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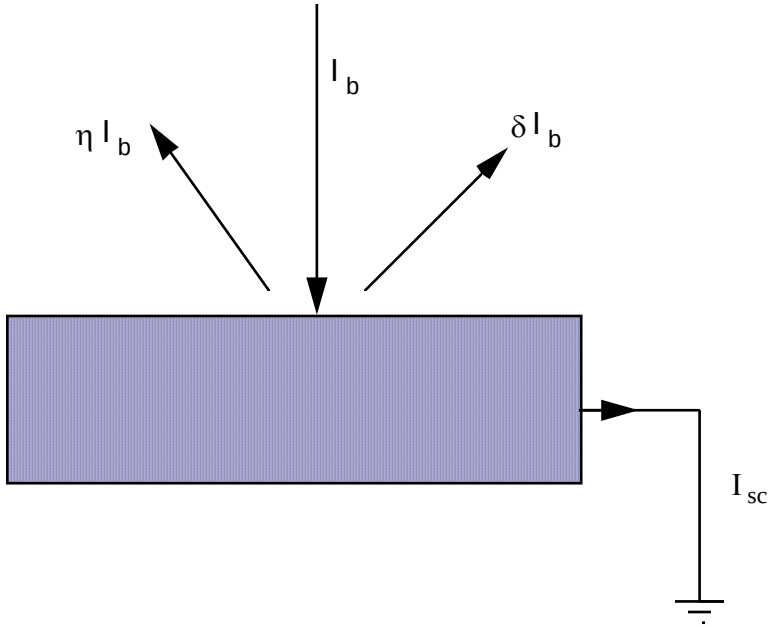
Charging Contamination

Charge balance in the sample

Electrons cannot be created or destroyed so currents at a point must sum to zero. The current flow to earth I_{sc} is the difference between the in and out currents



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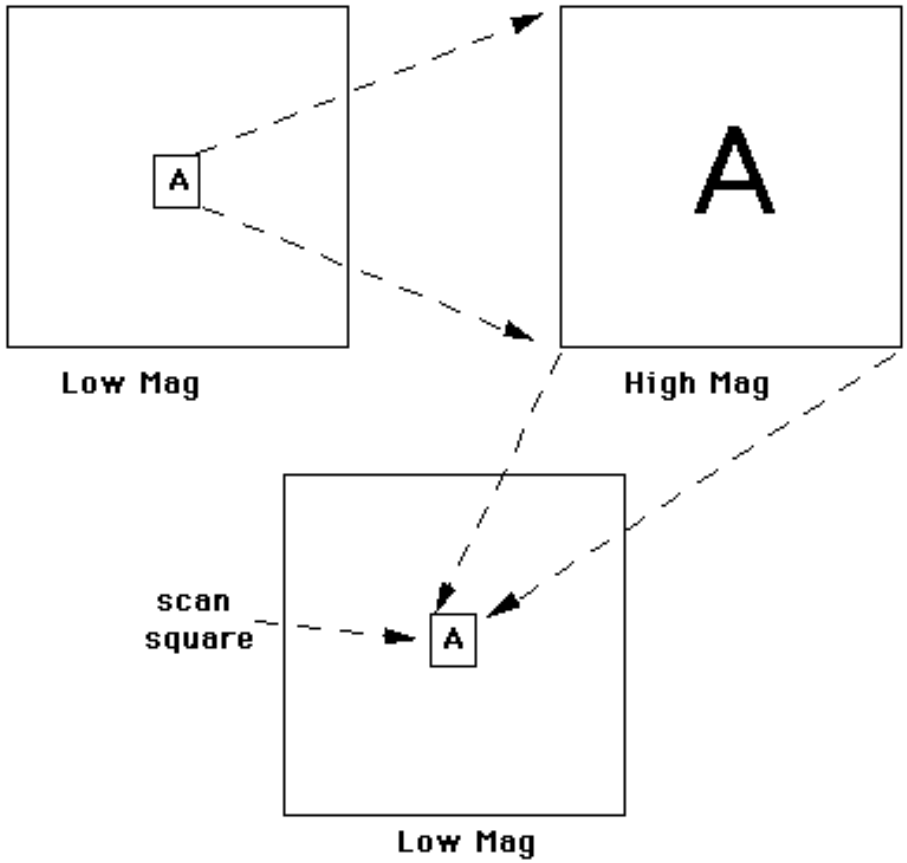
E2 values

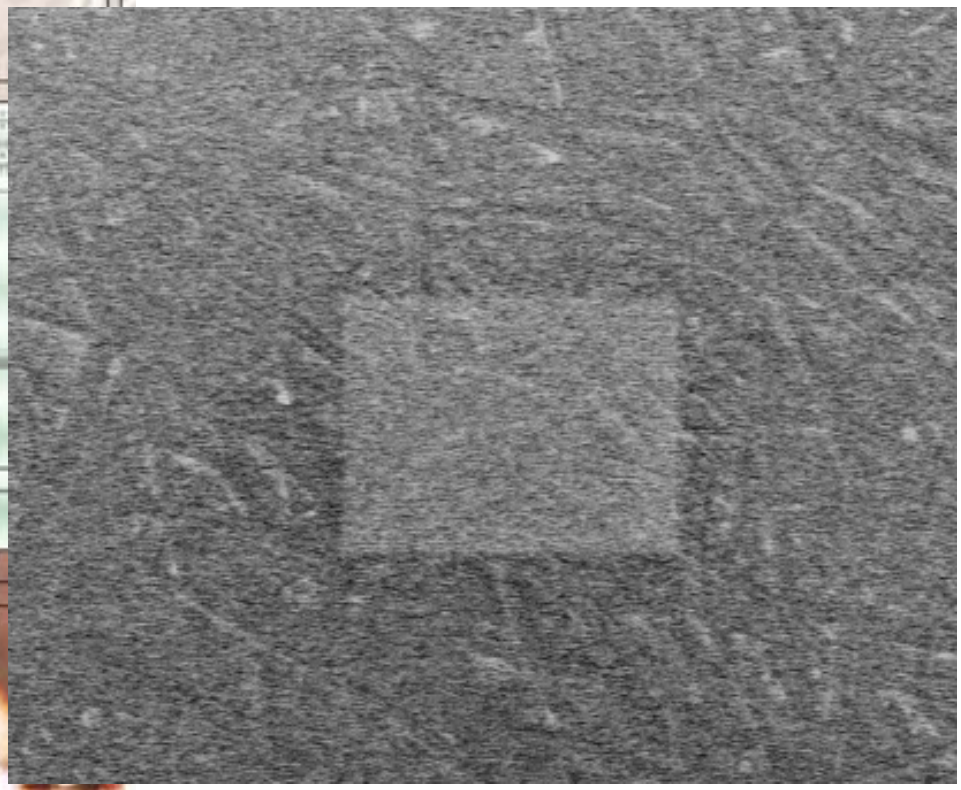
Material	E2(keV)	Material	E2 (keV)
Resist	0.55	Kapton	0.4
Resist on Si	1.10	Polysulfone	1.1
PMMA	1.6	Nylon	1.2
Pyrex glass	1.9	Polystyrene	1.3
Cr on glass	2.0	Polyethylene	1.5
GaAs	2.6	PVC	1.65
Sapphire	2.9	PTFE	1.8
Quartz	3.0	Teflon	1.8

