

STEREOSCAN 200

SCANNING ELECTRON MICROSCOPE

OPERATING INSTRUCTIONS

TL 2025-OM

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Applicable to Instruments  
Serial No 1001 onwards

Cambridge Instruments Ltd  
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AMENDMENT RECORD CERTIFICATE

This certifies that the following Amendments have been incorporated into this Manual.

Amendment Number	Brief Details of Amendment	Date	Incorp. By.
AM1	Incorporated before Issue.	4.1.84.	C.F.
AM2	Split Screen Page 32 Insert New Para.at 2.5.6. 9.7.84.		"
AM3			
AM4			
AM5			
AM6			
AM7			
AM8			
AM9			
AM10			
AM11			
AM12			
AM13			
AM14			
AM15			
AM16			
AM17			
AM18			
AM19			
AM20			

This publication summarises the recommended procedures for operating the described instrument. It supplements, but does not replace, the contents of an approved operator's training course. New users are strongly advised to participate in such a course before operating their instruments. Details of courses and enrolment procedures are available from accredited Cambridge distributors and service organisations and from Cambridge Instruments Ltd, Sales Department, Rustat Road, Cambridge, CB1 3QH, England.

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## WARNINGS RE USE OF S200

### 1. EHT VOLTAGES IN THIS INSTRUMENT ARE LETHAL

Interlocking safety circuits are incorporated both to safeguard the equipment during certain operating conditions, and to safeguard personnel during maintenance procedures on, for instance, the electron optical column. In addition WARNING notices are affixed at danger points. Any attempts to override these safety circuits will involve risk of contact with voltages of up to 30 KV.

Do not remove the cover from any electronic unit to gain access while the equipment is operating.

### 2. LINE VOLTAGE

Line voltage is present at various places in the instrument when connected to the electrical supply, even with the instrument power switch in the OFF position.

**ALWAYS ISOLATE THE INSTRUMENT FROM THE POWER BEFORE ATTEMPTING ANY MAINTENANCE WORK.**

### 3. X-RADIATION

The instrument is designed to ensure that X-radiation is less than that permitted under any international or federal legislation. To achieve this, appropriate materials are used throughout, and radiation shields are fitted.

Customers are advised to check X-radiation levels when installing their own experimental equipment, or when fitting accessories not supplied by Cambridge Instruments Ltd.

### 4. TRANSIT CLAMPS

To avoid damage to the equipment during transit, the gun, specimen stage, suspension system and the turbomolecular pump are fitted with transit clamps which must be removed during the installation procedure (see Chapter 6).

### 5. ROTARY PUMP EXHAUST

The discharge from the rotary pump will contain a small quantity of oil mist and this can contaminate the environment and create a health hazard when used in enclosed surroundings. To avoid this risk an exhaust system should be provided, as described in the Installation Recommendations.

### 6. SOLVENTS

Careless use of solvents (Arklone, Propanol, Methanol etc) can constitute a health hazard. Use minimal quantities and take all possible precautions to avoid spillage, skin and eye contact, and vapour inhalation. Before using any solvent be familiar with the published safety procedures (normally provided by the solvent manufacturers) and comply with them.

## 7. CORROSION

Although all components used in the S200 are protected from corrosion either by plating, painting or other anti-corrosion treatment, Cambridge Instruments Ltd accept no responsibility for corrosion caused by storing the S200 in adverse atmospheric conditions.

## 8. FANS

The instrument is totally air cooled and the input to, and the outlets from, the cooling fans should not be blocked or impaired. The removal of any of the instruments covers will upset the air flow and the instrument should not be run with any covers removed. Keep the fingers well away from the fans at all times.

## 9. WARN ALL USERS

It should be a matter of routine to bring these warnings to the notice of **ALL** and **EVERY** user. A user who initially comes for only five minutes may, and often does, find himself using the microscope unsupervised, even though this was definitely never intended to happen.

**IF IN DOUBT CONSULT AN AUTHORISED SAFETY OFFICER**

**S200 OPERATORS MANUAL****Contents**

Chapter 1	Operating routines
Chapter 2	The Control Console - What the Controls Do
Chapter 3	Maintenance
Chapter 4	Options
Chapter 5	Advanced Operating
Chapter 6	Installation
Chapter 7	The Scanning Electron Microscope

## CHAPTER 1 OPERATING ROUTINES

These instructions provide a simple guide to some frequently used operating routines. Before using these routines all operators should be familiar with chapter 2 of this manual, using this section only as a reminder of the sequence of operations.

When following these routines there may be a choice of paths. The operator must consider the relevant facts and choose the right path.

**Note A** Follow the routine carefully, one step at a time.

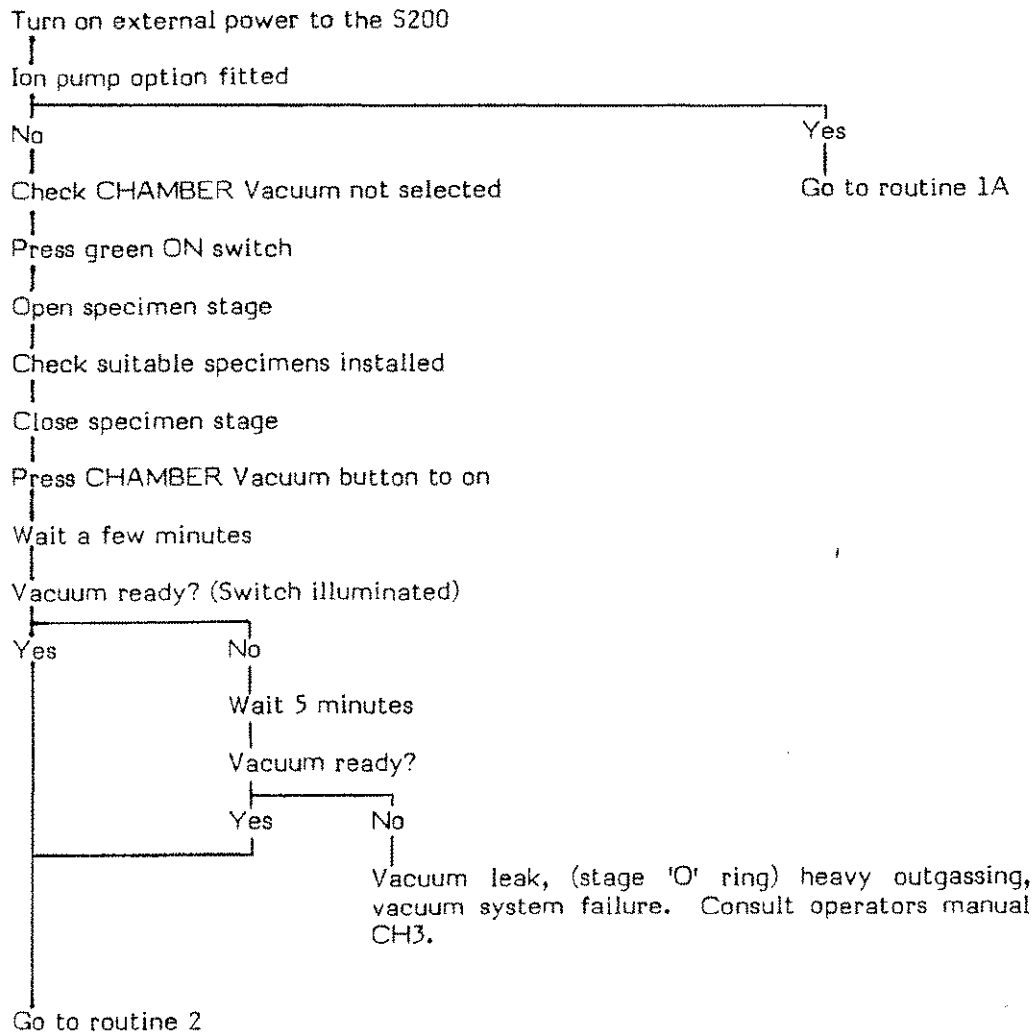
**Note B** The S200 should always be left in an evacuated state when not in use.

The routines covered by this chapter are as follows:-

Routine 1	Switching on and getting a working vacuum
Routine 1A	Switching on and getting a working vacuum (ion pump)
Routine 2	Obtaining a picture
Routine 3	Taking a Micrograph
Routine 4	Changing the Specimen
Routine 5	Switching off
Routine 6	Filament Change
Routine 7	Changing the Acceleration Voltage Range

## ROUTINE 1

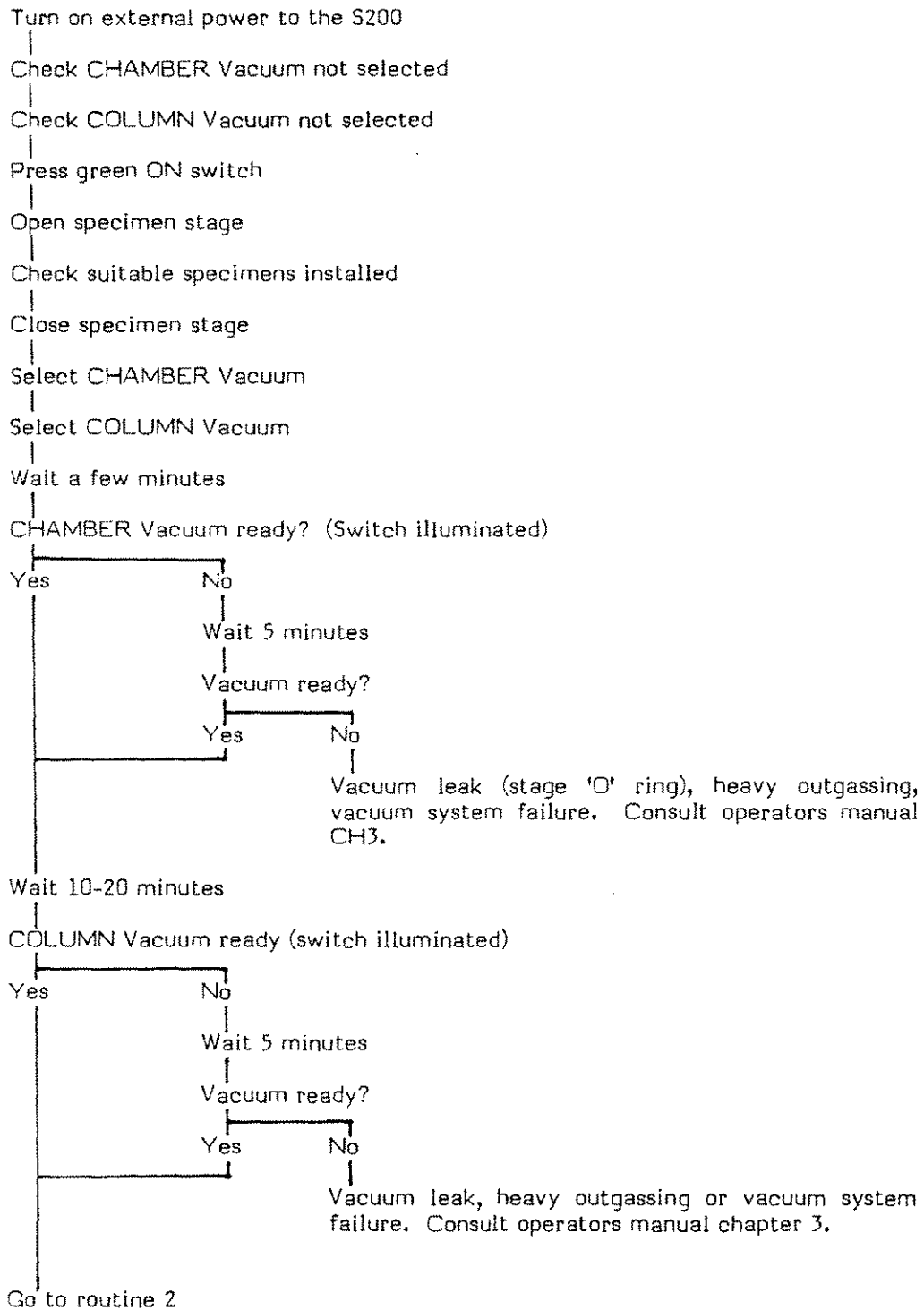
## SWITCHING ON AND GETTING A WORKING VACUUM





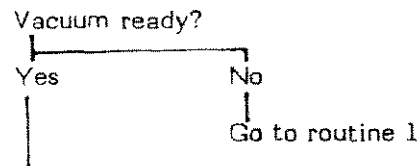
ROUTINE 1A

SWITCHING ON AND GETTING A WORKING VACUUM WITH ION PUMP



## ROUTINE 2

## OBTAINING A PICTURE

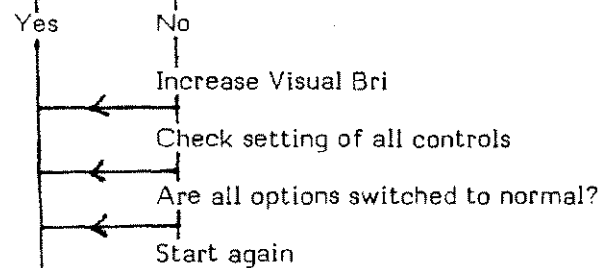


Select the following:- MAGNIFICATION Coarse, Visual Bri centred, SIGNAL LEVEL Auto, Auto Level centred, Contrast between 1st and 2nd mark, SCANNING MODE TV, STIGMATOR X and Y centred, FOCUS Coarse 10mm, EMITTER not LaB6, OPTIBEAM Normal, RESOLUTION Coarse 6, RESOLUTION Fine clockwise, ACCELERATION VOLTAGE 25, High, Fine clockwise, Filament minimum BEAM off, INPUT SELECT S.E., SIGNAL not INVERTed, SPLIT SCREEN off, GAMMA anticlockwise, DIFF anticlockwise, SCAN Normal, FOCUS MOD Off, EMISSION IMAGE off, all four GUN ALIGNMENT controls centred. Check that all options are selected OFF or NORMAL. The setting of any other controls is irrelevant.

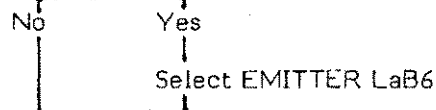
Turn OPERATE on

Wait 30 seconds

Raster visible?



LaB6 option fitted



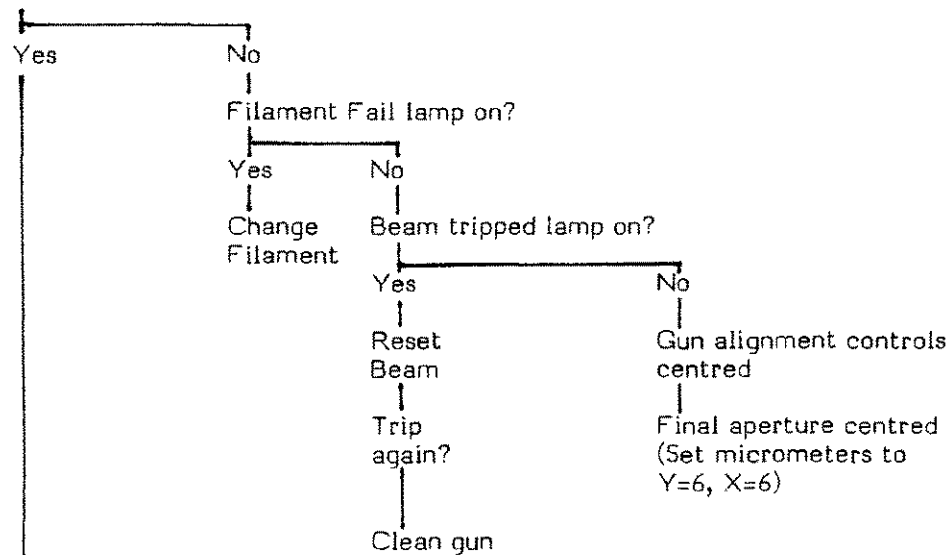
Switch BEAM on

Lab6 option fitted



Spin MAGNIFICATION Change Digiknob anticlockwise

Image of specimen visible?



Switch EMISSION IMAGE on

Set RESOLUTION to 2 or 3

Centre bright source on CRT using X and Y Shift

Reduce Auto Level so detail is visible in emission image

Adjust FILAMENT current to 1st or 2nd peak as required (section 2.6.4)

Set RESOLUTION to 6 or 7

Centre brightest part of source in the emission image using X and Y Tilt

Switch EMISSION IMAGE off

Using FOCUS COARSE obtain a sharp image

Using specimen stage controls select a suitable specimen area

Increase MAGNIFICATION

Adjust as necessary to obtain the required image:

FOCUS (Coarse at very low mag, Medium and Fine at higher mags)

MAGNIFICATION

RESOLUTION (turn clockwise to increase resolution at the expense of image noise)

SCANNING MODE (slower scan speeds reduce image noise)

Stage movements

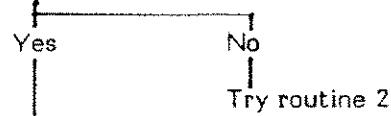
STIGMATORS

IMAGE SHIFT to give fine image movement at high magnification

## ROUTINE 3

## TAKING A MICROGRAPH

Suitable image on display?

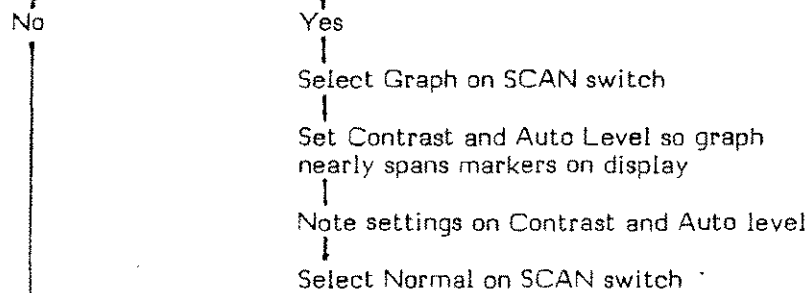


Check correct camera and film fitted

Select photo scan time (FAST is 50 seconds, SLOW is 200 seconds)

Check Photo number and Specimen number in data zone are correctly set

First photo of session

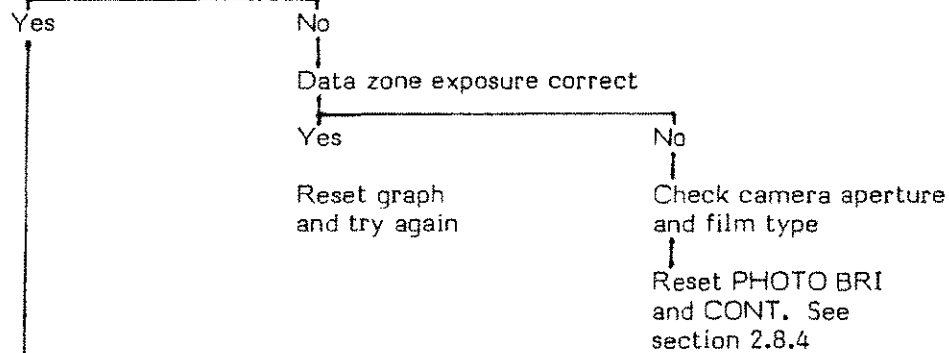


Open camera shutter (if it has one)

Press PHOTO START

When photo scan stops (scanning returns to visual mode) develop picture (Polaroid) or wind on film (roll film cameras)

Photo exposure correct

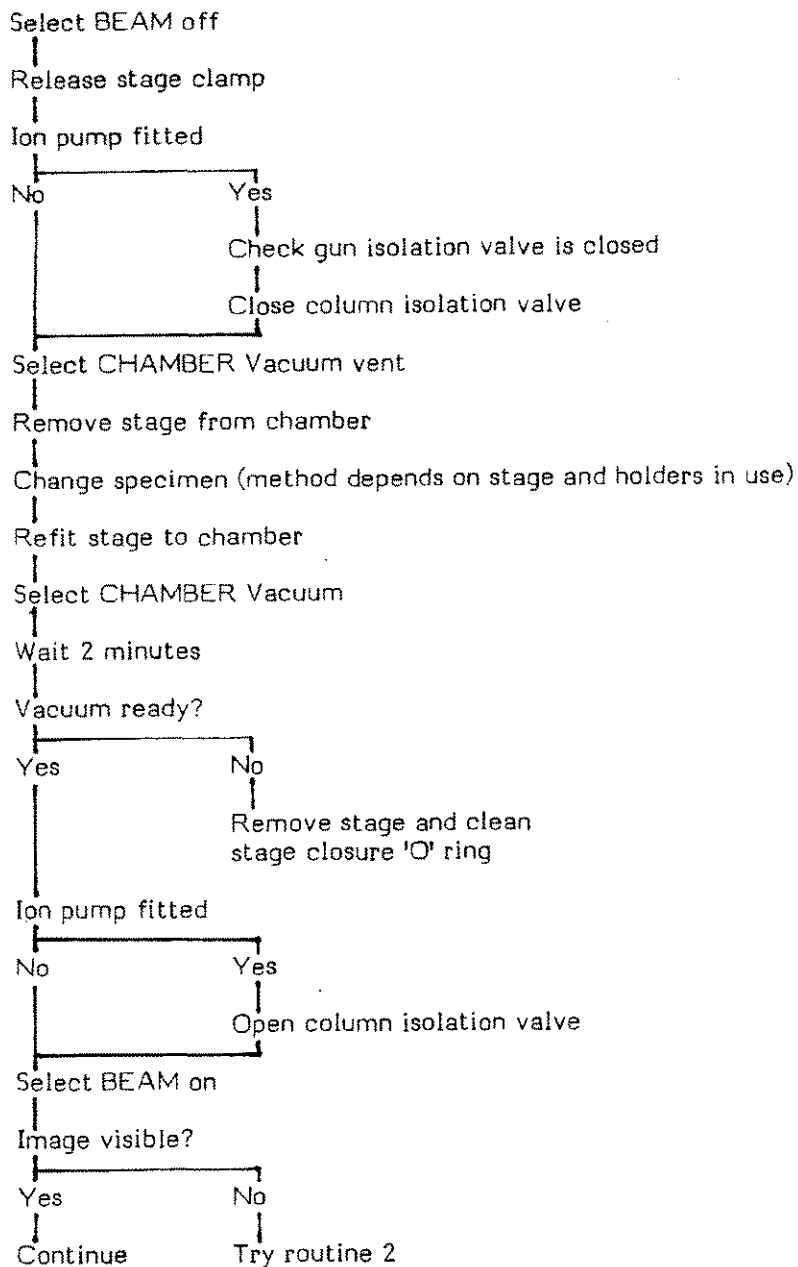


Now look for another suitable area of specimen

## ROUTINE 4

## CHANGING THE SPECIMEN

This routine can be used at any time provided power is switched ON



## ROUTINE 5

## SWITCHING OFF

Select BEAM off

The S200 may now safely be left for periods of several hours. For an overnight closedown:

Switch OPERATE off

The S200 may now safely be left for periods of several days. It is strongly recommended that the S200 is left under vacuum whenever possible. If it is absolutely necessary to switch the S200 off completely:

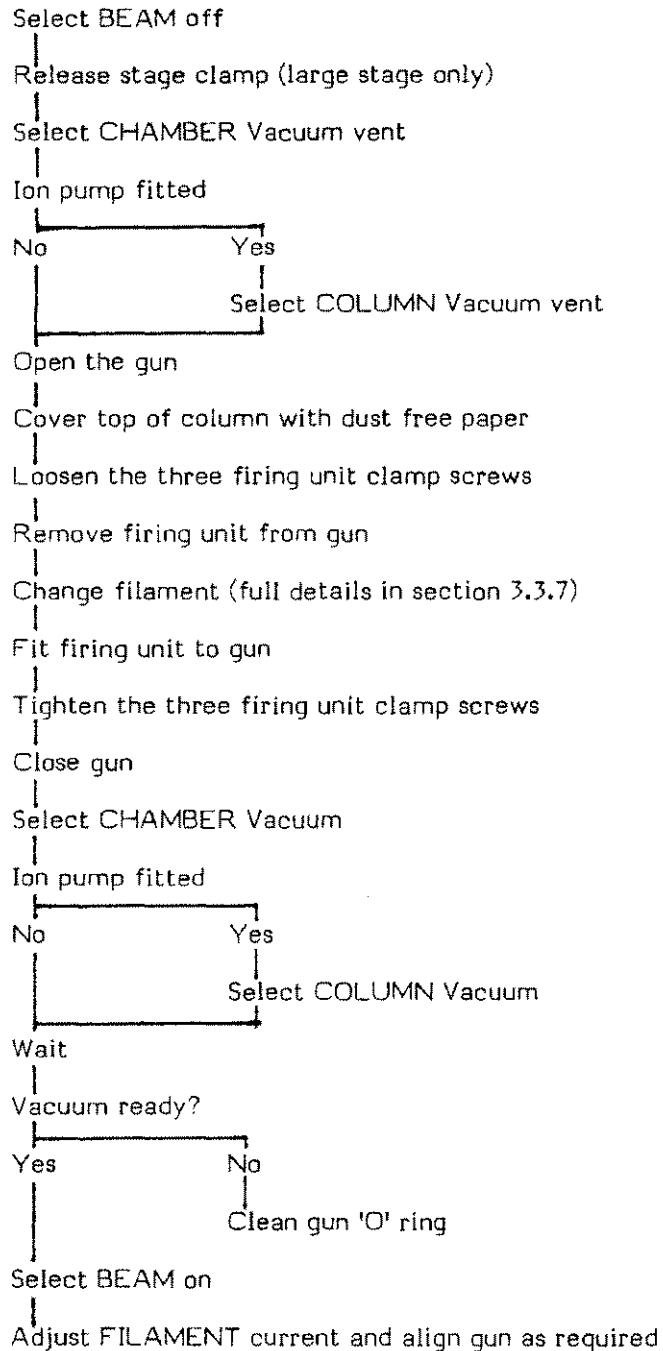
Switch power OFF

WITH POWER OFF THE COLUMN IS VENTED (if the optional baffle valve is not fitted). IF LEFT IN THIS STATE FOR LONG PERIODS IN AN ADVERSE ATMOSPHERE THE COLUMN MAY CORRODE. IF IT IS NECESSARY TO LEAVE THE S200 IN THIS STATE FOR LONG PERIODS IT SHOULD BE FILLED WITH A DRY INERT GAS e.g. DRY NITROGEN.

## ROUTINE 6

## CHANGING THE FILAMENT

This routine can be used at any time provided that power is ON



## ROUTINE 7

## CHANGING THE ACCELERATION VOLTAGE RANGE

THE S200 SHOULD NOT BE USED ON THE HIGH KV RANGE WITH THE LOW KV ANODE FITTED

Switch BEAM off

↓  
Select the desired KV range on the ACCELERATION VOLTAGE High control

↓  
Admit air to the column (Routine 6)

↓  
Open the gun

↓  
Lift out the anode

↓  
Fit other anode OR fit or remove anode spacer (the low KV anode is taller than the high KV one)

↓  
Close gun

↓  
Pump column (Routine 6)

↓  
Obtain an image (Routine 2)



## CHAPTER 2

### THE CONTROL CONSOLE - WHAT THE CONTROLS DO

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## 2. INTRODUCTION

This section of the manual is written as a "hands on" instruction. The person reading it is encouraged to sit in front of the S200 and operate it step by step. All control functions of the S200 are fully protected against operator error and it is not possible to damage the instrument by incorrect operation.

Almost all the controls of the S200 are situated on the console front panel. The frequently used controls are on the lower part of the panel, the less frequently used controls being grouped on the upper section.

There are three small groups of controls not mounted on the console front panel. These are:

- the vacuum controls, mounted on the front of the plinth

- the record unit calibration controls, mounted on the desk top to the right of the main control panel, behind the camera mounting

- the text keyboard. This is free standing and may be positioned anywhere on the S200 desktop.

There is some space left on the upper level of the control panel for option controls.

Some of the controls mounted on the vacuum control panel will only be used when the ion pump option is fitted (see chapter 4).

The standard front panel controls will be considered in four groupings. These are:

1. Power Switching
2. Electron Optics Control
3. Scanning Controls
4. Signal Level Controls

The less frequently used controls on the upper panel are:

5. Image Processing Controls (the left hand group)
6. Beam Processing Controls (the right hand group)

Controls not situated on the front panel are:

7. Vacuum Controls (on the front of the plinth)
8. Record Unit Controls (to the right of the main control panel)
9. Final Aperture Centring (mounted on the front of the column)
10. The Text Keyboard

Not really a control, but how you know what the controls are doing, is:

11. The Data Zone

### 2.1 Power Switching (figure 2.1)

Consists of two controls grouped at the left hand side of the control panel. These controls are:

### 2.1.1 Power (on the upper left control panel)

Consists of a green ON button and a red OFF button. When the S200 is connected to a source of mains power the red OFF switch is illuminated to warn the operator that some parts of the S200 may be live. (The only parts of the instrument which are powered in this condition are some parts of the main switching unit which are not accessible to the operator.) When the ON button is pressed, it will illuminate and the main supply to the instrument is switched on.

When the OFF Button is pressed the system will switch off.

Both switches are momentary acting non-latching types, so they give no positional indication of their status. If both switches are pressed simultaneously, the machine will switch off.

Power will normally be left on permanently and these controls will only be operated when the S200 is to be switched off for long periods. The S200 power system is designed for continuous running.

### 2.1.2 OPERATE (on the lower left of the front panel)

A push button controlling the power to the S200 electronics. Press the button once and the S200 electronics is ready to use (assuming you have power on and VACUUM ready, indicated by the column and chamber vacuum push buttons being illuminated). Press the control a second time and the electronics is turned off, the instrument is safe to leave for long periods, e.g. overnight or for weekends or longer periods.

The OPERATE switch will not function unless you have power switched on, but can be used with vacuum either pumped or vented. If vacuum ready has not been achieved then certain operate functions will be inhibited. These include supplies to the electron gun, collector system and displays. Thus, with operate on and vacuum not ready, the display tube will be blank and it is safe to change the specimen or the filament. It is normal practice to change specimens with the S200 in the operate mode.

When switching to OPERATE, or achieving vacuum ready, there will be a few seconds delay before a raster is seen on the visual display. This delay is to allow the electronics to "warm up".

## 2.2 Electron Optics Control (figure 2.2)

Consists of a group of fourteen controls and three indicators situated to the right of centre of the control panel. This seems a large number of controls (but in normal use) only a few of them are frequently used e.g. FOCUS, RESOLUTION and STIGMATOR.

### 2.2.1 BEAM.

A push button switch and two LED indicators. Press the button and the electron beam in the column is turned on. When the BEAM push button is depressed the acceleration voltage and filament current, at values selected by their respective controls, are applied to the electron gun.

Two "fault" indicators are positioned above the beam switch. These are:

- a. Filament Fail; the left hand one of the two indicators. If BEAM is selected on and no filament current is flowing, this LED will illuminate. This means that either the filament current control is turned to too low a value (set it to the centre graduation) or that the filament requires changing. (See Routine 6 in chapter 1 or 3.3.7 for details.)
- b. Trip; the right hand indicator. If BEAM on is selected and, for any reason, the current drawn from the eht supply exceeds a preset "safe" level, the eht will "trip" and the LED will illuminate. The trip is normally caused by a temporary current surge, or "flash over", in which case the beam can be reset to on by pressing the BEAM switch twice. If the trip persists it indicates that either the low KV anode is fitted with high KV selected, the gun components (grid and anode) need cleaning, the filament has moved off centre of the grid or, very rarely, that the eht set has malfunctioned. For details of gun component cleaning and performing a filament change, refer to chapter 3 of this manual.

### 2.2.2 ACCELERATION VOLTAGE.

A group of three controls labelled

- ACCELERATION VOLTAGE
- High
- Fine

#### ACCELERATION VOLTAGE

A set of six push buttons which selects the acceleration voltage applied to the gun. Press any button to select the acceleration voltage which is printed above the button (if the High button is depressed) or below the button if High is not selected.

High.

A single push button switch which selects the range of acceleration voltage available. Press the button (so that it remains in) and the range of acceleration voltage is from 4kv to 30kv. Press the button again (so that it remains out) and the range of acceleration voltage is from 0.3kv to 3.0kv.

### Fine.

The actual acceleration voltage applied to the gun also depends on the setting of the five position rotary switch labelled Fine. When this switch is fully clockwise the acceleration voltage will be the value selected on the acceleration voltage buttons. Turning this switch anticlockwise reduces the acceleration voltage in small steps. On the high ranges of acceleration voltage the switch changes the acceleration voltage in steps of 1kv. On the low range each step is 0.1kv.

By using these three controls the acceleration voltage can be changed in increments of 0.1kv from 300 volts to 3.0kv and in increments of 1kv from 4kv to 30kv. When an acceleration voltage of either 5kv or 0.5kv is selected some of the positions on the fine switch will have no effect.

