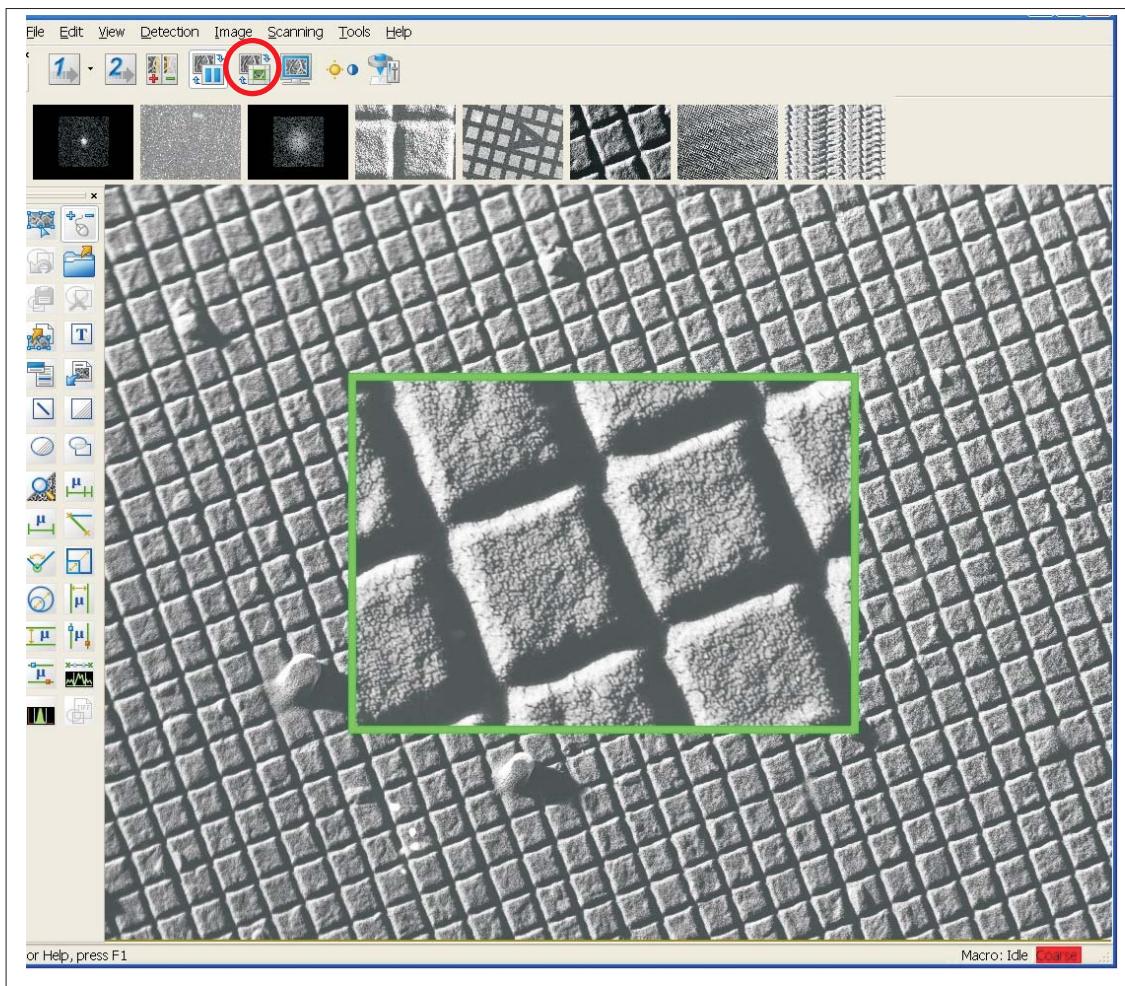


Fig.: 8 - 10 STEM image with a reduced scan field in the center

8.1.4.6 STEM astigmatism compensation

For astigmatism compensation the same recuced window mode can be applied.

- Select deflecting system *STEM Stig* in the *STEM* tab sheet (Fig. 8-9).
 - *STEM Stig* is displayed on the right hardpanel.
- Compensate astigmatism with knobs (1 and 6) on the right hardpanel.
 - Refocusing might be necessary during the astigmatism compensation.
 - After compensation there is no distortion of the graininess in under- and overfocus.

Two more features are very helpful for the daily work. These are *Split Screen* and *Dual Mag*.