Determining E2 in the Low Voltage SEM

1. Set the magnification to 100x and scan at TV rate
2. Increase the magnification to 1000x as quickly as possible
3. Count to five
4. Drop back to 100x magnification
5. Look at the scan square that is visible in the center of the screen .....



Negative charging



- If the scan square is brighter than the background then the sample is charging negative and the beam energy is greater than E2 (*or - just possibly - less than E1*)

Paper pulp at 2.5keV

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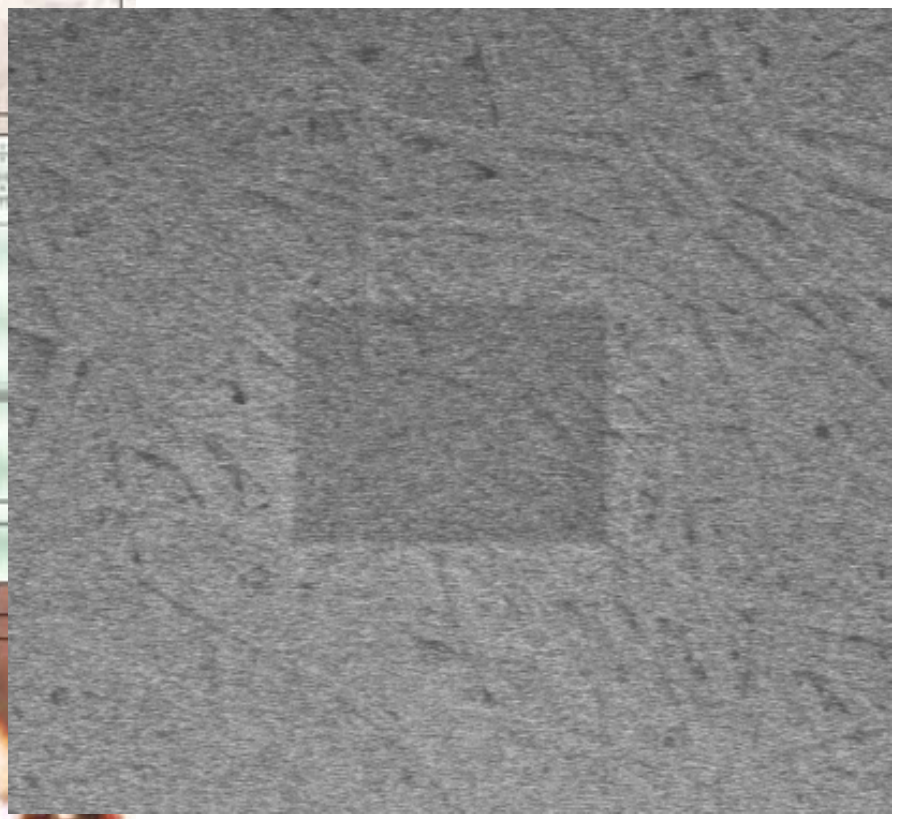
Positive Charging



- If the scan square is dark compared to the background then the sample is charging positive and the beam energy is less than E2 (and greater than E1)

Paper pulp at 1.2keV

Imaging non-conductors

- On a new SEM this will be the lowest available energy
- On older machines you must decide how low to go before the performance becomes too poor to be useful for the purpose intended
- The goal is to avoid implanting charge deep beneath the surface. If this is allowed to occur then stable imaging may never be achieved.
- Step #1 - Set the SEM to the lowest operating energy

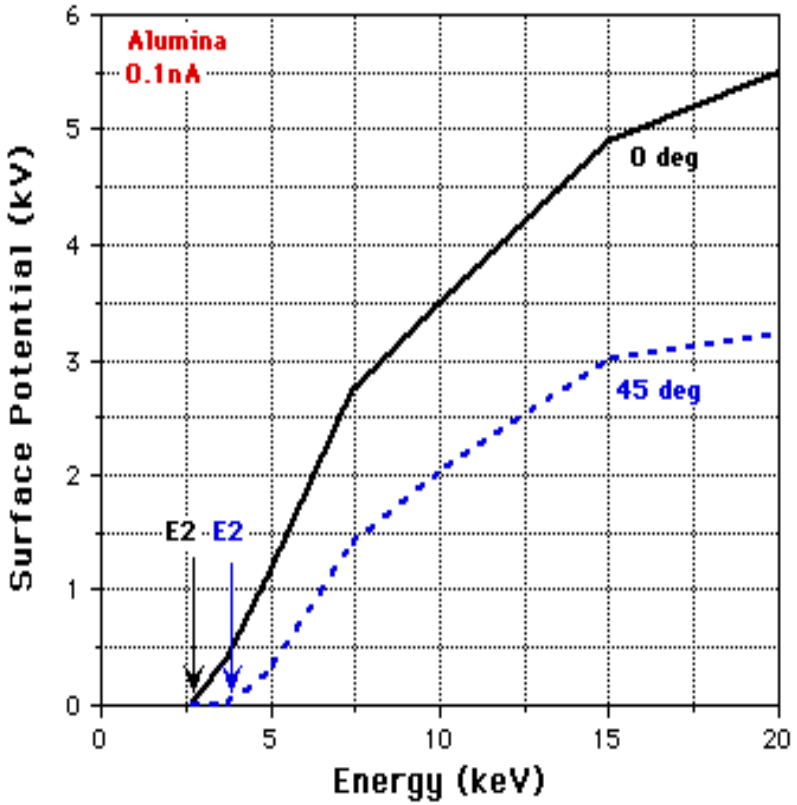
Next……..

