Talos on-line help manual -- Working with a FEG (X-FEG)

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1 FEG Safety

1.1 The column valves

The microscope is equipped with numerous safety features to ensure that no damage is done to the emitter tip. Nevertheless it is possible to cause temporary or permanent damage to the tip if a sudden vacuum breakdown occurs while the gun is open to the column. The microscope is therefore equipped with the ability for the user to open and close the column valves.

The gun area, including the intermediate block, accelerator and emitter area, is separated from the specimen area by valve Vac, one of the two column valves (the other, Vcp, separates the projection chamber from the column).

Note: It is advised to keep the column valves closed at all times possible :

- During exchange of specimens when the specimen holder is extracted from or inserted into the microscope;
- When the microscope is not actively used;
- When the IGPco vacuum is higher than 26 (log units).

Never introduce the specimen holder into the microscope with the gun valve open !

Make sure that the level of IGPco (Column) is at or below 26 (log units) before opening the column valves.

1.2 Optimal extraction voltage

For stable performance, the emitter is best operated at the optimal extraction voltage. This optimal value is the best balance between brightness, energy spread, and emitter lifetime. It is an emitter-dependent parameter which is set by the factory or service engineer. It is independent of acceleration voltage. The optimal extraction voltage V-optimal is briefly shown as a ‘tool-tip’ whenever the cursor moves over the extraction voltage input field.

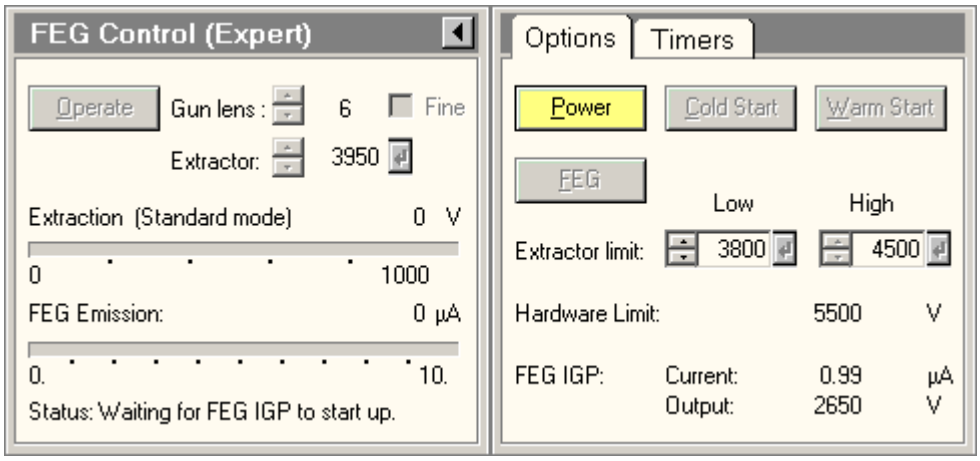
1.3 Dual Mode

For some applications, there is the need to decrease the extraction voltages to a value below V-optimal, for instance to obtain a higher energy resolution for fine EELS analysis. The software does allow the use of reduced extraction voltage, but the time in this mode (the so-called ‘Low Mode’) is limited to 12 hours (which can be extended by 4 more hours). After this, the Dual Mode software automatically brings the system to the ‘Standby’ sub-state, that is, at the optimal extraction voltage. After a certain amount of time (depending on the history), the Low Mode becomes available again. It is good practice to keep an X-FEG at the optimal extraction voltage overnight or whenever not attended for a longer period of time. This is done by using the Operate button to switch between Standby sub-state (which is at V-optimal) and Operate sub-state (which is at the user-defined extraction voltage setting).

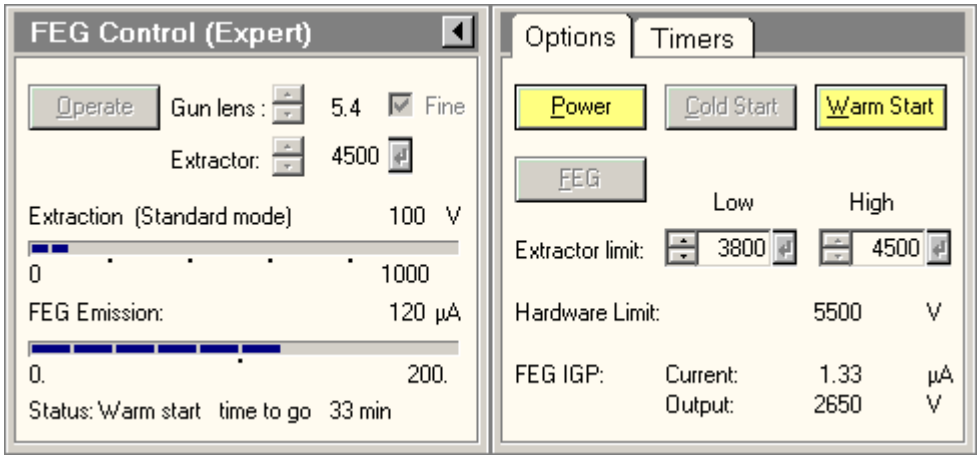
2 FEG States

The FEG has a number of operational states, described by the following conditions:

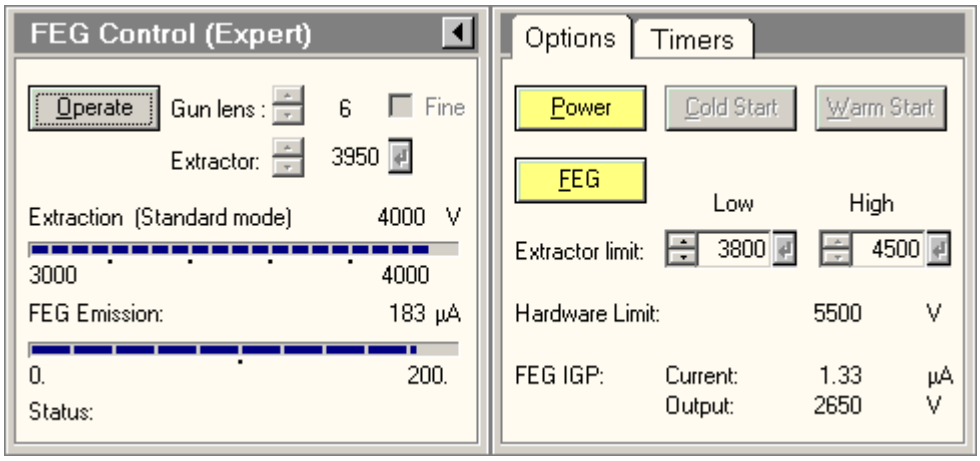
- **Power on** : IGPf is running (or attempting to start up), but otherwise all FEG electronics are off. The Power button is yellow, the FEG button is disabled. Warm and Cold Start are enabled once IGPf is running.



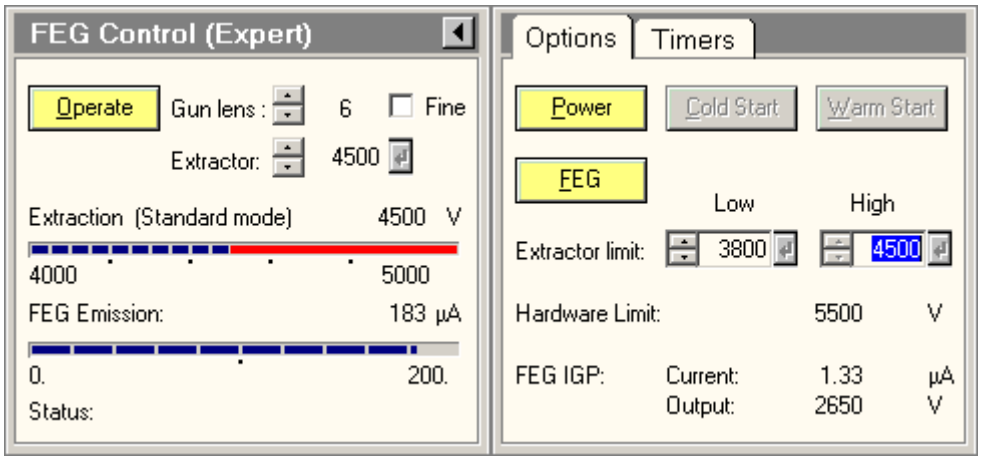
- **Cold Start** or **Warm Start** : FEG Start-up procedures, leading to FEG on.



- **FEG on** : all electronics are on. The Power and FEG buttons are yellow.
    1. **Standby** : a FEG-on substate with the extraction voltage at V-optimal. The Operate button is gray. It is disabled when the high tension is off and enabled when the high tension is on.

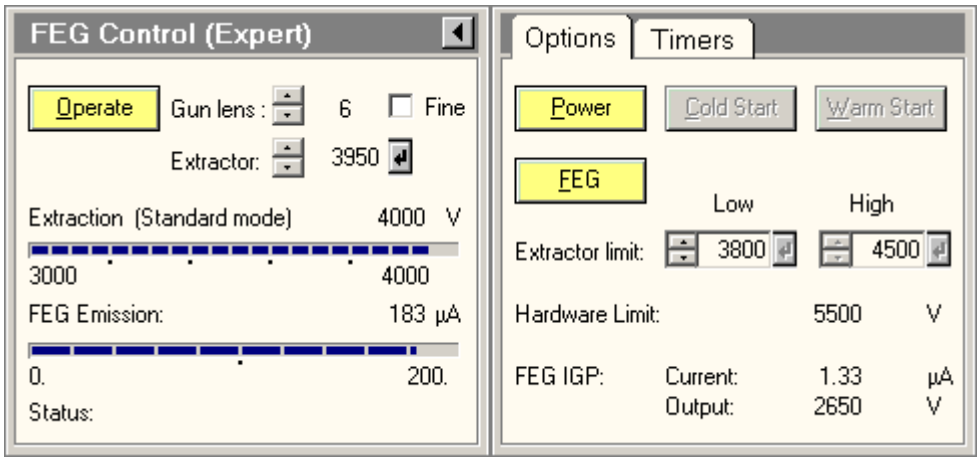


    2. **Operate** : a FEG-on substate with user-defined extraction voltage. The Operate button is yellow.