The actual acceleration voltage selected is always displayed in the Data Zone at the top of the visual display and the micrograph.

The acceleration voltage used in any particular situation will depend upon a number of factors determined by the nature of the specimen and the type of information you are trying to get from it. While you are learning to use the S200 it is easier to use a metallic sample and a high acceleration voltage. Try 25kv.

When using acceleration voltages of 3kv or less the low kv anode should be fitted to the gun. For details on changing the anode see Routine 7 in chapter 1 or section 3.7.

A few general rules are that a high acceleration voltage gives better resolution on robust, conductive specimens. A low acceleration voltage causes less charging and specimen damage on delicate, non-conductive samples (e.g. biological material). A high voltage is needed for the efficient generation of X-rays for specimen analysis, a low one for looking at the surface of semiconductor samples with minimum beam penetration. The selection of the correct voltage for different types of sample will be discussed more fully in chapter 5 of this manual.

All the S200 electron optics and scanning circuits are compensated for changes in acceleration voltage. When you select a different voltage the control circuits also change the effect of the RESOLUTION control (via the OPTIBEAM circuits), the MAGNIFICATION control and the FOCUS controls. The net result of this on the visual image is that nothing appears to change, the magnification, focus and picture brightness stay constant. Only the effect of beam energy on the sample changes.

### 2.2.3 Filament.

Controls the amount of current flowing through the filament, and so its electron emission characteristics. The centre calibration mark round the control corresponds to a filament current of 2.75 amps, each small division represents a change of 0.05 amps.

The correct method of setting the filament current requires the use of the EMISSION IMAGE and GUN ALIGNMENT controls and will be fully described in section 2.6.4 of this manual.

### 2.2.4 RESOLUTION Coarse and Fine.

A twelve position rotary switch labelled Coarse and a three turn potentiometer labelled Fine which controls the size and intensity of the electron beam reaching the sample. In normal operation the Fine control is kept in the "calibrated" position (fully clockwise) and Coarse only is used to control the beam intensity. To see the effect of the RESOLUTION control select a "low" resolution, about Coarse position 3 with the Fine fully clockwise, obtain a TV picture at low magnification and focus it. Increase the magnification, focusing as you go. Notice the clear, noise free image. Soon you will reach a magnification when the image will not come sharp in spite of your efforts to focus it. This will happen at about 5,000 times. Check that you have signal level auto selected. Turn the RESOLUTION up to about position 8 and refocus. The image will now be sharper and you can go to higher magnifications. At magnifications of 50,000 and above you will need a RESOLUTION setting of 11 or 12 to achieve the ultimate resolution performance.

You will notice that as RESOLUTION is increased the image will become more noisy, like looking through a snow storm. This is the unfortunate and unavoidable result of increasing the resolution. The operating technique to use is to start at low resolution at low magnifications and increase the resolution only as far as you must to achieve the picture quality you need. (The noise on the picture can be reduced by using slower scan speeds, Vis 2 or Vis 3. Try it. The noise reduction will be even greater on the micrograph when you take one.)

### 2.2.5 OPTIBEAM.

A control labelled Normal and an indicator labelled Change Aperture.

The OPTIBEAM system computes the best lens excitations for use under any operating conditions and sets up the lenses to give optimum performance. To achieve this OPTIBEAM must know many things about the operating conditions that you have set. Most of this information is fed in automatically, but there is one important parameter that OPTIBEAM does not know. This is, the size of the final aperture. The normal final aperture size for OPTIBEAM is 20 micron and if the NORMAL switch is depressed then OPTIBEAM assumes that this is the size in use. (You must

select the final aperture size using the final aperture selection controls on the column, see section 2.9. It is recommended that a 20 micron aperture is fitted in aperture position 2, with the aperture centring controls being set at about X=6mm, Y=6mm).

Under certain operating conditions, particularly at long working distances and low acceleration voltage OPTIBEAM may need to use a different size final aperture to achieve optimum performance. It tells you this fact by illuminating the Change Aperture indicator. To maintain optimum performance you must select the 50 micron aperture using the aperture changer controls. This is normally in aperture position 4 with the aperture changer micrometers set at about X=6mm, Y=18mm. (These settings may vary slightly between machines.) When you have selected the alternative aperture, tell OPTIBEAM by releasing the Normal switch.

For normal operating just check that OPTIBEAM is switched to Normal, the EMITTER switch is out, ie not LaB6. Ignore the Change Aperture LED, set the Fine control fully clockwise and just use the RESOLUTION control as described above.

#### 2.2.6 EMITTER LaB6

This switch has three functions, all of which are needed when the optional LaB6 emitter assembly is used. These functions are

- a. Change the level at which vacuum ready is indicated
- b. Change the emission current from the EHT set to the level required by the LaB6 emitter, and
- c. Correct the OPTIBEAM circuits for the different performance characteristics of the LaB6 emitter.

If the normal tungsten filament is in use set this control to the non LaB6 position (switch out). If you have the optional LaB6 filament fitted, switch to LaB6 and read section 4.2 of this manual.

**Note:** If a conjugate beam blanking option is fitted the use of the Normal and Change Aperture controls will be slightly different, see section 4.5.

#### 2.2.7 FOCUS - Coarse - Medium - Fine

A set of controls which allow the user to adjust the current in the objective lens, thereby adjusting the position in the chamber at which the electron beam is focused.

Select a very low magnification (switch the MAGNIFICATION to Coarse and spin the Change control several turns anticlockwise). Turn the FOCUS Coarse control. The image goes in and out of focus. Focus as well



as possible, increase the magnification and focus again. Repeat this process. At magnifications of 100 times and above it will not be possible to obtain an accurate focus using the Coarse control. When this point is reached, start using the Medium focus control. When the magnification reaches in the region of 10KX (as indicated on the data display on the top of the visual display) it will again be difficult to accurately focus. Start using the Fine control. Take care not to adjust Coarse or Medium focus when working at high magnifications or focus may be completely lost, requiring the operator to return to low magnifications and use the Medium focus again. (It will be necessary to increase the resolution as the magnification is increased.)

An image which is in focus at high magnifications will be in even better focus at lower ones. So, for the best pictures, turn the magnifications up, focus and then return to the required magnification.

#### 2.2.8 STIGMATOR

Two single turn controls affecting the amount of astigmatism correction applied to the electron beam. Astigmatism is when the focus of the image is good in one direction, but it looks smeared in a direction perpendicular to that. In a perfect electron microscope stigmators should not be needed, but in a "real" microscope small imperfections in the lens and contamination in the column means that stigmators are necessary. (Astigmatism can also be caused by many other varied effects, including magnetic samples).

A simple method of using of the stigmators will now be described.

Set both stigmator controls to their central positions with their knob pointers vertical. Obtain a well focused image at a magnification of 10,000 times. Now adjust one stigmator control, going first one side of centre and then the other. The image will go in and out of focus in one axis. Select the setting of the stigmator control which gives the best image. Now repeat this exercise with the other stigmator, selecting the setting which gives the best image. The image resolution may now be further improved by repeated small adjustments of focus and stigmators. For further details see chapter 5 of this manual.

### 2.3 SCANNING controls. (fig 2.3)

In the following section all scan times and line densities are approximate and are quoted for an S200 set for 50hz power. The numbers in brackets are for a 60hz machine.

The scanning controls consist of six controls grouped at the left hand end of the control panel. (This group is split in two by the Signal Level controls.) As each control is described, try it and see what it does. The controls are:

### 2.3.1 MAGNIFICATION.

Two controls, a push button switch labelled Coarse and a digiknob labelled Change. Turn the Change digiknob clockwise and the magnification increases, you see smaller and smaller parts of the sample in greater detail. Turn the Change digiknob anticlockwise and the magnification decreases.

Press the Coarse switch so that it stays depressed. The Change control now covers the full range of magnifications (from 30X to 300,000X at 30kv and 15mm working distance) in 15 coarse steps. Now go to low magnification and press the coarse switch and try the change control again. The magnification will now increase in much smaller steps, taking 211 steps to cover the full range. The interchange between coarse and fine can be made at any time.

The magnification selected is shown in the data zone at the top of the display. The magnification range available depends on the working distance and the acceleration voltage used. At long working distances it will cover a lower range e.g. from 7X to 55KX at 100mm working distance and 30kv. At low acceleration voltages the range covered will also be lower e.g. from 10X to 90KX at 3kv and 15mm working distance. Other typical values are

KV	Working Distance	Min Mag	Max Mag
30	15mm	30X	280KX
3	30mm	6X	50KX
300V	100mm	0.6X	5KX

If the lowest magnification is selected and then the acceleration voltage is increased the digiknob Change control will have to be turned some way before the magnification actually changes.

#### IMAGE SHIFT $\leftrightarrow$ and $\updownarrow$

Two controls which move the scanned raster across the specimen in the X and Y axes a distance of 40 microns (at 15mm working distance). These controls can be used to give apparent fine specimen movements at high magnifications when the stage micrometers have become too coarse. They are most useful at magnifications in excess of 10,000 times.

Turn the  $\leftrightarrow$  control clockwise and the image will move right across the display. Turn it anticlockwise and the image will move left. Now try the effect of the  $\updownarrow$  control.

### 2.3.2 SCANNING MODE

A bank of six push buttons labelled SCANNING MODE and a single push button labelled PHOTO Start/Reset.

The SCANNING MODE switch incorporates the following functions:-

#### TV.

When selected gives a standard television scan for flicker free viewing. A CCTV output is available in the S200 to allow images to be displayed on a remote TV monitor or recorded on a video tape recorder. The line density on TV is 625 lines on 50hz power, 525 on 60hz power.

(The CCTV output socket is mounted on the rear of the small trim panel to the left of the visual display.)

#### SMALL.

A 12 (15) frames per second 625 (525) line scan covering one ninth of the display area, in the centre of the display. This small noise free raster is most useful for critical focusing and astigmatism correction at high magnifications.

#### Vis 1.

A 12 (15) frame per second 625 (525) line full frame scan for less noisy flicker free viewing.

#### Vis 2.

A 3 (3.75) frames per second 625 (525) line raster for even better noise reduction on the image.

#### Vis 3.

A 5 (4) second per frame 625 (525) line raster for the ultimate in noise free visual observations of images.

#### Fast.

When depressed it selects the faster of the two record speeds. Fast is used for the majority of micrographs, when the ultimate in noise reduction is not needed. Slow is used for very high resolution work. It is also useful for signals with a poor frequency response e.g. cathodoluminescence, specimen current and x-ray analysis. It has no effect on any visual scans.

The difference in noise between the two scan speeds can be seen on graph. Select Graph on the SCAN switch on the Image processor on the left of the upper control panel. Obtain a noisy image (RESOLUTION setting 9 or above) and select Fast. Look at the noise on the signal. (Noise is the fine "grass" on the graph). Select Slow and see the noise on the signal decrease.

## PHOTO.

A single push button below the SCANNING MODE switch. When you have a good enough image on the visual display and you want a photograph of it, just press the PHOTO button. (Assuming that the record display is correctly calibrated, see section 2.8.4 and the camera loaded with a suitable film.) When you press the PHOTO button the visual display will go blank for a short period. This is to allow the auto photo calibration system to work without you seeing the rather funny things that happen to the S200 during this period. The auto setting procedure will now be described but your ability to operate the S200 will not be affected if the next paragraph is not fully understood.

If SIGNAL LEVEL Auto is selected the sequence is as follows. When you press the PHOTO button the scan control switches to Vis 1 scan speed, scans for a few frames and the autobrightness circuits average the signal from the specimen over this number of frames. This average signal is then adjusted to the level set by the Auto Level control (see section 2.4.1.) The autobrightness circuit is then locked at this value. Now the record display is switched on and a record scan (at the speed selected by the Fast switch see 2.3.2) started. At the end of the record scan the scanning system returns to the scan speed that you had set before pressing the photo button. This seemingly complex sequence happens automatically and ensures that all your photographs have the same exposure level.

If SIGNAL LEVEL Auto is not selected, or Hold is selected, the same sequence happens but the autobrightness system cannot adjust the average signal, so the photo will have the brightness set by the manual level control.

## 2.4 DISPLAY CONTROLS

Consists of six controls grouped to the left of centre of the control panel. These controls are:

### 2.4.1 SIGNAL LEVEL

A group of five controls. These are:

#### SIGNAL LEVEL Auto

Select Auto (switch pressed in) and the auto brightness system is functional. In this mode the average image intensity is set by the auto level control. This level will be maintained under all operating conditions. Set auto level and then adjust the resolution control, the image intensity does not change. To see the effect of autobrightness select auto level off and repeat the above test with the resolution control, using the Manual Level control to correct the image brightness.

Select non Auto (switch released) and the auto brightness system is off. The mean signal level is now controlled by the Manual Level control and will not be corrected for changes in signal level.

#### Auto Level

Sets the signal level to which the autobrightness system will adjust. It only works when signal level auto is selected.

Select signal level auto and turn the auto level clockwise, the brightness of the image on the display increases. Turn it anticlockwise and the image brightness decreases.

If any changes are made to the electron optics, final aperture or collector bias which changes the intensity of the electron input to the collector system, the autobrightness circuit will adjust the gain of the signal channel to maintain a constant signal level.

The normal setting for this control is so that, when graph is selected, the video signal almost spans the video level markers (see section 2.5.8 graph).

#### Manual Level

Sets the average signal level when SIGNAL LEVEL Auto is not selected. When in this mode the signal level will not be automatically corrected for changes in the input to the collector system.

#### Hold

When this button is depressed the Autobrightness system will be locked at its present level. Any changes to the signal will be seen on the display. When the button is released the autobrightness will continue functioning as normal.

When in the Hold mode the operator has no control of the signal level, it will hold the level set by the last autobrightness sample.

#### Contrast

If SIGNAL LEVEL Auto is selected, turning the Contrast control clockwise will increase the contrast of the displayed image. (The mean signal level will try to drop but the Autobrightness will adjust the gain to maintain the selected signal level.)

If Auto is not selected, operating the Contrast control will actually decrease the mean signal level. This allows the Manual Level control to be used to give greater contrast.

The contrast control can also reduce the "natural" contrast of the sample, so for normal viewing a small amount of contrast control must be used. The easiest way to set this "normal" contrast is to obtain a blank raster with no signal or noise showing (select non Auto signal level and turn the Manual Level to minimum). Increase Visual Bri until the blank raster

is visible. Adjust Contrast so that the blank raster is the same brightness as the background of the data zone. This setting will suffice for a large amount of microscopy and it is good operating practice to check this level at the start of each operating session.

#### 2.4.2 Display controls

A single control setting the brightness of the visual display CRT. This control is:

Visual Bri.

Sets the brightness of the visual display on visual speeds only. It has no effect on the record display. Set the signal level to its "average" value, using auto level and graph, and then adjust Visual Bri to give an image brightness that is comfortable to look at.

An alternative method of setting this is to select SIGNAL LEVEL non-Auto and set Manual Level to minimum to give a blank raster with no signal or noise showing. Adjust Visual brightness so that the background of the data zone is visible. Now set Contrast so that the raster is the same brightness as the data zone background. Adjust Visual Bri so that the blank raster is just visible in your ambient room lighting.

This level depends on ambient lighting conditions and operator preference, and may need to be altered frequently.

#### 2.4.3 THE KEY PAD

This can be used to enter the photograph and specimen numbers into the data zone and to control the linear measurement cursor. The function of the individual keys are:

A

Switches the output of the optional specimen current monitor into the top left corner of the data zone. If the specimen current monitor is in measure mode the sequence is:-

Press it once to switch this part of the data on.  
Press again and the specimen current monitor data is locked and will not respond to changes in specimen current until it is unlocked.  
Press the switch a third time and the specimen current monitor data is switched off.

If the specimen current monitor is switched to image mode the sequence is:-

Press once, data on.  
Press again, data still on.  
Press a third time, data off.

B

Sequences the Specimen number and the Photo number section of the data zone. Press it once and the specimen number will go into inverse video (black numbers on a white

ground). Keys 0 to 9 can now be used to set the Specimen number.

Press the key again and the photo number can be entered as above.

Press the key again and both number fields go back to normal video and keys 0 to 9 have no effect.

C

Press C once to enter cursor POS mode, when in this mode:

2 cursor lines will appear on the display, the micron marker size will be replaced by the cursor separation and POS will replace the micron marker.

E key moves the cursor lines (both together) to the right.

F key moves them both to the left.

Pressing the E or F keys momentarily moves the cursor one position.

Holding down E or F causes continuous movement. Releasing the key stops the movement.

**Note:** The cursor can only move one position during each frame scan, so cursor movements will be fast at TV scan and very slow at Vis 3 scan.

Press C again to enter the cursor sep mode. While in this mode: SEP appears in the data zone.

The left cursor line stays stationary while the right line moves.

E moves the line right.

F moves it left.

The keys work the same as for the pos mode above.

Press C a third time to exit the cursor mode and return to normal data.

While in the cursor mode the micron marker part of the display is replaced by a number representing the separation of the two cursor lines, which can be altered as described above. The cursor measurement is correct at all magnifications, acceleration voltages and working distances.

If using the cursor in the split screen mode, (see section 2.5.6 and 2.5.7) two things must be remembered.

- a. To make a sensible measurement, both cursor lines must be one half of the display.
- b. If the cursor is in the right half of the display then you must divide the separation number by the zoom factor.

D

Press the D key once and the entire data zone, including any text (see 2.11) will be switched off.

Press it again and the data zone will switch on again.

## 2.5 Image Processing Controls (fig 2.5)

This is the group of controls on the upper left control panel. It provides the following facilities:

- a. Split Screen and Dual Magnification
- b. Input Switching
- c. Video Processing
  - Signal Mixing
  - Differentiator
  - Signal Invert
  - Gamma
- d. Scan Processing
  - Normal
  - Graph
  - Line
  - Spot

### 2.5.1 INPUT SELECT

This is a 4 position switch which lets you select the type of signal you want to look at. The choices are:-

S.E.

Secondary Electrons from the photomultiplier head amplifier. Although called Secondary Electrons the output of the head amplifier may be either secondary or reflected electrons, as selected by the SE/RE switch. This is mounted on the side of the black plastic block protruding from the back of the chamber. When the switch is forward, toward the chamber, the collector cage is biased positively and both secondary and backscattered electrons will be collected. When the switch is pointing backward, away from the chamber, the collector cage is at ground potential and backscattered electrons only will be collected.

B.S.E.

Backscattered Electrons from an optional backscattered detector. Three types of Backscatter detector are available. These are:

- a. Four element solid state detector
- b. Annular solid state detector
- c. Scintillator backscatter detector

These are fully described in sections 4.10 to 4.12.

X-RAY

The output from the ratemeter of X-Ray processing equipment or from the optional X-Ray processor.

AUX - For any other input that you may have. The input sensitivity is 1v into 100ohms. If an optional Specimen Current Monitor is fitted it will be fed into this input.



The input sockets selected by these switches are mounted on the back of the small trim panel to the right of the visual display.

#### **2.5.2 SIGNAL MIX.**

Allows you to mix the signal selected by the INPUT SELECT switch with the secondary electron signal to produce a composite signal which appears on the display.

Turn the control fully clockwise to get a secondary electron image.

Turn the control fully anti clockwise to see the signal selected by the INPUT SELECT switch (assuming that you have an optional detector fitted.)

Turn the control to an intermediate position to get a linear mix of these two signals.

#### **2.5.3 INVERT.**

With this switch in the down position the signal is shown "normally" on the display. This means that areas of specimen which give a large signal to the detector appear as bright areas on the picture. When the switch is up the signal is inverted and the picture on the display will appear as a negative, i.e. with whites being black and vice versa. This facility may be used to invert specimen current images, making them easier to compare with reflected electron images.

If a normal image is inverted it can be photographed directly on negative film to make a slide for projection.

#### **2.5.4 GAMMA.**

When applied, GAMMA makes the video amplifier behave in a non linear fashion. Low level video signals are enhanced while high level video is attenuated. This allows the overall contrast of an image to be reduced while the contrast in low brightness areas is increased.

When this control is fully anticlockwise it has no effect on the picture. As it is turned clockwise the dark areas on the picture begin to get brighter, but the bright areas stay the same.

#### **2.5.5 DIFF.**

Processing the video signal by Differentiation enhances the detail in low contrast areas by "sharpening" the edges of areas of almost equal brightness. When used on low contrast specimens it shows the boundaries between different areas. When used on specimens with a large amount of fine detail it will enhance the detail.

When the control is fully anticlockwise it has no effect on the image. When turned clockwise it has maximum effect.

### 2.5.6 SPLIT SCREEN.

Sets the scanning system into the split screen mode, where the display is split vertically into two halves. The left half of the display always shows the secondary electron image at the magnification set by the magnification controls. The right half of the display can show one of the following:

- a. The same signal at a higher magnification selected by the ZOOM control.
- b. A different signal, selected by the INPUT SELECT switch, at the same magnification or a higher magnification selected by the ZOOM control.
- c. A mix of secondary electrons and one other signal, selected by INPUT SELECT and MIX, at the same magnification or a higher magnification selected by the ZOOM control.

When in the split screen mode, all video processing is done equally on both halves of the image.

AM2

### 2.5.7 ZOOM.

For best results on record use Split Screen on scan speeds of 2 and below.

Operates in conjunction with the split screen control. Select split screen and ZOOM X1. Both halves of the display are at the same magnification and the zoom factor displayed in the top right of the data zone says X1. Now select ZOOM X2. The right side of the display increases in magnification by a factor of 2 and the zoom factor in the data zone says X2. A bright rectangle on the left side of the display (the lower magnification side) shows you where the higher magnification image comes from. The bright "window" can be moved about on the left half of the display using the X and Y POS controls.

The X4 and X8 zoom factors work in the same way.

While in the split screen mode the magnification controls work as normal on the left side of the display, with the right half always zoomed by the selected factor.

If the cursor (section 2.4.3) is being used on the right side of the display, then you must divide the separation number by the zoom factor.

The cursor mode can only be used sensibly when both cursor lines are on the same half of a split screen image.

### 2.5.8 SCAN

A four position switch selecting

- Normal
- Graph
- Line
- Spot

#### Normal.

When this is selected the SCAN switch has no effect but one effect of the SCAN system can be seen on the display. Somewhere on the display should be a small white square, called the spot. Its position can be adjusted by the X POS and Y POS controls. It shows the position of Graph or Spot (see below).

In normal use this square can be left positioned under the data zone so it is not visible on the image.

#### Graph.

Press Graph and the picture on the display will be replaced by a wavy line. This is a graph displaying the video intensity of one line on the specimen. The graph is taken from a line on the specimen drawn horizontally through the white square spot. This can be moved using the Y POS control. (X POS will move the spot but will have no effect on the graph.)

Graph is drawn at the line speed of the record raster selected by SCANNING MODE and is not intensity corrected for photography.

On each side of the display are drawn two short horizontal red lines. When in the graph mode the lower line represents zero video level, or black on a micrograph. The top line represents the maximum useful signal level, or white on a micrograph. (Signals can go above or below the markers but since they are outside the recording range of the micrograph they are of no use.)

Select Graph and Auto SIGNAL LEVEL. Adjust Auto Level and Contrast so that the Graph almost spans the markers. Switch back to Normal. The image on display is now using the full dynamic range of the video system.

#### Line.

This gives a graphical display as described above. The image is a graphical representation of the signal intensity on a line drawn horizontally across the sample through the spot. The main difference between Graph and Line is that of speed. Graph is drawn quite fast, at record line speeds. Line is drawn slowly, each Line scan taking the frame time selected by the SCANNING MODE switch. Each line scan can be as long as 200 seconds if slow record is selected.

It is used to generate graphs of signals that require a slow sampling rate to give a statistically correct picture, particularly X-Rays and Cathodoluminescence.

When in the Line mode you will see that the line drawn consists of a number of discrete points. The signal is being "chopped" to ensure that the brightness on the visual display is not too high, and to control the photographic exposure on the record unit.

If Line is used on visual speeds, you should either lock the autobrightness or select manual brightness control. Also

Visual Bri may have to be increased to make the Line visible at the faster scan speeds.

Spot.

Switch back to Normal on the Image Processor. Move the X and/or Y POSITION controls to place the bright square on some feature on the picture. Switch off the autobrightness. Now switch to Spot. This will stop the scan in the column and park the beam at a point indicated by the centre of the square. The square itself will have a brightness equivalent to the signal at that point on the specimen.

**Note:** If an X-Ray processor option is fitted then the function of some of the SCAN controls will be altered. For details see section 4.3.

## 2.6 Beam Processing Controls (fig 2.6)

Consists of a number of controls grouped on the upper right control panel. It provides the following facilities:-

- a. FOCUS MODULATION                   - Focus wobble  
  - Dynamic Focus
- b. TILT CORRECTION
- c. GUN ALIGNMENT                       - EMISSION IMAGE  
  - X and Y Tilt  
  - X and Y Shift

### 2.6.1 FOCUS MOD

Off

This is the "off" switch. When it is selected the FOCUS MOD controls are inoperative.

Focus Wobble.

Allows the final aperture to be centred onto the electron optical axis of the column. Select TV and obtain a focused image at a magnification in the range from 1KX to 10KX. Switch to Focus Wobble. Turn up the CHANGE control. The picture swings in and out of focus, by an amount varied by the Change control. If the image also moves laterally on the display the final aperture is not centred. Adjust the aperture centring micrometers for minimum picture shift (as in section 2.9).

Dynamic Focus.

When a sample is viewed at a very high angle of tilt it will not be in focus all over. If it is in focus in the centre it will be out of focus at the top and bottom of the slope. Dynamic Focus corrects for this by adding some of the vertical scan on to the focus. The amount of scan added is adjustable with the CHANGE control. It works best where the plane of tilt is in the vertical axis of the screen. This will occur at 15mm working distance. At any other working distance, scan rotation can be used to rotate the scan to align with the

plane of tilt. This condition is fulfilled when the stage movements appear horizontal and vertical on the display. There are two Dynamic Focus switches; "+" and "-". Dynamic Focus + is regarded as normal in that the specimen is tilted towards the collector system i.e. the top of the picture is at longer working distance. Dynamic Focus - is for a specimen which tilts away from the collector.

There are two methods of setting up Dynamic Focus. For the first method switch to TV and focus the centre of the image, then at VIS 2 or VIS 3 scan speed use the CHANGE control to focus the picture at the bottom of the screen.

The second method uses some of the controls on the image processor. First use the Y POSITION control to put the spot at the centre of the screen's vertical axis. Now switch to Graph. You can then adjust the FOCUS controls on the main instrument to obtain nice sharp peaks and troughs on the Graph waveform. You will find this easier if you select slow scan speed. Next turn the Y POSITION control fully anticlockwise, and focus again, this time with the CHANGE control. You should end up with a nicely focused picture. If you need to change magnification there is no need to readjust the controls, as Dynamic Focus is compensated for changes in magnification (and accelerating voltage.)

#### 2.6.2 Change

Controls the amount of Focus Wobble or Dynamic Focus. Turn it fully anticlockwise and the Focus Wobble and Dynamic Focus have little effect. Turn it clockwise and they have maximum effect.

#### 2.6.3 TILT CORRECTION.

When a specimen is tilted, it appears to be shorter in the plane of tilt than it would be if viewed face on. The TILT CORRECTION control compensates for this by increasing the magnification in the vertical axis on the screen. Again, this works best with the specimen tilted in the vertical axis. With a flat specimen, you simply use this control by setting it to the same angle as the surface of the stub the operator must make his or her own judgement as to what angle to set, using the stage tilt angle as a guide.

#### 2.6.4 GUN ALIGNMENT CONTROLS

A set of five controls which allow the position of the electron source to be set on the electron optical axis of the column. They only have limited range and require that filament alignment (section 3.3.7) is done correctly.

##### Emission image

Switch it on and you see a pictorial representation of the emission profile of the filament (see figure 2.10). It is not an image of the end of the filament. When in the emission image mode you can use the GUN ALIGNMENT controls to adjust the filament emission to be on the axis of the column.

#### X and Y Shift

Allow the source of electrons emitted from the filament to be shifted laterally over the top of the column so that the brightest part of the source is projected onto the specimen. You should adjust these controls so that the bright emission image is in the centre of the visual display.

#### X and Y Tilt

The emission density from the filament will be a maximum at one particular point on its surface and in one particular direction. These controls allow you to "tilt" the gun to use the best emission angle of the filament. This is not a mechanical gun tilt but an electronic simulation of it. The correct setting is when the brightest part of the emission profile is in the centre of the emission image.

One method of setting the above controls is now given.

Obtain an image as described in chapter 1, routine 2. Select a RESOLUTION of 2 or 3.

Set the filament control to the centre mark.

Set the SIGNAL LEVEL switch to Auto and the Auto Level control to centre travel.

Switch on the EMISSION IMAGE.

Using the X and Y Shift controls in the GUN ALIGNMENT group, centre the bright area on the visual display.

Select a RESOLUTION of 7 or 8. Using the X and Y Tilt controls, centre the brightest part of the bright spot on the display. If the picture is too bright to allow detail to be seen in the emission image, turn down the AUTO LEVEL control.

Set the filament current to give the required operating conditions. The emission image will look something like those shown in figure 2.10. For normal operation the filament current should be adjusted to give an emission profile as shown in figure 2.10.3. This should be with the filament control knob somewhere near the centre marker of the scale. Do not worry if it is not at the centre marker as it varies with different filaments and with the age of the filament. As a filament gets older the Filament control should be reduced to maintain this emission profile. This setting should give good performance with a long filament life.

The ultimate resolution of the S200 will be better if the filament current is increased to give an emission profile as shown in figure 2.10.5 but the filament life will be shorter.

### 2.6.5 Scan Rotation

Scan Rotation consists of two controls and a digital readout.

Off/Course/Fine

When Off is selected, Scan Rotation has no effect, independent of the reading of the angle display.

When Fine is selected the Change control rotates the image in 1 degree increments continuously from 0 to 360 degrees.

When Coarse is selected the Change control rotates the image in 10 degree increments.

You can switch from Fine to Coarse, or back again, at any time. The coarse 10 degree increments will start at the angle set by the Fine control, and vice versa. The Scan Rotation can be left set at any predetermined angle and switched on and off.

The Scan Rotation will reset to zero when the S200 OPERATE is switched off.

CHANGE

Changes the angle of Scan Rotation.

## 2.7 VACUUM CONTROLS (fig 2.7)

This group of controls, mounted on the front of the plinth, switch and monitor the vacuum system of the S200. Some of the controls on this panel are only operative when the gun pumping option is fitted. The basic system is described here. If you have the ion pump option fitted please refer to 4.1.

The standard controls consist of:-

### 2.7.1. CHAMBER Vacuum

A green push button controlling the basic vacuum system of the S200.

Press it once (so that the button stays depressed) to start the pumping system and pump down the column and chamber. When a working vacuum has been achieved (vacuum ready) the switch is illuminated. Press the switch again to turn off the vacuum system and vent the column and chamber to air. (Release the clamp on the stage front plate if pressurised dry nitrogen backfilling is being used.)

This control will not operate until power is switched on, but it can be used independently of the operate switch. If the vacuum system is vented with operate selected, then certain operate functions will be inhibited. These include the supplies to the electron gun, collector system and the displays.

**Note:** It is good practice to check that the beam is switched off before venting the vacuum system. It is also advisable to keep the column and chamber under vacuum when the S200 is not in use. Prolonged exposure to air may contaminate the inside of the electron optical system, leading to long pump down times and possibly degraded instrument performance, particularly in atmospheres of high humidity.

The S200 vacuum system is designed for continuous operation and is fully protected against failure.

### 2.7.2 Pressure readout

A bar LED meter, calibrated in TORR, showing the vacuum in the column and chamber, as selected by the Column - Chamber switch.

### 2.7.3 Col-Chamber

Selects whether the vacuum readout displays the vacuum in the column or the chamber.

### 2.7.4 Column Vacuum

)

### 2.7.5 Valve Status

) Described in section 4.1  
)

## 2.8 Record Unit Controls and Calibration (fig 2.8)

These controls are mounted on the back of the record unit top panel, behind the camera mount.

### 2.8.1 Brightness

Adjusts the brightness of the record CRT only. It has no effect on the visual display.

### 2.8.2 Contrast

Adjusts the contrast of the record CRT only. It has no effect on the visual display.

### 2.8.3 Film/ASA

Adjusts the intensity of the record display to compensate for different types of film (assuming the camera has been set at the correct aperture). Since different types of film of the same nominal film speed vary in sensitivity to the blue light emitted by the record CRT the Film ASA correction cannot be made totally accurate and the system may have to be calibrated for different film types.

### 2.8.4 Calibration

This section outlines the complete procedure for calibrating the record unit. The settings resulting from this should not need to be adjusted frequently, but should be checked periodically (say once a month) or when good micrographs are not obtained consistently. Long term changes may arise due to the ageing of components such as the CRT.



