Stellingen behorende bij het proefschrift:

Ontwerp van een hoogspannings post-column afbeeldend energie filter

A.J. Gubbens

1. Het gebruik van een energie filter in de TEM zal net zo gebruikelijk worden als dat van het objectief diafragma.

2. Hoewel veelvuldig toegepast, is de inspectie van een energieverlies spectrum voorafgaand aan het opnemen van elementaire distributiebeelden niet altijd nuttig.

3. In tegenstelling tot wat de literatuur* lijkt te beweren is de transmissiviteit slechts een van de vele prestatie criteria voor afbeeldende energie filters en niet noodzakelijk het belangrijkste criterium. In het geval van het afbeelden van chemisch contrast, bijvoorbeeld, zal de non-isochromatie van het filter bepalen of het experiment wel of niet haalbaar is, onafhankelijk van de waarde van de transmissiviteit.


4. De vervanging van de fotografische film in de elektronenmicroscopie door CCD camera's wordt voornamelijk belemmerd door de afwezigheid van commercieel verkrijgbare CCD's met groot formaat en grote pixels. De ontwikkeling van deze CCD's zal gedreven moeten worden door vakgebieden als medical imaging, vakgebieden met genoeg geld om de enorme ontwikkelingskosten te kunnen betalen.

5. Met de opkomst van CCD's met groot formaat en grote pixels, kunnen microscopen ontworpen worden zonder de gebruikelijke fluorescentieschermen. Net zoals andere moderne instrumentatie, kan zo'n microscoop via een computer bediend worden en zijn de ergonomische aspecten van het ontwerp minder belangrijk. Dit staat dan het ontwerp toe van nieuwe hoog prestatie microscopen die compacter zijn en economischer zijn te vervaardigen.


7. Volgens The Economists Big Mac Index zal het besluit van McDonalds om in Amerika Big Macs te gaan verkopen voor $55c een verdere stijging van de dollar ten opzichte van andere valuta tewega brengen. Dit zou wel eens een verslechtering van de Amerikaanse economie kunnen veroorzaken en een verdere afname in de verkopen van de Big Mac.
Propositions added to the thesis:

The design of a high voltage post-column imaging energy filter

A.J. Gubbens

1. The use of an energy filter in the TEM will become as commonplace as that of the objective aperture.

2. Although generally practiced, the examination of an energy-loss spectrum prior to the recording of an elemental distribution map is not necessarily useful.

3. Unlike the literature* makes one believe, transmissivity is only one of many performance parameters that characterize imaging energy filters, and is not necessarily the critical parameter for an experiment.
   In the case of chemical imaging, for instance, the filter's non-isochromaticity will actually determine whether or not the experiment is feasible, regardless of the transmissivity.

4. The replacement of photographic film in mainstream electron microscopy by CCD cameras is mainly limited by the lack of commercially available large format, large pixel CCD’s. Development in this area will have to be driven by fields as medical imaging, a field with pockets deep enough to afford and drive the massive development efforts.

5. With the appearance of large format, large pixel CCD’S, microscopes can be built without the conventional viewing and camera chamber and without photographic film. Similar to other modern instrumentation, such a microscope can be fully operated from a computer making ergonomics a much less important design issue. This will allow new high performance yet compact and economical layouts to be considered.

6. The preparation of material science specimens for electron microscopy has much in common with the manufacturing of integrated semiconductor circuits. Good examples are chemical mechanical planarization (CMP) and plasma etching. TEM should benefit more from the tremendous efforts in this multi-billion dollar industry.

7. According to The Economist's Big Mac Index, the decision by McDonalds to start offering $5.50 Big Macs in the USA will cause a further strengthening of the dollar with respect to other currencies. This may actually cause a slow down in the American economy resulting in a further decline in Big Mac sales.

*****
Design of a High-Voltage
Post-Column Imaging Energy Filter
Ontwerp van een hoogspannings post-column afbeeldend energie filter

PROEFSCHRIFT

ter verkrijging van de graad van doctor aan de Technische Universiteit Delft, op gezag van Rector Magnificus Prof.dr.ir. J. Blauwendraad, in het openbaar te verdedigen ten overstaan van een commissie, door het College van Dekanen aangewezen, op maandag 24 maart 1997 te 13:30 uur door

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Introduction

Transmission electron microscopy has since long been a powerful tool in the study of materials and biological structures at high spatial resolution. As a result of the interactions between the beam electrons and the specimen atoms, many beam electrons lose some amount of energy. Although the spectrum of energy-losses contains a wealth of elemental and chemical information, the contribution of the energy-loss electrons to the image will be out of focus due to the chromatic aberration of the objective lens and will therefore deteriorate the quality of the image. Especially for thick specimens, where the fraction of energy-loss electrons can be large, the deterioration of the image can be significant.

To overcome this problem, imaging energy filters have been developed that allow imaging with only a narrow range of energies. They do so by transforming the unfiltered image into an energy-loss spectrum in which an energy-selecting slit selects a range of electron energies that are then transformed back into an energy-filtered image.

Not only does energy-filtering allow the image contrast to be improved, it also enables imaging using purely energy-loss electrons and allows powerful, new contrast techniques to be used. As the energy-loss spectrum contains elemental and chemical information, imaging using specific energy-loss ranges allows images with elemental and/or chemical contrast to be obtained. Although a lot of this information can also be probed with other analytical techniques such as energy dispersive x-ray spectroscopy (EDXS), electron energy-loss spectroscopy (EELS), or Auger spectroscopy, these techniques yield spectra of the area under the electron probe, and images can only be obtained by scanning the probe across the specimen and recording spectra at each position. This severely limits the size of the specimen area that can be imaged in a reasonable amount of time.

Since the introduction of the first in-column filter by Castaing and Henry in the early sixties, various imaging energy filters have been proposed and built. Until about ten years ago the performance of the in-column filters was limited as a result of large electron optical aberrations. The so-called post-column imaging filters first appeared in the early eighties. They were based on early electron energy-loss spectrometers and also had limited electron optical performance.

This thesis covers the design of a high voltage post-column imaging energy filter for use with the JEOL ARM1000 and ARM1250 high voltage transmission electron microscopes. I performed this work while employed at Gatan Inc. (California, USA) under the leadership of Dr. Ondrej Krivanek. Prior to this project I worked on the design of a High Voltage Parallel Electron Energy Loss Spectrometer (HV PEELS) and the design of the Gatan Imaging Filter (GIF), the first high performance post-column imaging energy filter, the GIF, which was commercially introduced at the MSA meeting in Boston in August 1992. Although this thesis covers in particular the design of the high voltage post-column imaging filter, many of the design principles and much of the experience were developed in these earlier projects.
1 Energy-Filtered HV TEM

1.1 Transmission electron microscopy

Transmission electron microscopy, commonly referred to as TEM, is widely used for the study of materials and biological structures at intermediate to high magnifications (about 1,000 to 2,000,000 times). TEM offers a vastly superior spatial resolution over conventional light microscopy, where the resolution is limited by diffraction to less than a μm. The very short wavelength of high energy electrons (order of pm versus μm for light) allows much higher spatial resolutions to be obtained. Unlike in the light microscope, in the TEM the (point) resolution is limited by the wave length of the electron as well as the spherical aberration of the magnetic electron lenses that are used(1). This currently limits the directly attainable spatial resolution (point resolution) to about 0.1 nm(2).

![Diagram of a modern transmission electron microscope](image)

Figure 1.1 Schematic overview of a modern transmission electron microscope.

The transmission electron microscope (figure 1.1) shows great similarity with the conventional light microscope, and consists of a source (electron gun), a system of condenser lenses, an objective lens and several image magnifying lenses (referred to as intermediate and projector lenses). Unlike the light microscope, the electron microscope column is under vacuum and magnetic lenses are used to focus the electrons. The electrons are typically created by thermal emission from a heated tungsten or LaB₆ filament, or by field emission from a sharp tungsten tip. By holding the electron gun at a high negative potential, the electrons are accelerated towards the grounded lens column. Once accelerated, the condenser lenses control the illumination of the specimen and allow the area as well as the angular convergence of the illumination to be varied. The specimen is immersed in the magnetic field of the objective lens. While traversing the specimen many of the electrons are
scattered by the specimen atoms. The very strong interaction between the incident electrons and the specimen atoms limits the useful specimen thickness from about 0.5 µm at 100 keV to several micrometers at several MeV. The back-focal plane of the objective lens contains a diffraction pattern. This diffraction pattern is basically a map of the angular distribution of the scattering. Each point in the diffraction pattern represents scattering over a particular angle.

The scattered electrons generate image contrast through a combination of two mechanisms. Amplitude or diffraction contrast results when the strongly scattered electrons are intercepted by an objective aperture placed in the back focal plane of the objective lens. As a result, areas of different composition, density or orientation are imaged with different intensities. Alternatively, phase contrast may result from the interference of the scattered and the unscattered incident electron wave. Amplitude contrast dominates in thicker, phase contrast in thinner specimens.

The electron microscope can be operated in the imaging or the diffraction mode. In the imaging mode, the first intermediate lens focuses on the objective lens' specimen image, which is then transferred by the other imaging lenses to a phosphor screen, electronic camera or photographic plate for image observation and/or recording. Alternatively in the diffraction mode, the first intermediate lens focuses on the back-focal plane of the objective lens and the diffraction pattern is finally projected onto the detector.

1.2 Electron scattering

Two types of electron scattering can be distinguished:

1. Elastic scattering occurs when the electron is deflected through a Coulomb interaction with the positively charged nucleus of a specimen atom. The term elastic reflects the fact that there is no appreciable energy-loss for small scattering angles, although in the case that the electrons are deflected over angles larger than 90° (back scattered electrons), the transfer of energy to the specimen is no longer negligible and may actually reach several tens of eV.

2. Inelastic scattering occurs as the result of Coulomb interactions with the atomic electrons surrounding the nucleus and is always associated with a loss of energy. This can happen in various ways:

- In the case of phonon scattering, the incident electron interacts with the thermal vibrations of the atoms, resulting in the creation or absorption of so called phonons. As the energy associated with the thermal vibrations is \(-kT\), where \(k\) is the Boltzmann constant and \(T\) the temperature in Kelvin, phonon scattering is limited to energy transfers below 0.1 eV.

- Plasmon scattering involves the excitation of collective oscillations of the valence electrons (conduction electrons in the case of a metal) and results in energy-losses ranging from 5 to 30 eV.

- Lastly, single electron scattering involves the transfer of energy from a beam electron to a single atomic electron. Here one can distinguish between excitation of valence electrons or inner shell electrons. Valence excitation may result in intra or inter-band transitions with energy losses ranging from several to tens or even hundreds of eV, or even to the ejection of weakly bound valence electrons (creating secondary electrons). Alternatively inner shell electrons may be excited to an
Energy-Filtered HV TEM

unoccupied energy state above the Fermi level of the solid. In both cases the atom is excited and may decay by filling the vacated state with an electron from an outer shell, leading to the emission of visible radiation (cathodoluminescence) or the emission of characteristic X-rays or Auger electrons.

1.3 The energy-loss spectrum

As a result of the inelastic scattering, the transmitted electrons will show a broad distribution of energies, or 'energy-losses'. If the energy-losses are sorted with a suitable energy-loss spectrometer, an energy-loss spectrum can be observed whose features reflect the various scattering mechanisms. The zero loss peak, the most prominent feature for thin specimens, contains those electrons that were either not scattered at all, or that were scattered elastically. The width of the zero-loss peak reflects the energy spread of the beam electrons before they reach the specimen. The spread is typically due to the electron gun and/or the Boersch effect (stochastic Coulomb interactions in beam crossovers) and amounts to about 1.5 eV (full width at half maximum) for a saturated LaB₆ filament but can be as low as 0.25 eV for a cold field emitter. The phonon scattering can not be distinguished from the zero-loss peak as its energy transfer is much smaller than the energy spread.

The remainder of the energy-loss spectrum is the result of the inelastic scattering. The plasmon and valence electron scattering cause peaks in the several to 50 eV range. On the smoothly decaying tails of these peaks one can distinguish edge-like features that are due to scattering from the inner electron shells. The onset of an edge, the ionization threshold, is approximately equal to the binding energy of the particular shell and is characteristic for each element. Identification of the energy-loss edges in the spectrum therefore allows the elemental analysis of the specimen. Elemental quantification is possible if the area underneath the energy-loss edge is measured and processed appropriately.

1.4 TEM aberrations

The magnetic lenses used in the TEM to focus the electrons suffer from several aberrations which limit the image quality. The two most important ones are the spherical and chromatic aberration.

The spherical aberration, Cₛ, causes the electrons that are scattered over larger angles to be focused closer to the lens. The resulting blur can be minimized through operation at a slight underfocus. Referred back to the specimen, the diameter dₛ of the circle containing 50% of the current is then given by(3):

\[ dₛ = 0.18Cₛβ^3 \]  

Here β is the angular divergence from the object. In the case of TEM, typically only the aberration coefficients of the objective lens are considered as the angular divergence decreases rapidly as the magnification increases going down the column. β is therefore defined by the objective aperture. With typical values for Cₛ being on the order of a few mm, β has to be restricted to about 10 mrad to guarantee a resolution on the order of nm.
The chromatic aberration, \( C_c \), expresses a dependence of the focal length of a lens on the energy of the electrons. As a result a lens can only be in focus for one electron energy. For an energy spread \( \Delta E \), the diameter \( d_c \) (referred back to the object plane) of the circle containing 50% of the current is given by (3):

\[
d_c = 0.34 C_c \frac{\Delta E}{E_0} \beta
\]

Here \( E_0 \) is the energy of the electrons for which the lens is actually focused.

As the objective lens is usually focused on the elastically scattered electrons, the inelastic scattering will cause a blurring of the image. For thick specimens (several times the mean free path for inelastic scattering, see section 2.7), the amount of inelastic scattering outweighs the elastic scattering and the deterioration of the image quality will be considerable. For instance, for a \( C_c \) of 2 mm, an average energy loss of 50 eV, a primary energy of 100 keV and an angular acceptance of 10 mrad, the blurring diameter equals 3.4 nm.

The chromatic blur can be decreased in several ways. The two methods of importance here are 1. by reducing the amount of inelastic scattering, either through an increase of the primary energy of the incident electrons or through a reduction of the specimen thickness, 2. by limiting the range of electron energies that are allowed to contribute to the final image by means of an energy filter.

### 1.5 High voltage transmission electron microscopy

The use of primary energies above 400 keV is typically referred to as High Voltage TEM. The smaller likelihood of inelastic scattering at high voltage operation results in a reduced blurring due to the chromatic aberration, allowing the study of thicker specimens, which are prepared more easily and are also better suited for the study of bulk effects or large (biological) structures.

At the same time the reduced relative energy spread \( \Delta E/E_0 \) and the narrower angular scattering distribution allow a more efficient collection of the scattering for a given resolution, although this is partially negated by the typically larger values for the aberration coefficients of HV TEM lenses.

Other advantages of high voltage TEM are easier specimen manipulation as a result of the larger space inside the naturally larger objective lens, the possibility to study the radiation damage induced by the large energy transfers for large angle elastic scattering, and the possibility of ultra high resolution TEM thanks to the smaller wavelength of the electron.

### 1.6 Electron spectroscopic imaging

The technique of imaging with only a narrow range of energies is commonly referred to as electron spectroscopic imaging (ESI) or energy-filtered transmission electron microscopy (EFTEM). ESI is possible through the incorporation or attachment of an imaging energy filter and has allowed powerful new operating modes an overview of which is given in (4).
1.6.1 Contrast enhancement

For one, by allowing only a narrow range or window of energy-losses to contribute to the image, the adverse effect of the chromatic aberration can be greatly reduced and the image contrast enhanced.

The simplest method of contrast enhancement is zero-loss filtering in which a narrow energy window (about 5 - 15 eV) is centered around the zero-loss peak. This is especially useful for biological specimens, where for the light matrix the amount of inelastic scattering is actually larger than the amount of elastic scattering. Particularly good results can also be obtained by the zero-loss filtering of convergent beam diffraction patterns. The significant reduction of the out of focus background allows more accurate quantification, as well as the use of thicker samples, which in turn results in sharper HOLZ lines(5).

Contrast enhancement through zero-loss filtering can only work well if there still is a considerable amount of elastic scattering. For very thick specimens, this is not the case and better results can be obtained by imaging with an energy window positioned elsewhere in the energy-loss spectrum. In 'most probable loss imaging', the total signal is optimized by placing the energy window at the maximum in the energy-loss spectrum. In 'contrast tuning', the position of the energy window is varied such as to optimize the contrast of the structures of interest. It should be noted though that even for specimen thicknesses of up to several times the mean free path, zero-loss filtering will still yield better contrast and resolution than most probable loss imaging, even though the total inelastic scattering already significantly outweighs the elastic scattering. It has been shown that the small fraction of elastic scattering still contains a significant portion of higher resolution phase contrast which becomes apparent once the inelastic scattering contribution is removed from the image(6).

Especially useful for biological samples is 'pre-carbon imaging' which employs an energy window placed just before the carbon K-edge at 284 eV energy-loss. This results in the relative suppression of the carbon contrast with respect to that of possible staining elements and/or other constituents.

1.6.2 Elemental mapping

The introduction of an energy window does not only decrease the effect of the chromatic aberration, but can also be used to generate elemental contrast if the energy window is chosen to coincide with an element's characteristic energy-loss edge.

ESI can therefore also be used for the creation of elemental distribution maps. In this case several images are recorded with energy windows placed on and before an element's characteristic core loss edge. Two or more images before the edge are used to determine the background intensity underneath the edge. This background is then subtracted from the edge image such that the true edge signal is obtained. Knowledge of the scattering cross-section (see below) and the specimen thickness, allow the element's absolute concentration to be determined.
1.7 Scattering angles

Both the elastically and inelastically scattered electrons are distributed over a range of scattering angles and it is important to have some understanding of the typical scattering angles prior to the design of instrumentation for ESI. Whereas it is desirable to collect as large a range of scattering angles as possible to maximize the signal, the collection of the very large scattering angles may result in a loss of spatial resolution due to the spherical and chromatic aberrations of the TEM objective lens as well any aberrations from the energy filter.

1.7.1 Elastic scattering

When using elastic scattering for imaging, one typically chooses between amplitude contrast and phase contrast. In amplitude contrast, scattering contrast is generated by intercepting the highly scattered electrons with the objective aperture. Typical semi-acceptance angles are smaller than 10 mrad at 100 keV and even lower at higher primary energies.

In the case of scattering from periodic arrays of atoms, Bragg’s law gives the requirement for constructive interference from diffracting planes with interplanar spacing $d$:

$$n\lambda = 2d \sin(\beta / 2) \quad 1.3$$

where $n$ is an integer, $\beta/2$ is the angle of incidence of the electrons on those planes, and $\lambda$ is the electron wavelength. The specimen image is now formed as a result of phase contrast and the objective aperture is either withdrawn entirely, or is sufficiently large to allow the first-order diffracted beams to contribute to the image. Typical scattering angles are for instance 10.8 mrad at 100 keV and 2.5 mrad at 1000 keV for the atomic planes of graphitized carbon (0.34 nm spacing). The current highest attainable point resolution of 0.1 nm at 1250 keV corresponds to a scattering angle of 7.4 mrad(2).

