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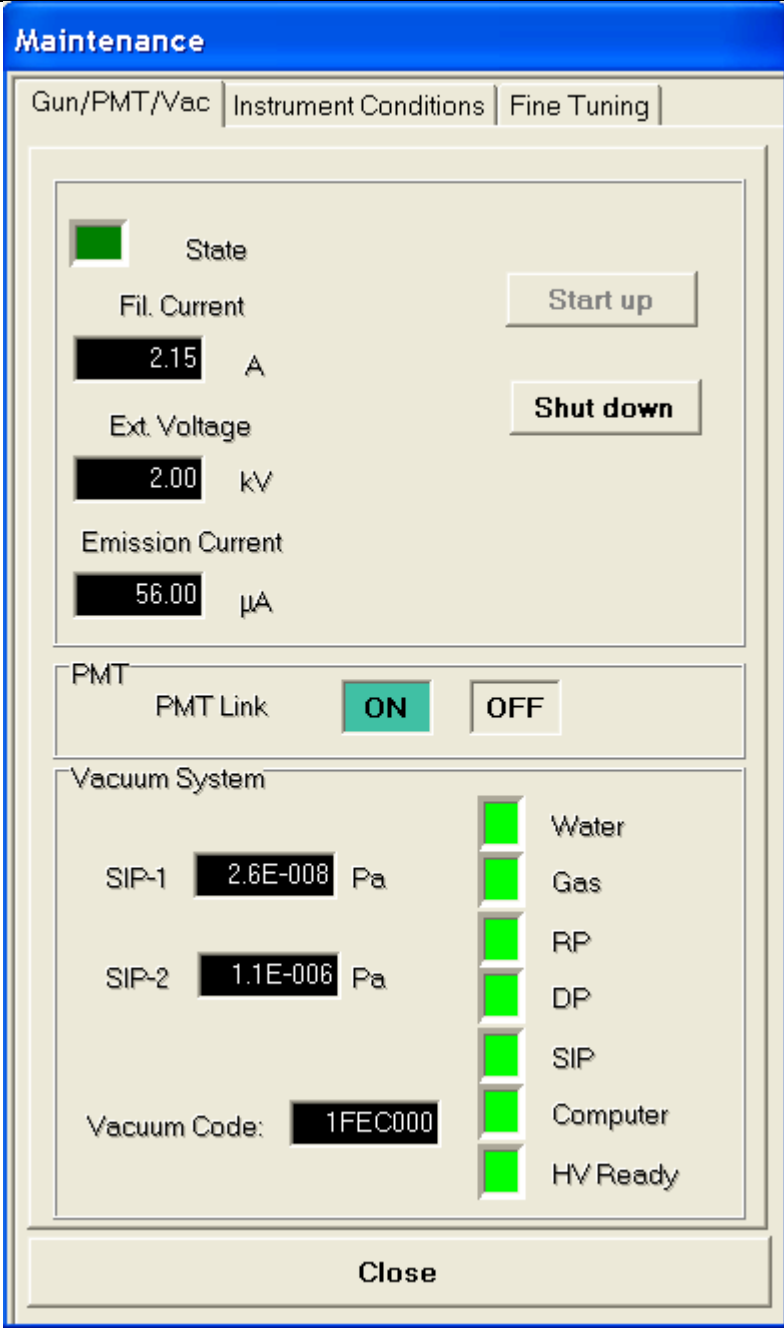
JEOL 7000F SCAN ELECTRON MICROSCOPE

+EDS

מכון ויצמן למדע
WEIZMANN INSTITUTE OF SCIENCE



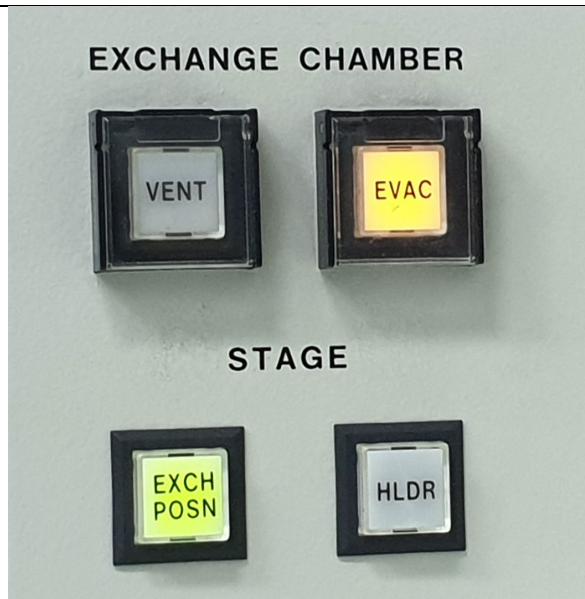
Machine availability check

1	<p>Activate the JEOL PC-SEM 7000 program if it is not activated.</p>	
2	<p>Open Maintenance menu in the main menu bar. Choose the Gun option.</p>	
3	<p>In the Gun/PMT/Vac label Make sure SIP1 and SIP2 are not higher than 3x10-8 Pa and 1x10-6 Pa, respectively.</p>	
4	<p>Make sure all lights of indicators are green. (If not, one of the machine requirements are not ready and you should contact the cleanroom staff).</p>	

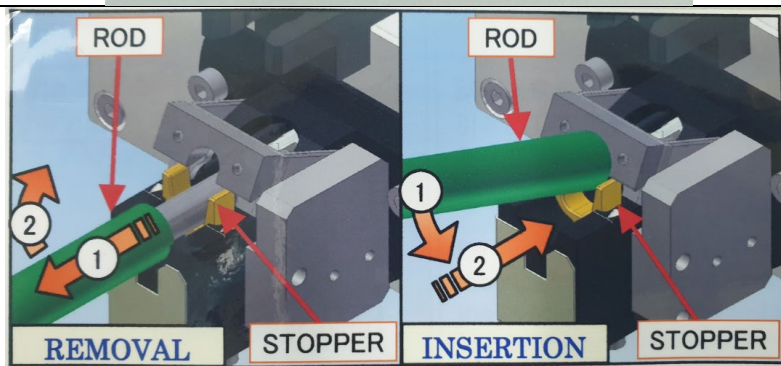
Sample mounting

1	<p>Make sure the HLDR-OFF button is OFF (if there is a specimen in the chamber it will be lighted).</p>			
2	<p>Loosen the clip on the side of the exchange chamber. Press and hold the VENT button until it start to blink.</p>			
3	<p>When VENT button is lighted the chamber has vented. Open the door and mount the sample holder (the arrows on the sample holder should be parallel to the load lock rail).</p>			
4	<p>Check that the O-ring around the door is in the right place. Close the door and shut the loadlock clip.</p>			

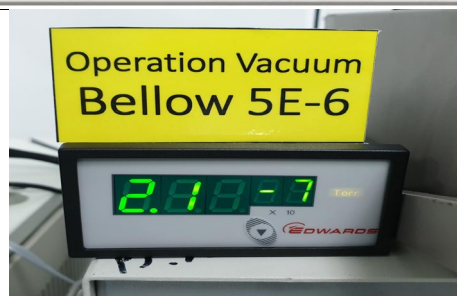
5 Press and hold the **EVAC** button until it start blinking.

