Tecnai G^2 20/30
Operating Manual

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PREFACE

About this Manual

The Operating Instructions of the Tecnai microscope are divided into a paper part (this manual) and a digital part (the on-line help). The digital part forms the major portion of the manual, with the paper part covering only the essentials (a chapter on safety aspects, one on getting started, and one on maintenance; the latter because it is not practical to use a digital manual for the operations required for maintenance).

A selection from the on-line help files is also assembled in Word documents, allowing easy selection of parts to be printed. These documents are located in a dedicated folder in the \Tecnai\tem_help folder on the hard disk of the instrument. Two versions of each Word document are made, one with "_A4" in the file name (covering the European A4 paper format), the other with "_Let" (covering the American Letter format). PDF versions, if preferred, of the same are also available in the same location.

This *Pinted Manual* for your Tecnai Electron Microscope is divided into the following chapters:

- **1. SAFETY AND HANDLING** provides important information for product and personal safety.
- **2. STARTING WITH THE TECNAI** gives procedures for system start-up.
- **3. MAINTAINING YOUR TECNAI** step by step cleaning procedures.

How to Use this Manual

At the beginning is a main Contents, List of Figures and a List of Tables covering the whole Manual. Each chapter has a Contents of the subjects specific to that chapter. Included in some chapters are easy-to-follow tables and figure number references are associated with the text. High-lighted text can also be found in descriptive paragraphs to aid association of items.

Notes, Cautions, and Warnings

Important highlighted information is indicated by either of the following three categories expanded into the left column:

NOTE: Notes give information or advice on general technical matters.

CAUTION! Cautions makes the user alert to possible instrument damage.

WARNING! Warnings makes the user alert to possible Health hazards. Where these dangers occur the relevant logo is displayed.

Finding What You Need

This manual has been organized so you can find information in several ways. You can read the manual from beginning to end (highly recommended but rarely done). Be sure to read the safety section, before operation, in Chapter 1.

Once your system is up and running, you can search for information in the main contents or the individual contents preceding each chapter.

Major headings have been hung in the left column to help you scan for the basics within a chapter. That column provides space for your own notes as well.

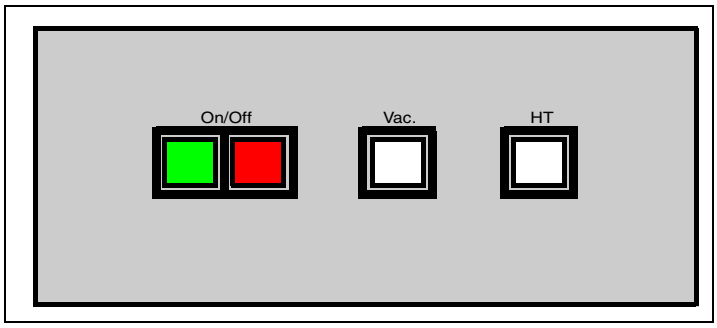
Tables and Figures are numbered within each chapter and are listed after the main Contents for the whole manual.

1 SAFETY AND HANDLING

Emergency controls

The microscope is equipped with several emergency controls, located in a separate panel on the right-hand side of the column support.

FIGURE 1-1 SYSTEM ON / OFF PANEL (SOOP)



These emergency controls have the following functions (from left to right):

TABLE 1-1 BUTTON FUNCTIONS

Label	Color	Function
ON	**Green**	Switches power supply to the microscope on, lit when microscope is off
OFF	**Red**	Switches power supply to the microscope off, lit when microscope is on
Vacuum	**White**	Vacuum on / off
HT	**White**	Enables/disables HT on (if this is off, HT cannot be switched on)

The Vacuum on / off and HT Enable controls provide safety and at the same time allow switching the HT on through software. For example, it is not possible to switch HT on from a remote site, while a service engineer has disabled it.

Emergency procedures

In the case of a fire or other emergency (building evacuation), perform the following actions **if time permits (do not risk your own safety for shutting the microscope down)**:

1. Press the OFF button on the Emergency Control panel. The microscope will shut down and thereby minimize any hazard to persons in its neighbourhood as well as itself.
2. Switch the PC off by pressing its on/off button. Do not use the normal Windows NT shutdown procedure.
3. Immediately leave the room containing the microscope.

Handling SF_6 gas

Introduction

SF_6* gas is used as insulating gas in the high-voltage tank and the emission chamber of the Tecnai 200 and 300 kV instruments. This gas has been used for many years in many industrial environments. It is non-poisonous at temperatures below 250°C.

- SF_6 Sulphur-hexafluoride gas, colourless, odourless, non-flammable, non-poisonous if not heated above 250°C.

The high-voltage tank and emission chamber of the Tecnai 200 and 300 kV instruments are both sealed volumes and normally there will be no leakage of gas from these parts. Nevertheless, International Regulations require that certain safety procedures regarding SF_6 gas must be known and implemented by the person responsible for the installation. Additionally, any local regulations concerning SF_6 gas must also be observed.

WARNING! In the event of fire, SF_6 will decompose to highly poisonous fluorine if the temperature exceeds 250°C. Ensure that the relevant local regulations are obeyed.

(The rest of this page has been left blank intentionally for the insertion of local fire regulations).

General

- SF_6 gas is heavier than air.
- The gas causes suffocation at high concentration levels.
- The gas breaks down when overheated (above 250°C) giving off highly toxic fluorine gas.

Therefore:

- The ventilation system must run (extract) **continuously**.
- **Smoking** in SF_6 sensitive areas is strictly **prohibited**.
- The SF_6 detector must always be in operation.
- Gas masks (2x) and rubber gloves (2 pairs) must be present. Gas masks should have filters of active charcoal and incorporated dust filter.

In the event of a Gas leakage

- Switch off all heat sources (including any heat-producing lights).
- Ventilate the area by opening all windows. **Do not open the door**, since this allows the heavy gas to flow into the building.
- Switch off all heat sources.
- Trace and stop the leakage.
- All parts contaminated with solid decomposition products must be handled with rubber gloves.

What to do if SF6 decomposes and Fluorine is given off

NOTE: Even the presence of very low concentrations of fluorine can be detected by smell.

- Put on a gas mask.
- Ventilate the area (open windows, but keep door closed!).
- Switch off all heat sources.
- Trace and stop the SF_6 gas leakage.
- All parts contaminated with solid decomposition products must be handled with rubber gloves.

Safety precautions

Ventilation of the area

- As SF_6 is heavier than air, it will sink and stay at floor level. Therefore, there should be an extraction ventilation channel opening about 10-15 cm above the floor. This ventilation channel should open directly to outside air and **may not be connected to the central ventilation system of the building under any circumstance.**

- All holes in the floor giving possible access to lower floors in the building should be closed.
- When a filament (Wehnelt) of the 200 and 300 kV LaB_6/W instruments is replaced, a certain amount of SF_6 gas must be pumped away with the pre-vacuum pump of the microscope.

Because of this, the outlet of the pre-vacuum pump should be connected in such a way that the pump vents directly to the atmosphere outside the building and under no circumstances to the central ventilation system of the building.

SF_6 detection

For full safety, an SF_6 detector should be present and operated continuously. The detector must be a suitably calibrated instrument based on the thermal conductivity principle.

Illumination of the area

As SF_6 gas decomposes when heated to temperatures above 250°C, fluorescent-tube illumination should be used and not conventional tungsten lamps.

Specifications

The SF_6 gas used must meet the following specifications:

TABLE 1-2 SF_6 GAS SPECIFICATIONS

	SF_6 (IEC)		Esaflon
SF_6 minimum contents	Weight %	99.9	99.4
Air	Weight %	max. 0.05	max. 0.01
CF_4	Weight %	max. 0.05	max. 0.05
Water	Weight ppm	max. 15	max. 2
Acidity, calculated as HF	Weight ppm	max. 0.03	max. 0.3
Hydrolysable fluoride, calculated as HF	Weight ppm	max. 1	max. 1
Mineral oil	Weight ppm	max. 10	max. 1

The MONTEDISON company is a manufacturer of SF_6 gas.