8.1.4.7 Split screen

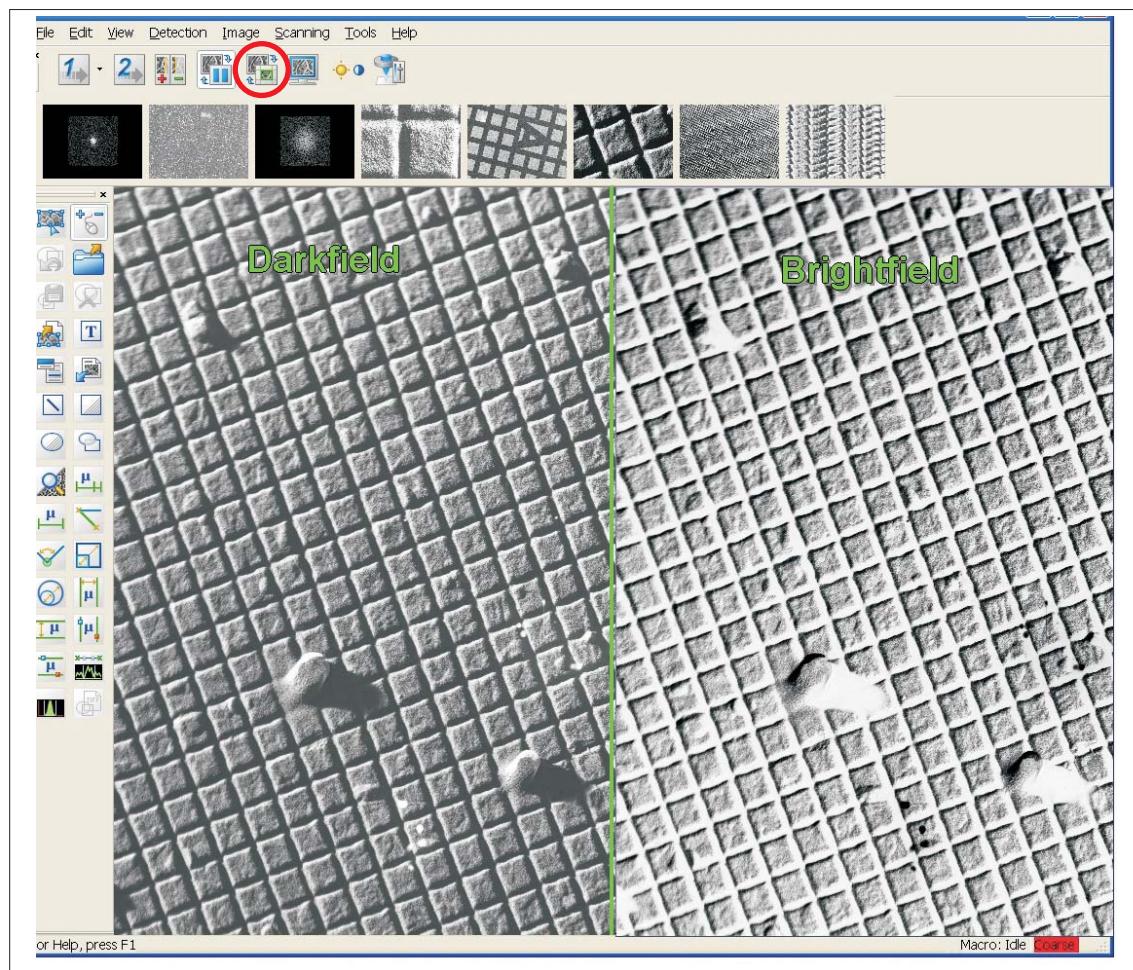


Fig.: 8 - 11 STEM split screen mode

This mode is recommended, if 2 different detectors are installed. E. g. a STEM brightfield and a STEM darkfield detector as shown in the above image. Much more information can be provided simultaneously.

- Click on the marked icon with the middle mouse button.
 - The screen is split in 2 frames. Each frame can show the image from a different source.

- Select the appropriate signals for Signal A and Signal B.
- Adjust the scan speed with icon ① or ② in the toolbar.



The split images can be frozen separately depending where the anchor symbol is located. The symbol can be moved to the desired frame.

- Move the anchor symbol on the image to be frozen.
- Freeze the image with icon ④ .
 - The scan can be restarted with another left mouse click on the icon.
 - The middle mouse click on icon ④ allows the freezing only.

8.1.4.8 Dual Mag

The *Dual Mag* function allows the display of 2 different magnifications simultaneously. Thus the overview and the detailed information is obtained with one shot.

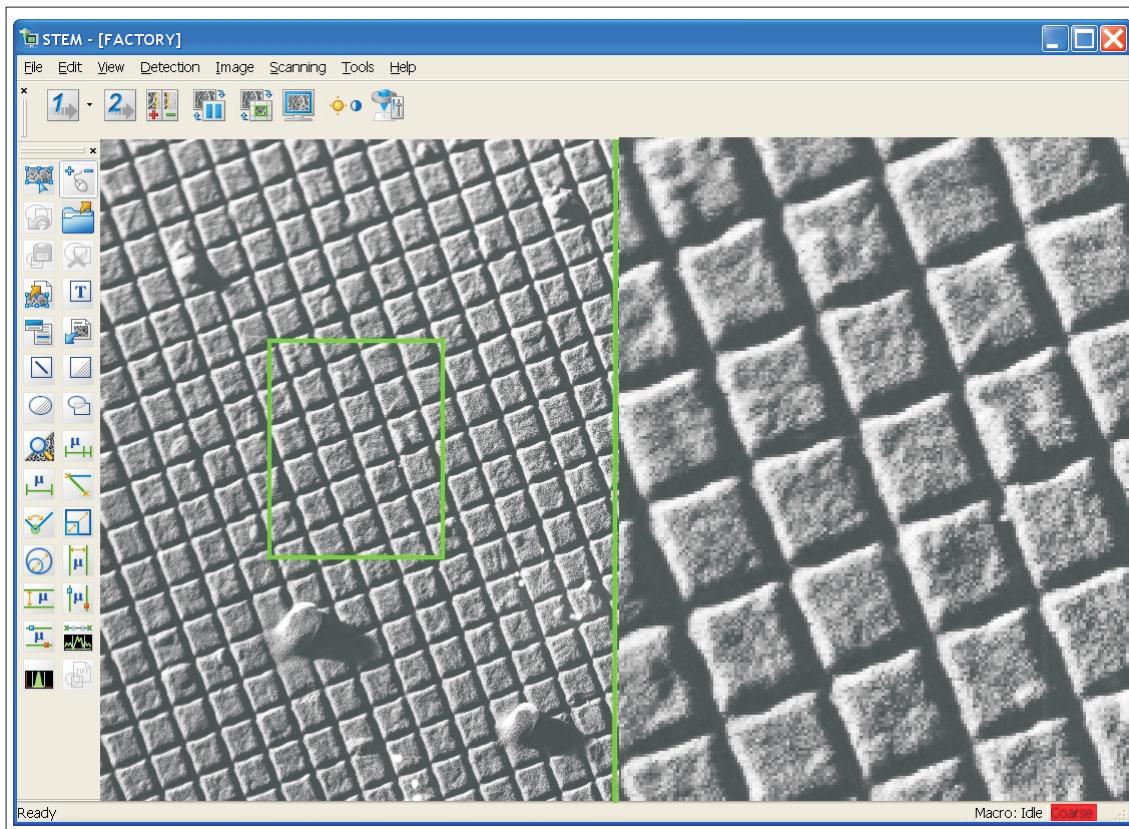
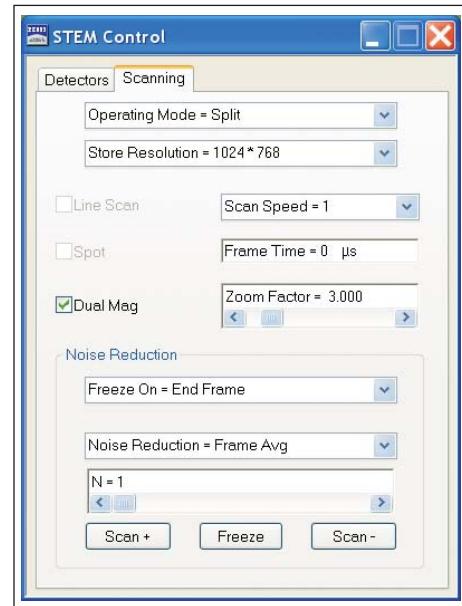


Fig.: 8 - 12 Dual Mag acquisition window

- Open the STEM control dialog window by clicking on the icon ⑥ with the middle mouse button.
- Activate the *Dual Mag* function and set the desired zoom factor.
 - The scan window appears as shown above.
- Freeze the image with a left mouse click on icon ④ in the toolbar.
- Save the image as described in 8.1.4.3.