The calibration procedure is:

1. Select the appropriate lens aperture for the film and camera back as follows:-

Film Speed	Camera Type	
ASA	405 or 545	70mm, 35mm, 120
50 to 400	f8	f11
800	f11	-
1600	f16	-
3200	f22	-

and check that the lens and spacers are in the correct position for the back being used (see figure 2.11).

2. Set the Record Bri and Record Cont controls to zero.

3. Select a black video level as follows:

Obtain an image at a resolution setting of about 6, set signal level to non auto and then select beam off. Select graph and adjust contrast so that the signal level displayed is level with the lower signal level markers. The graph should be a straight line.

4. Put a film in the camera, open the camera shutter (if it has one) and start a photo scan by pressing the PHOTO button.

5. As the scan runs increase BRIGHTNESS by a small amount, say 0.1 turns every 5 seconds. This will produce a grey scale on the photo. Note the BRIGHTNESS setting in relation to the scan position.

6. Develop the film. Study it and choose a BRIGHTNESS setting which just causes perceptible lightening of the film. Set BRIGHTNESS to this value and lock the control.

If no greys have been achieved, or the grey steps are too widely separated for an accurate setting to be made, repeat this test over a higher brightness range or using smaller increments.

7. Select a white video level by inverting the black level using the INVERT switch.

8. Load a film and start a photo scan. As the scan runs increase the CONTRAST control by a small amount, say 0.1 turns, every 5 seconds.

9. Develop the film and choose a CONTRAST setting which just produces a saturated white on the photo. Set this level on the CONTRAST control and lock it.

If the contrast steps are too small or covering the wrong range, repeat the test with smaller steps or over a different range.

10. Check the settings of BRIGHTNESS and CONTRAST by taking a micrograph of a suitable sample, setting the video signal level to cover (nearly) the full video level range, as described in section 2.5.8 Graph.

#### Uncal LED

If the Uncal LED is illuminated then the Record scan speed selected is too fast for the film speed selected by the Film ASA control. When this happens you must either select the slow photo speed or use a slower type of film.

### 2.9 Final Aperture Centring (fig 2.9)

Good images can only be obtained from an SEM in which the final aperture (or the projected final aperture) is correctly centred onto the electron optical axis of the final lens. If the final aperture is not correctly aligned, image shift will occur when the focus is adjusted.

The final aperture mechanism of the S200 holds four apertures which can be interchanged and adjusted under vacuum, by the two micrometer controls situated in the side of the column just above the chamber. The method of adjustment is as follows:-

1. Select the required aperture size. The recommended aperture sizes and the corresponding micrometer settings are

Position	Aperture Size	Micrometer X	Micrometer Y
1	20 micron	6	0
2	20 micron	6	6
3	50 micron	6	12
4	50 micron	6	18

The micrometer readings may vary slightly on different instruments. The aperture size fitted in any position may also vary, depending on particular requirements of the instrument operator.

2. Obtain a TV rate image at about 1KX magnification.
3. Select Focus Wobble and increase the CHANGE control (both on the FOCUS MOD switch) so that the image is "wobbling" in and out of focus.
4. Adjust the aperture centring controls to give zero image shift with changing focus.

5. Increase the magnification and repeat steps 4 and 5. Continue until the image shift is small at 100 KX.

## 2.10 THE DATA ZONE

In addition to the controls described above there is one other very important part of the S200 control system, the data display. This is an area across the top of the display showing all the important operating parameters of the S200. It looks like this:

```
27PA 101KX 25KV  WD:5MM  S:12345  P:67890
      200NM - - - - -                X4
```

The significance of this string of information is:

27PA

Information coming from the optional Specimen Current Monitor, if fitted. (See section 4.7.)

101KX

The magnification of a micrograph when taken on the standard 5 inch X4 inch Polaroid camera supplied with the S200. The magnification is correct at all acceleration voltages and working distances. The magnification on the visual display is approximately 1.6 times greater than this.

25KV

The value of the acceleration voltage selected.

WD:5MM

The distance from the point of the sample which is in focus to the final lens.

S:12345

An operator selected specimen number. For the method of selecting this number read section 2.4.3 THE KEYPAD.

P:67890

The number of the next micrograph to be taken. The start number of this sequence can be selected using the keypad, and the number is then automatically incremented each time a micrograph is taken.

200NM - - - - -

The micron marker. The length of the bar in the example shown is 200 nanometers. This marker is correct on both the visual display and the micrograph at any acceleration voltage and working distance.

If the data system has been put into the cursor mode the micron marker will be replaced by a number, in inverse video, representing the separation of the two cursor lines. The use of the cursor is described in section 2.4.3 THE KEYPAD.

(Inverse video is dark numerals on a light background.)

X4

The Zoom factor in use, if SPLIT SCREEN is switched on. In the data zone drawn above the magnification of the left image is 101KX, that of the right image is 404KX. (A rather optimistic example but the numbers were chosen to show the maximum number of characters available in the data zone.)

## 2.11 The Text Facility

This allows the user to write over the whole of the SEM display using the keyboard.

There are two main facilities provided. These are:-

- a. the ability to place text on the SEM screen in any position for the purpose of providing information about the specimen or operating conditions.
- b. the ability to store up to 16 title messages in a temporary memory and to recall them at will for giving titles to micrographs.

### 2.11.1 Text Screen Format

The Text writing area starts immediately below the Data Zone and consists of 24 rows of 50 characters. The characters available are:

Uppercase letters	A-Z
Numerals	0-9
Symbols	!"#\$%&"()=-'+;:<, >. ? / *

### 2.11.2 Writing Text

After the SEM power is switched on you will notice a question mark on a flashing video background in the top left corner of the screen immediately under the Data Zone. If the video level is low in this region then you may not notice the flashing background. This character is called the CURSOR and indicates the current writing position. (It may not be visible on a TV raster, try Vis 1).

If a key is typed on the keyboard this letter will replace the cursor and the cursor will be moved one position to the right. This process repeats as more characters are typed until the end of the line is reached. At this point the next character typed will replace the cursor as usual but the cursor will move to the beginning of the next line. If any key is held down the autorepeat facility will cause a continual string of letters to be printed.

If a new line is required before the end of a line then just press CR on the keyboard. A full screen of text can be typed in this way. When the bottom right corner of the text screen is reached the cursor returns to the top left corner.

### 2.11.3 Editing

There are five keys on the keyboard which are used in editing the screen text. These are:

DEL.....Delete key

If you mistype a character you can remove that character using the DEL key and type the correct character. Similarly, if you want to delete several characters or a whole word then hold down DEL and the cursor will backspace deleting characters as it goes.

To use the arrow keys, hold down the SHIFT key while pressing the arrow key.

RIGHT ARROW	Move cursor to the right
LEFT ARROW	Move cursor to the left
UP ARROW	Move cursor up one line
DOWN ARROW	Move cursor down one line

As the descriptions suggest, these keys move the cursor character in the appropriate direction without altering any of the text on the screen. If the cursor is moved on top of a character then this character is not lost but temporarily stored. When the cursor is moved away from this position using one of the cursor control keys then the original character is restored. In this way the cursor can be moved to any position on the screen, passing over any number of words or characters without altering the screen content.

If the cursor reaches the right edge of the screen then further right movement causes it to go to the start of the next line. Similarly, if the cursor reaches the left edge of the screen then further left movement causes it to go to the end of the next line above.

If the cursor reaches the top edge of the text screen then it re-appears from the bottom of the screen and vice versa.

The cursor can be used to move to a particular point on the screen to write a new word or it can be used to edit a mistake embedded in a block of text. This editing role is carried out as follows:-

- a. Using the cursor control keys, put the cursor over the character to be deleted.
- b. Type the correct character. (The cursor will move one place to the right.)
- c. Move the cursor away using the cursor control keys.

### 2.11.4 Video Mode

There is a choice of video background for the Text characters. The characters can either be superimposed on top of the SEM video or the SEM video can be turned off behind individual characters leaving the character setting in a box. The former case does not obscure much of the

specimen being viewed but the characters can be difficult to read on bright or high contrast areas of the specimen. The latter case gives clarity of reading but blanks off some of the image.

The video mode is selected by CTRL V. (This means press V while holding down the CTRL key.) This changes the cursor character from a question mark to an asterisk. While the cursor character is an asterisk any character typed on the screen will have a black background behind it. Pressing CTRL and V again will bring back the question mark cursor. The two modes can be used at any time to produce a mixture of text with a video background and text with a black background.

This video mode feature can be used with the cursor control keys to change the video mode of characters already on the screen by just running the cursor character over the text using the cursor control keys. This can be done as follows:-

Say that the word SPECIMEN is written on the screen (in normal characters) in an area where the video level is quite high, making the reading of the word difficult. We would like to turn off the video background behind these characters. Carry out the following steps:-

- a. Move the cursor character until it is over the first letter to be changed (using the cursor control keys).
- b. Change the video mode by pressing CTRL and V. The cursor character should now be an asterisk.
- c. Move the cursor over the letters to be changed using the cursor control keys. As the cursor passes over the letters, their video mode is changed.

Note that "black boxes" can be drawn with the asterisk cursor to mark areas of interest by just using the cursor control keys to move the cursor around the area. As the cursor moves it turns off the video leaving a trail of black boxes.

#### 2.11.5 Title Mode

In the TITLE MODE the top line of the text screen (immediately under the Data Zone) is reserved for placing titles and is underlined. There are three control keys which control the operation of this mode. They are two key operations which consists of holding down the CTRL key and simultaneously pressing a letter key. The operations are as follows:-

TITLE MODE ON/OFF.....CTRL T

Turns on the TITLE MODE. The first text line will be blank and the second line will be filled with hyphens. At this time the title memory is empty. The cursor will be moved from wherever it was located before entering the Title Mode to the top left under the line of hyphens. This is so that the cursor can easily be moved into the Title line for creating or editing titles.

Titles are created by moving the cursor into the title line and typing as normal. When the title is complete typing CR will bring the cursor out of the title line to the start of the line under the hyphens. The Title can now be stored.

When the Title Mode is not required anymore CTRL T will turn it off, erasing whatever was in the title line and also the line of hyphens. The titles that have been saved will still remain in memory as long as OPERATE remains on.

#### SAVE TITLE.....CTRL S

Will save whatever is in the title line in a temporary memory. This memory only retains its information while OPERATE remains on. The characters in the Title line can be in either Video Mode and they will be saved and recalled as such. Up to 16 titles can be stored and recalled at will.

#### EXAMINE TITLES.....CTRL E

Causes the next title to be displayed in the title line. The 16 titles form a continuous loop so that when the 16th title is reached the next CTRL E brings you back to the first title.

#### CLEAR SCREEN.....CTRL C

Clears the screen of all text. (When the S200 is first switched on the Text screen is automatically cleared.) All the text is lost, the title mode is turned off (title line and hyphens erased) but the title memory is left intact. The cursor is returned to the top left of the screen. The only way of clearing the title memory is by writing spaces in the title line and saving them by CTRL S, or by switching OPERATE off.

#### TEXT ON/OFF.....CTRL B

If the text is not wanted on the micrograph but still saved in memory then CTRL B turns off the text video. CTRL B also brings the text back again. This is similar in function to the key D on the console keypad. Whereas, CTRL B just affects the text, D on the keypad turns off both the text and the Data Zone.

## 2.11.6 Summary of Key Functions

Key	Function
LEFT ARROW .....	Moves the cursor left one position
RIGHT ARROW .....	Moves the cursor right one position
UP ARROW .....	Moves the cursor up one line
DOWN ARROW .....	Moves the cursor down one line
RETURN .....	Fills the rest of the line with spaces and moves the cursor to the next line
DEL .....	Moves the cursor left one position deleting the character it moves over
CTRL C .....	Clears the screen. Text is lost
CTRL B .....	Turns the Text off. Text is retained in memory
CTRL V .....	Changes the VIDEO MODE ? - Normal mode * - Video off
CTRL T .....	Turns the TITLE MODE on and off
CTRL S .....	Saves the current contents of the title line in memory
CTRL E .....	Examine and display the next stored title





Figure 2.1  
Power Switching Controls

# STEREOSCAN 200

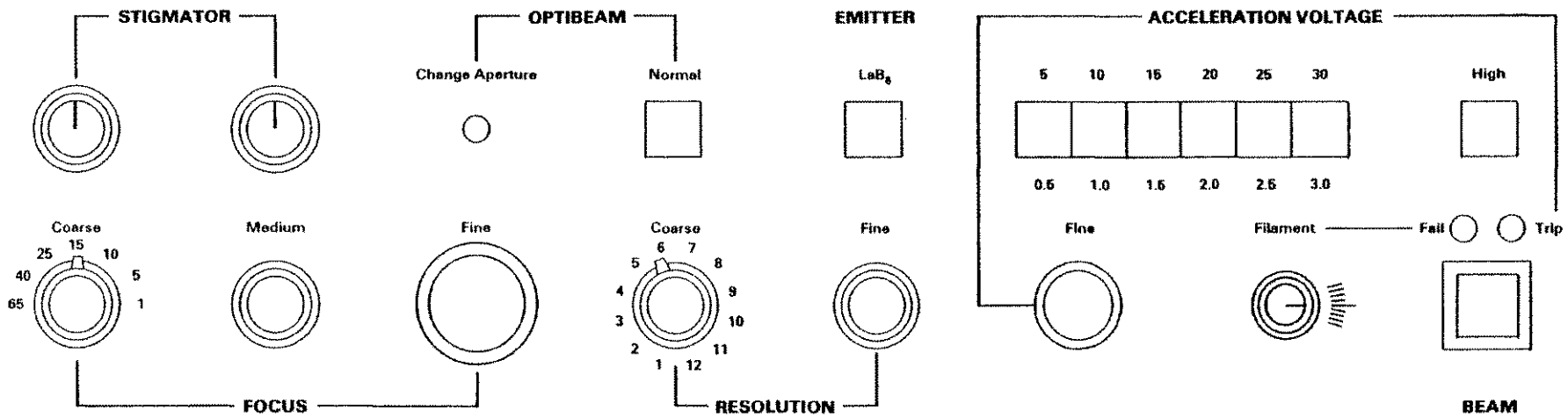


Figure 2.2  
Electron Optics Controls

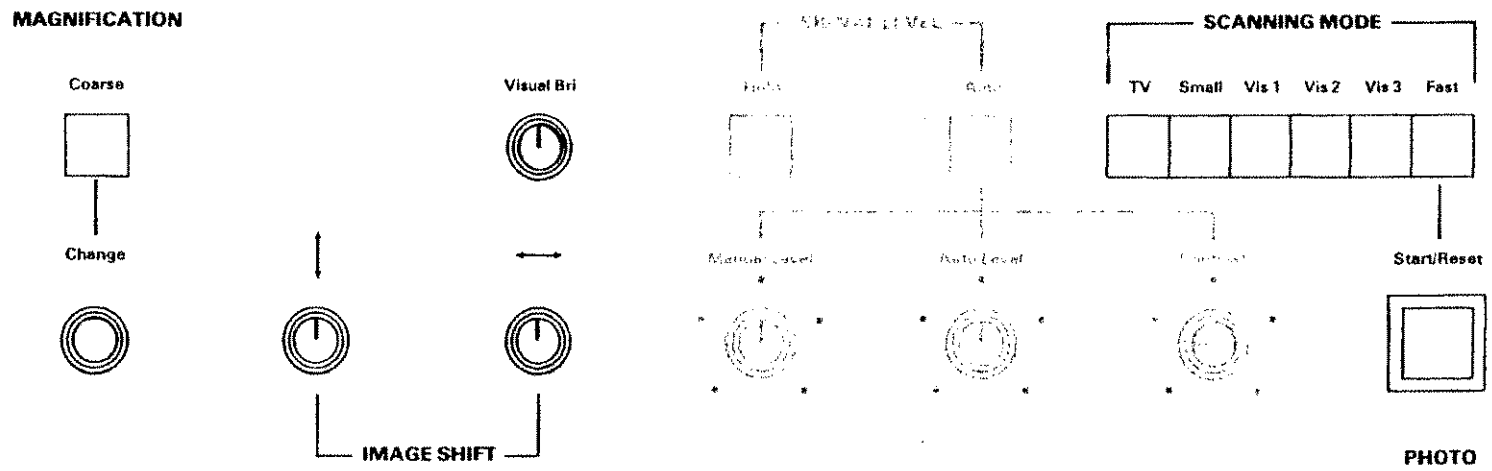


Figure 2.3  
Scanning Controls

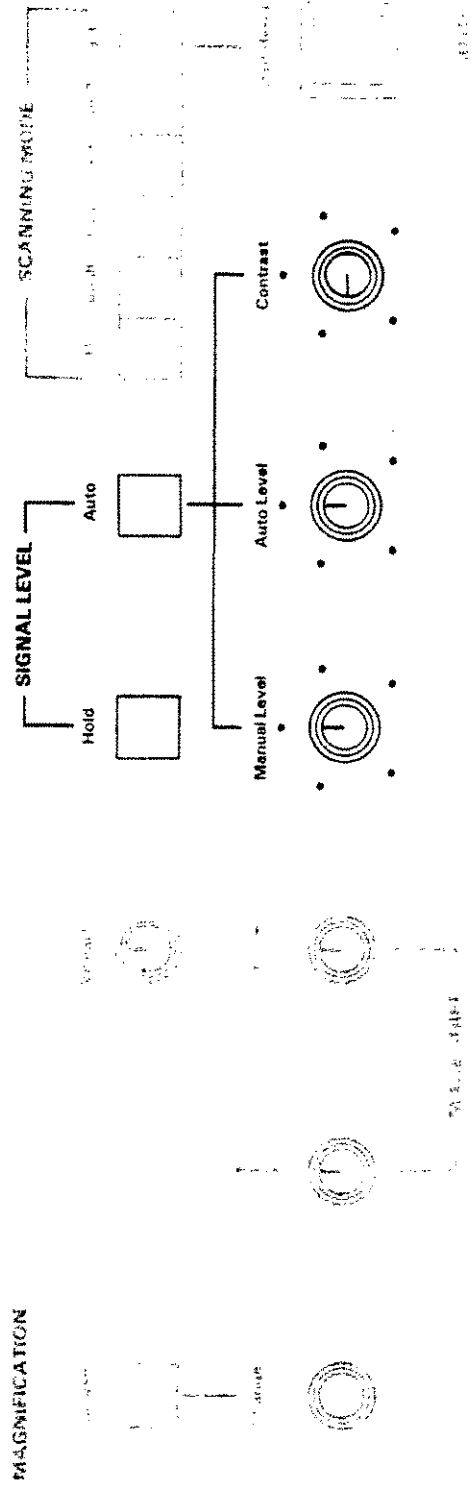


Figure 2.4  
Display Control

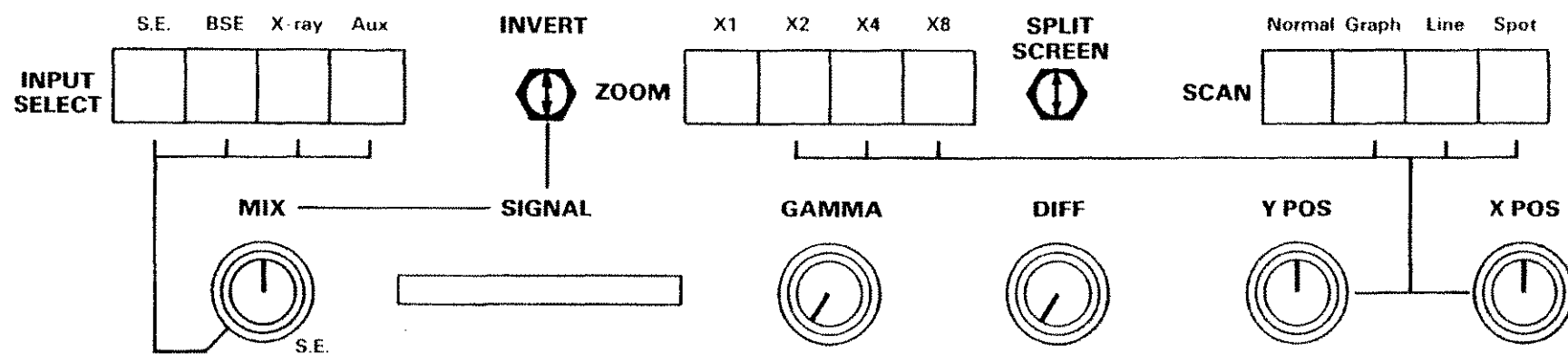


Figure 2.5  
Image Processing Controls

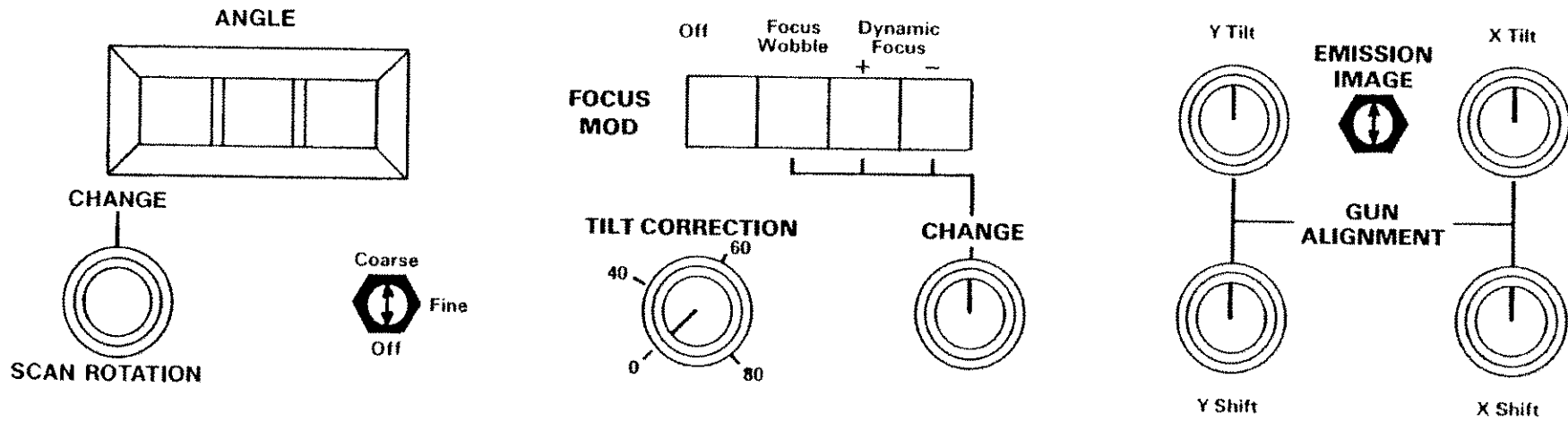


Figure 2.6  
Beam Processing Controls

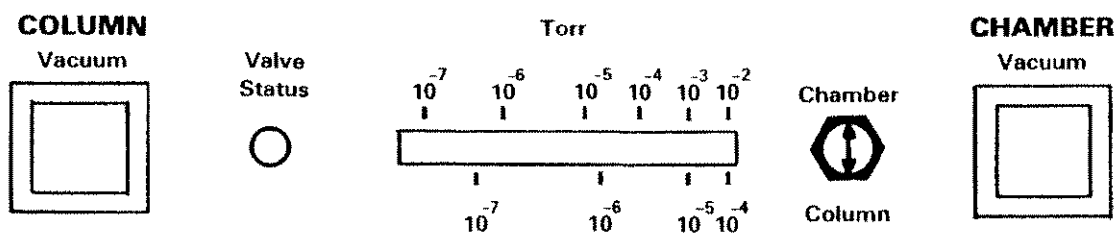
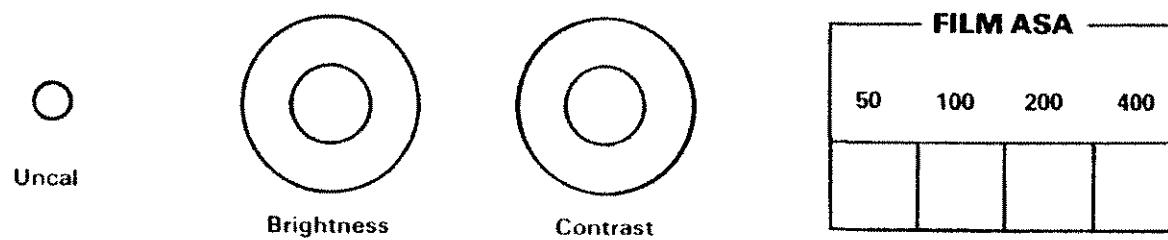


Figure 2.7  
Vacuum Controls



**Figure 2.8**  
**Record Unit Controls**



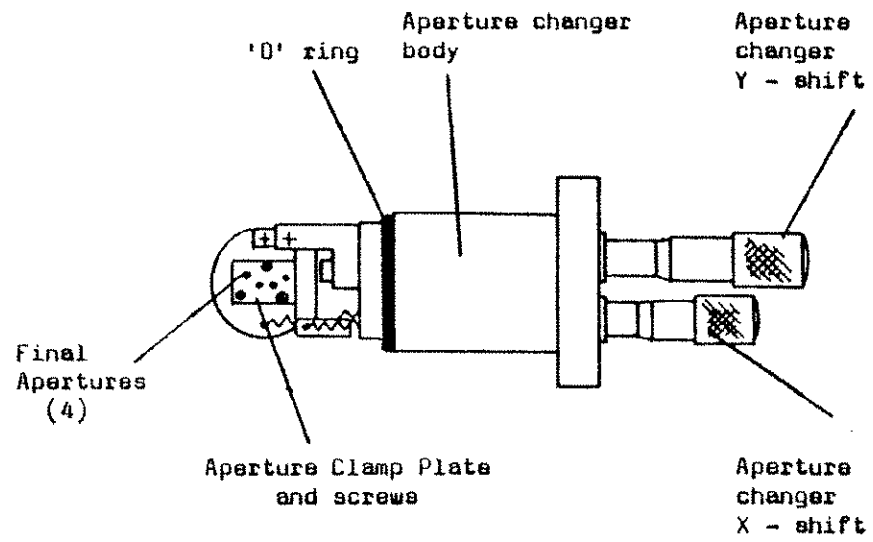


Figure 2.9  
Final Aperture Changer

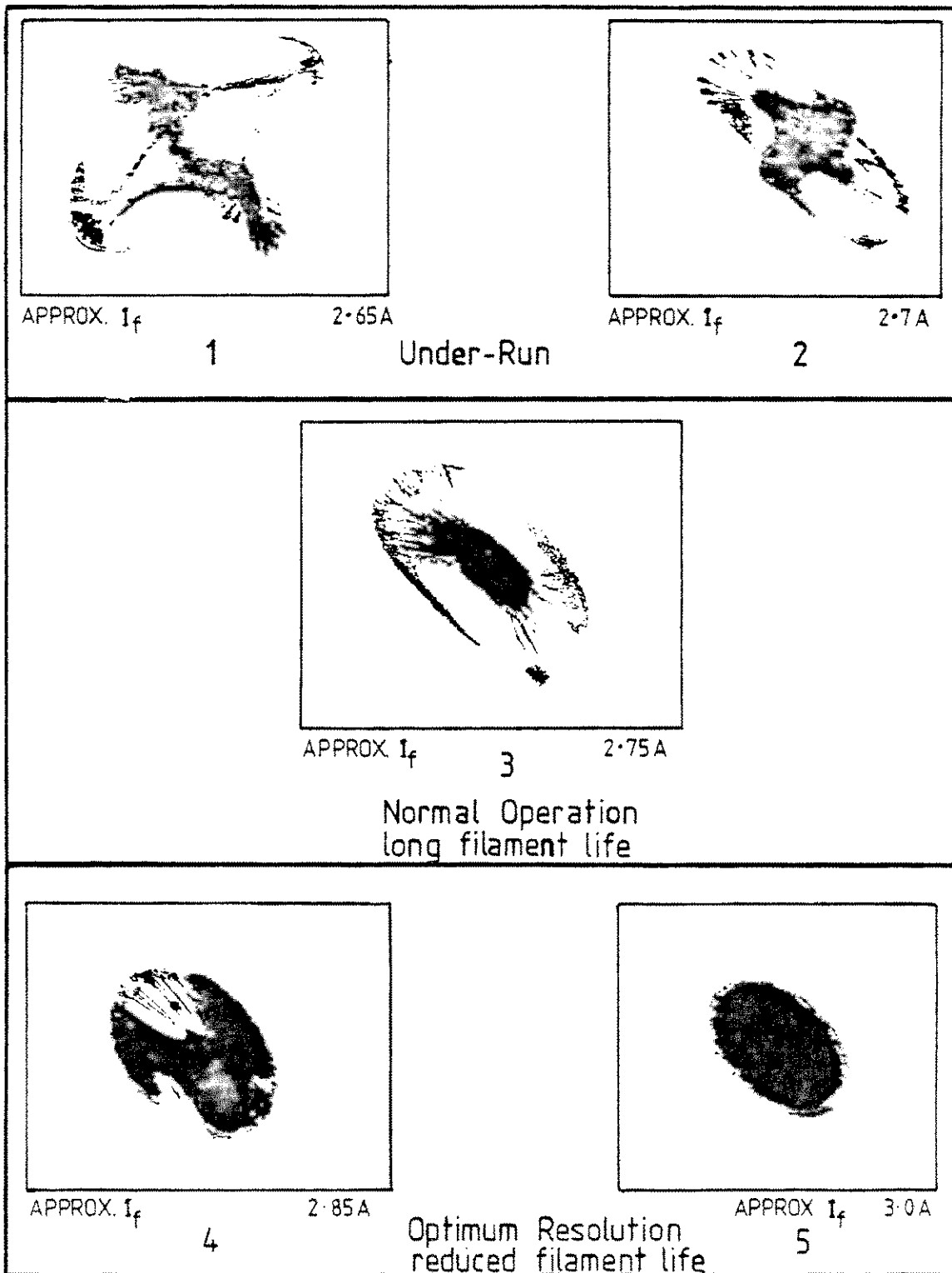


Figure 2.10

Setting the Filament Current

Note: The improvement in resolution at high filament current is marginal

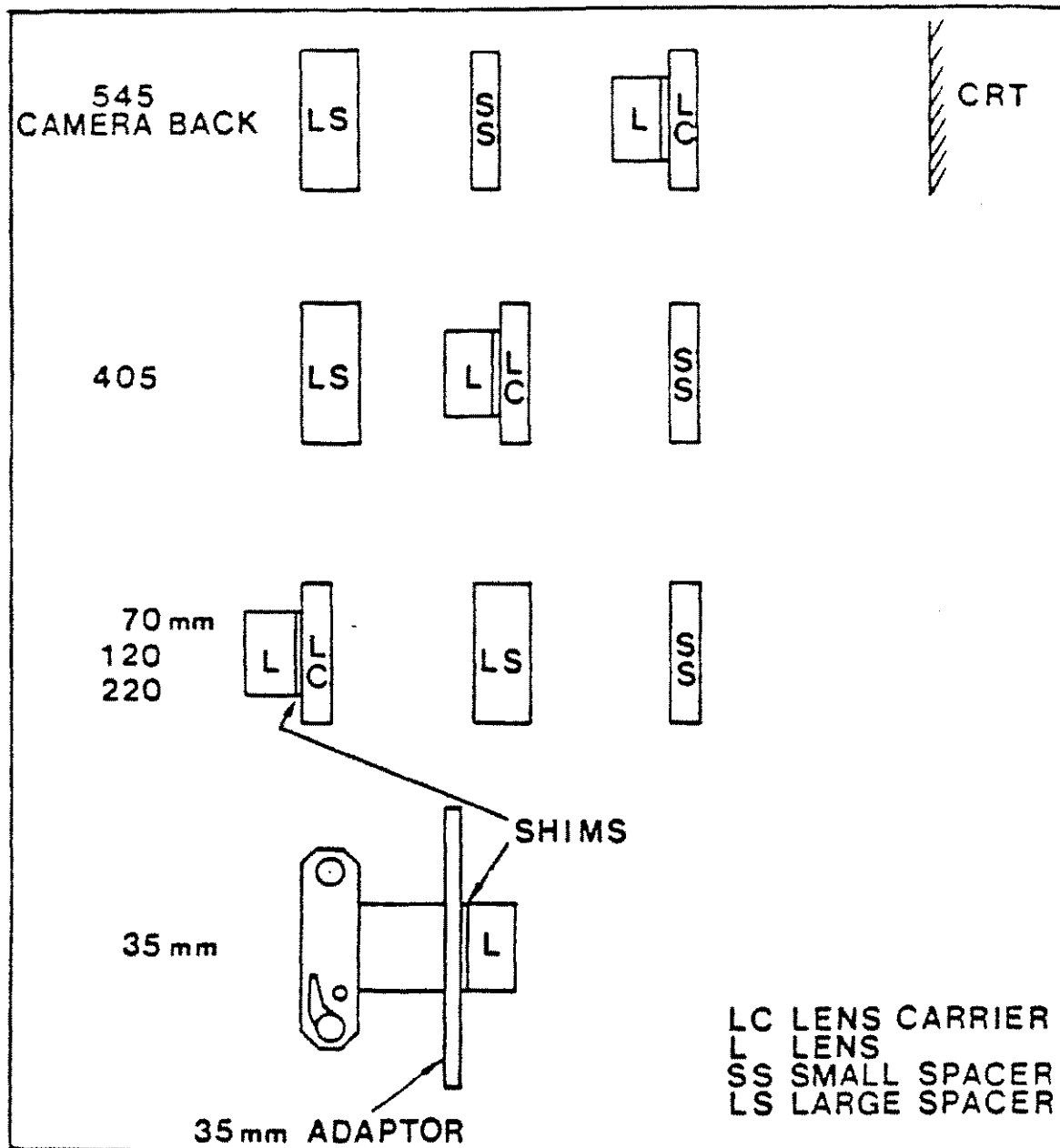


Figure 2.11

HRRU Lens Positioning

## CHAPTER 3 MAINTENANCE

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  - 3.1.1 Rotary pump oil level
  - 3.1.2 Air admittance drier assembly
- 3.2 6 MONTHLY MAINTENANCE
  - 3.2.1 Changing the rotary pump oil
  - 3.2.2 Rotary pump belt tension
  - 3.2.3 Changing the turbo pump oil
- 3.3 COLUMN SERVICING
  - 3.3.1 Cleaning recommendations
  - 3.3.2 Routine column cleaning
  - 3.3.3 Basic column dismantling, cleaning and assembly
  - 3.3.4 Complete column breakdown
  - 3.3.5 Cleaning the specimen chamber
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  - 3.3.7 Changing the filament
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- 3.4 REPLACING THE SWING DOOR STAGE
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- Figure 3.4 The Final Aperture Changer
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- Figure 3.5a Collector System (assembly sequence)
- Figure 3.6 Column Alignment Screws
- Figure 3.7 Stage Hinge Replacement

### 3.1 ROUTINE BASIC MAINTENANCE

#### 3.1.1 ROTARY PUMP OIL LEVEL

Check the oil level in the sight glass on the pump. The minimum oil level is the lower edge of the sight glass. The maximum level is 25mm below the top of the glass. When necessary top up with oil of the type specified for the particular pump in use. (i.e. Edwards no 15 for Edwards pump or Alcatel VPI for Alcatel pumps).