1.7.2 Inelastic scattering

For the analysis of the angular distribution of inelastic scattering it is useful to use the so called scattering cross-section. The scattering cross-section, $\sigma$, gives the probability for a certain scattering event to occur and is given by(7):

$$\sigma = \frac{N}{n_i n_i} \quad 1.4$$

Here N is the number of particular scattering events per unit volume, $n_i$ is the number of atoms per unit volume and $n_i$ is the number of incident electrons per unit area. Associated with the cross-section is a mean free path $\lambda$ which represents the distance an incident electron must travel to undergo an average of one particular scattering event:

$$\lambda = \frac{A}{\sigma N_0 \rho} \quad 1.5$$
Here $A$ is the atomic weight, $N_0$ is Avogadro's number, and $\rho$ is the density. The cross-section depends on the scattering angle $\theta$ and the energy-loss $E$. For $\theta << 1$ and $E << E_0$ the relativistically corrected differential cross-section which expresses the probability of scattering into a solid angle $d\Omega (=2\pi\sin\theta d\theta)$ with an energy-loss range $dE$ is given by (8, 9):

$$\frac{d^2\sigma}{d\Omega dE} = \frac{4a_0^2}{(E / R)(T / R)} \left( \frac{df}{dE} \right) \left[ \frac{1}{\theta^2 + \theta_E^2} + \frac{(v / c)^4 \theta^2 \theta_E^2}{(\theta^2 + \theta_E^2)(\theta^2 + \frac{\theta_E^2}{\gamma^2})^2} \right]$$

$$T = m_0v^2 / 2$$

$$\gamma = (1 - \frac{v^2}{c^2})^{-1/2}$$

$$\theta_E = E / (2\gamma T)$$

$f$, the generalized oscillator strength (GOS), represents the probability of an electron for undergoing a transition from its initial state to a particular state in the continuum and is effectively constant in the dipole region, $\theta << (2\theta_E)^{1/2}$. For primary energies $<250$ keV the first term in 1.6 will dominate and the angular distribution of the differential cross-section will be Lorentzian with a half-width $\theta_E$. The second, retardation, term in formula 1.6 becomes important for primary energies above 250 keV and intermediate scattering angles and will move the maximum in the angular distribution away from $\theta = 0$. This term is once again negligible for larger scattering angles.

Formula 1.6 can be integrated to find the cross-section for scattering up to a particular scattering angle. Figure 1.2 shows the results at 1.25 MeV for the K-edges from C, Na and P and a 100 eV energy interval starting at the edge onset. This graph is based on the parameterized data from Egerton, Appendix B was used (10) and clearly shows that for energy-losses of several keV virtually all scattering is contained within a 10 mrad scattering angle.
Figure 1.2 Integrated scattering cross-sections as a function of the scattering semi-acceptance angle for various K-edges and a 100 eV energy interval starting at the edge onset (C 285 eV, Na 1072 eV, P 2149 eV). 1.25 MeV primary energy.
2 Instrumentation for ESI

2.1 Principle of operation

Although the actual implementations may vary, each imaging energy filter performs three functions (figure 2.1):

1. Transform the unfiltered TEM image or diffraction pattern into an energy-loss spectrum.
2. Select a narrow energy window in the energy-loss spectrum using an energy selecting slit.
3. Transform the selected energy window into an energy filtered image or diffraction pattern and project it onto a suitable detector.

![Diagram showing the process of transforming a TEM image into an energy-filtered image](image)

Figure 2.1 Schematic representation of the various functional stages of an imaging energy filter.

The energy-loss spectrum is created by imaging a narrow beam cross-section using an energy-dispersive element. It is best to use the typically strongly demagnified diffraction pattern (TEM in image mode) or image (TEM in diffraction mode) near the back-focal plane of the last pre-imaging filter lens. This offers the advantage that 1) its small size in the energy loss spectrum will have minimal effect on the energy resolution and 2) as here the entire image or diffraction pattern is represented in a near point, any vignetting of the final energy-filtered image or diffraction pattern is minimized.
Once the energy-loss spectrum is created, the imaging energy filter can has two modes of operation.

1. In the imaging mode, the filter projects an energy-filtered version of the TEM image plane onto the filter's detector.
2. In the spectroscopy mode, the energy-selecting slit is withdrawn and the filter projects the energy-loss spectrum onto the detector.

These two operating modes and their conjugate planes are depicted in figure 2.2.

Figure 2.2 Conjugate planes for the Imaging and Spectroscopy modes of operation. The TEM is in the imaging mode.
2.2 Design parameters for imaging energy filters

The ideal imaging energy filter behaves almost like an ideal round lens and transforms the unfiltered TEM image into an achromatic (energy dispersion free), energy-filtered image, free of distortions and blurring and with a well defined energy window that is uniform across the object. Similarly the energy-loss spectrum at the energy selecting slit will be purely the result of the convolution of the energy-loss distribution introduced by the specimen and the energy spread in the electron beam before the specimen.

This is however not a practical reality and the performance of actual energy filters is limited by various factors.

2.2.1 Aberrations

As a result of the electron optical aberrations, an object point will typically be imaged into a spot or aberration figure (see figure 2.3). If large enough, the aberrations will limit the spectrum or image resolution.

![Diagram of object and image with coordinates x, y, z]

Figure 2.3 Blurring in the image due to the presence of aberrations. As expressed in formula 2.1 the imaging properties can be expressed as a function of the object coordinates \(x_0\) and \(y_0\), the slopes \(x'_0\) and \(y'_0\) given by \(dx_0/dz\) and \(dy_0/dz\) and the fractional momentum deviation \(\delta\).

The aberrations can be characterized by expressing an arbitrary electron ray's image coordinates \(x_1\) and \(y_1\), in polynomials of the ray's object coordinates \(x_0\) and \(y_0\), slopes \(x'_0\) and \(y'_0\), as well as the fractional momentum deviation \(\delta = \Delta p/p\), \(p\) being the central momentum:

\[
\begin{align*}
x_1 &= \sum_{i,j,k,l,m} A_{ijklm} x_0^i y_0^j x'_0^k y'_0^l \delta^m \\
y_1 &= \sum_{i,j,k,l,m} B_{ijklm} x_0^i y_0^j x'_0^k y'_0^l \delta^m
\end{align*}
\]

Note that this representation does not assume the system to be rotationally symmetric. Those constants \(A_{ijklm}\) and \(B_{ijklm}\) for which the sum of the indices \(i,j,k,l,m\) equals 1 are referred to as the first-order properties. They express the paraxial magnifications, focusing powers and energy dispersive properties of the system. All constants \(A_{ijklm}\) and \(B_{ijklm}\) for which the sum of the indices is greater than 1 are referred to as the aberrations of the system. Those with the sum equaling 2 are
referred to as second-order aberrations, those with the sum equaling 3 as third-order aberrations, etc. Aberrations are especially important for non-paraxial rays, i.e. larger objects and slopes.

A basic division can be made between geometric and chromatic aberrations, where the geometric aberrations are independent of the momentum deviation $\delta$ ($m=0$). The aberrations can further be classified by their dependence on the various object parameters(11):

Geometric aberrations

- Aperture aberrations are independent of the object point, and the aberration figure depends only on the slopes $x'_0$ and $y'_0$. Aperture aberrations cause a uniform blurring throughout the image.
- Distortions depend purely on the coordinates $x_0$ and $y_0$. Distortions do not cause blurring and do not exist on the optical axis.
- Field aberrations depend on both the coordinates and slopes and therefore do not exist on the optical axis.

Some special chromatic aberrations:

- Energy dependent magnifications depend on the momentum deviation $\delta$ and the coordinate $x_0$ or $y_0$.
- Axial chromatic aberrations depend on the momentum deviation $\delta$ and the slopes $x'_0$ and $y'_0$.

2.2.2 Non-isochromaticity

Both the size of the objective aperture in the energy-loss spectrum and the spectrum aberrations contribute to a variation of the selected energy across the image field. Referred to as the non-isochromaticity of the system, this variation determines the smallest energy window that can be selected without causing any vignetting in the image.

The size $d_\beta$ (eV) of the objective aperture in the energy-loss spectrum is given by:

$$d_\beta = 2\beta \frac{L}{M_T} \frac{M_s}{D}$$  

2.2

Here $\beta$ is the objective aperture semi-angle, $L$ the distance from the crossover to the object plane, $M_T$ is the TEM magnification at the filter's object plane, $M_S$ is the magnification from the crossover to the energy-loss spectrum at the energy-selecting slit, and $D$ the energy-dispersion at the energy-selecting slit (in m/eV), and $L/M_T$ is effectively the camera length referenced to the filter's object plane(12). The size of the crossover in the spectrum can be minimized by ensuring that the term

$$L \frac{M_S}{D}$$  

2.3

is as small as possible.
2.2.3 Transmissivity

Table 2.1 lists various resolution limiting factors in the energy-loss spectrum and the image as well as the elements of the overall system that affect them.

<table>
<thead>
<tr>
<th>Mode</th>
<th>Resolution limited by</th>
<th>Affected by</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spectrum</td>
<td>Beam energy spread</td>
<td>Gun</td>
</tr>
<tr>
<td></td>
<td>Boersch effect</td>
<td>Gun, TEM illumination optics</td>
</tr>
<tr>
<td></td>
<td>Spectrum aberrations</td>
<td>Entrance aperture, filter optics</td>
</tr>
<tr>
<td></td>
<td>Objective aperture size</td>
<td>Objective aperture</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Intermediate magnification</td>
</tr>
<tr>
<td>Image</td>
<td>Aperture aberrations</td>
<td>Objective aperture, filter optics</td>
</tr>
<tr>
<td></td>
<td>Field aberrations</td>
<td>Objective/entrance aperture, filter optics</td>
</tr>
<tr>
<td></td>
<td>Distortions</td>
<td>Entrance aperture, TEM optics</td>
</tr>
<tr>
<td></td>
<td>TEM objective aberrations</td>
<td>Objective aperture, TEM optics</td>
</tr>
<tr>
<td></td>
<td>Diffraction limit (Airy disc)</td>
<td>Objective aperture</td>
</tr>
</tbody>
</table>

Table 2.1 Resolution limiting factors for both the energy-loss spectrum and the image and the parameters can be changed to affect them. The intermediate magnification refers to the magnification of the TEM image in the filter's object plane.

The effect of many of the resolution limiting factors can be reduced by restricting the size of the various apertures. An exception is the Airy disc, which is the result of aperture diffraction and is actually larger for smaller objective apertures. It should be noted that although the resolution can typically be improved by using smaller apertures, this will result in a reduced angular acceptance and field of view.

An important measure of a filter's performance is its transmissivity $T$, which is the product of the imaged specimen area of radius $p$ and the solid scattering acceptance angle (13).

$$T = (\pi p^2)$$

2.4

The transmissivity of a filter is typically calculated for a specific spectrum and image resolution. Its concept is similar to that of phase space often used in spectrometer design (14). Phase space is the 6-dimensional particle density $p(x, p_x, y, p_y, z, p_z)$ covering the spatial coordinates and momenta of the electrons traveling through the system. Good filter design maximizes the transmissivity without compromising spectrum or image resolution.

For an imaging filter on the TEM the energy resolution is fundamentally limited by the energy spread of the electron gun. The image resolution is fundamentally limited by the aberration coefficients of the objective lens ($C_S$ and $C_C$). If the imaging filter were entirely aberration free, the only other energy resolution limiting parameter would be the size of the objective aperture in the energy-loss spectrum. To maximize this filter's transmissivity it should be placed at the end of the TEM column to maximize the intermediate magnification ($M_I$) and so to minimize the size of the objective aperture in the spectrum (formula 2.2).

This, however, is not the optimal configuration for a practical, aberrated filter. Placing the filter at the end of the column implies a large entrance aperture and poor spectrum (aperture aberrations) and possibly image resolution (field aberrations and distortions). To reduce these aberrations the filter's entrance aperture must be reduced. Effectively this means that the filter is moved up the
column. The intermediate magnification is reduced and the post-filter magnification is increased. Moving the filter up the column will however increase the size of the objective aperture in the spectrum, which may in its turn start limiting the resolution. Obviously there is a location where for a given resolution the transmissivity is optimized.

A filter's transmissivity is best optimized by minimizing the filter's spectrum and image aberrations, and minimizing the term of formula 2.3. Next its entrance aperture should be optimized for the desired resolution. The size of the detector will then determine the necessary post-filter magnification.

It is important to realize that a filter's transmissivity may have a maximum meaningful value. For one, the limited extent of the elastic and inelastic scattering distributions may imply a largest meaningful acceptance angle, especially in the case of operation at high voltage (see section 1.7). But also the TEM objective lens aberrations may impose a limit on the acceptance angle. Table 2.2 lists as a function of image resolution, the minimum overall magnification necessary to resolve this detail for a detector with 24 μm pixels, the corresponding Bragg scattering angle for elastic scattering, as well as the angles for which the blurring due to the spherical and chromatic aberration (formulas 1.1 and 1.2) equals the image resolution.

<table>
<thead>
<tr>
<th>Resolution nm</th>
<th>Magnification</th>
<th>Bragg angle mrad</th>
<th>Cs angle mrad</th>
<th>Cc angle mrad</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1</td>
<td>480000</td>
<td>7.4E+00</td>
<td>5.8E+00</td>
<td>2.1E+00</td>
</tr>
<tr>
<td>0.2</td>
<td>240000</td>
<td>3.7E+00</td>
<td>7.3E+00</td>
<td>4.2E+00</td>
</tr>
<tr>
<td>0.5</td>
<td>96000</td>
<td>1.5E+00</td>
<td>1.0E+01</td>
<td>1.1E+01</td>
</tr>
<tr>
<td>1.0</td>
<td>48000</td>
<td>7.4E-01</td>
<td>1.3E+01</td>
<td>2.1E+01</td>
</tr>
<tr>
<td>2.0</td>
<td>24000</td>
<td>3.7E-01</td>
<td>1.6E+01</td>
<td>4.2E+01</td>
</tr>
<tr>
<td>5.0</td>
<td>9600</td>
<td>1.5E-01</td>
<td>2.1E+01</td>
<td>1.1E+02</td>
</tr>
<tr>
<td>10.0</td>
<td>4800</td>
<td>7.4E-02</td>
<td>2.7E+01</td>
<td>2.1E+02</td>
</tr>
<tr>
<td>20.0</td>
<td>2400</td>
<td>3.7E-02</td>
<td>3.4E+01</td>
<td>4.2E+02</td>
</tr>
<tr>
<td>50.0</td>
<td>960</td>
<td>1.5E-02</td>
<td>4.6E+01</td>
<td>1.1E+03</td>
</tr>
<tr>
<td>100.0</td>
<td>480</td>
<td>7.4E-03</td>
<td>5.8E+01</td>
<td>2.1E+03</td>
</tr>
</tbody>
</table>

Table 2.2 Image resolution, corresponding minimum magnification and Bragg scattering angles (formula 2.1). Also shown are the acceptance semi-angles at which the blurring due to the spherical and chromatic aberrations of the objective lens equals the image resolution. 1.25 MeV primary energy, C_S = 2.8 mm, C_C = 3.5 mm, 50 eV energy window.

This table shows that already at 50 nm resolution the maximum useful semi-acceptance angle is limited by the spherical aberration to about 50 mrad.

### 2.3 Imaging energy filter designs

Over the past few decades, various energy filter designs have been proposed and built. These energy filters can be divided into two classes. The first class, including among others the imaging Wien Filter(15), uses electrostatic fields, often in combination with magnetic fields, and are precluded from use above about 80 keV due to the technical difficulties encountered when working with large electrostatic potentials. The second class uses only magnetic fields and does not have a principal limitation in the operating voltage. One can also distinguish between so
called in-column filters, which are incorporated into the imaging column of the TEM, and post-column filters which attach to the bottom to the TEM camera chamber.

As this thesis concerns the design of an imaging energy filter for use up to 1250 keV, the discussion will be limited to those filters employing magnetic prisms as the energy-dispersive element.

### 3.3.1 The magnetic prism

An electron beam entering a magnetic prism (figure 3.3) is deflected by the Lorentz force resulting from the magnetic field $B$ that is perpendicular to the electron beam. As a result the electrons will follow a circular trajectory the radius $\rho$ of which is given by:

$$\rho = \left( \frac{\gamma m_0}{eB} \right) v$$

where $\gamma = \frac{1}{\sqrt{1 - v^2/c^2}}$ is a relativistic correction factor and $m_0$ is the rest mass of the electron.

The dependence of the bending radius on the momentum of the electron causes the energy-loss electrons to experience a smaller bending radius than the zero-loss electrons and introduces the energy dispersion. Typically bending angles on the order of $90^\circ$ are used to obtain a reasonable dispersion.

Besides introducing energy dispersion, the magnetic prism also has a focusing effect on the electron beam. This can be understood by realizing that those electrons entering the magnetic prism on the left hand side of the optical axis in fig. 3.3, will travel in the magnetic prism for a longer distance than those entering on the right hand side. Both will therefore be deflected towards the optical axis. This focusing effect occurs throughout the magnetic prism, although only in the x, z plane (parallel to the pole faces).

By inclining the entrance and exit faces of the magnetic prism with respect to the optical axis, a local so called quadrupole effect can be introduced yielding a focusing effect in the x, z and a defocusing effect in the y, z effect or vice versa. It is also possible to yield continuous focusing effects in both directions through the use of wedge shaped magnets (16, 17).

Similarly, curving the entrance and exit faces or the pole faces will introduce so called sextupole effects that can be used to control various second-order aberrations.

### 2.3.2 In-column filters

The forerunner of the modern in-column imaging energy filters employing magnetic prisms was developed by Castaing and Henry (18, 19, 20). The 'Castaing-Henry' or 'prism, mirror, prism' filter uses a magnetic prism to bend the electron beam over $90^\circ$, an electrostatic mirror to reflect the electron beam and another $90^\circ$ magnetic prism to bend the beam back to the optical axis of the TEM. A focused spectrum present after the filter allows energy-selection. Various versions of the Castaing-Henry filter have been built (21, 22, 23). Zeiss successfully commercialized the design with their EM 902 Transmission Electron Microscope with Energy Filter (24). Thanks to symmetry of the fields and the fundamental rays, the image of the Castaing-Henry filter is free of second-order aperture aberrations and distortions, although it does suffer from large field aberrations which limit the field of view, as
well as from second-order chromatic aberrations in the energy-loss spectrum that significantly limit the isochromaticity in the energy-filtered image.

Because the electrostatic mirror made the Castaing-Henry filter unsuitable for use at voltages greater than about 80 kV, Senoussi started the development of a purely magnetic Omega filter (named after the Greek letter whose shape it resembles) for use on a high voltage microscope(25). Unfortunately, unlike the Castaing-Henry filter, this design lacked midplane symmetry and suffered from all second-order aberrations.

The first symmetric magnetic filter was proposed by Rose and Plies (26), figure 2.4. The first symmetric magnetic filter was actually built by Zanchi et al.(27). Designs were later also offered by Krah et al., Perez et al. and Oikawa et al(28, 29, 30, 31). Except for the latter, these systems were free of second-order aperture aberrations and distortions, although their performance suffered from the remaining second-order field aberrations.

Figure 2.4 Schematic overview of an in-column Omega filter.
The first fully corrected Omega filter was proposed by Rose and Pejas. (32). It used curved prism faces and three symmetrically arranged sextupoles to control the second-order field aberrations in the image and the second-order energy dependent magnification aberrations in the energy-selecting plane. This filter was built by Krahl(33) who found it was practically impossible to accurately align the instrument. As this was believed to be due to the curvatures of the entrance faces of the magnets, these were straightened in a new design and replaced by 7 symmetrically arranged separate sextupoles(34).

In order to find a simpler configuration with good performance, Lanio performed an extensive search for a filter with small aberrations and as few elements as possible(35). This search resulted in an optimum Omega and an optimum Alpha filter. The concept of the optimum Omega filter, which had one sextupole added to the symmetry plane to improve the energy resolution, was used by Zeiss in the design of their EM 912 Omega Analytical Energy Filtering Transmission Electron Microscope(36, 37, 38).

A promising in-column filter was recently introduced by Uhlemann and Rose(16, 17). Their Mandolin filter (named after the shape of the musical instrument) uses two wedge shaped and one conventional magnetic prism. This filter yields a high energy dispersion, is corrected for all second-order aberrations and has minimized third-order aberrations in the spectrum plane. Although promising on paper, this system has not yet been build and tested.
2.3.3 Post-column filters

Post-column filters attach to the camera chamber of the TEM. Unlike in-column filters they can easily be retrofitted to an existing TEM to add the functionality of energy-filtering.