- If the sample is charging positive (i.e. a dark scan square) then E1< E<E2. Increase the beam energy and proceed to image
- If sample is charging negatively (i.e. bright scan square) then E>E2.
- Since we cannot reduce E any further go on to step 3.

- Step #2 - Determine the charging state of the sample using the scan square test

Step 3

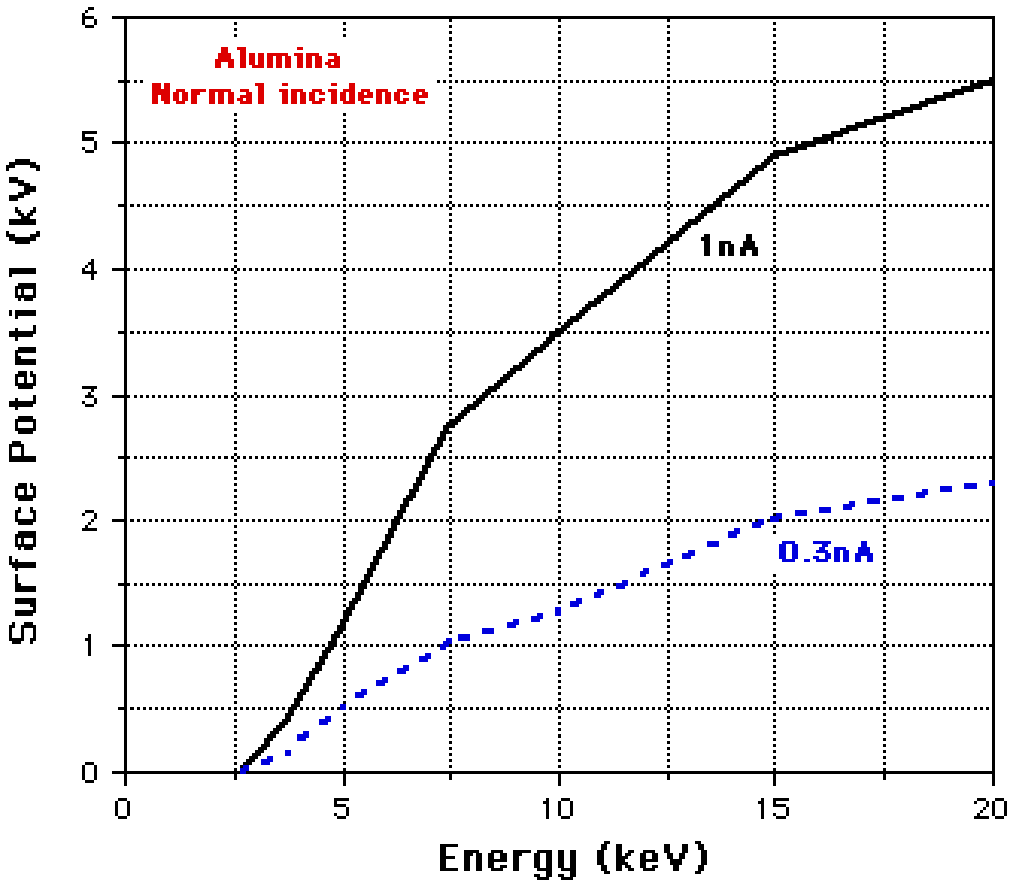
- Tilt the sample to 45 degrees and repeat the usual scan square test
- Can E2 be reached now?
- $E2(\theta) = E2(0)/\cos^2\theta$ so tilting by 45 degrees raises E2 by a factor of 2x
- But ..because E2 varies with the angle of incidence the 'no charge' condition can never be satisfied everywhere on the surface at the same time and charging will always occur



Note that tilting the sample reduces charging at all energies

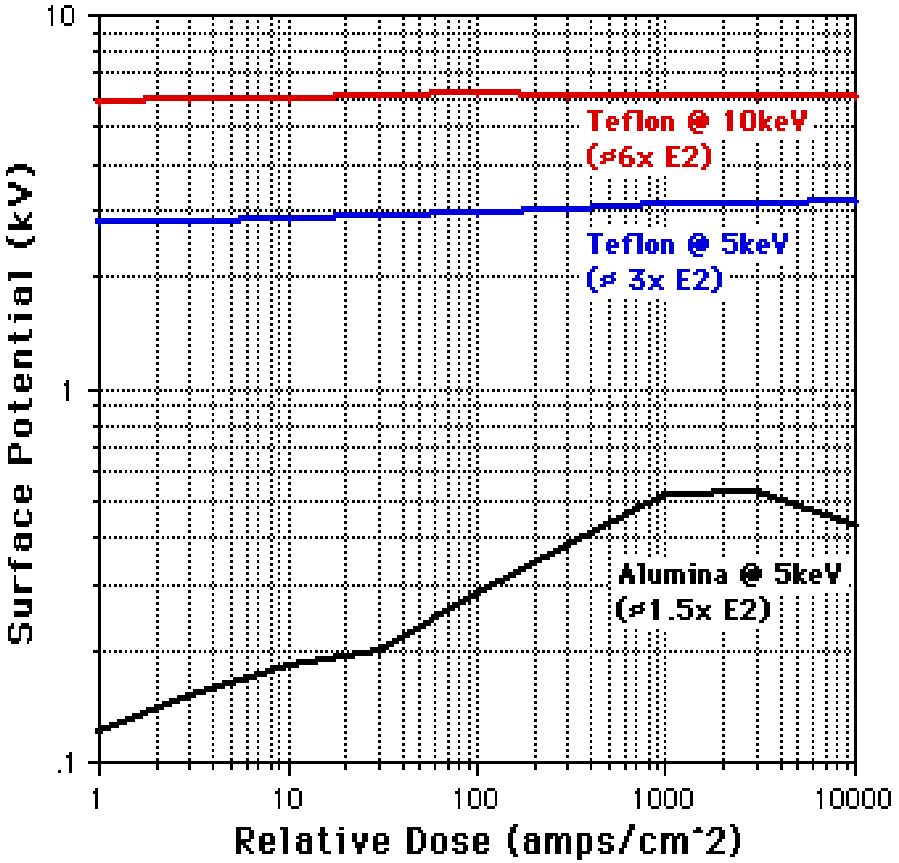
Living with charging - #1

- Reduce the beam current since the charging varies directly with I_B
- Use a smaller aperture, or reduce the gun emission current
- Reduces the S/N ratio so longer scan times may be required