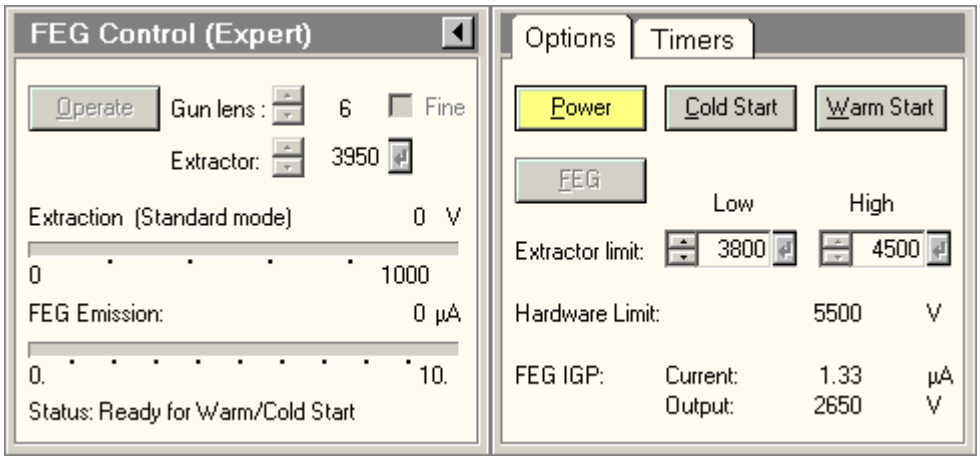
3 Starting the FEG

Note: Until the FEG has been started - through either the Warm or Cold Start procedure - it is not possible to switch the high tension to more than120 kV. Switching high tension on is possible once the extraction voltage is higher than 1000 V.



- Press the Power button. The software will now start IGPf (the Cold Start and Warm Start buttons will not be enabled until a current has been measured on IGPf ; after the initial current in IGPf, the value may drop to zero, but the initial current shows that the pump is truly running). If IGPf doesn't start within a certain amount of time, the software will switch off IGPa. This will slightly deteriorate the vacuum and make it easier for IGPf to start up. When IGPa is switched off, the system may display an error message 'Gun pressure too high' which can be ignored (click on the Enter button to remove it). Once IGPf has started, the software will (if it had been switched off) restart IGPa. Once IGPa is running again (checked by a significant lowering of the IGPf pressure), the system is ready for a Warm Start or Cold Start.

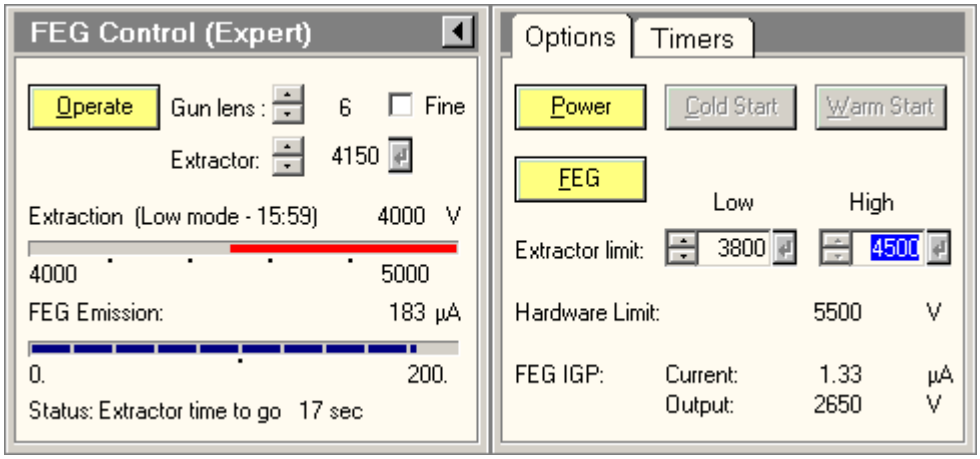
  For X-FEG, the Warm and Cold Start procedures are stopped if the FEG IGP pressure exceeds the value of 1 µA. It is recommended to let the pressure decrease to values below 0.5 µA (preferably ~0.1 µA) before the FEG is started via a Cold or Warm Start.

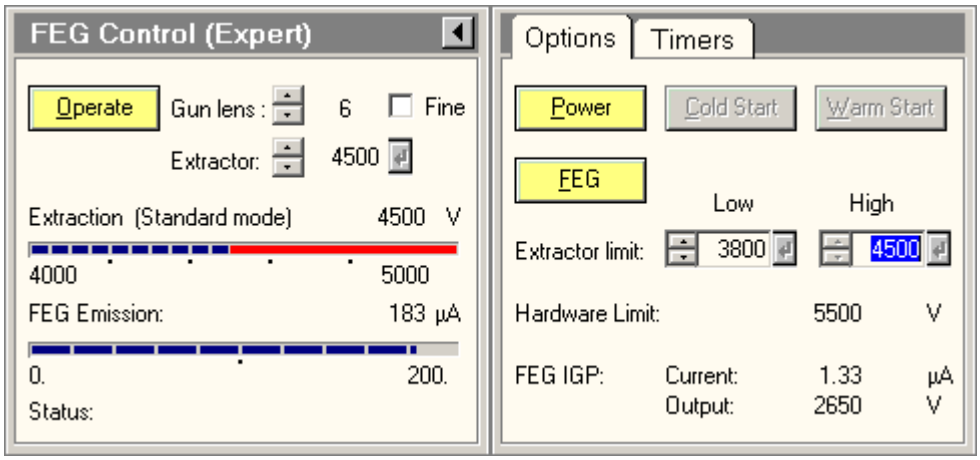


- Press the Warm Start (normal procedure if IGPa has not been off longer than 24 hours) or Cold Start to begin the FEG start procedure. The time required (under normal conditions) for the procedure is displayed. Note that the timing display calculation runs on the PC while the actual timing is done on

an electronics board so discrepancies of up several minutes in the timing display and the actual timing used may occur.

- Select the maximum allowed high tension (120 kV) and switch high tension on.
- The start-up procedure will lead to the FEG on, Operate state, with the extraction voltage at the optimal extraction voltage V-optimal.
- Change the Extraction voltage and gun lens settings to the values required for operation (the recommended extraction voltage is V-optimal).





4 Shutting the FEG down

Note: For X-FEG, it is discouraged to leave the FEG at an extraction voltage in Low Mode (low extraction voltage) when the microscope is not being used. You can use the Operate button to switch between the operating condition and the Standby sub-state, which is at the optimal extraction voltage V-optimal. Note that the FEG automatically leaves Low Mode and switches to Standby after a certain amount of time or when the column valves have been closed for 2 hours. If the extraction voltage is in the Standard mode, the FEG may be left in this operating condition.

The FEG can be shut down partially (all electronics except the power to IGPf off) or completely. To shut the FEG down but keep IGPf running, press the FEG button (this action must be confirmed before it is executed). To shut the FEG down completely, press the Power button (also subject to confirmation).

5 FEG Design

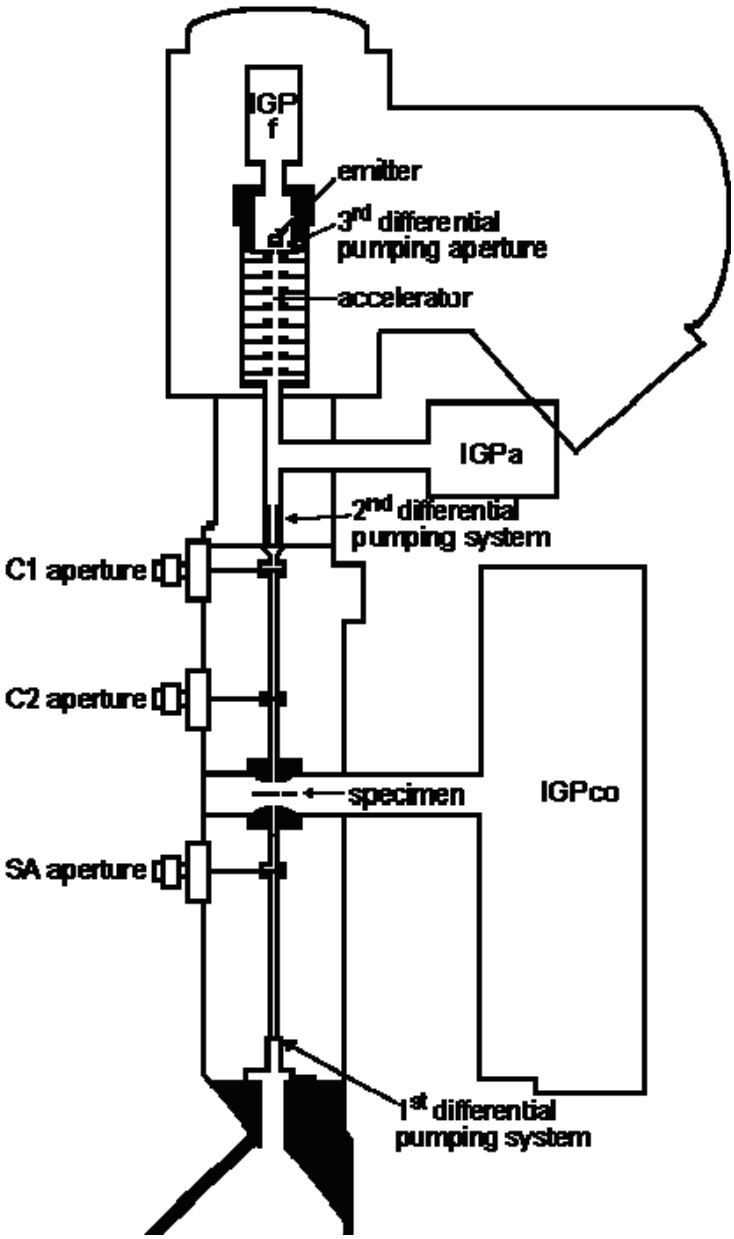
5.1 Electron source

The electron source of the TEM FEG microscope is a field emitter operating in the Schottky mode. The emitter consists of a tungsten single crystal in <100> orientation, covered with a thin layer of zirconia (zirconium-oxide). The zirconia lowers the work function of the emitter (the function that determines the energy level needed by electrons to make the jump from the tungsten metal to vacuum) when the emitter is heated to approximately 1800 K.

