INSTRUCTION MANUAL

FOR

SCANNING ELECTRON MICROSCOPE

MODEL: SIII-A(K)

IT IS IMPERATIVE THAT ALL OPERATORS OF THE SIII-A(K) READ THIS MANUAL THOROUGHLY BEFORE OPERATING THE EQUIPMENT.

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SUPER IIIA(K) SCANNING ELECTRON MICROSCOPE

1. INTRODUCTION

The SUPER IIIA(K) is a high performance Scanning Electron Microscope. Its fundamental performance is similar to that of any other SEM, however, maximum versatility, operational ease and simplicity of installation place it in a category by itself. The SUPER IIIA(K) functions on the following principle: An electron beam emitted from the electron gun is accelerated by a high voltage, focused by a three-stage electromagnetic lens system, and scanned over the specimen surface. Secondary electrons, backscattered electrons, and x-rays(optional) collected by their respective detectors are converted to video signals; these signals from the surface of the specimen are displayed on the cathode ray tubes (CRT) and magnified electronically in synchronization with the scanning beam in the electron optical column.

This manual contains instructions on the operation, service, and trouble shooting of the instrument and is arranged so that an inexperienced operator can understand all aspects with ease.

It is imperative that all operators read this manual carefully before attempting to operate the instrument. Errors in operation can cause temporary loss of performance capability and result in the need for time consuming maintenance. Even the experienced microscopist must make himself familiar with the characteristics of the SIIIA(K) so as to avoid operational errors and so as to obtain optimum results.

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2. CONTROL FUNCTIONS

It is important that this section is studied thoroughly prior to operation of the SUPER IIIA. The following information is intended to familiarize the operator with the control functions of the instrument.

Section	Control	Function
Column	Gun alignment (2)	Centers electron beam to column.
Column	Specimen stage controls. NOTE: Refer to pages 5-19, 5-20 for limitations.	Moves sample under electron beam.
Column	Stage clamp	Clamps stage to minimize vibration when working at high magnification.
Column	Vacuum system	Operation - cycles vacuum valve automatically to operating vacuum (#3 valve position). Shut - cycles vacuum valve to shut down position (#1 valve position). Air - cycles vacuum valve to emit air into column for sample change.
Display Console (right side)	Power	Applies power to vacuum circuits.
Display Console (right sid e)	Rotary Pump (RP)	Applies power to mechanical pump.
Display Console (right side)	Diffusion Pump (DP)	Applies power to diffusion pump heater.
Display Console (right side)	Operation	Applies power to all electronic circuits.
Display Console (right side)	Emission (manual)	Manually applies power to electron gun filament.

Section	<u>Control</u>	Function
Display Console (right side)	Meter Selector	Selects vacuum, emission or working distance reading on meter.
Display Console (right side)	Spot Size	Changes current in first condenser lens (clockwise decreases brightness of electron beam).
Display Console (right side)	Working Distance	Changes course range of objective lens current to achieve focus throughout working distance range.
Display Console (right side)	Focus (fine and coarse)	Changes objective lens current (focuses electron beam on sample).
Display Console (right side)	Alignment (X and Y)	Electromagnetically aligns electron beam to electron optics axis.
Display Console (right side)	Stigmator (X and Y)	Compensates for astigmatism in electron beam.
Display Console (right side)	Dynamic Focus	Corrects for defocusing at high tilt angles and low magnification. NOTE: This control should only be used at magnifi- cations of 5000X and lower.
Display Console (right side)	High Voltage (kV)	Selects accelerating voltage.
Display Console (left side)	Signal Mode Selector	See following explanation.
	Secondary Electrons (SE)	Couples secondary electrons to CRT when depressed.
	Backscattered Electrons (BSE)	Couples backscattered electrons to CRT when depressed.
	X-Ray	Couples x-ray pulse to CRT when depressed.

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Section	<u>Control</u>	Function
	Line Profile (LP)	Couples x-ray ratemeter signal to vertical axis of CRT when depressed.
	Spot	Disables scan on CRT and in column when depressed. (This mode is used with position controls).
	Line	Disables vertical scan on CRT and in column when de- pressed. This mode is used for positioning the horizontal scan with the Y position control for selecting area of interest for x-ray profile.
Display Console (left side)	<u>Auto Contrast/Brightness</u> (OPTIONAL)	See following explanation.
	Time Constant	Selects response time of ACB circuit.
	Contrast	Changes contrast of image when ACB is engaged.
Display Console (left side)	<u>Dual Magnification</u> (OPTIONAL)	See following explanation.
	Selector Switch	Xl - split screen imaging. Same magnification on both sides of CRT.
		X3 - split screen imaging. Magnification on right side of CRT is 2 times greater than left side.
		X5 - split screen imaging. Magnification on right side of CRT is 5 times greater than left side.
		X10- split screen imaging. Magnification on right side of CRT is 10 times greater than left side.

Section	<u>Control</u>	Function
	Postion X-Y	Positions window on left side of CRT (X3, X5, X10) that selects field of view displayed on right side of CRT.
Display Console (Left Side)	Scan Mode	Changes vertical and horizontal sweep speeds.
Display Console (Left Side)	Reduced Area	Provides reduced area scan when depressed and scan mode selector is in rapid position.
Display Console (Left Side)	Image Shift X and Y	Electronically moves raster on sample.
Display Console (Left Side)	Micron Marker	Displays micron marker on CRT.
Display Console (Left Side)	Start	Starts single sweep when scan mode is in Photo.
Display Console (Left Side)	Contrast	Changes contrast of image.
Display Console (Left Side)	Brightness	Varies brightness of image.
Display Console (Left Side)	Zoom	Continuously varies magnification
Display Console (Left Side)	Magnification	Changes magnification in 24 steps.

3. VACUUM SYSTEM START UP PROCEDURE

3.1 Before turning instrument on, <u>check that the controls are in the</u> positions indicated below. Controls and switches not mentioned can be set in any position.

Section	Control	Position
<u>Colum</u> n	Vacuum System	Shut YELLOW ON COLUMN ~
Display Console (right side)	Main Power	Off (lower half depressed)
Display Console (right side)	Diffusion Pump (DP)	Off (lower half depressed)
Display Console (right side)	Operation	Off (lower half depressed)
Display Console (right side)	Emission (manual)	Full counterclockwise
Display Console (right side)	Auto emission	Off
Display Console (right side)	Spot Size*	Set at 12:00 to 2:00 o'clock
Display Console (right side)	Alignment (X and Y)*	Set at 12:00 o'clock
Display Console (right side)	Stigmator (X and Y)*	Set at 5.0 on vernier dials
Display Console (right side)	Dynamic Focus	Zero
Display Console (left side)	Brightness*	Set at approximately 12:00 o'clock
Display Console (left side)	Contrast*	Set at approximately 12:00 o'clock
Display Console (left side)	Scan Mode	Rapid (reduced area or full area)
Display Console (left side)	Magnification	Set at low magnification

Section	Control	Position
Display Console (left side)	Secondary Electron (SE)	Depress
Display Console (left side)	Auto Contrast/Brightness (OPTIONAL)	Off
Display Console (left side)	Dual Magnification (OPTIONAL)	Off

*Once the position of these controls has been established for routine operation, they can be left in this position and need not be returned to the positions indicated for initial start up.

3.2 Check that the AC power cable is plugged in properly to a 115VAC receptacle (capable of supplying at least 1.5kVA).

NOTE: Before connecting the input power cable, the operator should check the line voltage. It can easily be checked by measuring voltage on the AC receptacle located at the rear panel of the display console.

> The line voltage must be within a range of 98V to 105V. If the line power voltage is not within this range, disconnect the input power cable from the outlet and adjust the tap of the stepdown transformer to obtain the required voltage.

3.3 Turn on the cooling water for the diffusion pump (DP) and make sure cooling water flows at about 1/2 gallon per minute.

- 3.4 Depress the upper half of the POWER switch to energize the vacuum system circuit. The red switch lamp comes on. Depress the RP (rotary pump) switch (this switch automatically returns to its original position when released) to energize the rotary pump for evacuation.
- 3.5 Depress the upper half of the DP switch. The red switch lamp comes on. Wait for about 15 minutes for warming up of the diffusion pump.
 3.6 Depress the VACUUM CONTROL OPER. switch. The rotary pump starts evacuating the microscope column. When the vacuum meter reads 50 to 80 (meter detector to vacuum), the valve automatically cycles to high vacuum.
- 3.7 When the vacuum meter reaches the green range, and the green pilot lamp (vacuum indicator lamp) comes on, the instrument is ready for operation. However, it is recommended to wait a few more minutes before turning on operation switch.

IMPORTANT:

 When power failure occurs, be sure to turn OFF the OPERATION switch, DP switch and POWER switch respectively, and depress the VACUUM CONTROL SHUT switch. In this condition, wait for about 15 minutes until the diffusion pump is cooled sufficiently. After the line power is restored again, proceed as instructed in 3.4 through 3.7 above for starting again.

2. When water flow stops, a thermostat is actuated to automatically de-energize the heater for the diffusion pump and turn off the DP switch lamp.

In such a case, be sure to turn OFF the OPERATION switch and DP switch.

Then depress the VACUUM CONTROL SHUT switch button. In this condition, wait for about 15 minutes to cool the diffusion pump. After water supply resumes, proceed as instructed in 3.5 through 3.7 for starting microscopy again.

NOTE: For resetting the thermostat, remove the rear cover of the column console and depress the thermostat pushbutton on the diffusion pump.

If the automatic valving system should malfunction, the vacuum system can be operated manually. Refer to section 23 of this manual for detailed instructions.