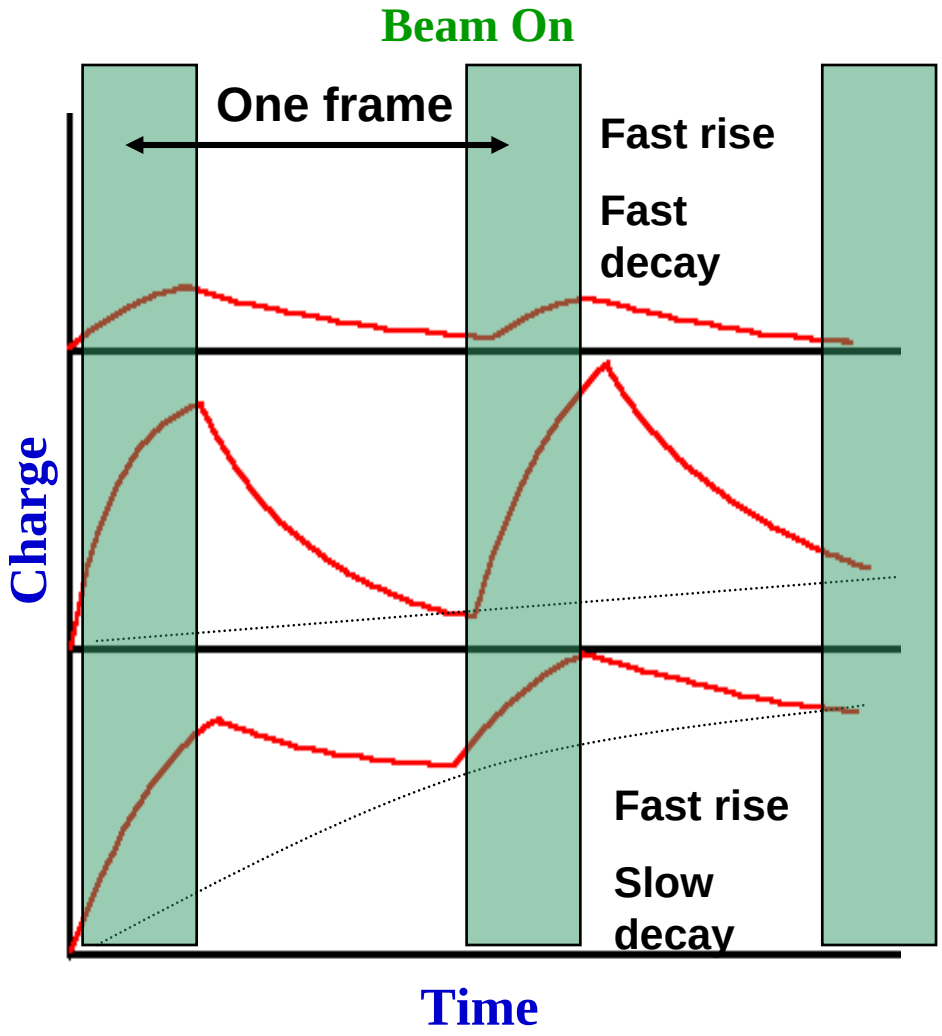
Living with charging - #2

- Reduce the magnification
- This minimizes Dynamic Charging (internal charge production from electron-hole pairs). The magnitude of this depends on the dose and hence on the magnification
- Dynamic charging is worst when E_0 is close to the E2 value
- Limits resolution by limiting magnification



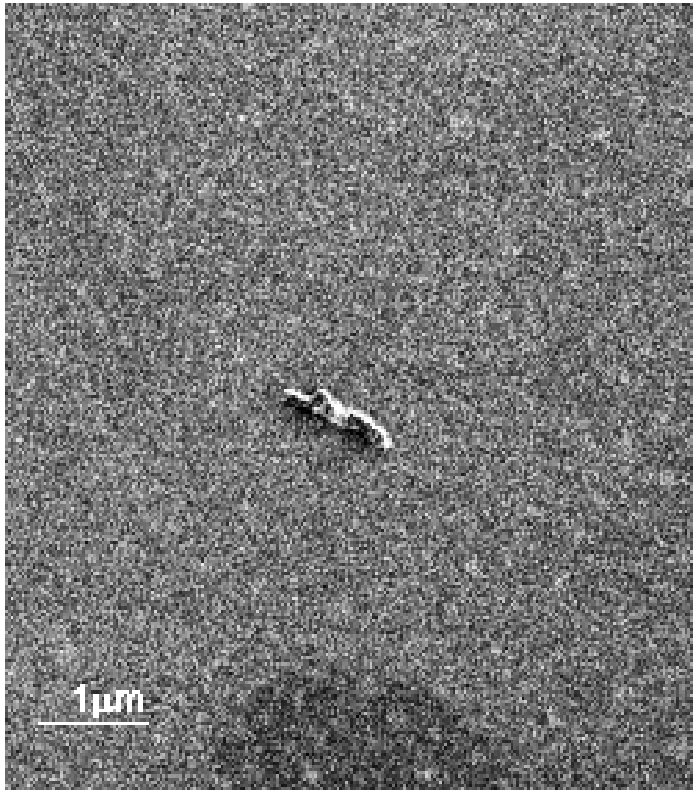
Living with charging - #3

- Charging is time dependent because the sample acts like a leaky capacitor
- Samples charge up more quickly than they discharge
- Depending on the scan rate and the relative charge up and decay rates samples can
  - float at a steady potential or
  - gradually acquire a charge
- Generally at TV scan rates sample potentials float in a stable manner so focussing and stigmation are possible