#### 3.1.2 AIR ADMITTANCE DRIER ASSEMBLY

If the air drier is allowed to become ineffective the pump down time of the 5200 will become longer than normal.

The assembly is mounted on the rear panel of the plinth. The colour of the desiccant in the assembly should be checked daily and if it shows signs of becoming saturated i.e. turning from blue to pink or white, it should be replaced or reactivated. To renew the desiccant:

- a. Unclip the drier from the rear of the plinth.
- b. Unscrew the large knurled retaining ring from one end of the assembly and remove the end cap. Remove the filter washer beneath it. The desiccant can now be poured out and either dried or discarded. Remove the remaining filter washer and the perforated metal support.
- c. Clean the parts by washing in a suitable solvent, e.g. liquid detergent, after which each part must be thoroughly rinsed and dried.
- d. Replace the perforated metal support with the concave side facing away from where the desiccant will be. Cover with two filter washers (shiny side away from the desiccant). Fill the assembly with new or reactivated desiccant. Fit a filter washer with the shiny side towards the desiccant. Fit the end cap and knurled clamp ring.
- e. Refit the assembly to the rear panel of the plinth.

### 3.2 6-MONTHLY MAINTENANCE

#### 3.2.1 CHANGING THE ROTARY PUMP OIL

It is recommended that the rotary pump oil is changed after the first 100 hours operation and thereafter at 6-monthly intervals, with intermediate checks on the oil level.

To change the oil:

- a. Select CHAMBER VACUUM vent and wait for the rotary pump to stop. Unplug cable number 6 to remove turbo pump power.
- b. Place a container of not less than 2.5 litres capacity under the oil drain plug at the bottom of the pump. Remove the plug and drain the oil.
- c. Remove the two screws holding the vacuum hose manifold to the rotary pump. Break the vacuum seal by carefully moving the hose sideways. Remove the pipe manifold, 0

ring carrier and O ring. With the rotary pump outlet partly sealed (e.g. by the thumb) select CHAMBER pump. The rotary pump will start and eject the remaining oil. Continue until the pump is empty.

**WARNING:** During this process oil will be ejected from the pump with great force. Avoid risk of inhalation or eye contamination.

- d. Pour a small quantity (e.g. 0.75 litres) of clean oil down the rotary pump inlet. Allow this oil to be pumped away before switching off the pump by selecting CHAMBER vent.
- e. Replace the drain plug and remove the filler plug at the top of the pump. Refill the pump with the oil recommended in the pump manufacturers handbook.
- f. Replace the filler plug and run the pump for about 30 seconds with the inlet open to air. Refit the O ring carrier, O ring, pump hose manifold clamp ring and 2 screws.
- g. Connect the turbo pump cable, number 6, and pump down the system.

### 3.2.2 ROTARY PUMP BELT TENSION

The direct drive rotary pump normally supplied does not have a drive belt. If a belt drive pump has been fitted the correct belt tension should be checked according to the pump manufacturers instructions.

### 3.2.3 CHANGING THE TURBO PUMP OIL

The turbomolecular pump bearing oil should be changed every 6 months using the instructions supplied by the pump manufacturer. It is very strongly recommended that this work is carried out by a Cambridge approved service engineer.

## 3.3 COLUMN SERVICING

The periods between cleaning will depend on the frequency of use, type of specimens and environmental conditions, etc. As a general rule, if the required performance can be achieved then leave well alone. Only if the resolution deteriorates and cannot be improved by adjustment is cleaning necessary. The degree of cleaning needed can only be determined by inspecting the column components.

Routine cleaning consists of cleaning the grid and anode and inserting clean apertures. If this does not restore the performance, then the whole column must be dismantled and cleaned. The extent of this depends on the severity of contamination, which can only be found by inspecting the column as it is dismantled.

### 3.3.1 CLEANING RECOMMENDATIONS

All swabs should be made from clean, absorbent, lint free material which will leave no dust or particles on the cleaned

surfaces. A low power binocular microscope is useful to enable dust particles to be seen. All cleaned components must be kept covered to protect them from dust in the atmosphere. Great care must be taken when handling any part of the column since all parts are machined to close tolerance. Nylon gloves must be worn when handling all polepieces, gun parts and other components exposed to the electron beam.

An aerosol can of freon or similar compressed gas is very useful for blowing the dust off each component as it is replaced in the column. Commercial compressed air should not be used as it contains oil vapour.

**WARNING: DO NOT ALLOW ANY LIQUID TO COME INTO CONTACT WITH THE GUN CERAMIC. IT IS ESSENTIAL TO AVOID MAGNETISING ANY PART OF THE COLUMN. THE STEEL USED FOR COMPONENTS IN THE MAGNETIC CIRCUITS (I.E. POLEPIECES) IS OF A VERY SOFT TYPE AND WILL RUST VERY QUICKLY IF LEFT IN THE ATMOSPHERE. WHERE POSSIBLE THE COLUMN SHOULD BE KEPT UNDER VACUUM.**

- a. STEEL AND STAINLESS STEEL, COPPER AND HIDURAL COMPONENTS. (Hidural is the coppery looking metal.)

In normal cleaning, wash in a 10% solution of quadralene in water, followed by distilled water. Rinse with propanol or other suitable solvent and dry with a hot air blower. In the case of severe contamination the initial cleaning in quadralene should take place in an ultrasonic cleaner for at least 20 minutes. Components which are severely contaminated may be cleaned with Hyprez (see next section).

- b. ALUMINIUM AND ALUMINIUM ALLOY COMPONENTS.

These components may be cleaned with Hyprez diamond compound grade 1-W-47 or, in the case of severe contamination, grade 4-W-47. Wash off all Hyprez with Arklone, methanol or propanol, preferably in an ultrasonic cleaner, and dry off using a hot air blower.

DO NOT USE QUADRALENE ON ANY COMPONENTS CONTAINING ALUMINIUM.

- c. MOLYBDENUM SPRAY APERTURES AND PLATINUM FINAL APERTURES.

These may be flashed by electrically heating the apertures in a molybdenum or platinum boat, in a vacuum, to white heat. They should be held at this temperature for a few minutes before being allowed to cool. Alternatively, spray apertures can be cleaned using method B.

d. MU METAL

These components should not be cleaned except for the removal of dust. It is important that the metal is not strained or dropped as this will reduce its effectiveness as a magnetic screen.

e. O RINGS

O rings may be cleaned with, but not soaked in, Arklone, methanol or propanol. The use of O ring grease is not recommended on any O rings, but a small amount may be used, if necessary, on moving seals e.g. in the specimen stage and aperture changer micrometers. The grease must be applied with a lint free tissue to avoid contamination with natural oils, using only enough grease to just put a shine on the O ring. Apiezon L grease is recommended.

f. O RING GROOVES AND FACES

Very fine abrasive paper may be used sparingly to remove any scratches. Wash any surfaces treated in this way in the appropriate solvents for the particular material.

**WARNING: ANY COMPONENT WITH O RING GROOVES AND/OR MATING SURFACES, AND WHICH IS TO BE ULTRASONICALLY CLEANED, SHOULD BE PLACED IN THE CLEANER TANK IN SUCH A WAY THAT THE GROOVE OR FACE DOES NOT TOUCH THE WALLS OF THE TANK, OR ANY OTHER COMPONENTS.**

### 3.3.2 ROUTINE COLUMN CLEANING (fig 3.2)

The numbers quoted thus (23) refer to the identification markers in figure 3.2.

**WARNING:** Certain screws (items B, D, E, H, I and J in figure 3.6) in the column are connected with the electron optical alignment of the column. If these screws are touched, column alignment will be affected, leading to time consuming realignment. Indiscriminate column dismantling should be avoided.

The first stage of column cleaning consists of cleaning the gun components and the final apertures. The method of doing this is:

- a. Vent the column and chamber to air.
- b. Open the gun.
- c. Loosen the three filament assembly clamp screws (1) and remove the filament assembly (2).
- d. Lift out the anode (3). Take care, the anode is a very good fit on the top of the alignment coils (4).



- e. Remove the four M3 screws holding the aperture changer (15) into the column, and remove this assembly from the column.
  - f. Close the gun while the components removed are cleaned. Take the filament assembly, anode and aperture changer to a clean area for cleaning.
  - g. Loosen the two screws (B) clamping the filament holder into the grid and separate the two components. (figure 3.3).
  - h. Loosen the four screws (C) holding the filament into its carrier and remove the filament (figure 3.3)
  - i. Remove the three screws holding the aperture clamp plate from the aperture blade. Lift off the clamp plate and remove the apertures from the carrier (figure 3.4).
  - j. Clean the grid and anode by method A above.
  - k. Clean the aperture blade and aperture clamp plate by method B above.
  - l. Clean the apertures by method C, or use new ones.
  - m. Replace the apertures in the aperture blade, fit the clamp plate and screws.
- Note:** The apertures have one flat side, the other side being funnel shaped. The apertures must be mounted in the blade with the flat side upwards when the aperture changer is mounted in the column. Clean the O ring on the aperture changer, put it in the column and replace the four screws.
- n. Put the anode on top of the alignment coil making sure that it is correctly seated onto its location boss. The anode should sit level on the alignment coil and be free to rotate, but not be loose.
  - o. If necessary change the filament, centre it in the grid and replace the assembly in the gun (as detailed in section 3.3.7).
  - p. Pump down the column and chamber and obtain a picture as detailed in chapters 1 and 2.

### 3.3.3 BASIC COLUMN DISMANTLING, CLEANING AND REASSEMBLY (fig 3.2)

If the routine column cleaning of section 3.3.2 is not sufficient, the second stage of column cleaning is detailed below.

- a. Remove and clean the filament assembly, grid and final apertures as detailed in 3.3.2.
- b. Mounted in the top of the anode section, under the anode, is the gun alignment coil (4). Remove the three M4

screws holding it down. Using the special tool, carefully lift the coil assembly out of the anode section VERTICALLY. (It has an O Ring seal on its lower end). The assembly is wired to the electronics by a cable entering the back of the anode section. As the assembly is lifted, ease the cable into the anode section. As soon as the socket connecting the cable to the coil is visible, disconnect it. Leave the cable laying in the anode section.

- c. Under the gun alignment coil is the anti contamination collar (5 and 6). Using a long hex socket driver, remove the three M3 screws holding it down. Using the tool provided lift the assembly out of the anode section. Invert the assembly and remove the O ring. Remove the two M2 screws and separate the inner (5) and outer (6) sections. Remove the two M2 screws from the top of the inner section and lift off the top "aperture".
- d. Protruding from the top of the lens is the lens liner tube (7). Using the special tool lift out the liner tube. Remove the top aperture clamp and top aperture (8). Remove the bottom aperture clamp and aperture (9). Close the gun.
- e. Carefully clean all components using cleaning recommendation A or B, ensuring that all traces of contamination and discoloration are removed from all components. This can be a time consuming task, but it must be done very carefully.
- f. Wash all parts in Arklone, methanol or ethanol in an ultrasonic cleaner. Visually check that all traces of cleaning materials are removed. Wash again in clean solvents. Now wash again in more clean solvent.
- g. Put a spray aperture in the top of the liner tube (the end with the pumping holes) and replace top aperture clamp. Fit the other spray aperture and the long nosed clamp (10) in the bottom of the liner tube. Store the assembly in a clean plastic bag or wrapped in clean tissue.
- h. Fit the top "aperture" to the anti contamination assembly inner section with two M2 screws. Fit the inner section (5) in the outer section (6) with two M2 screws. Clean the O ring and refit to the bottom of the assembly. Store the assembly in a clean environment.
- i. Check the bore of the gun alignment coils (4) are clean. Clean and fit the two O rings.
- j. Unwrap the lens liner tube and replace it in the column. Check that it is fully down in the lens. (The top two pumping holes should be just above the lens top plate with the lower pumping holes not visible).
- k. Check the O ring in the bottom of the anti contamination assembly. Fit the three M4 screws into the bottom flange and, using the special tool, carefully lower the assembly

into the column. Rotate the assembly until the screws are felt to line up with their holes. Tighten the screws.

- l. Check the gun align coil and its two O rings for dust. Connect cable 87 to the coil. Fit the three M4 screws into the flange. Using the special tool, carefully lower the coil VERTICALLY into the anode section. (Remember there is an O ring on the bottom of the coil which must enter into the top of the anti contamination assembly). Rotate the assembly until the screws are felt to line up. Tighten the screws.
- m. Refit the filament assembly, grid and aperture changer, as detailed in section 3.3.2.
- n. Pump down the column and chamber. Obtain an image as described in chapters 1 and 2.

#### 3.3.4 COMPLETE COLUMN BREAKDOWN (fig 3.2 and fig 3.6)

The numbers refer to figure 3.2, the letters to figure 3.6)

If the cleaning detailed in section 3.3.3 is insufficient then a complete column breakdown is required.

- a. Remove the gun hinge pin and earth strap. Remove the gun from the top of the column.
- b. Remove the four M4 screws holding the top of the pumping pipe to the anode section. Push the pipe backwards so that it just clears the anode section when it is lifted off. If you have an ion pump fitted, remove the four 6-32 UNC screws holding the magnet to the pump body. Remove the magnet. Note that these screws are a different size from all others on the S200 and must not be interchanged with any other screws from the machine.
- c. Remove the filament assembly, anode, gun align coil, final aperture changer, anticontamination assembly and lens liner tube as detailed in sections 3.3.2 and 3.3.3.
- d. Halfway down the column is a flat 25mm wide dark coloured metal band secured by a single screw (16). Loosen (do not remove) the screw, open the band slightly and slide it up the column a little way. This will reveal six M4 caphead screws (A). Remove these using the hexkey provided. If an ion pump is fitted you must support the anode section when removing the last of these screws. Lift off the anode section.
- e. Lift off the top trim ring from the mumetal shield. Remove the column isolation valve if fitted. Remove the vertical mumetal shield.
- f. Disconnect the six "in-line" connectors at the rear of the condenser lens. Check that the six M4 screws (I) housed in the recesses round the bottom of the condenser lens are tight. Loosen (do not remove) the four M3 grub screws (H) that are housed horizontally in the side of the large flange at the bottom of the condenser lens. Remove the

six M4 screws (C) (the outer ring, not the ones in the recesses) securing the condenser lens and lift off the lens. Store it laying on its side, standing it on end may damage the sealing faces.

- g. Unscrew the 3 screws (13) and remove the scan coil assembly. (The screws are deeply recessed into the scan coil pot). Remove the cable clamp from the scan coil pot. Unplug the connectors from the scan coil PCB. Remove the 2 screws which hold the scan coil onto the scan coil pot. Separate the two components. Remove the O ring clamp plate from the top of the scan coil. Remove the top and bottom O rings.

- h. Unscrew the 4 fixing screws (F) and lift off the final lens.

**Note:** The final lens fixing screws are the four situated on the outer ring on the lens flange. The screws on the inner ring hold the two parts of the final lens together. If they are loosened the lens alignment may be disturbed requiring extensive column alignment.

- i. Clean the scan coil and the scan coil pot using cleaning recommendation B.
- j. Clean the top and bottom faces of the condenser lens with solvent. Do not remove the top and bottom end plates of the lens if it is absolutely necessary i.e. to repair a vacuum leak in the O rings under the lens plates. If the plates are removed a complete column alignment will be required.
- k. Lay the final lens on the bench with the end that is normally in the chamber uppermost. Remove the lens protection plate. Inspect the bore of the lens. It may, if necessary, be wiped with a clean, lint free tissue soaked in solvent.

Only if the lens bore is severely contaminated may it be polished with hyprez grade 1-W-47 on a piece of soft balsa wood or other soft lapping stick. All traces of cleaning compound must be removed using a solvent. Take great care not to scratch the lens bore or the lens face over an area of about 2cm radius about the bore.

**IT IS STRONGLY RECOMMENDED THAT POLISHING THE FINAL LENS BORE IS ONLY DONE BY AN ENGINEER WHO HAS BEEN TRAINED AT CAMBRIDGE.**

If the lens bore is so badly contaminated that the upper and lower polepieces of the lens must be separated to allow efficient cleaning, the procedure is now given. It requires that the entire column be realigned after assembly, the method for this being given in section 3.3.8.

- l. Remove the 4 screws (E) holding the polepieces together. Stand the lens with the lens bore uppermost and lift off the lens plate vertically taking care not to twist or rock

the polepieces in a manner which might disturb the four alignment blocks.

**DO NOT DISTURB THE FOUR ALIGNMENT BLOCKS WHICH LOCATE THE LENS PLATE ONTO THE BODY. THEY CAN ONLY BE RESET IN CAMBRIDGE.**

- m. Clean the lens polepiece with hyprez 1-W-47 as described above.
- n. Reassemble the final lens and replace it on the column. Remember to check the O ring.
- o. Fit the O rings (top and bottom) and O ring clamp to the scan coil. Fit the scan coil pot on to the scan coil (with an O ring between them), checking that the location pins in the interface locate correctly. Connect the cables to the scan coil PCB, fit the cable clamp, put the scan coil assembly in the final lens (check the location pins) and screw it down. Route the cable through the column wall.
- p. Check the O ring on top of the scan coil pot, put the condenser lens on and screw it down.
- q. Fit the mumetal shield and top trim ring.
- r. Fit the anode section and screw it down.
- s. Fit the gun pumping tube. (Remember the O ring.)
- t. Fit the ion pump magnet and column isolation valve (if applicable).
- u. Fit the gun. Replace the gun hinge pin AND THE EARTH STRAP.
- v. Fit the rest of the column components as described in section 3.3.2 and 3.3.3.
- w. Check the column alignment and adjust if necessary (see 3.3.8).

### 3.3.5 CLEANING THE SPECIMEN CHAMBER.

Although it is possible to clean the chamber with the column intact, it is easier to do so with the column removed. It is therefore recommended that the chamber is cleaned whenever the column is removed for servicing.

- a. Remove the specimen stage.
- b. Unscrew the four M4 screws and remove the collector system mounting plate from the rear of the chamber.
- c. Clean all interior faces of the chamber with a lint free tissue soaked in Arklone, methanol or ethanol. After cleaning, dry the interior of the chamber with a hot air blower.

- d. If the contamination is severe, all options fitted to the chamber should be removed and cleaned separately. When replacing the options, clean all O rings and check them for damage.
- e. Refit the collector system and specimen stage.

### 3.3.6 REPLACING THE SCINTILLATOR AND LIGHT GUIDE (fig 3.5) and (fig 3.5a)

- a. Unscrew the 4 screws and remove the collector system mounting plate from the rear of the chamber.
- b. Slide back the two protective sleeves A and B. Loosen the three screws C, D and E on the backplate and remove the three wires from the sockets.
- c. Loosen the nylon screw F and remove the lightguide assembly from the backplate.
- d. Remove the front mesh G and focusing aperture H from the end of the collector cage. Loosen screw I and carefully slide the lightguide from the cage assembly. Take great care not to damage the wire connected to the brass ring on the end of the scintillator. If the wire breaks away from the brass ring it cannot be resoldered as this damages the lightguide.
- e. Clean all parts of the collector system (except the lightguide) with Arklone, methanol or propanol. Insert a new light guide into the collector cage, taking care not to touch the scintillator with the fingers (or anything else).
- f. Fit the front mesh and focusing aperture. Adjust the position of the collector cage assembly on the lightguide so that dimension J is 12mm as shown. Tighten screw I.
- g. Fit the lightguide assembly into the backplate and tighten screw F.
- h. Reconnect the three leads, not forgetting to fit the protective covers.
- i. Refit the collector backplate to the chamber.

### 3.3.7 CHANGING THE FILAMENT (TUNGSTEN GUN) (figure 3.3)

For details of the LaB<sub>6</sub> cathode change see section 4.2

- a. Vent the column
- b. Open the gun
- c. Loosen the three filament assembly clamp screws (A) and remove the filament assembly. Close the gun.
- d. If a spare filament assembly is available, fit it. If not replace the filament as follows.

Filament Replacement

- e. Loosen the filament holder clamp screws (B) and remove the filament holder from the grid.
- f. Loosen the filament alignment screws (C) and remove the filament.
- g. Clean the grid, anode and filament holder as required.
- h. Refit the filament holder into the grid and tighten the clamp screws (B).
- i. Put a new filament into the holder and gently tighten the filament alignment screws (C).
- j. Turn the assembly over, look at the filament through the grid hole and move the filament to the centre of the grid using the filament alignment screws (C). For the best instrument performance this must be done as accurately as possible. A low power microscope or a watchmakers eyepiece is very useful in allowing the alignment to be seen better.
- k. Using the three height setting screws (D), set the filament to be 0.5mm behind the front face of the filament.
- l. Refit the filament assembly to the column, pump down and obtain a picture as detailed in chapters 1 and 2.

### 3.3.8 ALIGNING THE COLUMN. (figure 3.6)

Various stages of column alignment may be needed, as indicated by the following criteria.

- a. If the C1/C2 lens assembly has not been lifted off the final lens (C3), and provided the four sets of column clamping screws A, C, E and I (6 in each set) and the three sets of column alignment screws B, D, G and H (4 in each set) have all remained untouched, and therefore are not loose, then no alignment is necessary. It may be a good idea to check the alignment (steps 70 onward).
- b. If only the 6 column to final lens clamping screws (C) and the 4 column to final lens clamping screws (F) have been loosened, then steps 7 to 37 and steps 70 onward should be done.
- c. If any of the other alignment or clamping screws have been loosened then the complete procedure will be required.

**Note:** Always leave all alignment screws tight at the end of each stage of alignment.

#### Tools required

- a. 12 M1.5 hexagon wrenches to fit M3 grub screws
- b. A felt tip pen which can write clearly on the display
- c. Short ended M3 hexagon wrench part no 716727
- d. Ball ended hexagon driver part no 429008

#### Other requirements

A well maintained specimen stage capable of moving in 1 micron increments.

A filament which is well centred in the grid.

Shift coils (cable 198 on the EO PCB) unplugged.

A digital multimeter, set to read volts, connected across testpoints TP15 and TP16 on the EO board.

#### Initial Screw Adjustments

**CAUTION** Do not perform steps 1 and 2 unless C1 and C2 poleplates have been removed or loosened.

- 1 Loosen the 6 vertical M3 cap head screws (J) accessed through the holes in the anode section mounting flange.
- 2 Loosen the 6 vertical M4 cap head screws (A) on the anode flange using the short hex key.
- 3 Release the 6 vertical M4 cap head screws (C) next to the slots and the 6 vertical M4 cap head screws (I) in the slots in C1/C2 body.
- 4 Set the 4 horizontal M3 grub screws (H) in the top of C3 to centre travel (not the ones in the clearance holes).

**CAUTION** Do not perform steps 5 and 6 unless C1 or C2 poleplates have been removed or loosened.

- 5 Set the 4 horizontal M3 grub screws (D) down the clearance holes in the top of C3 to centre travel.
- 6 Set the 4 horizontal M3 grub screws (B) in the tapped holes in the top of C1/2 to centre travel.

#### Start of alignment procedure

- 7 Set the Optibeam selector switch (the small DIL switch) on the EO PCB so that the pointer is towards the A on the switch. This sets the optibeam system to the align mode.
- 8 Set C3 reversing switch (on the EO board) to normal.
- 9 Set all 4 gun align controls to centre.
- 10 Turn fine acceleration voltage clockwise.
- 11 Select 20 KV
- 12 Adjust the fine resolution control so that the DVM on TP15 and TP16 reads 300 mV.
- 13 Set Resolution coarse to 8.



- 14 If you have beam down the column set the filament current to first peak. If not, go to step 17.
- 15 Adjust the specimen to 10mm working distance and 45 degree tilt.
- 16 Go to step 25.
- 17 If no beam is visible select emission image.
- 18 Adjust aperture centring and gun align to obtain a beam. If a beam cannot be found go to step 22.
- 19 Reset gun align as near centre of travel as possible to still maintain a beam.
- 20 Adjust final aperture.
- 21 Go to step 25.
- 22 Reset gun align to centre travel
- 23 Adjust C1 poleplate screws (B) to find the beam
- 24 Go to step 25

#### Finding C3 axis

During these procedures it will help if you put a small label on the hex keys used to adjust screws (D) so they are not confused with those in screws (H).

- 25 Using focus wobble, centre the final aperture.
- 26 Centre an easily recognisable feature on the screen. Pick something that can be recognised at a magnification of 100X and has 1 micron detail on it at high magnifications.
- 27 Focus accurately coming from the anticlockwise end of the medium focus control.
- 28 Centre the aperture using focus wobble.
- 29 Repeat from 26 if the reference feature is no longer visible.
- 30 Mark the position of the reference feature on the screen. Reverse C3 current using the reversal switch on the EO board.
- 31 Mark the new position of the feature on the screen. If it has moved less than 2 micron go to 35.
- 32 Using the stage, move the feature half way back to its original position.

- 33 Select focus wobble. Adjust the column alignment screws (H) and the aperture position so that you have simultaneously
  - a. zero image shift with focus wobble and
  - b. the reference feature in the centre of the screen
- 34 Repeat from step 30 until condition in 31 is met.
- 35 Switch the C3 current back to normal (not reversed).
- 36 Do not move the aperture or stage from now on. If C1 and C2 poleplate clamping has been loosened, go to step 38.
- 37 If C1 and C2 poleplates have remained clamped, alignment is complete. Check alignment as in step 70 onward.

Adjusting C2 axis to coincide with C3 axis.
- 38 Set lower gun align knobs central. Adjust upper gun align knobs for max brightness.
- 39 Set Resolution fine so DVM on C1 reads about 500mV.
- 40 Starting with C2 high, reduce the resolution coarse control adjusting C2 poleplate screws (D) to keep the beam going down the column. If at any time you get uncontrollable beam cut off go to 49.
- 41 At a resolution of 4 reset upper and lower gun align controls for maximum brightness.
- 42 Focus C3
- 43 Adjust C2 poleplate (D) to achieve zero shift with focus wobble.
- 44 Set resolution coarse to 9.
- 45 Refocus C3
- 46 Adjust column adjusting screws (H) for zero shift with focus wobble.
- 47 Set resolution coarse to 4.
- 48 Repeat from step 42. After the third time through go to 49.

Getting C1 onto the axis of C2 and C3.
- 49 Set to gun align mode.

- 50 Set resolution coarse to 4.
- 51 Set resolution fine so DVM reads 800 mV.
- 52 Use Shift to centralise source image in aperture image.
- 53 Use Tilt to centre gun align image.
- 54 Reduce C1 current (resolution fine), adjusting C1 poleplates (B) to keep the aperture like dark image from encroaching on the bright source image. The adjustment will at the same time recentre the gun align image on the screen, but:
- 55 If further adjustment is needed use the tilt controls, these will have an increasing effect as the lens currents are lowered.
- 56 Continue adjustments until the source image collapses and expands as you go through the final aperture with no cutoffs.
- 57 Staying in the gun align mode, switch the lenses back to optibeam.
- 58 Adjust resolution coarse. If cut off occurs at low resolution numbers, adjust shift controls to centralise the aperture image and the tilt controls to centre the bright part of the emission profile in the aperture.
- 59 Select alignment mode again.
- 60 Repeat 50 to 58 inclusive.
- 61 Repeat 39 to 48 inclusive.
- 62 Set resolution coarse to 4 and fine so the DVM reads 800 mV.
- 63 Obtain an image of an interesting feature in the centre of the screen at 10KX.
- 64 Swing resolution fine control over its full range. If the feature moves more than 2 microns go to 68.
- 65 Repeat 64 until the condition is met.
- 66 Repeat 63 to 65.
- 67 Test column alignment as in 70 onward.
- 68 Focus C3. Move features halfway back to the centre using C1 poleplate adjustment screws (B).
- 69 Repeat from 63.

## Testing column alignment

- 70 Check that C3 reversal switch is set to normal and the alignment switch is set to optibeam.
- 71 Set resolution coarse to 11.
- 72 Obtain an image with a recognisable feature in the centre of the screen at 10KX.
- 73 Adjust the resolution controls. The feature must stay on the screen at all settings of the resolution controls.
- 74 Repeat 71 to 73 at other KV settings. The feature should stay on screen at all KV settings.

**Note:** There will be some image shift as KV is changed so the feature will have to be recentred on the display at each KV.

- 75 If column alignment test is met go to 79.
- 76 If specification is not met try step 58.
- 77 If it is still not met try 49 to 58.
- 78 If it is still not met start again.

## Locking the column together

- 79 Obtain an image of a recognisable feature at 10KX at a resolution of 5.
- 80 Carefully tighten all four rings (A, C, F and J) of 6 clamping screws a little at a time in an order such that the image does not shift.
- 81 Centre the final aperture using focus wobble.
- 82 Check column alignment using steps 70 onward.
- 83 Check that the lens offset controls are correctly set. Set optibeam to align with the DIL switch on the EO PCB. Set resolution coarse to 1 and the fine control fully clockwise. Select OPTIBEAM normal. Adjust RV8 on the EO PCB such that the image of the aperture is at its smallest (the lens crossover is in the aperture). Now select aperture not normal (switch released) and adjust RV7 so the image of the aperture is again at its smallest. Select optibeam normal with the DIL switch on the EO PCB.

### 3.4 REPLACING THE SWING DOOR STAGE (fig 3.5) and (fig 3.5a)

1. Obtain the swing door stage and the 4 securing screws for the hinge block.
2. Vent the chamber and remove whatever is mounted on the front of it.
3. Set the stage Z control to its lowest setting. Set the Y control to a low reading (25mm) and the X control to centre (50mm).
4. Offer up the stage to the chamber, engage the hinge block location pin in the slot in the chamber, and attach the block loosely to the chamber with the four M5 screws (see figure 3.5). Engage the stage door fastener with the hook on the side of the chamber. Tighten the screws in the hinge block.
5. Check that as the stage door is opened and closed the location pin on the right side of the door fits smoothly into the slot in the chamber. If it does not slacken the rear facing grub screw in the rear of the hinge and adjust the right facing cap head screw at the back of the hinge clockwise to raise the door, anticlockwise to lower it. Lock the grub screw.
6. Slacken the four hinge block screws half a turn. Pump down the chamber. Tighten the hinge block securing screws.

### 3.5 Changing the Anode

The acceleration voltage range is split into two, above 4KV and below 3KV. This change is selected by the ACCELERATION VOLTAGE High switch. To achieve a better gun geometry and brightness and hence better instrument performance the anode should be lifted towards the grid when the low KV range is selected. To do this

1. Admit air to the gun. Open the gun.
2. Lift out the anode.
3. Fit the anode spacer on the top of the gun align coils. Take care, this is a high precision fit.
4. Fit the anode back on top of the spacer. This is also a good fit.
5. Select Low ACCELERATION VOLTAGE range (see 2.2.2).
6. Close the gun, pump down and obtain an image.