5.2 Vacuum system

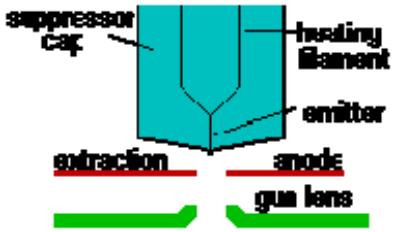
The vacuum system of the TEM FEG contains four stages with differential pumping apertures between them:

- The detection unit (projection chamber), pumped by the turbo-molecular pump.
- The specimen environment, pumped by the main ion-getter pump IGPco to a level of ~10^{-7} Torr.
  Prepumping is done by the turbo-molecular pump (and its backing diaphragm pump). The turbo-molecular pump also pumps on the specimen-holder airlock.
- The intermediate block and accelerator, pumped by a second ion-getter pump IGPa to a level of ~10^{-8} Torr.
- The emitter area, pumped by the third ion-getter pump IGPf mounted directly above the emitter to a level of 10^{-8} to 10^{-9} Torr.



Schematic diagram of the four-stage vacuum system of the TEM FEG.

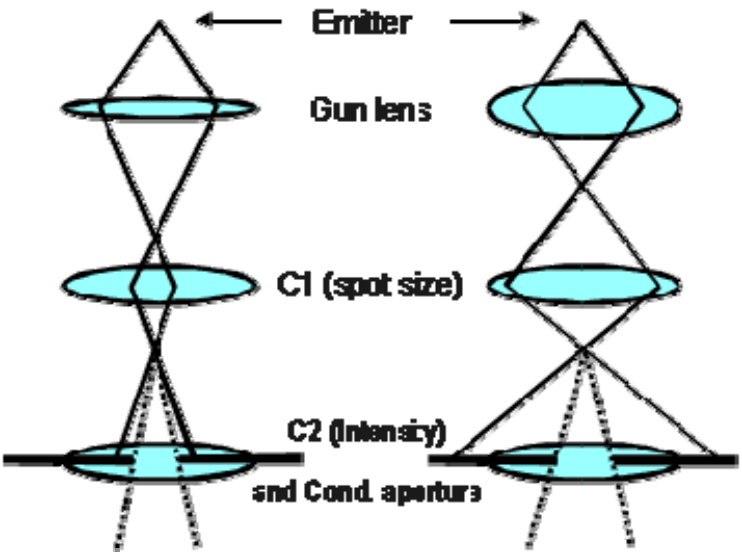
5.3 Gun unit



The gun unit contains the field emitter, a suppressor cap, an extraction anode and an electrostatic gun lens. The field emitter is mounted on a tungsten hairpin used for heating. The suppressor cap around the emitter (only the tip of the emitter itself protrudes out of a small hole in the front of the suppressor cap) blocks electrons emitted by the heating hairpin or the shaft of the emitter and is at a constant negative voltage. The extraction anode provides the extraction voltage in the range of 1.8 to 5.5 kilovolts, that controls electron emission from the emitter. Electron emission (and the gun brightness, see below) increases with extraction voltage up to an emission level of about 500 μA.

5.4 Mechanical design of the Schottky FEG module

The electrostatic gun lens converges the electron beam. If the gun lens is weakly excited, the cross-over occurs lower in the column and more electron current passes through the condenser aperture. If the gun lens is more strongly excited, the cross-over moves up the column and less electron current passes through the aperture. At the same time, the image of the source is more demagnified, allowing the smallest probes to be obtained. The gun lens thus operates quite similar to the spot size (C1 lens).



Schematic diagram showing the effect of the gun lens on electron-optical conditions.
Left: gun lens weakly excited.
Right: gun lens strongly excited.

5.5 Emitter life

The zirconia on the emitter slowly disappears during electron emission. The rate of zirconia loss is determined by the intensity of electron emission and by the quality of the vacuum. When the stock of zirconia runs out, the emitter will stop working (the electron emission drops to a very low level) and must be replaced (by Service)
Some variability exists between individual emitters in the emission currents as a function of extraction voltage. Average emission currents are 200 - 400 μA at the optimal extraction voltage, but lower or higher values do also occur. If excessive emission currents are found, even at low extraction voltages, please contact Service.

5.6 Brightness, high-resolution imaging and small probes with high currents

The performance of an electron gun depends critically on two factors: the energy spread (for the temporal coherence in high-resolution imaging and for high- resolution energy-loss spectroscopy) and the brightness (for the spatial coherence in high-resolution imaging and for small probes with high currents).

Brightness is a parameter that describes the electron current density per unit solid angle. Often the reduced brightness B_r is used, which is normalized to the acceleration voltage:

$B_r = 4\ I / \pi^2\ \alpha^2\ d_g^2\ V^*$

where I is the probe current, α the convergence half angle, d_g the geometrical probe size, and V* the acceleration voltage (with relativistic correction). For an ideal system, without aberrations or Coulomb interactions, the reduced brightness is a preserved quantity.

Brightness describes the relation between beam current, opening angle, and probe size. Given a certain probe current I and convergence half angle α, the geometrical probe size is given by:

$d_g = 2\ \sqrt{(I / B_r\ V^*)} / \pi\ \alpha$

The higher the brightness, the more current we can put into an electron beam of given size. Alternatively, with higher brightness, a smaller (geometrical) probe size can be obtained with the same current (note that there are also other factors, apart from brightness, which influence the definitive probe size, like lens aberrations and system disturbances).

The reduced brightness of an S-FEG is typically in the order of 0.5-2 10^7 [A/ m^2 sr V] and depends on the extraction voltage. An X-FEG typically has a reduced brightness of 0.7-1.5 10^8 [A/ m^2 sr V] at the optimal extraction voltage.

For more details on small probes and high currents, see P.B. Kenway (1987) Probe size and current: some universal curves. Inst. Phys. Conf. Series 90, 101.

5.7 FEG Gun Lens Settings

5.7.1 Gun Lens Setting

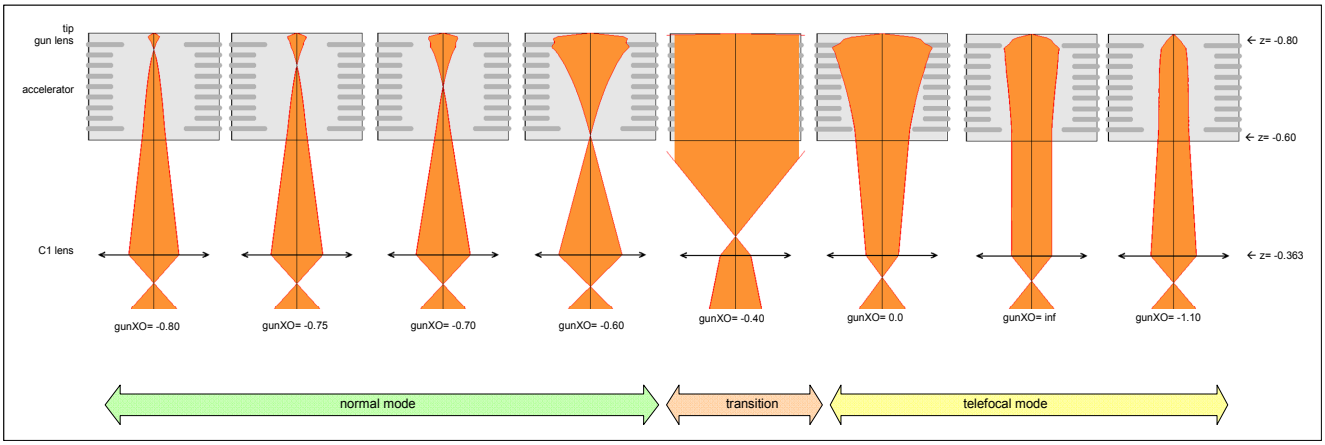
The strength of the gun lens can be changed by changing the gun-lens setting (GL) in discrete steps, from 1 to 8. With ‘fine gun lens’ enabled, the resolution can be increased to stepsizes of 0.2, thus making available intermediate gun lens strengths. Fine Gun Lens is enabled when the ‘Fine’ checkbox in the control panel is checked.

The focusing strength of the gun lens depends on the gun lens setting, the extraction voltage, and the acceleration voltage.

5.7.2 Normal and telefocal imaging

The position of the first cross-over in the condenser system depends on the focusing strength of the electron gun, which is an electrostatic lens composed of the extractor, gun lens electrode, and the first anode of the accelerator. If the lens is sufficiently strong, an intermediate image of the source is formed in front of the C1 lens. This is the preferred situation.

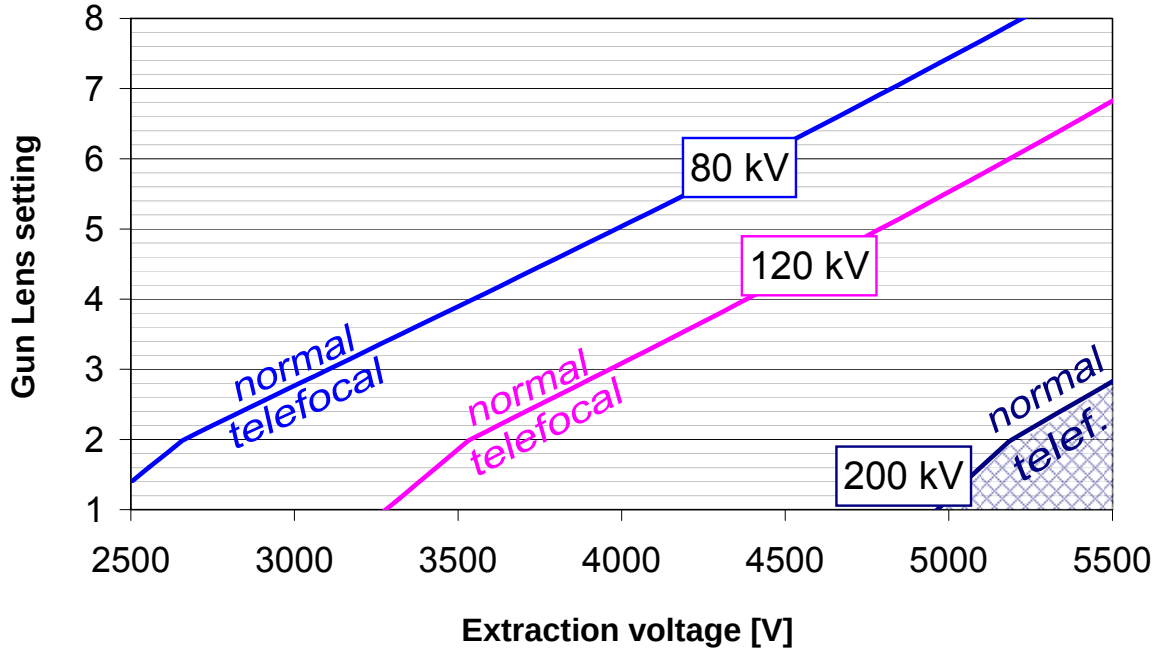
If the gun lens is weak, no intermediate image is formed in front of C1. This is called the telefocal mode. The gun optics can be difficult to align in telefocal mode, so are correctors. In the transition region between normal and telefocal, the gun-optics can not be aligned at all.