![Diagram of post-column filters]

Figure 2.5 Schematic overview of a post-column imaging filter. The two sets of conjugate planes are marked * and **.
The development of the post-column filter followed that of the single prism electron energy-loss spectrometers. The first post-column filter was built by Shuman(39) who used a single magnetic prism followed by an energy selecting slit and two round lenses. A similar system, consisting of only an energy-selecting slit, a drift tube and a camera, was built by Ajika et al.(40). The predecessor of the modern post-column filter was built by Krivanek et al. who used an energy-selecting slit, a lens assembly consisting of 3 quadrupoles and an intensified TV rate camera(41, 42).

All of the early designs suffered from aberrations in either or both the energy-loss spectrum and the image. This severely restricted the angular acceptance, field of view and the size of the energy window that could be used.

The development of the modern post-column imaging filter (figure 2.5) started in 1990 and lead to the commercial introduction of the Gatan Imaging Filter in 1992.(43, 44, 45, 46, 47, 48, 49). Unlike its predecessors this design incorporated sextupole elements to control the important second-order aberrations in the image.

A post-column filter can basically be separated into pre- and post-slit optics. The pre-slit optics consists of a 90° bending magnet and possibly a dispersion magnifying quadrupole lens. It transfers the TEM projector lens crossover into a doubly focused (x and y direction) energy-loss spectrum at the energy-selecting slit. The energy dispersion in the spectrum ranges from about 2 to 10 μm/eV for the various different implementations and operating energies.

The energy resolution in the spectrum is determined by the prism's aperture aberrations. To allow a large entrance aperture while ensuring good energy resolution, the important second-order aperture aberrations in the energy-loss spectrum are corrected by appropriately inclining and curving the prism's entrance and exit faces.

The imaged area as selected by the entrance aperture is transferred by the pre-slit optics into separate (virtual) images for the x- and y-direction, the one for the x-direction being dispersion free or achromatic. The post-slit optics consists of several quadrupole and sextupole lenses and can be operated in two modes. In the imaging mode the quadrupoles transfer the (energy-filtered) x and y images from the pre-slit optics to the detector with appropriate magnifications. The sextupole lenses are used to cancel any important second-order image aberrations. In the spectroscopy mode, the energy-selecting slit is withdrawn and the quadrupoles project an image of the energy-loss spectrum at the slit plane onto the detector. The energy dispersion can be varied by adjusting the magnification of the quadrupole lens system. The sextupoles are turned off as the important second-order spectrum aberrations were already corrected by the bending magnet.
3 Electron Optical Calculations

3.1 Methods

The design of a post-column imaging energy filter is an iterative process: an optical design is proposed, its optical properties determined and evaluated. Depending on the outcome of the evaluation, a modified design may be proposed and the process starts again. Given the complexity and the many parameters involved, the help of a powerful computer program is essential in this process.

Generally the electron optical properties are deduced from the electron trajectories. These follow from solving the equation describing the motion of an electron in the magnetic field. The equation of motion follows from the Lorentz force on the electron

$$\vec{F} = -e\vec{v} \times \vec{B}$$

where \(\vec{v}\) the speed of the electron and \(\vec{B}\) the magnetic induction. Equation 3.1 can be developed further to yield(50):

$$\frac{d^2 \vec{r}}{dt^2} = -\frac{e}{P} \left( \frac{d\vec{r}}{dT} \times \vec{B} \right)$$

Here \(\vec{r}\) is the electron's position vector, and \(T\) the distance traveled.

In order to solve equation the equation 3.2, the magnetic field must be known. For static magnetic fields in a current free space, the magnetic potential \(\Phi\) must satisfy the Laplace equation

$$\nabla^2 \Phi = 0$$

The Laplace equation can either be solved analytically or numerically. In the numerical case, finite difference or finite elements methods may be used. These methods do not yield an analytical expression for the potential distribution, and ray tracing through direct integration of the differential equation of motion is often used to yield the electron trajectories. The optical properties of the system are then determined by following many individual trajectories and interpreting their intersections with the image plane. This method is powerful for complex, non-symmetrical systems, but does not offer good insight into the optical properties and their dependence on the various design parameters.

If the field distribution can be expressed analytically, it may be possible to find an analytical solution for the equation of motion. A proper choice of the coordinate system is key here. First the central trajectory or optical axis is determined. Next a Taylor expansion describing an arbitrary trajectory around the central trajectory is proposed and substituted in the differential equation of motion. Depending on the desired accuracy, terms up to a certain order are retained. The various order differential equations are then solved to yield the various Taylor coefficients.

For a more extensive discussion of electron optical calculations in general see for instance (11, 14, 50, 51, 52).
3.2 Transport

3.2.1 Matrix formalism

In the case of the post-column imaging filter, it is possible to divide the system into well separated, distinct optical elements. The optical properties of the elements have been well described and the electron trajectories through these elements are well known. It is therefore possible to express the trajectory parameters at the exit of such an element as a function of the parameters at the entrance.

This is the basis for a design method employing a matrix formalism. Here each element in the optical system is described by a transfer matrix $R$ that transforms an electron's trajectory vector $X(0)$ at the entrance into the trajectory vector $X(1)$ at the exit.

$$X(1) = RX(0)$$  \hspace{1cm} 3.4

The overall transfer matrix $R_T$ for a system of $n$ elements, is given by the multiplication of the individual transfer matrices:

$$R_T = R_n \cdots \cdot R_2 R_1$$  \hspace{1cm} 3.5

The matrix formalism allows a fast and easy way to follow an electron's trajectory through an optical system by examination of the coefficients of the accumulative transfer matrix.

This method was used here using TRANSPORT(50, 53, 54, 55), a matrix multiplication computer program for the calculation of static-magnetic beam transport systems with midplane symmetry. Although initially intended for application in high energy physics, Transport lends itself very well as a tool in design of imaging energy filters: imaging energy-filters posses midplane symmetry, use only static magnetic fields and use drift spaces, bending magnets, quadrupoles, sextupoles and octupoles, all of which are supported by Transport.

Using Transport notation equation 3.4 can be written as:

$$\begin{bmatrix} x_1' \\ x_1 \\ y_1' \\ y_1 \\ l_1 \\ \delta_1 \end{bmatrix} = \begin{bmatrix} R_{11} & R_{12} & R_{13} & R_{14} & R_{15} & R_{16} \\ R_{21} & R_{22} & R_{23} & R_{24} & R_{25} & R_{26} \\ R_{31} & R_{32} & R_{33} & R_{34} & R_{35} & R_{36} \\ R_{41} & R_{42} & R_{43} & R_{44} & R_{45} & R_{46} \\ R_{51} & R_{52} & R_{53} & R_{54} & R_{55} & R_{56} \\ R_{61} & R_{62} & R_{63} & R_{64} & R_{65} & R_{66} \end{bmatrix} \begin{bmatrix} x_0 \\ x_0 \\ y_0 \\ y_0 \\ l_0 \\ \delta_0 \end{bmatrix}$$  \hspace{1cm} 3.6
The trajectory vector $X$ contains the positions, angles and momentum of the arbitrary trajectory with respect to an assumed central trajectory (see fig. 3.1) that lies entirely in the midplane:

- $x = \text{the radial displacement of the arbitrary ray with respect to the assumed central trajectory}$
- $x' = \text{the angle this ray makes in the radial plane with respect to the assumed central trajectory}$
- $y = \text{the transverse displacement of the ray with respect to the assumed central trajectory}$
- $y' = \text{the transverse angle of the ray with respect to the assumed central trajectory}$
- $l = \text{the path length difference between the arbitrary ray and the central trajectory}$
- $\delta = \Delta p/p$ is the fractional momentum deviation of the ray from the assumed central trajectory

![Diagram showing right-handed, curvilinear coordinate system](image)

Figure 3.1 The right-handed, curvilinear coordinate system used in Transport, consisting of unit vectors $x$, $y$ and $z$.

The matrix coefficients of equation 3.6 follow from the Taylor expansion, truncated to first-order, describing an arbitrary trajectory about the central trajectory. The matrix formalism can be extended to the second- and third-order by including the higher order terms in the Taylor expansion:

$$X_i(1) = \sum_j R_{ij} X_j(0) + \sum_{jk} T_{ijk} X_j(0) X_k(0)$$

$$+ \sum_{jkl} U_{ijkl} X_j(0) X_k(0) X_l(0) = R_i X(0)$$
\( T_{ijk} \) and \( U_{ijkl} \) are the elements of the second-order matrix \( T \) and third-order matrix \( U \). For instance for \( x_1 \):

\[
T_{x_1} = \begin{bmatrix}
T_{111} \\
T_{112} & T_{122} \\
T_{113} & T_{123} & T_{133} \\
T_{114} & T_{124} & T_{134} & T_{144} \\
T_{115} & T_{125} & T_{135} & T_{145} & T_{155} \\
T_{116} & T_{126} & T_{136} & T_{146} & T_{156} & T_{166}
\end{bmatrix}
\]

\[
U_{x_1} = \begin{bmatrix}
U_{111} & U_{112} & U_{122} \\
U_{113} & U_{123} \\
U_{133} & U_{133} & U_{133} & U_{133} \\
U_{114} & U_{124} & U_{124} \\
U_{134} & U_{134} & U_{134} & U_{134} & U_{134} \\
U_{144} & U_{144} & U_{144} & U_{144} & U_{144} \\
U_{115} & U_{115} & U_{115} & U_{115} & U_{115} & U_{115} \\
U_{116} & U_{116} & U_{116} & U_{116} & U_{116} & U_{116} & U_{116} & U_{116} & U_{116}
\end{bmatrix}
\]

The coefficients of the \( T \) and \( U \) matrices describe the many possible aberrations of the optical system, i.e. the deviations of the non-paraxial properties from the paraxial properties. For various reasons most of these coefficients are zero. For instance as a result of the midplane symmetry, the magnetic potential \( \Phi \) is an odd function in the \( y \) coordinate

\[
\Phi(x, y, z) = -\Phi(x, -y, z)
\]

and the magnetic field must follow the relationships
Electron Optical Calculations

\[ B_x(x, y, t) = -B_x(x, -y, z) \]
\[ B_y(x, y, t) = B_y(x, -y, z) \]
\[ B_z(x, y, t) = -B_z(x, -y, z) \]

resulting in

\[ B_x(x, 0, z) = B_y(x, 0, z) = 0 \]

indicating that the magnetic field is perpendicular to the mid-plane. To first order, the bend plane coordinates \( x \) or \( x' \) can therefore not depend on the non-bend plane coordinates \( y \) or \( y' \) and vice versa, and in the first-order matrix:

\[ R_{13} = R_{14} = R_{23} = R_{24} = R_{31} = R_{32} = R_{41} = R_{42} = 0 \]

Also for reasons of symmetry

\[ R_{36} = R_{46} = R_{53} = R_{54} = 0 \]

Because the elements of the trajectory vector are independent of the path difference

\[ R_{15} = R_{25} = R_{35} = R_{45} = R_{65} = 0 \]

Because the systems under discussion employ only static magnetic fields, the momentum is a constant of motion resulting and

\[ R_{61} = R_{62} = R_{63} = R_{64} = R_{65} = 0 \]

This reduces the first-order matrix to:

\[
R = \begin{bmatrix}
R_{11} & R_{12} & 0 & 0 & 0 & R_{16} \\
R_{21} & R_{22} & 0 & 0 & 0 & R_{26} \\
0 & 0 & R_{33} & R_{34} & 0 & 0 \\
0 & 0 & R_{43} & R_{44} & 0 & 0 \\
R_{51} & R_{52} & 0 & 0 & 1 & R_{56} \\
0 & 0 & 0 & 0 & 0 & 1
\end{bmatrix}
\]

Similarly most of the second- and third-order coefficients are zero. The non-zero coefficients for \( x_1 \) and \( y_1 \) are:

in the bend plane \((x, t)\),

\[
T_{111}, T_{112}, T_{122}, T_{133}, T_{134}, T_{144}, T_{116}, T_{126}, T_{166}
\]

\[
U_{1111}, U_{1112}, U_{1122}, U_{1123}, U_{1133}, U_{1134}, U_{1134}, U_{1234}, U_{1144}, U_{1244}
\]

\[
U_{1126}, U_{1226}, U_{1336}, U_{1336}, U_{1446}, U_{1366}, U_{1466}, U_{1666}
\]

and in the non-bend plane \((y, t)\),

\[
T_{313}, T_{323}, T_{314}, T_{324}, T_{336}, T_{346}
\]

\[
U_{3113}, U_{3123}, U_{3223}, U_{3333}, U_{3114}, U_{3124}, U_{3224}, U_{3334}, U_{3334}, U_{3344}, U_{3444}
\]

\[
U_{3136}, U_{3236}, U_{3146}, U_{3246}, U_{3366}, U_{3466}
\]

For a detailed description on how the values of the matrix elements relate to the physical parameters of the optical elements, see(50).
3.2.2 Fitting

In the optical design process the individual matrices of the optical components are combined to determine the overall transfer matrix. The optical properties follow from the matrix coefficients. The various design parameters of the system are then varied to control the matrix coefficients and to obtain the desired optical properties.

To aid in this process, Transport allows constraints to be imposed on the coefficients of a transfer matrix, and the physical parameters of the optical elements to be varied in order to meet these constraints. For instance, the excitation of one or more quadrupoles can be varied to ensure that at a particular plane $R_{12} = R_{34} = 0$. This specifies first-order stigmatic imaging as the image coordinates $x_1$ and $y_1$ are independent of the angles $x_0'$ and $y_0'$ in the object plane. Similarly, magnifications for the x- and y-direction may be specified through $R_{11}$ and $R_{33}$, or first-order achromatic imaging may be specified with $R_{16} = 0$. If a particular energy-dispersion is desired it is specified through $R_{16}$. The energy dispersion $D$ (m/eV) of the electron beam follows from $R_{16}$ through:

$$D = kR_{16}$$  \[3.18\]

with $k$ given by

$$k = \beta^2(eV + m_0c^2)^{-1}$$  \[3.19\]

$$\beta = \frac{v}{c} = 2\frac{\sqrt{eV(1 + eV)}}{1 + 2eV}$$  \[3.20\]

$$\epsilon = \frac{e}{2m_0c^2} = 0.978 \times 10^{-6}$$  \[3.21\]

Here $v$ is the speed of the electron, $V$ the accelerating potential, $e$ the charge of the electron, $m_0$ its mass and $c$ is the speed of light. The above formulas include relativistic effects, as for operation at high primary energies, the speed of the electrons starts approaching the speed of light ($\beta \sim 0.95$ at 1.25 MeV, see appendix).

Basically Transport can vary elements up to third order to fit matrix coefficients of that order. For instance Transport can vary the curvatures of a prism's entrance and exit faces to control second-order aberrations in the energy-loss spectrum, or vary the excitation of an octupole to correct a certain third-order aberration in the image.

3.3 The optical elements

The various optical elements that are used in the post-column filter will be briefly described.

3.3.1 Drift space

The optical elements are separated by field free drift spaces. The transfer matrix for a drift space of length $l$ is simply given by:
Electron Optical Calculations

\[
R = \begin{bmatrix}
1 & 0 & 0 & 0 & 0 & 0 \\
0 & 1 & 0 & 0 & 0 & 0 \\
0 & 0 & 1 & 0 & 0 & 0 \\
0 & 0 & 0 & 1 & 0 & 0 \\
0 & 0 & 0 & 0 & 1 & 0 \\
0 & 0 & 0 & 0 & 0 & 1 \\
\end{bmatrix}
\]

The electron trajectories through a drift space are straight and the coordinates at the end of a drift are simply given by the ones at the start plus the slope of the trajectories times the length of the drift space.

3.3.2 Multipoles

The post-column filter uses a variety of multi-poles (figure 3.2). In general, for a magnetic multi-pole of order \( n \), the magnetic potential \( \Phi \) in cylindrical coordinates along a cylinder centered on the optic axis is given by:

\[
\Phi_n(r, \varphi) = \Phi_n \left( \frac{r}{a} \right)^n \sin n \varphi
\]

where \( r \) is the distance from the optic axis and \( \varphi \) is the angular coordinate.

The force exerted on the electron beam is proportional to the gradient of the magnetic potential or to:

\[
\left( \frac{r}{a} \right)^{n-1}
\]

The force is therefore of order \( n-1 \), and will show the same sinusoidal dependence on the angular coordinate as the magnetic potential.

For various values of \( n \) we find:

- \( n=1 \) dipole
- \( n=2 \) quadrupole
- \( n=3 \) sextupole
Figure 3.2 Dipole, quadrupole and sextupole elements implemented by shaped pole faces evenly divided around the optical axis.

The multipole elements can be created in several ways:

Dipoles
- 2 poles 180° apart
- 2 extended poles faces (magnetic prism)

Quadrupoles
- 4 poles 90° apart
- rotated entrance or exit face of a magnetic prism
- transverse slope of bending magnet pole face

Sextupoles:
- 6 poles 60° apart
- curved entrance and exit face of a magnetic prism
- second-order transverse curvature of a magnetic prism pole face

3.3.3 Dipoles

\[ \Phi(r, \varphi) = \Phi_1 \frac{r}{a} \sin \varphi \]  \hspace{1cm} 3.25

For a dipole the force is independent of the distance from the optic axis and the effect is therefore a uniform beam deflection.
3.3.4 Quadrupoles

\[ \Phi(r, \varphi) = \Phi_2 \frac{r^2}{a^2} \sin 2\varphi \]

For the quadrupole the force on the electron is proportional to the distance from the optic axis causing the quadrupole to have a focusing effect in one and a defocusing effect in the perpendicular direction.

For an ideal quadrupole the hyperbolic iso-potential planes are described by:

\[ xy = \frac{a^2 \Phi_2(x, y)}{2\Phi_2} \]

In reality the hyperbolic pole faces are typically approximated by cylinders. The radius \( R \) of the cylinders is then given by(14, 56):

\[ R = 1.1468a \]

This essentially eliminates the 5th order component. The first higher order component will be of 9th order.

In electron optical calculations, the quadrupoles are often approximated by block fields of length \( L \). The magnetic field is assumed to be zero outside the effective length, whereas at the effective boundary it suddenly increases to a constant value. The effective length \( L \) can be defined by(56):

\[ L = \frac{1}{B_z(0)} \int B_z(z)dz \]

Where \( B_z(z) \) is the amplitude of the transverse component of the magnetic induction \( B \) at a distance \( r \) from the z- or optical axis. The effective length \( L \) will be longer than the length of the pole faces and can be closely approximated by:

\[ L \approx l + 1.1a \]

Where \( l \) is the length of the pole faces and \( a \) is the inscribed radius of the pole faces.

The first-order matrix for a quadrupole is given by:

\[
R = \begin{bmatrix}
\cos(kqL) & \frac{1}{kq} \sin(kqL) & 0 & 0 & 0 \\
-kq \sin(kqL) & \cos(kqL) & 0 & 0 & 0 \\
0 & 0 & \cosh(kqL) & \frac{1}{kq} \sinh(kqL) & 0 & 0 \\
0 & 0 & kq \sinh(kqL) & \cos(kqL) & 0 & 0 \\
0 & 0 & 0 & 0 & 1 & 0 \\
0 & 0 & 0 & 0 & 0 & 1
\end{bmatrix}
\]
where

\[ L = \text{the effective length of the quadrupole} \]

\[ k_q^2 = \frac{B_0}{a}(1/B\rho_0) \]

\[ a = \text{the radius of the aperture} \]

\[ B_0 = \text{the magnetic induction at radius } a \]

\[ B\rho_0 = \text{the magnetic rigidity of the central trajectory} \]

Typically the effective length of a quadrupole (or higher order element) does not need to be known precisely as its strength is proportional to the product of the effective length and the field at the pole tips. Any small errors in the effective length can therefore easily be compensated by an adjustment of the magnetic induction at the tip.