9 Specification LIBRA 120

Resolution

| | | |
|------------------------------|---------------------------------|--|
| Basic instrument | Point Resolution | 0.34 nm |
| | Lattice Resolution | < 0.20 nm |
| | Energy Resolution | < 1.5 eV (limited by energy spread of emitter) |
| STEM Attachment (DSA) | Global and Elastic BF/DF | 1.5 nm |
| | SE | 2.0 nm |
| | BSE | 2.0 nm |

Modes of Operation

| | |
|---|---|
| EFTEM Imaging | - Global and elastic BF / DF - Elemental contrast - Structure sensitive contrast - Contrast tuning - Conical illumination - Low dose |
| TEM Diffraction | - Global and elastic SAED - Global and elastic CBED/LACBED - Global and elastic micro- and nano-diffraction - Electron spectroscopic diffraction |
| TEM Analysis (Image Analysis System required) | - Electron spectroscopic imaging - Image EELS - Parallel EELS - Lateral and angular resolved EELS - EDS (EDX system required) |

| | |
|-------------------------|--|
| STEM (Option) | <ul style="list-style-type: none"> - Global and elastic BF / DF / HAADF - Electron spectroscopic BF / DF - Spectrum imaging (image analysis system required) - SE / BSE - EDS mapping (EDX system required) |
|-------------------------|--|

Electron Emitter

Type Single-stage accelerator for thermal emitter (Tungsten or LaB₆). Pneumatic gun lift.

Gun supply **Stability / Drift:** $\Delta U/U < 2 \times 10^{-6}$ (ripple) / ≤ 0.25 eV/min.

Max. Beam Current / Stability: $I_{\max} > 50$ μA / ≤ 1 % (1h).

Accelerating Voltage: 80, 120 kV (40 kV for SE / BSE only in SEM mode); 60, 100 kV on request.

Variable in steps of 10 to 1,000 V; range ± 15 kV (option).

Correction of lens currents every 30 V.

Filament: Selectable settings for maximum current or maximum energy resolution. Automatic ramp-up and safety procedures; filament overheat protection; filament hour meter.

Emitter Alignment: Double deflection system for independent emitter shift and tilt. Beam blanker function.

Emitter Control: Fully digital control, optimum safety and reproducibility.

Beam Blanker: above the specimen

Illumination System

Two illumination systems are available: Advanced Köhler Illumination System and Conventional Illumination System

Advanced Köhler Illumination System Highly flexible with 4 lenses: condenser 1 to 3 with multi-hole C3 aperture, and condenser objective (C-O) pre-field lens.

C-O Pre-field Coefficients: $C_s = 2.2$ mm, $C_c = 2.2$ mm

Condenser Zoom: Fully digitally controlled spot size/illumination angle (brightness) steps. Separate, HT specific data sets for MAG/LM and spot/parallel wide-field (TEM) illumination, respectively. Automatic ΔE compensation. Exactly reproducible and pre-selectable electron dose.

Variation of Illumination Aperture (Electron Dose):

Independent of illumination field size and vice versa.

Selection of TEM and Spot Illumination: Push button selection between parallel wide-field (TEM) and spot (Nanoprobe) mode.

Constant Brightness Mode: Brightness automatically adapted to magnification.

Probe Size:

Parallel Wide-Field TEM-Mode: 1 μm to 75 μm (Illumination aperture from 0.02 to 5 mrad). Display of probe size, illumination angle and dose.

Spot (Nanoprobe) Mode: 1.6 to 100 nm

AIS (ZEISS Patent): Automatic Illumination-aperture Selection System. Magnification dependent restriction of illuminated specimen area to field of view on screen for optimum radiation protection of specimen.

MIS: Manual Illumination-aperture Selection System. Selection of illuminated area by the user to minimise specimen drift and prevent stray or diffracted electrons to be imaged. Allows easily quantifiable diffraction patterns to be recorded even from areas < 1 μm Ø.

Independent selection of brightness setting in both modes.

Illumination Tilt and Shift: Completely independent of each other, with double deflection system positioned above C-O pre-field lens; no shift compensation required when tilting and vice versa; no compensation required when switching from TEM to Spot (Nanoprobe) mode.

Beam Shift (MAG Mode): $\pm 100 \mu\text{m}$

Beam Shift (LM Mode): $\pm 1 \text{ mm}$

Minimum Dose Beam Shift: $\pm 25 \mu\text{m}$

Maintains true parallel illumination or selected convergence angle (requires MDF option).

Darkfield Illumination: With variable offset in Cartesian and polar coordinates (data sets can be stored).

Hollow Cone Illumination: Push button.

Illumination Stigmator: Factory aligned for different HTs. Recalable and user specific memories for MAG, Low Mag, TEM, and Spot.

Condenser Aperture: Metal bellows and click stop mechanism, operating as illumination field aperture. LEO patented drift minimised version. (Optional: pneumatic remote controlled high accuracy relocation aperture selection with high precision motorised fine positioning).

Aperture Sizes: One 5 hole aperture (37.5, 75, 150, 300 and 600 µm). Computer controlled setting (see AIS and MIS section). One additional position for standard 3 mm apertures (default 20 µm; optional: Top hat EDX aperture 30 µm or 5 µm for micro-diffraction and spot-scan).

Optional: 2nd multi hole aperture (800, 500, 320, 200, 125, 80 and 50 µm) with computer controlled setting (see AIS and MIS section) for use of condenser aperture also in low mag mode (requires pneumatically controlled aperture drive).

Unique Use of Condenser Apertures: Köhler illumination allows to use condenser apertures as selected area (SA) apertures. The illuminated specimen area defines the SA diffraction region on the specimen without selection error for SA areas (~ 1.25 µm for 37.5 µm and 0,17 µm for 5 µm aperture).

Standard Illumination System (Option)

The Standard Illumination System offers the same illumination characteristics known from a conventional TEM illumination system. In this system the illumination field is varied by the control of the final condensers. Due to a smooth beam profile and the continuous variation of the intensity, this system offers a high flexibility regarding the illuminated field. The known advantages of the condenser objective (C-O) pre-field lens design remain unchanged.