The Gun Lens setting at which the transition occurs between normal and telefocal mode is indicated in the next graphs. This GL setting is a function of both extraction voltage and accelerating voltage. The normal mode is at the upper left side of the line indicating the transition; telefocal mode is found at the lower right side of this line. Note that the exact position of the transition can differ slightly from system to system due to mechanical and electrical tolerances.

5.7.3 X-FEG

The graph for a 200 kV X-FEG accelerator is shown below. At 200 kV, the gun lens is in normal mode for all extraction voltages and gun lens settings, except for very high extraction voltages with low gun lens settings. For lower HT values, the region of telefocal mode increases; the transition shifts to lower extraction voltages and higher gun lens settings.



6 FEG Operating Modes

This section is intended to offer guidance on starting settings for achieving various modes of operation of the microscope. In general terms:

- Aperture sizes influence coherence, convergence, spot size and total current
- A strongly excited gun lens minimizes the aberrations of the gun giving the smallest probe sizes but also limiting the total current in the probe.

Thus the settings given below represent a compromise of competing factors. The user is free to deviate from these settings in order to achieve the desired results for his or her application.

In practice for small-spot work the gun lens and C1 (spot size) are somewhat interchangeable: if a certain probe current is obtained, then a corresponding spot size is achieved. However, the best operating conditions with respect to aberrations are achieved by balancing gun lens and spot size. Thus the combinations gun lens 4 + spot 8, gun lens 6 + spot 6 and gun lens 8 + spot 4 may result in the same beam current, but the gun lens 6 + spot 6 will typically provide a higher-quality spot than the other combinations. Hence, when changing to smaller or larger spots, change both gun lens and spot size.

The optimal extraction voltage, V-optimal, is set by the factory and service engineer. It is advised to use V-optimal for most applications, as it gives the best balance between brightness, energy spread, and lifetime of the emitter. At the optimal extraction voltage, the emission current can be from 200-400 uA, although lower and higher values can also occur.

6.1 TEM bright-field and dark-field imaging

Mode	TEM BF (microprobe)
C1 aperture	2 mm
C2 aperture	150/100/50 μm
Objective aperture	10 μm or larger if necessary
Extractor	V-optimal
Gun Lens	3
Spot number	2

Conditions are chosen to achieve good intensity for viewing of high-contrast specimens, typically requiring ~30 nA of total beam current. For applications where a higher beam current is needed (weak-beam imaging, elemental mapping with Imaging Filter) the maximum beam current is advised, obtained at gun lens 1 and C2 aperture 150 μm. In general re-alignment of the gun is not of critical importance. Only if the beam is cut-off not only by the C2 aperture, but also by the XFEG aperture (which can be seen when there appear to be two apertures limiting the beam), the gun needs to be re-aligned.

6.2 High-resolution imaging

Mode	TEM BF (microprobe)
C1 aperture	2 mm
C2 aperture	100/50 μm
Objective aperture	100 μm (if used or any other to select a specific range of diffracted beams)
Extractor	V-optimal
Gun Lens	3
Spot number	2
Magnification	500 kx to 750 kx
Exposure time	1 - 5 seconds

Conditions are chosen to maximize the spatial coherence (approximately 0.1 mrad) at a beam current that allows a reasonable exposure time at high magnification. V-optimal is recommended as extraction voltage, but eventually a lower extraction voltage can be selected in order to minimize the energy spread.

The C2 aperture size can be chosen dependent on the stability of specimen. Sometimes it is better to use a larger aperture and defocus the beam (especially if the specimen contaminates, since the contamination will be trapped at the edge of the beam, which is then far from the interesting area with a large beam). A large aperture will make orientating the specimen more difficult, however (larger discs in convergent beam diffraction pattern). Also, if the specimen charges, it is better to use a smaller aperture to reduce the total beam current on the specimen.

6.2.1 HRTEM image stigmation

Because of the high coherence of the FEG, it can be difficult to judge where minimum contrast focus is when considerable astigmatism is present. The easiest method is to go to higher magnification (200 000x or higher) and focus the beam somewhat (to something like the 4 cm circle on the screen). By viewing the image through the binoculars the high spatial frequencies normally visible in the image on the TV monitor are much reduced and the image looks quite like that obtained on a LaB_6 instrument. Now it is much easier to focus to minimum contrast and correct the astigmatism.

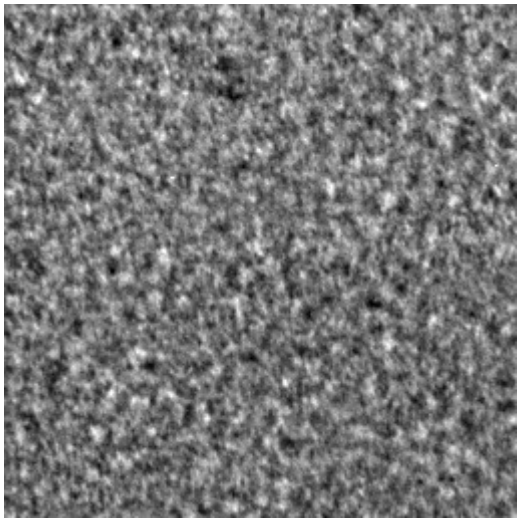
The correct procedure for correcting the astigmatism is as follows. The criterion for astigmatism correction is always attaining minimum contrast on an amorphous area of the specimen (notwithstanding the claims of some microscopists, it is impossible to correct astigmatism on crystalline areas). Usually, the thinner the amorphous area the better. Beware that in some cases the amorphous rims of specimens may have structure. In that case minimum-contrast focusing still works, but trying to minimize the structure visible will not.

First try to focus as close to minimum contrast as possible. In this condition, the Fresnel fringing at the specimen edge is minimized and no preferential direction should be visible in the image of amorphous material. If astigmatism is strong, find one line focus (image streaked in one direction), press Reset defocus, find the line focus in the other direction and read off the amount of defocus. (Note: if the line foci do not change direction, it is not astigmatism; instead you may have vibrations or specimen charging.) Set the defocus to half this value (correct focus lies halfway between the two astigmatic line foci). Now activate the stigmator and first change MF-X. If considerable astigmatism appears, then first try to make the image sharper. If no more improvement is seen when changing MF-X, then do the same thing with MF-Y. Repeat this procedure (set minimum contrast focus, MF-X, MF-Y) if necessary. When astigmatism is close to zero, the aim is to make the image contrast disappear (getting true minimum contrast focus). Repeat focusing, changing MF-X and MF-Y until this is achieved. Note that weak astigmatism close to minimum-contrast focus is characterized by a tweed-like structure (two weak preferential directions visible in the image). It is often helpful to switch (once or a few times) to another stigmator channel during astigmatism correction and then copy the contents of the other (good) channel into the current one. It provides an easy fall-back position if for some reason things are getting worse. It is also possible to correct astigmatism for the two channels independently. If a comparison between the two channels (simply switch over while observing the image or read off the values) shows a lot of difference, at least one of the settings is off.

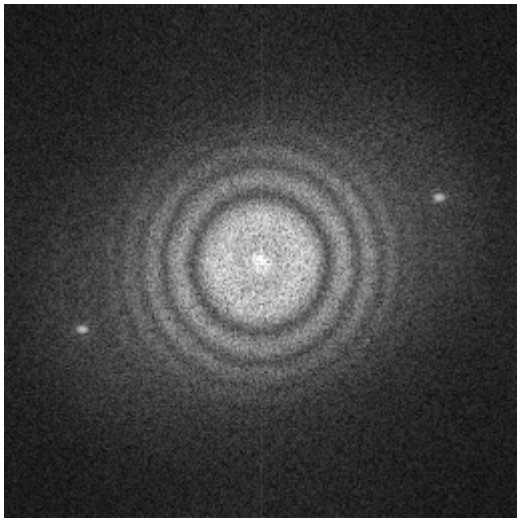
6.2.1.1 Focusing and astigmatism correction using a CCD camera

With CCD cameras generally available nowadays, the most useful method for correcting astigmatism and focusing is by using the on-line FFT functionality available. The FFT (Fast Fourier Transform) effectively is a representation of the distribution of image spacings. Any distance visible in the image

becomes a pixel in the FFT such that the direction of the distance gives the direction in the FFT and the size of the distance becomes the inverse (so a large distance ends up in the center of the FFT while a small one ends up at the edges).



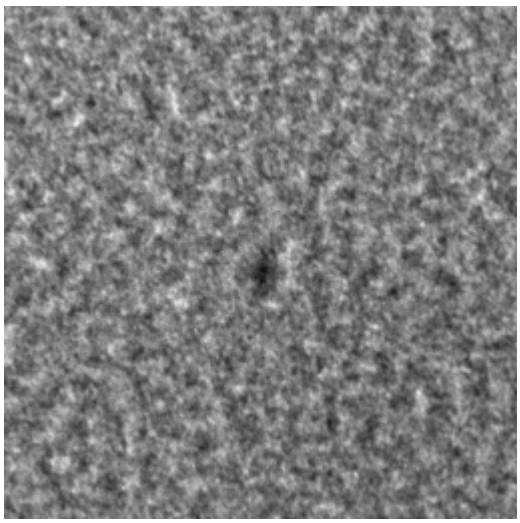
High-magnification image of carbon foil.

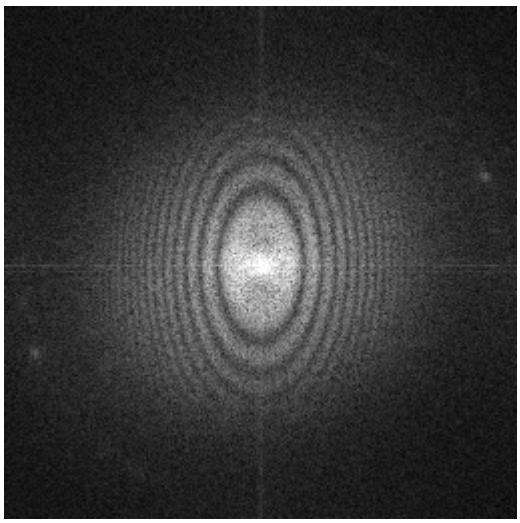


FFT of the image.