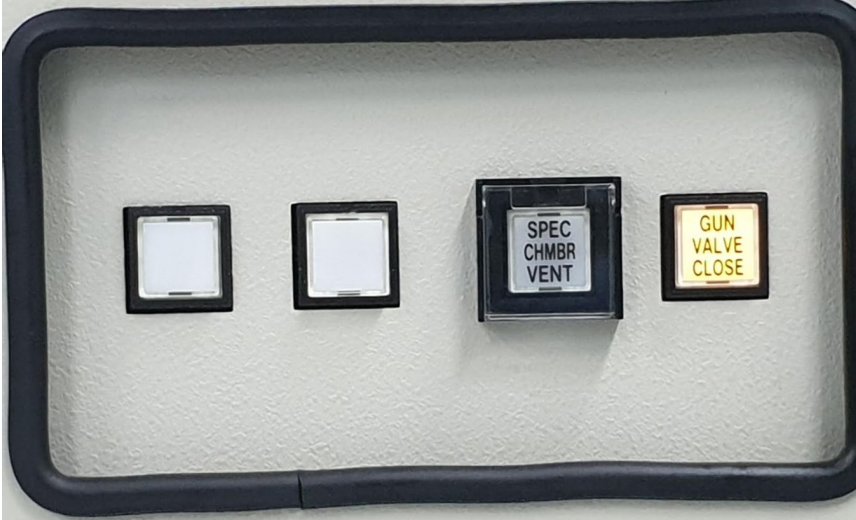
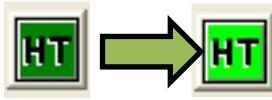


6 When **EVAC** button is lighted the chamber has vacuumed.
Stand up by the machine and pull the rod up, swing it in 90 degrees **carefully and smoothly**. Slide the rod in completely and pull it back to its place.
Look at the chamber internal camera and see that the sample is loaded correctly to the stage.
Caution: if a loud BEEP is heard - STOP! - Something is wrong.
Check if the stage is not in the exchange position (HLDR light should be on which means no sample is loaded).



7 Wait for the vacuum in the main chamber will be below 5×10^{-6} Torr.


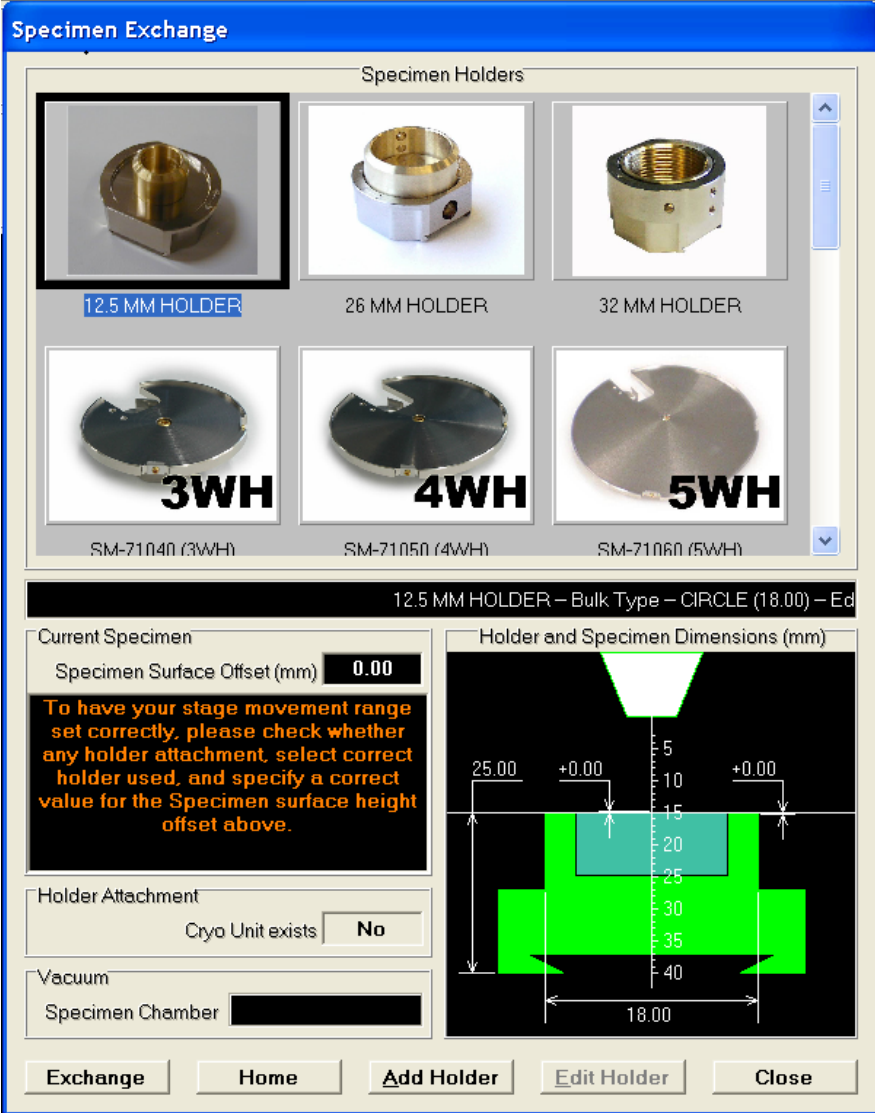
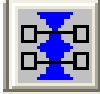


8	<p>Push the lighted "GUN VALVE CLOSE" button on the front of the microscope panel. The button will pop-out, still lighted.</p>	
9	<p>Turn ON the High Tension by pressing HT on the main screen. HT background button will change from dark green to bright green.</p>	