Requirements: Metal bellows and click stop mechanism pre-field aperture drive, with 3 single hole apertures (15, 30, 60 µm)

Projector aperture drive for SA diffraction

Condenser aperture drive with 30 µm top-hat aperture for EDS

Brightness control: via brightness knob on hard panel

Minimum Spot Size:

TEM Mode: 1 µm

Spot Mode: 7 nm (1.6 nm with active condenser zoom)

Illumination angles:

0.15 – 2.5 mrad (with 15 µm pre-field aperture)

0.15 – 10 mrad (with 60 µm pre-field aperture)

Objective Lens

| | | |
|--|---|--|
| Type | Truly symmetrical condenser objective lens. Special design concerning water flow, and symmetrical heat dissipation without any thermal effects to the specimen. | |
| Characteristics | | |
| Focal Length: | 3.0 mm | |
| Spherical Aberration (Cs): | 2.2 mm | |
| Chromatic Aberration (Cc[*]): | 2.2 mm | |
| Current Stability: | $\leq 1 \times 10^{-6}$ | |
| <small>* non relativistic</small> | | |
| Calibration Function (Autofocus): Normalization of objective lens field to calculated optimised value for eucentric reproducibility and focused spot. | | |
| Focusing | Continuous Focus: Automatic coarse/fine switching. Magnification depending focus step. Constant Focus: Over the entire range in LM- and SA-MAG-mode. Manual Defocus: Focus step size 0.5 nm to 60 nm, independent of magnification. Display of defocus on monitor. Mechanical Z-focus: Mechanical fine focusing of Z-axis. Focus Aid: to adjust Gaussian focus. Alignment of beam incidence: current centre, voltage centre and coma-free (assisted by image analysis software). | |
| Objective Aperture | Metal bellows and click stop mechanism; three single hole thin-film apertures (30 µm, 60 µm, 90 µm) located in the back-focal plane of the objective lens throughout the entire SA-MAG range (4,000x to 315,000x). | |
| Anticontamination Device | Standard anticontamination device integrated into the objective area connected to the dewar located in the pumping column. | |
| EDX Collection Angle | 0.13 sradi (Oxford EDX detector system, 30 mm ² detector) | |

Specimen Stage

| | |
|-------------------|--|
| Type | Eucentric side entry goniometer, ZEISS patent; 5 axes motorised: X, Y, Z, and α -tilt and optional β -tilt or rotation. |
| Characteristics | <p>Maximum Holder Tilt Angle*: $\pm 75^\circ / \pm 30^\circ \alpha / \beta$</p> <p>* corresponding holder spec compliance required</p> |
| | <p>Specimen Movements: X = 2 mm, motorised Y = 2 mm, motorised Z = ± 0.4 mm, motorised</p> |
| | <p>Specimen Drift: ≤ 1 nm/min</p> |
| | <p>Accuracy of Position Display: 0.1 μm</p> |
| Digital Control | Digital control, display, storage and relocation of all 4 (5) axes. Help function for eucentric positioning and objective lens normalisation. |
| Operation | Joystick or trackball (option) for X and Y positioning. Step width tracks with magnification. Step button for tilt control; variable tilting speed. Mechanical Z fine focusing by focus knob. |
| Specimen holders | <p>Standard Holder: Fast exchange specimen holder for two 3 mm grids.</p> <p>Low Background Holder (EDX) (Option): for 1 grid with Faraday cup.</p> <p>Double Tilt Holder (Option): $\alpha = \pm 75^\circ$; $\beta = \pm 30^\circ$, all axes motorised and digitally controlled; variable tilting speed.</p> <p>Other holders available on request.</p> |
| Special Functions | Automatic detection of holder type for optimum safety and instrument security at all tilt angles (ZEISS patent pending). Optional 90° goniometer tilt for cryo lock-in from front side. |

Imaging System

| | |
|----------------------|--|
| Type | Magnification Zoom for imaging, diffraction and spectroscopy with 7 imaging lenses: objective, 3 intermediate lenses, Omega spectrometer, 2 projector lenses |
| Magnification Ranges | <p>Total Range: 8x to 630,000x.</p> <p>Low Mag Range: 80x to 2,000x.</p> <p>Mag (SA) Range: 4,000x to 315,000x. (Parfocal).</p> |

High Mag Range: 400,000x to 630,000x

EELS Spectrum Imaging: 20x to 315x.

Overview Function (Option): Continuous or stepwise decrease of the magnification or the camera length down to a factor of 0.1 of the normal magnification range without affecting objective lens settings and without removing objective aperture, e.g. reaching 500x Mag from original 5,000x by push of a button. Used to e.g. adjust the field of view (originally imaged on a sheet film) to the size of a CCD detector.

Objective Lens

Magnification: 30x

Camera Length

SA Mode: (24mm*) 240 mm to 6,000 mm (*with overview function)

Low Angle Diffraction: (1m*) 10 m to 800 m (*with overview function)

Lens Programs: Optimised for every high voltage; free lens program for user definition (option).

Electromagnetic Image Shift System

Constant Shift: Independent of imaging mode and magnification.

Maximum Image Shift: ± 100 mm on viewing screen; separately memorised values for MAG image and diffraction, LM image, diffraction and spectrum imaging.

Stigmators

Separately memorised values for MAG image and diffraction and Low-Mag/Low angle diffraction.

Selector or Intermediate Aperture (Option)

For optimum contrast in Low Mag mode, additional selected area apertures or LACBED. Click stop, metal bellows type aperture rod. Three 3 mm disk apertures (50, 100, 200 μm or defined by user). Apertures in focus throughout the entire magnification range.

Spectrometer

Type

In-column OMEGA type with 4 sector magnets and 1 multipole correction element in the plane of symmetry. 2nd order aberration optimised factory alignment.