3.8

4. SPECIMEN EXCHANGE

- 4.1 Turn the EMISSION control fully counterclockwise. Turn OFF the OPERATION switch. The lower half of the switch is depressed and the switch lamp goes off.
- 4.2 Depress the VACUUM CONTROL AIR switch. Several seconds later, the COLUMN LEAK valve starts operating to admit air into the microscope column. Internal pressure of the column reaches the atmospheric level in about 15 seconds.
- 4.3 Turn the STAGE CLAMP knob clockwise to the UNCLAMP position on the specimen chamber and set the tilt control at 0°.
- 4.4 Unlock the snap lock located on the left side of the specimen chamber and pull the specimen chamber door toward you. Now the entire specimen chamber stage will be exposed.
 - NOTE: Do not expose the specimen chamber interior to the atmosphere for a long period of time.
- 4.5 After loosening the specimen holder set screw, pull out the specimen holder (cup 15mm in diameter) upward (see fig. 4-1), turn the R (rotating) control until the set screw is conveniently positioned for loosening.



- 1. Specimen set screws M2
- Specimen holder (cup 15mm)
- 3. Hole 6mm
- 4. Specimen holder base
- 5. Gear
- Specimen holder set scew M3

Fig. 4-1

4.6 After loosening the two specimen set screws used for fixing the specimen stub, take out the specimen stub from the specimen holder (see fig. 4-2).





- 1. Specimen
- 2. Specimen set screws M2
- 3. Specimen cup
- 4. Specimen stub
- 5. Specimen should be in same plane as top of cup.
- Fix the specimen stub (on which a specimen is attached) to the specimen holder, following the notes given below. The two specimen set screws must be securely tightened to prevent vibration during operation.
 - NOTE 1: The specimen stub must be fixed to the specimen holder so that the top surface of the specimen is aligned with that of the specimen holder on a horizontal plane.
 - NOTE 2: Mount sample on specimen stub of the correct size to fit specimen cup selected. Each specimen should be attached to the specimen stub by methods best suited to the individual specimen. Silver conducting paint is commonly used. Non-conductive specimens must be coated to prevent charging.
- 4.8 Insert the specimen holder (on which a specimen is fixed) into the specimen holder base and securely tighten the specimen holder set screw. Make sure that the screw is securely tightened to prevent vibration. The specimen holders having diameters of 15mm to 3 inches can be mounted in the same way.
- 4.9 Close the specimen chamber door and lock the snap lock.
- 4.10 Depress the VACUUM CONTROL OPER. switch to start evacuating the microscope column with the rotary pump. When the vacuum meter indicates 50 to 80 (meter selector to vacuum), the valve is auto-matically cycled to the high vacuum position.

4-12

4.7

- NOTE: Periodically check the o'ring seal between the stage and chamber for cleanliness. Lint, hair, etc. could cause a vacuum leak.
- 4.11 When the vacuum level reaches the green range, the green pilot lamp comes on. At this point the instrument is ready for operation. However, it is recommended to wait a few more minutes before turning on operation switch.

5. HANDLING OF SPECIMEN STAGE

(FOR ADDITIONAL INFORMATION, REFER TO FIG. 5-1) 5.1 **Specifications** Specimen size 76.2mm (dia.) x 25.4mm (high) Size of specimen holders (four types) (1) 15mm (dia.) x 15mm (high) (2) 31.8mm (dia.) x 25.4mm (high) (1-1/4" x 1") (3) 76.2mm (dia.) x 25.4mm (high) (3" x 1") (4) 76.2mm (dia.) x 0.5mm (high) (3" dia. for IC wafer) 5.2 Specimen shift range (1) X direction (by using the X control) 40mm (0 \sim 400 on digital counter) (2) Y direction (by using the Y control) 43mm (0 \sim 430 on digital counter) (3) Rotation (by using the R control) 360° continuous (0 \sim 99 on digital counter) 36°/division (4) Tilting (by using the T control) -10° ∿ 70° (5) Working distance Z (by using the WORKING DISTANCE control) 8 ∿ 38mm 5.3 Connectors provided (1) 1 coaxial connector for measuring specimen current. (2) Connector (having 25 terminals) for applying voltage to specimen. 5.4 Detector ports 1 for secondary electron detector 3: 1 spare 1 for x-ray detector 5.5 Specimen movement A specimen is moved by using the X, Y controls and R (rotation) control. By turning the X control, the image is

moved in the right-left direction on the cathode ray tube (CRT). When the X control is turned clockwise, the image moves from the left to the right. By turning the Y control, the image is moved in the up-down direction. When the Y control is turned clockwise, the image is shifted upward. The image can be rotated on the CRT by using the R control. By turning this control clockwise, the image is rotated counterclockwise on the CRT.

REMARKS: When the 15mm Ø specimen holder is used, specimen center is positioned at the center of the CRT by setting the X counter at 320 and Y counter at 200.

IMPORTANT: The X control should be turned only within a range of 0 - 430. When a large specimen holder is used, the specimen chamber door should be opened and closed with readings around X = 200 and Y = 200 to prevent the specimen holder from striking the specimen chamber.

5.6

Tilting of a specimen is very effective for observing convex and concave forms on its surface. For this purpose, the specimen stage can be tilted by turning the T (tilting) control.

Tilting angle is indicated taking the plane perpendicular to the electron beam as 0° (the reading is coincident with position of the T control read on its scale).

IMPORTANT: Do not turn the T control while the stage clamp knob is set at the CLAMP position. Before opening or closing the specimen chamber door, be sure to set the T control at the O position and unclamp stage.

5.7 When it is desired to use a large specimen holder or observe images at a low magnification, a long working distance should be used. The ₹ (working distance) control permits changing working distance from 8mm to 38mm. Magnification is reduced to 1/3 by changing working distance from 8mm to 38mm, for example, if the magnification dial is set to 30X, minimum magnification is 10X. NOTE: When working distance is changed by turning the ₹ (working distance) control, the correct magnification can be determined with the WORKING DISTANCE FACTOR meter.

EXAMPLE: Focus an image of a specimen, then set the meter selector switch at the WDF position and read the meter indication. Actual magnification can be obtained by multiplying the meter reading by the magnification indicated on the panel (when the meter selector switch is set at the WDF position, read indication on the magnification factor scale graduated in red). Actual magnification:

= magnification reading on panel

x magnification factor (WDF)

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Determining the magnification with the working distance factor is only accurate with the zoom control in the full clockwise or full counterclockwise position. When the zoom control is in the counterclockwise position, the magnification is X1 the magnification dial reading and when the zoom control is in the full clockwise position, the magnification is X3 the magnification on the dial reading.

IMPORTANT: The Z control should be turned only within a range of 8 \sim 38mm.

Do not turn the Z (working distance) control with the stage clamp knob set at the CLAMP position.

IMPORTANT: Specimen movement is limited for each specimen holder. Be sure to use the T, X, Y, R and Z controls within the ranges specified for them respectively below:

(1) When specimen holder 15mmØ x 15mm H is used:

(1-a) $Z = 8 \sim 15.5$ mm (useable range of the T control may be limited depending on position of the Y control; see the graph on page 5-19-20).

-	300	* ^ ^
1 .	- 103 0	. 40 .
1.	-10 1	70

- X: 15mm
- Y: 15mm
- R: 360°
- (1-b) Z = 15.5 ∿ 23mm
- T: -10° ∿ 50°
- X: 15mm
- Y: 15mm
- R: 360°

(1-c) $Z = 23 \sim 28mm$ -10° ∿ 60° Τ: Χ: 15mm Υ: 15mm 360° R: (1-d) Z = 28 \sim 38mm -10° ∿ 70° T: Χ: 15mm Υ: 15mm 360° R: When specimen holder 31.8mmØ x 25.4mm H (1-1/4" x 1") is used. (Useable range of the T control may be limited depending on position of the Y control; see the graph on page 5-19). T: $0 \sim 70^{\circ}$ Χ: 24mm Υ: 32mm 360° R: Z: $28 \, \sim \, 38 \text{mm}$ When specimen holder 76.2mmØ x 25.4mm H (3" x 1") or 76.2mmØ x 5.11 0.5mm H (3" for IC wafer) is used. (Useable range of the T control may be limited dependent on position of the Y control; see the graph on page 5-20). $0 \sim 70^{\circ}$ Τ: Χ: 40mm Y: 43mm R: 360° 28 ∿ 38mm Z:

5.10





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5.12 The terminal used for measuring specimen current is located at the right lower part of the specimen chamber door. A connector is plugged in the terminal for grounding it (for grounding the specimen holder) while the terminal is not used for measuring specimen current.

NOTE: If this plug is not connected to the terminal, specimen

5.13

is not grounded and charging of the sample will occur. For measuring specimen current, connect the furnished plug (BNC-P-55U) to a cable. Then connect the other end of the cable to the input side of a commercially available micrometer (see fig. 5-1).



- 1. Specimen chamber
- Connector for measuring specimen current
- 3. Micrometer

Fig. 5-1

5.14

Focus an image on the CRT in the procedure instructed in Section 6, "E.G. Alignment", and then depress selector switch to the SPOT position. The tilt control should preliminarily be set at the O position. Specimen current is dependent on position of the SPOT SIZE control. When the control is turned from its minimum position to maximum position, beam spot size and specimen current changes as follows:

(Accelerating voltage 25kV):

Electron beam spot size: approximately 60 Å \sim 560 Å Specimen current: approximately 3.5 x $10^{-12} \sim 1 \times 10^{-9}$ A NOTE: When a specimen stub is used in the specimen holder, only half the actual specimen current is effective.

5.15

When it is required to accurately measure specimen current, use a Faraday cup in place of a specimen stub in the specimen holder (see fig. 5-2).



1. Electron beam

2. Faraday cup

3. Specimen holder

Fig. 5-2

Referring to Section 6, "E.G. Alignment", form an image of the Faraday cup at a suitable magnification on the CRT (position the opening of the Faraday cup at the center of the CRT), depress selector switch to the SPOT position. By turning the POSITION X and Y controls, adjust the electron beam spot to the center of the opening of the Faraday cup. Measure specimen current as instructed in 5.13 and 5.14.

5.16

Terminals for applying voltage to specimen.

These terminals are to be used for observing a specimen in such a condition that a bias voltage is being applied to semiconductor or similar specimens.