Choosing a detector

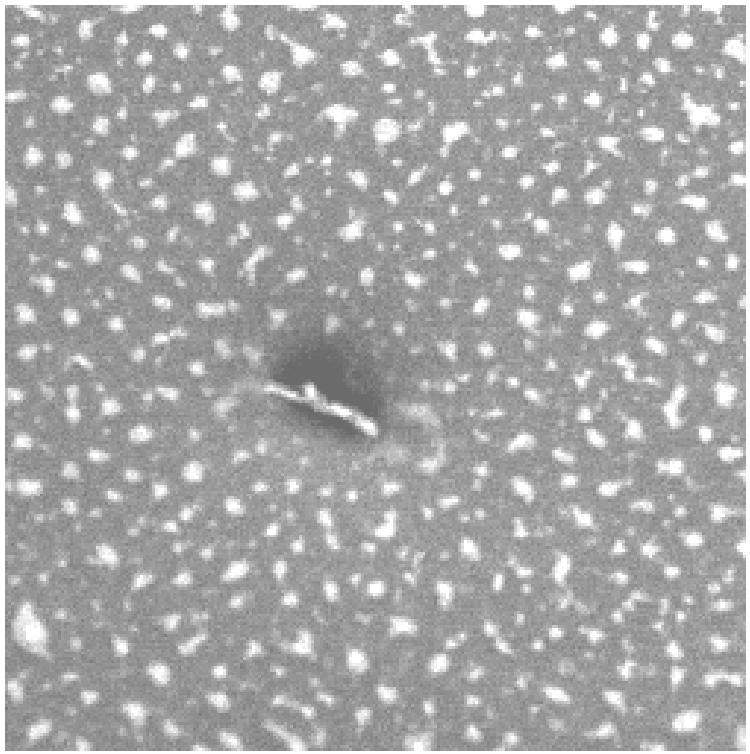
- The choice of detector can have a significant effect on the apparent severity of charging
- The conventional ET (Everhart - Thornley) detector is much less sensitive to charging than…



Individual polymer macro-molecules on Si at 1.5keV - Lower (ET) detector

Upper detector

- ...a through the lens detector. This is because TTL systems act as simple SE spectrometers and preferentially select low energy electrons
- Note however that charging can be a useful form of contrast mechanism when properly employed



Same area as before, TTL detector

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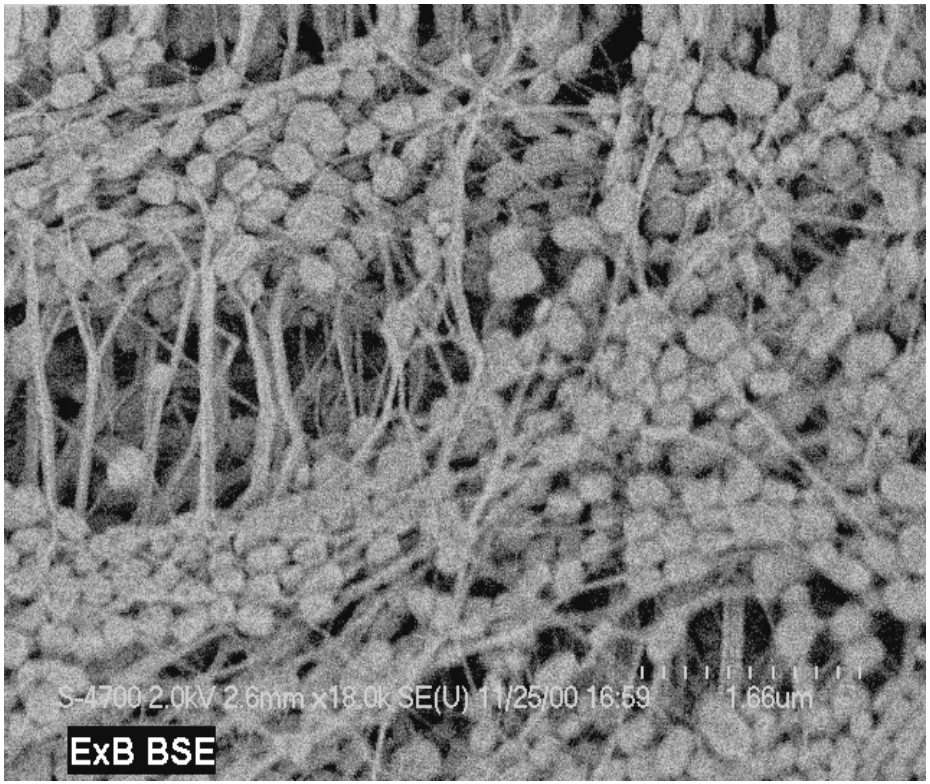
BSE imaging to avoid charging