*** Operation panel



Sample Observation

<p>1</p> <p>If you see no image on the screen look at the OPERATION panel and if the FREEZE button is turned on turn it off by pressing the FREEZE button.</p>	
<p>2</p> <p>Open the stage option in the main menu and choose the Exchange option. Choose the correct sample holder you are using</p>	 <p>The software window 'Specimen Exchange' displays a grid of specimen holders. The first row shows three cylindrical holders: '12.5 MM HOLDER' (highlighted), '26 MM HOLDER', and '32 MM HOLDER'. The second row shows three circular holders: '3WH' (SM-71040), '4WH' (SM-71050), and '5WH' (SM-71060). Below the grid, the current specimen is identified as '12.5 MM HOLDER - Bulk Type - CIRCLE (18.00) - Ed'. The 'Current Specimen' section shows 'Specimen Surface Offset (mm)' set to '0.00'. A warning message states: 'To have your stage movement range set correctly, please check whether any holder attachment, select correct holder used, and specify a correct value for the Specimen surface height offset above.' The 'Holder Attachment' section has 'Cryo Unit exists' set to 'No'. The 'Vacuum' section has 'Specimen Chamber' set to an empty field. A 'Holder and Specimen Dimensions (mm)' diagram shows a cross-section of the holder and specimen with dimensions: 25.00 mm width, 18.00 mm specimen width, and a vertical scale from 0 to 40 mm. The specimen is 18.00 mm wide and 15 mm high. The holder has a 25.00 mm diameter opening. The diagram also shows a +0.00 offset for the specimen surface. At the bottom of the window are buttons for 'Exchange', 'Home', 'Add Holder', 'Edit Holder', and 'Close'.</p>
<p>3</p> <p>Open the Column window by clicking the Column button in the CONTROL MENU.</p>	

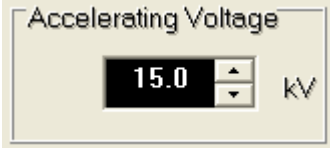





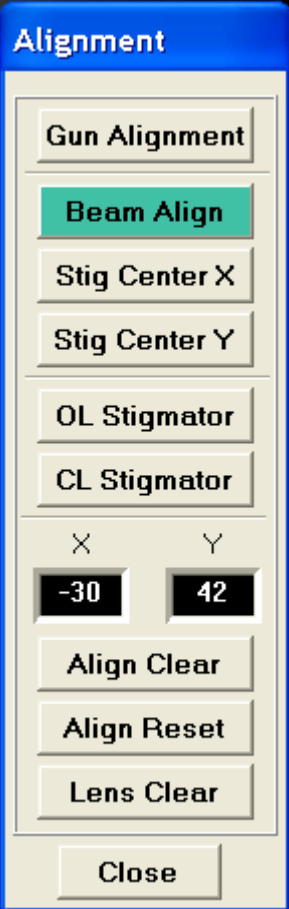
4	<p>Choose the Acceleration voltage you wish to work at the acceleration voltage section.</p> <p>Increase and decrease the voltage in steps of no more than 5 kV.</p>	
5	<p>→Adjust Contrast and Brightness manually or press the ACB button in the operation panel (Auto contrast brightness).</p>	
6	<p>→Move stage to find any area to focus on using joystick and mouse.</p> <p>Adjust the focus by rotating the focus wheel at all times.</p> <p>** WD will be 40.0.</p> <p>→Move the stage and rotate the magnification wheel to find and zoom on the area of interest (the specimen) and focus on it at all times.</p>	 <p style="text-align: center;"> Joystick Magnification Wheel Focus Wheel </p>
5	<p>Lift the sample and rotate the WD (Working Distance) so the WD will be 10.0 and the image focused.</p> <p>** Caution: Look at the screen showing the sample in the chamber and make sure you don't touch the objective lens with the sample.</p>	 <p style="text-align: center;">WD 10.0mm</p>

Table 3.3 Relationship between magnification, accelerating voltage, and working distance


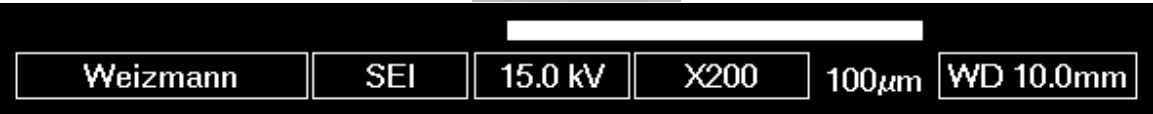
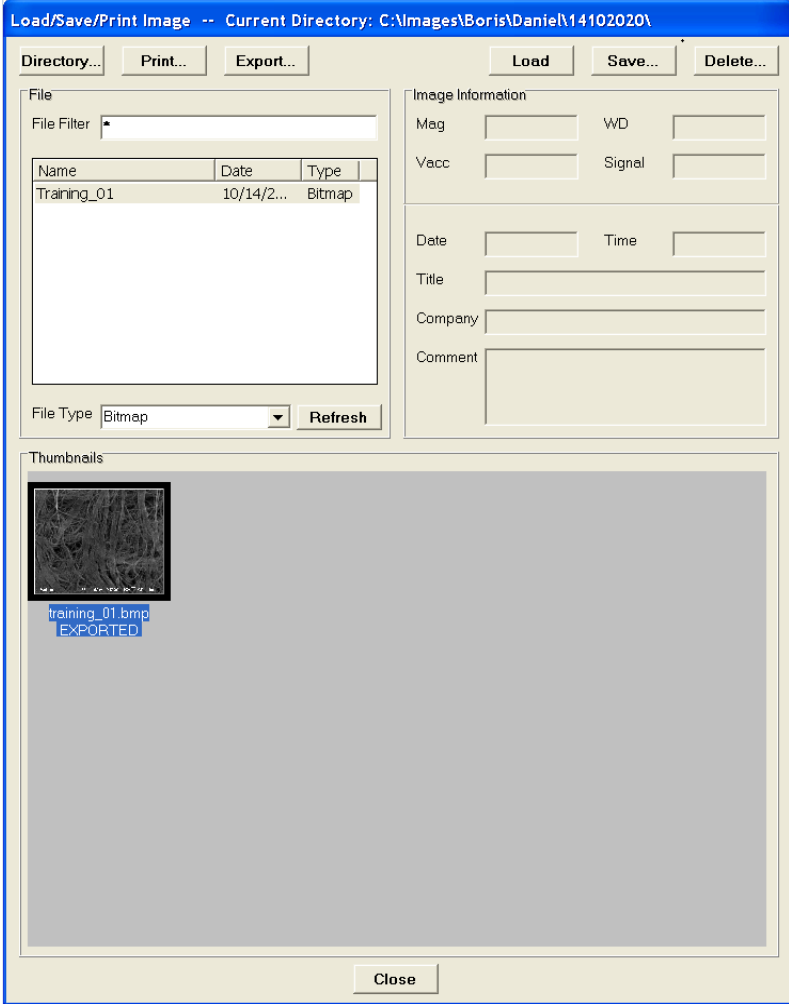