2 STARTING WITH THE TECNAI

Getting started

Logging in

In order to use the microscope you must have an account on the Windows 2000 computer. To start using the microscope simply log in to Windows 2000 (the standard procedure: press Ctrl+Alt+Del, enter your name and password). From this log-in the microscope knows who you are, what your level of microscope user interface is (user or expert), and where to find your personal settings for microscopy.

In general the microscope user interface will start up automatically after you have logged in. This is, however, not required (it simply means that the Tecnai user interface program is in the general or user Startup folder of the Start menu). As an alternative, your microscope supervisor may have changed these settings and you may have to start the user interface by selecting it from the Start menu (or a shortcut on the Windows 2000 task bar).

If you need the PC of the microscope but do not require the microscope user interface (e.g. for processing data or transferring them via a network to elsewhere), you can close the user interface. This has no effect on the running of the microscope.

NOTE: The microscope will keep running even if the user interface is closed. The only thing the user interface does is allow you to interact with the microscope.

If the microscope is not running, contact the microscope supervisor (starting the microscope software requires Windows 2000 privileges you may not have).

Making the first image

1. If necessary, start the Tecnai user interface.
2. Fill the cold trap with liquid nitrogen and check the vacuum.
3. Make sure HT enable is on and the high tension is on at the required high tension value.
4. Insert a specimen into a specimen holder and insert the holder into the microscope.
5. Switch the filament on to the required setting.
6. If the vacuum in the specimen area is good enough, open the column valves. The beam should now be visible.

7. Select a reasonable magnification (Magnification knob; not too high, initially the LM range is often good).
8. Center (with the left-hand track ball) and adjust (with the Intensity knob) the beam, and focus (Focus knob) the image.
9. Find a suitable specimen area with CompuStage goniometer.
10. Select a suitable magnification and focus the image.
11. Select the workset tab containing the plate camera control panel.
12. Adjust the Intensity setting to give a normal exposure time (1 second or more).
13. Press the Exposure button (the screen will be lifted, a plate loaded, the exposure recorded, the plate unloaded and the screen turned down again).

You now have finished recording your first image.

Logging out

When you are finished with working with the microscope, you simply log off (in the Start menu, select Log Off). **Do not select shut down the computer because that will also effectively shut down the microscope.** If you only log off, the microscope will keep running normally (except that nobody can change settings until logged in).

On-line help

The microscope is equipped with a comprehensive system of on-line help, accessible under the F1 function key of the PC keyboard. The on-line help system consists of a series of html (Hypertext Mark-up Language: Internet browser) files. The contents of these files are displayed in the on-line help window of the Tecnai user interface (but they can equally well be 'browsed' off-line using an Internet browser). The entry point is a file called Index.htm

The Help system has many ways of giving you information. If you want information on a certain topic such as a button or other control in one of the Control Panels, you can use the on-line help function (F1) to jump there immediately (click in the relevant Control Panel and press F1). The on-line help also contains an alphabetical and a topics index.

Part of the on-line help is available in digital form as (Acrobat) PDF documents, located in a dedicated folder in the \Tecnai\tem_help folder on the hard disk of the instrument. Two versions are made of each document, one with "_A4" in the file name (covering the European A4 paper format), the other with "_Let" (covering the American Letter format). This makes it easier to print without having to reformat everything.

3 MAINTAINING YOUR TECNAI

3.1 Maintenance Procedures

Overview

The table below summarises a series of maintenance activities which can be performed by the operator. Some of these combine the appropriate cleaning procedures, which are described later in this chapter, with standard operational sequences already described..

TABLE 3-1 OPERATOR MAINTENANCE

Maintenance activity	Section	Procedures required
Flushing with dry Nitrogen	3.2 “Flushing with dry nitrogen”	Flushing with dry Nitrogen
Cleaning the Wehnelt	3.5 “Exchanging the filament (and wehnelt aperture)” 3.3.3.1 “Cleaning the Wehnelt assembly”	Exchanging the filament (and Wehnelt aperture). Cleaning the Wehnelt assembly.
Cleaning apertures and aperture holders	3.4.3 “Dismounting aperture holders” 3.4.6 “Removing and replacing apertures in the holders” 3.3.3.2 “Cleaning aperture holders” 3.3.3.3 “Cleaning apertures” 3.4.4 “Remounting aperture holders”	Dismounting aperture holders Removing and replacing apertures in the holders Cleaning aperture holders Cleaning apertures Remounting aperture holders
Cleaning specimen holders	3.3.3.4 “Cleaning specimen holders”	Cleaning specimen holders
Cleaning film and plate holders	3.3.3.5 “Cleaning film and plate holders”	
Cleaning viewing surfaces	3.3.3.6 “Cleaning viewing surfaces”	

CAUTION! All parts operating in vacuum should be handled carefully using nylon gloves and stored in suitable containers when not in use.

3.2 Flushing with dry nitrogen

In all cases where the high-vacuum parts of the electron optical column must be opened, e.g. when the Wehnelt assembly or aperture holders have to be removed, dry-nitrogen flushing is strongly recommended.

The inlet to the emission chamber is provided with a suitable connector for this purpose. Dry-nitrogen supply should be delivered by a flexible tube and the supply regulated to a pressure of 0.1 to 0.2 Bar.

Flushing should be continuous from the moment of operating:

- Gun Air
- Col Air
- All Air

Once the emission chamber or column tube has been opened, it is recommended to continue flushing until evacuation begins again (when the inlet will again be closed)

NOTE: This will not apply when, for any reason, the column is to be left open for a longer period of time.

3.3 General cleaning procedure

Regular checks are essential to maintain the best performance.

It is stressed that cleaning is more successful if carried out before heavy deposits of contamination have been allowed to build up.

Moreover, charge effects on parts that have become heavily contaminated by the electron beam eventually cause instability of the image or illumination.

The goal is to clean the microscope parts with maximum efficiency during corrective or preventive maintenance, using a minimum of instruments, consumables and chemicals. The topics described in the text following are:

- Cleaning equipment and materials required
- Brand names "CIF" and “Soft Scrub”
- Warnings, danger classification and safety advice
- Code of practice
- Five-phase cleaning flow chart
- Explanation of the chart
- Summary

This is a five-phase cleaning procedure, highly effective, using a minimum of equipment and materials, and producing good results when the instructions are strictly followed.

NOTE: The instructions given here supersede all previous cleaning instructions. Ignoring these, or only carrying them out in part will result in poor performance of the microscope and will necessitate recleaning.

3.3.1 Cleaning equipment and materials required

Instruments:

- Ultrasonic cleaner
- Ultrasonic vapour degreaser (if available)
- Storage bottles (1l. size)
- Beakers, EM liner tube size and smaller
- Petri dishes
- Stereo light microscope (magn. 5x to 50x)
- Tweezers
- IR heating lamps
- EM special tools (removing of liner tube and diaphragms)

Consumables:

- Rubber gloves
- Safety goggles
- Lint-free clothes
- Lint-free gloves
- Dust-free tissue paper
- Grease-free cotton wool
- Wooden spills
- Aluminium foil
- "Gas Jet Duster" spray can

Chemicals:

- De ionised or distilled water
- Ethanol - C_2H_5OH
- Ethanol Pro Analysis (99.8% pure) - C_2H_5OH
- Industrial Soap (ph neutral)
- KOH (Potassium Hydroxide) or NaOH (Sodium Hydroxide)
- EXTRAN - MA02 (neutral cleaning fluid)
- CIF or SOFT SCRUB (household fine abrasive cleaner)

NOTE: Only use recommended solvents.