### 3.3.5 Sextupoles

\[ \Phi(r, \phi) = \Phi_3 \frac{r^3}{a^3} \sin 3\phi \]

For sextupoles the force is proportional to the square of the distance from the optic axis. A sextupole is therefore a second-order element and will couple to the second-order coefficients.

The first-order matrix of a sextupole equals that of a drift space as an ideal multipole can only have an effect on its fundamental and higher orders.

The poles face for sextupoles are typically cylinders or even flat. The deviation from the ideal iso-potential planes will give rise to higher order effects, but these can typically be neglected.
3.3.6 Bending magnet

Figure 3.3 Magnetic prism. Indicated are the bending radius \( \rho_0 \), the bending angle \( \alpha \), the entrance and exit face tilts and curvatures \( \varepsilon_1 \) and \( \varepsilon_2 \), and \( 1/R_1 \) and \( 1/R_2 \) and the pole face gap \( 2G_0 \).

Figure 3.3 gives an overview of a typical bending magnet. Its primary purpose is to introduce energy dispersion in the electron beam. It does so by bending the electron beam over a large angle. The slower electrons (lower energy) will be bend more, the faster ones (higher energy) less. The bending magnet has a constant magnetic induction perpendicular to its pole faces:

\[
B_y = B_0 \\
B_x = B_z = 0
\]

The first-order matrix for a bending magnet with \( \varepsilon_1 = \varepsilon_2 = 0 \), bending radius \( \rho_0 \) and bending angle \( \alpha \) is given by:
\[
R = \begin{bmatrix}
\cos \alpha & \rho_0 \sin \alpha & 0 & 0 & 0 & \rho_0(1 - \cos \alpha) \\
-\frac{1}{\rho_0} \sin \alpha & \cos \alpha & 0 & 0 & 0 & \sin \alpha \\
0 & 0 & 1 & 0 & 0 & 0 \\
0 & 0 & 0 & 1 & 0 & 0 \\
-\sin \alpha & -\rho_0(1 - \cos \alpha) & 0 & 0 & 1 & \rho_0(\alpha - \sin \alpha) \\
0 & 0 & 0 & 0 & 0 & 1
\end{bmatrix}
\]

The magnetic induction is related to the bending radius via:

\[
B_0 \rho = \frac{p}{e} = 2\frac{m_0 c}{e} \sqrt{\epsilon(1 + \epsilon V)}
\]

where \(B_0\) is the constant magnetic induction well inside the magnet.

The bending magnet has a focusing action in the energy dispersive x-direction but no focusing action in the y-direction. If a focusing action is desired in both directions, the entrance and or exit faces can be rotated. The first-order matrix for an entrance or exit face rotated over an angle \(\varepsilon_i\) is:

\[
R = \begin{bmatrix}
1 & 0 & 0 & 0 & 0 & 0 \\
\frac{1}{\rho_0} \tan \varepsilon_i & 1 & 0 & 0 & 0 & 0 \\
0 & 0 & 1 & 0 & 0 & 0 \\
0 & 0 & -\frac{1}{\rho_0} \tan(\varepsilon_i - \psi) & 1 & 0 & 0 \\
0 & 0 & 0 & 1 & 0 & 0 \\
0 & 0 & 0 & 0 & 0 & 1
\end{bmatrix}
\]

This matrix includes a correction \(\psi\) to the focusing strength in the non-bend direction as a result of the fringing fields at the magnet's entrance and exit face. For an ideal magnet the magnetic field will drop abruptly to zero at the magnet boundary. For a realistic magnet this will not be the case and the field will fall to about 10% of the constant value inside the magnet over a distance approximately equal to the pole face gap, but a long tail will extend over a much larger distance. Magnetic clamps are often used to terminate the magnetic field at some distance \(G_0\) to \(2G_0\) from the magnet boundary. This improves the accuracy of simulations of the magnetic design, as well as make the optical properties less sensitive to distortions of the magnetic field by magnetic materials surrounding the magnet.
Transport's adjustment $\psi$ to the focusing strength in the non-bend plane is given by:

$$\psi = K_1 \left( \frac{G_0}{\rho_0} \right) \left( 1 + \sin^2 \varepsilon \right) \left( \frac{\cos \varepsilon}{\rho_0} \right) + \text{higher order terms in} \frac{g}{\rho_0}$$

3.36

Here the factor $K_1$ describes the effect of the particular clamp geometry. Typical values for $K_1$ range from 0.3 to 1.0 depending on the particular magnetic design.

3.4 Aberrations for imaging energy filters

3.4.1 Spectrum aberrations

As discussed previously (figures 2.2 and 2.5), the energy-loss spectrum at the energy-selecting slit is an energy dispersed image of the projector lens crossover and must have minimal aberrations to guarantee good energy resolution. The aberrations in the non-dispersion, $y$, direction do not affect the energy resolution and can be ignored. The size of the objective aperture in the spectrum (formula 2.2) overshadows any aberrations depending on the object coordinate $x_0$, which can therefore be neglected. This leaves only the aperture aberrations, axial chromatic aberrations and higher order dispersions. Up to third-order they are:

Geometric:

$$T_{122} x_0^2, T_{144} y_0^2$$ 3.37

$$U_{1224} x_0 y_0^2, U_{1244} x_0 y_0$$ 3.38

Chromatic:

$$T_{126} x_0 \frac{\Delta p}{p}, T_{166} \frac{\Delta p^2}{p}$$ 3.39

$$U_{1226} x_0^2 \frac{\Delta p}{p}, U_{1446} y_0^2 \frac{\Delta p}{p}, U_{1266} x_0 \frac{\Delta p^2}{p}, U_{1666} \frac{\Delta p^3}{p}$$ 3.40

The $T$ and $U$ coefficients follow from the optical transfer matrices from the projector lens crossover to the energy-loss spectrum at the energy-selecting slit. The maximum value for $x_0$ and $y_0$ is determined by the filter’s entrance aperture when the TEM is in the imaging mode, and by either the TEM objective aperture or the filter's entrance aperture if the TEM is in the diffraction mode.
3.4.2 Image aberrations

In the imaging mode, the post-column energy filter transfers the TEM image or diffraction pattern to the detector. The many aberrations introduced in the image by the magnetic prism must either be corrected or minimized to ensure good spatial resolution, even for large scattering acceptance angles and large energy windows.

To facilitate the calculation of the image aberrations, the regular Transport aberration coefficients must be further developed as follows:

The angular semi-divergence $\gamma$ in the TEM image is given by:

$$\gamma = \frac{\beta}{M_T}$$  \hspace{1cm} 3.41

where $M_T$ is the magnification from the specimen to the TEM image plane, and $\beta$ is the system’s electron scattering acceptance semi-angle as determined by the objective aperture. As each electron ray passing through a TEM image point $x_0, y_0$ must originate from the projector lens crossover, it can only have slopes $x_0', y_0'$ in the range:

$$x_0' = \frac{x_0}{L} \pm \frac{\beta_x}{M_T}$$  \hspace{1cm} 3.42

$$y_0' = \frac{y_0}{L} \pm \frac{\beta_y}{M_T}$$  \hspace{1cm} 3.43

where $L$ is the distance between the TEM image and projector lens crossover, and $\beta_x$ and $\beta_y$ are the objective aperture acceptance semi-angles in $x$ and $y$ respectively.

The overall second-order aberrations $\Delta x_1$ and $\Delta y_1$ can be determined for combinations of Transport coefficients. By substituting $x_0'$ and $y_0'$ by formulas 3.42 and 3.43 and sorting the various terms by their dependence on the object plane coordinates $x_0, y_0$, the scattering angles $\beta_x, \beta_y$ and the fractional momentum deviation $\Delta p/p$ we find:
**Geometric aberrations**

x-direction:

\[ T_{111}, \ T_{112}, \ T_{122} \]

\[ \Delta x_1 = T_{111} x_0^2 + T_{112} x_0 y_0 + T_{122} y_0^2 \]

\[ = T_{111} x_0^2 + T_{112} x_0 \left( \frac{x_0}{L} + \frac{\beta_x}{M_T} \right) + T_{122} \left( \frac{x_0}{L} + \frac{\beta_x}{M_T} \right)^2 \]

\[ = \left( T_{111} + \frac{T_{112}}{L} + \frac{T_{122}}{L^2} \right) x_0^2 + \left( T_{112} + 2 \frac{T_{122}}{L} \right) \frac{\beta_x}{M_T} x_0 + T_{122} \frac{\beta_x^2}{M_T^2} \]

\[ T_{133}, \ T_{134}, \ T_{144} \]

\[ \Delta x_1 = T_{133} y_0^2 + T_{134} x_0 y_0 + T_{144} y_0^2 \]

\[ = \left( T_{133} + \frac{T_{134}}{L} + \frac{T_{144}}{L^2} \right) y_0^2 + \left( T_{134} + 2 \frac{T_{144}}{L} \right) \frac{\beta_y}{M_T} y_0 + T_{144} \frac{\beta_y^2}{M_T^2} \]

y-direction:

\[ T_{313}, \ T_{314}, \ T_{323}, \ T_{324} \]

\[ \Delta y_1 = T_{313} x_0 y_0 + T_{323} x_0 y_0 + T_{314} x_0 y_0 + T_{324} x_0 y_0 \]

\[ = \left( T_{313} + \frac{T_{323}}{L} + \frac{T_{314}}{L} + \frac{T_{324}}{L^2} \right) x_0 y_0 \]

\[ + \left( T_{314} + \frac{T_{324}}{L} \right) \frac{\beta_y}{M_T} x_0 + \left( T_{323} + \frac{T_{324}}{L} \right) \frac{\beta_x}{M_T} y_0 \]

\[ + \frac{T_{324} \beta_x \beta_y}{M_T^2} \]

**Chromatic aberrations**

x-direction:

\[ T_{116}, \ T_{126}, \ T_{166} \]

\[ \Delta x_1 = T_{116} x_0 \frac{\Delta p}{p} + T_{126} x_0 \frac{\Delta p}{p} + T_{166} \left( \frac{\Delta p}{p} \right)^2 \]

\[ = \left( T_{116} + \frac{T_{126}}{L} \right) \frac{\Delta p}{p} + \frac{T_{126}}{L} \frac{\beta_x}{M_T} \frac{\Delta p}{p} + T_{166} \left( \frac{\Delta p}{p} \right)^2 \]

y-direction:

\[ T_{336}, \ T_{346} \]

\[ \Delta y_1 = T_{336} y_0 \frac{\Delta p}{p} + T_{346} y_0 \frac{\Delta p}{p} \]

\[ = \left( T_{336} + \frac{T_{346}}{L} \right) \frac{\Delta p}{p} + \frac{T_{346}}{L} \frac{\beta_y}{M_T} \frac{\Delta p}{p} \]
Similar results obtained for the third-order aberrations are shown in table 5.8. The above shows that only if the various Transport coefficients are combined can their actual effect on the image be revealed. For instance, the three combinations of \( T_{111}, T_{112} \) and \( T_{122} \) in equation 3.44, result in a distortion

\[
(T_{111} + \frac{T_{112}}{L} + \frac{T_{122}}{L^2}) x_0^2
\]

3.49

a field aberration

\[
(T_{112} + 2 \frac{T_{122}}{L}) \frac{\beta_x}{M_T} x_0
\]

3.50

and an aperture aberration

\[
T_{122} \frac{\beta_x}{M_T^2}
\]

3.51

This actually complicates the calculations as Transport does not allow constraint on combinations of coefficients, but only on individual coefficients. This makes it impossible to have Transport correct an effective aberration like the distortion of equation 3.49.

It is possible to look at these effective aberrations in a different way. Table 3.1 shows what happens to the regular first- and second-order matrix coefficients when the object plane is shifted from the TEM image plane to the projector lens crossover plane. The coefficients from the projector lens crossover plane are in **bold**.
Electron Optical Calculations

\[ R_{11} = R_{11} \]
\[ R_{12} = R_{11}L + R_{12} \]
\[ R_{16} = R_{16} \]
\[ R_{21} = R_{21} \]
\[ R_{22} = R_{21}L + R_{22} \]
\[ R_{33} = R_{33} \]
\[ R_{34} = R_{33}L + R_{34} \]
\[ R_{43} = R_{43} \]
\[ R_{44} = R_{43}L + R_{44} \]

\[ T_{111} = T_{111} \]
\[ T_{112} = 2T_{111}L + T_{112} \]
\[ T_{122} = T_{111}L^2 + T_{112}L + T_{122} \]
\[ T_{133} = T_{133} \]
\[ T_{134} = 2T_{133}L + T_{134} \]
\[ T_{144} = T_{133}L^2 + T_{134}L + T_{144} \]
\[ T_{116} = T_{116} \]
\[ T_{126} = T_{116}L + T_{126} \]
\[ T_{166} = T_{166} \]
\[ T_{313} = T_{313} \]
\[ T_{323} = T_{313}L + T_{323} \]
\[ T_{314} = T_{313}L + T_{314} \]
\[ T_{324} = T_{313}L^2 + T_{323}L \]
\[ + T_{314}L + T_{324} \]
\[ T_{336} = T_{336} \]
\[ T_{346} = T_{336}L + T_{346} \]
\[ T_{111} = T_{111} \]
\[ T_{112} = T_{112} - 2T_{111}L \]
\[ T_{122} = T_{111}L^2 - T_{112}L + T_{122} \]
\[ T_{133} = T_{133} \]
\[ T_{134} = T_{134} - 2T_{133}L \]
\[ T_{144} = T_{133}L^2 - T_{134}L + T_{144} \]
\[ T_{116} = T_{116} \]
\[ T_{126} = T_{116} - T_{116}L \]
\[ T_{166} = T_{166} \]

Table 3.1 Relation between the first- and second-order Transport coefficients as calculated from the projector lens crossover (bold) and from the TEM image plane.

Table 3.1 shows how a single coefficient of a system calculated from the projector lens crossover plane, is equivalent to a combination of coefficients when the same system is calculated from the TEM image plane. This greatly facilitates the calculations. For instance the distortion of equation 3.49 can now be corrected by calculating the system from the projector lens crossover and ensuring that \( T_{122} = 0 \). Table 3.2 shows all the effective second-order aberrations, calculated from the projector lens crossover and from the TEM image plane. Similarly results can be obtained for the effective third-order aberrations.
<table>
<thead>
<tr>
<th>From TEM image plane</th>
<th>From projector crossover plane</th>
<th>Character</th>
</tr>
</thead>
<tbody>
<tr>
<td>((T_{111} + \frac{T_{112}}{L} + \frac{T_{122}}{L^2})x_0^2)</td>
<td>(\frac{T_{122}}{L^2}x_0^2)</td>
<td>Distortion</td>
</tr>
<tr>
<td>((T_{112} + 2\frac{T_{122}}{L})\frac{\beta_x}{M_T}x_0)</td>
<td>((2\frac{T_{122}}{L} - T_{112})\frac{\beta_x}{M_T}x_0)</td>
<td>Inclination of image plane</td>
</tr>
<tr>
<td>(T_{122}\frac{\beta_x^2}{M_T^2})</td>
<td>((T_{111}L^2 - T_{112}L + T_{122})\frac{\beta_x^2}{M_T^2})</td>
<td>Second-order aperture aberration</td>
</tr>
<tr>
<td>((T_{133} + \frac{T_{134}}{L} + \frac{T_{144}}{L^2})y_0^2)</td>
<td>(\frac{T_{144}}{L^2}y_0^2)</td>
<td>Distortion</td>
</tr>
<tr>
<td>((T_{134} + 2\frac{T_{144}}{L})\frac{\beta_y}{M_T}y_0)</td>
<td>((2\frac{T_{144}}{L} - T_{134})\frac{\beta_y}{M_T}y_0)</td>
<td>Field astigmatism</td>
</tr>
<tr>
<td>(T_{144}\frac{\beta_y^2}{M_T^2})</td>
<td>((T_{133}L^2 - T_{134}L + T_{144})\frac{\beta_y^2}{M_T^2})</td>
<td>Second-order aperture aberration</td>
</tr>
<tr>
<td>((T_{313} + \frac{T_{323}}{L} + \frac{T_{314}}{L} + \frac{T_{324}}{L^2})x_0y_0)</td>
<td>(\frac{T_{324}}{L^2}x_0y_0)</td>
<td>Distortion</td>
</tr>
<tr>
<td>((T_{314} + \frac{T_{324}}{L})\frac{\beta_y}{M_T}x_0)</td>
<td>((\frac{T_{324}}{L} - T_{323})\frac{\beta_y}{M_T}x_0)</td>
<td>Field astigmatism</td>
</tr>
<tr>
<td>(T_{324}\frac{\beta_x\beta_y}{M_T^2})</td>
<td>(\frac{T_{324}}{L^2}\frac{T_{313}L^2 - T_{323}L}{-T_{314}L + T_{324}}\frac{\beta_x\beta_y}{M_T^2})</td>
<td>Second-order aperture aberration</td>
</tr>
<tr>
<td>((T_{116} + \frac{T_{126}}{L})x_0\frac{\Delta p}{p})</td>
<td>(\frac{T_{126}}{L}x_0\frac{\Delta p}{p})</td>
<td>Energy dependent magnification</td>
</tr>
<tr>
<td>(T_{126}\frac{\beta_x\Delta p}{M_T p})</td>
<td>((T_{126} - T_{116}L)\frac{\beta_x\Delta p}{M_T p})</td>
<td>Axial chromatic aberration</td>
</tr>
<tr>
<td>(T_{166}\frac{\Delta p^2}{p})</td>
<td>(T_{166}\frac{\Delta p^2}{p})</td>
<td>Second-order dispersion</td>
</tr>
<tr>
<td>((T_{336} + \frac{T_{346}}{L})y_0\frac{\Delta p}{p})</td>
<td>(\frac{T_{346}}{L}y_0\frac{\Delta p}{p})</td>
<td>Energy dependent magnification</td>
</tr>
<tr>
<td>(T_{346}\frac{\beta_y\Delta p}{M_T p})</td>
<td>((T_{346} - T_{336}L)\frac{\beta_y\Delta p}{M_T p})</td>
<td>Axial chromatic aberration</td>
</tr>
</tbody>
</table>

Table 3.2 The effective second-order aberrations calculated from the filter's object plane as well as the projector lens crossover plane.
4 Design Specifications

The high voltage post-column imaging filter was designed for use with the JEOL ARM1000 and ARM1250 high voltage transmission electron microscopes. These microscopes operate at 1.0 and 1.25 MeV respectively.

Prior to the design the following specifications were drawn up.

4.1 Boundary conditions

As with any retrofit design one has to work within certain boundary conditions.

- Distance between projector lens crossover and the effective entrance face of the magnetic prism
  
  $1.175 \text{ m}$

  The JEOL ARM microscopes have a pit in the floor behind the microscope for the filter to fit in. This allows the x-rays coming from the filter to be shielded by covering the pit with lead blocks. In order for the lens sections of the filter to sit in the pit, the effective entrance face of the magnetic prism is located 1.175 m from the projector lens crossover.

- System length
  
  $\leq 1.5 \text{ m}$

  The system length as measured perpendicular to the optical axis of the TEM is limited by the length of the pit in the floor.

- Detectors

  Primary detector: 1024 x 1024 pixel High Voltage Slow-Scan CCD camera with 24 $\mu$m square pixels(57).

  Secondary detector: pneumatically retractable TV rate CCD camera located in front of the Slow-Scan camera.

- Entrance apertures

  A small entrance aperture will be provided for selected area spectroscopy.

  A 25 hole reference mask will be provided for assessing the optical performance.