Accelerating voltage (kV)	Maximum magnification					
	WD (mm)					
	4	6	10	15	25	40
0.5	150 K	140 K	120 K	110 K	90 K	70 K
1	230 K	220 K	190 K	170 K	130 K	100 K
2	350 K	330 K	270 K	230 K	180 K	140 K
5	500 K	400 K	400 K	350 K	250 K	200 K
10		500 K	500 K	450 K	350 K	250 K
15				500 K	400 K	300 K
20		450 K	350 K			
25		500 K	370 K			
30		500 K	400 K			

Beam Alignment

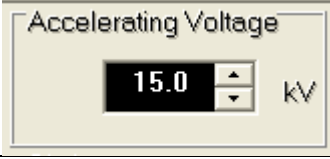
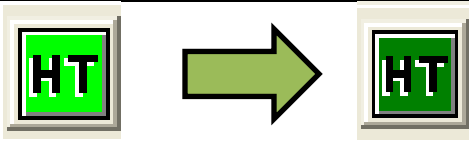
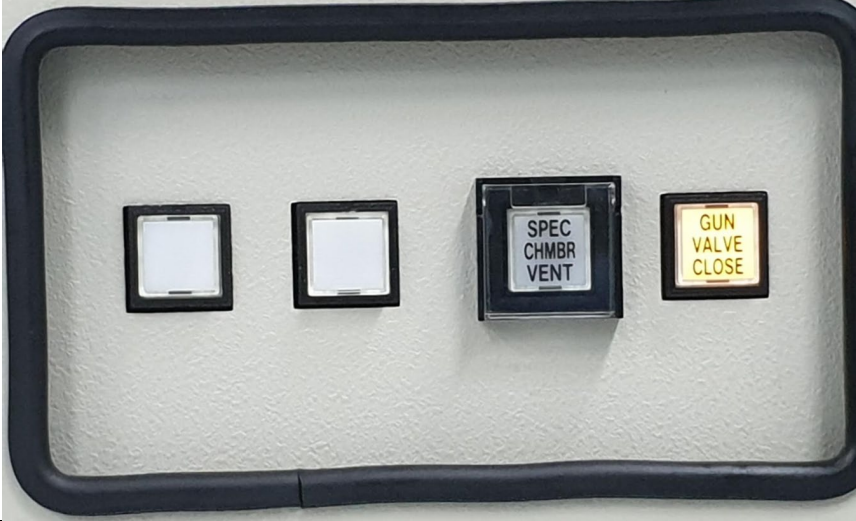
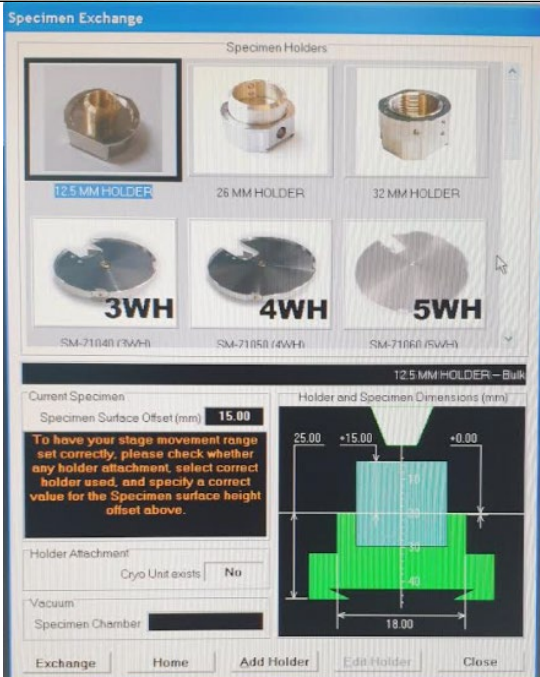
1	Make sure the image is the sharpest you can achieve.	
2	When STIG is on turn the X and Y knobs until the image is the sharpest you can achieve.	 <p>The image shows a control panel labeled 'ALIGNMENT' with two horizontal lines to its right. Below the label are three buttons: 'WOBB', 'ALIGN', and 'STIG'. The 'STIG' button is illuminated with a green light. Below the buttons is the text 'ALIGN OFF'. At the bottom of the panel are two large, light-colored knobs labeled 'X' and 'Y'.</p>
3	If the image is moving while doing focus, do the wobb procedure (continue the next step).	
4	Focus a defined pattern (circle, square...)	
5	Press the WOBB button.	 <p>The image shows the same control panel as in step 2, but with a blue arrow pointing to the 'WOBB' button. The 'STIG' button remains illuminated.</p>
6	Turn the X and Y knobs until the pattern goes in and out of focus all direction simultaneously and you get the minimum movement of the picture. (Pay attention to X and Y values before you start to rotate and follow it).	


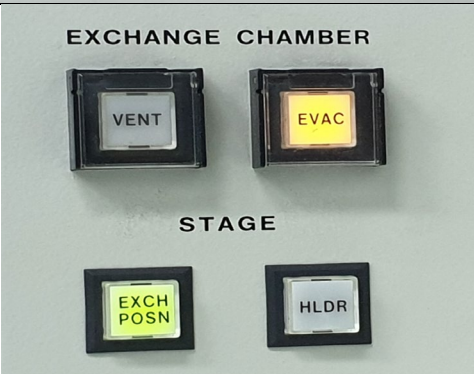
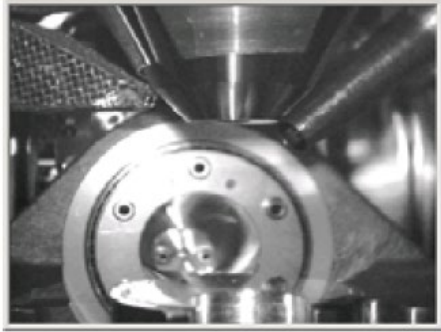


7	<p>If the pattern goes in and out of focus in one direction:</p> <p>→ Press the Stig Center X on the screen, press the WOBB button. Turn the X and Y knobs until the pattern goes in and out of focus all direction (Pay attention to X and Y values).</p> <p>→ Press the Stig Center Y on the screen, press the WOBB button. Turn the X and Y knobs until the pattern goes in and out of focus all direction (Pay attention to X and Y values).</p>	
8	<p>When done: press STIG button and focus.</p>	

Taking a picture





<p>1</p>	<p>Press PHOTO on the OPERATION panel (it will take about 1 min to scan).</p> <p>Image will be saved with micron bar.</p>	 
<p>2</p>	<p>When finishes Load/Save/Print Image window will be open. Press EXPORT... Choose the desired path and save.</p>	
<p>3</p>	<p>If you wish to avoid distortion caused by drifting in low scan, press FREEZE before taking picture.</p>	