Danger Classification:

R11 Highly inflammable

R35 Causes severe burns

Safety advice:

S2 Keep out of reach of children

S7 Keep stored under sealed conditions

S16 Keep away from igniters

Do not smoke

S26 By contact to the eyes, rinse profusely with water,

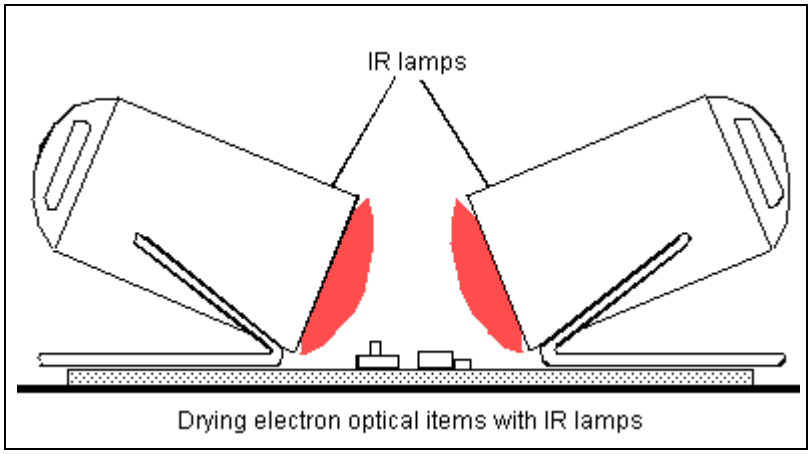
S37/39 Wear protective gloves and facial protection devices.

WARNING! As cleaning solvent Ethanol is highly inflammable, do not use open flames, or smoke while cleaning.
KOH and NaOH are corrosive solvents, prevent skin contact.
Ventilate the room properly.

3.3.2 Code of practice

- Do not open the column unless it is really necessary, but check the performance regularly.
- Always work in a clean and ***ventilated*** room
- Use a clean working table with good illumination
- Wear lint-free clothes
- Always wear clean gloves when handling "vacuum" parts
- Always use CLEAN solvent
- Before mounting, inspect parts under a light microscope
- Be sure that special tools are clean before use and packed after use in aluminium foil
- Do not mix materials of different compositions in the cleaning baths
- Strip the assemblies as far as possible
- Pack items in aluminium foil after cleaning
- Collect all cleaning items and have them on site before opening column
- Vacuum parts that cannot be remounted immediately can best be wrapped in aluminium foil and kept under an infra-red lamp.

FIGURE 3-1 USE OF IR LAMPS



Drying electron optical items with IR lamps

3.3.3 Specific cleaning procedures

The frequency of cleaning required will depend on many factors. The following check list is a recommendation only.

TABLE 3-2 CLEANING FREQUENCY

Components	Frequency
Projection chamber windows	As necessary
Binocular	As necessary
Wehnelt aperture	Every new filament
Wehnelt assembly	Once a year (W)
	Once every three years (LaB_6)
Aperture holders	As necessary, depending on astigmatism
Apertures	
Specimen holder (and specimen)	

3.3.3.1 Cleaning the Wehnelt assembly

Wehnelt cylinder and aperture

(Figure 3-2, items 8 and 10)

1. Remove the Wehnelt aperture-securing ring (11) and support ring (9) together with the Wehnelt aperture (10).
2. Remove the spring ring (13).
3. Follow the general cleaning procedure given in section 3.3 “General cleaning procedure”, starting with phase 1.

Wehnelt main assembly (1)

filament-securing ring (6) and spring washer (7); aperture support (9) and securing ring (11); Wehnelt cylinder spring ring (13) (Figure 3-2)

Clean, following the procedures given in section 3.3 “General cleaning procedure” starting with phase 2.

NOTE: When changing a filament, it is advised that the Wehnelt aperture should be changed at the same time and save cleaning of the apertures until the complete Wehnelt assembly is cleaned.

FIGURE 3-2 WEHNELT ASSEMBLY

3.3.3.2 Cleaning aperture holders

Follow the procedure given in section 3.3 “General cleaning procedure”, starting with phase 1 or 2 depending on the degree of pollution.

3.3.3.3 Cleaning apertures

3 mm Platinum type

Use one of the following methods:

Method 1

Heat the aperture (held in special tweezers with platinum points) in a clean gas flame until white hot.

Ensure that the aperture does not melt or become stuck to the tweezers.

Method 2

Connect a V-shaped (approx. 2 cm long) platinum-foil boat across the low-voltage secondary winding of a transformer, e.g. shadow coating unit, and heat until white hot.

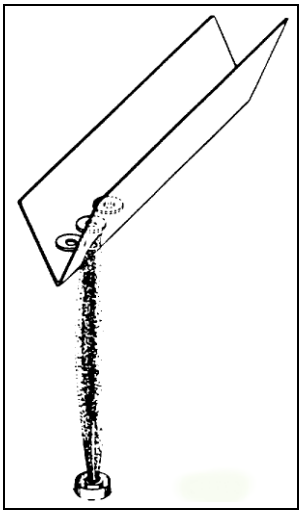
Place the aperture on the white-hot foil for a few seconds.

Take care that the aperture does not melt or become stuck to the foil.

Method 3 (see Figure 3-3)

Place the aperture on edge in a V-shaped platinum-foil boat (approx. 2 cm long) and hold the boat in a clean gas flame, using the special platinum-tipped tweezers, until the aperture is heated to a white colour. Maintain colour for 15 - 30 s.

FIGURE 3-3 CLEANING APERTURES



Method 4

Place the aperture on a V-shaped piece of molybdenum foil which has been connected to the source leads of a shadow coating unit using a vacuum of 10^{-5} Torr.

Heat the aperture to a cherry red colour and maintain this condition for 30 seconds. Allow the aperture to cool and then remove it from the foil.

Thin-foil type

In general, these do not need cleaning. The manufacturer suggests that, if necessary, they can be cleaned in the microscope by heating the edge with the focussed beam. Care is required in this kind of treatment or they could melt.

CAUTION! These apertures are very fragile and are easily damaged by tweezers or sudden rushes of air into the vacuum system.
Do not clean using the techniques described for platinum apertures.

3.3.3.4 Cleaning specimen holders

Since contamination of the specimen holder can have a considerable adverse influence on the image quality, a regular check of cleanliness is necessary.

When mounting a specimen, it is advisable to check whether there are (in general brown) deposits or parts of former specimens remaining on the holder. If there are, cleaning is necessary.

It is strongly advised that this is done by the service engineer during a preventive maintenance visit.

3.3.3.5 Cleaning film and plate holders

During normal operation in a laboratory, the containers for photographic material (plate holder, plate magazine) will be stored for longer or shorter periods in the darkroom where the conditions are, in general, not ideal for components which are intended for high-vacuum use. Even normal handling will introduce skin grease and humidity into the vacuum system.

Therefore, ensure that these components are free of such contamination before introducing them into the vacuum and to check their cleanliness regularly. If necessary they should be cleaned with a degreasing solvent such as ethanol p.a. in an ultrasonic cleaner.

3.3.3.6 Cleaning viewing surfaces

Clean the outside of the viewing chamber windows and the outside of the binocular glasses regularly. This is done by rubbing with a soft cloth or lens paper. Excessive dirt or grease is removed by using any commercial domestic glass cleaning solvent.

3.4 Apertures

3.4.1 Types of aperture holder

Two types of aperture holder are employed:

- The Diffraction and Objective lens aperture holders are identical and constructed of copper-aluminium.
- The Condenser aperture holder is thicker and constructed of copper.
- The Objective lens aperture holder for the S-TWIN lens is thinner than the normal holder to allow a greater tilt angle. This holder contains 8 apertures of different sizes.