4.2 Performance specifications

4.2.1 Electron spectroscopic imaging

- Geometric distortions

  The total of all geometric distortions as well as uncorrectable misalignments to result in less than 5% distortion (the distortion is given by the ratio of an image point’s displacement from the aberration free position and the distance from the optic axis times 100%).
• Geometric blur
  The total geometric blur to be less than 1 pixel (24 μm).

• Chromatic aberrations
  The total of all chromatic aberrations as well as uncorrectable misalignments to result in less than 2 pixels blur at the edges of the image field for an energy-window of 50 eV at 1.25 MeV.

A large energy-window will allow more core-loss signal to be collected. This is especially important at the higher energy-losses where the signal level is extremely low. A large energy-window will however also result in a loss of spatial resolution due to the chromatic aberration of the objective lens. 50 eV is taken to be the largest window one will typically use for energy-filtered imaging.

• Scattering acceptance semi-angle
  10 mrad

Chapter 2 showed that the largest useful scattering angle for elastic imaging is about 7.5 mrad. For inelastic imaging the scattering angles may actually be larger especially for high energy losses. It was shown that most scattering is contained within a 10 mrad semi-angle.

• Non-isochromaticity
  ~2 eV.

The non-isochromacity does not need to be much smaller than the energy resolution (see below) as reducing the non-isochromacity any further will not lead to a significant reduction of the energy resolution in the image.

• Energy dispersion at the energy-selecting slit
  ≥ 2.5 μm/eV at 1.25 MeV

The energy-dispersion at the energy-selecting slit needs to be large enough such that the roughness of the slit edges does not affect the accuracy of the energy-selection or causes streaks in the energy-filtered images. Previous experience has proven a dispersion >2.5 μm/eV to be adequate. Because the energy dispersion is inversely proportional to the primary energy, the dispersion at lower primary energies is actually higher.

4.2.2 Electron energy-loss spectroscopy

• Energy-resolution
  ≤3 eV full width at half maximum at 1.25 MeV.

This includes the contribution of all aberrations as well as the stabilities of all the important power supplies.

• Aberrations
  Spectrum to be free of second-order aperture aberrations and axial chromatic aberration in the energy-dispersive direction.
Design Specifications

- Range of energy dispersions at the detector
  1.0 eV to 0.05 eV/ pixel
- Spectrum width at the detector
  150 pixels wide.

This represents a compromise between dynamic range and readout speed. By spreading the spectrum over more pixels, the dynamic range of the detection is increased, but the readout speed is decreased because more pixels need to be read out.

4.3 Component specifications

Prior to starting the electron optical design process, various component specifications were drawn up:

- Bending magnet

Gatan has extensive experience with the 10 cm radius bending magnet used in its Serial and Parallel EELS spectrometers and intermediate voltage imaging energy filters. The 10 cm prism is used up to 400 keV and yields a dispersion of 1.8 μm/eV at 100 keV. For the HV filter it was decided to use a 25 cm bending magnet for mainly two reasons:

1. In order to guarantee a good agreement between calculations and reality, it is essential to prevent any magnetic saturation in the bending magnet. It is known that the 10 cm bending magnets do not show saturation below 400 keV. The momentum of 1.25 MeV electrons is about 2.25 times that of 400 keV electrons, so by increasing the bending radius to 25 cm a similar magnetic induction can be used as with the 10 cm bending magnet at 400 keV and no magnetic saturation should occur. Further this offered the benefit that the existing current supply could be used.

2. The dispersion from the bending magnet decreases with primary energy. As a result, a 10 cm bending magnet used at 1.25 MeV has a dispersion of only 0.2 μm/eV and a magnification of about 12.5 times is necessary to ensure the required dispersion of 2.5 μm/eV at the energy-selecting slit. Because the dispersion is directly proportional to the effective length of the prism, a 25 cm magnet there instead, will give a dispersion of about 0.5 μm/eV needing a further magnification of only 5 times.

- Quadrupoles and sextupoles

The quadrupoles will have cylindrical pole tips to reduce the magnitude of higher order aberrations. The radius will be determined using equation 4.27.

The pole tips should be kept as close to the optic axis as possible to maximize the strength. Given the vacuum considerations, the pole tips will be on a 12.5 mm diameter circle.

The maximum tip induction allowed in the calculations will be 2.5 kGauss. Experiments have shown signs of saturation with pole tip inductions over 2.5 kGauss.
- Magnetic shielding
  As a consequence of the magnification between the entrance aperture plane and the Slow-Scan camera, the size of the 24 μm CCD pixels referred back to the viewing screen will be just a few μm. An image movement of only one μm amplitude induced by stray AC magnetic fields penetrating into the viewing chamber will therefore cause a noticeable deterioration in the resolution of the final image, or a reduction of the effective number of pixels. As a result the minimum overall magnification necessary to resolve a certain spatial resolution will increase and the field of view will correspondingly decrease. This makes it essential that the HV GIF as well as the TEM viewing and camera chambers will be well shielded against such stray fields.

- Vacuum
  The filter will be pumped by the TEM vacuum pumps through the viewing and camera chamber. In order not to limit the pumping speed at the slow-scan camera too much, the drift tubes will have an internal diameter of 10 mm.

- Water cooling
  Water cooling will be added to remove the heat generated by the various lens elements as well as the Peltier cooler used in the slow-scan camera. The water will be supplied at room temperature by one of the TEM's water chillers.
5  The Electron Optical Design

5.1 The design process

Referring back to figure 2.4, the post-column filter has two modes of operation, 1. the Imaging Mode, and 2. the Spectroscopy Mode. In either mode, the filter transfers the projector lens crossover into an energy-loss spectrum at the energy-selecting slit. In the Imaging Mode it simultaneously transfers the TEM image or diffraction pattern into an energy-filtered image or diffraction pattern at the detector. Alternatively, in the Spectroscopy Mode operation, the filter transfers the energy-loss spectrum at the energy-selecting slit to the detector with variable magnification.

In total it therefore takes three sets of matrices to describe the operation of the filter:

1. the matrices describing the transfer of the projector lens crossover into the energy-loss spectrum at the energy-selecting slit,
2. the matrices for the Imaging Mode, describing the transfer from the TEM image plane in the viewing chamber to the CCD detector, and
3. the matrices for the Spectroscopy Mode, describing the transfer of the energy-loss spectrum at the slit to the CCD detector.

Only the first two matrices will be described here.

As mentioned before, the filter can be split into pre- and post-slit optics. The purpose of the pre-slit optics is to create the energy-loss spectrum at the energy-selecting slit, the purpose of the post-slit optics is to transfer the energy-filtered image or diffraction pattern, or alternatively the energy-loss spectrum to the detector.

It is possible to calculate the pre- and post-slit optics separately. In order to do so, the constraints on the various important matrix coefficients need to be established.

5.2 The matrix coefficients

5.2.1 Projector crossover to energy-selecting slit

The projector lens crossover must be focused onto the energy-selecting slit in both the x- and y-direction. It is useful to have a double focus (x and y) as this will lower the demands on the quality of the slit's knife edges. Similarly, as previously determined, the dispersion should be at least 2.5 μm/eV at 1.25 MeV to further reduce the quality demands. Using formula 3.18 and the appendix this means that \( R_{16} \geq 4 \).

To ensure good energy resolution, the spectrum should have minimal aberrations. Referring back to section 3.4.1, and neglecting those aberrations that depend on the object coordinates, there are 4 second-order and 6 third-order coefficients of concern:

\[
T_{122}, T_{144}, T_{126}, T_{166} \\
U_{1222}, U_{1244}, U_{1226}, U_{1446}, U_{1266}, U_{1666}
\]

Since the design of the early serial electron energy-loss spectrometers it has been known that good energy-resolution for reasonably large acceptance angles, can be
obtained by correcting $T_{122}$, $T_{144}$, $T_{126}$(9). The magnitude of the second-order dispersion and third-order aberrations are then on the order of the energy spread from the filament. These three second-order aberrations can be corrected simultaneously by an appropriate choice of the first-order trajectories through the prism and by inclining and curving the prism’s entrance and exit faces.

This leads to the following first- and second order constraints:

\[
\begin{align*}
R_{12} &= R_{34} = 0 \\
R_{16} &\geq 4 \\
T_{122} &= T_{144} = T_{126} = 0
\end{align*}
\]

5.2

5.2.2 TEM image to detector.

The filter must provide achromatic and stigmatic imaging, with equal magnifications for the x- and y-direction, imposing 5 first-order constraints:

\[
\begin{align*}
R_{12} &= R_{34} = 0, \\
R_{11} &= \pm R_{33} = M \\
R_{16} &= 0
\end{align*}
\]

5.3

Note that if the imaging is achromatic, the image will automatically be in focus in the dispersion (x) direction, so really there are only four first-order constraints. The ± sign indicates that it does not really matter if the image is flipped around in the x- or the y-direction. The filter uses a digital camera and the image can easily be flipped back electronically if desired.

Ideally the image will be entirely free of any aberrations. This imposes 15 second-order (table 3.2) and 35 third-order constraints (table 5.8). For the case of a post-column filter this would require a lens system with many quadrupoles, sextupoles and octupoles. Clearly such a system is too complex to operate and for a practical imaging filter it is important to see how the number of optical elements can be minimized.

To start, the aberrations need not all be individually zero. More important the total of all aberrations must be small compared to the pixel size of the detector. Only the larger aberrations therefore need to be considered and possibly corrected. A great simplification is obtained by neglecting all aperture and field aberrations (effectively all aberrations depending on β). This seems a reasonable assumption for large TEM magnifications where the angular divergence in the TEM image is extremely small, but will also prove to be valid at low magnifications.

The remaining 6 second-order and 10 third-order distortions, energy dependent magnifications and higher order dispersions can now be calculated from the projector lens crossover as according to table 3.2 each of these effective aberrations is entirely described by just one aberration coefficient:

\[
\begin{align*}
T_{122}, T_{144}, T_{324} \\
T_{126}, T_{346}, T_{166} \\
U_{1222}, U_{1244}, U_{3444}, U_{3224} \\
U_{1226}, U_{1266}, U_{1666}, U_{1446} \\
U_{3246}, U_{3466}
\end{align*}
\]

5.4
5.3 The design of the pre-slit optics

A 25 cm radius bending magnet used at 1250 keV yields an energy dispersion of about 0.5 μm/eV. In order to reach the target dispersion of 2.5 μm/eV at the energy-selecting slit, a magnification of about 5 times is needed. To keep the filter as simple as possible only one quadrupole is used. To allow double focusing of the energy-loss spectrum at the energy selecting slit, the quadrupole is placed between the prism’s x- and y-crossovers. The matrix representation for the energy-dispersive stage calculated from the projector lens crossover plane to the energy-selecting slit is then given by:

\[ [X] = [R][X_0] \]

with

\[ [R] = [D_3][Q_1][D_2][PRISM][D_1] \]

where
- \( D_i \) = drift space matrix
- Prism = prism matrix
- \( Q_i \) = quadrupole matrix

The elements in formula 5.6 that are indicated in bold are varied to meet the constraints of formula 5.2.

First for a range of prism entrance and exit face rotations, the location of the prism’s x- and y-crossovers were determined by varying the drift space behind the prism to get \( R_{12}=0 \) and \( R_{34}=0 \) respectively. Next, calculating from the projector lens crossover to the x-crossover plane, the necessary entrance and exit face curvatures were calculated for \( T_{122}=T_{144}=0 \). Figures 5.1 and 5.2 show the results and the value of \( T_{126} \) in each case. Figure 5.3 shows that the aberrations can only be corrected with a convex entrance and a concave exit face.

In order to leave adequate room for the quadrupole, the x- and y-crossovers are separated by 6 cm. To allow the quadrupole to be located outside the frame supporting the prism, the crossovers must lie at least 10 cm away past the exit face of the prism. The exit face angles therefore have to lie in the 40°-60° range (figure 5.1).
Figure 5.1 Location of the x-crossover with respect to the effective exit face (x -10°, 15°, 20°), as well as the distance of the y-past the x-crossover (xy - 10°, 15°, 20°) for various entrance and exit face angles.

Figure 5.2 Value of $T_{126}$ for various entrance and exit face angles. $T_{122} = T_{144} = 0$ by curving the entrance and exit faces of the prism.
Figure 5.3 Entrance (1/R1) and exit face (-1/R2) curvatures required for $T_{122}=T_{144}=0$ for various entrance and exit face angles.

From figures 5.1 through 5.3 various prism designs can be chosen, each of which will yield the desired first and second-order properties. To keep the system as short as possible while providing 5x magnification in the x-direction, the quadrupole will be diverging in the x-direction, implying a real y-crossover closer to the prism and a virtual x-crossover 6 cm further away from the prism. The final design parameters are summarized in table 5.1, the axial trajectories (beam envelope) are displayed in figure 5.4.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Prism entrance face angle</td>
<td>14.8°</td>
</tr>
<tr>
<td>Prism exit face angle</td>
<td>50.0°</td>
</tr>
<tr>
<td>Prism entrance face curvature</td>
<td>4.477 m⁻¹</td>
</tr>
<tr>
<td>Prism exit face curvature</td>
<td>-7.826 m⁻¹</td>
</tr>
<tr>
<td>X-crossover location</td>
<td>0.276 m</td>
</tr>
<tr>
<td>Y-crossover location</td>
<td>0.216 m</td>
</tr>
<tr>
<td>Dispersion at x-crossover</td>
<td>0.64 μm/eV</td>
</tr>
<tr>
<td>Quadrupole location</td>
<td>0.260 m</td>
</tr>
<tr>
<td>Energy-selecting slit location</td>
<td>0.333 m</td>
</tr>
<tr>
<td>Dispersion at energy-selecting slit</td>
<td>2.62 μm/eV</td>
</tr>
</tbody>
</table>

Table 5.1 Design and electron optical properties of the energy dispersive stage. Distances listed are from the effective exit face of the prism. Primary energy 1.25 MeV.
Figure 5.4 Axial rays (beam envelope) from the projector lens crossover to the energy-loss spectrum at the energy-selecting slit. The x-trajectory through the prism is only shown schematically.
Table 5.2 lists all relevant coefficients for the transfer matrices from the projector lens crossover to the energy-selecting slit.

<table>
<thead>
<tr>
<th>1st order</th>
<th>2nd order</th>
<th>3rd order</th>
</tr>
</thead>
<tbody>
<tr>
<td>$R_{11} = -2.43$</td>
<td>$T_{111} = 7.77E0$</td>
<td>$U_{1111} = -1.47E2$</td>
</tr>
<tr>
<td>$R_{12} = 0.00$</td>
<td>$T_{112} = 1.43E1$</td>
<td>$U_{1112} = -4.27E2$</td>
</tr>
<tr>
<td>$R_{21} = -27.59$</td>
<td>$T_{122} = 0.00$</td>
<td>$U_{1122} = -3.98E2$</td>
</tr>
<tr>
<td>$R_{22} = -0.41$</td>
<td>$T_{133} = -2.50E1$</td>
<td>$U_{1222} = -1.94E2$</td>
</tr>
<tr>
<td>$R_{16} = 4.24$</td>
<td>$T_{134} = -3.53E1$</td>
<td>$U_{1133} = -1.67E2$</td>
</tr>
<tr>
<td>$R_{26} = 47.72$</td>
<td>$T_{144} = 0.00$</td>
<td>$U_{1233} = -7.37E2$</td>
</tr>
<tr>
<td>$R_{33} = 0.45$</td>
<td>$T_{116} = 1.17E1$</td>
<td>$U_{1134} = -1.11E3$</td>
</tr>
<tr>
<td>$R_{34} = 0.00$</td>
<td>$T_{126} = 0.00$</td>
<td>$U_{1234} = -2.03E3$</td>
</tr>
<tr>
<td>$R_{43} = 10.62$</td>
<td>$T_{166} = -1.53E1$</td>
<td>$U_{1144} = -9.33E2$</td>
</tr>
<tr>
<td>$R_{44} = 2.22$</td>
<td></td>
<td>$U_{1244} = -1.34E3$</td>
</tr>
</tbody>
</table>

Table 5.2 Matrix coefficients for the various transfer matrices from the projector lens crossover to the energy-selecting slit.

5.4 Evaluation of the pre-slit optics

5.4.1 The aberration figure

As a result of the prism’s aperture aberrations, the different rays coming from the projector will yield slightly shifted energy-loss spectra at the energy-selecting slit. The surface described by the shifts is referred to as the aberration figure and is described by the aperture aberrations $U_{1222}$ and $U_{1244}$:

$$ U_{1222}x_0^3 + U_{1244}x_0^2y_0 $$

5.7

The height of the aberration figure can be minimized by defocusing the spectrum at the energy selecting slit. In this case an $R_{12}$ term is added to formula 5.7:

$$ R_{12}x_0 + U_{1222}x_0^3 + U_{1244}x_0^2y_0 $$

5.8
It is useful to determine the height of the aberration figure corresponding to the image on the CCD. It can be derived that the height $d_{ab}$ in eV is minimized for:

$$R_{12} = -(U_{1222} + \frac{1}{2} U_{1244}) \left( \frac{r_{ap}}{L} \right)^2$$

and equals:

$$d_{ab} = \frac{1}{4} \sqrt{2} \frac{U_{1244}}{D} \left( \frac{r_{ap}}{L} \right)^3$$

$r_{ap}$ is the radius of the entrance aperture in the filter's object plane whose image fits just around the square detector. $L$ is the distance from the object plane to the projector lens crossover.

### 5.4.2 The transmissivity

The transmissivity of the system depends on the desired non-isochromaticity $\Delta E$. A larger non-isochromaticity will allow a higher transmissivity. $\Delta E$ follows from the sum of the contributions of the projector lens crossover ($d_{ab}$) and the aberration figure ($d_{ab}$):

$$\Delta E = d_{\beta} + d_{ab}$$

Here $d_{\beta}$ is given by formula 2.2. Combining formulas 2.2, 5.10 and 5.11 it follows:

$$\frac{\beta}{M_T} = \frac{D}{2M_s} \frac{1}{L} \left( \Delta E - \frac{1}{4} \sqrt{2} \frac{U_{1244}}{D} \left( \frac{r_{ap}}{L} \right)^3 \right)$$

By rewriting formula 2.4 for the transmissivity as

$$T = \left( \frac{\pi \beta r_{ap}}{M_T} \right)^2 = \pi^2 \left( \frac{\beta}{M_T} \right)^2 r_{ap}$$

and combining formulas 5.12 and 5.13 we find:

$$T = \left( \frac{\pi D}{2M_s} \right)^2 \left( \frac{r_{ap}}{L} \right)^2 \left( \Delta E - \frac{1}{4} \sqrt{2} \frac{U_{1244}}{D} \left( \frac{r_{ap}}{L} \right)^3 \right)^2$$

Figure 5.5 plots the transmissivity as a function of the entrance aperture angle ($r_{ap}/L$) for various energy resolutions.
Figure 5.5 Transmissivity of the HV post-column filter as a function of the entrance aperture angle $r_{ap}/L$ for various energy resolutions.

Formula 5.14 and figure 5.5 show that for small apertures the transmissivity is proportional to the aperture size. When the aperture aberrations start dominating the energy resolution, a further increase of the entrance aperture size will actually decrease the transmissivity.

For each non-isochromaticity there exists therefore an optimum size of the entrance aperture, or equivalently an optimum intermediate magnification $M_I$. Table 5.3 shows the optimum transmissivity and optimum entrance aperture angle for various operating energies and non-isochromaticities. Note that thanks to the much higher dispersion, the optimum transmissivity is some 300 x larger at 100 keV than at 1250 keV operation.