Sample removal

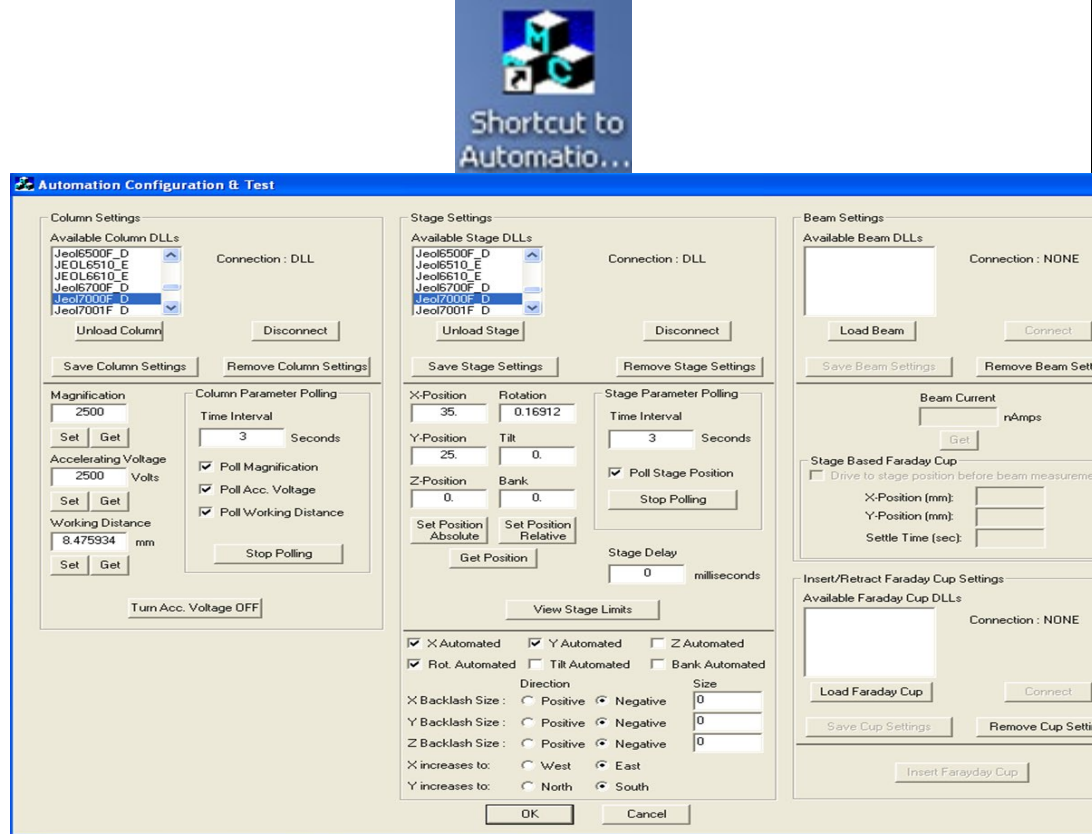
1	Decrease the acceleration voltage in steps of no more than 5 kV.	
2	Reduce magnification to minimum.	
3	Switch off the High Tension by pressing HT on the main screen. HT background button will change from bright green to dark green .	
4	Push the lighted "GUN VALVE CLOSE" button on front of the microscope panel. The button will be pressed in, still lighted.	
5	Open the STAGE menu in the MAIN menu and choose the EXCHANGE OPTION . In the SPECIMAN EXCHANGE window click on the Exchange button (the stage will start moving, you can see it by looking at the X control and Y control).	

6	<p>Set the WD control to 40.0 on the microscope body on the right side by turning the wheel. SET the stage tilt to 0.0.</p>	
7	<p>Make sure the EXCH POSN indication has turned on.</p>	
8	<p>Pull the rod up swing it in 90 degrees carefully and smoothly slide the rod in completely. Look at the chamber internal camera and see that the sample is unloaded.</p>	
9	<p>Loosen the clip on the side of the exchange chamber. Press and hold the VENT button on side of the chamber until blinking.</p>	
10	<p>When Vent button is lighted the chamber has vented. Open the door and remove the specimen holder.</p>	
11	<p>Close the exchange chamber and fasten the clip. Press and hold the EVAC button until blinking.</p>	
12	<p>DO NOT CLOSE THE JEOL SOFTWARE!</p>	

EDS SCAN

1	<p>Insert the sample to the SEM and Zoom to the area you wish to diagnose (horizontally). Follow the instruction of sample mounting, sample observation and beam alignment with SEM.</p>	
2	<p>Place the sample at 10 mm Working distance.</p>	
3	<p>Turn off the Infrared Camera (infrared interferes with the measurements).</p>	
4	<p>Transfer the control to the EDS computer by pressing on Button 2 on the KVM.</p>	
5	<p>Activate the EDS software (NSS) if it is not activated.</p>	

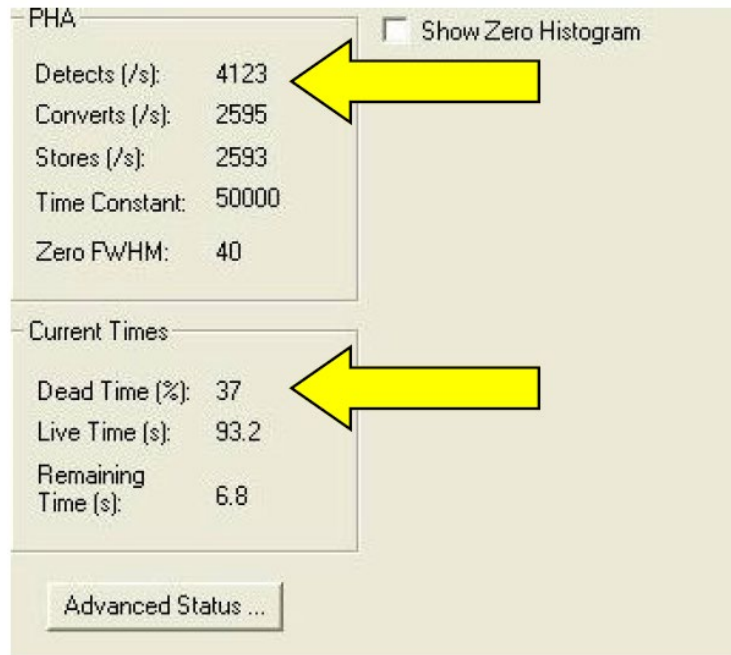
- 6 If the EDS was not activated:
1. Open the **Automation Configuration & Test**.
 2. under column Settings + stage Settings **Choose Jeol/ 7000F_D and Connect.**
 3. In the **Column Parameter Polling type Time Interval 3 seconds tick:**
 - ✓ Poll Magnification
 - ✓ Poll Acc. Voltage
 - ✓ Poll Working Distance
 4. **Click start Polling and OK.**
 5. Check these values from SEM.