Holders should not be exchanged between lenses, even when mechanically identical, since the adjustment of the end stops for the axial fine centering movements are made individually for each holder.

CAUTION! All aperture holders should be replaced in the lenses from which they were taken.
The aperture holders operate in high vacuum and should therefore be handled carefully using nylon gloves.

3.4.2 Types of apertures

Three types of apertures are used:

TABLE 3-3 APERTURE MATERIALS AND APPLICATIONS

Type	Lens	Advantages	Maintenance
Platinum (normal)	Objective Diffraction	No electron leakage; low cost; low contamination	Cleaning sometimes required
Platinum (thick)	Condenser when carrying out X-ray work	Stops all unwanted electrons and hard X-Rays	Cleaning sometimes required
Thin foil	Objective aperture	Remains clean; very well defined edge; available in a wide range of sizes; long life	At high voltages; electron leakage, thus loss of contrast; white spot effect at low magnification

NOTE: Thin foil apertures should be checked to ensure that they do not contain additional perforations.

3.4.3 Dismounting aperture holders

NOTE: Venting the column for aperture-holder dismounting requires Supervisor priveleges.

1. Switch the HIGH TENSION off.
2. Remove the specimen holder from the microscope.
3. Operate the aperture-displacement levers as necessary, to remove the objective and selected area apertures from the beam. Select the largest condenser aperture.

CAUTION! Steps 2 and 3 must be carried out in order to avoid possible damage to the specimen and apertures when venting the column.

4. Select the Vacuum Control Panel and press the flap-out button (arrow top right). Select the Control tab in the flap-out
5. Press Column Air
6. Wait for the column to reach atmospheric pressure.

NOTE: It is strongly advised that Nitrogen flushing be applied to the inlet on the column to prevent moisture from condensing on the inner surfaces of the microscope (see section 3.2 “Flushing with dry nitrogen”).

WARNING! Make sure that the N_2 pressure is very low (<0.1 Bar), otherwise the holder may be forced out with great speed which could be very dangerous.

7. Insert an Allen wrench (metric 2.5) into the Allen screw at the end of the holder and loosen the screw.
8. Next, completely unscrew the knob by hand at the ribbed ring at the end and carefully pull the holder straight out.

3.4.4 Remounting aperture holders

1. Replace the holder in the column (in the same lens from which it was taken) ensuring that the guide pin on the holder engages the slot in the aperture holder mechanism.
2. Screw in the knob at the end of the holder by hand until it is really tight, then tighten the Allen screw at the end of the holder.
3. Press the Column Air button in the Vacuum Control Control Panel. The column will now be pumped down again.
4. Close Nitrogen flushing (if applied).

3.4.5 Selecting the aperture sizes

Each aperture holder incorporates an aperture selector enabling a choice of (generally) four different aperture sizes.

Recommended aperture sizes are as follows:

TABLE 3-4 APERTURE SIZES

Aperture selector	Aperture sizes (µm) installed in each position on delivery			
	1	2	3	4
Condenser	200†	100	50	30
Objective TWIN	100	40	20	10
Objective S-TWIN‡	100 80	60 50	40 30	20 10
Objective U-TWIN	150	100*	60*	20*
Diffraction	800	200	40	10

*Thin foil apertures

†**Warning!**The largest condenser aperture size allowed is 200 µm. Larger apertures are not guaranteed to be X-ray safe under all operating conditions.

‡Eight-aperture blade

Special use

The Condenser aperture size should be chosen with regard to the application. In general, the larger the aperture, the greater the intensity for a given set of conditions, but the less the coherence of the illumination.

If extremely low intensities are required, together with small spot sizes (e.g. for beam sensitive specimens), a very small aperture should be used.

For X-Ray analysis, special low-background apertures are available.

Recommended condenser-aperture sizes for special purpose use are as follows:

- 100 µm - High-intensity operation with thick specimens.
- 50 µm - General observation; TEM X-Ray work; low-intensity operation.

CAUTION! Thin-foil Au apertures must never be used as condenser apertures in 200 and 300 kV machines.

The optimum **Objective aperture size** is a compromise between the small aperture required to limit spherical-aberration effects and the large aperture needed to eliminate the diffraction error (the so-called Abbe limit).

It is not always necessary or even desirable to use the optimum aperture size as the choice can also be influenced by contrast conditions. Theoretically, the larger the aperture size, the lower the contrast of the image and thus small structures are less visible. Consequently, for a specimen of low contrast, it is advisable to use a very small aperture in order to improve the contrast at a given accelerating voltage.

Recommended objective-aperture sizes for special purpose use are as follows:

- 100 µm High-resolution work.
- 40 µm Materials science.
- 20 µm Thin biological specimens.
- 20 µm Thick biological specimens and dark-field imaging.

The **Diffraction aperture** size is selected either for optimum imaging in the LM mode or for diffraction work.

Recommended aperture sizes for special purpose use are as follows:

- 800 µm - LM contrast.
- 200 µm - Selected Area Diffraction.
- 40 µm - Selected Area Diffraction.
- 10 µm - Selected Area Diffraction.

3.4.6 Removing and replacing apertures in the holders

CAUTION! The aperture holders operate in high vacuum and should therefore be handled carefully using nylon gloves. The apertures should be handled with clean, pointed tweezers.

1. Take the holder in one hand as shown in Figure 3-4.
2. Using the thumbnail, push the ring (1) against the spring to the left.
3. Raise the tip of the holder slightly and tap gently to allow the aperture to slide down the guide towards the opening (2).
4. Using tweezers in the other hand, take the apertures out through the opening (2). Take care not to damage the apertures with the tweezers.
5. Apertures are replaced by reversing the above procedure, taking care that the plain side of the aperture, in each case, is facing upwards towards the electron source and the conical side downwards (Figure 3-5).

FIGURE 3-4 CHANGING APERTURES

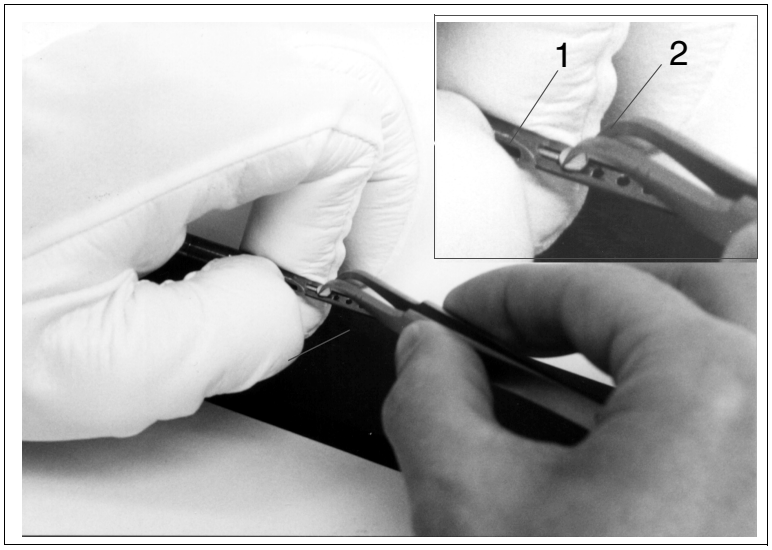
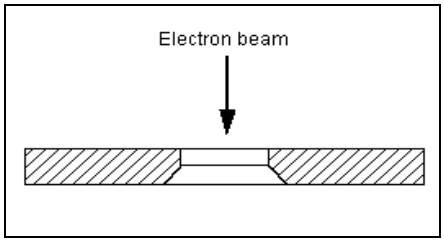


FIGURE 3-5 APERTURE CROSS-SECTION



Aperture cross-section showing profile of aperture.