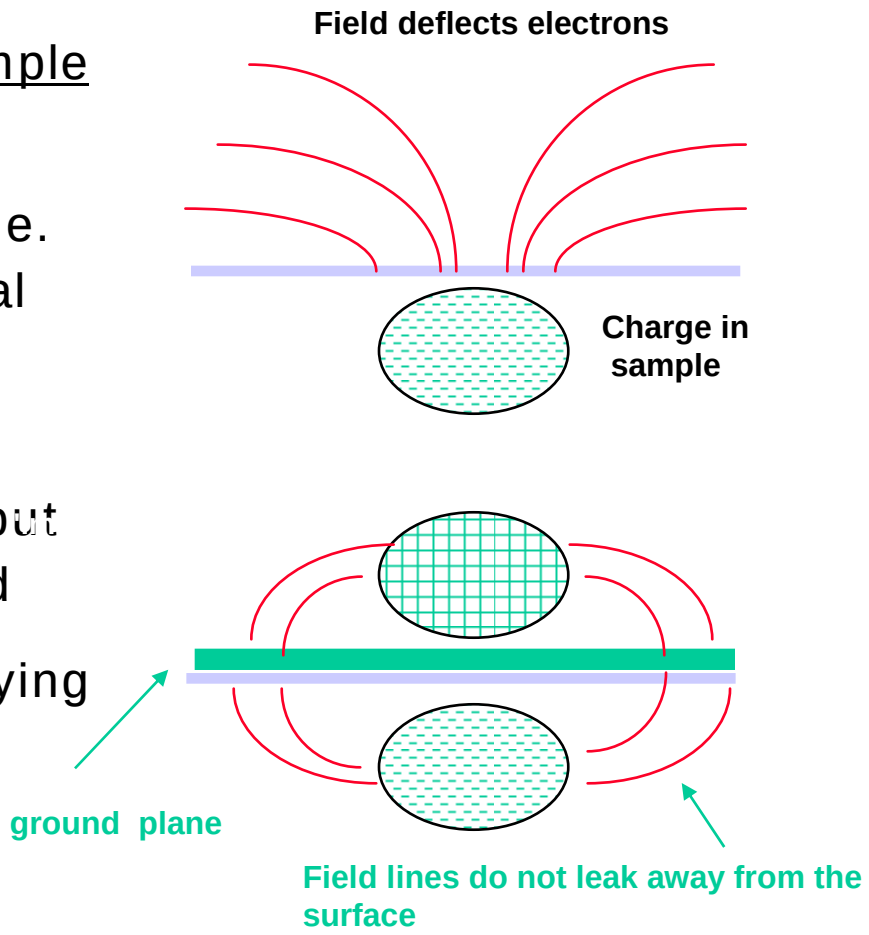
- Backscattered electrons are less affected by charging and offer the same resolution at LV
- Newer technologies such as conversion plates, and ExB filters, for BSE actually improve in efficiency as the beam energy is reduced, so using this mode to avoid charging problems becomes a good choice



Uncoated Teflon
S-4700 ExB BSE image

If all else fails.....coat the sample

- Coatings do not make the sample a conductor
- They form a ground plane - i.e. the free electrons in the metal move so as to eliminate the external field
- The charge is not eliminated but the disruptive field is removed
- Successful coating means paying attention to the details...



To ensure good coatings (for high resolution or low voltage)

- Keep it clean - wipe the glass vessel clean after every run, and clean the anodes weekly
- Keep it slow - reduce the gas pressure and/or the anode voltage till plasma just stays on
- Keep it thin - thicker coatings do not work better and they obscure surface detail. Aim for no more than 5nm of Au/Pd, 2nm of Cr
- Keep it dry - the argon gas (never air!) must be perfectly dry. Check the color of the plasma.

and furthermore…..

- All that glitters - do not use pure gold because of its high surface energy. Use Au-Pd, Ir, or Pt to ensure good, thin, particulate, films
- Evaporated Carbon is a contaminant not a coating. Carbon is a poor conductor, produces only a small number of secondary electrons, evaporated carbon is usually mixed with a filler compound, and the thickness cannot be well controlled. Either use a ion sputter coater for C or avoid it altogether

Unwanted Beam Interactions

Radiation Damage

Ionization
Displacement
Heating

Intrinsic to electron beam irradiation

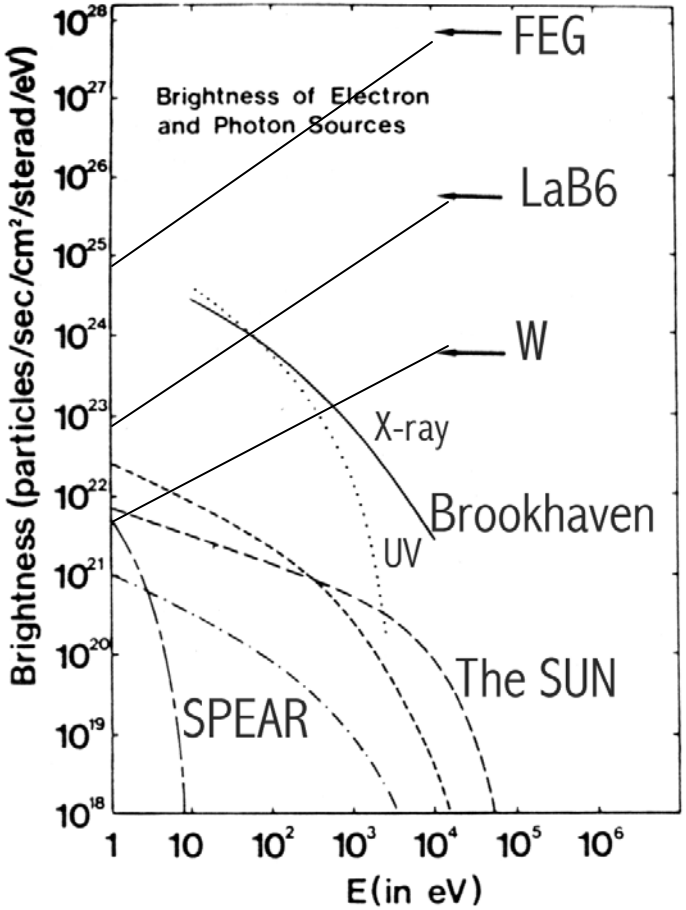
Contamination
Etching

Results from vacuum problems

Both are usually important

Radiolysis

- Ionization damage is most important threat to organic, and some inorganic, materials.
- Electrons are the most intense source of ionizing radiation available - the typical dose in an SEM is equivalent to standing 6 foot from a 10 megaton H-bomb