7 **Choose or make a folder.**

File > Project Explorer

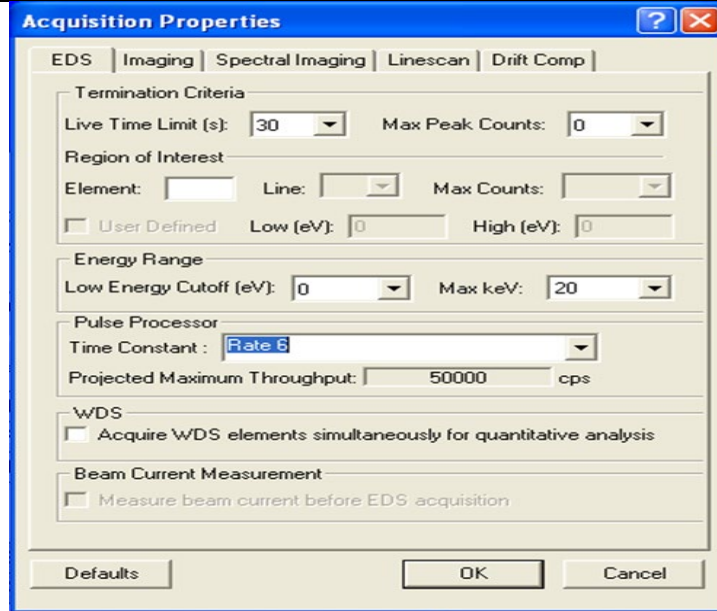
- 8 Set the **Acceleration voltage to 20 KV** at least
- Check the **PHA (SR) detects is at least 3,000 and DT is less than 35%.**
- If the values are not as desired, change the acceleration voltage and the probe current or turn & tilt sample to the inspector until you have reached the values needed.



9

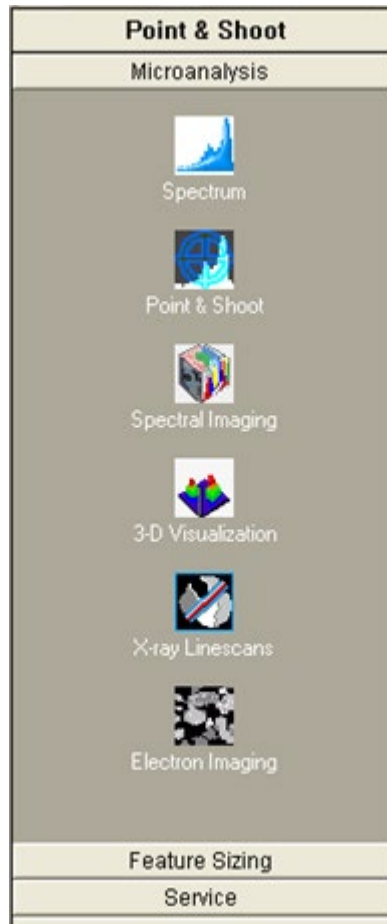
Click Edit > Acquisition Properties > EDS.

Set the **Live Time Limit** (detection time), **Max Peak Counts** and **Time Constant** (Rate 6).



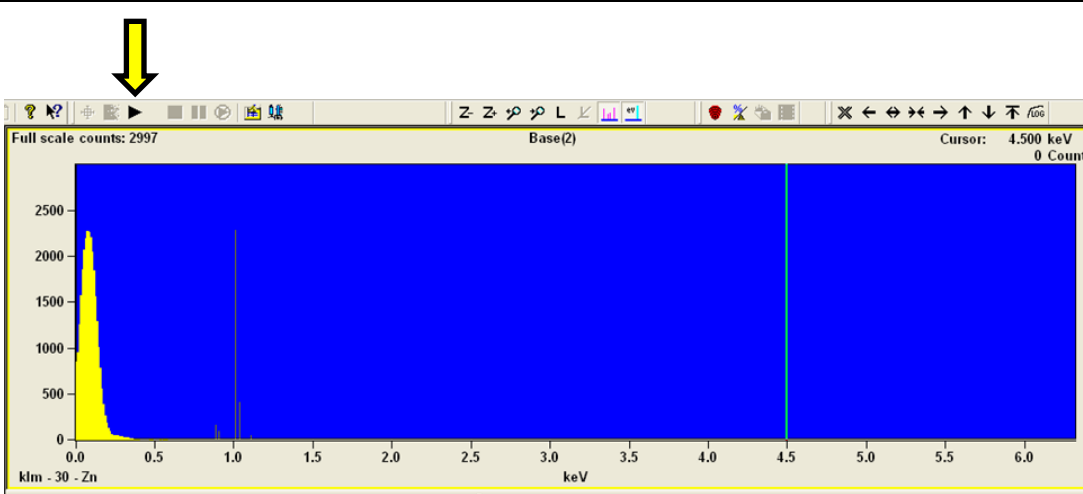
10

In the Microanalysis panel **choose** the desired scan option and **press** the **Acquire an averaged electron image** icon to take a picture.



10a. Spectrum
 Gives only counts vs. KeV of the all scanned area.

Press the **Start Acquisition** (Play) icon to run the EDS scan.




10b. Point & Shot
 Take a SEM picture and gives only counts vs. KeV of the specified area chosen from the SEM picture.


Press the **Start Acquisition** (video) or **Acquire an averaged electron image** (picture) icon.

Choose a point. You can wait the scan to finish and choose one/more points.

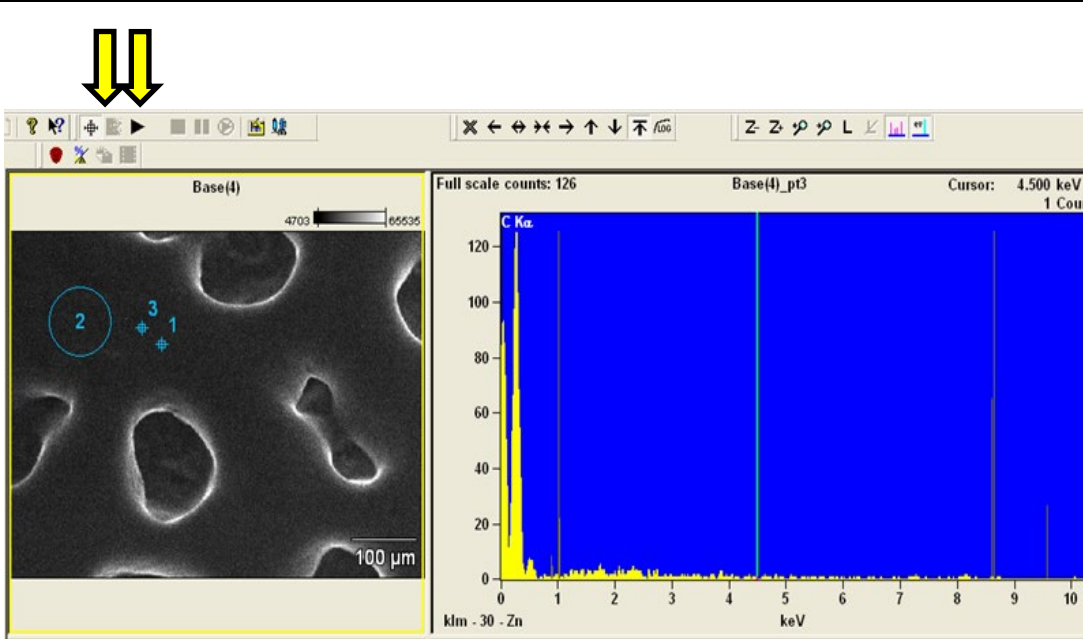
We can also choose area from the SEM picture