At the lower left part of the specimen chamber door, a total of 25 terminals (one of which is grounded) are provided.

These terminals should be protected with the dust cap (supplied as standard equipment) when they are not used.

When using the terminals, connect a cable to the furnished plug (DB-25S) for feeding in and taking out signals.

Terminals are provided inside the specimen chamber for connections to a specimen, reference numbers which correspond to the terminal numbers are provided (see fig. 5-3).

This wiring system is electrically rated as follows:

Rated current: 1 A

Dielectric strength: DC 300 V

Insulation resistance: 5000MQ or higher



Terminals outside specimen chamber



Terminals inside specimen chamber

Fig. 5-3

5.17 Specimen Stage Clamp.

Since the microscope column console is mounted on an antivibration base, external vibrations are greatly minimized during operation at high magnification. If external vibrations should affect performance, the operator should use the specimen stage clamp mechanism built in the instrument.

The control knob (STAGE CLAMP) for this mechanism is located on the right side of the specimen chamber. The clamp mechanism becomes effective by turning the knob counterclockwise. The control knob should be turned lightly as far as it can go and should not be manipulated forcibly after it is stopped. A focus change and slight image shift will occur when stage is clamped.

NOTE 1: While the specimen stage is kept in the clamped condition, do not turn the T (tilting) control or Z (working distance) control.

The X and Y controls may be manipulated when the specimen stage is clamped.

NOTE 2: Do not close the specimen chamber door for evacuating the specimen chamber while the specimen stage is kept in the clamped condition.

Be sure that the specimen stage clamp is released before pressing the operate switch.

6. ELECTRON GUN ALIGNMENT

- 6.1 Be sure that instructions in Sections 3 and 4 have been completed and are thoroughly understood.
- 6.2 Before turning on the operation switch, check that the operating controls and switches are set as listed below:

Location	Name of Control	Setting
Display Console (right side)	EMISSION control	Fully counterclockwise
	Meter selector switch	EMISSION
	HIGH VOLTAGE control	As desired
	DYNAMIC FOCUSING control	0°
	STIGMATOR X and Y controls	5.0 on dials
	ALIGNMENT X and Y controls	12:00 o'clock
(left side)	SPOT SIZE control	12:00-2:00 o'clock
فلجبل ثلا	AUTO CONTRAST/BRIGHTNESS	OFF
	MAGNIFICATION setting	100X or lower
	DUAL MAGNIFICATION	OFF
	SCAN MODE selector switch	RAPID
	Image selector switch button	SE
· ·	CONTRAST control	12:00 o'clock

9:00-12:00 o'clock

NOTE: When the AUTO EMISSION pilot lamp is lit, AUTO EMISSION is ON and the emission control is disengaged.

BRIGHTNESS control

6.3 Turn ON the OPERATION switch by depressing its upper half. The red switch lamp comes on.

The EMISSION meter pointer gradually deflects as listed below depending on the accelerating voltage selected:

Accelerating Voltage	Emission Current
30kV	Approximately 60 µA
15kV	Approximately 30 µA
2kV	Approximately 4 uA

6.4 Gradually adjust the BRIGHTNESS control until a scanning area measuring about 5cm high x 6cm wide is observed at the center of the CRT. Adjust the BRIGHTNESS control to a comfortable level.

Turn the EMISSION control clockwise until the EMISSION meter deflection is observed. Continue turning the EMISSION control until the EMISSION meter reading no longer increases by turning the EMISSION control clockwise. This is referred to as saturating the emission current.

EMISSION meter reading in this condition varies with operation hours of the electron gun cartridge used. Normal emission current is 90 to 175 μ A. The meter reading will generally be high immediately after replacement of the electton gun cartridge but lower as it is used for a longer time.

NOTE: If the EMISSION meter does not deflect at all by turning the EMISSION control fully clockwise, the filament is burned out and the electron gun cartridge must be replaced with a new one.

6.6

6.5

While monitoring indication on the PHOTO METER, turn the gun Augument alignment controls clockwise or counterclockwise to displace the electron gun cartridge relative to the anode until the indicator reads a maximum value (the scanning area is brightest on the CRT). This adjustment should be performed by alternating turning the alignment controls which are diagonally located with each other.

CONTRAST

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NOTE: When it is hard to monitor pointer deflection on the PHOTO METER indicator (change in brightness of the scanning area on the CRT) turn the CONTRAST control clockwise and the SPOT SIZE control counterclockwise.*

SPOT SHREE

HARD TO MONTAL

If the PHOTO METER reading reads full scale (the scanning area is too bright on the CRT), turn the SPOT SIZE control clockwise and the CONTRAST control counterclockwise until the PHOTO METER indicates 40 \sim 50 μ A (proper brightness is obtained on the CRT). Then repeat step 6.6 above and confirm that reading is reduced on the PHOTO METER (the scanning area darkened on the CRT) by turning the alignment controls in any direction.

Adjust the ALIGNMENT X and Y controls until a maximum reading. is obtained on the PHOTO METER (maximum brightness on the CRT). If the ALIGNMENT X and Y controls are turned fully clockwise or counterclockwise for this adjustment, or if it is impossible to obtain maximum brightness on the CRT, repeat steps 6.5 and 6.7 once again.

NOTE: If it is impossible to properly align the electron gun cartridge with the anode, the filament is misaligned in the electron gun cartridge. Refer to "Replacement of Electron Gun Cartridge" in Section 18 and properly center the filament.

*An alternate method for aligning and saturating the filament is described in Section 7.

- 6.9 When reading does not increase any more on the EMISSION meter by turning the EMISSION control clockwise in step 6.5, the emission current is saturated. If emission signal decreases with a further increase (clockwise) with the emission control, proper alignment has not been achieved. When this occurs, repeat steps 6.5 through 6.8
- 6.10 After the alignment has been completed, turn the COARSE FOCUS control until some image becomes visible on the CRT. Set the SCAN MODE selector switch at the NORMAL position to enlarge

6.8

6.7

the scanning image over the entire range of the CRT. NOTE: If the image can not be focused by turning the FOCUS control, check the items listed below:

1. Whether the requirement specified in section 4.7 is met.

- Whether the specimen is positioned within the range specified in section 5.9.
 When the specimen stub 15mmØ is used, centering position for specimen corresponds to readings of 320 on the X counter and 200 on the Y counter.
- 3. Whether Z motion of specimen properly corresponds to position of the WORKING DISTANCE control.

For checking this item, the following table gives a general guide:

WORKING DISTANCE	Z MOTION
8mm	8mm ~ 12mm
1 5mm	12 mm ~ 19 mm
23mm	19mm ∿ 28mm
<u>38mm</u>	28mm ∿ 38mm

- 6.11 At this point, a focused image should be observed on the CRT screen. To check the alignment, turn the EMISSION control fully counterclockwise and turn OFF the OPERATION switch by depressing the lower half. The switch lamp goes out.
- 6.12 If control settings are not changed, the image can be focused again on the CRT simply by turning ON the OPERATION switch and then gradually turning the EMISSION control clockwise until the emission current is saturated.

7. **#** USE OF WAVE FORM MONITOR

7.1 To use the Wave Form monitor, turn the wave form monitor switch ON located on the rear of the display console. When the line switch is also depressed, the vertical sweep in the column and CRT is disengaged. In this condition a signal from the horizontal line is coupled to the vertical axis of the CRT. The position of the horizontal line can be changed with the Y position control to display a signal from any area in the field of view.*

- 7.2 Emission current can be saturated and the beam aligned with the use of the wave form monitor. Set the controls as described in Section 7.1 and focus the signal to obtain the sharpest peaks. Saturation is obtained when maximum amplitude of the wave form is obtained with the emission control. If the amplitude decreases when the emission control is turned further clockwise, the beam is not correctly aligned. When this occurs, leave the emission control at this position and align the beam with the mechanical controls on the column. Be sure the alignment controls (X and Y) on the control console are at the 12:00 o'clock position before aligning the beam with the mechanical controls. After the beam is aligned with the controls on the column, fine alignment can be made with the electronic alignment controls (X and Y). Alignment is completed when the amplitude of the wave form does not change when the emission control is turned beyond the point of saturation.
- 7.3 The wave form can also be used to set the correct contrast and brightness for photographing the image when the controls are set as described in Section 7.1. After a series of micrographs are taken to determine the correct exposure as described in Section 16 of this manual, engage the wave form monitor without changing the contrast or brightness controls. Make reference marks on the

*When the line switch is depressed and the wave form monitor switch is off, the horizontal line on the CRT represents the area in the field of view that corresponds to the x-ray line profile (LP).

viewing CRT identifying the upper and lower limit of the wave form as illustrated in figure 7-1. The contrast control changes

the amplitude (peak to peak) of the wave form and the brightness control shifts the wave form vertically on the screen. To reproduce correct exposure,



Fig. 7-1

position the wave form between the upper and lower limit using the contrast and brightness controls.

8. VIEWING THE IMAGE

8.1 Make sure that the procedures instructed in Section 6 have been completed.

Set the operating controls as follows:

1. Depress the SE and SE/BSE switches.

-2. Turn off AUTO CONTRAST/BRIGHTNESS, if instrument is so equipped.

3. Turn off DUAL MAGNIFICATION, if instrument is so equipped.

4. Set magnification at X100 or lower.

8.2 Turn on the operation switch. After a few minutes of warm up period, adjust brightness control to a level where the scan is just visible on the CRT. Then saturate the filament current as described in Section 6 and/or 7 and focus the image.

8.3 Set the scan mode switch to the rapid position, contrast to approximately 12:00 o'clock and the spot size to the 2:00 o'clock position for viewing the sample. The TV mode is also a convenient mode for viewing the sample. When viewing in the TV mode, however, the spot size control should be positioned at approximately 9:00 o'clock.

NOTE: When TV mode is used, the Dynamic Focus control must be at 0° .