Dispersion

Energy Dispersive Plane: 1.17 $\mu\text{m/eV}$ at 120 kV

Final Image Plane: 23 $\mu\text{m/eV}$ to 370 $\mu\text{m/eV}$ (variable in 11 steps and continuously)

Energy Range: 0 to 2,500 eV

| | |
|---------------------------------------|--|
| Energy Loss Adjustment | Step Width: 0.2 eV (digital) |
| Image Isochromaticity | At Filter Achromatic Image Plane: 2.3 eV in 0.5 mm At Final Image Plane: (Max. FOV specimen area) 10.0 eV in 100 mm Ø = Plate (10 µm) 2.0 eV in 50 mm Ø = 2kx2k SCCD (5 µm) 1.0 eV in 25 mm Ø = 1kx1k SCCD (2.5 µm) |
| Transmissivity | 0.285*10 ⁻³ µm ² at 120 kV and ΔE = 2.0 eV. |
| Acceptance Angle | In Diffraction Mode with Detector Optimised CL: $\Theta_{obj} = 80 \text{ mrad}$ ($\Delta E = \pm 6 \text{ eV}$). |
| Spectrum Stability | Drift: < 0.5 eV/min |
| Distortion | Deviation from circular diffraction pattern: < ±1.0% |
| Entrance Aperture | Type: Click stop, metal bellows aperture rod (1000, 800, 100 µm) |
| Energy Selecting Slit Aperture | Standard: click stop, metal bellows type aperture rod with slit widths in two groups of 2, 4, 8, 12, 500 and 20, 30, 50, 70, 500 eV. One 1000µm fix aperture for parallel EELS. Different slit widths within a group are selected by a deflection system. Option 1: click stop, metal bellows type aperture rod variable from 0 to 85 eV (at 120 kV) with 1000µm fix aperture for parallel EELS. Option 2: High precision, fully automatic, remote controllable slit selection system with slit widths of 2, 4, 8, 12, 20, 30, 50, 70 and 500 µm and one 1000µm fix aperture for parallel EELS. |

Accessory Chamber and Viewing Chamber

| | |
|-----------------------------------|---|
| Shutter | Electromagnetic (below specimen) |
| Ports for Accessories | Two large wide angle ports (110 x 90 mm) for TV- and/or SCCCD-camera and/or third party HAADF detector. Image on monitor and on screen are simultaneously in focus. |
| Central Beam Stop (Option) | Pneumatically actuated with micro positioning in x / Y direction. Installs into right hand wide angle port. Beam stop will be above side-mount TV / SCCCD camera or third party HAADF detector. |
| Binoculars | Magnification: 9x, ± 35 mm shiftable in X direction |

| | |
|-------------------------------|--|
| Chamber Viewing Window | Circle section 215 mm Ø, 147 mm height. |
| Fluorescent Screens | Large screen 145 mm diameter; small screen 70 x 20 mm for focusing; automatic and remote operated pneumatic lift. |
| Bottom Port Flange | Opening large enough to accept a 4k x 4k bottom-mount SCCC-camera with ~ 20 µm pixel size or other detectors (BF /DF, TV cameras) in on-axis position. |

Vacuum System

| | |
|--------------------------|---|
| General | Dry and fast split-flow turbo-molecular pumped vacuum system with integrated adsorption trap in pumping column. Optional differential pumping system with ion-getter pump for gun area. |
| Vacuum Pumps | Pre Pump: 5 m ³ /h oil-free scroll pump with 20 l buffer tank. Split-flow Turbo-Molecular Pump: 250 l/sec for camera, viewing chamber, spectrometer and high vacuum in column/gun. Ion Getter Pump (optional): 50 l/sec for high vacuum in gun. |
| Pressures | End Vacuum Column: < 5x10 ⁻⁷ hPa (TMP pumped) End Vacuum Emitter Area: < 5x10 ⁻⁷ hPa (TMP/IGP pumped) End Vacuum Camera: <1x10 ⁻⁴ hPa (TMP pumped) |
| Air Lock | Automatic controlled Lock-in: <20 sec Lock-out: without delay Repeated specimen exchange without wait-necessity possible. |
| Special Functions | User selectable N2 flush cycles for air lock Manual closure of the column valve(s). IGP low pressure switch off override. |
| Vacuum Control | Fully automated, separate microprocessor control unit with bi-directional data link to host computer. Vacuum menu and operation controls on monitor. Stand alone operation of the vacuum (energy saving mode). |

Instrument Control

| | |
|--|---|
| General | Mouse driven system operation by menu-oriented graphical user interface (GUI). Hard panel control for most common functions. Complete data record storage and personalised storage of user parameters on hard disk. |
| Expert Operating System | Driving electron optics and controlling the subsystems; interface and safety filter for remote operation; comprising a host computer with Windows® operating system. |
| Dedicated Autonomous Subsystems | Autonomous microprocessor subsystems for monitoring and control of vacuum system, goniometer, high voltage and system status. Immediate protection circuit response and host communication. |
| Multilayer Management System | Event-driven, bi-directional communications between host computer and subsystems through optical CAN-bus. Direct control of lens and deflection systems through multiplexing control system. Full remote capability through 2x RS232 and 1x TTL interface. |
| User Alignment Support | Wobble high voltage; wobble focus (objective lens current); focusing aid (tilt axis wobbler), coma free alignment (by third party image analysis software); adjust eucentric axis (automatic stage tilt) |
| Factory Aligned Calibration Functions | For automatic alignment of the microscope to service settings: Image shift; illumination shift and tilt; magnification; image and illumination stigmators. |
| Store/Recall/Reset User Parameters | Image shift; illumination shift and tilt; focus (optimum objective current); magnification; brightness; spot size; energy loss value; spectrum magnification; defocus values; user macros; image stigmators; illumination stigmators; all operating modes; AIS; DF; dose; emitter alignment; goniometer; camera and MDF. |
| Software Options | <ul style="list-style-type: none"> - Tilt, focus, and exposure time series - Minimum Dose Focusing (MDF) - User defined calibration of magnification values - Free lens control - Measurement software - Remote control (2 RS232 and 1 TTL Interface possible) - Electron dose measurement - Overview function - Spot- and slitscan illumination |

Integrated Computer Environment

| | |
|--------------------------|--|
| Central Processor | Type: Minimum Intel 2.8 GHz, 512 MB RAM. |
| Operating System | Microsoft® Windows® 2000 / XP. |
| External Comms. | Serial, parallel, and USB. |
| Network | 10/100 base Tx on board. |
| Disk Drives | Hard-Disk: Minimum 120 GB EIDE standard. Floppy-Disk: 1.44 MB drive. CDRW-DVD: EIDE standard. |
| User Interface | Keyboard: Windows® enhanced type. Mouse: Three button serial mouse for instrument control. Control Panels: Hard-keys, knobs and dedicated rotary knobs for most common functions. Monitor: High brightness 18" TFT flat screen monitor; maximum pixel resolution 1280 x 1024. |