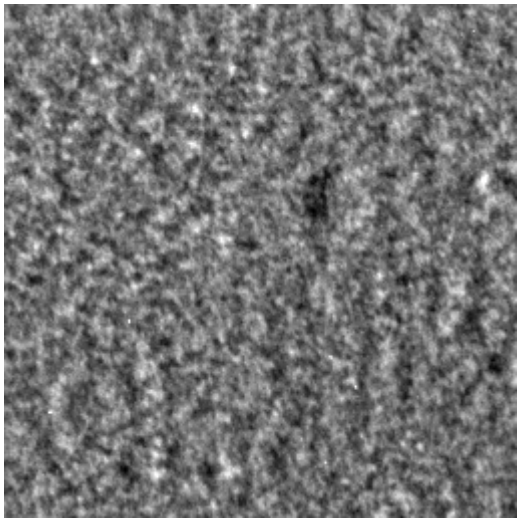
Since electron microscopes suffers from the spherical aberration of the objective lens, the FFT of the image is affected by the Contrast Transfer Function (CTF). When an amorphous specimen is used, as e.g. a carbon foil, with many different spacings in all directions, we observe a representation of the Contrast Transfer Function itself. When there is no astigmatism the FFT will therefore shows concentric rings that are circular, but with astigmatism present the rings become elliptical or of more complex shape (astigmatism is a difference in focus in different directions so the Contrast Transfer Function differs). Below is a description of what can be seen during astigmatism correction and focusing.

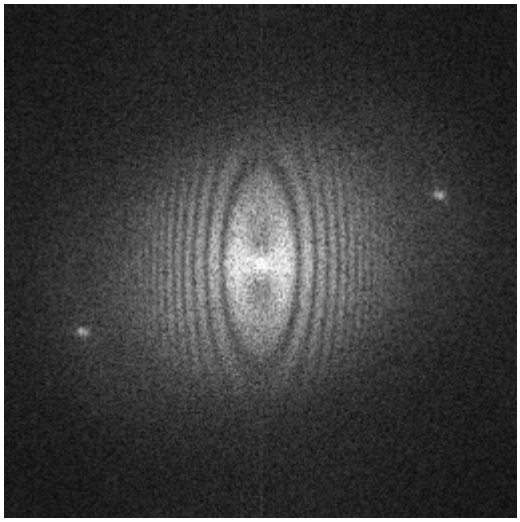
For more information about astigmatism in electron lenses, reference is made to the many text books on electron optics, for example:
Transmission Electron Microscopy (2ed.), L. Reimer (1989), Springer-Verlag, Berlin.
Experimental High Resolution Electron Microscopy (2ed.), J. C. H. Spence, Oxford University Press, New York, Oxford.



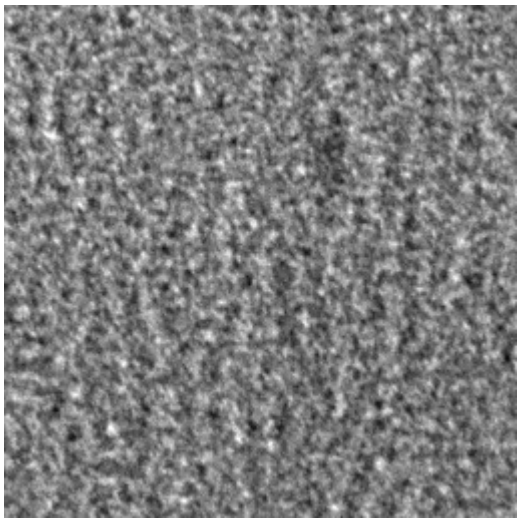


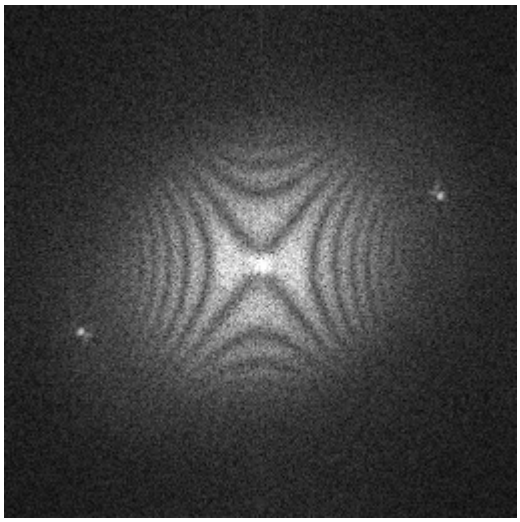
Above: HR-TEM image and FFT several hundreds of nanometers underfocus and with strong astigmatism. The rings in the FFT are very elliptical. As a start (with this amount of astigmatism) use the objective stigmator to make the rings more circular.



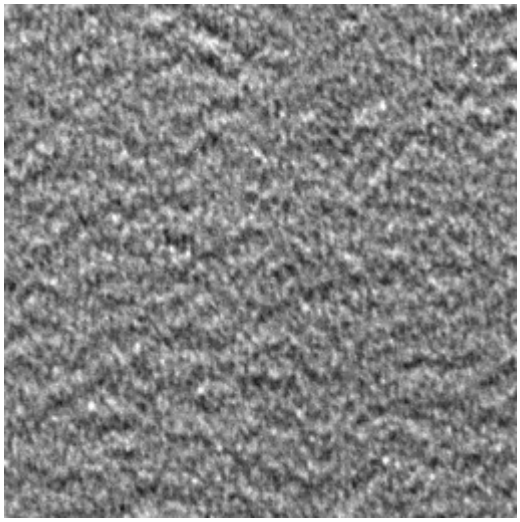


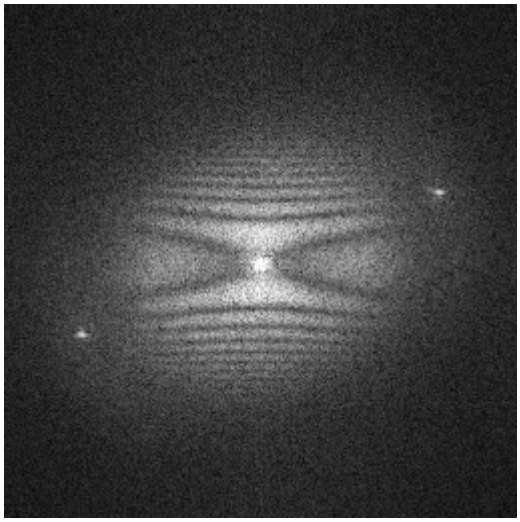
Closer to focus the ellipticity of the rings becomes more pronounced (because the focus difference caused by the astigmatism is a larger proportion of the total focus. The two spots on either side come from a gold-palladium particle.



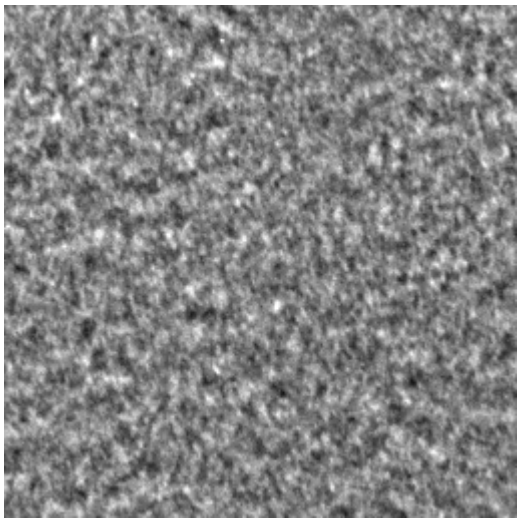


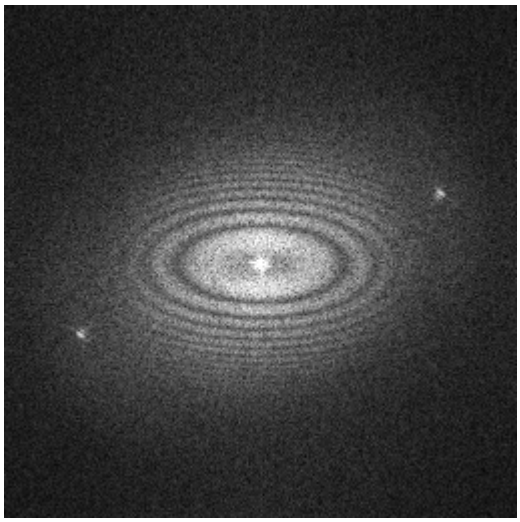
Even closer to focus there are no rings any longer but a cross-like pattern appears. That is because in one direction we have underfocus and in the perpendicular direction we have overfocus. With a pattern like this it is easier to go further underfocus to make the rings more circular than try to stigmate at this particular focus. The image shows a tweed-like pattern.





Overfocus the elliptical rings, though still complex, appear again.





Further overfocus the rings are elliptical.