8.4

Position the specimen for observation of the area of interest by using the X, Y controls and R control as instructed in Section 5. When it is desired to observe convex and concave forms on the specimen surface, tilt the specimen by using the Tilt (T) control.

NOTE: Focus is dependent on specimen height. As specimen height changes, the image will have to be refocused.

8.5

Select a magnification desired by using the MAGNIFICATION selector switch.

Magnification selected is indicated by the MAGNIFICATION control. The magnification reading is correct when zoom control is full counterclockwise and the working distance is 8mm. By using the combination of the magnification and zoom control, it is possible to select a range of $30X \sim 200,000X$ at a working distance of 8mm. When the working distance is at the lowest position*, the minimum magnification is 10X. Since it is difficult to shift the specimen at a high magnification, it is recommended to use the IMAGE SHIFT X and Y controls. Both the controls permit shifting the beam within a range of $\pm 20\mu$ on the specimen in the X and Y directions respectively.

NOTE: In step 8.4 above, focused condition varies each time a higher magnification is selected. Therefore, bring the image in good focus by turning the FOCUS COARSE control at each magnification level.

After the specimen area of interest becomes visible on the CRT, adjust the SPOT SIZE control (See NOTE below) and set the SCAN MODE switch at the RAPID position. By turning the FOCUS FINE and COARSE controls, bring the image in perfect focus. When the image is focused, it is very sharp and shows the fine structure of the specimen. For bringing the image in the best focused condition, it is recommended to select a magnification two steps higher.

NOTE: Signal on the CRT changes by turning the SPOT SIZE control. The beam spot is the minimum diameter and electron beam intensity is at minimum when the SPOT SIZE control is turned fully clockwise (to the MIN position). When the SPOT SIZE control is turned fully counterclockwise (to the MAX position), the beam spot has the maximum diameter and electron beam is most intense to make the image bright on the CRT.

The following table provides a general guide for setting the SPOT SIZE control that should be used to obtain high quality images. Referring to the data shown on the next page:

* See Section 5.7.

8.6

Magnification	Position of SPOT SIZE Control
<u>30X 5000X</u>	10:00 o'clock position
<u>7000X</u> 20,000X	12:00 o'clock position
28,000X 60,000X	3:00 o'clock position
80,000X or higher	MIN position

8.7

If the image is not sharp or if the image shifts in one direction and then in another direction (almost perpendicular to the former) when focusing, it is necessary to compensate for astigmatism. To compensate for astigmatism, adjust focus until the image on the CRT is between over focus and under focus (between the shift) as mentioned. Then adjust the X and Y Stigmator Controls until the sharpest image is obtained. Refocus and repeat until image is sharp and perpendicular shift is eliminated.

8.8 Repeat step 8.7 at a magnification one to two steps higher than the desired magnification required for investigation. Until an operator becomes familiar with the instrument, it is recommended that astigmatism is checked, and if necessary, compensated with a known specimen at the start of the day. To obtain optimum results, astigmatism should be checked on all specimens where viewing and micrographs are required at magnifications over 2000X.

REMARKS: Procedures for correcting astigmatism are detailed below:

- When astigmatism is gross, it will be easier to correct it step by step while changing magnification from lower to higher levels.
- 2. Astigmatism can not be corrected properly in a condition where the image is sweeping on the CRT. Be sure to set the focus controls at such positions that positive focusing is obtained (the image does not sweep, though it is blurred).
- 3. Adjust the STIGMATOR X control so as to obtain a sharp image.

- Adjust the STIGMATOR Y control so as to make the image sharpest.
- 5. Now the image should have been brought in better focus than in Step 2 above. Repeat setp 8.7 above once again. If the image still sweeps, repeat steps 2 through 4 above.
- 6. When the image does not sweep any more after repeating step 8.7 above, select a higher magnification and further repeat steps 2 through 4 above. However, it becomes rather hard to judge sweeping of the image since fine structures are enlarged at the higher magnification. Therefore, it is recommended to shift the image area or use a specimen which has fine structures matched with the magnification level selected for correcting astigmatism.
- REMARKS 2:

If the image still sweeps even after both or either of the STIGMATOR X and Y controls are turned to the 0 or 10.0 position (astigmatism can not be corrected completely), it can be assumed that astigmatism is too gross to correct. Further, when astigmatism can be corrected only by turning both or either of the STIGMATOR X and Y controls beyond the 7.0 position or within the 3.0 position, image quality will generally be too poor for high magnifications. Such gross astigmatism can be traced to the following:

- 1. Emission current is too saturated (See Section 6).
- 2. Specimen is charged or magnetized.
- 3. Lens aperture, sleeve, etc., are contaminated (see REMARKS 3 below).

REMARKS 3:

Causes which produce serious astigmatism can be checked as follows (in the order of seriousness):

A. Dust in the objective aperture.

- B. Dust on the top surface of the objective aperture holder (especially fibers).
- C. Dust inside the objective aperture holder (especially

fibers).

- D. Contamination on top surface of the objective aperture holder (brownish contamination).
- E. Dust in the bore (lmmØ) or on the top surface of aperture 2 located on sleeve joint 4.
- F. Dust or contamination in the bore (100 β) or on the top surface of the condenser lens aperture.
- G. Dust inside the condenser lens aperture holder (especially fibers).
- H. Dust in the bore (0.6mmØ) or on the top surface of aperture 1 located on sleeve joint 3 (condenser lens aperture holder).
- I. Dust inside sleeve joints 1, 2, 3 and 4 (especially fibers).
- J. Dust inside sleeves 1, 2, 3 and 4 (especially fibers).
- K. Dust around the hole in the anode (especially fibers). The parts mentioned above can be cleaned as instructed in Section 26.
- When specimen or image is sweeping after focusing the image as instructed in 8.6, it may be required to carry out step 8.6 above once again to bring the image in good focus. Further, it may also be required to correct astigmatism as instructed in 8.7 and 8.8 above when a specimen is charging or magnetized (especially at high magnification).
- It is often desired to observe a side surface or convex and concave conditions of a specimen at a low magnification. If the center of an image is focused and the specimen is tilted for this purpose, the upper and lower parts of the image will be blurred due to insufficient depth of focus. For correcting such a sympton, the DYNAMIC FOCUSING control can be used (set the SCAN MODE selector switch at the NORMAL position for checking).

Read the tilting angle on the specimen stage and adjust the DYNAMIC FOCUSING control to this angle. By turning the FOCUS controls, bring the center of the image in focus. The entire

8.9

8.10

image area can be brought in good focus by this procedure. NOTE 1: The entire image area can generally be focused properly by reading the tilting angle and setting the DYNAMIC FOCUSING control at that angle as instructed above. When the specimen proper is not perpendicular to the stub, however, it is recommended to adjust the DYNAMIC FOCUSING control until the entire image is very sharp while monitoring the image on the CRT. NOTE 2: The DYNAMIC FOCUSING control is not always necessary for imaging since stereoscopic impression may be enhanced when upper and lower parts of an image are somewhat defocused. NOTE 3: Since the DYNAMIC FOCUSING control is required exclusively for imaging at high tilting angles and low magnification levels, it is automatically made inoperative when high magnification levels are selected. The DYNAMIC FOCUSING control is ineffective at the magnifications of 7000X and above.

For obtaining a highly contrasted image, turn the CONTRAST
 control clockwise (turning the BRIGHTNESS control a little counterclockwise can enhance this effect).

When the index on the CONTRAST control is turned clockwise beyond the 2:00 o'clock position, however, the image becomes noisy.

When it is desired not to emphasize contrast but obtaining an image of soft tone, turn the CONTRAST control counterclockwise. For brightening the entire image area, turn the BRIGHTNESS control clockwise and vice versa. Since image contrast is changed also by turning the BRIGHTNESS control, it is practical to adjust the CONTRAST and BRIGHTNESS controls as a combination for obtaining a high quality image.

NOTE 1: When image is too noisy by turning the CONTRAST control clockwise for increasing contrast, but image contrast is insufficient by turning the BRIGHTNESS control clockwise for brightening the entire image area, turn the SPOT SIZE control counterclockwise. In this case, however, the image must be focused as instructed in Section 6. It may also be

8.11
required to correct astigmatism by following the instruction given in 8.7 and 8.8.

However, note that electron beam diameter increases and will degrade resolution by turning the SPOT SIZE control counterclockwise at a high magnification level (See NOTE in section 8.6). When image contrast is too high by turning the CONTRAST control counterclockwise or the entire image is too bright by turning the BRIGHTNESS control counterclockwise, turn the SPOT SIZE control clockwise. Also in this case, the image must be focused again. It may also be required to correct astigmatism. NOTE 2: The functions of the CONTRAST and BRIGHTNESS controls can be understood well when a line profile of the video signal is displayed on the CRT (refer to Section 7).

Amplitude of the profile is increased by turning the CONTRAST control clockwise, and vice versa. The entire profile moves upward by turning the BRIGHTNESS control clockwise, and vice versa. This can be regarded as coordinates on which contrast and brightness are plotted and is therefore useful for adjustment of image quality.

For obtaining a backscattered electron image, depress the IMAGE MODE BSE switch button and then turn the SPOT SIZE control counterclockwise. An image which is different in quality from a secondary electron image will appear on the CRT. In some cases, it is required to correct astigmatism, in the procedures instructed in 8.7 and 8.8 above. For displaying a secondary electron image again on the CRT, be sure to turn the SPOT SIZE control clockwise to its original position and depress the IMAGE MODE SE switch button.

8.12

9. DIGITAL MAGNIFICATION AND MICRON MARKER

9.1 The automatic digital magnification display is coupled to all operating parameters, thereby providing correct magnification at all times. To obtain optimum accuracy, focus the image as described in section 8 and read the magnification directly from the display unit. The reading is interpreted as illustrated below:



Decimals are displayed at the lower left side of the digits. See examples below:

.200kX: Magnification is X200.

2.00kX: Magnification is X2000.

20.0kX: Magnification is X20,000.

200kX: Magnification is X200,000.