The S200 should not be used on the High ACCELERATION VOLTAGE range with the anode spacer fitted.

### 3.6 Looking for Vacuum Leaks

A few simple rules

1. Before looking for a vacuum leak give the vacuum system time to pump. If you have just changed a specimen in a very humid atmosphere the pumpdown may take longer. If the specimen is wet it may take several minutes (up to 1 hour with a very large, very wet specimen has been known.) If the stage has been left open for long periods it will take longer to pump. If the column or chamber have been washed with solvents then it may take several hours to achieve a good vacuum.

2. Vacuum leaks rarely happen, they are often caused. If you have just done anything to the column or chamber then that is the most likely cause of the leak. If the specimen has just been changed, check the stage door O ring. Similarly if the filament has been changed check the gun O ring.
3. The most frequent cause of leaks is dust or fibres on O rings. O rings may be cleaned with a fluff free tissue dampened with Arklone, methanol or ethanol. O ring grease should only be used on sliding seals in the aperture changer and specimen stage. Grease traps fibres and may cause leaks.
4. Do not use any metal tools to remove O rings from their grooves. A small scratch in the bottom of a groove causes a big leak.
5. If the pump down is slow, check the air admittance drier and renew the desiccant before looking for a leak.

#### Detecting a leak.

The first requirement is a method of measuring the vacuum. Some methods, starting from the simplest, are

1. Use the built in vacuum indicator, this is not really sensitive enough for leak hunting.
2. Add a more sensitive meter to the internal vacuum gauge. This can be done in two ways.
  - a. To measure the chamber pressure using the built in Penning gauge, connect a voltmeter from the junction of R2 and R3 on the vac aux PCB 852628 to ground.
  - b. To measure the vacuum more accurately in the column when an ion pump is fitted, connect a voltmeter from testpoint ATE 3/3 on the ion pump control PCB 852626 to ground.
3. A still better method is to disconnect the Penning gauge from the vac interlock PCB and connect to a commercially available Penning gauge box, e.g. a Penning 8 gauge box available from Edwards High Vacuum Ltd.
4. The best, and most expensive method is to fit a commercial leak detector system to the chamber, e.g. a mass spectrometer. This only proves to be required in very rare cases.

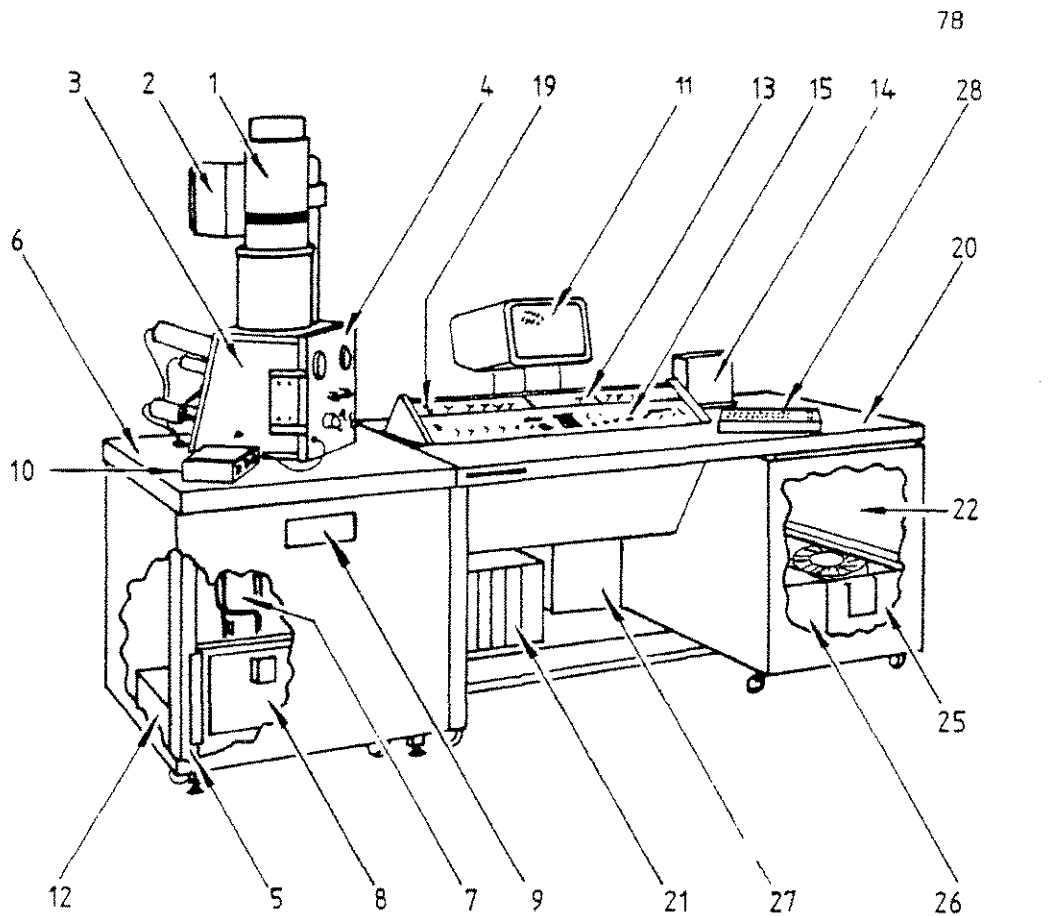
The second thing you need is to find the leak. This is done by putting some liquid or gas onto the leak. The liquid or gas used must do two things. It must quickly find its way through the leak and it must cause a reaction on the vacuum gauge.

If using a mass spectrometer leak detector the normal gas to use is helium.

If you are using the internal penning gauge as a leak detector then the normal leak detecting fluids include Arklone (trichlorotrifluoroethane) and Freon. Aerosal cans of various chloroflouroethanes, sold as dusters or propellants for spray guns are also good for leak detecting.

#### Method of finding the leak

1. Set up the vacuum measuring equipment
2. Get some leak detecting fluid
3. Put a small amount of leak detecting fluid on a place in the vacuum system that is vacuum sealed.
4. Look for some reaction on the vacuum gauge. This reaction is normally an increase in pressure but it can sometimes be a decrease in pressure. (If the fluid washes a piece of dust, or some O ring grease, into the hole causing the leak it may seal it).
5. If no leak is found, test each vacuum seal in the system in turn. Do this slowly as it may take the vacuum gauge several seconds to respond.
6. If a leak is found, take the vacuum joint apart and renew the seal.



- |   |   |
|---|---|
| 1 ELECTRON OPTICAL COLUMN                   | 15 CONTROLS PANEL                                     |
| 2 ION PUMP (OPTIONAL)                       | 16  |
| 3 SPECIMEN CHAMBER                          | 17  |
| 4 SPECIMEN STAGE                            | 18  |
| 5 PLINTH                                    | 19 POWER CONTROL                                      |
| 6 PLINTH DESK TOP                           | 20 CONSOLE DESK TOP                                   |
| 7 TURBO PUMP                                | 21 GUN EHT CRT & PM EHT                               |
| 8 VACUUM CONTROL UNIT                       | 22 OPTIONS SPACE                                      |
| 9 VACUUM SYSTEM CONTROLS                    | 23  |
| 10 SPECIMEN CURRENT AMPLIFIER<br>(OPTIONAL) | 24  |
| 11 VISUAL DISPLAY                           | 25 CAPACITOR CHASSIS<br>(POWER CAPACITOR DIODES etc.) |
| 12 TURBO CONTROLLER                         | 26 ION PUMP CONTROL UNIT<br>(OPTIONAL)                |
| 13 OPTIONS PANEL                            | 27 MAINS UNIT   |
| 14 RECORD DISPLAY                           | 28 TEXT KEYBOARD                                      |

Figure 3.1

Location of Major Items



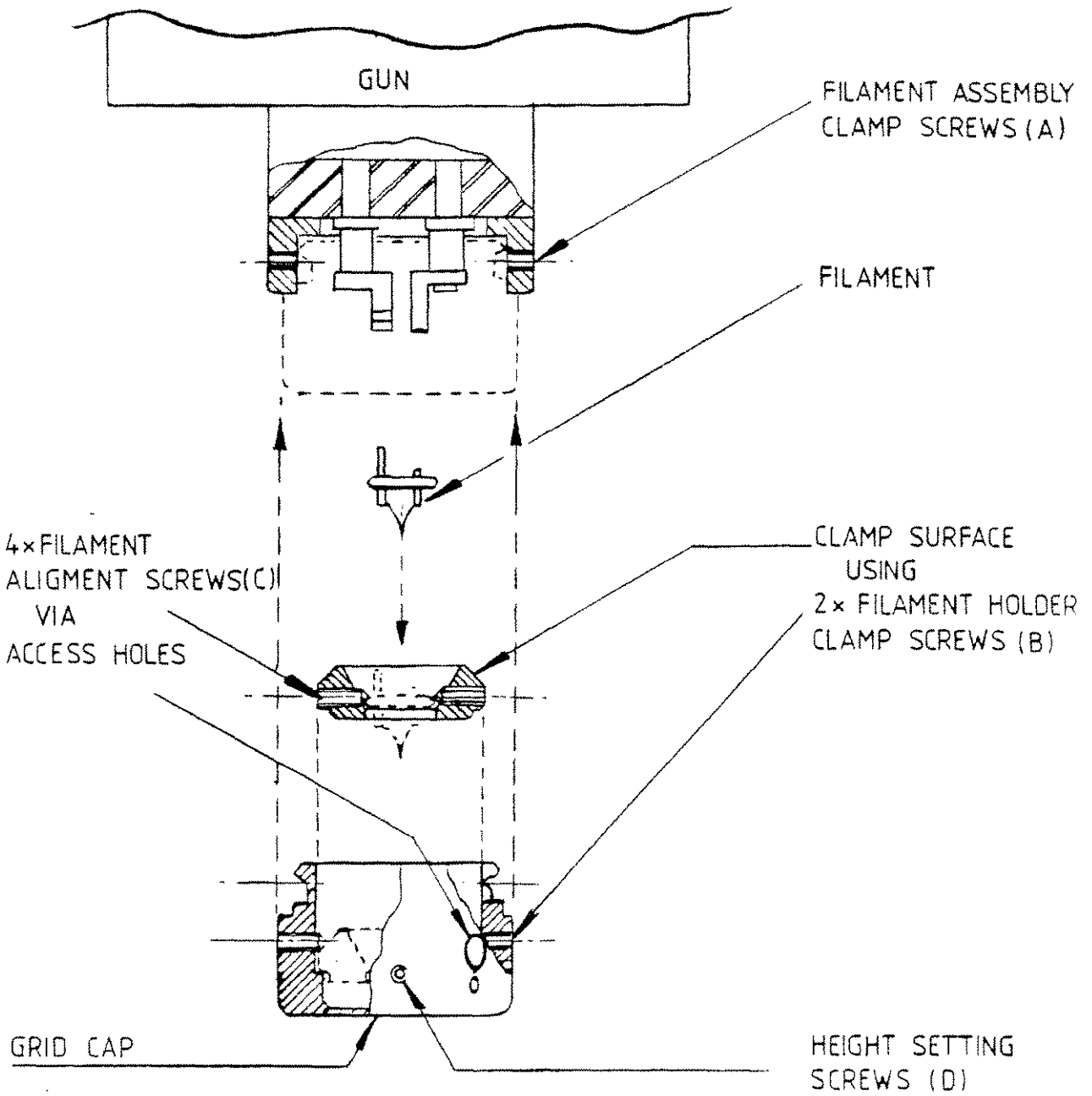


Figure 3.3  
The Firing Unit

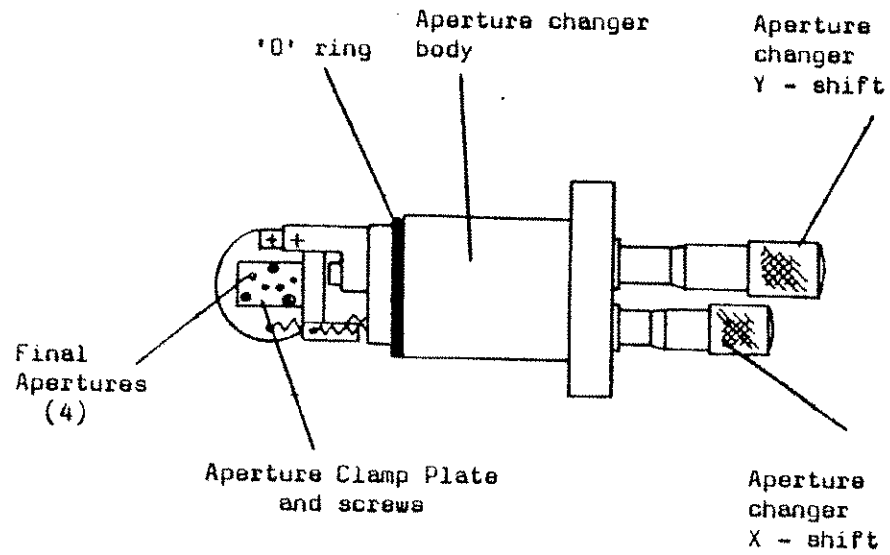


Figure 3.4

Final Aperture Changer

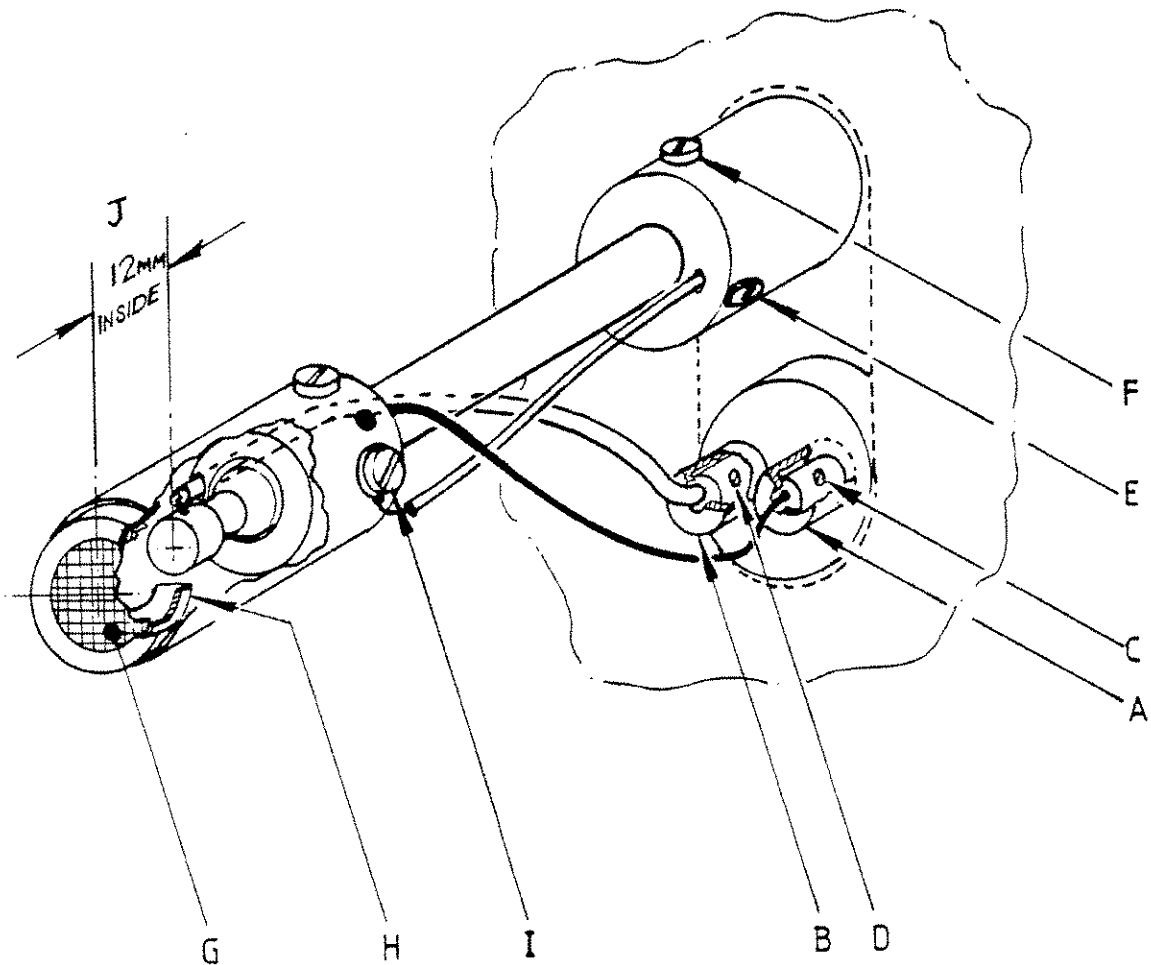
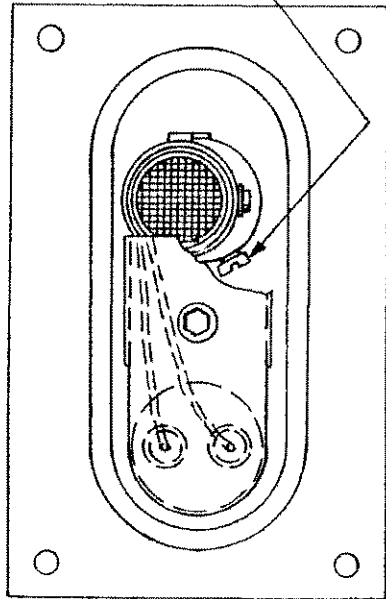


Figure 3.5

Collector System

(Shield-omitted for clarity - must be replaced)

Nylon Clamping  
Screw



Check this Dimension  
is 12mm

Mesh. Ref.

Pipe Support

Check End of Light Pipe  
is Flat. The End of the  
Light Pipe MUST Touch  
Window

Light Pipe

Window

99.0 Ref.

Mounting  
Plate

SUGGESTED ASSEMBLY SEQUENCE TO GIVE GOOD CONTACT BETWEEN END OF LIGHT PIPE AND WINDOW

Insert Light Pipe Through Pipe Support, Ensuring that it Protrudes 13mm Beyond Sealing Face of Pipe Support. Lightly Tighten Nylon Clamping Screw. Attach Pipe Support to Mounting Plate and Tighten Three Attachment Screws, Finally Tighten Nylon Clamping Screw.

**Figure 3.5a**  
**Collector System**  
**(Assembly Sequence)**

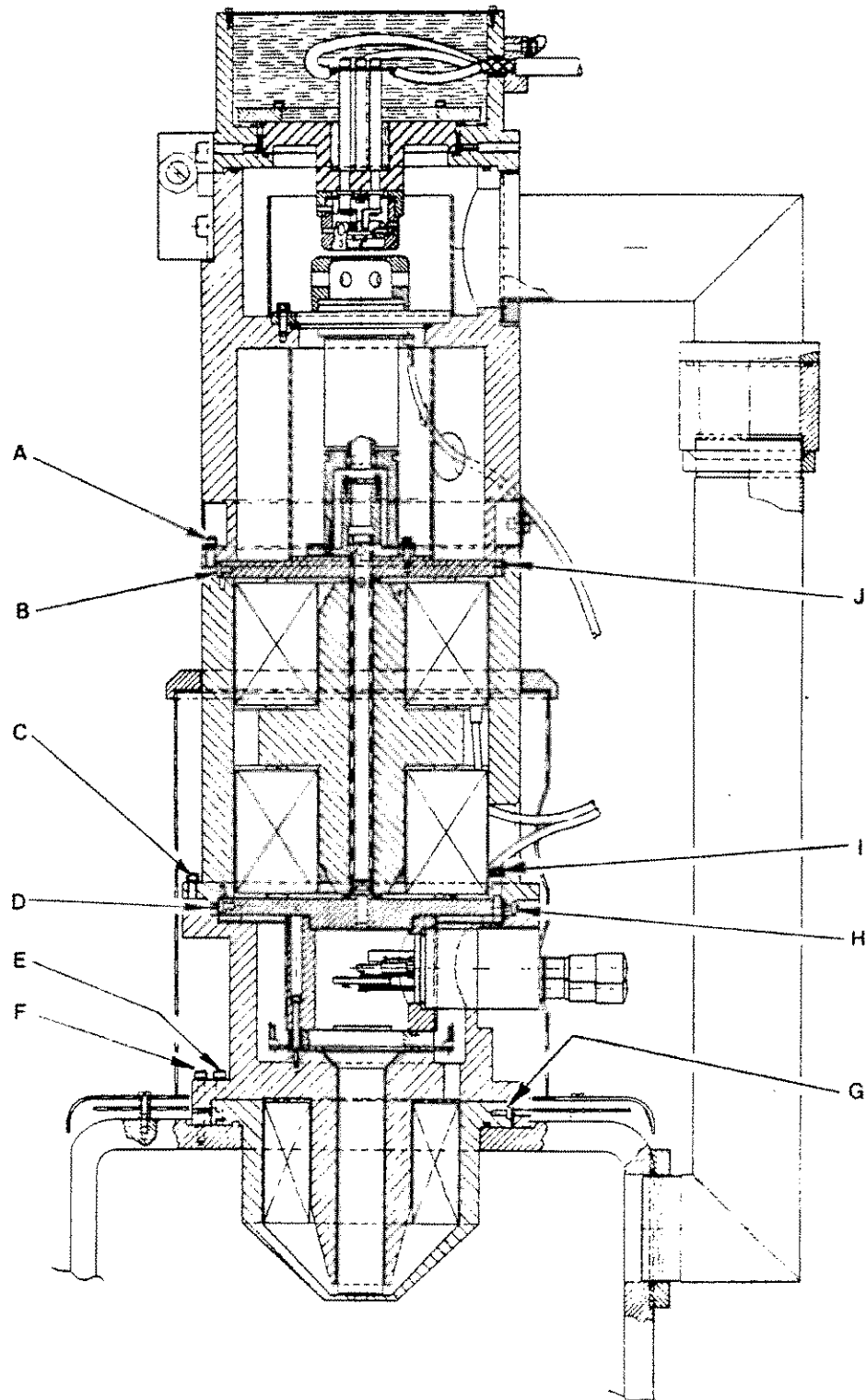


Figure 3.6

Column Alignment Screws

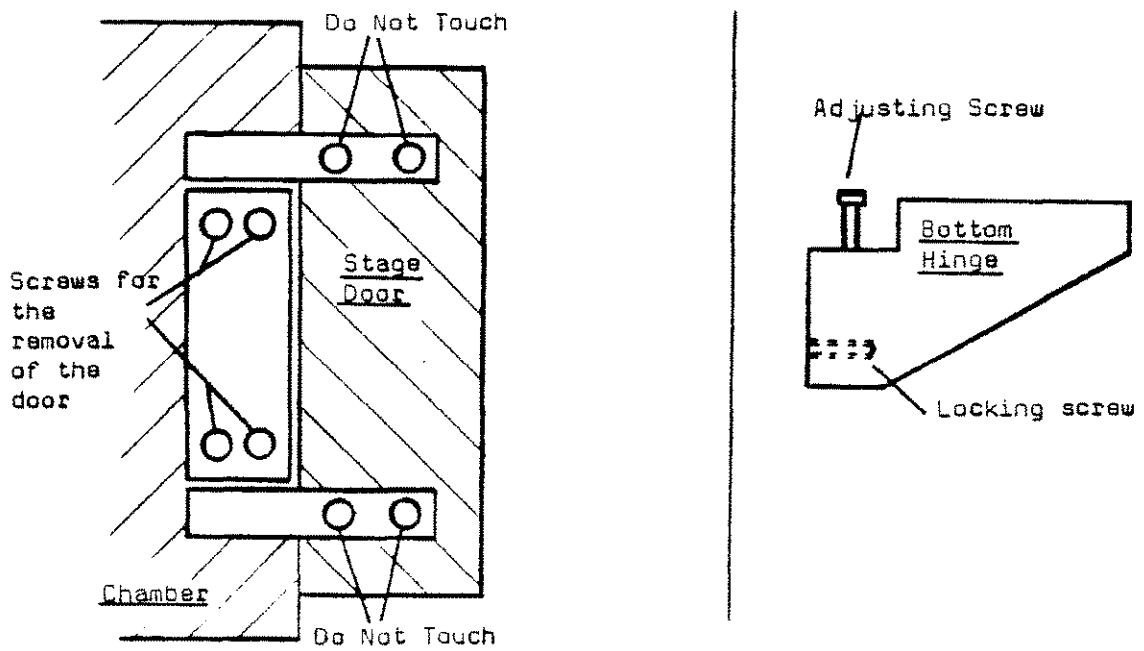


Figure 3.7  
Stage Hinge Replacement

## CHAPTER 4 OPTIONS

## CONTENTS

- 4.1 INDEPENDENT COLUMN AND PUMPING (200-ICPI) (\*)
- 4.2 LaB6 (200-LaB6)
- 4.3 X-RAY PROCESSOR (200-XPU) (\*)
- 4.4 AUTOFOCUS (200-AF5) (\*)
- 4.5 BEAM BLANKING (200-EBBU) (\*)
- 4.6 CHAMBER ISOLATION VALVE (200-CIV) (\*)
- 4.7 SPECIMEN CURRENT MONITOR (200-SCM) (\*)
- 4.8 IMAGE ANALYSIS INTERFACE (200-IAI9) (\*)
- 4.9 CAMERAS (\*)
- 4.10 FOUR ELEMENT BACKSCATTER DETECTOR (200-4BSD) (\*)
- 4.11 SCINTILLATOR BACKSCATTER DETECTOR (200-SBSD) (\*)

Sections marked (\*) are still being written.

Any sections not included in this issue of the manual will be included with the option when it is supplied.

## 4.2 LaB6

WHEN HANDLING THE LaB6 EMITTER ASSEMBLY SUITABLE CLEAN FIBRE FREE GLOVES MUST BE WORN. THE LaB6 TIP ON THE FILAMENT IS VERY FRAGILE. GREAT CARE MUST BE TAKEN NOT TO TOUCH THE TIP OR DAMAGE IT IN ANY WAY.

### 4.2.1 Installation Requirements

The LaB6 emitter assembly can be fitted to any S200 provided that it has the Independent Column Pumping and Isolation system fitted (200-ICPI). All controls used for LaB6 are fitted to the basic S200 and the EHT set and OPTIBEAM are already configured for LaB6.

### 4.2.2 Installation

1. Carefully remove the LaB6 emitter from its packing box. Using an Aerosol duster, clean any dust and loose particles from the emitter assembly.
2. Inspect the assembly for any signs of transit damage.
3. Vent the column and open the gun.
4. Loosen the three emitter assembly clamp screws and remove the tungsten emitter assembly. Store it in the box that the LaB6 emitter assembly came in.
5. Check the centralizing and height setting of the LaB6 tip in the firing unit (see Section 4.2.5).
6. Fit the LaB6 emitter assembly into the gun and tighten the emitter assembly clamp screws.
7. Close the gun. Select CHAMBER Vacuum and COLUMN Vacuum.
8. On the control unit front panel set the EMITTER control to LaB6. This resets the EHT set emission current and the OPTIBEAM system for LaB6 use.

USING THE LaB6 CATHODE WITHOUT HAVING LaB6 SELECTED ON THE CONTROL UNIT FRONT PANEL WILL SHORTEN THE WORKING LIFE OF THE CATHODE.

9. When both chamber and column have reached vacuum ready, indicated by both the chamber and column vacuum switches being illuminated, the system may be used.

LaB6 EMITTERS SHOULD NEVER BE USED IN A VACUUM WORSE THAN  $2 \times 10^{-6}$  TORR. IF ICPI IS FITTED AND COLUMN VACUUM IS SELECTED THEN THE S200 CANNOT BE USED UNTIL THIS VACUUM IS ACHIEVED, USING LaB6 WITHOUT ICPI OR WITH A VACUUM WORSE THAN  $2 \times 10^{-6}$  TORR WILL SEVERELY SHORTEN THE LaB6 EMITTER LIFE AND SHOULD NEVER BE ATTEMPTED.



#### 4.2.3 Initial Adjustment

The S200 is supplied already configured for LaB6 operation and no adjustment should be required.

The emitter assembly is supplied with an emitter fitted and adjusted. If any adjustment is thought necessary the method is described in Section 4.2.5.

#### 4.2.4 Operation

1. Install the emitter assembly as described in Section 4.2.2.
2. Select COLUMN Vacuum and wait for column vacuum ready to be achieved. This is shown by the COLUMN Vacuum switch being illuminated.

**Note:** The COLUMN Vacuum switch will be illuminated at the chamber vacuum ready pressure if COLUMN Vacuum is not selected. This indicates a vacuum which is good enough for tungsten filaments but is not good enough for LaB6 cathodes.

**LaB6 CATHODES SHOULD NEVER BE USED WITHOUT COLUMN VACUUM SELECTED AND READY.**

3. Select Column on the vacuum meter switch and check that the column vacuum is better than  $2 \times 10^{-6}$  Torr.
4. Select LaB6 on the EMITTER switch in the electron optics control group.
5. If using a new cathode perform steps 6 to 10. If the cathode has been used go to step 11.
6. Select 0.5kV ACCELERATION VOLTAGE.
7. Turn the filament control fully counter clockwise. Select BEAM on. Select EMISSION IMAGE on and centre all four GUN ALIGNMENT controls.
8. Slowly turn the Filament control clockwise, watching the column vacuum meter as you do so, taking approximately 1 minute to turn control from fully counter clockwise to  $\frac{3}{4}$  clockwise.
9. If the column pressure starts to rise, stop turning the filament control. Do not increase the filament control until the column vacuum again reaches  $2 \times 10^{-6}$  Torr.
10. Repeat steps 7 and 8 until an emission profile is as shown in figure 4.2.4C. Maintain this condition for about 5 minutes. Turn the filament control to minimum.
11. Select the required ACCELERATION VOLTAGE.
12. Select BEAM on. Slowly increase the Filament control until an emission image is seen (see Figure 4.2.4.A).
13. If the column vacuum increases during step 12, wait for it to recover before proceeding.
14. Increase the Filament control until the emission profile is as shown in Figure 4.2.4.D. This should be when the filament control is at centre scale.
15. Use the S200 in the normal manner. After a few minutes operating, when the LaB6 emitter assembly has reached stable operating conditions, check the emission profile

again. It may be possible to reduce the filament control while still maintaining the correct profile. This will increase the emitter life.

If the LaB6 emitter is run with the filament current too low, then you will get multiple images. If the filament current is only just too low, the multiple images may not be seen, but the resolution will suffer. If the filament current is too high the LaB6 tip will be too hot and its life will be shortened.

The optimum filament setting will be different for different cathodes. The actual setting required can only be found using the emission profile image.

The filament control should be set at the lowest value which gives a solid looking emission profile as Figure 4.2.4.D.

16. At the end of an operating session turn the filament control to minimum before switching the beam off.
17. If venting the column wait 1 min after turning filament control off to allow LaB6 cathode to cool.

#### 4.2.5. Routine Maintenance

If the S200 image becomes unstable and sudden image shifts or drifting focus can be seen then it may be that the emitter assembly needs cleaning or the LaB6 cathode needs replacing.

##### 4.2.5.1. Replacing the Emitter (See Figure 4.2.1.).

1. Admit air to the column and chamber. Open the gun, remove the emitter assembly, close the gun and pump down the system.
2. Take the emitter assembly to a clean area for cleaning and reassembly.
3. Place the assembly on a clean sheet of paper with the grid aperture downward. Using the flat metal key (1) unscrew the height adjusting ring (2). As this is done the cathode will move away from the grid.
4. Grip the cathode pins with the tweezers provided and lift the cathode assembly out of the grid cap.
5. Release the cathode clamp screws (visible in the four large holes) and, using the tweezers, lift out the cathode.
6. If the cathode assembly or grid aperture is not contaminated a new cathode can be fitted. If any contamination is present, dismantle the unit and clean the components as described below.
7. Using the tweezers, put the cathode into the cathode holder and gently tighten the cathode clamp screws.
8. Replace the cathode assembly in the grid cap. Replace the height adjusting ring and screw it down until it touches the sleeve.
9. Adjust the cathode height and centring as

#### 4.2.5.2. Dismantling the Assembly.

1. Remove the cathode as described above.
2. Turn the grid cap over and press the centre of the grid aperture. This should separate the aperture from the grid cap.

#### 4.2.5.3. Cleaning the Components.

##### **DO NOT ATTEMPT ANY CLEANING OF THE LAB6 CATHODE.**

All emitter assembly components can be cleaned using the method below.