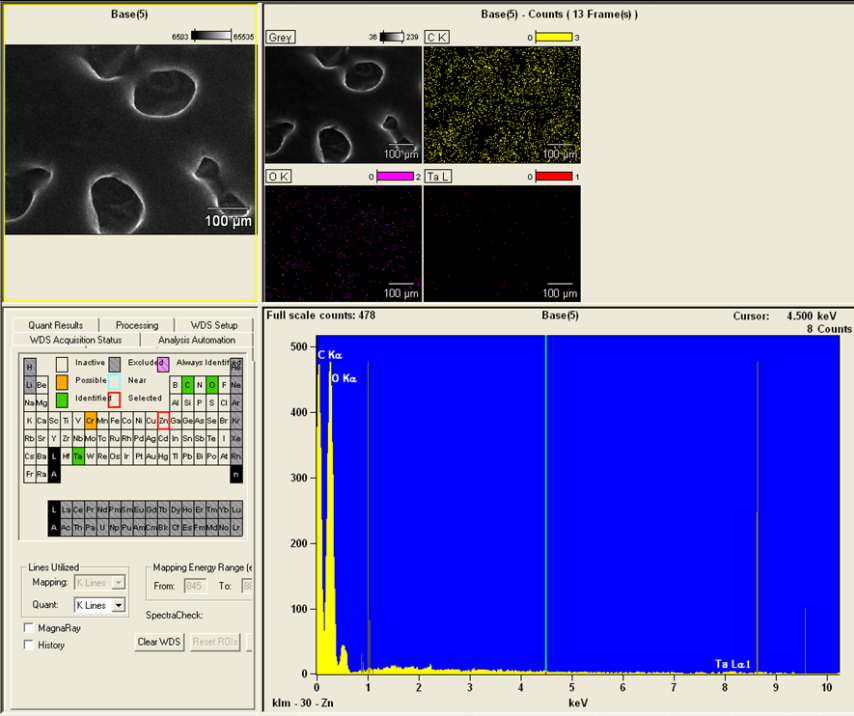
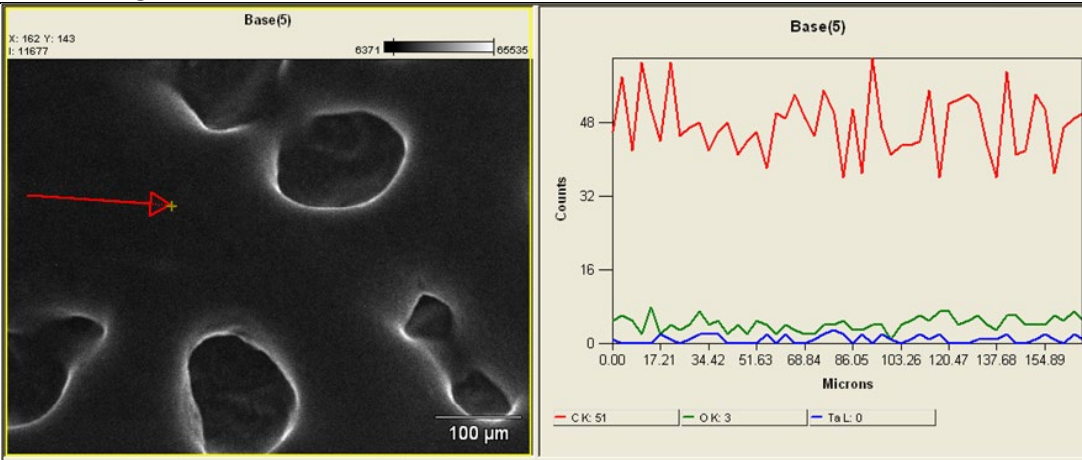



Press quantify spectrum



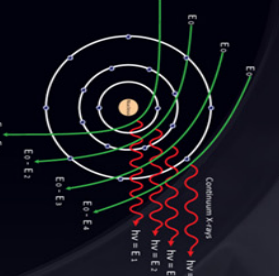
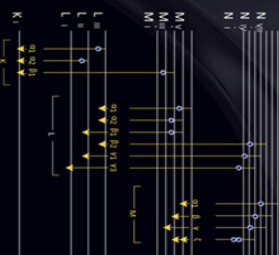
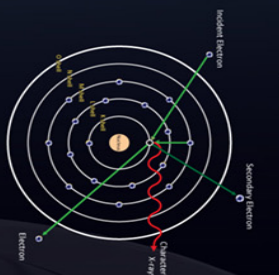
> Quant results.



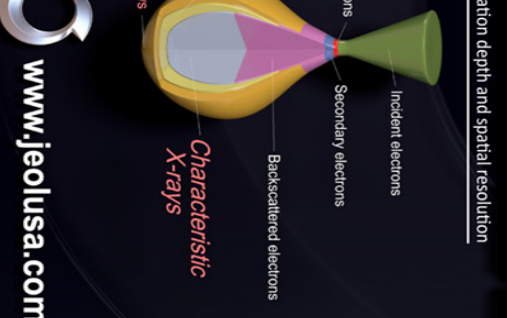
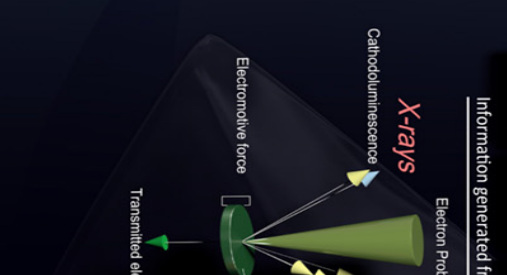
<p>10c.</p> <p><u>Spectral imaging</u></p> <p>Gives counts vs. KeV peaks and mapping by elements.</p> <p>Press the Play icon to run the EDS scan.</p>		
<p>10d.</p> <p><u>3-D Visualization</u></p>		<p>Not working</p>
<p>10e.</p> <p><u>X-Ray Line scans</u></p> <p>Gives counts vs. Microns on the image.</p> <p>Choose the cross and set arrow on desired part.</p>		
<p>10f</p> <p><u>Electron imaging</u></p>		<p>Working like SEM in the EDS. Not really in use.</p>
<p>11</p> <p>Export data by pressing the word icon on the top left of the screen.</p>		
<p>12</p> <p>When finished:</p> <ul style="list-style-type: none"> →Set the Acceleration voltage to 5 KV and the probe current to 7. →Turn on the IR camera. →Exchange the holder position and extract the sample from the instrument. 		