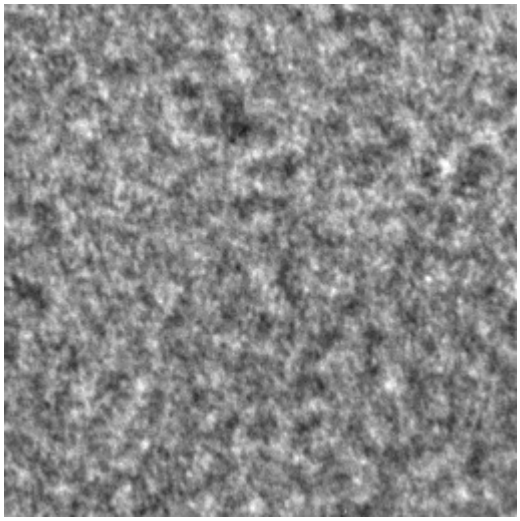
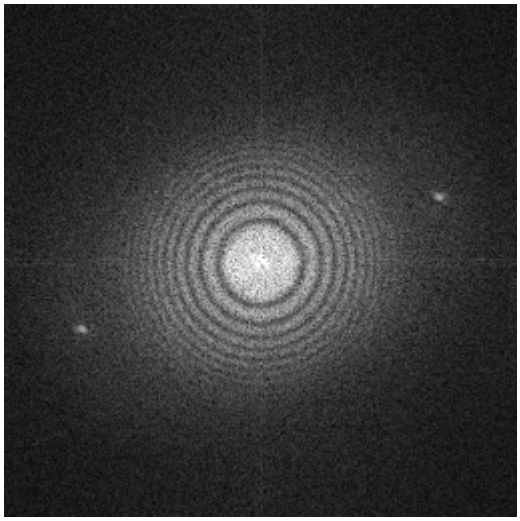
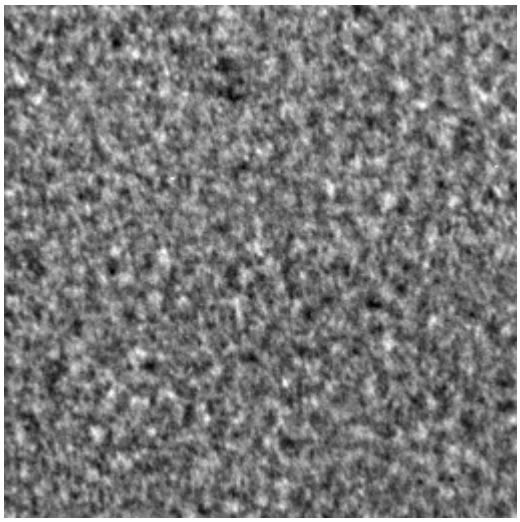
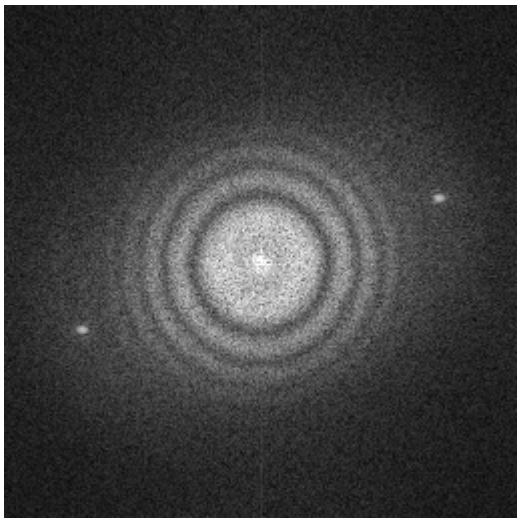
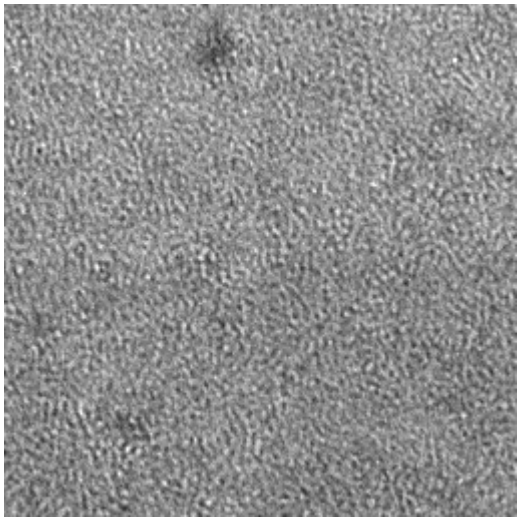
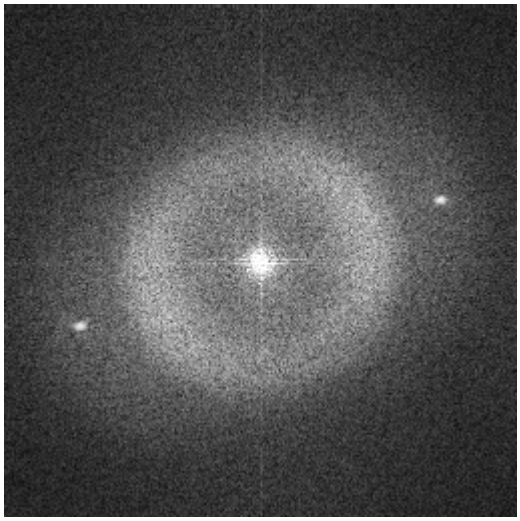



Image and FFT far underfocus but with very little astigmatism. The rings are almost perfectly round.

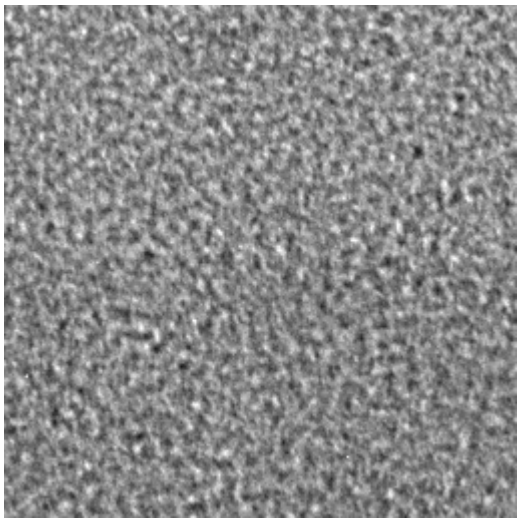



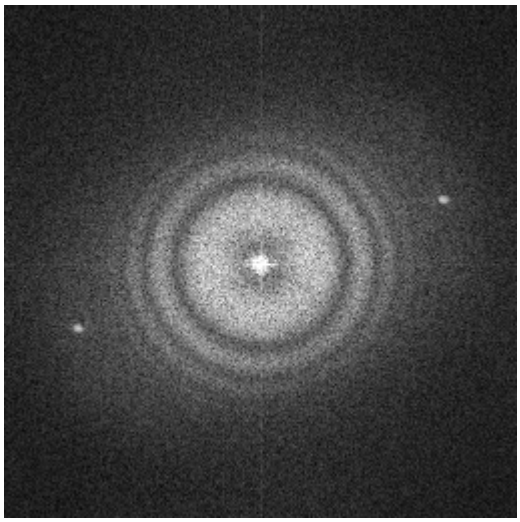
Closer to focus. The image appears "finer-grained" than further underfocus.



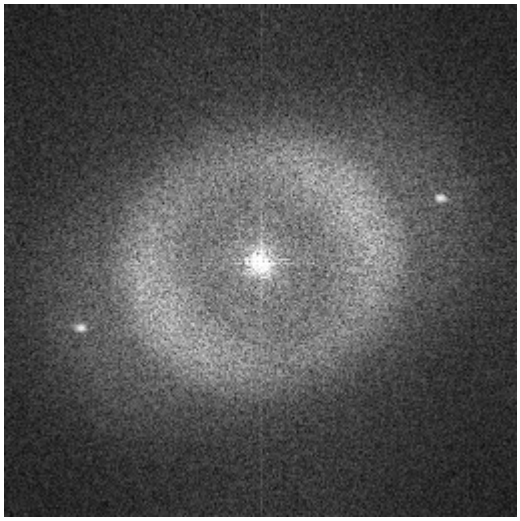


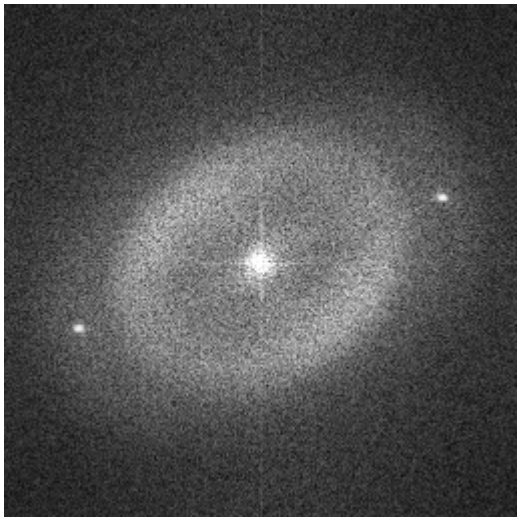
Minimum contrast focus shows very little in the image. At this focus setting the image and FFT are very sensitive to astigmatism so this is the best focus to do the final corrections.

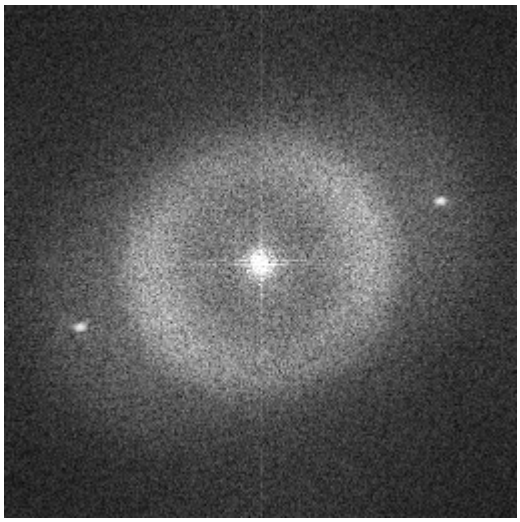




Overfocus the rings reappear. There is a slight cut-off of the FFT in the vertical direction (the outer, fuzzy ring doesn't extend as far) which has nothing to do with the focus or astigmatism but instead is the result of either mechanical instability or drift.







Three FFTs taken at minimum contrast where small changes in astigmatism show up very clearly. Look for circularity in the inner (dark) ring and the ring around it, but ignore the cut-off.

6.2.2 Coma-free alignment

Alignment of the electron-beam incidence angle is very important for the results of high-resolution imaging. For this purpose the coma-free alignment has been incorporated into the microscope.

Note: Coma-free alignment is an alignment method for the objective lens. As with the rotation center, the result is alignment of the incident beam with the optical axis - a beam tilt. **Both coma-free alignment and rotation center affect exactly the same alignment parameter!** The only difference between the two methods is the criterion used for the alignment (change in focus while wobbling the beam slowly; modulating the objective-lens current). There is therefore no point in repeating rotation center - coma-free alignment and then rotation center again (the same setting gets adjusted back to what it was).

As with stigmation, coma-free alignment is easiest done with the beam somewhat focused and using the small focusing screen. Under these conditions, it is essential, however, that the coma-free pivot points are set as near to perfect as possible, otherwise the convergence of the beam may result in actual misalignment if the center of the beam for the two tilts is not (nearly) coincident. Because the pivot points are sensitive to the objective-lens current, it is by far the easiest to make sure that the preset for the objective lens (Eucentric focus button) has been set correctly in the HM Image alignment procedure and

then bring the specimen into focus with the Z height after pressing Eucentric focus. This procedure minimizes having to adjust the coma-free pivot points continuously.