3.5 Exchanging the filament (and wehnelt aperture)

The procedures that follow refer to the Universal Wehnelt assembly designed for use with either the standard tungsten filament or high-brightness filaments, e.g. pointed tungsten or LaB_6

Wehnelt aperture exchange is included in this section as a recommended procedure. Used apertures should be retained for cleaning when maintenance is carried out.

CAUTION! All parts described operate in ultra-high vacuum and should therefore be handled carefully using nylon gloves and stored in suitable containers when not in use.
When using a LaB_6 filament, special care must be taken in mounting and operating with a new filament. A running-in procedure is included in section 3.5.10 "New filament - heating-up procedure".

The Wehnelt assembly is accessed by bringing the emission chamber to atmospheric pressure and raising the gun assembly. After removing the Wehnelt assembly, it is strongly recommended that the gun is immediately placed back into the emission chamber. If the Wehnelt is kept outside the microscope for an extended period, the emission chamber can be closed and pumped down again.

3.5.1 Mechanical controls

The reference numbers associated with each control refer to annotations in Figure 3-7.

CAUTION! All mechanical controls operate smoothly and can be adjusted without particular effort. If a high mechanical resistance is encountered, do not force the control otherwise damage may result. First find the cause of the resistance and, if necessary, call your local service organisation.

3.5.1.1 Electron gun and column

(Figure 3-6)

Earthing strip

Discharges to earth any residual charge on the electron gun when the gun is withdrawn from the emission chamber. The earthing strip is located behind the cover underneath the gun-lifting lever (103). It is not visible until the gun has been withdrawn from the emission chamber.

102) Electron gun securing screws

Two red-capped screws securing the gun to the emission chamber. Before the gun can be removed they must be loosened completely. When the gun has been replaced they must be tightened completely.

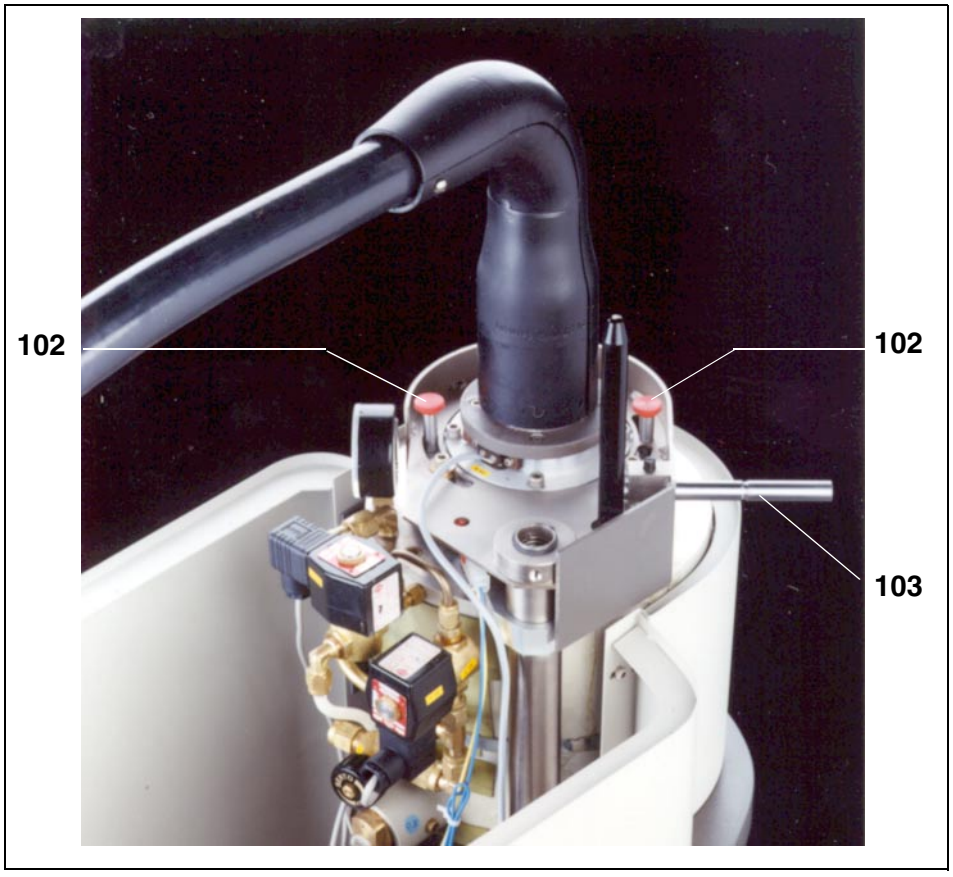
NOTE: Do not loosen the double nuts that are used for aligning the gun assembly with the column (factory alignment).

103) Gun-lifting lever

Lifts the gun assembly from the emission chamber (once air has been admitted).

Once the securing screws (102) are completely loosened and the vacuum has been broken by placing one of the gun securing screws in the gun lifting position, push the lever up as far as it will go, then turn it towards the back of the microscope and turn to the left until it locks in position. Ensure that the gun is in contact with the earthing strip. The gun assembly is lowered into the emission chamber in the reverse order.

FIGURE 3-6 ELECTRON GUN - MECHANICAL CONTROLS.



3.5.2 Removing the gun assembly from the emission chamber

(see Figure 3-7)

NOTE: Venting the gun for filament exchange requires Supervisor priveleges.

1. Move the gun lifting lever (item 103) to its operational position by pushing it from the inside to the outside, so it projects outside the body of the microscope, and can be used to lift the gun assembly.
2. Ensure that the High Tension is switched off; Check that the pressure of the nitrogen gas (if used), does not exceed 0.1 Bar.

NOTE: It is strongly advised that Nitrogen flushing be applied to the air inlet on the column to prevent moisture condensing on the inner surfaces of the microscope (see section 3.2 "Flushing with dry nitrogen").

3. Select the Vacuum Control Panel and press the flap-out button (arrow top right). Select the Control tab in the flap-out.
4. Press Gun Air

   First valve V59 will close, then valve V58 will open and the SF_6 gas in the gun isolation chamber will be pumped out by the prevacuum pump. At this moment unscrew the red-capped gun securing screws one or two turns. This is done now as the pressure from the SF_6 has been released and the pressure from flushing nitrogen gas has not had time to build up.

   When the pressure in the gun isolation chamber is low enough, valve V11 (gun air inlet valve) will open automatically.

CAUTION! Do not attempt to raise the gun assembly while the gun isolation chamber is being pumped down (valve V11 is still closed).

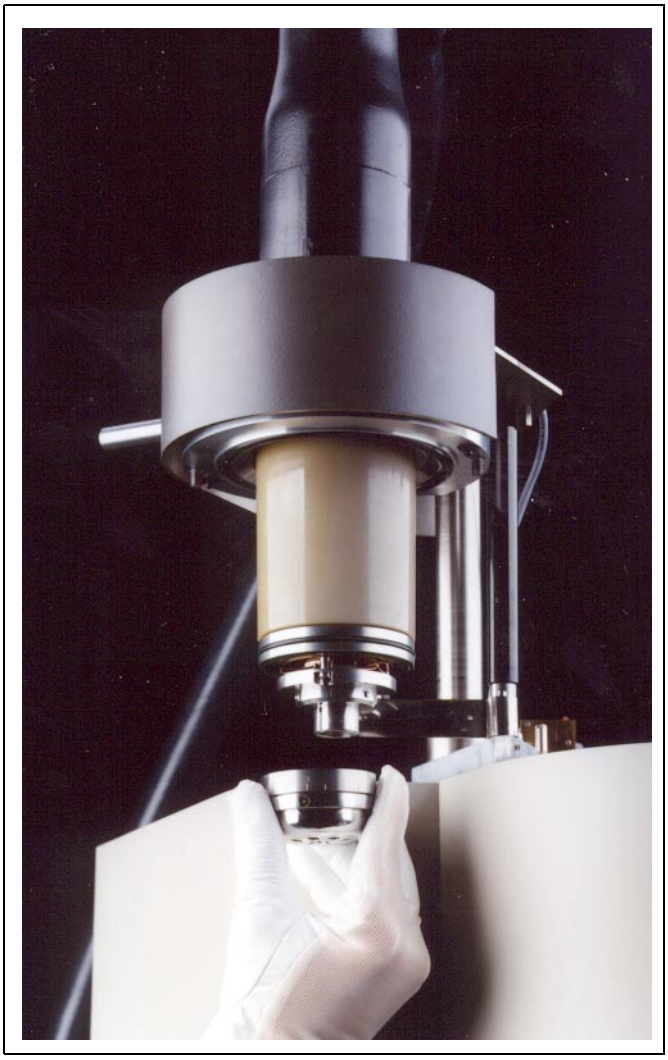
5. Completely unscrew one of the red-capped gun securing screws and insert this in the gun-lifting position (hole at 90^o to the gun securing positions, towards the front of the microscope). Screw it down until the movement begins to feel stiff, then release the other gun securing screw by one turn.