Periodic Table for EDS Analysis

1	H	Hydrogen	1.008	0.00014
2	He	Helium	4.0026	0.00018
3	Li	Lithium	6.941	0.53
4	Be	Beryllium	9.0122	1.85
5	B	Boron	10.811	2.34
6	C	Carbon	12.011	2.26
7	N	Nitrogen	14.0064	1.14
8	O	Oxygen	15.9994	1.57
9	F	Fluorine	18.9984	1.5
10	Ne	Neon	20.1797	2.01
11	Na	Sodium	22.98976928	1.041
12	Mg	Magnesium	24.304	1.253
13	Al	Aluminum	26.9815386	2.7
14	Si	Silicon	28.0855	2.33
15	P	Phosphorus	30.973762	2.013
16	S	Sulfur	32.06	2.07
17	Cl	Chlorine	35.45	3.16
18	Ar	Argon	39.948	3.49
19	K	Potassium	39.0983	0.87
20	Ca	Calcium	40.078	1.55
21	Sc	Scandium	44.955912	2.99
22	Ti	Titanium	47.88	4.5
23	V	Vanadium	50.9415	5.87
24	Cr	Chromium	51.9961	7.2
25	Mn	Manganese	54.938044	7.43
26	Fe	Iron	55.845	7.86
27	Co	Cobalt	58.933195	8.71
28	Ni	Nickel	58.6934	8.8
29	Cu	Copper	63.546	8.93
30	Zn	Zinc	65.38	6.92
31	Ga	Gallium	69.723	5.91
32	Ge	Germanium	72.630	5.48
33	As	Arsenic	74.9216	5.73
34	Se	Selenium	78.96	4.8
35	Br	Bromine	79.904	4.2
36	Kr	Krypton	83.80	3.4
37	Rb	Rubidium	85.4678	1.694
38	Sr	Strontium	87.62	2.6
39	Y	Yttrium	88.90584	4.48
40	Zr	Zirconium	91.224	6.49
41	Nb	Niobium	92.90638	6.88
42	Mo	Molybdenum	95.94	10.22
43	Tc	Technetium	98.9062	10.27
44	Ru	Ruthenium	101.07	12.1
45	Rh	Rhodium	102.9055	12.48
46	Pd	Palladium	106.3631	12.16
47	Ag	Silver	107.8682	10.47
48	Cd	Cadmium	112.411	8.65
49	In	Indium	114.818	7.29
50	Sn	Tin	118.710	7.28
51	Sb	Antimony	121.757	6.68
52	Te	Tellurium	127.603	6.25
53	I	Iodine	126.90545	4.94
54	Xe	Xenon	131.29	5.54
55	Cs	Cesium	132.90545196	4.286
56	Ba	Barium	137.327	3.5
57-71	Lanthanide			
72	Hf	Hafnium	178.49	13.5
73	Ta	Tantalum	180.94788	16.69
74	W	Tungsten	183.84	19.3
75	Re	Rhenium	186.207	21.02
76	Os	Osmium	190.23	22.5
77	Ir	Iridium	192.222	22.42
78	Pt	Platinum	195.084	21.4
79	Au	Gold	196.966569	19.3
80	Hg	Mercury	200.59	19.3
81	Tl	Thallium	204.38	11.85
82	Pb	Lead	207.2	11.34
83	Bi	Bismuth	208.9804	9.78
84	Po	Polonium	209	9.19
85	At	Astatine	210	9.19
86	Rn	Radon	222	9.73
87	Fr	Francium	223	12.009
88	Ra	Radium	226	13.8
89-103	Actinide			
104	Rf	Rutherfordium	261	13.37
105	Db	Dubnium	262	13.37
106	Sg	Seaborgium	263	13.37
107	Bh	Berkelium	264	13.37
108	Hs	Hassium	265	13.37
109	Mt	Moscovium	268	13.37
110	Ds	Darmstadtium	271	13.37
111	Rg	Roganium	272	13.37
112	Cn	Copernicium	285	13.37
113	Nh	Nihonium	286	13.37
114	Fl	Flerovium	287	13.37
115	Mc	Mendelevium	288	13.37
116	Lv	Livermorium	293	13.37
117	Ts	Tennessine	294	13.37
118	Og	Oganesson	294	13.37



79	Au	Gold	196.966569	19.3
80	Hg	Mercury	200.59	19.3
81	Tl	Thallium	204.38	11.85
82	Pb	Lead	207.2	11.34
83	Bi	Bismuth	208.9804	9.78
84	Po	Polonium	209	9.19
85	At	Astatine	210	9.19
86	Rn	Radon	222	9.73
87	Fr	Francium	223	12.009
88	Ra	Radium	226	13.8
89	Ac	Actinium	227	13.7
90	Th	Thorium	232.0377	11.7
91	Pa	Protactinium	231.03688	15.37
92	U	Uranium	238.02891	18.9
93	Np	Neptunium	237.0481733	20.25
94	Pu	Plutonium	244.06422	19.84
95	Am	Americium	243.061381	13.68
96	Cm	Curium	247.07647	13.51
97	Bk	Berkelium	247.07125	10.74
98	Cf	Californium	251.079589	10.74
99	Es	Einsteinium	252.08322	10.74
100	Fm	Fermium	257.10358	10.74
101	Md	Mendelevium	258.1051054	10.74
102	No	Noelium	259.1055832	10.74
103	Lr	Lavrentium	262.10932	10.74



Castaing's formula

$$Z_m = 0.033 \left(E_0^{-1.7} - E_c^{-1.7} \right) \frac{A}{\rho Z}$$

Z_m : Electron penetration range (um)
 E_0 : Accelerating voltage (kV)
 E_c : Critical excitation energy (keV)
 A : Atomic mass
 ρ : Density (g/cc)
 Z : Atomic number

Interaction volume in iron (g/cc)

WOOD
 PLASTICS
 FIBERS
 ROCKS
 CERAMICS
 GLASS
 SI, Al, Ti, Ge, Fe, Ag
 Au

Interaction volume in iron

1.5um @ 20kV
 0.21um @ 5kV