1. Remove all signs of contamination using 1 micro Hyprez diamond compound or other very fine grit abrasive. The abrasive should be carried on a soft fabric or a cotton bud. The hole in the grid aperture may be cleaned with abrasive on a cocktail stick or soft wooden stick. The faces of the aperture are best cleaned by rubbing on some abrasive spread on a sheet of CLEAN fine surfaced paper laid on a FLAT surface (eg, a piece of glass).
2. Wash all signs of abrasive from all components with Arklone, propanol or methanol, (or other suitable solvent) preferably in an ultrasonic cleaner.
3. Wash all components again in clean solvent.
4. Repeat step 3.
5. Dry all components using a hot air blower if possible.

#### 4.2.5.4. Reassembly of the Emitter Assembly.

1. Wear gloves when handling any part of the clean emitter assembly.
2. Fit the grid aperture into the grid cap. Fit the spring which holds the aperture in. Check that the aperture is correctly seated in its recess in the grid cap. If any gap can be seen between the aperture and the grid then it is not seated properly (See Figure 4.2.2.).
3. Using the key, screw down the height adjusting ring until it contacts the sleeve.
4. Adjust the cathode tip position as described in 4.2.5.5.

#### 4.2.5.5. Adjusting the Tip Position

1. Using a low power magnifier and a good source of illumination, look through the grid aperture hole and locate the LaB6 tip. It may be necessary to adjust the height adjusting ring to move the cathode forward so that the tip can be seen. Do not move the tip forward so that it touches the grid.
2. As soon as the tip can be seen through the grid aperture hole, adjust the cathode centring screws to centre the tip in the grid hole.

3. Adjust the height setting ring so that the tip of the cathode is between 0.1mm and 0.15mm behind the front face of the grid aperture (see Figure 4.2.3.). When in this position the tip is inside the hole in the grid, so care must be taken to ensure that the tip is correctly centred before adjusting its height.

One way of seeing when the tip is at the right height is to look obliquely across the face of the grid aperture so that the top near side and the bottom far side of the grid hole are in line (see Figure 4.2.3.). Now adjust the tip height so that the tip can just be seen. When the tip is in line with the top near side and the far bottom side of the grid hole, it is set 0.125mm below the top face. When doing this take great care to ensure that you are seeing the absolute tip of the cathode, it is very small. This operation cannot be done satisfactorily without using a magnifier to see the tip.

4. When the tip height has been set, check the tip is still accurately centred in the grid. Make fine adjustments to the cathode centring screws if required.

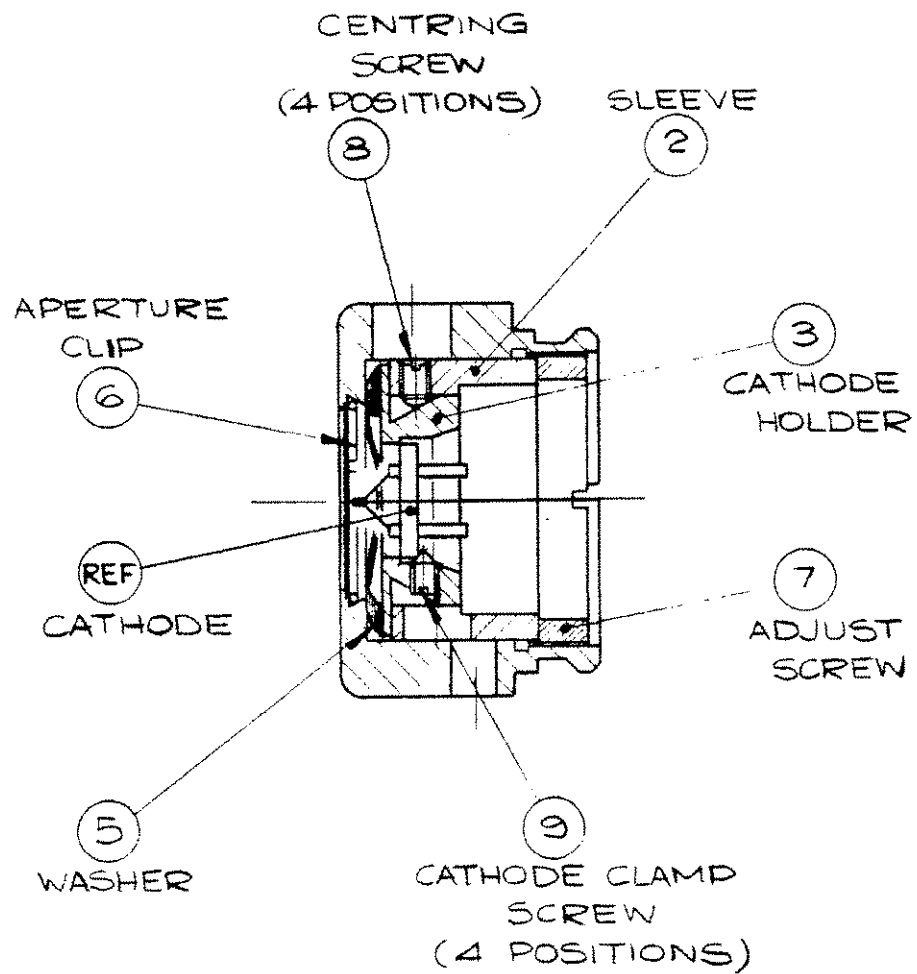


Figure 4.2.1

Emitter Assembly

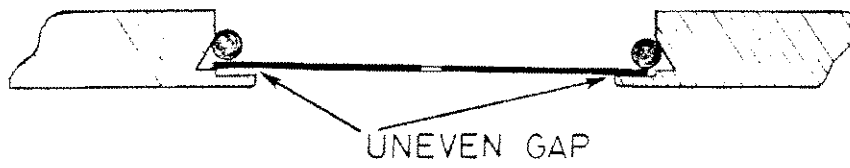


Figure 4.2.2  
Grid Aperture

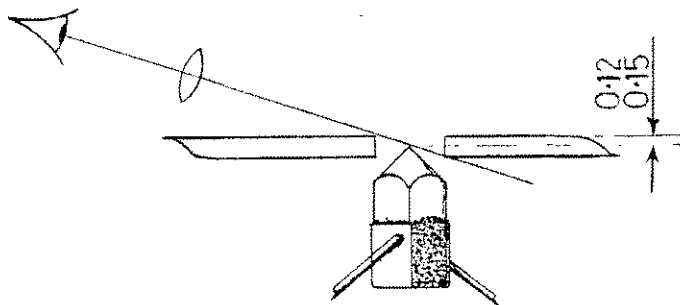
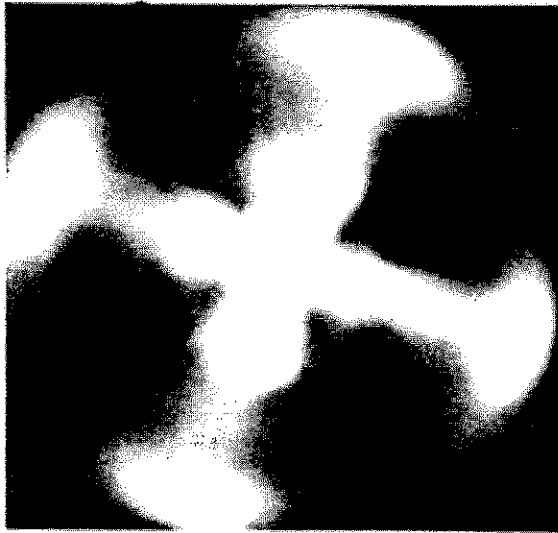
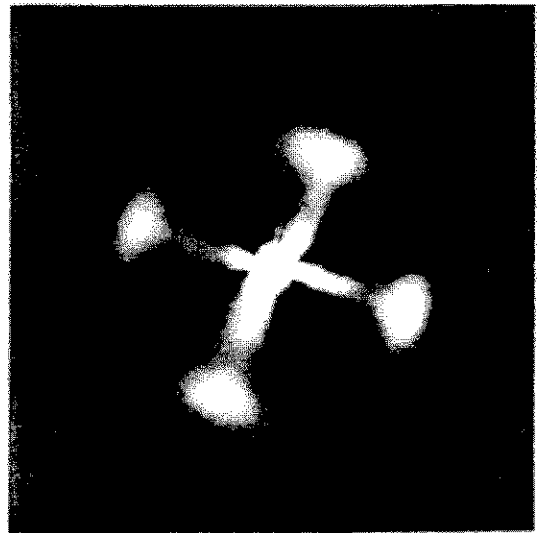


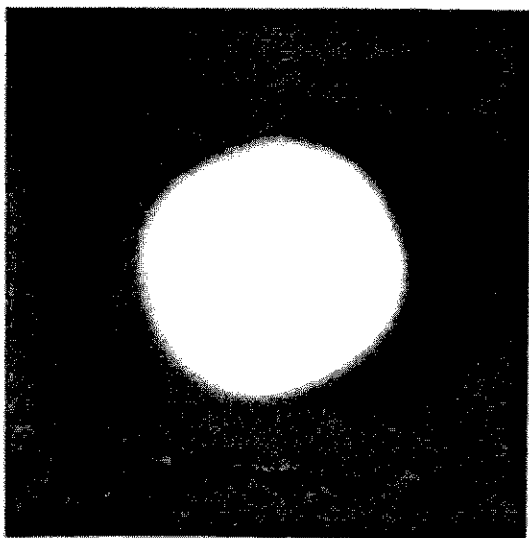
Figure 4.2.3  
Cathode Height Setting



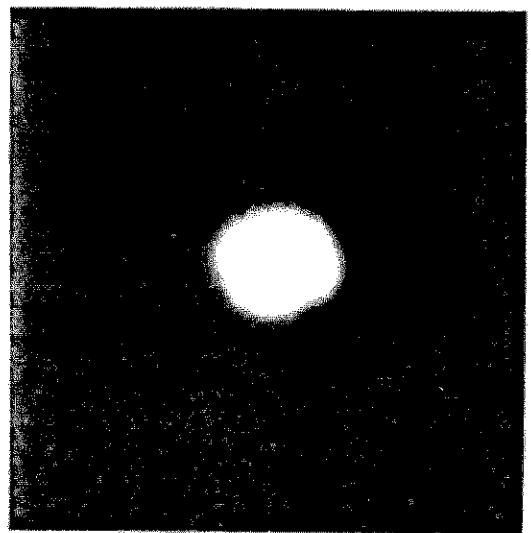
A



B



C



D

Figure 4.2.4

## 4.12 Cathodoluminescence

### Introduction

When certain specimens are irradiated with electrons they emit photons in the visible and infra-red spectrum. These emissions can be detected by the Cathodoluminescence system and imaged in the normal way. Two versions of this option are produced. They are:

- 1) Normal Response Cathodoluminescence (Sales Code CL)

It has a spectral response from 350 nanometers to 630 nanometers

- 2) Extended Response Cathodoluminescence (Sales Code CLR)

It has a spectral response from 350 nanometers to 850 nanometers.

Since the only difference between the two systems is the spectral response of the photomultiplier tube used, this manual applies to both.

### 4.12.1 Installation Requirements

To allow the Cathodoluminescence system to see the specimen it is designed to be mounted on the right hand port on the chamber back plate.

Cathodoluminescence cannot be fitted with a Microspec.

The CL system consists of two components. These are:

- 1) The Detector Assembly: this contains the photomultiplier and head amplifier assembly.

- 2) The Cathodoluminescence Control Unit: this contains the CL pcb 852810, the SE Detector and PM EHT psu's, and a low voltage psu.

### 4.12.2 Installation

#### WARNING

1) Never use any window other than the lead glass window supplied. To do so would create a serious X-Ray hazard. The lead-glass supplied is very soft and care should be taken to prevent it being damaged. It must be kept free of scratches, fingerprints and moisture.

2) When the Control Unit is plugged in and Vac Ready is achieved, the cover must NOT be removed: dangerous HIGH VOLTAGES are to be found inside.

3) There is a safety interlock via cable 231 to prevent the Scintillator EHT voltage from being turned on until Vac ready is achieved.

Installation is as follows:

- 1) Vent the chamber to air. Remove the chamber blanking plate. Clean the O rings on the head amplifier assembly. Fit the assembly to the chamber taking care not to damage the



plastic lens on the front of the Detector. Fit a suitable CL specimen onto the stage. Pump down the chamber.

2) Remove the cover to the Control Unit.

3) Check the transformer primary tapping in the Control Unit, it should be set to the Mains Voltage used.

4) Position the CL Control Unit on the rear console desk top to the left of the visual display. It is free-standing and not held down in any way.

5) Connect the following cables:

a) Cable 231 from the Control Unit to the vac interlock PCB 852728 in the plinth

b) Cable 43 from the CL head amplifier to the pcb (852810) in the Control Unit.

c) Cable 24 from the CL video PCB socket 26 to socket 130 on the S200 auxiliary input panel to the right of the visual display.

d) Cable 19 from the head amplifier to the CL PCB (852810)

e) Remove cable 134 (Scintillator/SE Detector EHT) from the Wallis Unit in the bottom of the console, and plug it into the Scintillator D/P socket in the Control Unit.

f) Connect cable 82 from the Cathodoluminescence Head Amp to the PM EHT D/P socket in the Control Unit.

6) Refit the Control Unit cover.

7) Ensure the SE (Secondary Electron) Detector button is NOT depressed. Plug cable 154 into the Mains Unit; the Control Unit is now powered if Vac Ready is achieved.

#### 4.12.3 Initial Adjustments

The Control Unit and the Detector Assembly are tested in the factory prior to despatch, and should require no further adjustment.

When Vac Ready is achieved check the SE Detector is ON (switch up, not illuminated), select SE on the Video Processor, and obtain a secondary electron image.

Turn off the SE Detector (switch illuminated), select AUX input on the Video Processor Unit, and obtain a Cathodoluminescence image.

#### 4.12.4 Operation

An image is obtained in the same way as for secondary electrons, but with the following changes:

a) select AUX input instead of SE

b) because the AUTO LEVEL will not work it should be deselected. The SIGNAL LEVEL and CONTRAST controls on the Control Unit should be adjusted to give the correct brightness and contrast on the visual display.

If in doubt, refer to the S200 Operating Instructions, Chapter 1 Routine 2.

#### 4.12.5 Routine Maintenance

**WARNING:** when the Control Unit is plugged in, and Vac Ready is achieved, dangerous HIGH VOLTAGES are generated inside. All maintenance to the Control Unit must be performed only by personnel competent work with high voltages.

The only routine maintenance to the Control Unit is to periodically inspect the EHT cables for signs of damage. If this is found immediately refer to the factory.

#### Changing The PM Tube (CLR only):

---

Should this require changing, the maximum output voltage from the PM EHT Unit in the Control Unit will have to be adjusted. This should be done as follows:

- 1) Switch the S200 from OPERATE.
- 2) Remove the cover from the Control Unit, and unplug cable 82 from the PM EHT Unit.
- 3) Turn the LEVEL control fully anti-clockwise, set RV1 (local adjust) on the PM EHT power supply fully clockwise. Connect a DVM, capable of reading approximately 1.5kV, between pins 2 and 3 of the potted assembly on the output of the PM EHT power supply. Note: pin 2 = output voltage, pin 3 = 0V.
- 4) Check the test data sheet, of the tube to be fitted, for anode-cathode voltage (Va/k) required for 2000A/Lumen; this is the maximum voltage to which the tube may be subjected.
- 5) Turn the Control Unit to OPERATE, beware of HIGH VOLTAGES.
- 6) By adjusting RV2 in the PM EHT Unit, set the output voltage, when the LEVEL control is fully clock-wise, to +/-2% of the 2000A/Lumen voltage. If the correct voltage cannot be set by adjusting RV2, set the output as close as possible using RV2, and finally set by RV1 (local adjust). Care must be excersized not to exceed the maximum working voltage of the DVM, especially when the LEVEL control is turned clock-wise.
- 7) Turn the LEVEL control fully anti-clockwise, switch the Control Unit off, plug in cable 82 and refit the cover.

## CHAPTER 5 ADVANCED OPERATING

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- 5.2 NOISE
- 5.3 DEPTH OF FOCUS
- 5.4 SPECIMEN VIBRATION
- 5.5 FINAL APERTURE CENTRING
- 5.6 SPECIMEN PHENOMENA
- 5.7 RESOLUTION
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- 5.12 SUGGESTED READING

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## CHAPTER 5 ADVANCED OPERATING HOW TO GET THE BEST PERFORMANCE

### 5.1 INTRODUCTION AND SUMMARY OF IMAGE DEFECTS

When you have got this far in the manual you should be quite adept at getting an image on the S200, but a number of effects may still be limiting the image quality. In this chapter these effects are described in some detail, as are ways of reducing or eliminating them. Complete elimination of all problems is often impossible since the art of good Scanning Electron Microscopy is one of continuous compromise, e.g. you cannot achieve best resolution and maximum depth of focus simultaneously. The main types of image defects are shown in table 5.1 and the main types of cause in table 5.2.

In the following chapter the interacting parameters are discussed but, in the end it is the operator who must decide what is required in the final image and who has to put up with the compromises.

### 5.2 NOISE

Image quality may be degraded by a "snowstorm" effect across the whole image, usually referred to as noise. The ratio of the wanted signal to the unwanted noise depends on several parameters:

#### 5.2.1 ELECTRON PROBE CURRENT.

This is determined by the selection of acceleration voltage, final aperture size, resolution control setting. If the probe current is small the signal to noise ratio is low, the overall image contrast is reduced and the amount of noise increases.

#### 5.2.2 THE ELECTRON EMISSION COEFFICIENT OF THE SAMPLE

The more electrons emitted by the sample and collected, the better the signal to noise ratio. This will be discussed in section 5.7.

#### 5.2.3 THE VIDEO AMPLIFIER RISE TIME

The longer the video amplifier rise time, the better the signal to noise ratio. In the S200 this is automatically linked to the scan time. So, for a better signal to noise, use a slower scan speed.

#### 5.2.4 THE SCINTILLATOR

The electron collector scintillator has a limited lifetime and, when it becomes discoloured or damaged, can lead to a degraded signal to noise ratio.

#### 5.2.5 PHOTOMULTIPLIER

Excessive noise may be caused by leakage paths round the photomultiplier tube caused by dirt or fingerprints on the tube.

### 5.3 DEPTH OF FOCUS.

This can be considered to be the allowable variation in specimen height, either side of the true electron beam focus, within which the specimen appears to remain in sharp focus. It is a function of working distance and final aperture size (figure 5.1).

To obtain a large depth of focus, use a long working distance or a smaller final aperture.

### 5.4 SPECIMEN VIBRATION.

Specimen vibration is usually only apparent at magnifications greater than about 10,000 times. It is characterised by small image shifts showing up in the line scan direction, usually as a repetitive "sawtooth" effect. It is caused by spurious movement of the specimen relative to the electron beam.

True specimen vibration may be due to inadequate fixing of the specimen to its supporting stub (see section 5.10) to lack of specimen stage clamping or to a vibration source external to the S200 (see section 5.9).

Similar image defects can arise from mild specimen charging (see section 5.5.1) or from specimen heating and distortion (see section 5.6.2)

### 5.5 FINAL APERTURE CENTRING.

If the final aperture is not correctly aligned to be concentric with the electron optical axis, focus adjustment will lead to an apparent lateral image shift, as well as astigmatism and a generally poor image quality. The final aperture should be centred using the aperture alignment micrometers and a "wobbling" focus, as described in section 2.9. The use of FOCUS WOBBLE makes aperture alignment easier.

### 5.6 SPECIMEN PHENOMENA.

#### 5.6.1 SPECIMEN CHARGING.

Electrons incident upon the sample which do not escape as backscattered or secondary electrons are absorbed by the sample. Unless these absorbed electrons can find their way to ground (as in a conductive sample) they will remain in the sample resulting in a negative charge build up, abnormal image contrast, image shift and beam instability.

Dust and other debris on the sample surface can also charge up, and appear as a bright area on the image surrounded by a dark region. The charged debris deflects the incident electron beam slightly, and so there is reduced electron emission from the area round the debris.

If the sample itself is charging, beam deflection will occur during scanning, the effect being seen as a slow image drift followed by a jump back to its original position. Intense sample charging can deflect the beam into the bottom of the final lens, the electron collector system, or any other part of the specimen chamber or stage.

If a non conductive sample is first observed at a high acceleration voltage so that it charges up and subsequently at a lower acceleration voltage, the lower energy beam will be easily deflected by the charge on the sample. In this case it will be necessary to remove the sample from the chamber and discharge it.

There are several ways to prevent specimen charging:-

- a. Coat the sample with a conductive layer (e.g. C, Au, Au/Pd, Al.) by evaporation or sputter coating.
- b. Use a low acceleration voltage
- c. Use short scan times
- d. Use backscattered electron imaging
- e. Reduce the collector bias to zero volts.

Further details concerning specimen preparation are given in section 5.10.

#### 5.6.2 SPECIMEN DAMAGE.

The loss of beam energy in the sample occurs mostly as heat generated at the point of impact. This can cause physical damage (e.g. bending) or chemical damage (e.g. depolymerisation) in the specimen.

The amount of heat generated depends on:

- a. Electron beam power, determined by acceleration voltage and specimen current.
- b. Scanned area. If you scan a large area the heat is distributed.
- c. The thermal conductivity of the sample.
- d. Scanning time. The longer the beam is left in one place the hotter it will get.

Biological samples and polymers are especially susceptible to the heating effect. To avoid damaging delicate specimens, the following precautions should be taken:

- a. Use a low acceleration voltage.
- b. Use a low beam intensity (high RESOLUTION setting and small final aperture) or select EMITTER LaB6 and use a tungsten emitter. This reduces the emission current of the gun from 300 microamps to 80 microamps.
- c. Reduce the scanning time, even though this may result in noisy images.
- d. Photograph large areas at low magnifications.

- e. Ensure that there is adequate specimen coating of a conductive material.

### 5.6.3 SPECIMEN CONTAMINATION.

When the electron beam is on the same part of the specimen for a long time any residual organic material in the vacuum environment or on the sample may be decomposed by the electron beam and form a build up of contamination on the sample surface.

Common causes of contamination are:

- a. Cleaning solvents. You need to allow adequate evacuation time to pump out residual vapours after cleaning the column before attempting high resolution work.
- b. Organic or polymeric samples which are unstable in the vacuum.
- c. The use of too much vacuum grease on "O" rings.
- d. Unstable organic adhesive used to fix sample to stub.
- e. Fingerprints on inside surfaces of the column and chamber.

The effects of contamination are:

- a. Dark areas left on the sample caused by the reduction in secondary electron emission.
- b. Decreased resolution caused by the contamination layer covering the microstructure.
- c. Reduced detectability of low atomic number elements during X-ray microanalysis.

## 5.7 RESOLUTION.

Resolution can be defined as the amount by which two adjacent objects can be seen to be separated.

It is found that the resolution observable in a scanning electron microscope depends on several factors. These are itemised below, and subsequently discussed more fully.

- a. Electron probe diameter (RESOLUTION Setting)
- b. Acceleration voltage
- c. Angular aperture diameter
- d. Working distance
- e. Aberrations (including astigmatism)
- f. Electron gun alignment
- g. Final aperture alignment
- h. Signal type
- i. Specimen tilt angle and direction
- j. Photographic frame period
- k. Vibration, charging



a. Electron probe diameter (RESOLUTION setting)

We have seen earlier that the spot size is a function of the RESOLUTION setting. The greater the RESOLUTION, the greater the demagnification. The smaller the spot size, the better the resolution. The final lens also has an effect on the demagnification. Again the greater the lens current (corresponding to a short working distance) the greater the demagnification.

There is no advantage, however, in using a small spot size at low magnifications, since image detail will be lost.

The effect of spot size on the resolution is shown in figure 5.3.

b. Accelerating Voltage

The best resolution is usually obtained at high accelerating voltages because at these voltages the effect of chromatic aberration is minimised and the emission from the electron gun is brighter. Diffraction, is also reduced because the electron wavelength is less at higher voltages. If the accelerating voltage is too high, however, there are certain detriments:

- i. Lack of surface detail
- ii. Edge-effects (penetration)
- iii. More likelihood of specimen charging
- iv. Specimen damage

Generally, finer surface structures can be seen with lower accelerating voltages. At high accelerating voltages, beam penetration and the diffusion volume becomes larger, resulting in unnecessary signals (e.g. reflected electrons) being generated within the specimen thus reducing image contrast.

c. Angular aperture

d. Working distance

e. Aberrations (including astigmatism)

These three have an interacting effect, mainly as a result of the semi-angular aperture. Electron lenses suffer from lens aberrations (e.g. spherical, chromatic and diffraction) as well as astigmatism. Astigmatism has been adequately discussed and is eliminated by the microscope stigmator controls.

The effect of the aberrations is to increase the incident electron probe diameter ( $d_0$ ) as shown in Table 5.3.

$$d_{\text{resultant}} = (d_o^2 + d_s^2 + d_c^2 + d_d^2)^{\frac{1}{2}}$$

where  $d_s$ ,  $d_c$  and  $d_d$  are the probe diameters due to the effects of spherical, chromatic and diffraction aberrations respectively.

$$\text{Also } d_s = \frac{1}{2} C_s \alpha^3$$

$$d_c = C_c \alpha \left( \frac{\Delta E}{E} + 2 \frac{\Delta I}{I} \right)$$

$$d_d = \frac{1.22 \lambda}{\alpha}$$

where

$\lambda$	= electron wavelength = $(12.12/\sqrt{E})\text{\AA}$
$C_s$	= coefficient of spherical aberration
$C_c$	= coefficient of chromatic aberration
$\Delta E$	= thermal spread of EHT emission energy
$E$	= accelerating voltage
$\Delta I$	= instability in final lens current
$I$	= final lens current

$C_s$  and  $C_c$  are both functions of the working distance, and to reduce their severe effects, the working distance should be short.

Table 5.3  
Aberrations and Probe Diameter

Thus it is found, when all the interacting effects are considered, that for the best resolution, one must have a small angular aperture and a short working distance, thus precluding maximum depth of focus. A smaller angular aperture would give rise to excessive noise. A typical working distance is 5mm.

- f. Electron gun alignment.
- g. Final aperture alignment.

If either of these are incorrect reduced resolution will result. Poor gun alignment reduces the amount of electron beam hitting the sample, leading to reduced emission and, eventually, noisy images. Misaligned apertures produce astigmatism effects.

- h. Signal Type

When an electron beam interacts with a sample, each individual electron can undergo an elastic or inelastic interaction involving small or large (respectively) energy losses.

The depth to which an incident electron can penetrate (typically 1-5 $\mu$ m) a sample before losing all its energy is a function of both the incident electron energy and the atomic number of the sample. This penetration volume is shown diagrammatically in figure 5.4.

Elastic scattering by the atomic nucleus involves a small momentum (and hence energy) change of the incident electron which can escape from the sample as a backscattered (reflected, primary) electron. Such backscattered electrons have a high energy (close to the incident electron beam energy) and are collected from up to several microns depth in the sample. The number of backscattered electrons emitted is a function of the atomic number of the samples.

Inelastic scattering involves a collision between the incident electrons and the orbital electrons of the sample atoms. This involves a large energy loss, and the resultant secondary electrons have a very low energy (e.g. less than 200 eV) and are easily absorbed by the sample. They are only collected from the top few hundred Angstroms of the sample surface and thus should be collected for best resolution (see figure 5.4). Too large a penetration volume may effectively reduce the ultimate resolution.

- i. Specimen tilt angle

Normal SEM images are influenced by the sample surface topography, which contributes to the collection efficiency of the electron collection system. Backscattered electrons travel in straight lines and are only collected if they happen to be travelling towards the electron collector. Secondary electrons can be deflected towards the collector by having a

positive bias on its grid. Surface features which are pointing directly towards the electron collector appear bright and thus suffer least from noise effects under high resolution conditions (e.g. point B in figure 5.4)

An angle in the region of  $45^{\circ}$  is generally used, but it is advisable to adjust the value and observe the effect on the image.

j. Photographic frame period

The longer the frame period, the longer the amplifier rise time can be without reducing resolution. Long rise times reduce the effects of noise, and thus long frame periods should be selected when taking high resolution micrographs.

k. Vibration and charging

Resolution will be severely limited if either of these two effects are present. Vibration of the sample is due to poor sample preparation. External vibrations are due to poor installation procedures. Sample charging can be minimised as discussed in Section 5.4.

## 5.8 PHOTOGRAPHIC CONSIDERATIONS

A good micrograph is sharp and noiseless with optimum contrast and brightness. These parameters are very user dependent. The camera and record CRT should be correctly calibrated as described in chapter 2. The waveform (as observed in GRAPH) should be evenly distributed between the video level markers, nearly spanning the markers but never going outside the limits set by them.

## 5.9 EXTERNAL INFLUENCES.

The two most troublesome external influences are:

- a. Magnetic fields
- b. Mechanical vibration

They give rise to image distortion and jagged edges. These problems will not exist if the room in which the S200 is installed is a room which meets the conditions laid down in the Stereoscan 200 installation recommendations.

## 5.10 SPECIMEN PREPARATION.

Specimens which are studied in the SEM can be divided into two main categories, namely Conductors and Non-Conductors. Factors to consider during specimen preparation are given in table 5.4.

### 5.10.1 Conductors

These fall into two groups:

- a. Metallic these are generally excellent conductors and need no preparation.

- b. Semi conductors--samples with a resistance of less than  $10E10$  ohms can be examined without special preparation.

#### 5.10.2 NON-CONDUCTORS.

This group includes all samples which are not electrically conducting, e.g. those generally not containing volatiles e.g. fibres, plastics, polymers, semi-conductors with a resistance greater than about  $10e10$  ohms.

Those generally containing volatiles e.g. biological and botanical material.

##### 5.10.2.1 NON-VOLATILE, NON-CONDUCTORS

For most non-conductors which contain no volatile components, e.g. water, that would outgas in the vacuum system it is sufficient to coat the sample with a thin layer of conducting medium such as Au, C, Au/Pd, Al etc.

This layer is typically 200-300 angstroms in thickness. There are several reasons for this coating:-

- a. Increased conductivity of the sample, thus minimising sample charge-up, which results in deflection of the incident beam and severe degradation of the final image. (See Section 5.6.1 on Specimen Charging Effects.)
- b. Increased mechanical stability of the sample due to increased heat conduction.
- c. Increase in primary and secondary electron emission.
- d. Decrease in beam penetration, resulting in better spatial resolution.

The two important current techniques of applying a coating are vacuum evaporation and ion sputtering.

Gold is generally used for the following reasons:

1. High secondary emission coefficient
2. High conduction of electrons and heat
3. Does not oxidise
4. Good granularity of evaporated or sputtered particles.

Carbon coating by evaporation is generally used if X-Ray microanalysis is to be undertaken on the sample unless, of course, the element under investigation happens to be carbon. Aluminium could be used in this case.

More recently Pt/Pd and Au/Pd have been used since their granularity is smaller. Al can also be used, but it has low mechanical strength and can oxidise.

#### 5.10.2.2 VOLATILE, NON CONDUCTORS

Biological and botanical samples, by their nature, require relatively more complex preparation procedures. The samples fall into two main categories: a) hard, b) soft.

- a. Hard sample (e.g. bone, teeth, wood).  
These can be washed to remove extraneous fluids such as blood and mucus, dried in air and coated in the normal way.
- b. Soft Samples
  - i. Untreated

Soft tissue needs more specialised treatment. Most soft tissue contains up to 90% water which must be removed without altering the structure. If this is not done, there will be difficulty in achieving adequate vacuum in the SEM, and complete or partial sample collapse and distortion would occur.

Some botanical specimens can be observed successfully for short periods provided that thought is given to the selection of instrument parameters, e.g. using a low accelerating voltage and beam current.

- ii. Replication

Although it is usually only adequate for low magnification work and for comparatively simple surface topography, replication has the advantage that the sample can be totally preserved. One method of replication uses an elastomeric material such as silicone rubber to obtain an impression. A positive replica is then obtained from the impression by coating it with a low-viscosity polymethylmethacrylate solution, allowing this to dry and then

stripping the resultant film away. Coating and examination follow in the usual manner.

With suitable modification, transmission electron microscope replication techniques can be utilised.

### iii. Chemical Pre-treatment

This technique involves chemical fixation of the material to strengthen the tissue. There is a large range of chemicals used in this process (e.g. glutaraldehyde and osmium tetroxide) and there are numerous publications discussing the benefits of each. After fixation, it is necessary to displace the water in the sample by a solvent to aid drying. The method must be such that the specimen suffers no physical change. The commonest drying agent used is a series of ethanol/water mixtures through to 100% ethanol. Having replaced the water present in the sample there is a choice of three methods for drying:

#### Freeze Drying

This is a complicated procedure, and may not be too successful. The sample is quench-frozen and maintained at low temperatures (about  $-130^{\circ}\text{C}$ ) until the sublimation process is complete.