If the coma-free pivot points are far off, first align them at a lower magnification. Thereafter go to a magnification around 500 000x (or the magnification to be used for high-resolution imaging) and adjust the pivot points as accurately as possible.

Always check the centering of the C2 aperture before the coma-free alignment.

If the coma-free alignment is far off, coma-free alignment may be difficult. It is then easier to start with the rotation center and thereafter the coma-free alignment. Once the coma-free alignment has been done, check the astigmatism correction (as beam tilt looks like astigmatism).

The coma-free alignment method aim is to minimize focus differences for two tilted incident-beam directions. If the focus is the same for both directions, the beam is incident along the true optical axis. Use amorphous areas as for astigmatism correction. Do not look for details in the images. Although the focus should be the same, the beam still passes through the specimen in a different way and patterns seen in the image for one tilt direction may not be visible in the other. Instead, "stare" at the images and get an overall impression of the focus.

6.2.3 Focus

In microscopy there are three important focus values: Gaussian (0), Scherzer ($c * [Cs \lambda]^{0.5}$) where c is a constant – 1.2 or 1.0 depending on the definition used) and minimum contrast focus. The latter has no fixed definition but in practice is the only one that can be recognized on the microscope (although different operators tend to have slightly different opinions on what is minimum contrast, especially on a FEG instrument). Minimum contrast is roughly equal to 0.4 x Scherzer.

Focusing can be difficult on a FEG instrument, especially on crystalline areas as the same type of image tends to repeat itself without any major changes over a large range of focus. Some tips:

- The amorphous rim is a good reference point (but remember that for wedge-shaped or tilted specimens the beam exit - lower - surface of the specimen away from the amorphous edge may actually be at a different height and thus different defocus). Not only should the amorphous image at the rim help in focusing; if there are lattice fringes far out into the vacuum you are away from useful focus settings like minimum contrast or Scherzer. The lattice fringes go out into the vacuum due to a process called delocalization (see Otten and Coene and references therein for further explanation).
- On the FEG strong Fresnel fringing effects are seen at interfaces. Close to the Scherzer the apparent width of interfaces or precipitates due to the Fresnel fringes is small, while further away from focus the Fresnel fringes move out in both directions.

You should be aware of delocalization of lattice fringes in images (especially when looking at interfaces which could be either crystalline or glassy). In general, very small lattice spacings have stronger displacements, so if delocalization is troublesome, it may be worthwhile to limit the visibility of the small, strongly delocalized lattice fringes by cutting them off with the objective aperture.

6.2.4 Further reading for high-resolution imaging

M.T. Otten and W.M.J. Coene (1993) High-resolution imaging on a field emission TEM. Ultramicroscopy 48, 77-91.

6.3 EDX microanalysis

Mode	Nanoprobe
C1 aperture	30 μm / 2 mm (beam limiting / not beam limiting)
C2 aperture	150 μm / 50 /70 μm (not beam limiting / beam limiting)
Extractor	V-optimal
Gun Lens	5 - 8 (depending on current required)
Spot size:	5 (depending on current required)

The beam current should be adjusted to suit the specimen and EDX count rate. If the specimen is beam-sensitive, it is usually better to use a relatively low beam current (or defocus the beam somewhat) so that the operator has more time to control the EDX analyzer without too much damage to the specimen in the meantime. If the specimen is thick, reduce the beam current so the EDX count rate remains below the optimum for the particular detector in use (typically ~10000 cps). Higher count rates mean more X-ray counts being rejected and thus fewer stored while any beam damage will be related to the electron dose - not the number of X-rays detected.

The operating conditions listed above give the overall best results with regard to in-hole count and system background. This does not mean that the microprobe mode cannot be used for EDX analysis or that the C2 aperture cannot be used as the beam-defining aperture.

6.3.1 Microprobe versus nanoprobe

The electron optics of the column with regard to the two modes result in a large field of view in the microprobe mode (with the beam able to illuminate a wide area) whereas in the nanoprobe mode the beam cannot be defocused further than several micrometers. Stray electrons (scattered at the edge of apertures) can only occur within the maximum area that can be illuminated by the beam. In nanoprobe any stray electrons are therefore confined to a few micrometers around the electron beam itself. Especially in ion-milled or jet-polished specimens, the stray electrons strike only thin parts of the specimen and thus do not have a significant effect on the EDX spectrum (which in principle should contain only information from the area within the beam itself). In the microprobe mode, the stray electrons can strike the specimen over a much larger area, including thick areas where a single incident electron can generate up to tens of X rays. The nanoprobe mode is therefore better suited for EDX analysis.

6.3.2 C1 aperture versus C2 aperture

Some electrons from the beam are scattered at the edge of the aperture. These stray electrons do not follow the path of the beam anymore but occur outside it. They can thus strike the specimen in areas outside the area intended for analysis. In the case of using the C1 aperture as the beam-defining aperture, there are two effects that reduce the number of stray electrons relative to the situation where the C2 aperture is used to define the beam:

- Stray electrons scattered at the edge of the C1 aperture are blocked from going further down the column by the C2 aperture (which acts as a so-called spray aperture).
- The C1 aperture occurs in higher vacuum than the C2 aperture and is often cleaner than the latter (and the amount of stray electrons depends on the cleanliness of the edge of the aperture).

The EDX spectrum may therefore be cleaner when the C1 aperture is used than when the C2 aperture defines the beam. The disadvantage of using the C1 aperture is the relative large (~0.8 nm FWHM) smallest spot size attainable.
The choice of C1 or C2 as beam-limiting aperture depends on practical considerations. If the in-hole count of the microscope is negligible, use the C2 aperture. If not, switch to the C1 aperture.

6.4 Diffraction

On the FEG, diffraction can be done in a number of ways. Selected-area diffraction is of course still possible, but is subject to the normal Cs-induced aberrations (beams of higher scattering angles actually may come from specimen areas outside the SA aperture). Also smaller SA apertures cut off visibility of the diffraction pattern away from the transmitted beam. Therefore microdiffraction or convergent beam diffraction is more reliable (the two terms are used here interchangeably, although some people use convergent beam diffraction for patterns with structure inside the diffraction discs and microdiffraction for other cases). In essence though, these techniques are electron-optically the same, relying on a focused (or a just slightly defocused) electron beam with a small size.

The two essential microdiffraction modes are microprobe mode (named this way because traditionally the beams are larger than in nanoprobe mode – though even in microprobe mode the FEG instruments can produce nanometer-size beams) with a generally somewhat larger spot size and small convergence angle, and the nanoprobe and STEM modes with smaller spot sizes and larger convergence angles. Thus the latter are used when the small spot size is critical and larger discs in the diffraction pattern are no problem (or even desirable in the case of convergent beam diffraction where structure inside the discs is sought or for coherent convergent-beam diffraction). For large-unit-cell materials or superstructures, high convergence angles give disc overlap and masking of weak diffracted beams (the same intensity is smeared out over a disc instead of being compressed into a spot) so the microprobe mode is better.

6.4.1 Selected-area diffraction

Mode	Microprobe
C1 aperture	2 mm
C2 aperture	any
Extractor	V-optimal
Gun Lens	3
Spot number	any

Insert the selected-area aperture and spread the beam (with the Intensity knob) until the pattern contains spots.

6.4.2 Small-angle microdiffraction

Mode	Microprobe
C1 aperture	2 mm
C2 aperture	smallest
Extractor	V-optimal
Gun Lens	5 - 8
Spot number	7

In this mode, spot sizes down to about 1-2 nm can be used while in the diffraction pattern the minimum convergence angle is obtained.

6.4.3 Smallest-spot microdiffraction

Mode	Nanoprobe
C1 aperture	2 mm
C2 aperture	smallest
Extractor	V-optimal
Gun Lens	5 - 8
Spot number	7

In this mode the smallest spots are obtained but at the expense of angular resolution in the diffraction pattern (larger discs). Spot sizes obtainable can be down to about 0.2 nm or less (depending somewhat on the objective-lens type).

6.4.4 General diffraction information

There are two limiting cases to beam convergence, one with parallel illumination on the specimen and a cross-over in the back-focal plane (spot diffraction pattern), the other with the cross-over on the specimen (that is, a focused beam) and thus a disc diffraction pattern. In the latter case no amount of defocusing of the diffraction pattern will produce a spot pattern (the spot - that is, the focused beam - occurs at the image plane!) and the correct diffraction focus should be set by focusing the edge of the diffraction disks. In intermediate cases of convergence it usually is possible to obtain a spot pattern but only at an incorrect diffraction focus! If you still wish to use such settings, remember that the camera length may well be off the value stated by the microscope or calibrated by yourself, and that the pattern may display considerable distortions.

If the C2 aperture is not completely clean, some electrons may be scattered outside the focused beam and fall on other specimen areas. This may give rise to unexpected diffracted beams. However, this effect is always recognizable from the fact that these scattered beams appear hollow (only the outside ring of the diffracted disc is visible).

Switching from imaging to diffraction may cause the beam to shift slightly on the specimen. In STEM, the microscope is continuously in diffraction mode so no beam shift will occur. Because of the beam shift for diffraction work in TEM or nanoprobe mode, it is best to leave the beam somewhat defocused on the area of interest while in image mode and thus obtain in diffraction a shadow image (an image that is visible inside the transmitted and diffracted beams; note that shadow-image contrast reverses between beam under- and overfocus). Often the shadow image shows the details sought for very effectively, in many cases even better than in the image anyway. Once the area of interest has been identified in the shadow image, center the area (with beam shift or goniometer) inside the (transmitted- or diffracted-beam) disc and then focus the beam. Focus can be identified easily because the apparent magnification of the shadow image increases and then decreases again once the beam has gone through focus. At beam focus, the magnification in the shadow image is infinite. It is even possible to correct condenser astigmatism in the shadow image since a streaky shadow image will be seen in an astigmatic beam close to focus. The streakiness changes direction by 90° as the beam goes through focus.
The shadow image can also be used to obtain multiple dark-field pattern (images for many diffracting conditions at the same time) that can be used e.g. for rapid Burgers vector determination. Be aware, however, that the image in one diffracted-beam disc may still be influenced by scattering from or to other (especially strong) diffracted beams.