<table>
<thead>
<tr>
<th>$\Delta E$ (eV)</th>
<th>D ($\mu$m/eV)</th>
<th>$r_{ap}/L$ (mrad)</th>
<th>$T$ ($\mu$m$^2$)</th>
<th>$r_{ap}/L$ (mrad)</th>
<th>$T$ ($\mu$m$^2$)</th>
<th>$r_{ap}/L$ (mrad)</th>
<th>$T$ ($\mu$m$^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.0</td>
<td>22.9</td>
<td>2.3</td>
<td>6.4E-4</td>
<td>1.5</td>
<td>2.4E-5</td>
<td>1.1</td>
<td>2.0E-6</td>
</tr>
<tr>
<td>2.0</td>
<td>6.64</td>
<td>2.9</td>
<td>4.1E-3</td>
<td>1.9</td>
<td>1.5E-4</td>
<td>1.4</td>
<td>1.3E-5</td>
</tr>
<tr>
<td>3.0</td>
<td>2.62</td>
<td>3.3</td>
<td>1.2E-2</td>
<td>2.2</td>
<td>4.5E-4</td>
<td>2.6</td>
<td>3.8E-5</td>
</tr>
</tbody>
</table>

Table 5.3 Optimum transmissivity and entrance aperture angle for operation at 100, 400 and 1250 keV.

When choosing the desired value of the non-isochromaticity the energy spread from the gun must be taken into account. It does not make sense to make the non-isochromaticity smaller than the energy spread from the gun as this will not improve the overall energy resolution significantly. Also the experiment of interest may affect the choice of the non-isochromaticity. An experiment involving the mapping of the fine structure of an element's edge will require a small non-isochromaticity.
Elemental mapping using large energy windows to maximize the signal there instead can tolerate a rather larger value for the non-isochromaticity.

In this particular design, the non-isochromaticity has been set to 2.0 eV. This dictates a 1.4 mrad entrance aperture angle and results in a transmissivity of $1.3 \times 10^{-5}$ μm at 1250 keV.

The transmissivity is mainly limited by the large distance $L$ between the projector lens crossover and the filter's object plane ($L=1.383$ m, see section 6.5), and the rather low dispersion of the magnetic prism of 0.64 μm/eV at 1.25 MeV. The distance $L$ is mainly determined by the mechanical design of the microscope and can not be decreased much. The dispersion can not be increased much further either as the prism of 25 cm bending radius is about the largest that will fit underneath the ARM1250.

The only way to further increase the transmissivity is by correcting the third-order spectrum aberrations at the energy-selecting slit. This will then allow a larger entrance aperture and a higher intermediate magnification. For a design that is free of spectrum aberrations, formula 5.14 reduces to:

$$T = \left( \frac{\pi D}{2M_s} \right)^2 \left( \frac{r_{op}}{L} \right)^2 \Delta E^2$$

5.15

Formula 5.15 seems to indicate that the entrance aperture angle can be chosen as large as possible. However, for larger entrance apertures the quality of the image will degrade as a result of the image aberrations (see sections 5.5 & 5.6). For the earlier 1.4 mrad entrance aperture angle, formula 6.15 results in a transmissivity of $5.6 \times 10^{-6}$ μm at 1250 keV.

### 5.4.3 Chromatic aberrations

Table 5.4 shows the calculated chromatic aberrations in the energy-loss spectrum for both a 50 and a 1000 eV energy window at 1.25 MeV. The 50 eV window is the largest used for energy-filtered imaging, the 1000 eV window represents the largest energy-loss spectrum that can be recorded in the spectroscopy mode. All aberrations should be evaluated with respect to the energy-dispersion at the energy-selecting slit, 2.62 μm/eV at 1.25 MeV. As can be seen all aberrations are negligible except for $T_{166}$ which causes a 2.3 eV (5.9/2.62) non-linearity over a 1000 eV field of view.

<table>
<thead>
<tr>
<th>Coefficient</th>
<th>Energy window</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>50 eV</td>
</tr>
<tr>
<td>$T_{166}$</td>
<td>1.5E-02</td>
</tr>
<tr>
<td>$U_{1226}$</td>
<td>2.6E-03</td>
</tr>
<tr>
<td>$U_{1446}$</td>
<td>9.7E-04</td>
</tr>
<tr>
<td>$U_{1266}$</td>
<td>8.5E-04</td>
</tr>
<tr>
<td>$U_{1666}$</td>
<td>3.5E-06</td>
</tr>
</tbody>
</table>

Table 5.4 Blurring ($U_{1226}, U_{1446}, U_{1266}$) and non-linearity ($T_{166}, U_{1666}$) in μm as a result of the various second- and third-order chromatic aberrations for a 50 and a 1000 eV window at 1.25 MeV primary energy.
5.5 The design of the post-slit optics

The post-slit optics consists of four quadrupoles to control the effectively four imaging constraints. Although in principle fewer quadrupoles can be used to establish the four constraints, in the alignment of the filter there must be four independent and easily adjustable parameters to assure the 4 imaging constraints can actually be met.

The post-slit optics is calculated from the projector lens crossover. The transfer matrix representation from the projector lens crossover plane to the detector plane is given by:

\[ [X_1] = [R] [X_0] \]

\[ [R] = \begin{bmatrix} D_8 \| Q_5 \| D_7 \| Q_4 \| D_6 \| Q_3 \| D_5 \| Q_2 \| D_4 \| Q_1 \| D_3 \| PRISM \| D_1 \end{bmatrix} \]

The four first-order imaging constraints for calculation from the projector lens crossover follow from equations 6.3 in combination with table 3.1:

\[ R_{16} = 0 \]
\[ R_{12} = \pm LM \]
\[ R_{34} = \pm LM \]
\[ R_{33} = \pm M \]

The ± indicates again that it is allowed for the image to be flipped in the x- and/or the y-direction.

The TEM image plane that serves as the object plane for the filter is conjugate to the virtual achromatic image plane of the pre-slit optics (figure 3.4). The location of the achromatic plane and its conjugate TEM image pane can be found by adjusting the lengths of drift space \( D_2 \) in:

\[ [X_1] = [R] [X_0] \]

\[ [R] = [D_2] [PRISM] [D_1] \]

until \( R_{16} = 0 \). This lead to a negative value for \( D_2 \) and \( L = 1.383 \) m, indicating that the filter’s object plane actually lies inside the magnetic prism (the prism’s entrance face is located 1.175 m from the projector lens crossover).

From section 5.4.2 it followed that the optimum entrance aperture angle for a non-isochromaticity of 1 eV is 1.4 mrad. This is implemented by placing a 3 mm diameter aperture 1.075 m away from the projector lens crossover. The magnification from the aperture to the CCD is chosen to be 12.5x such that the image of the aperture fits well around the 25 mm square CCD. This results in \( M = 9.72 \) (12.5 \( *1.075/1.383 \)) for the magnification from the object plane, and in \( R_{11}L = R_{12} = 13.44 \).

Finally the first-order constraints for the matrix from the projector lens crossover are:

\[ R_{16} = 0 \]
\[ R_{12} = \pm 13.44 \]
\[ R_{34} = \pm 13.44 \]
\[ R_{33} = \pm 9.72 \]
Understandably many systems can be designed that will satisfy these first-order constraints. Not each one can actually be implemented, however, given the physical limitations. Nor will each first-order solution yield acceptable higher order properties. Each first-order design therefore needs to be examined for its second- and third-order properties. If necessary, sextupoles and possibly even octupoles can be added in the drift spaces to control any large aberrations.

In the ideal design, each sextupole will have an affect on only one second-order coefficient and the elements are said to be orthogonal. Typically, though, a sextupole will have an effect on various coefficients. By carefully choosing the first-order solution the orthogonality of the sextupoles can be maximized. The introduction of highly stigmatic trajectories, intermediate images, beam crossovers, as well as areas of high and low dispersion will allow greater orthogonality. For instance, sextupoles that should only affect an aberration dependent on the y coordinate are then placed at x-crossovers. Sextupoles that should affect the chromatic aberrations are placed in areas where the energy dispersion is high, whereas those that should mainly affect the geometric aberrations are placed in areas where the energy dispersion is low.

The imaging stage is designed with several considerations in mind:

1. Keep the beam envelope as narrow as possible to minimize the introduction of higher order aberrations. This includes aberrations introduced by the fringing fields of lens elements, as well as those due to any misalignments or from mechanical or magnetic imperfections of the pole tips.

2. Reduce the energy dispersion immediately following the energy-selecting slit to avoid the introduction of chromatic aberrations. The first quadrupole lens following the energy-selecting slit must therefore be converging in the dispersive (x) direction. As this will increase the width of the beam envelope in the y-direction, the next quadrupole should be converging in the y-direction.

3. The last quadrupole should act as a projector type lens and transfer the final intermediate x and y images to the detector. This requires the last quadrupole to be located between the final intermediate x and y images.

4. The requirement for the beam envelope to be as narrow as possible throughout the system demands a rather large magnification from the final quadrupole. A sizable drift space must therefore be added to limit this quadrupole's excitation.

It should be noted that the design of the pre-slit optics is likely to greatly affect the design of the post-slit optics. This has not been explored in this research. Three different solutions using the above principles have been explored for the post-slit optics:

The first solution uses only three quadrupoles, although if actually implement a fourth, fine tuning quadrupole, would be added to allow control of all four first-order imaging properties. The first two quadrupoles are converging and diverging in the dispersion direction respectively as discussed above. The projector quadrupole is converging in the dispersion direction, implying that there is a real intermediate image in the x-direction and a reversal of the energy dispersion. This solution allows upright imaging for both the x- and the y-direction \((R_{11} = R_{33} = +9.72)\) and has a very small beam envelope. Similar to the other solutions discussed here, this
system offers good second- and third-order properties. Only some minor distortions will be noticeable. The main disadvantage of this solution is its excessive overall length (>1.5 m) preventing it from fitting in the available space. The long length is mainly the result of the rather long drift space (0.4 m) between Q4 and Q5 which is necessary to allow stigmatic imaging. Although this drift space can be shortened by locating Q3 as close to the energy-selecting slit as possible, this significantly complicates the mechanical design of the energy-selecting slit. The system can also be shortened by increasing the strength of the projector quadrupole, either through a reduction of the internal diameter at the pole tips, or by increasing the excitation. Unfortunately neither method leads to a design that adheres to the boundary conditions and component specifications defined in chapter 4.

The second solution is very similar to the first one, but uses a projector quadrupole that is diverging rather than converging in the dispersion direction. Here there is no intermediate x image and the energy dispersion decays monotonously to zero at the detector. This and an additional intermediate image in the y image, results in reversed imaging for both x and y \( (R_{11} = R_{33} = -9.72) \). For this solution to work at all, the second diverging quadrupole must be located immediately following the first one. Furthermore, this solution requires either a very long drift space between the second and the projector quadrupole or a very long drift space to the detector. In either case the total system length significantly exceeds the maximum of 1.5 m.

The third solution was actually build and employs four quadrupoles. The added flexibility of the fourth quadrupole allows the overall system length to be reduced to 1.4 m while allowing stigmatic imaging with reasonable excitations of the various quadrupoles. As this solution introduces an intermediate x- and y image, it results in upright imaging for \( x \) and reversed imaging for \( y \) \( (R_{11} = +9.72, \ R_{33} = -9.72, \) figure 5.6). The narrow width of the beam envelope of about 2 mm throughout the system (see figure 6.7) helps avoid the introduction of higher order aberrations from imperfections and fringing fields of the various quadrupoles. The second- and third-order properties are very good (see next section on the evaluation of the aberrations).

The important \( R \) matrix elements calculated from the TEM image plane are:

<table>
<thead>
<tr>
<th>Coefficient</th>
<th>Value</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>R11</td>
<td>9.72</td>
<td>m</td>
</tr>
<tr>
<td>R12</td>
<td>0.00</td>
<td>m</td>
</tr>
<tr>
<td>R16</td>
<td>0.00</td>
<td>m</td>
</tr>
<tr>
<td>R21</td>
<td>22.29</td>
<td></td>
</tr>
<tr>
<td>R22</td>
<td>0.10</td>
<td>m</td>
</tr>
<tr>
<td>R33</td>
<td>-9.72</td>
<td>m</td>
</tr>
<tr>
<td>R34</td>
<td>0.00</td>
<td>m</td>
</tr>
<tr>
<td>R43</td>
<td>-23.25</td>
<td>m</td>
</tr>
<tr>
<td>R44</td>
<td>-0.10</td>
<td>m</td>
</tr>
</tbody>
</table>

Table 5.4 First-order properties for the imaging mode calculated from the filter's object plane to the camera plane.
Fig. 5.6 Axial rays from the TEM image plane which is located about 20 cm after the entrance face of the bending magnet. Necessarily where the energy-dispersion reverses polarity there is an intermediate image for the x-direction.

Figure 5.7 Beam envelope and the energy-dispersion throughout the system. As can be seen the beam is about 4 mm wide through the bending magnet after which it reduces to less than 2.0 mm until it increases rapidly after the final quadrupole which functions as a projector lens. The energy dispersion increases from 2.62 μm/eV at the slit to about 4 μm/eV at the first postslit quadrupole where it is then reduced. Note that the dispersion actually reverses polarity just before Q5 and increases to 0.5 μm/eV before it finally decays to zero at the detector.
5.6 Evaluation of the image aberrations

Jumping a little ahead of the discussion, two versions of the filter have been analyzed:

1. The above design without any correction.
2. The above design corrected for the second-order geometric distortions $T_{122}$, $T_{144}$, $T_{324}$, through the addition of three sextupoles as displayed in figures 5.6 and 5.7.

Table 5.5 lists for both cases the values for the various aberration coefficients calculated from the TEM image plane.

<table>
<thead>
<tr>
<th>Coefficient</th>
<th>Uncorrected</th>
<th>Corrected</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>$T_{111}$</td>
<td>4.66E+00</td>
<td>-1.25E+02</td>
<td>1/m</td>
</tr>
<tr>
<td>$T_{112}$</td>
<td>1.28E+01</td>
<td>2.88E+02</td>
<td></td>
</tr>
<tr>
<td>$T_{122}$</td>
<td>8.59E+00</td>
<td>-1.59E+02</td>
<td>m</td>
</tr>
<tr>
<td>$T_{133}$</td>
<td>-5.14E+01</td>
<td>1.16E+02</td>
<td>1/m</td>
</tr>
<tr>
<td>$T_{134}$</td>
<td>-5.67E+01</td>
<td>-1.74E+02</td>
<td></td>
</tr>
<tr>
<td>$T_{144}$</td>
<td>-1.38E+01</td>
<td>1.85E+01</td>
<td>m</td>
</tr>
<tr>
<td>$T_{116}$</td>
<td>2.42E+03</td>
<td>2.77E+03</td>
<td></td>
</tr>
<tr>
<td>$T_{126}$</td>
<td>-3.31E+03</td>
<td>-3.73E+03</td>
<td>m</td>
</tr>
<tr>
<td>$T_{166}$</td>
<td>-4.20E+03</td>
<td>-4.47E+03</td>
<td>m</td>
</tr>
<tr>
<td>$T_{313}$</td>
<td>4.74E+01</td>
<td>-1.32E+02</td>
<td>1/m</td>
</tr>
<tr>
<td>$T_{323}$</td>
<td>5.75E+01</td>
<td>1.74E+02</td>
<td></td>
</tr>
<tr>
<td>$T_{314}$</td>
<td>-1.39E+01</td>
<td>3.43E+01</td>
<td></td>
</tr>
<tr>
<td>$T_{324}$</td>
<td>2.80E+01</td>
<td>-3.67E+01</td>
<td>m</td>
</tr>
<tr>
<td>$T_{336}$</td>
<td>-9.55E+02</td>
<td>-8.08E+02</td>
<td></td>
</tr>
<tr>
<td>$T_{346}$</td>
<td>1.32E+03</td>
<td>1.24E+03</td>
<td></td>
</tr>
</tbody>
</table>

Table 5.5 Values of the second-order aberration coefficients calculated from the filter’s object plane for the uncorrected filter, and the filter corrected for the second-order distortions $T_{122}$, $T_{144}$, $T_{324}$.

5.6.1 Second-order geometric aberrations

Not much can be said about the real magnitude of the various aberrations until they are reworked into the effective forms of table 3.2. Table 5.6 shows the effective geometric aberrations and their maximum blurring or displacement in μm for the filter without any correction and for the filter where the geometric distortions $T_{122}$, $T_{144}$ and $T_{324}$ have been corrected. Where applicable, a value of 1.3 mm (25 mm/2*9.72, corresponding to the corners of the CCD) is used for $x_0$ or $y_0$, a TEM magnification of 1000x (9720x at the CCD), and a semi-acceptance angle $\beta$ of 10 mrad.
<table>
<thead>
<tr>
<th>From TEM image plane</th>
<th>From projector lens</th>
<th>Uncorrected µm</th>
<th>Corrected µm</th>
</tr>
</thead>
<tbody>
<tr>
<td>((T_{111} + \frac{T_{112}}{L} + \frac{T_{122}}{L^2})x^0)</td>
<td>(\frac{T_{122}}{L^2}x^0)</td>
<td>29.4 (0.2%)</td>
<td>0.0</td>
</tr>
<tr>
<td>((T_{112} + 2\frac{T_{122}}{L})\frac{\beta_x}{M_T}x^0)</td>
<td></td>
<td>6.2</td>
<td>14.2</td>
</tr>
<tr>
<td>(T_{122}\frac{\beta_x}{M_T^2})</td>
<td></td>
<td>0.1</td>
<td>1.5</td>
</tr>
<tr>
<td>((T_{133} + \frac{T_{134}}{L} + \frac{T_{144}}{L^2})\frac{\beta_y}{y_0})</td>
<td>(\frac{T_{144}}{L^2}y_0)</td>
<td>159.2 (1.3%)</td>
<td>0.0</td>
</tr>
<tr>
<td>((T_{134} + 2\frac{T_{144}}{L})\frac{\beta_y}{M_T}y_0)</td>
<td></td>
<td>18.8</td>
<td>36.1</td>
</tr>
<tr>
<td>(T_{144}\frac{\beta_y}{M_T^2})</td>
<td></td>
<td>0.1</td>
<td>0.2</td>
</tr>
<tr>
<td>((T_{313} + \frac{T_{314}}{L} + \frac{T_{324}}{L^2}x_0)\frac{\beta_y}{y_0})</td>
<td>(\frac{T_{324}}{L^2}x_0y_0)</td>
<td>149.5 (1.2%)</td>
<td>0.0</td>
</tr>
<tr>
<td>((T_{314} + \frac{T_{324}}{L})\frac{\beta_y}{M_T}x_0)</td>
<td></td>
<td>1.6</td>
<td>1.9</td>
</tr>
<tr>
<td>((T_{323} + \frac{T_{324}}{L})\frac{\beta_x}{M_T}y_0)</td>
<td></td>
<td>19.1</td>
<td>36.3</td>
</tr>
<tr>
<td>(T_{324}\frac{\beta_x\beta_y}{M_T^2})</td>
<td></td>
<td>1.1</td>
<td>1.4</td>
</tr>
</tbody>
</table>

Table 5.6 Blurring (µm) and displacement (µm) due to the various second-order geometric aberrations for the uncorrected filter, and the filter corrected for the second-order distortions \(T_{122}, T_{144}, T_{324}\). \(L=1.383\) m, \(M_T=1000\) m, \(\beta=10\) mrad.

As can be seen, the uncorrected filter has very small aberrations overall. Already for a TEM magnification of 1000 x are all aperture aberrations and field aberrations smaller than a pixel (24 µm) and should therefore not be noticeable. The distortions are very small as well with a maximum value of 1.3% at the edges of the image.

These distortions can be corrected through the incorporation of the three sextupoles. Calculations showed that two sextupoles are best placed just after \(Q_6\) where there is little energy dispersion and the coupling of sextupoles to the chromatic aberrations will be minimal. One might expect that the third sextupole is best placed at the dispersion free intermediate x image just in front of \(Q_5\). This does indeed allow the distortions to be corrected but leads to increased second-order aberrations overall. Empirically it was found that this sextupole is best placed between \(Q_5\) and \(Q_6\).