#### Air Drying

It is worthwhile first transferring the specimen to a solvent of low volatility (e.g. amyl acetate) as the last stage of dehydration. The solvent is then allowed to evaporate from the sample under carefully controlled temperature conditions. As this is a gentle process, there is little chance of any specimen damage.

#### Critical Point Drying

The specimen is dehydrated as previously described and the solvent replaced with a liquified gas in a small pressure vessel. The vessel is then heated to above the critical temperature of the selected gas. Under these conditions the liquid and vapour phases have the same physical properties, so that on venting, the liquid vapourises across cell boundaries and therefore minimum sample distortion occurs.

The sample may, however, undergo some mechanical shock during the venting stage. The choice of gas for this process is limited by the number of available gases which have a critical temperature relatively close to ambient, a safe pressure level and low toxicity, whilst at the same time being completely miscible in all proportions with the solvent selected in the final stages of dehydration. It is important to replace the solvent completely with the liquified gas (usually carbon dioxide) before venting.

#### iv. General Considerations

The methods described above for soft tissue preparation are mainly for secondary electron imaging. The problems facing the biologist or botanist who wishes to undertake X-ray microanalysis are different in that the requirements in this case are to maintain the element(s) of interest in their original position in the sample.

There is not one major preparative technique for biological/botanical samples. Where possible, several combinations should be tried for a particular type of sample, giving prime consideration to the information sought. Once a technique has been established, instrument parameters and specimen coating methods must be carefully considered.

### 5.10.3 ATTACHING THE SPECIMEN TO THE STUB

The specimen may be attached to the stub by using any suitable adhesive, but care must be taken to ensure that the specimen is not electrically insulated from the stub. Colloidal silver (Silver Dag) or colloidal graphite (Acquadag) are often useful for making electrical contact between the edge of the specimen and the stub, and are supplied by Acheson Colloids Ltd.

For lightweight samples (e.g. fabrics, powders, fibres, small pieces of metal etc), the "dags" mentioned above are usually adequate as an adhesive. For heavier and bulkier specimens (e.g. large pieces of metal), it may be necessary to mix some glue with the "dag". The glue should be rapid drying, not outgas and be easily soluble in a suitable solvent for subsequent sample removal and cleaning (e.g. Durofix).



#### 5.10.4 SPECIMEN EARTHING (GROUNDING)

It is possible to earth the specimen either directly through the specimen stage or via a specimen current amplifier. This facility is selected on the stage by a plug situated on the left hand side of the stage mechanism looking from the front of the stage.

If the SCM is not fitted, or the SCM is fitted but not in use, it is essential that the specimen is earthed through the specimen stage, otherwise charging phenomena may be observed.

Even with coated specimens it is doubtful if the best results will be obtained using an accelerating voltage greater than 25kV and for the majority of investigations 10kV or 5kV is adequate (whereas best results on conducting samples such as metals are generally obtained using 25kV).

It must be emphasised, however, that the operating conditions which give the best results on one sample will not necessarily give best results on another sample. All major parameters such as accelerating voltage, spot size are readily variable.

#### VARY THEM

### 5.11 SUMMARY

In order to get the best results from a scanning electron microscope it is important to consider the following points:

a. Routine Operation

Read carefully and understand the operating instructions supplied with the instruments and ensure that you are trained by an experienced S200 operator (e.g. by participating in a Cambridge Instruments Ltd approved training course. See the front of this publication for further details).

Ensure that the correct routine maintenance has been carried out so that the S200 will generate the results that you require -e.g. clean or replace the apertures if the image is astigmatic.

b. Correct Parameter Choice

Read and understand Chapter 5 to appreciate the fundamental considerations. Refer to published textbooks, journal and conference proceedings concerning SEMs (some suggested literature sources are given in Section 5.12). Communicate with people doing similar investigations and find out their techniques. (The Cambridge Instruments Ltd Stereoscan users list may help to locate relevant installations.)

Experiment on your own samples to find the best conditions to use. The S200 controls reflect in choice and location the many years of SEM experience incorporated in the Stereoscan, and will amply repay considered use.

c. Specimen Preparation

Incorrect or incomplete sample preparation cannot be compensated for by mal-adjustment of the S200, so careful consideration of the preparation procedures and adequate care in their use are pre-requisites to success in the use of the instrument.

The important instrument parameters, their effect, and their optimum conditions are summarised in the following tables, together with some general comments. Use the table only as an aide-memoire, and refer to the rest of Chapter 5 for further information.

d. Resolution

Resolution limits in an SEM are thoroughly considered by Goldstein and Yakowitz 1975 (see Section 5.12, 5.8). They discuss a theoretical resolution limit assuming a 25% contrast level, a secondary electron collection efficiency of unity and certain values of gun brightness, field aperture diameter and working distance. They emphasise that the theoretical resolution **cannot** be achieved on all specimens or even with all objects in the same field of view of a particular specimen. This is especially true of specimens that do not produce strong secondary topographic contrast.

Resolution may be degraded by boundary and edge effects, both of which are due to the electron collector "seeing" a broader electron probe due to scattering effects. The resolution may not be the same in two different directions in the same image. For example, if a sample is tilted towards the electron collector, the electron beam on the sample surface may appear elliptical in shape, elongated towards the collector.

Remember, optimum resolution is only obtainable from optimum specimens.

#### 5.12 SUGGESTED READING.

The tremendous growth of scanning electron microscopy in the years since the advent of the first commercially available instrument (the first \* of the long line of "Cambridge" Stereoscans) has inevitably generated a prodigious bibliography of instrumental, applications and techniques literature. To attempt anything like a complete listing of this in the present volume would obviously be inappropriate. Instead, the Stereoscan user is referred to a number of books which provide a general background to scanning electron microscopy, together with a selection of more specialised publications. Probably the most useful items listed in this section are the various series of national and international conference proceedings. The serious reader is strongly recommended to become familiar with these, and will soon identify those series whose future events are most likely to be of special relevance, by virtue of content, sponsoring body or geographical location.

While Cambridge Instruments are pleased to be able to provide this introductory list of publications, and are happy to have been associated directly or indirectly with the work described in many of these, they regret that they are unable to provide copies of the publications cited. Instead the reader is referred to the relevant publishers, especially of the conference proceedings, or to suitable national technical libraries.

For convenience the listed publications are classified into a number of groups under various headings, but it will be appreciated that many of the volumes and articles cover a wider field than the heading under which they are given. New readers will almost certainly find that just one or two references will provide them with almost all they routinely need. Table 5.8 gives titles of relatively recent text books which can be regarded as key integrating publications, some with a useful review of the evolutionary backgrounds to equipment and techniques. Table 5.9 summarises the most important continuing series of SEM conferences, seminars and meetings, and their organising societies. Table 5.10 lists specific references under a number of sub-titles, and Table 5.11 lists some journals which may occasionally include articles of interest.

Courses on electron microscopy (including scanning electron microscopy) are available at many universities and elsewhere. Details of those held in the UK are usually available from the Royal Microscopical Society (33/38 St Clements, Oxford, OX4 1AJ, England) or from the Institute of Physics (47, Belgrave Square, London SW1X 8QX, England) either of whom may be able to advise how to locate details of courses elsewhere in the world.

New S200 users are strongly advised to participate in an approved operator's training course. Details of courses and enrolment procedures are available from accredited Cambridge distributors and service organisations, and from Cambridge Instruments Ltd, Sales Dept, Rustat Road, Cambridge, CB1 3QH, England.

\* eg Stewart A D G and Snelling M. A., "A new scanning electron microscope", Proc 3rd European Regional Electron Microscopy Conference, Prague, 1964, pp 55-56.

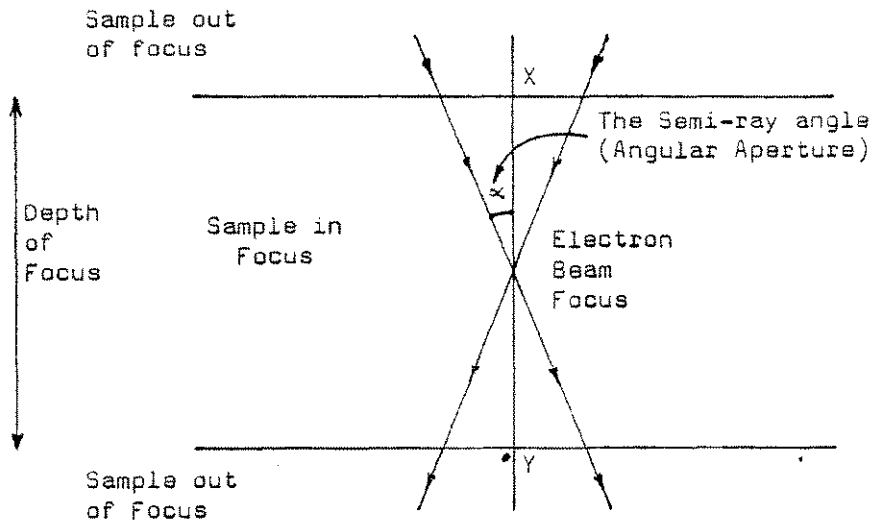


Figure 5.1  
Depth of Focus

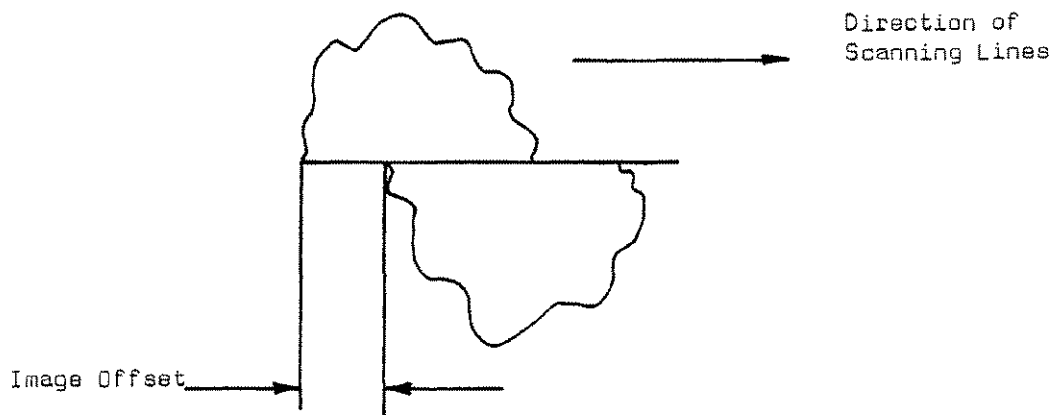


Figure 5.2  
Image offset

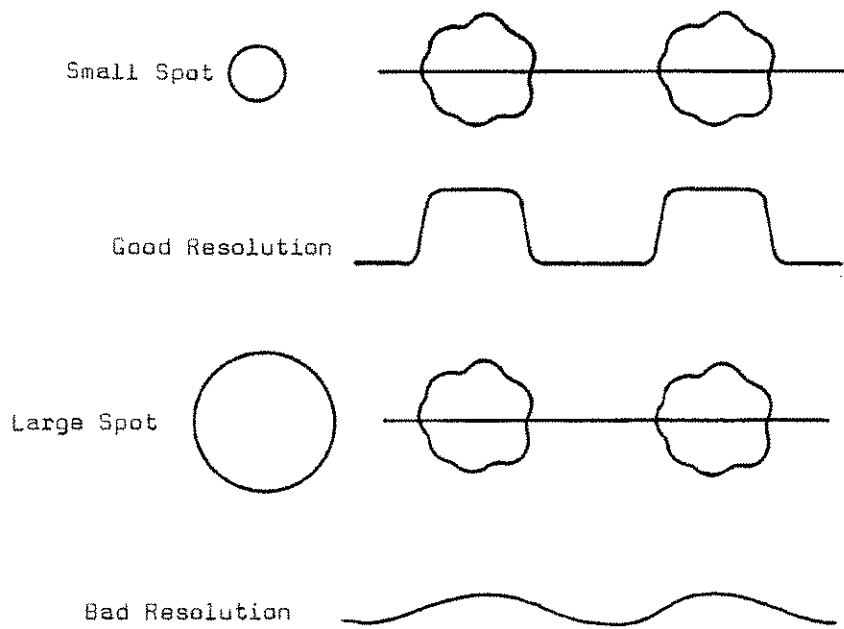


Figure 5.3

Effect of Spot Size on Resolution

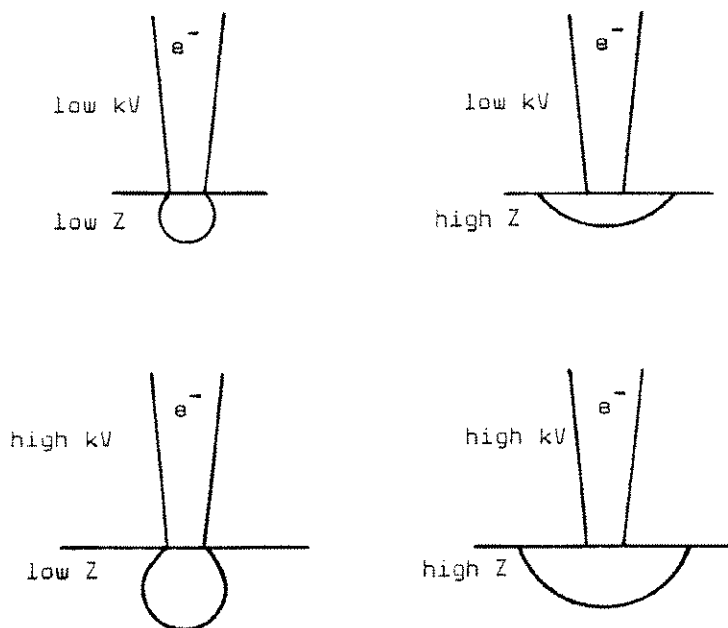


Figure 5.4

Depth of Electron Penetration

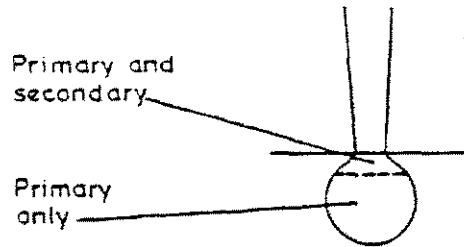


Figure 5.5

Regions of electron collection from a sample

<u>Collector Bias</u>		<u>Appearance of:</u>	
		<u>Region A</u>	<u>Region B</u>
+ve	backscattered and secondary electrons collected	dark	bright
-ve	backscattered electrons only collected	black	bright

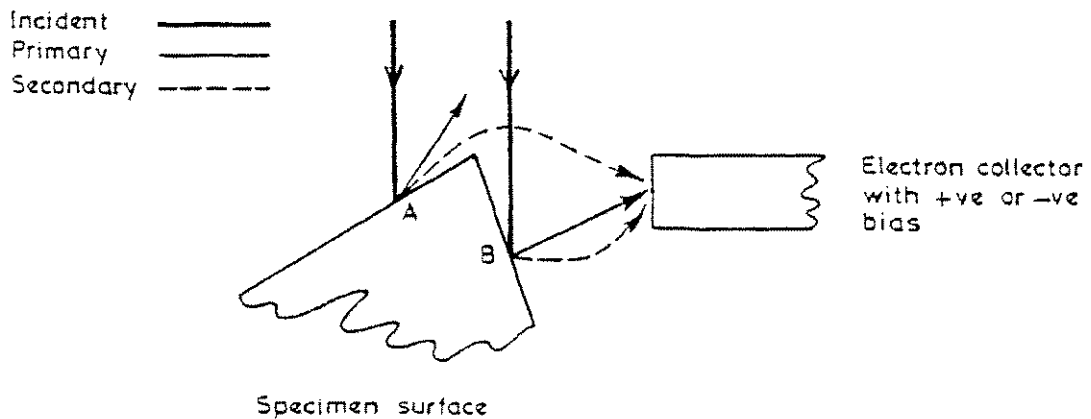


Figure 5.6

Collection of backscattered and secondary electrons

<p><b>(A) Lack of Image Sharpness</b></p> <ul style="list-style-type: none"> <li>Astigmatism not corrected</li> <li>Spot Size too large</li> <li>Misaligned final aperture</li> <li>*Angular aperture too large</li> <li>Working distance too long</li> <li>Poor focusing</li> <li>Unsaturated filament</li> <li>Filament not aligned, or at incorrect height</li> <li>Too low accelerating voltage</li> <li>kV instability due to dirty gun/anode</li> <li>Sample charge - up or vibration</li> <li>Magnetic fields (from sample or external)</li> <li>Poor depth of focus</li> <li>Too much noise</li> <li>Specimen contaminated or of low potential resolution</li> </ul> <p><b>(B) Noisy Image</b></p> <ul style="list-style-type: none"> <li>Spot size too small for resolution required</li> <li>Too short frame period</li> <li>Filament not aligned, or at incorrect height</li> <li>Unsaturated filament</li> <li>Too low accelerating voltage</li> <li>Incorrect electron collector bias and voltage</li> <li>Sample not facing electron collector</li> <li>Inadequate rise time</li> <li>Faulty scintillator and/or photomultiplier</li> <li>Too much CONTRAST, incorrect LEVEL</li> <li>Dark regions of sample being observed</li> <li>*Too small an angular aperture size</li> </ul>	<p><b>(C) Poor Final Image Quality</b></p> <ul style="list-style-type: none"> <li>Insufficient resolution</li> <li>Incorrect LEVEL &amp; CONTRAST settings</li> <li>Incorrect accelerating voltage</li> <li>Incorrect beam (probe) current</li> <li>Inadequate specimen preparation</li> <li>Incorrect HRRU calibration</li> </ul> <p><b>(D) Image Distortion</b></p> <ul style="list-style-type: none"> <li>Specimen charge-up</li> <li>Specimen magnetism</li> <li>Specimen vibration</li> <li>Specimen damage</li> <li>External magnetic fields</li> <li>FCF in use on highly tilted samples</li> <li>External vibration</li> </ul> <p>*Optibeam normally sets the angular aperture correctly.</p>
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**Table 5.1**  
**Image Defect Types**

<b>(A) Operator error</b>		<b>(C) Sample type and preparation</b>	
<b>CAUSE</b>	<b>DEFECT</b>	<b>CAUSE</b>	<b>DEFECT</b>
(1) Under-run filament	Lack of image sharpness and lack of signal	(1) Atomic number effect	Reflected and secondary electron images brighten with increasing atomic number
(2) Incorrect probe current	Low contrast, lack of signal (if too low), possible specimen damage (if too high)	(2) Charge-up	Image shift and distortion, abnormal contrast and unstable image
(3) Incorrect gun and final aperture alignment	Lack of image sharpness, image shift when focusing	(3) Overheating	Deformation and cracking of specimen, coating peeling
(4) Wrong angular aperture size and working distance	Lack of resolution, Lack of depth of focus, noisy images	(4) Incorrect or inadequate sample coating	Charge-up or sample damage
(5) Insufficient astigmatism correction	Less image sharpness in one direction, poor resolution	(5) Contamination	Surface coating leading to poor image quality
(6) Wrong scanning period	Noisy image (too short), beam deflection on charging sample (too long), specimen damage	(6) Incorrect sample preparation (especially soft tissues etc)	Sample deformation, damage and charge up
(7) Wrong level selected	Poor image quality		
(8) Wrong contrast selected	Poor image quality		
(9) Wrong accelerating voltage	Influence on resolution, penetration and charging		
<b>(B) External influences</b>		<b>(D) Machine Faults</b>	
<b>CAUSE</b>	<b>DEFECT</b>	<b>CAUSE</b>	<b>DEFECT</b>
(1) Magnetic fields	Image distortion, jagged edges in image	(1) kV instability	Jagged edged images, focus and brightness drift
(2) Vibration	Jagged edge in image	(2) Gun emission instability	Focus and brightness drift
		(3) Faulty condenser and objective lens supplies	Focus and brightness drift
		(4) Scintillator kV discharge	Unstable image, noise
		(5) Column contamination	Lack of sharpness, bad astigmatism and image drift.
		(6) Scratched final lens	Bad, uncorrectable astigmatism

**Table 5.2**  
**Image Defect Causes**



1. The size of the specimen must be reduced (if necessary) to fit the available specimen holders and specimen stages, but there is often an advantage in selecting an even smaller sample size for ease of observation.
2. The object must be able to withstand being in the high vacuum of the SEM; it must not change its shape, and it must not outgas. A cold stage may help here.
3. It should be clean, i.e. free of dust, oils and greases (Their presence can lead to charging and contamination effects).
4. It should be treated to improve the secondary electron yield if this is low e.g. coating of the sample with gold.
5. Any disturbances in the surface structure caused by preparation procedures should give rise to surface details which are too fine to be resolved at the magnification used to record the images.
6. If an artefact is suspected as a result of a preparative procedure, a control specimen should be utilised.
7. The specimen stub should be in good electrical contact with ground potential.
8. There should be good electrical contact between the surface of the specimen and the specimen stub, i.e. attach sample to stub with conducting paint such as Silver-Dag and coat the specimen adequately if necessary.
9. The specimen stub should give rise to as few backscattered and secondary electrons as possible. Al stubs are normally used, although C stubs are used for some X-ray microanalysis applications.
10. Very small particles are best mounted on a low mass foil to give rise to minimal interfering signals e.g. nylon film stretched over Al ring.
11. The sample must be attached to the specimen holder (stub) so that it does not move whilst being irradiated by the electron beam.
12. The sample should be attached to the specimen holder (stub) so that all the surface can be studied using the existing stage movements (e.g. tilt, rotate, X, Y, Z).

**Figure 5.4**  
**Factors to consider during specimen preparation**

<u>PARAMETER</u>	<u>EFFECT(S)</u>	<u>OPTIMUM CONDITIONS</u>	<u>COMMENTS</u>
Filament - Grid Distance ("Height")	Number of electrons in crossover; SNR (Signal to Noise Ratio)	0.4 - 0.6mm	Influences Resolution and Filament life
Filament Centring	Source Geometry; SNR	Correctly Centred	Filament may drift early in its life
Filament Current	Controls Emission; SNR	Approx. 2.8A (New fil.)	If too high - shortens fil life. If too low -reduces resolution
Beam Current	Number of electrons in crossover; SNR	High as possible	Use less for beam sensitive samples
Accelerating Voltage	Resolution; sample damage; penetration; signal emanation depth; contrast; x-ray emission	Sample dependant	Found by expt. Low kV produces low resolution, contrast, SNR
Grid/Anode Cleanliness	Stability of Beam	Must be kept clean	Dirt encourages flashovers

**Table 5.5**  
**Electron Source Parameters**

<u>PARAMETER</u>	<u>EFFECT(S)</u>	<u>OPTIMUM CONDITIONS</u>	<u>COMMENTS</u>
C1/C2 Currents	Beam diameter, resolution, SNR, probe current	Small for resolution, beam sensitive samples. Large for XRMA, BSD, CL.	If noise is excessive check gun alignment, collector, sample CL.
Final Aperture Diameter	Beam diameter, aberrations, resolution, depth of focus, SNR, probe current	General: - 20um Long W.D: - 50um CL, XRMA: - 50um D.o.f.: -20um + "50um"	May have to trade D.o.f. vs resolution
Final Aperture Centring	Resolutions, aberrations, image shift,	No image shift while focusing	Effects most obvious at high magnification
Spray and Final Aperture Cleanliness	Astigmatism	Should be clean	Effects worse at low kV
Stigmators	Astigmatism reduction	Adjusted to minimise astigmatic effects	More correction required at low kV
Frame Period	SNR, Charging	Long - Noise reduction Short - Charge reduction	May have to compromise
Vacuum System	Filament life, EHT stability, contamination	Best vacuum possible	LN <sub>2</sub> traps aid pump down speed

Table 5.6  
Electron Optical Column Parameters

TABLE 5.7 - SPECIMEN PARAMETERS

PARAMETER	EFFECT	OPTIMUM CONDITIONS	COMMENTS
Mounting on to specimen stub	Charging, vibration	Securely attached	Use quick - drying adhesive (Preferably conducting)
Conductivity and grounding	Charging	Good conduction to earth	Use conducting paint (Dag)
Sensitivity to beam damage	Specimen distortion	Use:- Low beam current, short frame period. Focus away from area of interest	Optimum conditions may result in poor SNR
Coating	Conductivity	No Charging	Charging seen as: local intensity changes; astigmatism; image distortion; dark micrographs
Coating	S.E. signal	20nm Metal coating	If too thick can mask detail
Coating	B.S.E. signal	Carbon coating	If too thick will absorb signal
Coating	C.L. signal	Carbon, Aluminium coating	If too thick will absorb signal
Coating	X-ray signal	Carbon, Aluminium coating	Avoid interference with element(s) of interest
Coating	Specimen current	C - Material contrast Au - Topography	Use inverted S.C. image for best topography
Coating	Resolution	Gold	Grain size of coating may be resolution limit
Mechanical Stability	Resolution; Cracks in coating	Vibration amplitude less than required resolution	Reduce kV, and beam current
Eucentric Position	Specimen tilting, Stereo pairs	No translation motion during tilting	Use Eucentric goniometer stage for best results
Specimen Tilting	Topography, SNR	About 45° for E-T detector. Zero for BSD	Found by expt. Depends on sample
Specimen Tilting	Stereo viewing	Tilt difference 5-10°	Use low tilt diff. for large height differences
Specimen Tilting	X-ray analysis	Depends on detector geometry	More critical for WDS
Working distance	Resolution	Short	Beware of final lens damage
Working distance	Depth of focus	Long	Limit = Loss of resolution
Working distance	Electron collection	Generally better at long WD	Poorer resolution at long WD
Working distance	Beam aperture Angle ( $\alpha$ )	Small	Reduces aberrations. Good for D.o.F.

<u>PARAMETER</u>	<u>EFFECT(S)</u>	<u>OPTIMUM CONDITIONS</u>	<u>COMMENTS</u>
Correctly calibrated Record system	Information content from micrographs	Acceptable brightness and contrast for all mags and frame period.	More important for Polaroid
Film Speed	"Grain" of image	Medium/Slow 50-400 ASA	Limited by film available for camera in use

Table 5.8  
Photographic Parameters

## THE USE OF THE SCANNING ELECTRON MICROSCOPE

J.W.S. Hearle, J.T. Sparrow, P.M. Cross  
Pergamon Press (1972)

## THE SCANNING ELECTRON MICROSCOPE

C.W. Oatley  
Pergamon Press (1972)

## SCANNING ELECTRON MICROSCOPY

O.C. Wells  
McGraw Hill (1974)

## PRACTICAL SCANNING ELECTRON MICROSCOPY

J.I. Goldstein, H. Yakowitz  
Plenum Press (1975)

## SCANNING ELECTRON MICROSCOPY IN BIOLOGY

R.G. Kessel, C.Y. Shih  
Springer Verlag (1976)

## QUANTITATIVE SCANNING ELECTRON MICROSCOPY

D.B. Holt, M.D. Muir, P.R. Grant, I.M. Boswarva  
Academic Press (1974)

## INTRODUCTION TO BIOLOGICAL SCANNING ELECTRON MICROSCOPY

M.A. Aayat  
University Park Press, Baltimore (1978)

Topics relevant to the use of the scanning electron microscope are covered in the indicated parts of:

## "PRACTICAL METHODS IN ELECTRON MICROSCOPY"

Ed. Audrey M. Glauert, North Holland Publishing Co. (1974 onwards)

Vol. 1, Part I	Specimen Preparation in Materials Science P.J. Goodhew
Vol. 2,	Principles & Practice of Electron Microscope Operation A.W. Agar, et.al.
Vol. 3, Part I	Fixation, Dehydration and Embedding of Biological Specimens A.M. Glauert
Vol. 4,	Designing the Electron Microscope Laboratory R.H. Alderson
Vol. 5, Part II	X-ray Microanalysis in the electron microscope J.A. Chandler

**Table 5.9**  
**General Textbooks**

There are a number of conferences held throughout the world for which proceedings are published. The most important of these are the proceedings of the IITRI conference, edited by O. Johari and published by IIT Research Institute, (IITRI), Chicago. Known as "Scanning Electron Microscopy, 19.." (1968 onwards) they are generally regarded as one of the best sources of papers on S.E.M., usually containing extensive bibliographies. (Recent volumes have been issued by Scanning Electron Microscopy Inc., and may be so listed in libraries). In the Table 3 references these proceedings are referred to as "IITRI/68" etc.

There were also a series of "Stereoscan" Colloquia held in the USA from 1968 onwards, for which proceedings were published by Engis Equipment Co. Inc., or Kent Cambridge Scientific Inc.

Other international conferences have been:-

Electron Microscopy, 1970 (7th International Conference, Grenoble - French Society of Electron Microscopy)

Electron Microscopy 1972 (EMCON 72 - 5th European Congress on Electron Microscopy, Manchester, England - Institute of Physics)

Electron Microscopy 1974 (8th International Congress on Electron Microscopy, Canberra, Australia - Australian Academy of Science)

Electron Microscopy 1976 (6th European Congress, Israel - Tal International Publishing).

Electron Microscopy 1978 (9th International Congress on Electron Microscopy, Toronto, Canada - Microscopical Society of Canada).

The Electron Microscopy and Analysis Group (EMAG) of the Institute of Physics, London, England, also hold conferences and publish proceedings, e.g:

- |         |  |
|---------|--|
| EMAG 71 | Electron Microscopy and Analysis (Inst. Physics)                                     |
| EMAG 73 | Scanning Electron Microscopy: Systems and Applications. (Inst. Physics)              |
| EMAG 75 | Developments In Electron Microscopy and Analysis, Ed. J.A. Venables (Academic Press) |
| EMAG 77 | Developments in Electron Microscopy and Analysis, Ed. A.L. Misell, (Inst. Physics)   |

The Electron Microscopy Society of America also publish proceedings, e.g.

- |         |   |
|---------|---|
| EMSA 76 | 34th Annual meeting of EMSA (Claitor's Publishing Division) |
|---------|---|

Abstracting journals may also be useful, e.g:

Bulletin Signaletique Section 761 - Microscopie Electronique  
CNRS/EMSA Rue Boyer 26, 75971 Paris.

**Table 5.10**  
**Conference Proceedings**

**Table 5.11**  
**Specific References**

**5.11.1 General Papers on Scanning Electron Microscopy**

1. The SEM - Principles and Applications  
D. Joy  
IITRI/73 743-750
2. Fundamentals of the SEM  
R.E.W. Pease  
IITRI/71 9-16
3. Introduction to Scanning Electron Microscopy  
W.C. Nixon,  
IITRI/69 1-10
4. Fundamentals of SEM for biologists  
M.D. Muir  
IITRI/74 1011-1018
5. Fundamentals of SEM for physicists  
R.F. Greer  
IITRI/76 669-674
6. How to get the best from your SEM  
G.E. Pfefferkorn, A. Boyde, R. Blaschke  
IITRI/78/1 1-12
7. The physics of the SEM for biologists  
D.C. Joy, C.M. Maruszewski  
IITRI/78/2 379-390
8. X-ray analysis of biological specimens  
J.R. Coleman  
IITRI/78/2 911-926

**5.11.2 Specimen Coating**

1. The rationale and mode of application of thin films to non-conducting materials.  
P. Echlin, P.J.W. Hyde  
IITRI/72 137-146
2. A cool sputtering system for coating heat-sensitive specimens  
P.N. Panayi, D.C. Cheshire, P. Echlin  
IITRI/77/1 463-470
3. Coating techniques for scanning electron microscopy and X-ray microanalysis  
P. Echlin  
IITRI/78/1 109-132
4. S.E.M. of materials without conductive coatings  
R.S. Gerdes  
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J.A. Murphy  
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I.K. Arenberg et. al.  
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A. Boyde,  
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11. Specimen preparation techniques  
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A. Boyde  
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A.J. Saubermann, W. Riley, P. Echlin  
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A.W. Robards, P. Crosby  
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A.P. MacKenzie  
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Periodical and journal articles relating to scanning electron microscopy are as wide-spread as its varied applications would suggest. The reader will know best which specialist publication to consult within his own discipline. General articles, and those relating to equipment and accessory principles and design, are to be found in publications such as the following:

Journal of Physics D: Applied Physics  
Journal of Physics E: Scientific Instruments  
both published by the Institute of Physics

Journal of Microscopy  
published by Blackwell Scientific Publications, Oxford, for the Royal Microscopical Society

Proceedings of the Royal Microscopical Society

Scanning - International Journal of Scanning Electron Microscopy & Related Methods  
published by Gerhard Witzstrock Publishing House, Inc. N.Y. & Baden-Baden  
(from 1978 onwards)

**Table 5.12**

**Periodicals**

## CHAPTER 6 INSTALLATION

### CONTENTS

#### 6.1 INSTALLATION

Initial installation of the Stereoscan 200 should only be attempted by a Cambridge service engineer or an approved Cambridge agent. Any attempt by any other person may invalidate the warranty.