6.4.5 Convergence angles

Convergence angles are generally displayed by the microscope. You can also measure the angle in diffraction patterns. Record a diffraction pattern with the focused beam, under the conditions required

(C1/C2 aperture, TEM mode, nanoprobe or STEM mode). The convergence half-angle is measured from the radius R of the disc of the central beam divided by the camera length (both in the same units, e.g. mm's), giving the angle in rad.
(The formula for this measurement is derived from Bragg's Law $2\sin\theta = \lambda / d$ and the formula used for measuring d spacings in diffraction patterns $D \cdot d = L \cdot \lambda$, where D is the distance in the diffraction pattern and L the camera length; $L \cdot \lambda$ is also called the camera constant).

6.5 STEM

Mode	Nanoprobe or STEM
C1 aperture	2 mm
C2 aperture	50 um (Titan), 70 um (Tecnai/Talos)
Extractor	V-optimal
Gun Lens	4-6
Spot number	8

STEM can be somewhat hard to set up with regard to centering of the C2 aperture and gun alignment, so it is easier to do this setup in nanoprobe mode (which electron-optically is nearly identical) and afterwards switch to STEM.

When going into STEM, a choice must be made for the camera length to be used. For most materials a camera length between 200 and 300 mm is suitable for standard bright-field/dark-field imaging while a camera length around 100 mm is suitable for high-angle annular dark-field imaging. Especially for work at lower magnifications, some camera lengths are less suitable because of strong Cs-induced (in this case Cs of the diffraction lens) movement of the diffraction pattern. Whether or not a camera length is suitable is easily seen by observing the diffraction pattern while the beam is scanning and the STEM image magnification is at a low value (10000x). If the diffraction pattern remains nearly stationary, the camera length is a good one. If it moves, giving a pattern looking like a bird with spread wings, then it is less suitable.

The beam shift pivot points are probably best set in the HM-STEM procedure and should not require modification by direct alignment. The same criteria for the rotation center can be used as for nanoprobe. If this proves difficult, the standard procedure in diffraction mode can be used (the same as in the HM-STEM procedure). Center the beam on a detail in the shadow image as it appears while the beam goes through focus and make sure that that detail remains in the center of the shadow image. After rotation centering, center the diffraction pattern.

If stigmation is difficult, it is also possible to switch to TEM image mode by pressing the Diffraction button while in STEM, stop the beam and stigmate the beam this way.

Remember to scan reasonably slowly while focusing and stigmating on STEM images, otherwise the beam stability may be too poor and the image too noisy so focusing and stigmation will be impossible.

6.6 Electron energy-loss spectroscopy

Mode	Nanoprobe or STEM
C1 aperture	2 mm (possibly smaller to cut off side-lobes)
C2 aperture	50 um
Extractor	V-optimal (possibly lower to obtain a higher energy resolution for fine analysis)
Gun Lens	4-6 (depending on current required)
Spot number	5 (3-8) (depending on current required)

Electron energy-loss spectroscopy can have two major goals: elemental microanalysis and investigations of electronic structure in the spectrum. In the latter case, the resolution in the spectrum is important, in the former it usually is not. These two applications are therefore considered separately.

6.6.1 High energy resolution

The energy resolution of the microscope as seen on the Imaging Filter/Spectrometer is determined by a large number of factors: the stability of the high-tension chain (tank and accelerator), the Boersch effect (electron-electron interactions) on the emitter and in the first cross-over, the electron-optical conditions, the stability and resolution of the Imaging Filter/Spectrometer and interferences from outside (stray fields). Under the best conditions it is possible to get down to about 0.8 eV or less with the FEG instruments, provided that all these elements are perfect.
Separate conditions apply to the microscopes equipped with a monochromator. See the monochromator documentation.

Tank and accelerator. The high-tension chain has a number of performance characteristics, called ripple, noise and drift. Ripple is a high-frequency interference, while noise concerns random changes in high tension. The effects of ripple are always such that they are faster than the read-out of the Imaging Filter/Spectrometer so the effect is more or less constant and independent of the read-out time. Drift concerns the slow change in high tension over a period of at least several minutes.

Boersch effect. The Boersch effect is the result of Coulomb interactions between electrons in the beam (an electron in front will get accelerated by the push from an electron behind and thus get a higher energy, while the electron behind is decelerated). This effect is reduced by making sure that the extraction voltage (or rather emission current) is not too high: if too many electrons leave the surface of the emitter together, then they start interfering with each other, pushing up the energy spread. In general, the Boersch effect is not a significant factor for emission current <80 μA.

Electron-optical conditions. The electron optics play a role in achieving the minimum energy spread because of the following. On the Image Filter/Spectrometer CCD, there is not only a spectrum, but the spectrum itself consists of a diffraction pattern or an image that has been stretched out according to the energy distribution. Whether it is an image or diffraction pattern depends on what is at the Imaging Filter/Spectrometer entrance aperture. If the microscope is in diffraction mode, the Imaging Filter/Spectrometer 'sees' an image and vice versa (hence the terms diffraction-coupling when in image mode and image-coupling when in diffraction mode). If the image or diffraction pattern at the entrance aperture is small, the diffraction pattern or image at the Imaging Filter/Spectrometer CCD is the inverse, that is, large, and energy resolution will not be good. Thus it is in principle impossible to obtain at the same time high energy resolution and high currents. The following conditions have been found to be ideal for obtaining high energy resolution: nanoprobe diffraction mode, camera length not too small (>=150 mm or so), beam not completely focused and diffraction pattern focused to a spot pattern, Imaging Filter/Spectrometer entrance aperture 1 mm.

Imaging Filter/Spectrometer. Make sure the Imaging Filter/Spectrometer control unit is not too close to the microscope (at least 2 meters away), especially the power supply which is either inside the main unit or comes as a separate (flat) box. Make sure the power is supplied from the microscope. The stability will have been tested during installation and should remain alright. The scintillator of the Imaging Filter/Spectrometer causes a certain amount of beam spreading and thus loss of energy resolution. AC compensation on may help to reduce ripple effects although on good instruments the improvement shouldn't be more than 0.1 eV.

Outside interferences. Stray fields can have considerable effects on the energy resolution. One source of such stray fields consists of TV monitors. The frequency of these stray field often may be slightly off relative to the Imaging Filter/Spectrometer and microscope 50 or 60 Hz frequency. The result is a slowly beating interference that is often visible if the Imaging Filter/Spectrometer is used with smallest read-out time. If the zero-loss peak goes up and down somewhat with a periodicity of a few seconds, it is likely that there is interference.

6.6.2 Microanalysis

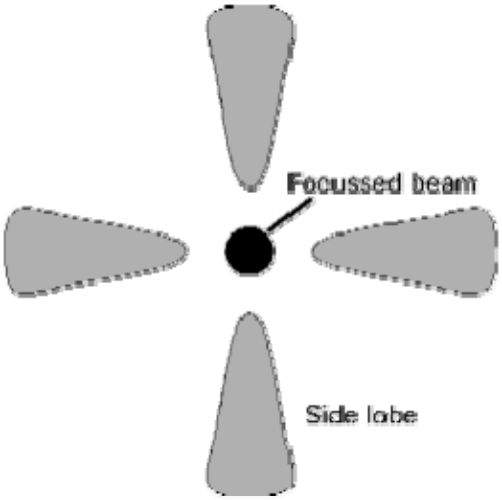
For EELS microanalysis the conditions can be chosen as one wishes although it should be realised that for high reproducibility of the analyses it is advisable to stick to the same conditions, especially with regard to the β acceptance angle for the spectrometer.

One point should be mentioned here regarding the FEG. Due to the emission process on the emitter, there is not only electron emission from the front surface but also some emission (typically below 1% of the total) from four crystal faces on the emitter tip at an angle to the front surface (see below). Some of the electrons emitted from these crystal faces scatter back through the extraction anode and gun lens after having lost some energy. In the image these electrons are focused outside the main beam due to the chromatic aberration of the condenser system, forming a cross that can be seen at low magnifications. Under some conditions these energy-loss electrons can contribute to the energy-loss spectra, although they carry of course no specimen information. The contribution is pronounced at very low camera lengths and large Imaging Filter/Spectrometer entrance apertures. It is therefore best to avoid such extreme conditions. Usually most of this contribution can be removed by inserting a selected area aperture around the small beam used for microanalysis, but don't use the smallest aperture, otherwise energy-loss electrons from the specimen are also removed. The gun contribution can be easily detected by collecting a spectrum in a hole. The gun contribution can also be removed from the specimen spectrum if necessary by subtracting the hole spectrum.

6.6.3 Side-lobe emission and EELS spectroscopy or diffraction

Although the Schottky FEG has many excellent characteristics that make it highly suitable as an emitter for TEM applications, there is one feature that is somewhat problematic: the side-lobe emission (note: this is generic to all Schottky emitters and is due to the relatively large size of the crystal faces as compared to cold FEGs where the tip is much sharper and thus these crystal faces smaller). Side-lobe emission occurs when crystal faces other than the (100) front face of the emitter also emit electrons. These faces are tilted relative to the (100) face and the electrons do not enter the beam normally but fly off at an angle. These electrons then hit the extractor anode plate and are either backscattered themselves or cause emission of secondary electrons. Some of these move back on trajectories that, depending on their energy, may cause them to pass through the central hole in the extraction anode and then get accelerated and come down the microscope. The problem is caused by the fact that the energy of these electrons is not the same as that of the primary electrons.

Due to the chromatic aberrations in the electron optics of the column, these electrons are focused differently from the primary-beam electrons and are spread around the central beam. Because emission occurs from only four crystal faces, the pattern that emerges is that of four lobes of electrons with increasing energy loss away from the central beam. The maximum value of the energy losses depends on the extraction voltage and gun lens setting and is approximately given by $(V_{\text{extraction voltage}} - V_{\text{gun lens}})/2$.



In most applications of the TEM, the side lobes have no measurable effect, but in two there are some troublesome effects. Both occur in diffraction, where the side lobes are also visible around the central beam. In diffraction applications where the specimens have very large spacings (as in biology), the lobes may hamper visibility of diffraction spots close to the central beam. In EELS the side lobes also enter the spectrometer at small camera lengths and/or large EELS entrance apertures and create a background of energy-loss electrons that have nothing to do with the specimen (and are thus undesirable). In the latter case it may be possible to subtract the 'in-hole' EELS contribution by simply recording a spectrum under the same conditions in a hole and subtracting the resulting spectrum from the real data.

On Titan microscopes with a monochromator the side lobe emission is removed by the entrance aperture of the monochromator.