As can be seen from the table, the corrected filter is indeed distortion free and has minimally increased aberrations overall. Only some field aberrations are
significantly larger and in some cases exceed the pixel size. It should be realized however that these values are calculated for a 10 mrad semi-acceptance angle. Given the 24 nm size of the pixel referred back to the specimen, the largest Bragg elastic scattering angle that can be captured is limited by Nyquist theorem to 3.8 mrad, for which the aberrations are smaller again than the pixel size. Only for inelastic imaging can large scattering angles be captured without aliasing, and only at low magnifications will the aberrations then just be larger than the pixel size.

5.6.2 Second-order chromatic aberrations

Table 5.7 shows the maximum blurring or displacement in μm due to the second-order chromatic aberrations for the filter without any correction and for the filter where the geometric distortions $T_{122}$, $T_{144}$ and $T_{324}$ have been corrected.
<table>
<thead>
<tr>
<th>Aberration</th>
<th>Energy window eV</th>
<th>Uncorrected μm</th>
<th>Corrected μm</th>
</tr>
</thead>
<tbody>
<tr>
<td>((T_{116} + T_{126})x_0 \frac{\Delta p}{p})</td>
<td>5</td>
<td>0.1</td>
<td>0.3</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>0.2</td>
<td>0.5</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>0.4</td>
<td>1.1</td>
</tr>
<tr>
<td></td>
<td>50</td>
<td>1.0</td>
<td>2.7</td>
</tr>
<tr>
<td>(T_{126} \frac{\beta \Delta p}{M_T p})</td>
<td>5</td>
<td>0.1</td>
<td>0.1</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>0.2</td>
<td>0.2</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>0.4</td>
<td>0.5</td>
</tr>
<tr>
<td></td>
<td>50</td>
<td>1.0</td>
<td>1.2</td>
</tr>
<tr>
<td>(T_{166}(\frac{\Delta p}{p})^2)</td>
<td>5</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>0.2</td>
<td>0.2</td>
</tr>
<tr>
<td></td>
<td>50</td>
<td>1.0</td>
<td>1.1</td>
</tr>
<tr>
<td>((T_{336} + T_{346})y_0 \frac{\Delta p}{p})</td>
<td>5</td>
<td>0.0</td>
<td>0.4</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>0.0</td>
<td>0.7</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>0.0</td>
<td>1.4</td>
</tr>
<tr>
<td></td>
<td>50</td>
<td>0.1</td>
<td>3.5</td>
</tr>
<tr>
<td>(T_{346} \frac{\beta \Delta p}{M_T p})</td>
<td>5</td>
<td>0.1</td>
<td>0.1</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>0.2</td>
<td>0.2</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>0.3</td>
<td>0.3</td>
</tr>
<tr>
<td></td>
<td>50</td>
<td>0.8</td>
<td>0.8</td>
</tr>
</tbody>
</table>

Table 5.7 Blurring (μm) as a function of the size of the energy window due to the various second-order chromatic aberrations for the uncorrected filter, and the filter corrected for the second-order distortions \(T_{122}, T_{144}, T_{324}\). \(L=1.383 m, M_T=1000x, \beta=10\) mrad.

In both cases are the aberrations negligible with respect to the pixel size. It is interesting to compare the blurring coming from the filter's largest axial chromatic aberration, in this case \(T_{126}\), with the chromatic aberration of the objective lens(17). Setting the two equal to each other:

\[
C_c \frac{\Delta E}{E} \beta = T_{126} \frac{\beta}{M_T} \frac{\Delta p}{p} \frac{1}{M_T M_F}
\]

5.21

where \(M_F\) is the magnification from the filter's object plane to the detector. Solving the above equation for \(M_T\):
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\[ M_T = \sqrt{\frac{T_{126}}{C_c} \frac{\Delta p}{p} \frac{E}{\Delta E} \frac{1}{M_F}} \]  \hspace{1cm} 5.22

With

\[ \frac{\Delta E}{E} \frac{p}{\Delta p} = 1.3 \]
\[ C_c = 3.5E - 3 \text{ m} \]
\[ T_{126} = 3733 \text{ m} \]
\[ M_F = 9.72 \]

it follows that above a TEM magnification \( M_T = 378x \) (3671 at the CCD), the blurring in the final image from the objective lens chromatic aberration surpasses that of the filter.

5.6.3 Third-order aberrations

The formulas for the third-order aberrations together with their values for the corrected filter are shown in table 5.8. As can be seen all but one aberration are smaller than the pixel size resulting in a 0.3% distortion.
<table>
<thead>
<tr>
<th>Aberration</th>
<th>Corrected μm</th>
</tr>
</thead>
<tbody>
<tr>
<td>( (U_{1111} + \frac{U_{1112}}{L} + \frac{U_{1122}}{L^2} + \frac{U_{1222}}{L^3})x_0 )</td>
<td>32.6</td>
</tr>
<tr>
<td>( (U_{1112} + 2 \frac{U_{1122}}{L} + 3 \frac{U_{1222}}{L^2}) \frac{\beta_x}{M_T} x_0 )</td>
<td>1.6</td>
</tr>
<tr>
<td>( (U_{1122} + 3 \frac{U_{1222}}{L}) \frac{\beta_x^2}{M_T} x_0 )</td>
<td>-0.1</td>
</tr>
<tr>
<td>( U_{1222} \frac{\beta_x^3}{M_T^2} )</td>
<td>0.1</td>
</tr>
<tr>
<td>( (U_{1133} + \frac{U_{1233}}{L} + \frac{U_{1134}}{L} + \frac{U_{1144}}{L^2} + \frac{U_{1234}}{L^2} + \frac{U_{1244}}{L^3})x_0 y_0^2 )</td>
<td>-10.8</td>
</tr>
<tr>
<td>( (U_{1134} + \frac{U_{1234}}{L} + 2 \frac{U_{1144}}{L^2} + 2 \frac{U_{1244}}{L^2}) \frac{\beta_y}{M_T} x_0 y_0 )</td>
<td>0.4</td>
</tr>
<tr>
<td>( (U_{1233} + \frac{U_{1234}}{L} + \frac{U_{1244}}{L^2}) \frac{\beta_x}{M_T} y_0^2 )</td>
<td>5.2</td>
</tr>
<tr>
<td>( (U_{1234} + 2 \frac{U_{1244}}{L}) \frac{\beta_x \beta_y}{M_T} y_0 )</td>
<td>-0.5</td>
</tr>
<tr>
<td>( (U_{1144} + \frac{U_{1244}}{L}) \frac{\beta_y^2}{M_T^2} x_0 )</td>
<td>0.0</td>
</tr>
<tr>
<td>( U_{1244} \frac{\beta_x \beta_y^2}{M_T^2} )</td>
<td>0.0</td>
</tr>
<tr>
<td>( (U_{3333} + \frac{U_{3334}}{L} + \frac{U_{3344}}{L^2} + \frac{U_{3444}}{L^3})y_0^3 )</td>
<td>1.0</td>
</tr>
<tr>
<td>( (U_{3334} + 2 \frac{U_{3344}}{L} + 3 \frac{U_{3444}}{L^2}) \frac{\beta_y}{M_T} y_0^2 )</td>
<td>-5.2</td>
</tr>
<tr>
<td>( (U_{3344} + 3 \frac{U_{3444}}{L}) \frac{\beta_y^2}{M_T} y_0 )</td>
<td>0.2</td>
</tr>
<tr>
<td>( U_{3444} \frac{\beta_y^3}{M_T^3} )</td>
<td>0.0</td>
</tr>
<tr>
<td>( (U_{3113} + \frac{U_{3114}}{L} + \frac{U_{3123}}{L^2} + \frac{U_{3223}}{L^2} + \frac{U_{3124}}{L^3} + \frac{U_{3224}}{L^3})x_0^2 y_0 )</td>
<td>-7.6</td>
</tr>
</tbody>
</table>
\[
\begin{array}{|c|c|}
\hline
\frac{U_{3123}}{L} + \frac{U_{3124}}{L} + 2 \frac{U_{3223}}{L} + 2 \frac{U_{3224}}{L^2} \frac{\beta_x}{M_T} x_0 y_0 & -0.4 \\
\hline
(U_{3114} + \frac{U_{3124}}{L} + \frac{U_{3224}}{L^2}) \frac{\beta_y}{M_T} x_0^2 & -0.3 \\
\hline
(U_{3124} + 2 \frac{U_{3224}}{L}) \frac{\beta_x \beta_y}{M_T} x_0 & 0.0 \\
\hline
(U_{3223} + \frac{U_{3224}}{L}) \frac{\beta_x^2}{M_T^2} y_0 & 0.2 \\
\hline
\frac{U_{3224}}{L} \frac{\beta_x \beta_y}{M_T^3} & 0.0 \\
\hline
(U_{1116} + \frac{U_{1126}}{L} + \frac{U_{1226}}{L^2}) \frac{\Delta p}{p} x_0^2 & 1.6 \\
\hline
(U_{1126} + 2 \frac{U_{1226}}{L}) \frac{\beta_x}{M_T} \frac{\Delta p}{p} x_0 & -0.4 \\
\hline
\frac{U_{1226}}{L} \frac{\beta_x^2}{M_T^2} \frac{\Delta p}{p} & 0.6 \\
\hline
(U_{1166} + \frac{U_{1266}}{L}) \frac{(\Delta p)^2}{p} x_0 & -0.2 \\
\hline
\frac{U_{1266}}{L} \frac{\beta_x}{M_T} (\frac{\Delta p}{p})^2 & 1.1 \\
\hline
\frac{U_{1666}}{L} \frac{(\Delta p)^3}{p} & 0.7 \\
\hline
(U_{1336} + \frac{U_{1346}}{L} + \frac{U_{1446}}{L^2}) \frac{\Delta p}{p} y_0^2 & 9.7 \\
\hline
(U_{1346} + 2 \frac{U_{1446}}{L}) \frac{\beta_y}{M_T} \frac{\Delta p}{p} & -1.9 \\
\hline
\frac{U_{1446}}{L} \frac{\beta_y}{M_T} \frac{(\Delta p)^2}{p} & 0.1 \\
\hline
(U_{3136} + \frac{U_{3236}}{L} + \frac{U_{3146}}{L} + \frac{U_{3246}}{L^2}) \frac{\Delta p}{p} x_0 y_0 & -1.0 \\
\hline
\end{array}
\]
<table>
<thead>
<tr>
<th>Term</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>( U_{3236} \frac{\beta_x \Delta p}{M_T \ p} y_0 )</td>
<td>18.7</td>
</tr>
<tr>
<td>( (U_{3146} + \frac{U_{3246}}{L}) \frac{\beta_y \Delta p}{M_T \ p} x_0 )</td>
<td>0.1</td>
</tr>
<tr>
<td>( U_{3246} \frac{\beta_x \beta_y \Delta p}{M_T^2 \ p} )</td>
<td>-0.2</td>
</tr>
<tr>
<td>( (U_{3366} + \frac{U_{3466}}{L}) \frac{(\Delta p)^2}{p} y_0 )</td>
<td>0.9</td>
</tr>
<tr>
<td>( U_{3466} \frac{\beta_y}{M_T} \frac{(\Delta p)^2}{p} )</td>
<td>-0.2</td>
</tr>
</tbody>
</table>

Table 5.8 The effective third-order aberrations calculated from the filter's object plane. The values (\( \mu \text{m} \)) of the aberrations are listed for the design corrected for the second-order distortions \( T_{122}, T_{144}, T_{324} \). \( L=1.383 \text{ m}, M_T=1000x, \beta=10 \text{ mrad} \).
5.7 Alignment

5.7.1 Spectrum focus at the energy-selecting slit

The alignment of the energy-loss spectrum is depicted in the flow chart of figure 5.8.

![Flow chart](image)

Figure 5.8 Flow chart for the first-order spectrum alignment and the correction of the second-order spectrum aberrations ($R_{12}, R_{34}, T_{122}, T_{144}, T_{126}$).

It starts by exciting the magnetic prism to the calculated value. The prism current is then adjusted until the electron beam can be seen on either of the cameras. Next, the first quadrupole, Q1, is excited to its calculated setting. The energy-selecting slit is inserted and the zero-loss is positioned right on the bottom edge of the energy-selecting slit. By observing the shadow of the slit edge on the cameras, the first-order focus in the dispersion direction and the second-order aberrations can be studied. A well focused, aberration free energy-loss spectrum will look like figure 5.9. The zero-loss peak being straight and focused, the shadow of the bottom slit edge can not be distinguished: if the zero-loss peak is scanned over the bottom slit edge, the entire image will appear / disappear uniformly.
Figure 5.9 A focused, aberration free energy-loss spectrum at the energy-selecting slit. First picture, the actual zero-loss peak in the plane of the energy-selecting slit. Second picture, the shadow of the slit edge in the image on the cameras. Third picture, as picture 2 but with the 25 hole reference mask inserted.

If the spectrum is out of focus in the dispersion direction, a clear shadow of the bottom slit edge will be seen as in figure 5.10.

Figure 5.10 Various manifestations of a defocused spectrum at the energy-selecting slit. First picture, the actual zero-loss peak in the plane of the energy-selecting slit. Second picture, the shadow of the slit edge in the image on the cameras. Third picture, as picture 2 but with the 25 hole mask inserted.

If the energy-loss spectrum suffers from the second-order aberrations $T_{122}$ and $T_{144}$ the zero-loss peak will be cup-shaped and images will look like figure 5.11.

Figure 5.11 An energy-loss spectrum focused in the dispersion direction but suffering from the second-order aberrations $T_{122}$ and $T_{144}$. First picture, the actual zero-loss peak in the plane of the energy-selecting slit. Second picture, the shadow of the slit edge in the image on the cameras. Third picture, as picture 2 but with the 25 hole mask inserted.

The excitation of Q1 is first adjusted to focus the energy-loss spectrum in the dispersion direction. If any second-order aberrations can be seen, the pre-prism sextupole is adjusted to correct them. If $T_{122}$ and $T_{144}$ can not be corrected simultaneously, the magnetic prism is slightly rotated, mainly to change the sextupole moment of the exit face. As this will also move the crossovers of the prism, Q1 needs to be adjusted to maintain the focus in the dispersion direction.
Once both $T_{122}$ and $T_{144}$ are corrected and the image looks like figure 5.8, the spectrum also needs to be focused in the non-dispersive direction. This is done by moving a vertical edge (parallel to the dispersion direction) that is part of the energy-selecting slit mechanism into the electron beam and observing its shadow in the image. Similarly to the x-focus, a clear shadow will be seen when the spectrum is not focused in the y-direction, whereas no shadow can be seen when the image is properly focused in the y-direction. If the y-focus is off, a weak pre-prism quadrupole is adjusted in combination with Q1 until the spectrum is focused in both the x- and the y-direction. The effect of small adjustments of the pre-prism quadrupole on the second-order aberrations $T_{122}$ and $T_{144}$ is negligible.

The only remaining second-order spectrum aberration $T_{126}$, which expresses itself as an inclination of the spectrum plane, can be checked in the Spectroscopy Mode at a low energy dispersion. If present, $T_{126}$ will cause the focus of the zero-loss peak to vary with its position on the detector.

### 5.7.2 First-order image alignment

Table 5.9 shows the change of the important first-order imaging properties for a small change in the excitation of the imaging quadrupoles.

<table>
<thead>
<tr>
<th></th>
<th>Normal</th>
<th>$\Delta Q_3$</th>
<th>$\Delta Q_4$</th>
<th>$\Delta Q_5$</th>
<th>$\Delta Q_6$</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>R11</td>
<td>9.72</td>
<td>-0.06</td>
<td>11.41</td>
<td>8.18</td>
<td>10.24</td>
<td>m</td>
</tr>
<tr>
<td>R12</td>
<td>0.00</td>
<td>13.47</td>
<td>-2.31</td>
<td>2.45</td>
<td>-0.13</td>
<td>m</td>
</tr>
<tr>
<td>R16</td>
<td>0.00</td>
<td>17.03</td>
<td>-2.92</td>
<td>3.10</td>
<td>-0.17</td>
<td>m</td>
</tr>
<tr>
<td>R33</td>
<td>-9.72</td>
<td>-10.47</td>
<td>-5.80</td>
<td>-10.50</td>
<td>-10.00</td>
<td>m</td>
</tr>
<tr>
<td>R34</td>
<td>0.00</td>
<td>0.94</td>
<td>-4.88</td>
<td>0.90</td>
<td>-0.05</td>
<td>m</td>
</tr>
</tbody>
</table>

Table 5.9 Effect of the imaging quadrupoles on various first-order properties. 1 Gauss change for Q3 and Q4, 10 Gauss change for Q5 and Q6.

This table shows that both Q5 and Q6 act as a projector lenses. Q6 has a similar effect on the x- and y-magnification ($R_{11}$ and $R_{33}$) and hardly affects the focus ($R_{12}$ and $R_{34}$) or the chromaticity ($R_{16}$) of the image. Q5 has a somewhat different effect on $R_{11}$ than on $R_{33}$ and can be used to change the x- versus y-magnification or aspect ratio of the image. Q5 also has a small effect on the chromaticity due to the fact that their is a small amount of energy-dispersion left in the electron beam.

Q4 has a strong effect on the chromaticity as well as the focus of the system ($R_{12}$ and $R_{34}$).

Q3 finally has a very strong effect on all imaging properties, especially the chromaticity and the astigmatism ($R_{12}$ versus $R_{34}$).

Given these properties, Q3 is best used to adjust the astigmatism, Q4 to control the chromaticity, Q5 the aspect ratio and Q6 the magnification. As each quadrupole has some effect on each first-order property, the above will of course be an iterative process.

The alignment procedure is shown in figure 5.12.
The alignment uses the 25 hole reference mask as this allows an easy measurement of the various imaging properties. First the lenses are excited to their calculated settings. Next some energy variation is introduced in the electron beam by applying a 50 eV modulation to the TEM high voltage. If the image has some chromaticity, two separate images can be seen. Q4 is then adjusted to make the two images coincide and the imaging achromatic (figure 5.13).

Next Q5 is adjusted to balance the x and y magnifications, and Q6 is adjusted to set the magnification to the desired value.

Next it is verified whether or not the imaging is stigmatic. This can be done by applying a square wave modulation to a pre-prism quadrupole (Krivanek, O.L., private communications). If the imaging is exactly stigmatic, the aspect ratio of the
25 hole mask will change in exactly the same fashion for a positive or a negative change of the pre-prism quadrupole, although in perpendicular directions (figure 5.14).

![Image of the 25 hole pattern with a fast square wave modulation applied to the pre-prism quadrupole. The pattern consisting of the solid dots results from the positive swing, the pattern consisting of the empty dots results from the negative swing of the pre-prism quadrupole. First picture, astigmatic imaging. Second picture, stigmatic imaging. Only for stigmatic imaging will the aspect ratio of the mask image change similarly for the negative and the positive swing.]

If the imaging is found to be astigmatic, Q3 is slightly adjusted and the alignment continues as shown in the flow chart.

### 5.7.3 Second-order image alignment

Section 5.4 showed that all chromatic aberrations can be neglected and that only the second-order aberrations $T_{122}$, $T_{144}$ and $T_{324}$ may need correction.

Table 5.10 shows the change of these aberration coefficients for a 1 Gauss change in the excitation of the sextupoles.

<table>
<thead>
<tr>
<th></th>
<th>$T_{122}$</th>
<th>$T_{144}$</th>
<th>$T_{324}$</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>-0.1</td>
<td>1.5</td>
<td>-0.3</td>
<td>m</td>
</tr>
<tr>
<td>S2</td>
<td>-5.4</td>
<td>3.1</td>
<td>-8.2</td>
<td>m</td>
</tr>
<tr>
<td>S3</td>
<td>-10.4</td>
<td>8.4</td>
<td>-18.7</td>
<td>m</td>
</tr>
</tbody>
</table>

Table 5.10 Effect of a 1 Gauss change of each of the sextupoles on the three important various second-order coefficients.