The standard S200 is configured for a power supply of between 110V and 250V, single phase. If it is required to run the instrument from two phase power it may be necessary to add an additional fuse into the instrument "neutral" line. For details of this see section 6.1.

These instructions are included mainly for the use of the approved installation engineer, but may be of some use to a suitably qualified user who wishes to move his S200 from one place to another.

When removing any packing material, check that it has not been used as a place for hiding some small, vital part of the S200.

Do not throw away **any** packing material until the installation has reached a successful conclusion.

**IF IN DOUBT CONSULT AN APPROVED REPRESENTATIVE OF  
CAMBRIDGE INSTRUMENTS LTD**

List of Illustrations

Fig 6.1 Floor Plan

## 6.1 INSTALLATION

Check that the room that you are going to install the S200 in meets the conditions set out in the STEREOSCAN 200 INSTALLATION RECOMMENDATIONS.

Unpack all packing crates and carefully check their contents against the packing list.

Check all packing crates really are empty. Store them somewhere until installation is complete.

Remove all external wrapping material from the electronics console and the column and plinth.

Position the electronics console in its correct position, and put the plinth on the left hand side of the console. (See figure 6.1).

Open stage and remove transit clamp and packing.

Fit head amplifier assembly to rear plate of the chamber. Remember to fit the insulating gasket under the assembly and the insulating bushes under the screws to ensure that the head amplifier is not in electrical contact with the chamber.

Remove the gun transit clamp. Open the gun and fit the high KV anode onto the top of the lens polepiece assembly. Close the gun (the high KV anode is the shorter one of the two supplied).

Remove the foam rubber packing from between the fan and the turbomolecular pump.

Remove the four plinth suspension system clamp bolts and spacers.

**THE TRANSIT BOLTS AND SPACERS MUST BE REFITTED IF THE INSTRUMENT IS MOVED.**

If the plinth is moved without the transit spacers fitted the rubber suspensions may be damaged.

Remove any sealing material from the rotary pump, air admit and turbo pump inlets. Locate the rotary pump and its vibration isolation block either behind the S200 or on its left hand side. Connect one of the vacuum hoses from the block to the rotary pump, the other from the block to the turbo pump. The "O" rings, carriers and clamp rings are attached to the isolation block.

Connect the rotary pump outlet to the outside world with a suitable exhaust pipe. For details of a suitable pipe see the STEREOSCAN 200 INSTALLATION RECOMMENDATIONS.

Remove protective packing from all cables in the plinth.

Slide back the two catches (one each end) securing the control unit in the console and lower the control unit to its servicing position. Remove all packing material from inside the control unit.

Remove all protective packing from the cables in the control unit and the lower part of the console. Remove any other packing material you find.

When connecting the cables between the console and the plinth they must be correctly routed. Those going into the control unit enter the console through the top cable inlet, those going to the EHT set and power supply going through the lower cable inlet. The majority of cables are permanently connected at their sources, they only need to be connected to their destinations.

Install the following cables:

Cable No	Source	Destination
7	\rotary pump	vacuum chassis
42	\DSA	column
43	\head amplifier	video PCB
49	\column	electron optics PCB
56	\column	column scan amp
57	\column	column scan amp
58	\column	column scan amp
59	\column	column scan amp
60	\gun	EHT set filament
61	\gun	EHT set filament
62	\gun	EHT set grid
66	\EO PCB	column
67	\vac chassis	EHT set
67B	xvac chassis	ion pump control
79	\head amplifier	regulator PCB
82	\head amplifier	EHT set
87	\scan processor	column
132	xvac chassis	ion pump control
134	\chamber	EHT set
142	\plinth	mains unit

Check that all connectors in the control unit are correctly connected.

Check that all other cables in the console and the plinth are correctly connected.

Fit the plinth rear panel and connect the air admit hose from the vent valve to the dessicator. Check that the dessicant is still a deep blue colour. Replace it if it has turned pink.

Check with the customer to see if it is necessary to install the "neutral" fuse modification (see separate page "THE NEUTRAL FUSE").

Connect the power cable no 1 to a suitable source of power.

Obtain a working vacuum as described in chapter 1 routine 1.

Remove the turbo pump clamp by removing the 4 screws at the base of the turbo pump and the two M6 screws at the rear of the plinth.

Adjust the plinth and console positions so that they are positioned as in figure 6.1.

Screw down the plinth feet so that the castors are just above the floor. Screw down the console jacking feet so that the console and plinth tabletops are level (when fitted).



Check the positions of the console and plinth again.

Fit all panels to the machine. Check that no panels are touching the floating platform and that nothing is shorting out the anti vibration mounts.

Raise the control unit to its working position, fit the console desktop and control unit end cheeks. Begin the performance checks.

Details of how the controls should function are contained in chapter 2.

## THE NEUTRAL FUSE

The standard S200 is supplied with overcurrent protection in the "live" power connection only. Under certain local conditions it may be necessary to run the instrument from a power source which requires a fuse to be installed in the "neutral" line.

### INSTALLATION

1. CHECK THAT THE S200 IS NOT CONNECTED TO ANY EXTERNAL SOURCE OF POWER.
2. Remove the lower front panel from the console. Slide out the mains unit and remove its cover. (It may be necessary to uncable the mains unit to gain access.)
3. Locate wire Blue 10 on the back of fuse FS3. Remove it from the back of FS3 and connect it to one of the tags on the side of FS3. Do not move any other wires from the back of FS3.
4. Similarly, move Blue 12 from the back of FS5 to the side of FS5.
5. Move Blue 11 from the back of FS7 to the side of FS7.
6. Fit fuses as follows:

Fuse No	240V	Voltage	110V
FS3	10A		20A
FS5	5A		10A
FS7	10A		15A

7. Fit the top on the mains unit, cable it up and replace it in the console.

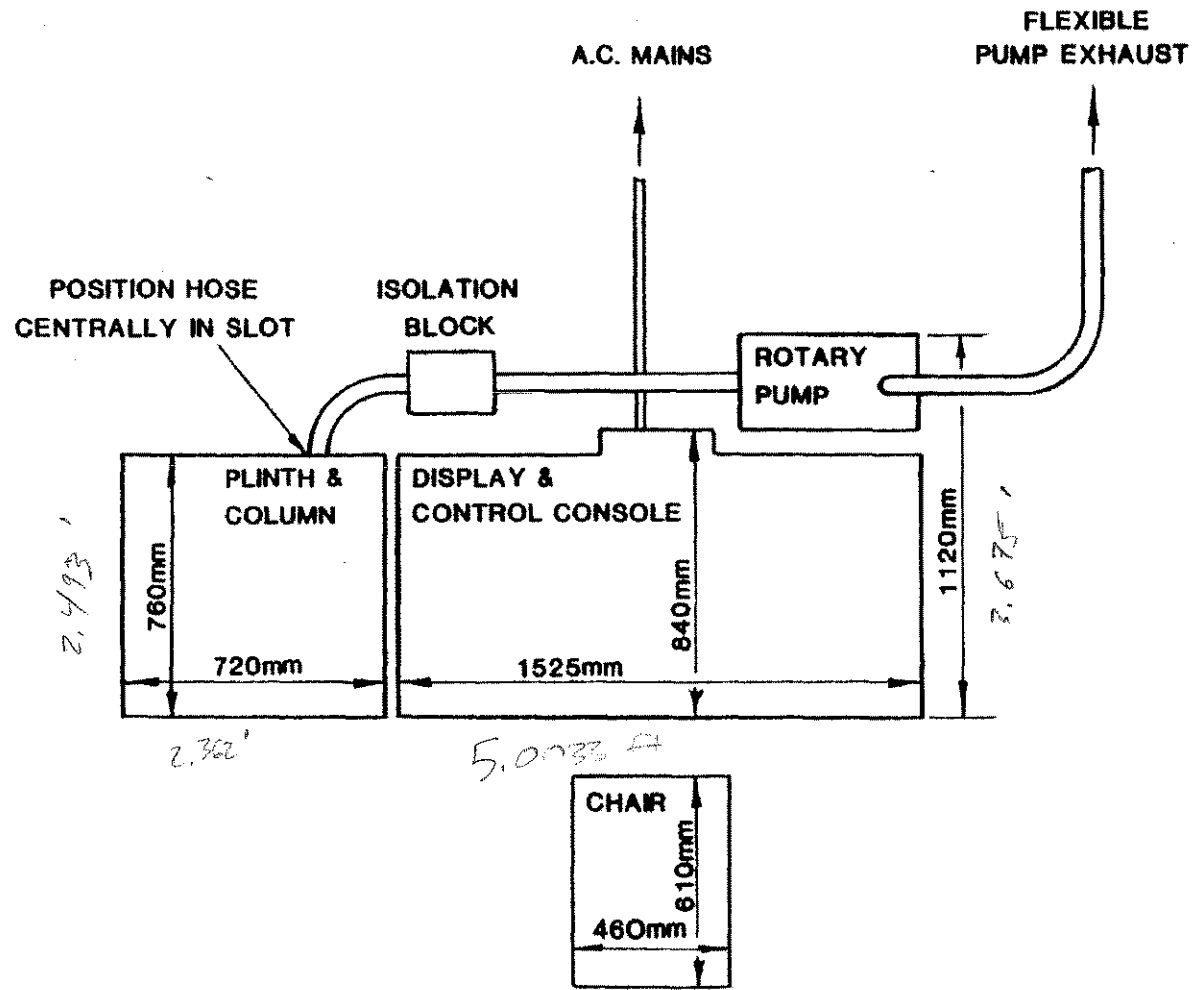


Fig. 6.1

S200 FLOOR PLAN

## CHAPTER 7 THE SCANNING ELECTRON MICROSCOPE

### Contents

- 7.1 What an SEM is.
- 7.2 How it Works.
- 7.3 What it can do.

### List of Illustrations

- Figure 7.1 A Basic SEM
- Figure 7.2 The Electron Gun
- Figure 7.3 The Lens
- Figure 7.4 Stigmators
- Figure 7.5 Scanning
- Figure 7.6 Electron Collection

## 7.1 What an SEM is.

The basic function of a Scanning Electron Microscope, or SEM, is to produce an image of three dimensional appearance derived from the action of an electron beam scanning across the surface of a specimen. The size and shape of features on the surface of solid bulk samples can be examined, as can surfaces whose roughness renders their observation very difficult or impossible by light optical or transmission electron microscopy. The resolution is better than 4nm under suitable conditions, with a depth of focus that is at least 300 times greater than that of a light microscope at the limit of resolution. The SEM can have a magnification range from a few times (typically times 10) to several hundred thousand times. The upper magnification is limited only by the resolution available. Samples as large as 7 inches in diameter can be handled routinely, without being damaged by the SEM.

## 7.2 How it works

The basic operating principle of an SEM is shown in figure 7.1. It can be seen that the SEM consists of five main components.

These are:

1. **THE ELECTRON GUN** produces a large, high intensity electron beam which is fitted into
2. **THE COLUMN** which controls and shapes the beam into a size useable for scanning microscopy.
3. **THE SCANNING SYSTEM** scans the beam over the sample in a television type raster. The beam scanning over the sample releases electrons from it.
4. **THE ELECTRON COLLECTOR AND DISPLAY** collects these electrons and converts them to an image which can be viewed by you, the operator.
5. **THE CONTROL ELECTRONICS** allows you to control the performance of items 1 to 4.

A more detailed description of how the SEM works will now be given. It is not necessary to fully understand this at this stage, and many operators may prefer to leave further study until some operating experience has been gained.

### 7.2.1 The electron gun (see fig 7.2)

The electron gun, at the top of the electron optical column, produces a beam of electrons with an effective source diameter of about 30 microns. The electrons are emitted from a heated tungsten filament or a piece of Lanthanum hexaboride (LaB<sub>6</sub>). The emitted electrons are accelerated towards the specimen by the acceleration voltage applied to keep the filament at a high negative potential with respect to the earthed anode. The current in the electron beam is controlled by a bias voltage applied to the grid. This beam is not small enough to produce the definition required in an SEM.

The necessary reduction of beam diameter is done in an electron optical system, called the column.

### 7.2.2 The column

The column consists of three electromagnetic lenses acting on the electron beam. The action is very similar to that of a light source shining into a convex lens producing an image of smaller diameter than the original source (fig 7.3). The successive action of the three lenses produces the required diameter of beam to scan the specimen. Unlike optical lenses the SEM lenses can easily be changed in strength to produce any required beam diameter. This is done by changing the current flowing in the lens coil. The first two lenses (mechanically combined into a single unit in the S200) produce most of the beam demagnification while the third lens focuses the beam onto the sample surface.

Also in the column is a beam shaper called the stigmator. The beam, by the time it reaches the sample, may not be exactly circular causing a phenomenon known as astigmatism. Astigmatism is present when the image is in focus in one axis but is smeared in the direction perpendicular to this. The stigmator can create a magnetic field around the beam to restore it to its original circular cross section, so removing the astigmatism from the image (see fig 7.4).

For best performance the slenderness of the cone of electron illumination reaching the sample needs to be controlled. This "angular aperture" which is very similar in many ways to the variable aperture in a camera, is obtained by projecting a cone of electron illumination through a defining aperture called the final aperture.

The size of the electron beam on the sample is controlled by the condenser lenses, and the strength (or focal length) of these is determined by the current flowing through the lens coil. Low lens currents give a weak, long focal length lens giving a large, high intensity beam. The image produced by such a beam has a very low noise content (it looks "clean") but is of limited resolution. This condition is best for low magnification images, X-Ray analysis and cathodoluminescence.

As the lens current is increased the beam diameter becomes smaller and the potential image resolution increases, allowing higher magnification images to be obtained. But the noise content of the image also increases, making the image more difficult to see. It is like looking through a snow storm. Fortunately slow scan rates as used for photographing images can overcome this problem.

### 7.2.3 The scanning system

To produce an image on the display the beam must be scanned over the specimen and the display tube in

synchronism (figure 7.5). Information coming from any point on the sample can then be reproduced in the same relative position on the display, building up a point by point reproduction of the sample surface. The magnification of the displayed image is defined as

$$\frac{\text{linear dimension of scan on the display}}{\text{linear dimension of scan on the sample}}$$

Since the display is of fixed size, the magnification is controlled by varying the scan size on the sample.

Various speeds of scan are required, fast for flicker free viewing, slow for optimum photographic results, and these are provided by the scanning system electronics.

#### **7.2.4 Electron collection and video processing. (fig 7.6)**

When the sample is struck by the electron beam many things happen. Some of the incident beam will be reflected as high energy reflected electrons, and some will be absorbed by the specimen, flowing to ground through the specimen current contact. The sample will emit low energy secondary electrons. It may also give off X-Rays and light. All of these things can be collected by some form of collector system and used by the SEM to provide you with information about the sample, but the main one considered here is electron emission, collected by the electron collector.

The electrons leaving the sample are first attracted to a grid biased positively with respect to the specimen. This bias increases the collection efficiency for secondary electrons. The grid voltage may be held at zero volts to decrease the collection of secondary electrons, resulting in an image formed mainly of reflected primary electrons. After passing through the grid the electrons are accelerated onto a scintillator, biased at 12KV where they cause light to be emitted. The light is optically coupled to a photomultiplier, or PM, which converts the light to electrons and amplifies them. The signal from the PM is further amplified by the head amplifier and video amplifier before being used to modulate the intensity of a cathode ray tube display.

#### **7.2.5 The control electronics**

This contains all circuits necessary to control an SEM. What these electronics consist of is not really of any use to the SEM operator, all he or she needs to know is how to use the controls provided to drive the SEM and this will be fully explained in chapter 2 of this manual. If you are really interested in how the control electronics work, full details can be found in the S200 technical manuals.

### **7.3 What it can do**

The basic function of an SEM is to produce on a cathode ray tube an image of three dimensional appearance, derived from the action of an

electron beam scanning the surface of a specimen. This technique is fundamentally different from the methods of imaging used in optical or transmission electron microscope.

Many different types of information can be obtained from the sample.

#### **7.3.1 Topography.**

The most common use of the SEM is in the study of the shape and size of the specimen surface, called topography. This is done using secondary electrons, emitted from the sample surface with low energy when it is hit by the electron beam. The topography of areas as large as 23mm x 18mm can be presented on a single micrograph. Details as small as 4 Nanometers can be micrographed. (One Nanometer is one millionth of a millimeter.)

#### **7.3.2 Elemental analysis.**

All samples will emit X-Rays when struck by the electron beam. These X-Rays are characteristic of the element from which they were emitted. By the use of special detectors sensitive to X-Rays the sample may be analysed for its constituent elements. The results may be either a plot of the concentration of all the elements present, or the spatial distribution of a chosen element on the sample surface. Particles as small as 1 Micron may be analysed. (A Micron is one thousandth of a millimetre.)

#### **7.3.3 Cathodoluminescence.**

Light is generated in some samples by the electron beam hitting electroluminescent material within it. A special detector can look at light being emitted from these samples, giving an image of the spatial distribution of such material in the sample.

#### **7.3.4 Specimen current.**

All specimens absorb some of the electron beam incident on them. These electrons normally flow out of the sample to ground through the specimen stage earth. If a specimen current amplifier is put in this earth return, images may be formed of the specimen current. This can provide valuable information about what is happening below the surface of the sample. In the case of semiconductor samples e.g. integrated circuits, it may be the only method available for getting such information.

#### **7.3.5 Transmission.**

If a thin sample is mounted on a suitable specimen stage, with an electron collector mounted below the sample, then an image can be formed of the electrons transmitted through the sample. Although the resolution of an SEM working in the transmission mode is not as good that of a dedicated transmission electron microscope, it can give useful pictures of much thicker samples.



### 7.3.6 Backscattered electrons.

Some of the electron beam incident on the sample will be reflected back from it, the amount reflected depending on the atomic weight of the part of the sample reflecting them. These are called backscattered electrons. They can be collected and used to provide images of the variations of atomic number in the sample surface.

Various types of backscatter electron detectors are available (Annular, 4 element, scintillator etc) and any type may be fitted to an S200.

### 7.3.7 Others.

As scanning electron microscopy develops new detectors and methods of retrieving and processing information are being made available. The above represents the most commonly used. Information on these, and other related topics can be obtained from the publications listed in the bibliography in chapter 5 of this manual.

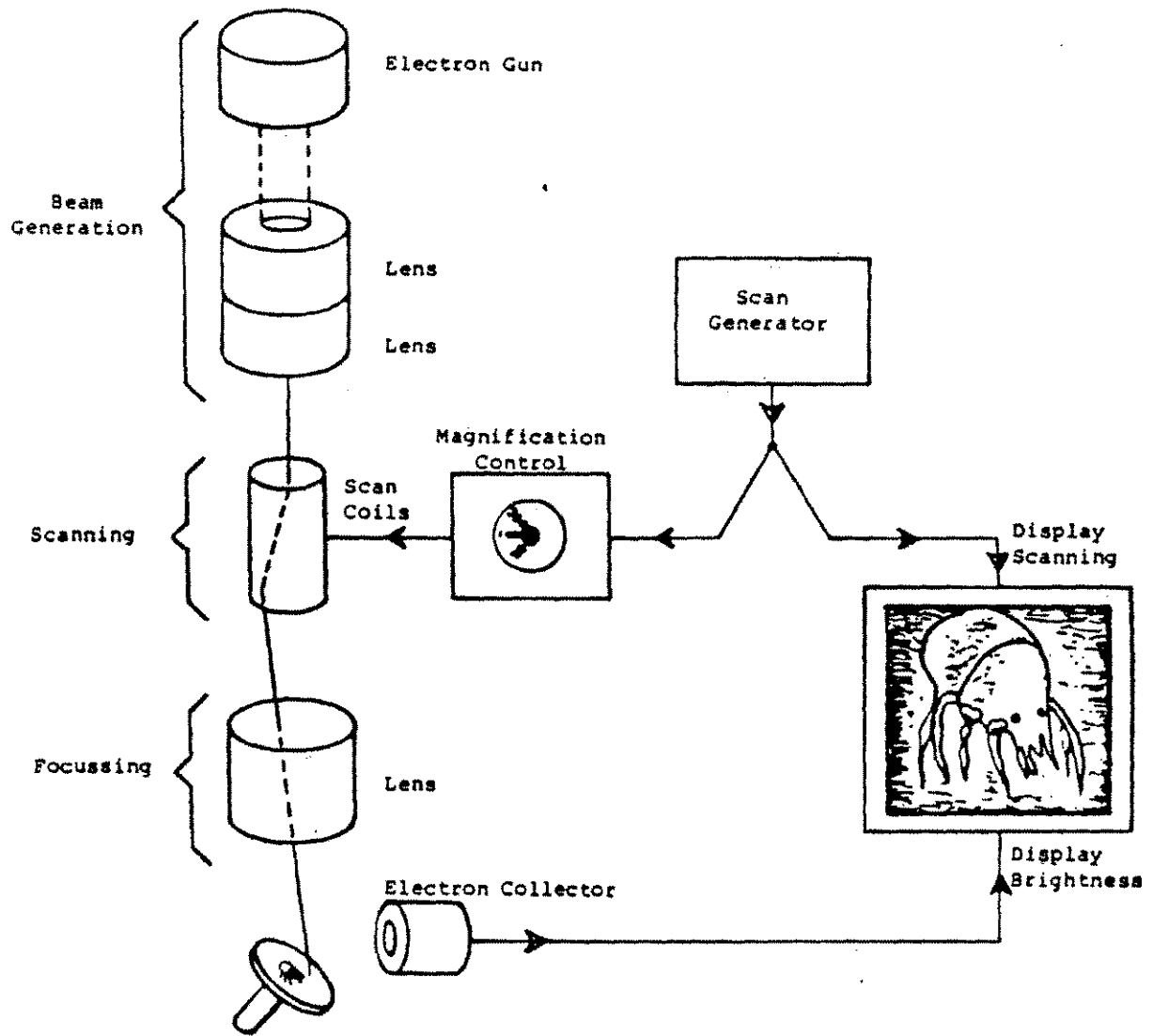


Figure 7.1  
Basic SEM

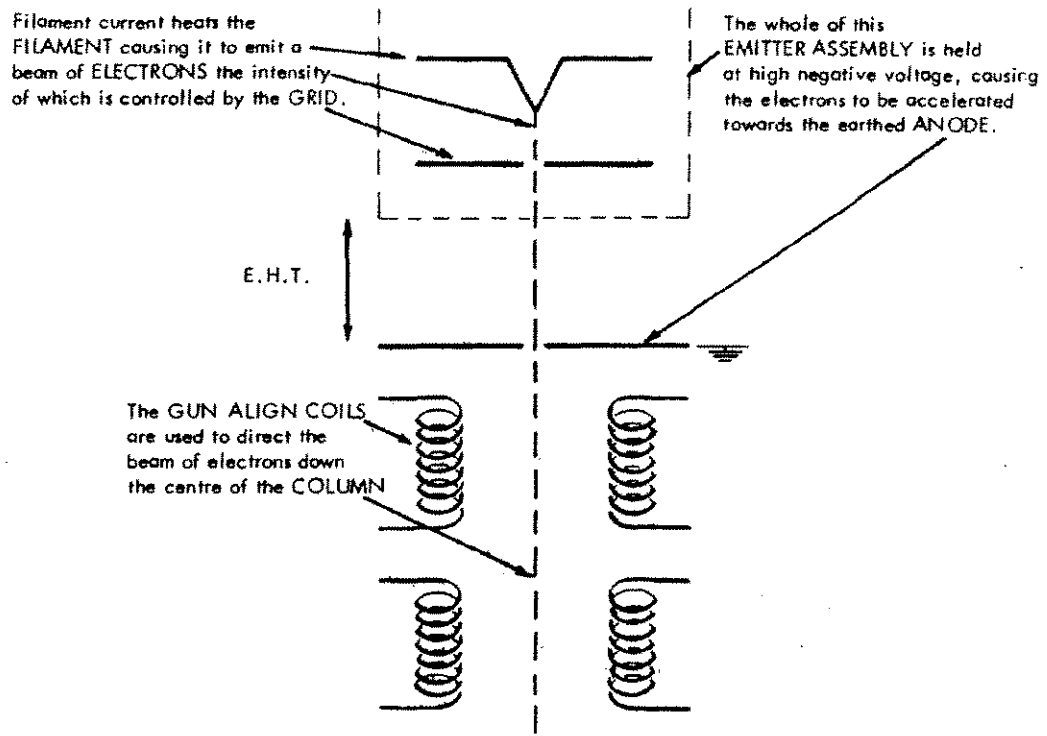


Figure 7.2  
Beam Generation

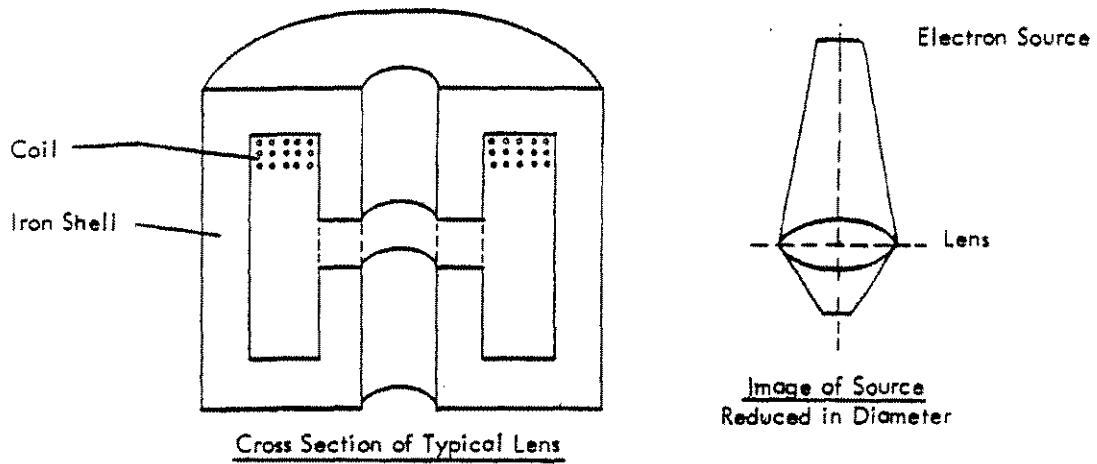


Figure 7.3

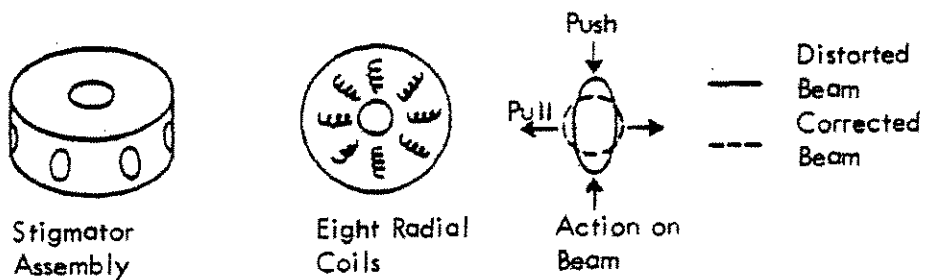


Figure 7.4

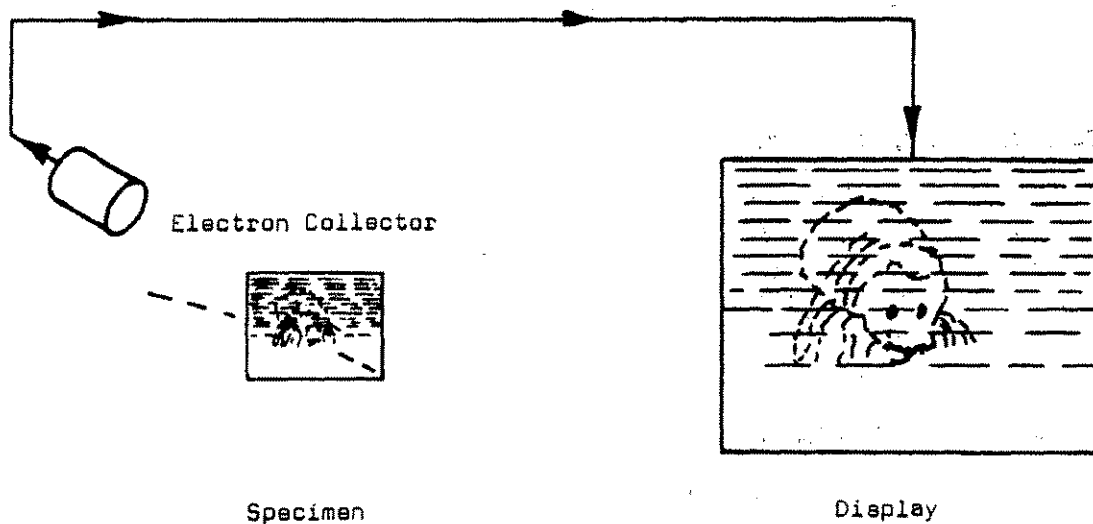
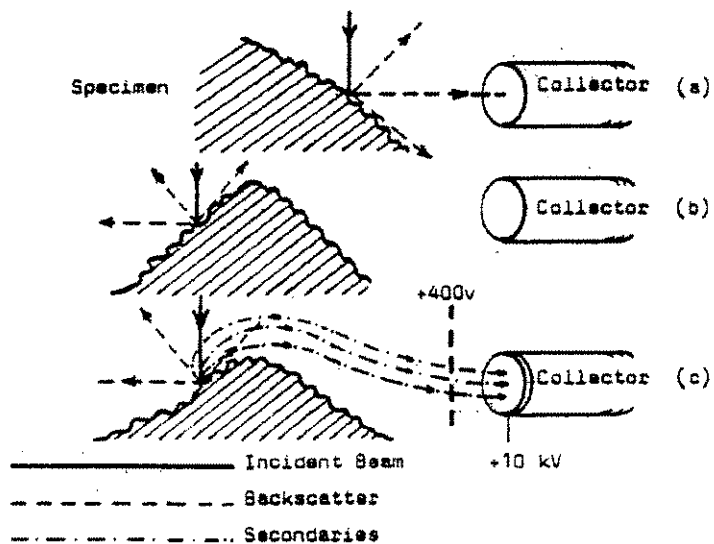
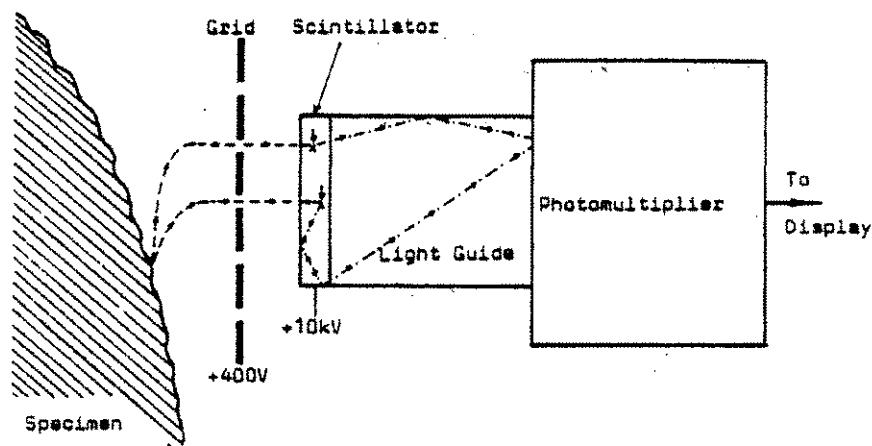


Figure 7.5



Electron Collection



Electron Collector

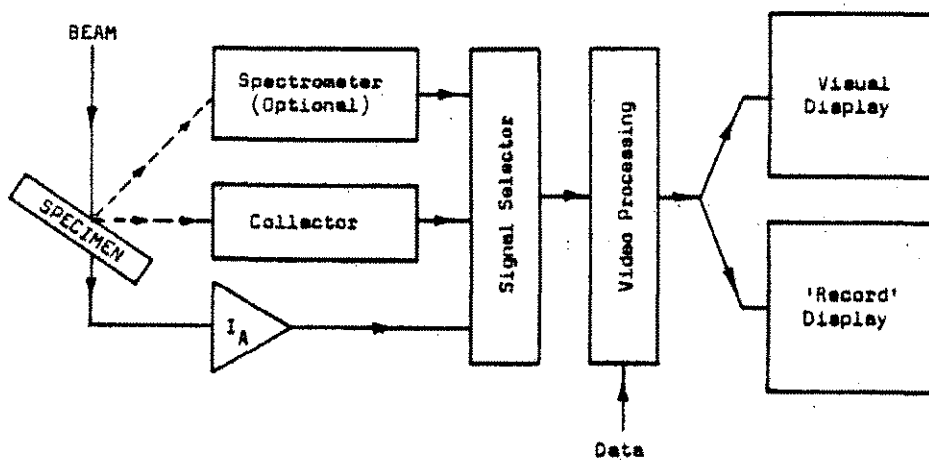


Figure 7.6 Typical Use of Basic Video System