Safety Features

| | |
|--------------|---|
| Types | Fully protected power line failure. Water failure circuit. Continuous monitoring of vacuum system operation with emergency shut down and error display on monitor. Air pressure failure circuit. Electronic and lens temperature failure circuit. Subsystems (emitter, goniometer, camera, vacuum) with failure circuits independent of the microscope controller (PC). Safety check for correct operation of remote control commands from external computers. |
|--------------|---|

Sheet Film Camera (Option)

| | |
|----------------------|---|
| Type | Fully automated separate microprocessor control unit with bi-directional data link to host computer. |
| Film Capacity | Max. 45 sheet films 3.25" x 4" or 20 plates or sheet films 65 x 90 mm. Digital display of film supply on monitor. |
| Exposure Time | Fully automatic measurement or manual input. Shutter speed 0.2 to 100 sec for automatic exposure; 0.2 to 300 sec for fixed exposure time. |

Data Recording

The following data-set is automatically included in the negative:
User initials, negative No., magnification or camera length,
 μ -marker (image mode) or spectrum magnification (spec. mode),
high voltage, energy loss (E), electron collector current or
defocus or tilt angle, date, user defined 16 alphanumeric
characters via keyboard, data recording on printer or hard disk
with additional comments for documentation (option).

Special Exposure Modes

Automatic Defocus Modes:

- a) Optimum magnification dependent underfocus (low, med., high).
- b) Scherzer defocus compensated for acceleration voltage.
- c) Fixed defocus selectable from 2 nm to 30 μ m.
- d) Display of defocus value on monitor.

Multiple exposure. Automatic through focus, tilt and exposure time series (options).

Control Functions

Exposure time out of range (over and under exposure). Film supply. Sheet film transport.

Digital STEM Attachment (Option)

General

Enables digitally controlled scan of focussed probe for image formation from detectors above and below the sample. Switch over from TEM to STEM via push button, no change of condenser aperture, no variation of focus, stigmation, and specimen position: Fully digital and thus reproducible manual and remote control. All STEM modes available using the energy filter.

Resolution

See resolution section.

Magnification Range

50x – 1,000,000x

Deflection System

As a standard built in to the basic microscope.

Choice of Detectors

Parallel BF and annular DF STEM or 3rd party HAADF (option).

Scintillator type SE-detector (option).

Solid state BSE detector in place of the objective pre-field aperture (option).

| | |
|---------------------------|--|
| Signal Input | Free selection of detectors. Automatic or manual contrast and brightness control. Scan speeds: 15 non-interlaced scan speeds from 0.09 sec/frame to 42 min/frame. Scan modes: Normal raster, line scan and spot mode (freely defined within frozen raster image). Noise reduction: Frame/line average or integration modes. |
| Store Resolution | 512 x 384, 1024 x 768, 2048 x 1536, 3072 x 2304; maximum display resolution is 1024 x 768 on 18" TFT monitor. |
| Image Input/Output | Format: BMP, JPG, or TIFF (8-/16-bit). Printers: ZEISS uses standard Windows® printer drivers allowing flexibility in choice of local or networked printers. |

Additional Options

EDS-Systems
Image Analysis Systems, Image Processing, Image Archive
Side Mount SSCCD cameras
Side Mount TV cameras
Pneumatically operated beam stop
VarioSpeed, and MultiScan, SSCCD bottom mounted cameras
Image Plate System
Cryo transfer and cryo preparation systems
Water recirculators
Compressor
Preparation equipment (Plasma cleaner, etc) for TEM
Additional options on request

10 Installation

10.1 Room specifications

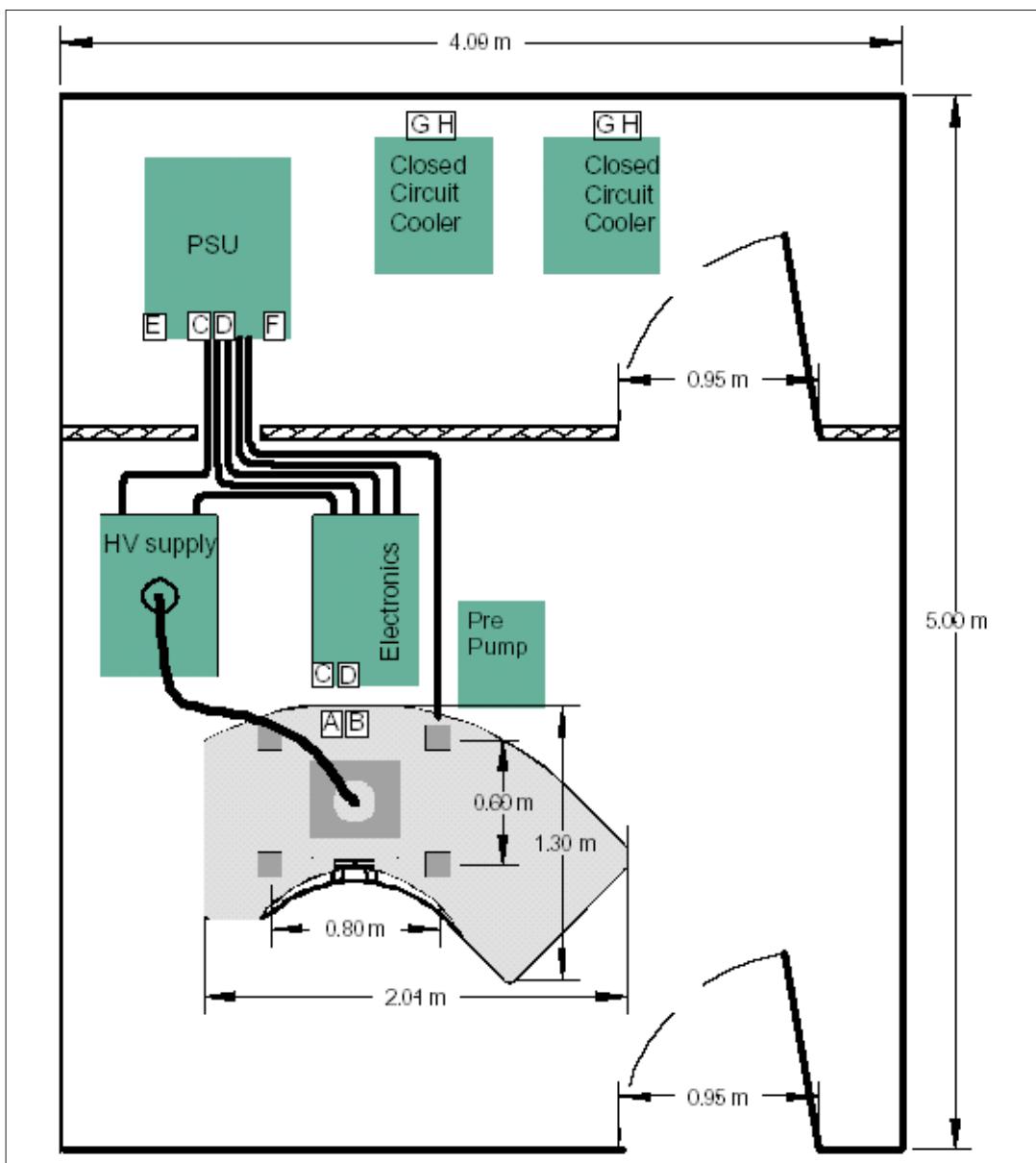
10.1.1 Room size



NOTICE:

It is strongly recommended to use an acoustically separated room ("utility room": minimum 1.5 m x 4.0 m) for installation of power supply unit and closed circuit cooler.

| | |
|----------------------------|--|
| <i>Total room area</i> | recommended: 5.0 m x 4.0 m Minimum: 4.5 m x 4.0 m |
| <i>Ceiling height</i> | recommended: 2.8 m Minimum: 2.6 m |
| <i>Minimum floor space</i> | foot print: 2.1 m x 2.1 m |
| <i>Distances</i> | from power supply unit to TEM column: min. 2.0 m, max. 3 m from HV supply unit to TEM column: min. 1.5 m, max. 2.8 m from HV supply unit to control electronics: min. 0.5 m, max. 1 m |
| <i>Accessibility</i> | right/left of console: min. 0.8 m front of console: min. 1.2 m back of console: min. 1.2 m |
| <i>Statics of the room</i> | The allowed static load must be checked by a stress analyst. For dimensions and weights of the electron microscope see section "Product data". |



| Supplies | | |
|----------|---|--|
| A | Compressed Dry Air (CDA) | |
| B | Nitrogen (N ₂) | |
| C | Cooling Water IN | |
| D | Cooling Water OUT | |
| E | Main Power IN | |
| F | Ground | |
| G | Closed Circuit Cooler – Power IN | |
| H | Closed Circuit Cooler– Cooling Water IN/OUT | |

10.1.2 Crane system

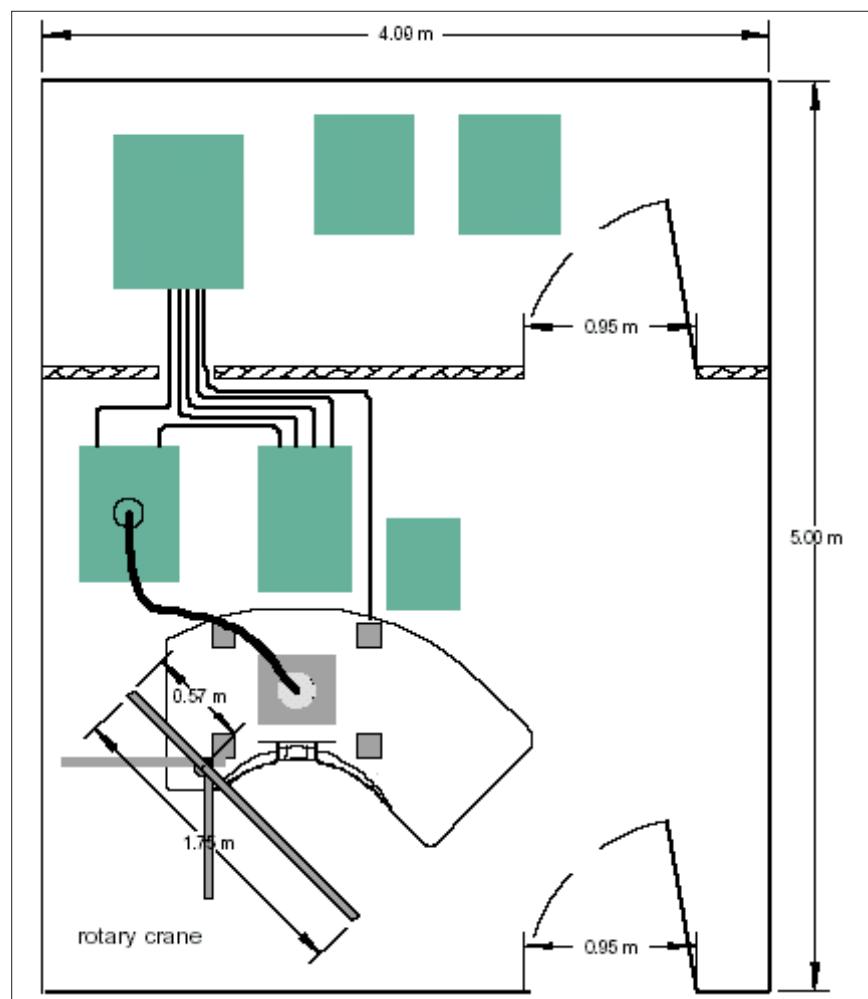


A crane system is required for installation and service of the electron microscope i.e. for lifting the TEM column. It is recommended to install the following fixed crane system:

- Rotary crane (one sided fixed swivel arm system, see figure on this page).
The minimum distance required between top of the TEM column (2460 mm for differential pumping system, 2390 for non-differential pumping system) and crane hook is 260 mm.
Please contact ZEISS SMT for more details.

Required lifting capacity: 600 kg min.

Example of floor plan for the rotary crane:



10.1.3 Transport passages

| | |
|---------------------------|---|
| <i>Door opening</i> | height: min. 1.9 m width: min. 0.8 m |
| <i>Passage clearances</i> | passage height: min. 1.9 m passage width: min. 1.2 m |

10.1.4 Environmental conditions

Electromagnetic stray fields, mechanical vibrations (floor vibration) and acoustic noise will have a negative effect on the performance of the electron microscope. The compliance with the environmental requirements is within the responsibility of the customer and has to be provided at the time of installation.

Select locations far away from sources of electric and magnetic stray fields such as transformers, facility power lines, large electrical machines, railways or street cars.

Place the microscope in the basement of the building. Separation of the microscope foundation from the rest of the building is recommended. Avoid rooms near potential sources of vibration such as heavy machines, compressors, air conditioning supplies, busy roads, underground railway, lift shafts etc. Do not place the microscope in the direct flow of an air conditioning system. Restrict air flow to a minimum.