   Continue tightening the first screw and releasing the second screw until the gun assembly is released. The vacuum seal between the assembly and the microscope is now broken and the flow of nitrogen gas is audible. This procedure is needed so that the pressure difference in the gun chamber, either due to the nitrogen gas, or the vacuum left after the SF_6 is pumped away, is released in a controlled manner.
6. Raise the gun assembly using the lifting lever (item 103, Figure 3-7), shift the lever towards the rear of the microscope and then further to the left until it locks in position.

The gun is now clear of the emission chamber and the wehnelt cylinder can be dismantled or remounted as described in sections 3.5.3 “Dismounting the Wehnelt assembly” and 3.5.4 “Remounting the Wehnelt assembly”.

The gun assembly can be replaced as described in section 3.5.5 “Replacing the gun assembly in the emission chamber”.

FIGURE 3-7 DISMOUNTING AND REMOUNTING THE WEHNELT ASSEMBLY



3.5.3 Dismounting the Wehnelt

(Figure 3-7)

Grasp the Wehnelt assembly with one gloved hand and pull to detach it from the gun assembly. The Wehnelt assembly is held in place by an internally mounted spring ring. A small amount of force will be required to overcome the spring pressure.

CAUTION! The use of clean gloves for this operation is essential.

3.5.4 Remounting the Wehnelt

(Figure 3-7)

1. Replace the Wehnelt assembly on the receiver, ensuring that the pin (1) on the Wehnelt engages with one of the slots on the receiver (item 1 in Figure 3-8).

Press firmly into place. This is a reasonably tight fit and the two parts must be placed together carefully.

FIGURE 3-8 SECURING THE WEHNELT ASSEMBLY

3.5.5 Replacing the gun assembly in the emission chamber

1. Replace the gun in the emission chamber by raising the gun-lifting mechanism a few millimetres (Figure 3-7), turning it towards the front of the instrument and then to the right as far as it will go and allowing it to descend so that the insulator fits into the emission chamber. Check that the earthing strip does not obstruct its path.
2. Insert the two gun-securing screws in the gun-securing holes and screw them down, consecutively and equally, a few turns at a time, until they are hand-tight.

NOTE: Tightening the gun-securing screws must be carried out by hand. Never use any tool to exert force on the screws.

3. Press Gun Air in the Vacuum Control Panel to restart the automatic pumping sequence.
4. When the sequence is finished and the gun is under vacuum, re-tighten the gun-securing screws (by hand!).

NOTE: Conditioning is required before switching on the H.T. above 160 kV.

When the vacuum levels in the entire microscope are back to normal, check that the SF_6 pressure is higher than 5 Bar.

3.5.6 Dismounting the filament and Wehnelt aperture

(Figure 3-2)

1. Prise the Wehnelt cylinder (8) off the main body of the Wehnelt assembly (1) with the blades (17,18). The Wehnelt cylinder is held in place by a spring-clip arrangement and some force is required to overcome the spring pressure.
2. Using gloves, unscrew the filament securing ring (6) with spring washer (7) and remove them from the body of the assembly (1). The filament is now free and can also be removed.
3. Using end 15 of the key supplied, unscrew the aperture securing ring (11) from the outside of the Wehnelt cylinder (8). Remove the securing ring (11) and the Wehnelt aperture (10).
4. If it is intended to clean the components of the assembly, use the end 16 of the key to unscrew the aperture support ring (9) from the inside of the Wehnelt cylinder (8).

NOTE: Instructions for cleaning are given in section 3.3.3.1 “Cleaning the Wehnelt assembly”.

FIGURE 3-9 WEHNELT ASSEMBLY

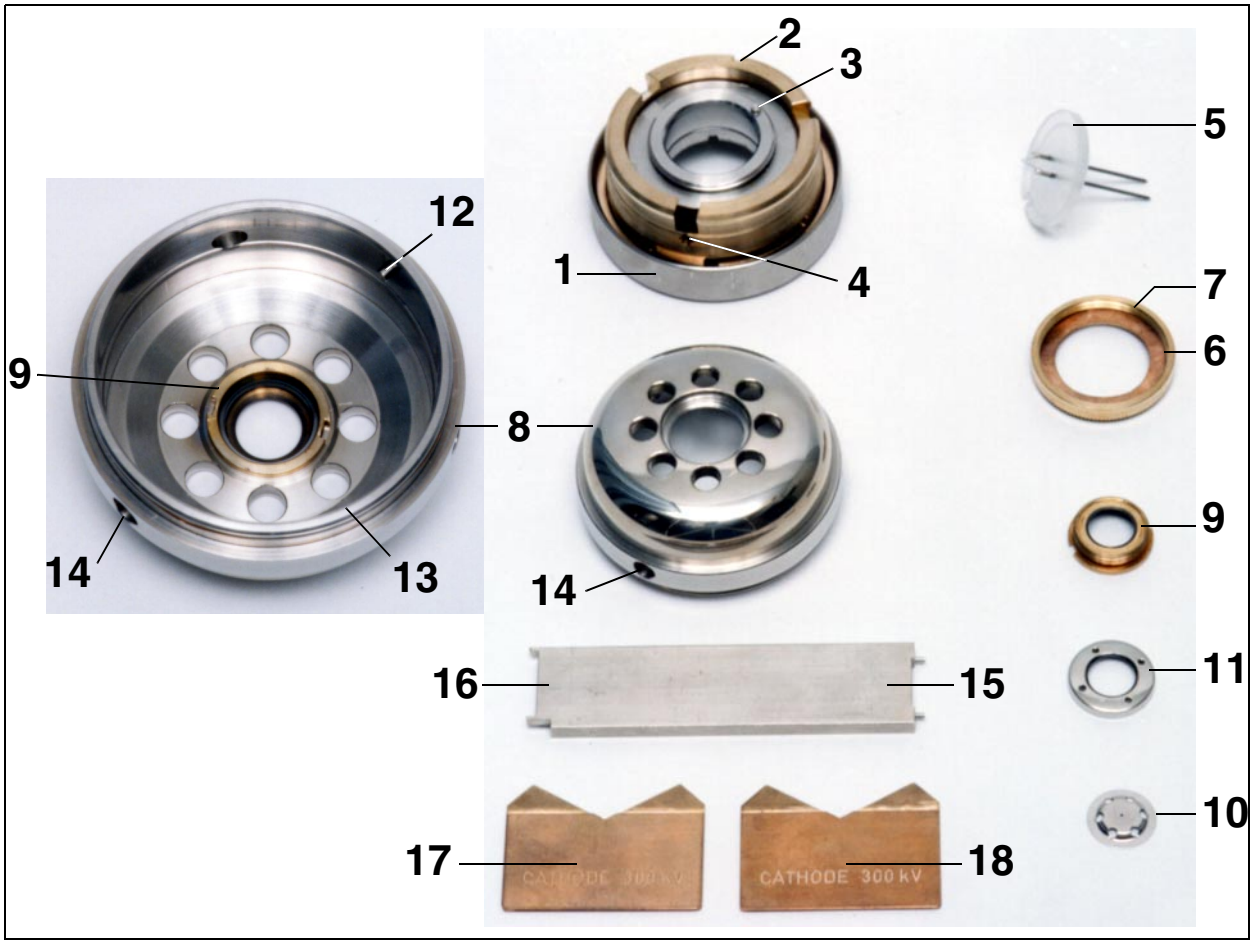
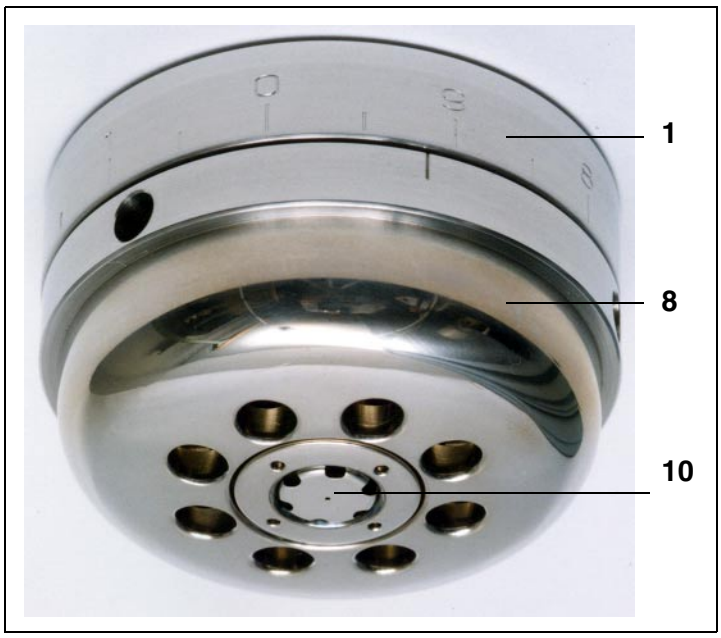