- 9.2 When using the dual magnification mode, the magnification displayed is for the low magnification image only. To obtain the high magnification, multiply the displayed value by the factor being used (X3, X5, X10).
- 9.3 The micron marker is displayed on the viewing CRT and the Photo CRT when engaged with the switch on the control panel. This feature provides a permanent record of the magnification on the micrograph. As with the digital display, the micron marker is coupled to all operating conditions, thereby providing accurate magnification at all times.
- 9.4 The micron marker is recorded on the micrograph in the lower right corne

A coding system that identifies the value of the micron bar as the magnification is changed is used in this system. The code is interpreted as follows: The μbar represents 100 μ when followed by 3 dots. 1. The μ bar represents 10μ when followed by 2 dots. 2. 3. The μ bar represents $l\mu$ when followed by 1 dot. 4. The ubar represents 0.1µ when followed by no dots. * Scale for calculating magnification. Magnification can be calculated from the micron bar by using the following relationships: 10X = 1mm when the µbar represents 100µ ---= 1mm when the ubar represents 10μ --100X 1000X = 1mm when the μ bar represents $|\mu|$ -10000X = 1mm when the ubar represents 0.1μ Example: If the micron bar is preceded by 2 dots and is 6mm long, the magnification is 600X.

Length of μ bar(mm) x 100X = magnification.

9.5

NOTE: When using dual magnification, the ubar indicates the magnification of the low magnification image.

10. USE OF DYNAMIC FOCUS

10.1 Although use of the DYNAMIC FOCUS control was described briefly in section 8.10, this section provides more detailed information with respect to this feature. Basically, this control is used when a sample is viewed at low magnifications and high tilt angles. When the tilt angle is high, the vertical distance of the beam travels could exceed the depth of field of the objective aperture. If this should be the case, the top and bottom of the image will be defocused. The DYNAMIC FOCUS control compensates for this by keeping the beam focused throughout the vertical scan. 10.2 To use this feature, set the DYNAMIC FOCUS control to correspond with the tilt angle indicated on the specimen stage. While observing the center of the viewing CRT, focus the image as accurately as possible. The image should now be in focus from top to bottom.

- 10.3 Although the DYNAMIC FOCUS is generally used as described above, good top to bottom focus may not be obtained. If this occurs, the cause is due to the sample surface not being parallel to the specimen stub, in which case the true tilt angle differs from the indicated tilt angle on the specimen stage. When this occurs, adjust the DYNAMIC FOCUS control until the entire image from top to bottom is in focus while observing the image in the normal scan mode.
- 10.4 In some cases, it may be advantageous to not use the DYNAMIC FOCUS as the depth effect may be enhanced by viewing an image that has some defocusing. When this is desired, set the DYNAMIC FOCUS control to 0°.

11. USE OF SPOT, LINE, L.P. AND X-RAY SWITCHES

11.1 The SCAN MODE SPOT, LINE, L.P. and X-RAY switch buttons are used for x-ray elemental analysis using a non-dispersing type x-ray detector. For this analysis, signals are supplied from the multichannel analyzer of the x-ray analysis system to the connector (PULSE AMP) located on the left side rear of the display console. Also, the output of the rate meter of the x-ray analysis system can be connected to the L.P. connector. By following the instructions in Sections 6 through 8, form an image on the CRT. The specimen should be tilted toward the x-ray detector.

11.2 When it is desired to observe elemental distribution in the specimen, depress the SCAN MODE X-RAY switch button. Then an x-ray image (map) is formed on the CRT.

The multi-channel analyzer of the x-ray analysis system provides output in the following conditions:

Pulse height: 5Vp-p

11.3

Signal polarities are optional since a selector switch is provided inside the instrument for matching polarities.

(refer to "PC Board Check Point and Adjustment Table"). NOTE: Brightness on the CRT is dependent only on the pulse height and does not change by turning the BRIGHTNESS controls.

- If it is desired to analyze elements within a specific point on a specimen, depress the SCAN MODE SE switch button. Using the afterglow of the secondary electron image as a guide, locate the area of interest. Then depress spot button and position the spot at the area requiring elemental analysis by turning the POSITION X and Y controls, analyze elements within the area by operating the x-ray analysis system.
- 11.4 When it is desired to analyze elemental distribution along a certain line (in horizontal direction) on a specimen, depress the SCAN MODE SE switch button and search for an area to be checked on a secondary electron image. When the area of interest is located, depress the line switch and position the

line (using the afterglow of the CRT) with the Y position control to the area selected. Then depress the Line Profile switch (LP). The X-RAY LINE PROFILE will appear at the bottom of the CRT from the area selected.

Both x-ray mapping and x-ray Line Profile can be displayed on both

11.5

the viewing and photo CRT.

11.6 When performing x-ray analysis, it is recommended to monitor specimen current. Refer to Section 5 of the manual for details.

13. OPERATION OF DUAL MAGNIFICATION (OPTIONAL)

- 13.1 The instrument is equipped with a system which permits simultaneous observation of a low magnification image on the left half and a high magnification image on the right half of both the viewing and photo CRT's.
- 13.2 To engage dual magnification, turn the selector switch to X3, X5 or X10, as desired. Dual magnification is displayed in the rapid, normal and photo scan modes. Dual magnification is automatically disengaged when the scan mode is in the TV scan position.
- 13.3 When dual magnification is on when a rectangular frame is displayed on the low magnification (left side) image. The area within the frame represents the field of view displayed on the right side of the CRT and changes size when the dual magnification is changed. X and Y position controls are used to locate the frame anywhere within the low magnification field of view.
- 13.4 To focus and correct astigmatism with dual magnification on, turn the scan mode switch to rapid and depress the reduced area button. The reduced area scan will shift to the right side of the CRT so that focusing and astigmatism corrections are performed on the high magnification image.
- 13.5 The procedure for obtaining a micrograph of a dual magnification image is the same as with dual magnification off. (Refer to Section 16 of the manual).

14. SIMULTANEOUS IMAGING DIFFERENT SIGNALS (OPTIONAL)

14.1 Two different signals can be imaged simultaneously with the SUPER IIIA when the dual magnification selector is in the X1 position. Secondary electrons (SE), backscattered electrons (BSE) and x-ray can be displayed on the right side of the CRT when the corresponding button is depressed which is located below the viewing CRT.

14.2

The signal desired on the left side of the CRT is selected with the signal selector located below the dual magnification controls which are as follows:

SE/BSE Displays a secondary electron or backscatter image depending on which signal was selected on right side of CRT.

X-RAY Displays the same x-ray signal as on the right side of the CRT.

Displays signals from optional accessories EXT. such as specimen current imaging, cathodoluminescence imaging, etc.

NOTE: Options connected to the external input have separate contrast controls.

14.3 Dual magnification can be used in conjunction with simultaneous imaging of two different signals. Focusing the image, astigmatism correction and photographing the image is carried out as described in sections 13.3 and 13.4.

15. OPERATION OF AUTO CONTRAST/BRIGHTNESS CONTROLS (OPTIONAL)

- 15.1 Normally, the image quality is adjusted by turning the Contrast, Brightness, Spot Size controls for optimum viewing and photography. In practice, however, there may be times when it may be difficult to obtain constant contrast and brightness levels, especially when taking a micrograph because of large signal level changes.
- 15.2 To use the Auto Contrast/Brightness, start with a normal image on the viewing CRT. Leaving the manual Contrast and Brightness controls in the normal operating positions, set the Auto Contrast control to read approximately 50 (middle of the green range) on the Contrast meter. Then adjust the photo meter to read approximately 50 with the recessed screw driver adjustment located to the left of the Auto Contrast on/off switch.
- NOTE: Auto Contrast/Brightness is inoperative in the TV scan mode. 15.3 When viewing the image, if it is determined that the contrast level is too high, turn the Auto Contrast control counterclockwise to obtain a lower reading on the Auto Contrast meter. If the contrast is too low, increase the reading of the Auto Contrast meter.
- 15.4 When the brightness level of the image is too high, turn the recessed Brightness control to obtain a lower reading on the photo meter. If brightness is too low, turn the Brightness control to obtain a higher reading on the meter.
- 15.5 To photograph the image using the Auto Contrast/Brightness system, proceed as described in Section 16 of this manual. Image contrast and brightness desired must be determined by taking several micrographs while making adjustments with the Auto Contrast/Brightness controls. Once the correct settings are determined, which are usually within the mid-range on the meters, lock the Auto Contrast control and record the contrast and brightness meter readings. Contrast and brightness can be continually reproduced on micrographs, even though the signal level changes from area to area or sample to sample. Minor changes in the manual Contrast and Spot Size controls can be made as well without affecting the image quality. However, the ACB system has a limited range and, as a

result, will not compensate for extreme changes in the manual operating controls.

15.6

The Auto Contrast/Brightness system also incorporates a time constant control. The image contrast and brightness is dependent on response time for signal processing. When an image contains excessive bright or dark regions due to large signal level differences, the response time of the system may produce unusual effects. View the image in the normal scan mode and select the time constant that produces the best results. The time constant position will vary with the specimens and magnifications. Time constant values are as follows:

Position	1:	0.6	seconds
Position	2:	1.2	seconds
Position	3:	6.0	seconds
Position	4:	12.0	seconds
Position	5:	60.0	seconds
Position	6:	120.0	seconds

15.7

Turn ACB switch off to operate without the Auto Contrast/Brightness. For future use of this system, simply turn Auto Contrast/ Brightness switch on. Contrast and brightness initially obtained will be reproduced. From time to time, however, minor adjustments of the Auto Contrast/Brightness controls may be required.