Compare SEM to Sun and SPEAR

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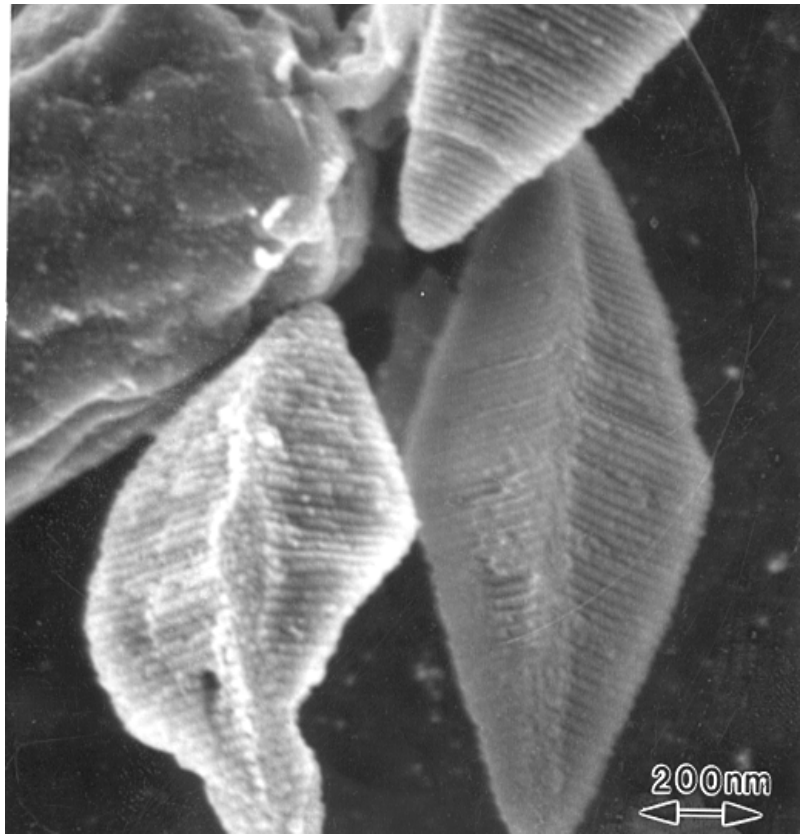
Effects of radiolysis

- Destroys the crystalline structure of polymers, and other organic crystals, leaving them amorphous
- The probability of radiolysis is 10x to 100x bigger than the chance of generating an X-ray
- Damage competes with signal generation - damage usually wins

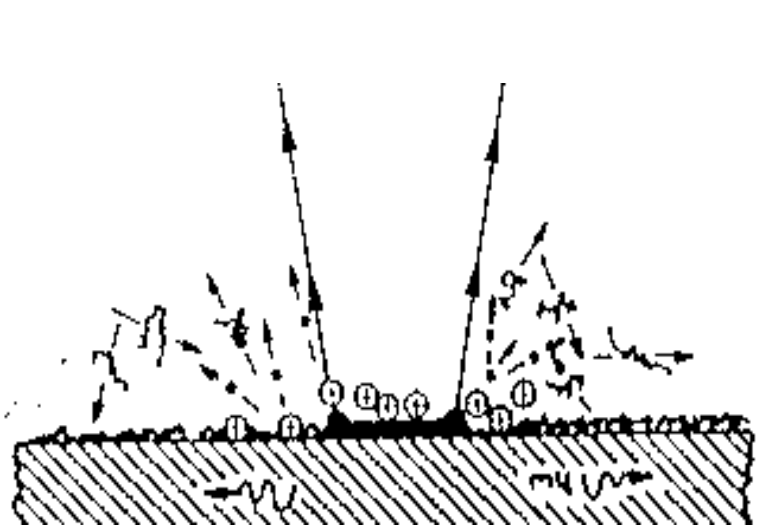


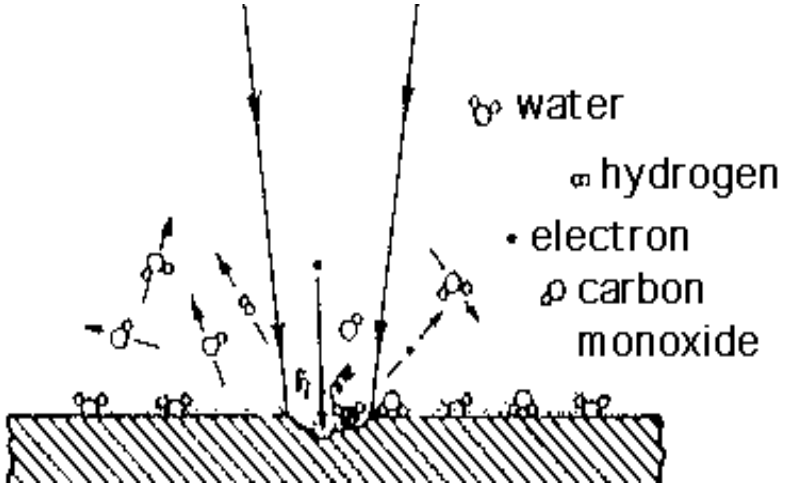
Damage to Protein Protoxein crystals from imaging

Contamination - Etching

- Contamination is beam induced polymerization of the hydrocarbons present on the sample surface
- Etching is the removal of surface layer by impact of ions ($C + H_2O^{2-} \rightarrow CO + H_2$)
- Both phenomena are affected by surface charging and often occur together
- Both are temperature dependent

Contamination and Etching

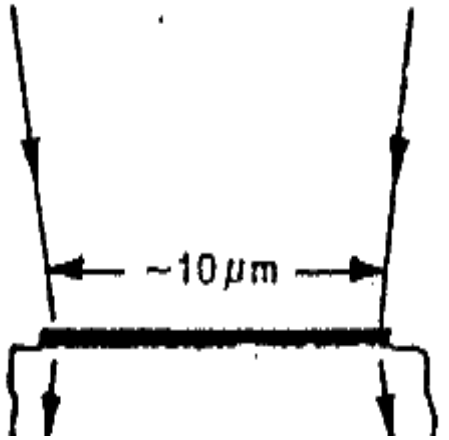




Electrons break down the hydrocarbon film. The residue charges +ve and the field pulls in fresh material for radiolysis. If water vapor is present then H_20^{2-} ions go to the + ve charged region and etch that area away