The microscope room should provide as close as possible an 'acoustically dead' environment.

The ZEISS SMT Technical Service will be happy to assist the customer by identification of the environmental state: Qualification measurements will be performed by a trained ZEISS service engineer using certified ZEISS analysis instrumentation.

Electromagnetic stray fields

(*x, y and z direction*) Maximum values for B (peak to peak), measured in heights of 1.50 m, 1.90 m and 2.50 m:

$$B_x = \sqrt{\frac{f}{50\text{Hz}}} \cdot 100 \text{nT} \quad B_y = \sqrt{\frac{f}{50\text{Hz}}} \cdot 100 \text{nT} \quad B_z = \sqrt{\frac{f}{50\text{Hz}}} \cdot 200 \text{nT}$$

That means e. g. for a frequency of **f = 50 Hz**:

$$B_x = 100 \text{nT} \quad B_y = 100 \text{nT} \quad B_z = 200 \text{nT}$$

| | | |
|---|---|---------------------------------|
| <i>Mechanical vibrations</i> | 0 - 5 Hz and 12 - 20 Hz: | < 5.0x10 ⁻³ mm/s rms |
| | 5 - 12 Hz: | < 2.0x10 ⁻³ mm/s rms |
| | > 20 Hz: | < 2.0x10 ⁻² mm/s rms |
| <i>Acoustic noise level</i> | 150 Hz to 300 Hz: | max. 48 dB. |
| | All other Frequencies: | max. 50 dB. |
| <i>Room illumination</i> | <ul style="list-style-type: none"> - bright illumination source for servicing - dimmed illumination source for adjustment of room light down to complete darkness - red light illumination for plate camera exchange | |
| <i>Room temperature</i> | operation: + 21 °C ± 4 °C storage or transport: - 10 to + 50°C | |
| <i>Temperature fluctuation during operation</i> | max. 1° C/h | |
| <i>Relative humidity</i> | max. 65 %, dew point below 18°C | |
| <i>Air conditioning capacity</i> | microscope room: 2.0 kW utility room: with air-cooled closed circuit cooler WK 3200/A: 6.5 kW with water-cooled closed circuit cooler WK 3200 W/B: 1.0 kW | |
| <i>Floor covering</i> | anti-static, washable material, no carpets (essential to obtain the required cleanliness) | |
| <i>Cleanliness</i> | clean zone according to ISO 7 or higher Clean the floors twice a week. Use air conditioning with air flow filters. No brittle or porous material for ceilings and room walls. | |

10.2 Utilities

10.2.1 Electrical supplies

Make sure to avoid systemic ground loops in the electrical installation near or in the microscope room. Ensure strict “star shaped” grounding of all electrical equipment. Check for ground loops/compensation currents on heat/air conditioning pipes.

| | |
|-----------------------------------|---|
| <i>Electrical supplies</i> | European standard: 3 phases, 400 V ± 10% / 50 Hz US standard: 3 phases, 208 V ± 10% / 60 Hz in combination with transformer. |
| <i>Frequency</i> | European standard: 50 Hz ± 3,5 Hz US standard: 60 Hz, ± 3,5 Hz |
| <i>Connectors</i> | European standard: CEE 32 A, 3/N/PE 400V/230V AC US standard: 3/PE |
| <i>Fuse protection</i> | European standard: 32 A (slow-blow fuse) US standard: 40 A (slow-blow fuse) |
| <i>Voltage fluctuation</i> | European standard: ± 10% US standard: ± 10% |
| <i>Line breakdown</i> | max. 20 ms |
| <i>Apparent power consumption</i> | 5.5 - 6.5 kVA (max. 13 kVA) |
| <i>Grounding</i> | Protective ground: 10 mm ² Separate second grounding is essential. |

10.2.2 Compressed air supply

Compressed air is required for damper pneumatics and valve operation.

Compliance with SEMI standard requires lockable faucets for compressed air.

| | |
|---------------------|-----------------|
| <i>Air pressure</i> | 6-7 bar |
| <i>Air purity</i> | 0.97 (oil free) |

Air consumption max. 1 l/min

Type of connector PU4 hose, 4 mm diameter inside

10.2.3 Nitrogen supply

Gaseous nitrogen is required for ventilating the column chambers. When handling nitrogen, take the appropriate safety precautions. Compliance with SEMI standard requires lockable faucets for nitrogen supply.

Nitrogen pressure 0.1 – 0.3 bar

Nitrogen content min. 99.96%, water content less 5 vpm

Type of connector PU4 hose 4 mm diameter inside, 6 mm diameter outside

10.2.4 Cooling water supply

There are two loops for cooling water: 1) Power supply unit , 2) System electronics and lenses. Compliance with SEMI standard requires lockable faucets for cooling water supply.

Water pressure 5 bar max.

| | | |
|-------------------|---|---------|
| <i>Water flow</i> | Loop 1 (power supply unit): | 300 l/h |
| | Loop 2 (system electronics and lenses): | 300 l/h |

Type of connector ½" Nozzle

| | | |
|----------------------------------|---------|--------|
| <i>Required cooling capacity</i> | loop 1: | 3.2 kW |
| | loop 2: | 3.2 kW |

If above specification cannot be met by a in-house cooling water supply it will be necessary to install a closed circuit cooler.

ZEISS recommends LAUDA closed circuit coolers: Lauda WK 3200/A (air cooled) or Lauda WK3200 W/B (water cooled). Each cooling water loop requires a closed circuit cooler, i.e. two coolers are required.

10.3 Product data

| Dimensions and weights | Length (mm) | Depth (mm) | Height (mm) | Weight (kg) |
|------------------------------------|----------------|---------------|----------------|----------------|
| Basic instrument with table top | 2010 | 1310 | 2460 | 875 |
| Basic instrument without table top | 980 | 835 | 2460 | 860 |
| Column with base plate | 850 | 650 | 1700 | 585 |
| Support frame | 930 | 730 | 730 | 295 |
| High voltage unit | 780 | 560 | 1608 | 250 |
| Power supply | 800 | 720 | 1550 | 330 |

Conformity

The electron microscope complies with the following standards and directives:

89/336/EEC Electromagnetic Compatibility 73/23/EEC

Low Voltage Directive changed by 93/68/EEC

X-Ray information

During operation X-rays are generated within the electron microscope. Therefore the user of the electron microscope must observe the specific national X-ray regulations on installation, start-up, operation and maintenance.

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