16. PHOTOGRAPHING THE IMAGE

- 16.1 After an area is selected where a micrograph is desired, focus the image, correct the astigmatism and set the magnification to the desired position as described in Section 8.
- 16.2 For operating procedures of the camera, refer to the instruction supplied with the camera. Polaroid 4 x 5 sheet film is required with the camera supplied. Type 52 or PN-55 is used. An f stop of 8 to 5.6 is generally used.
- 16.3 Set the SCAN MODE switch at the RAPID position. Turn the CONTRAST control fully counterclockwise. Then gradually turn the BRIGHTNESS control clockwise from its maximum counterclockwise position. Scanning lines will become visible on the CRT and the PHOTO METER pointer starts deflecting at the same time. Set the BRIGHTNESS control at a position where the PHOTO METER pointer reads $30 \sim 40 \mu A$.
- 16.4 Then turn the CONTRAST control gradually clockwise. An image becomes visible on the CRT and the PHOTO METER pointer starts deflecting further at the same time. Set the CONTRAST control so the PHOTO METER reading increases to $55 \sim 60 \mu$ A.
- 16.5 Turn the SCAN MODE control to PHOTO, pull the film cover out till it stops and depress the photo start switch. When the scan line on the CRT disappears at the bottom of the viewing CRT, slide the film cover in and process the film as instructed in the camera manual. The SCAN MODE control can be returned to one of the other modes for viewing.
- 16.6 If the contrast level on the micrograph is too high, turn the contrast control counterclockwise to a lower reading on the photo meter and vice versa. If the brightness is too high or low, adjust the brightness control in the same manner. Generally, several micrographs will have to be taken to obtain the correct contrast and brightness settings. Once the correct settings have

been determined, the reading should be recorded for future use.

When taking low magnification (2-3000X and lower) micrographs, it is recommended that type 52 film be used. This film has a much higher resolution and as a result will record the fine detail displayed on the photo CRT. At the higher magnifications, type PN-55 film is quite adequate. It must be remembered, however, that different contrast and brightness settings will be required when changing film types.

16.8

Taking micrographs with the Auto Contrast/Brightness system is the same except that manual contrast and brightness controls are not used. If the Auto Contrast/Brightness controls have been adjusted as described in Section 15, all that is required is to engage the Auto Contrast/Brightness system. Proceed as described in this section with the exception of steps 16.3, 16.4 and 16.6.

17. SHUTDOWN PROCEDURES

Turn the EMISSION control fully counterclockwise, if Auto 17.1 Emission is used turn off, turn OFF the OPERATION switch by depressing its lower half. The switch lamp will go off.

- Depress the VACUUM CONTROL SHUT switch button. Turn OFF the 17.2 DP switch button by depressing its lower half. The switch lamp will go off. Wait for about 15 minutes for the diffusion ~ WAIT 15 MMU pump to cool.
- Turn OFF the POWER switch by depressing its lower half. The 17.3 RP and POWER switch lamps will go out and the rotary pump stops operating at the same time. The RP leak valve is automatically actuated to admit air into the rotary pump.
- Turn OFF the cooling to the diffusion pump. 17.4

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18. REPLACEMENT OF ELECTRON GUN CARTRIDGE AND ANODE

- 18.1 Turn the EMISSION control fully counterclockwise or turn OFF Auto Emission, if used. Turn OFF the OPERATION switch by depressing its lower half. The switch lamp will go out.
- 18.2 Depress the VACUUM CONTROL AIR switch button. Several seconds later, the COLUMN LEAK valve starts operating to introduce air into the microscope column and electron gun chamber. In about 15 seconds, internal pressure of the microscope column reaches atmospheric level.
- 18.3 This instrument incorporates a flip top gun. Gently tip the gun to the left till it comes to rest on the stop. Be sure that the discharge bar makes immediate contact with the electron gun cartridge. (Refer to fig. 1 of this Section). <u>CAUTION</u>: In performing the following steps, take care not to drop dust or foreign material into the electron gun chamber and microscope column.
- 18.4 Firmly grasp the electron gun cartridge and remove from cartridge connector. For some time after use, the cartridge will be very hot (200°C or higher). Therefore, the operator should grasp the electron gun cartridge with a piece of cloth to prevent a serious burn. Fit a new cartridge into the socket. The replacement cartridge can be plugged in in either direction. 18.5 To replace the anode, lift out the anode from the anode spacer
- located in the electron gun chamber.



- 1, Anode
- 2. Anode seat
- 3. Anode spacer

Fit a clean anode into the anode space. NOTE 1: Be sure to wear vinyl gloves in handling new electron

gun cartridge and anode. Handling them with bare hands may cause discharging after the electron gun cartridge and anode are assembled in the instrument.

NOTE 2: When the electron gun cartridge, anode and socket for the electron gun are badly contaminated, they must be cleaned. Contamination of the electron gun cartridge and anode may cause discharging. The anode spacer must be cleaned as well. (For cleaning procedures, refer to Section 21).

18.6

Lift the discharge bar (See Fig. 1) so that it does not get caught in between the electron gun housing and anode chamber. Tip the gun housing back in place into the column.

18.7 Evacuate the instrument as instructed in Section 4. NOTE: When the pump down time is slow, it is possible that dust or foreign material such as lint is adhering to the vacuum gasket located between the electron gun chamber and electron gun housing.

- 18.8 To replace filament, remove 2 screws holding electron gun flat ring to electron gun base. Lift off flat ring and grid cap. Clean these parts if necessary as described in Section 21. Loosen 4 alignment set screws in base of electron gun cartridge and remove burned-out filament.
- 18.9 Insert new filament in electron gun cartridge base. Replace grid cap and flat ring. Next replace and tighten (loosely) the two screws holding the flat ring to the electron gun base. Adjust the four alignment set screws so that the tip of the filament is perfectly centered in the hole of the grid cap. Check with 5X to 10X eyepiece. Tighten four alignment set screws and two screws holding down the electron gun flat ring. Re-check alignment. If alignment has moved, re-center. The electron gun cartridge is now ready for use.

19. REPLACEMENT OF LENS, APERTURES, SLEEVES AND SLITS

- 19.1 Admit air into the instrument as when changing a sample. Refer to Section 4.
- 19.2 Fit the aperture holder tool in the groove of the objective aperture holder located at the center of the lower pole piece. By turning the tool counterclockwise, the objective aperture holder, objective aperture (200 β) and objective aperture stop can be removed as an assembly.
- 19.3 Successively, sleeve 4, sleeve joint 4, aperture 2 (lmmØ), sleeve 3, sleeve joint 3 (condenser lens aperture holder) condenser lens aperture stop, sleeve joint 2, aperture 1 (0.6mmØ), sleeve 2, sleeve joint 1 and sleeve 1 can be removed in this order from the microscope column (See fig. 19-1).
- 19.4 Replace with spare ones which have been cleaned. It is recommended that clean parts be handled with vinyl gloves. Check for contamination or dust on each part, use a 5-10X eye piece. Before assembling clean column parts, check using the same procedure as described in Section 21. To remove the objective aperture, first remove the objective aperture stop by turning it counterclockwise. Then remove the objective aperture by inserting the thinner . end of the furnished aperture removing rod from the lower side of the objective aperture holder. Place a new objective aperture in such a direction that the face having the smaller hole diameter comes in contact with the objective aperture stop (when being observed through an eye piece, the hole is in a conical form on the larger diameter side; see fig. 19-1). Place the new objective aperture in its proper position by pushing it with the thicker end of the rod and then tighten the objective aperture stop (for cleaning of the aperture, refer to Section 21).

19-52



 0.2 or 0.1mm in diameter (200/100 micron)

Fig. 19-1

For removing the condenser lens aperture, first take out sleeve joint 2 and condenser lens aperture stop from the upper side of the condenser lens aperture holder (sleeve joint 3). Then remove the condenser lens aperture by inserting the thinner end of the furnished aperture removing rod through the lower side of the condenser lens aperture holder. Set a new condenser lens aperture $(100 \ \beta)$ in such a direction that the face having the smaller hole comes into contact with the condenser lens aperture stop (when being observed through an eye piece, the hole is in a conical form on the larger diameter side). Set the new condenser lens aperture in position by pushing it with the thicker end of the lens aperture remover rod. Then drop the condenser lens aperture stop and sleeve joint 2 into their proper positions respectively (for cleaning of the lens aperture, refer to Section 21). Place clean parts in the microscope column in reverse order. Evacuate the instrument as when changing a sample. Refer to Section 4.

If pump down is slow, check vacuum seal between chamber and stage for lint, hair, etc. Also, be sure stage clamp is in the unclamped position.

19.5



19-53(b)

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20. PROCEDURE FOR CHANGING SCINTILLATOR

- 20.1 Periodically the scintillator has to be changed to maintain optimum performance of the SUPER IIIA. If bright spots or horizontal lines are observed on the CRT, this is an indication that the aluminum coating on the scintillator is deteriorating. When the Spot Size control has to be positioned past 11:00 o'clock (in counterclockwise direction) to maintain a sufficient noise free signal, is an indication that the efficiency of the scintillator plastic is deteriorating. Also, it must be kept in mind that rotating the Spot Size control counterclockwise increases the electron beam spot size which will result in a decrease of resolution. When any of the mentioned symptoms occur, the scintillator must be changed to regain optimum performance.
- 20.2 Admit air into the instrument as when changing a sample. Refer to Section 4. Set stage clamp knob on the specimen chamber at the UNCLAMP position.
- 20.3 Referring to fig. 20-1, slide back the rubber light shield (located at the end of the preamplifier) toward the photomultiplier cover until the two preamplifier set screws are exposed. After loosening these two screws, carefully pull out the preamplifier from the photomultiplier cover. The preamplifier and the photomultiplier can be removed as assembly and set aside.
- 20.4 After loosening the four detector set screws, remove the detector. Be careful not to bump the internal parts of the detector against the stage opening. Remove the collector mesh from the light guide (note distance between mesh and scintillator) and remove the scintillator cap from the light guide. Take out the scintillator while taking care not to contaminate the scintillator cap and light guide with fingerprints or dust. It is recommended that vinyl gloves be used.
 20.5 With the coated aluminum surface kept toward the sample, mount a new scintillator on the light guide. Replace the scintillator cap onto the light guide. Place a small drop of silver conducting paint at the inner edge of the scintillator retaining ring to make contact between the scintillator and ring. Be careful that the paint does

not flow between the scintillator and the light pipe as damage could result to the light pipe from the solvent in the silver paint. Also, be sure the conductive paint has thoroughly dried before installing the detector. Attach the collector mesh to the mount on light guide, always taking care not to contaminate the parts with dust or fingerprints. For removing the dust from the parts, blow with Freon gas, etc. Carefully insert the preamplifier together with the photomultiplier into the photomultiplier cover as far as it can go, and then tighten the set screws in the preamplifier. Slide the light shield back in place.