FIGURE 3-10 ADJUSTING THE FILAMENT TO WEHNELT DISTANCE



3.5.7 Remounting the filament and Wehnelt aperture

(Figure 3-9)

CAUTION! When mounting a LaB_6 filament, great care must be taken when handling the filament, particularly when adjusting the filament-to-Wehnelt distance. If the filament tip is allowed to come into contact with the Wehnelt aperture, it can very easily be damaged.

1. Locate the filament in the body of the Wehnelt assembly (1) ensuring that the slot on the filament base (5) engages with the pin (3) in the body of the assembly.
2. Screw down the filament securing ring (6) and spring (7) to secure the filament. The ring should be finger-tight.
3. If the aperture support ring (9) has been removed for cleaning, remount it on the inside of the Wehnelt cylinder (8) and screw it down tightly using the end (16) of the key supplied.
4. Fit the Wehnelt cylinder (8) without the aperture onto the body of the Wehnelt assembly with the pin (12) fitting into the groove (2).
5. Using a binocular microscope, view the filament tip in the opening of the aperture securing ring (9). Insert the three hexagonal keys provided into the three screws (4) which are reached through the holes (14) in the Wehnelt cylinder then adjust the tip to an estimated centre point.
6. Turn the Wehnelt cylinder (8) clockwise with respect to the body of the Wehnelt assembly (1) until the tip of the filament is level with the outside surface of the aperture support ring (9).
7. Place the appropriate aperture* in the recess in the aperture support ring (9) with its raised centre protruding outwards.

NOTE: The following diameters are recommended:

- **For Standard Tungsten filaments and LaB6 : 0.5 mm.**
- **For EELS work where the minimum energy spread is required: 0.3 mm.**

8. Replace the aperture securing ring (11) and screw it down using the end (15) of the key.
9. Using the binocular microscope, check that the filament tip is visible through the aperture (10). If not, remove the aperture and repeat steps 6 - 8 as necessary.

3.5.8 Setting the position of the filament to the Wehnelt cap

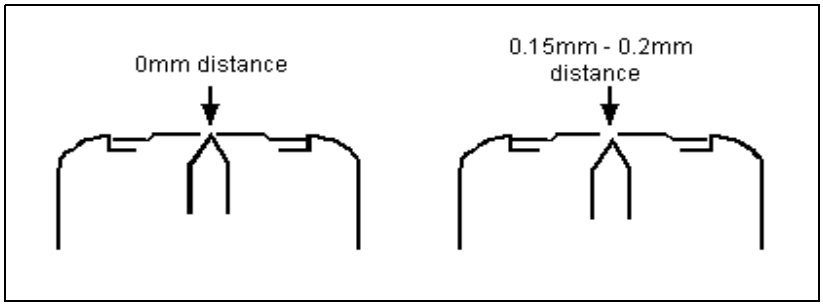
(Figure 3-9, Figure 3-10)

CAUTION! Great care should be taken with this operation as the filament tip can very easily be damaged if it is allowed to come into contact with the Wehnelt aperture

1. Using a binocular microscope, accurately centre the tip of the filament in the aperture of the Wehnelt aperture (10) by means of the three screws (4) reached through the holes (14) in the Wehnelt cylinder (8).
2. Turn the Wehnelt cylinder (8) counter-clockwise with respect to the body of the Wehnelt assembly (1) until the tip of the filament is level with the outside surface of the aperture (10) as seen through the binocular microscope. Continue observing the filament tip through the binocular microscope while turning the Wehnelt cylinder.
3. Turn the Wehnelt cylinder (8) clockwise with respect to the body of the Wehnelt assembly (1) until the tip of the filament is set to a position 0.15 to 0.2 mm* below the outside surface of the Wehnelt aperture (10).

   This can be measured on the scale on the main body of the assembly. Each large division represents 0.10 mm and each small one 0.05 mm. Turning the ring from a higher to a lower indication retracts the filament.
4. Re-centre the filament if necessary (see step 1).

FIGURE 3-11 SETTING THE POSITION OF THE FILAMENT



NOTE: A distance of this order is recommended as optimum for normal operation. Shorter distances will give greater brightness but decreased filament life and larger energy spread of the electrons in the beam. The minimum distance (which can vary from one microscope to another) is determined by the maximum allowed emission current (8 μA at emission step 1, 16 μA at 2, etc., doubling each time). If this emission current is exceeded, a safety in the high tension tank will lower the high tension (note that the LED above the high tension button will remain on). The emission will then be reduced and the safety will be released.

If the emission then again exceeds the safety value, the same sequence will follow, giving a typical up-down-up-down effect. Increase the filament-to-wehnelt distance if this occurs.

3.5.9 Conditioning the gun

After venting the gun, conditioning is necessary for attaining High Tension. Proceed as follows:

1. Select the High Tension Control Panel.
2. Press the flap-out button (arrow top-right).
3. Select H.T. setting 40 kV.
4. Wait until vacuum status is ready.
5. Switch High Tension on.
6. Increase H.T. to 160 kV using the regular steps slowly, each time waiting until the emission meter reading has settled down. If severe discharges occur (strong fluctuations of the emission-meter reading), go one step down in H.T. and proceed as listed below.
7. Press the Conditioning checkbox in the Conditioning Control Panel.
8. Select a 1 kV step size.
9. Click the ">" button to increase the High Tension. If the emission reading increases more than by about 5 scale readings, wait until it settles down. If severe instabilities occur, press the "10kV lower" button to reduce the High Tension quickly by 10kV.
10. When the High Tension has reached 220 kV, leave the microscope in this setting for about 15 minutes. The High Tension should stabilise during this period.
11. Press the Conditioning checkbox. The High Tension will go to its normal operating voltage and the microscope is ready for use.