Low magnification

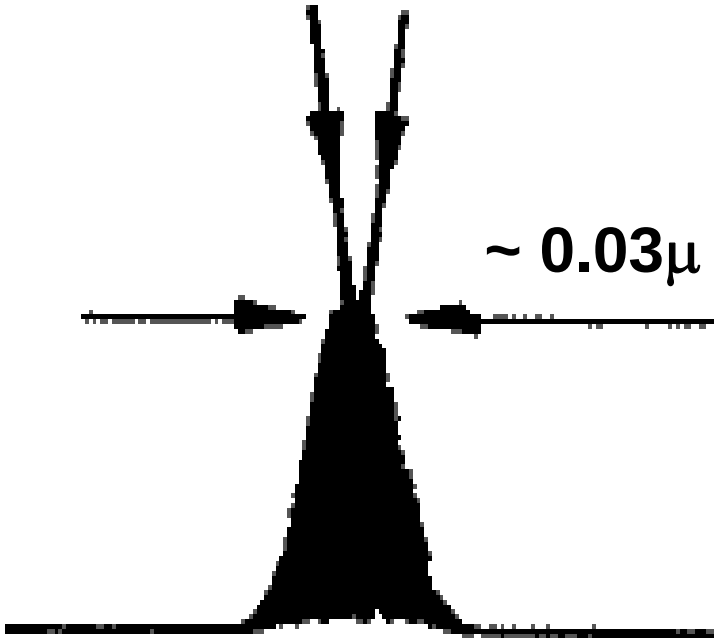
- At low magnification the hydrocarbon film is polymerized into a thin sheet.
- This will charge positive (and so look black in the SE image) but is not a serious problem



Schematic of contamination build-up at low magnification scans

High magnification

- At high magnification the contamination grows a cone which prevents the beam reaching the surface
- So avoid spot mode - always keep the beam scanning the sample
- Try and pre-pump samples before use
- Keep your hands off the sample



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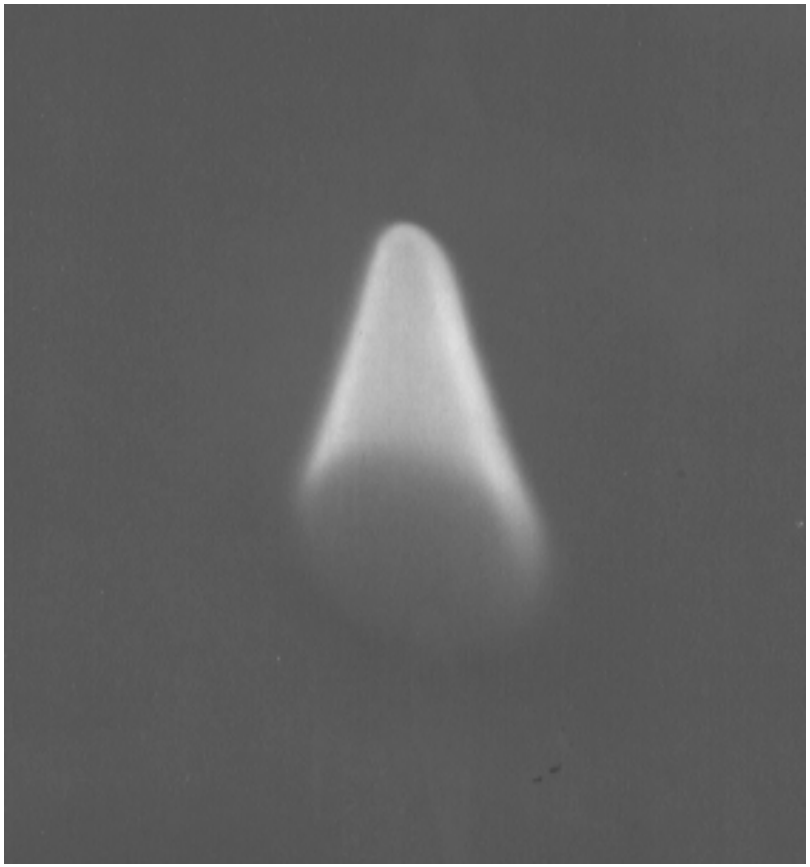
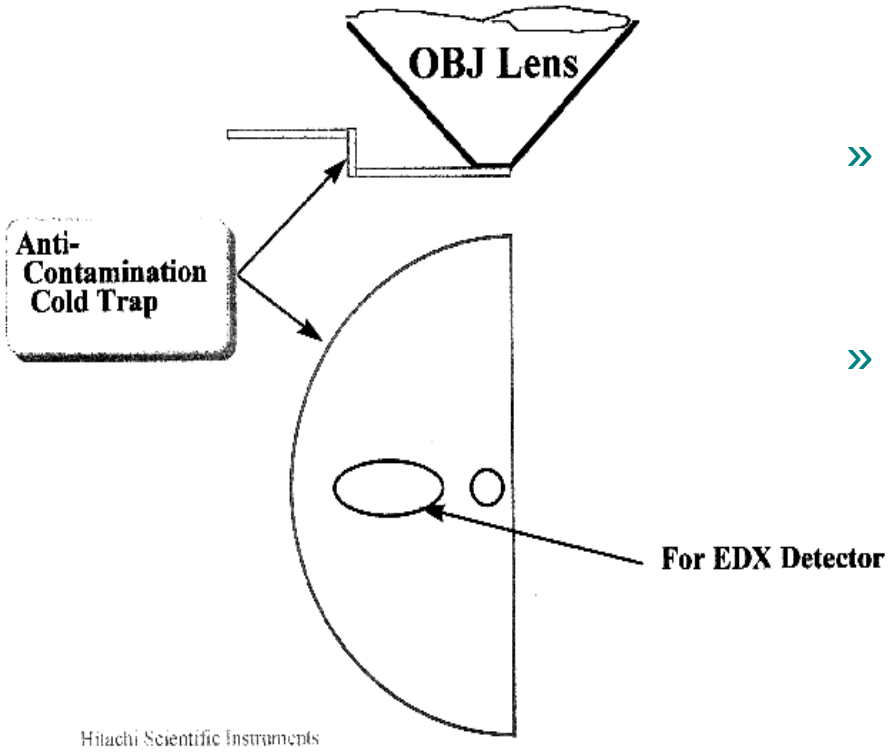
Virtue of necessity..



300A high cone grown on a silicon wafer in 5 minutes exposure

- Contamination cones can grow to a height of hundreds of angstroms and are very tough - used for high resolution AFM tips
- Prevent this growth by irradiating the area at low magnification before going up to a high magnification

The Cold Finger



- Standard fitting on S-4800 & S-4700, available as an option for the S-4500
- The finger is held at LN2 temperatures, a few mm from the specimen surface
- After filling the cold finger allow the sample enough time to reach thermal equilibrium before starting to image