20.6

Evacuate the instrument as when changing a sample. Refer to Section 4. When the pumping speed is slow, it is possible that foreign material such as lint is adhering to the vacuum gasket of the detector base.

20.7 IMPORTANT: Do not clean the surface of the scintillator by using reagent or wiping with cloth. When lint or other foreign materials are adhering to the surface, remove with air stream, etc. Do not clean the light guide (made of acrylic resin) with any type of reagent. When the light quide is contaminated, clean it with a piece of soft gauze while taking care not to damage or scratch the end surface. 20.8 To check the scintillator high voltage contact, turn on the instrument and leave the emission control in the counterclockwise position. Adjust brightness to a comfortable level and turn contrast control clockwise. If proper high voltage contact has been made to the scintillator, no noise (bright spots on CRT) should appear on the CRT till the contrast control is in the 2-3 o'clock position. Should noise appear before this position is reached with the contrast control, indicates a problem in the high voltage contact to the scintillator. If the contact between the scintillator and ring is causing a problem, it can usually be rectified by applying a small drop of silver conducting paint at the aluminum/ring interface. CAUTION: Do not allow silver paint to come in contact with light guide.

Fig. 20-1



21. CLEANING OF ELECTRON GUN CARTRIDGE, ANODE, SLEEVES AND APERTURES

21.1 One of the most important requirements to obtain the best results from any electron optical column is cleanliness. Therefore, the SUPER IIIA is no different in this respect. The following procedure is recommended for thorough and effective cleaning of the column parts.

The following parts must be cleaned when they are badly contaminated:

(1) Electron gun cartridge grid cap

Filament holder

(2) Socket for the electron gun cartridge

(3) Flat ring for the electron gun cartridge

- (4) Anode aperture
- (5) Sleeves 1, 2, 3 and 4
- (6) Sleeve joints 1, 2, 3 and 4
- (7) Slits 1 and 2
- (8) Condenser lens aperture stop
- (9) Objective aperture stop
- (10) Objective aperture holder
- (11) Condenser lens aperture $(100\mu\emptyset)$
- (12) Objective aperture $(200\mu\emptyset)$
- 21.2 Disassemble the column liner, electron gun cartridge and objective aperture holder. Save the objective and condenser apertures for cleaning, using a different procedure.
- 21.3 Clean all parts with a metal polish such as Wenol (CAUTION: do not use Wenol with PlusK additive). This polish is available from all microscope accessory suppliers. Use a cotton swab saturated with metal polish to clean the inside diameter of the sleeves, sleeve joints, etc. The smaller diameters can be cleaned by wrapping a little tissue such as Kimwipes around a wooden stick. To clean the outer surfaces, saturate tissue or cloth and polish till

all contamination is removed. Clean the bores of the 600μ and 1000μ apertures with polish and a wooden stick shaved down to a small diameter. In the case of the electron gun cartridge and anode where the contamination may be quite heavy, a cleaning agent such as Comet, Ajax, etc., can be used with water. Remove all traces of the cleaning agent and place parts in a beaker of acetone or equivalent solvent in an ultrasonic cleaner for several minutes. As the ultrasonic cleaning action removes the remaining cleaning agent, the solvent will discolor. Keep exchanging the solvent until it remains clean, then follow up with a final rinse in alcohol and dry the parts thoroughly.

- 21.4 Prior to assembling the parts, inspect with a 5X to 10X magnifying lens. A speck of dust or lint in a critical area could defeat the entire cleaning procedure.
- 21.5 Depending on what they are made of, there are several procedures for cleaning apertures. A platinum aperture can be cleaned very effectively by holding in a carbon free flame such as an alcohol burner or propane torch with a pair of platinum tipped tweezers. The aperture should be heated until it is cherry red for 30-60 seconds. To clean molybdenum apertures, place in tungsten boat in vacuum evaporator. Again, heat until cherry red for a minute or two or until color of entire aperture is uniform. Turn off heat and let cool before letting air into the system. If air is let into the evaporator before the apertures cool down, the apertures will oxidize.

21.6 When the column parts are cleaned successfully, the stigmator controls should be near the center of the controls when the astigmatism is corrected. Many times when the quality of the image deteriorates, one has a tendency to over-react and go through the entire cleaning process. Quite often this may not be necessary. If severe astigmatism is encountered and can not be corrected with the controls, generally the problem is dust or lint on the objective aperture or in the objective aperture holder. When the stigmator

controls gradually shift towards one end, usually this is an indication of contamination buildup. In either case, cleaning or replacing the objective aperture and/or cleaning the objective aperture holder may be all that is required. However, when astigmatism is corrected and the controls are near the center but the quality of the image is poor, usually the column liner is the cause and requires cleaning.

21.7

IMPORTANT: Do not clean the bore of the microscope column with a solvent such as acetone. If the center hole is cleaned with a solvent or reagent, its component parts may be dissolved to make the microscope column unusable. The center hole of the microscope column does not require cleaning.

21.8 Clean the anode with the procedures instructed in 21.3. The surface of the anode aperture must thoroughly be cleaned. To prevent discharging after assembly in the instrument, dry the anode and anode aperture using a blow dryer after cleaning has been completed.

CAUTION: <u>DO NOT</u> pour any type of solvent into the bore of the column. The column bore does not require cleaning.

22. CONDENSED OPERATING INSTRUCTIONS AUTOMATIC VACUUM SYSTEM INITIAL START UP PROCEDURE

- 22.1 Turn on diffusion pump cooling water.
- 22.2 Check that the yellow shut button is depressed (#1 valve position).
- 22.3 Turn on main power switch.
- 22.4 Depress rotary pump switch (RP) momentarily to start pump. The red lamp will remain on when the switch is released.
- 22.5 Wait about 20-30 seconds, then turn on diffusion pump (DP).*
- 22.6 After the diffusion pump has been on for 15 minutes, depress the blue operate button. The vacuum valve will go to the roughing position (#2 valve position). When vacuum level reaches 50 - 70 on the vacuum meter, the valve will automatically cycle to the high vacuum position (#3 valve position).
- 22.7 When the green vacuum indicator lamp comes on and the meter is in the green area, the operation switch can be turned on.

SPECIMEN CHANGE PROCEDURE

- 22.8 Turn emission control fully counterclockwise and turn off operation switch.
- 22.9 Depress red air button to admit air into the instrument.
- 22.10 Unclamp stage (if used) and release door lock.
- 22.11 Exchange sample.
- 22.12 Close specimen chamber door and lock. Be sure specimen stage clamp is in unclamped position.
- 22.13 Depress blue operate button. When the green vacuum indicator lamp comes on and the meter is in the green area, the operation switch can be turned on.

SHUTDOWN PROCEDURE

- 22.14 Turn emission control fully counterclockwise and turn off operation switch.
- 22.15 Depress yellow shut button.
- 22.16 Turn off diffusion pump (DP).
- 22.17 Wait 10-15 minutes for diffusion pump to cool, then turn off main power.
- 22.18 Turn off diffusion pump cooling water.

* If water failure should occur, the diffusion pump is protected with a thermal switch which is mounted on the back side of the pump. When the light in the diffusion pump switch fails to light, push the reset button. The thermal switch is reset when a click is heard.

23. INSTRUCTIONS FOR OPERATING VACUUM SYSTEM MANUALLY

23.1 If the automatic valving system should malfunction, the vacuum system can be operated manually. Remove the cover on top the column assembly. To disengage the automatic valving system, depress the shut button. Place the switch on the vacuum control logic chassis (located under table) in the manual positions. Then disengage the motor from the vacuum valve by pushing the rod to the left of the vacuum valve as viewed from the front. The vacuum valve can be moved manually by rotating the knob on top of the vacuum valve assembly. Refer to the following instructions for manual operation.

INITIAL START UP PROCEDURE

- 23.1 Turn on diffusion pump cooling water.
- 23.2 Check that the shut switch is depressed.
- 23.3 Check that the vacuum valve control is in the #1 position.
- 23.4 Turn on main power switch, then depress rotary pump switch (RP) momentarily to start pump.
- 23.5 Wait about 20-30 seconds, then turn on diffusion pump (DP).
- 23.6 After the diffusion pump has been on for 15 minutes, rotate the vacuum valve control counterclockwise to the #3 position.
- 23.7 When the vacuum level reaches 50-70 on vacuum meter, rotate the vacuum valve control counterclockwise to the #3 position.
- 23.8 When the green vacuum indicator lamps come on and the meter is in the green area, the operation switch can be turned on.

REMEMBER: THE VACUUM VALVE <u>MUST</u> BE MOVED FROM ONE POSITION TO ANOTHER IN SEQUENCE 1-2-3, 1-2-3, 1-2-3. ALSO, THE AIR SWITCH MUST NOT BE USED UNLESS THE VACUUM VALVE IS IN THE NUMBER ONE POSITION.

SPECIMEN CHANGE PROCEDURE

- 23.1 Turn emission control fully counterclockwise and turn off operation switch.
- 23.2 Rotate the vacuum valve control clockwise to the #1 position.
- 23.3 Admit air into the column with the small switch located to the front of the vacuum valve assembly.
- 23.4 Exchange sample.
- 23.5 Turn off air switch, then rotate valve clockwise to the #2 position.
- 23.6 When vacuum level reaches 50 70 on vacuum meter, rotate the vacuum valve control counterclockwise to the #3 position.
- 23.7 When the green vacuum indicator lamp comes on and the meter is in the green area, the operation switch can be turned on.