NOTE: During conditioning the filament can not be switched on.

Leaving the microscope to attain ultra-high vacuum for an extended period is no substitute for conditioning. Not only is conditioning more efficient for obtaining ultra-high vacuum, it also prevents extreme discharges.

3.5.10 Heating-up procedure for a new filament

NOTE: Execution of the following procedure requires Supervisor privileges.

1. Select the Gun Setup Control Panel and press the flap-out button (arrow top right).
2. Select the Settings tab in the flap-out.

If a new filament has been installed:

1. Press Reset count to reset the filament-hour counter to zero.
2. If necessary, change the filament type (LaB_6 <> W).

3. Open the Column valves, if necessary.
4. Slowly turn up the filament heating.
5. Once some emission current is seen from the display, try finding the beam using the gun alignment (gun tilt). If no beam is visible initially, turn both gun tilt controls (Multifunction-X,Y) to their maximum. Keep turning the Multifunction-Y through its range, each time changing Multifunction-X a little when Multifunction-Y reaches its limit. Repeat until the beam is found.

NOTE: Never exceed filament heating step 20 if no intensity becomes visible on the screen.

NOTE:If a beam is found but the gun tilt appears to be out of range, the true gun tilt setting may lie at the other end of the range of the Multifunction knob that has reaches its limit. If this is not the case, the filament is not centered properly in the Wehnelt.

Explanation of the flow chart

Phase 1:

Heavily polluted metal parts:

Rub cleaning must be done using "CIF" or "SOFT SCRUB", a household fine abrasive.

Rinsing in water.

Wehnelt (cylinder, apertures):

Ultrasonically cleaned for 30 min. in a 6% dilution of NaOH or KOH in distilled water at 20 °C, rinsing done in water.

The KOH and NaOH dilutions are corrosive solvents, prevent skin contact. Use a fume cupboard.

Raising the water temperature increases the cleaning effect.

Phase 2:

Less polluted metal parts and parts coming from phase 1:

Ultrasonically cleaned for 5 min. in a 5% dilution of industrial soap (ph neutral) in water. Raising the temperature increases the effect.

This soap solution binds the dirt and removes it from the surface of the part.

Rinsing is done in distilled water; raising the water temperature increases the effect.

Phase 3:

O-rings, synthetic materials and assemblies (which cannot be stripped down in the field) start their cleaning sequence from this point.

Ultrasonic cleaning in Ethanol for 5 min. at 20 °C, to remove the water.

Rinsing in a new clean bath of Ethanol for 2 min.

Ethanol is highly flammable, do not use open flames, or smoke while cleaning.

Phase 4:

This phase must be skipped for ***O-rings and synthetic materials***.

Ultrasonic cleaning in Ethanol - pro analysis (99.8% pure), to be sure all water and traces left by Ethanol in phase 3 are removed.

Rinsing for 2 min. in a new bath with Ethanol p/a.

If available, an ultrasonic vapour degreaser can be used.

Phase 5:

The parts must be dried with the aid of two 150 W Infra Red lamps. To reach a temperature of 80 °C, heating by the IR lamps must be maintained for a minimum of 15 min., or, for heavy items and the emission chamber, for 2 hrs or more.

If available, a vacuum oven can be used and set to 80 °C.

When mounted into the microscope and external heating is applied, the temperature must kept under 100 °C, otherwise damage to encapsulated items will occur.

O-rings must not become warmer than 70 °C and can best be dried in air.

Parts should only be handled after they have cooled down below 50 °C.

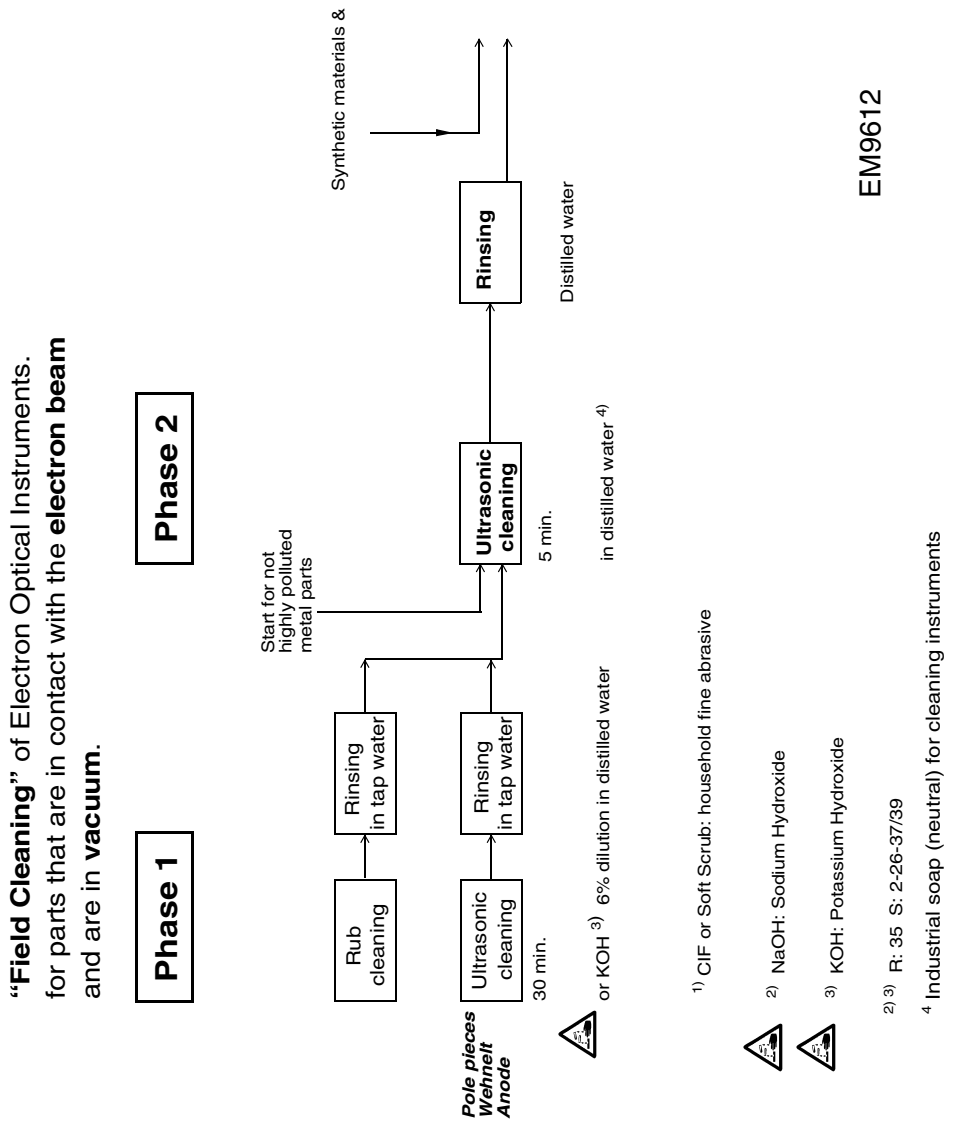
Spray the part with a gas jet duster, to remove any dust particles or fibres before inspection.

After inspection under a light microscope, mount the parts directly into the Electron Microscope and spray with the gas jet duster again before closing the microscope.

If parts are not mounted but stored, pack them in aluminium foil.

Field Cleaning Flow Chart(part 1)

"Field Cleaning" of Electron Optical Instruments.
for parts that are in contact with the **electron beam** and are in **vacuum**.



Field Cleaning Flow Chart(part 2)