REMEMBER: THE VACUUM VALVE MUST BE MOVED FROM ONE POSITION TO ANOTHER IN SEQUENCE 1-2-3, 1-2-3, 1-2-3. ALSO, THE AIR SWITCH MUST NOT BE USED UNLESS THE VACUUM VALVE IS IN THE NUMBER ONE POSITION.

SHUT DOWN PROCEDURE

- 23.1 Turn emission control fully counterclockwise and turn off operation switch.
- 23.2 Turn off diffusion pump and rotate vacuum valve control clockwise to the #1 position.
- 23.3 Wait 10-15 minutes for the diffusion pump to cool.
- 23.4 Turn off main power switch.
- 23.5 Turn off diffusion pump cooling water.

To re-engage the automatic valving system, check that the shut down button is depressed. Manually position the vacuum valve in the #1 position. Place the switch on the vacuum control logic chassis in the auto position. If valve motor continues to run, readjust valve at the #1 position. Engage motor by pulling rod. The vacuum valve may have to be rocked back and forth slightly to allow the gears to engage.

24. SUPER IIIA SPECIFICATIONS

Resolution:	70 Angstroms guaranteed (at working		
	distance of 8mm and accelerating		
	voltage of 25kV).		
Magnification:	10X to 200,000X (zooming X1 to X3		
	possible at each step). Working		
	distance factor meter (WDF) and		
	accelerating voltage factor compen-		
	sation built in.		
Accelerating Voltage:	2kV, 15kV, 30kV.		
Electron Lens:	Three-stage, electromagnetic.		
Alignment:	Mechanical, electromagnetic deflection.		
Stigmator:	Electromagnetic, 8 poles		
Electron Beam Scanning:	Electromagnetic double deflection.		
Dynamic Focusing:	Tilting possible within a range of		
	$-10 \sim +70^{\circ}$.		
Electron Gun:	Hairpin type tungsten filament		
	(cartridge type for easy replacement).		
Specimen Holder Assembly			
and Specimen Chamber:	Universal type TXYRZ		
	Specimen size:		
	76.2mmø x 25.4mm t		
	76.2mmØ x 0.5mm t		
	31.8mmØ x 25.4mm t		
	15mmØ x 15mm t		
	Specimen shift: 40mm in X direction and		
	43mm in Y direction.		
	Specimen rotation: 360° continuous		
	Specimen tilting: ~10° \sim +70° continuous		
	Specimen working distance (Z): 30mm		
	(WD 8mm \sim 38mm continuously variable).		

Specimen current measuring terminals: 2 (one for grounding). Voltage application terminals (1A, 300V): 25 pins.

Signal Detection Mode:

Microscopic Observation and Micrography:

Scanning Mode:

Photography of Image: Simultaneous Display of Low and High Magnification Images: Secondary electron. Backscattered electron. X-ray signal input.

8 inch CRT for observation: 1 8 inch CRT for micrography: 1 Rapid scan (over the entire screen): 1 frame/0.7 seconds Rapid scan (reduced area): 1 frame/0.2 seconds TV scan (over the entire area): 1 frame/50 or 60 HzNormal mode (over the entire screen): 1 frame/7 seconds Photo mode (over the entire screen): 1 frame/80 seconds Line mode Spot mode (X and Y) Wave form monitor mode 4" x 5" Polaroid film camera.

Ratio between low and high magnifications: 1/1, 1/3, 1/5 and 1/10 (selectable in 4 steps) Position indication for high magnification image: A frame is indicated on low magnification image for the selection of visual field for high magnification image:

On low magnification image, optional portion is

selectable for high magnification observation. Simultaneous observation of different types of images (detectors available as optional accessories). Photo Meter for Micrography: Optimum exposure time can be read on photo meter. Indicator: Vacuum/emission/WD (selectable). Electromagnetic Image X, Y: 40μ (at accelerating voltage Shift: of 25kV). Micron Marker: 3 standard magnification scales: 50μ , 5μ , and 0.5μ) are displayed on image. Working Distance Calibration for Objective 8, 15, 23, 38mm (selectable in 4 steps). Lens Evacuation System: Fully automatic motor drive type Safety devices against power failure, water supply failure and poor vacuum level. Oil diffusion pump 100%/sec (with meter cooled baffle...] Oil rotary pump 50%/min...1 Power supply: single phase, AC 115 VAC Site Requirements: 50/60 Hz, 1.5kVA Grounding: grounding resistance lower than 100Ω . Cooling water: flow rate approximately 1.4 /minute with open drain Magnetic field: less than 3 milligauss Vibration: Less than +1 micron (freq. 5-50 Hz) Humidity: Less than 80%








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<u>A D D E N D U M</u>

All I.S.I. Scanning Electron Microscopes currently use an improved scintillator. The scintillator is a glass disc, which is first coated with phosphorus, then with aluminum. Advantage of this scintillator is improved efficiency and now longer life.

The <u>PROCEDURE FOR CHANGING SCINTILLATOR</u>, as described in the instruction manual, is the same.

CAUTION: HANDLE SCINTILLATOR BY THE EDGE WITH FINE TWEEZERS.

ISI - SPUTTERER-COATER

Model P-SI (Polaron E 5100)

INSTRUCTION MANUAL

ULPACKING ALD ASSEMBLY

Remove Model P-S1 Sputterer-Coater System from crate and take off packing material. Remove the front and rear panel and inspect interior for any loose components. Check that the circuit board is firmly seated in the socket.

In addition to the equipment the following parts are included:

a. Vacuum Hose (approximately 1/2 meter in length.)

b. Vacuum hose to mechanical purp adaptor.

c. 2 Hexagonal socket wrenches (1 for removing specimen holders and the other for removing specimen mounts).

Check that the voltage indicated on adhesive label corres-

ponds with main voltage in the laboratory.

The Sputterer-coater is designed so that the mechanical pump can be installed in the rear of the cabinet.

To install the pump, remove the back panel of the cabinet. Cut off the existing plug on the mechanical pump and connect to the terminal block provided on the chassis in the cabinet. The green wire in the pump cable must be connected to the same terminal that has a green wire connected on the back side. The black and white wire in the pump cable can be connected either way to the other two terminals on the chassis.

Connect a standard 3-prong plug to the main power cable of the Sputterer-Coater. Be sure to connect the green wire to the U-shaped pin.

-1-

Slide the vacuum hose onto the open vacuum connection inside the cabinet under the vacuum chamber. Next slide the L-shaped adaptor into the other end of the vacuum hose and connect the adaptor to the intake of the mechanical pump. Clamps are not provided as they are not necessary. Carefully position the mechanical pump in the back of the cabinet. The pump will have to be tilted to get it in. Finally, bolt the pump to the floor of the cabinet.

Finally connect the gas tubing to the gas inlet tube (smallest connection) on side panel and to the Argon cylinder (which must be provided by the customer.) Admit gas into tubing under pressure of approximately 5 psi.

NOTE: The cylinder must be fitted with a regulator of the type which indicates the pressure in the gas tubing.

The instrument is now ready for use.

Please note the following:

 Water cooling of the specimen holder block is generally unnecessary. It is only used in cases where a temperature of say 20 to 30 degrees, changes the structure of the specimen (e.g. chocolate)

2. If Argon gas is not available, then Nitrogen gas may be used. In the case of Nitrogen the discharge time will be approximately double as compared with Argon gas. If neither Argon gas nor Nitrogen is available the discharge can be run in air. In this case the Specimen holder shield must be fitted to the Specimen holder block TO prevent build up of contamination. WE DO NOT RECOMMEND THAT THE UNIT IS USED IN THIS WAY.

-2-

OPERATING PROCEDURE

- Set operation switch to pump position.
 Evacuate chamber until Pirani guage is reading better than 0.07 Torr (70 microns). When the pressure reaches approximately 0.2 Torr (200 microns) a 'click' is audible as the contact switch on the overload protective relay.
 This relay ensures that, if the pressure suddenly rises during the course of a run, the HT and hence the discharge are automatically cut off.
- Rotate leak valve approximately 1 revolution in an anticlockwise direction and close again, quickly. When Pirani gauge has recovered to original value (0.07 Torr) repeat again.

This ensures that the residual gas in the chamber is essentially Argon.

- 3) Select 'SET HT' position by turning Operation Switch. Rotate HT control until pointer indicates 1.2KV. There will be a slight surge indicated by the milliammeter, which should still read nearly zero. (this has selected the operating voltage).
- 4) Rotate the leak valve anti-clockwise slowly until the milliammeter indicates a reading of about 40. A discharge will now be visible between the specimen holder and the

-3-

gold anode.

These are the experimental conditions necessary for the gold coating of the most non-thermo sensitive materials.

 Select timer position by rotating operation switch.
 Set timer at 2 minutes by depressing triangular knob and rotating clockwise.

The timer is activated by pressing the black push button. The discharge will now operate for a period of 2 minutes indicated by the red pointer which will gradually rotate to zero.

6) When the timed discharge is cut off the specimen should be coated with a layer of gold.

Set leak value to zero position and turn HT control anti-clockwise to off position.

Rotate operation switch to select 'off position'. Admit air to chamber by lifting air admittance valve in top plate. CAUTION: DO NOT LEAVE SYSTEM UNDER VACUUM. The chamber may be opened up and the specimen carefully removed.

THICKNESS OF COATING.

An approximate measure of the thickness of the deposited gold film may be derived from the following equation:

d – MA x KV x t x k

-4-

Where:

KV is the kilovolt setting.

t is the time of the discharge in minutes.

k is a constant which is approximately 5 for Argon and 2 for Nitrogen and Air.

d is the thickness of the film in Angstroms.

For example: MA=40., KV - 1.2, t = 2 minutes

Argon gas discharge

 $d - 40 \times 1.2 \times 2 \times 5$

-5-

- 500 Å

Generally a gold film of between 200 and 500 Å is used.

(recommond 50 to 100 A)