The sextupoles can be adjusted manually and the aberrations corrected visually. As table 5.10 indicates, it is best to first correct $T_{122}$ and $T_{324}$ by adjusting S2 and S3 after which $T_{144}$ can be corrected with S1.

The process can also be automated. In this case the sextupole coupling coefficients \( \frac{\partial T_i}{\partial S_j} \)

5.23

to each of the aberration coefficients is determined. The "corrected" aberration coefficients $T_{ijkC}$ can then be described as the linear combination of the sextupoles excitations with the coupling coefficients as weight factors:

\[
T_{122C} = T_{122U} + \frac{\partial T_{122}}{\partial S_3} S_3 + \frac{\partial T_{122}}{\partial S_4} S_4 + \frac{\partial T_{122}}{\partial S_5} S_5
\]

\[
T_{144C} = T_{144U} + \frac{\partial T_{144}}{\partial S_3} S_3 + \frac{\partial T_{144}}{\partial S_4} S_4 + \frac{\partial T_{144}}{\partial S_5} S_5
\]

\[
T_{324C} = T_{324U} + \frac{\partial T_{324}}{\partial S_3} S_3 + \frac{\partial T_{324}}{\partial S_4} S_4 + \frac{\partial T_{324}}{\partial S_5} S_5
\]
The subscript $U$ specifies the uncorrected aberration coefficient. Setting the left hand side of 5.24 to zero, the required sextupole excitations can be found by solving the set of equations.

This process is depicted in figure 5.15.

Figure 5.15 Flow chart for the automated correction of the second-order geometric aberrations, $T_{122}, T_{144}$ and $T_{324}$. 
6 Mechanical Design

The design of an earlier version of the filter has been reported on before (58). Although the filter (figure 6.1) was designed especially for use with the JEOL ARM1000 and 1250 microscopes, its design can easily be adapted to other modern high voltage microscopes. The filter is mounted to the camera chamber of the electron microscope. A gate valve allows the filter vacuum to be preserved when the camera chamber is vented to replace the photographic film plates. A pumping port mounted beneath the gate valve allows the filter to be pumped even when the camera chamber is vented.

The pneumatic entrance aperture mechanism is computer-controlled and contains 3.0, 2.0 and 0.6 mm apertures, as well as the 25-hole reference mask used for evaluating and/or correcting the various image distortions produced by the filter.

The magnetic prism of 25 cm bending radius, has curved entrance and exit faces, 90° bending angle, 1.25 cm pole face gap. A weak quadrupole and sextupole are located in front of the magnetic prism, such that the quadrupole and sextupole moment of the entrance face can be fine-tuned. The prism is mounted such that it can be rotated over about ±5° about an axis perpendicular to the bending plane, allowing some further control of the quadrupole and sextupole moments of the entrance and exit face.

By rotating the prism and adjusting the weak pre-prism sextupole and quadrupole, the first and second-order spectrum focusing properties can be fine-tuned for precise cancellation of the aberrations in the spectrum plane. The drift tube leading through the sector drift tube is electrically isolated. A voltage applied to the drift tube (de)accelerates the electrons while they are inside the prism and allows the energy-loss spectrum to be precisely shifted which is used for alignment purposes and for calibration of the energy scale.

The lens assembly following the magnetic prism contains two quadrupoles. Although in the calculations only one quadrupole was used to magnify and focus the spectrum created by the prism, in the actual design a second quadrupole is added such that the focus of the spectrum at the slit can be fine-tuned without affecting the second-order aberration correction.

The energy-selecting slit is computer controlled and can be pneumatically retracted for unfiltered imaging or spectroscopy. The slit consists of a fixed bottom edge and a piezo-electrically actuated top edge. The movement of the top slit is calibrated and the user can select a slit width in eV by simply entering it in the FilterControl software.

The energy-selecting slit is followed by three lens assemblies that contain the 4 quadrupoles and 3 sextupoles necessary to either transform the spectrum formed at the slit into an achromatic image that is free of all important second-order aberrations and distortions, or project a magnified image of the spectrum onto the filter's detectors with a range of dispersions.

A fiber-optically coupled TV-rate camera can be pneumatically inserted into the beam just in front of the slow-scan camera and allows TV-rate observation of images and spectra. This camera is mainly used for searching and alignment purposes. It lacks the sensitivity to record the very faint core-loss images.
For this purpose the use of a slow-scan CCD camera is essential. A special lens-coupled slow-scan CCD camera was developed specifically for high voltage applications(57). The need for the new design arose because slow-scan CCD cameras that use a thin scintillator mounted on a fiber-optic plate(57, 59) perform poorly when used at primary energies greater than 500 keV. They suffer from beam spreading that produces pronounced "tails" in the point-spread function of the scintillator, as well as unacceptable radiation damage in the fiber optic plate. This design therefore uses a thin powder phosphor (P20 or P43) deposited on the beam-exit side of a thin, self-supporting Al foil (thickness < 20 μm) mounted on a solid rim. The light image produced by the scintillator is transferred to the CCD chip with unity magnification by a 90° aluminum mirror and a pair of front-to-front, f/2 lenses. The advantages of this arrangement are: 1) it keeps the lenses and the CCD camera out of the intense forward-moving beam of X-rays generated by relativistic energy electrons hitting a solid target; 2) the greatest part of the incident beam energy is deposited in an aluminum mirror rather than an easily damaged glass window or lens; 3) it shortens the overall length of the instrument. Advantage 1) is especially important in core-loss imaging where the low-loss region in the spectrum is intercepted by the energy selecting slit. Even though a Beryllium beam trap is used, the dissipation of practically all the beam energy in a small area results in a very intense source of X-rays. Having the CCD at 90° and some 30 cm above the beam allows enough lead to be placed between the energy selecting slit and the CCD such that these X-rays will not affect the image quality.

The camera uses a 1024 x 1024 pixel CCD sensor with 24 μm square pixels. The image detected by the CCD chip is digitized with 14 bits dynamic range. The sensitivity is such that one single primary electron produces on average about one digitized count, giving a maximum intensity of about 16000 primary electrons per pixel before saturation is reached. The captured images are sent over a 16-bit direct memory access (DMA) interface to a Macintosh computer where they are immediately processed and displayed by Gatan's DigitalMicrograph software and can be analyzed. A more complete description of the camera design and test results obtained at 800 keV have been given elsewhere(57).

The slow-scan camera and the lens assemblies are water cooled. The water is provided at room temperature by one of the TEM water chillers. The water first enters the slow-scan camera and then runs along the length of the lens assemblies to the front of the filter from where it returns to the chiller. This arrangement ensures that the temperature of the water going into the slow-scan camera does not vary with the operating mode of the filter while only one chiller is needed.

X-ray shielding is provided by orienting the filter squarely towards the back of the microscope and placing the horizontal portion of the filter in a trench in the floor covered by lead blocks. The thick X-ray walls at the front and the sides of the microscope shield the operator from X-rays generated in the upper parts of the filter and the lead blocks prevent X-rays generated in the lower part of the filter from radiating outside the trench.

The weight of the filter is supported by the TEM camera chamber flange and by a support that attaches the lens assemblies to the steel microscope foundation. The support consists of a steel rod, a steel belt that wraps around the lens assembly and a turn buckle for adjustment purposes.
Figure 6.1 Schematic overview of the HV imaging filter.
7 Experimental Results

Experimental results were obtained at 800 keV, 1.0 MeV and 1.25 MeV using filters mounted on three different JEOL ARM microscopes: 1) the ARM 1000 at Kyoto University, Kyoto, Japan; 2) the ARM 1250 at the Max-Planck-Institut für Metallforschung, Institut für Physik, Stuttgart, Germany; 3) the ARM 1000 at the National Research Institute for Metals, Tsukuba, Japan. The results presented are a mixture of the results obtained with the earlier design (58) and the design discussed here.

7.1 The spectroscopy mode

Figure 7.1a shows a spectrum obtained at 1.25 MeV with an emission current <1 μA using an undersaturated filament, 0.6 mm entrance aperture, dispersion of 0.1 eV/pixel and an exposure time of 0.1 seconds. The full-width at half-maximum of the zero loss peak is 0.6 eV and the peak clearly shows the characteristic asymmetry of thermionic sources. The energy spread from a strongly undersaturated LaB₆ filament is about 0.4 eV. Adding the principal resolution-limiting factors (source energy spread, HT instabilities and sector current instabilities) in quadrature shows that the short-term stability of both the high voltage of the microscope and of the power supply of the filter's magnetic sector had to be better than 0.3 parts per million to permit 0.6 eV resolution.

![Figure 7.1 Zero-loss peaks recorded at 1.25 MeV. (a) Undersaturated filament, 0.6 mm diameter entrance aperture, 0.1 s. exposure time: fwhm 0.6 eV. (b) Saturated filament, 2.0 mm diameter entrance aperture, 20 s. exposure time: fwhm 2.1 eV.](image)

Typical experimental conditions at 1.25 MeV, such as a saturated LaB₆ filament, a 2.0 mm diameter aperture and a 20 seconds integration time resulted in zero-loss peak widths around 2 eV (figure 7.1b), showing the excellent longer-term stability of the two critical power supplies, and the usefulness of the instrument for general EELS.
7.2 The imaging mode

7.2.1 The aberration figure

An estimate for the filter's aberrations was determined by scanning the zero-loss peak over the bottom slit edge in 0.1 eV steps (60) and recording an image at each step. From the acquired images a new image was constructed. The value of each pixel in the new image reflects the voltage at which its intensity dropped to 50% of the starting value.

Figure 7.2 The aberration figure. Full height is 0.9 eV.

This aberration surface is shown in figure 7.2. Its full height is 0.9 eV.
7.2.2 Uncorrected performance

The imaging performance of the filter was tested both with and without second-order distortion correction. Figure 7.3 shows an uncorrected 512 x 512 pixel image of the 25-hole test pattern inserted in front of the filter. The listed distortions follow from the distance between the actual and the predicted positions, divided by the distance of the predicted positions from the central hole, times 100 %. The actual hole positions were determined using a center of gravity algorithm(60). The central hole is used as the reference and its distortion is therefore zero.

<table>
<thead>
<tr>
<th>Hole</th>
<th>Distortion</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.4</td>
</tr>
<tr>
<td>2</td>
<td>1.3</td>
</tr>
<tr>
<td>3</td>
<td>1.2</td>
</tr>
<tr>
<td>4</td>
<td>0.6</td>
</tr>
<tr>
<td>5</td>
<td>0.0</td>
</tr>
<tr>
<td>6</td>
<td>0.4</td>
</tr>
<tr>
<td>7</td>
<td>1.2</td>
</tr>
<tr>
<td>8</td>
<td>1.5</td>
</tr>
<tr>
<td>9</td>
<td>1.3</td>
</tr>
</tbody>
</table>

Figure 7.3 Uncorrected image of the 25 hole test pattern located in the entrance aperture assembly. The distortions are listed for the 9 numbered holes and is 1.5 % maximum.

As can be seen the about 1.5% distortions are mainly due to $T_{144}$ (curvature) and $T_{324}$ (trapezoid).

From the image of figure 7.3, the actual optical coefficients can be determined using a least square fitting routine. Table 7.1 contains the results for the coefficients up to second-order. It can be seen that the overall magnification was set slightly too large resulting in $R_{12}=13.85$ and $R_{34}=-13.82$ rather than 13.44. The aberration coefficients, which were recalculated for the larger magnification, show a reasonable match with the measured values. The error bars on the measured coefficients follow from the least square fitting routine. The error bars on the calculated coefficients reflect the possible offset currents in the electronics, which can cause the sextupoles to be excited even when they are supposedly off. In the particular experimental configuration it is possible that S1 may actually have been excited up to ±20 Gauss, and S2 and S3 up to ±10 Gauss. From table 5.10 it can be seen that this could change $T_{122}$ and $T_{324}$ by as much as -156 and -275 m while at the same time increasing $T_{144}$ by 145 m. It should be noted that the actual coupling coefficients will likely differ from those in table 5.10 as only a crude approximation was used to specify the effective length of the sextupoles in the calculations.
<table>
<thead>
<tr>
<th>Coefficient</th>
<th>Calculated</th>
<th>Measured</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>m</td>
<td>m</td>
</tr>
<tr>
<td>$R_{12}$</td>
<td>13.82</td>
<td>13.85±0.01</td>
</tr>
<tr>
<td>$R_{34}$</td>
<td>-13.82</td>
<td>-13.82±0.01</td>
</tr>
<tr>
<td>$T_{122}$</td>
<td>40(±156)</td>
<td>-44±6</td>
</tr>
<tr>
<td>$T_{144}$</td>
<td>-213(±145)</td>
<td>-239±6</td>
</tr>
<tr>
<td>$T_{324}$</td>
<td>200(±275)</td>
<td>141±6</td>
</tr>
</tbody>
</table>

Table 7.1 Calculated and measured aberration coefficients for the uncorrected filter.

### 7.2.3 Second-order corrected performance

Figure 7.4 shows a second-order corrected 25-hole test pattern. The adjustment of the sextupole lenses was done manually. Also the quadrupoles were changed slightly. The distortions are only 0.6% maximum and are well within the design specification of 5%.

![Image of the 25 hole test pattern located in the entrance aperture assembly. The sextupoles are excited. The distortions are listed for 9 of the holes (corners, center etc.) and is 0.6 % maximum.]

<table>
<thead>
<tr>
<th>Hole</th>
<th>Distortion</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.3</td>
</tr>
<tr>
<td>2</td>
<td>0.6</td>
</tr>
<tr>
<td>3</td>
<td>0.4</td>
</tr>
<tr>
<td>4</td>
<td>0.6</td>
</tr>
<tr>
<td>5</td>
<td>0.0</td>
</tr>
<tr>
<td>6</td>
<td>0.3</td>
</tr>
<tr>
<td>7</td>
<td>0.1</td>
</tr>
<tr>
<td>8</td>
<td>0.2</td>
</tr>
<tr>
<td>9</td>
<td>0.4</td>
</tr>
</tbody>
</table>

Figure 7.4 Image of the 25 hole test pattern located in the entrance aperture assembly. The sextupoles are excited. The distortions are listed for 9 of the holes (corners, center etc.) and is 0.6 % maximum.

<table>
<thead>
<tr>
<th>Coefficient</th>
<th>Measured</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>m</td>
<td></td>
</tr>
<tr>
<td>$R_{12}$</td>
<td>13.94±0.01</td>
<td></td>
</tr>
<tr>
<td>$R_{34}$</td>
<td>-13.93±0.01</td>
<td></td>
</tr>
<tr>
<td>$T_{122}$</td>
<td>49.3±6.4</td>
<td></td>
</tr>
<tr>
<td>$T_{144}$</td>
<td>-14.0±6.4</td>
<td></td>
</tr>
<tr>
<td>$T_{324}$</td>
<td>27.0±6.9</td>
<td></td>
</tr>
</tbody>
</table>

Table 7.2 Calculated and measured aberration coefficients for the corrected filter.
Table 7.2 shows the measured aberration coefficients. Although $T_{144}$ and $T_{324}$ were reduced, $T_{122}$ was actually increased showing that the fine tuning of the aberrations coefficients is best automated.

Figure 7.5 shows an image of the test pattern while a square wave oscillation of 50 V amplitude was applied to the microscope high voltage to simulate the filter's imaging performance for a 50 eV wide energy window. No visible blurring can be distinguished. To quantify the achromaticity of the filter, two images of the test pattern were recorded with a 50 eV difference in energy. For both images the positions of the holes were determined using the same routine as used in the determination of the distortions. The chromatic movement of the 9 basic holes is listed in pixels for the $x$ and $y$ direction and shows the total chromatic blurring to be smaller than 1 CCD pixel. This represents the combined effect of any remaining first-order dispersion and all chromatic aberrations.

Figure 7.5 Image of the test pattern acquired while a 50 V oscillation was applied to the microscope high voltage.

Movement of P1:  0.0, -0.8  
Movement of P2:  0.0, -0.6  
Movement of P3:  -0.4, -0.4  
Movement of P4:  -0.8, -0.0  
Movement of P5:  -0.8, -0.0  
Movement of P6:  -1.0, -0.2  
Movement of P7:  0.8, -0.4  
Movement of P8:  0.4, 0.4  
Movement of P9:  0.2, 0.4
7.3 Applications results

Figure 7.6 shows a zero-loss filtered image of gold particles on amorphous carbon recorded at 1.25 MeV using a 10 eV wide slit centered on the zero-loss peak. The insert shows the computed Fourier transform of the image. The microscope magnification was 80,000 times and the total magnification on the filter's CCD camera was about 1.4 million times.

![Image](image_url)

Figure 7.6 No-loss high resolution image of gold particles on amorphous carbon recorded by the filter at 1.25 MeV. The insert shows the computed diffractogram of the image.

The diffractogram insert shows discrete spacings of 0.23, 0.20, 0.14, 0.123 and 0.118 nm, corresponding to (111), (200), (220), (311) and (222) gold spacings. There is also an intensity halo due to the amorphous carbon that extends out to about (0.11 nm)^{-1} in all directions. This demonstrates that stray magnetic fields, whose effect would have been to decrease the attainable resolution at the filter's CCD predominantly in one direction, were properly shielded by the steel viewing chamber of the microscope and the μ-metal shield surrounding the magnetic sector.
Experimental Results

The absence of dark bands in the diffractogram shows that the image was recorded close to Scherzer defocus and illustrates the excellent interpretable resolution performance of the microscope.

Figures 7.7 through 7.9 illustrate elemental mapping and spectroscopic analysis of a practical material with the filter. Figure 7.7 shows an unfiltered image of a ceramic material consisting of Si₃N₄ and SiC grains, bound together by a glassy mixture of alumina and yttria. A zero-loss filtered image of the same area was also recorded, but it showed poorer intensity and contrast. This demonstrates that one of the advantages of high voltage operation is that the low chromatic aberration preserves the contrast due to low-loss electrons. Some of the crystalline grains are diffracting and show strong contrast. Other grains are not in a Bragg orientation and show up only weakly. In the thicker part of the sample on the left side of the image, it is almost impossible to distinguish individual grains.

![Figure 7.7 Unfiltered image of a Si₃N₄/SiC ceramic sample recorded through the filter at 1.25 MeV. Sample courtesy Dr. J. Mayer, Max-Planck-Institut for Metallforschung, Institut für Werkstoffwissenschaft, Stuttgart, Germany.](image)
Figure 7.8 shows spectra acquired from roughly the area of figure 7.7. Figure 7.8a shows a spectrum containing the carbon, nitrogen and oxygen K-edges. Quantification of the spectrum using an AE⁻ʳ-type (A and r are constants and E is the energy-loss) background extrapolation and subtraction gave the elemental ratios shown in the insert. Figure 7.8b shows the higher energy-losses.

Figure 7.8 Energy-loss spectra from the sample of Figure 7. Primary energy 1.25 MeV. The medium losses (left) and high losses (right) were acquired separately.

Figure 7.9 shows elemental maps acquired with the filter from the specimen area shown in figure 7.7. All the maps were obtained using the three-window mapping method, in which two energy-filtered images are acquired before an edge and one after the edge.
Figure 7.9 Elemental maps of carbon, nitrogen and oxygen of the ceramic sample. Primary energy 1.25 MeV.
The two pre-edge images allow the background to be modeled as $AE^{-f}$, where $A$ and $r$ are evaluated at each pixel. Extrapolating and subtracting the contribution of the background at each pixel of the post-edge image results in an elemental map of the element of interest. The total recording times were $3 \times 20$ seconds for the carbon map and $3 \times 30$ seconds for the nitrogen and oxygen maps. The width of the energy selecting slit was $30$ eV. The carbon map shows the SiC grains, the nitrogen map shows the $\text{Si}_3\text{N}_4$ grains and the oxygen map shows the inter-granular glassy phase.

7.4 Further work

In total three of the filters have been built. They are currently in use at Kyoto University, Japan, the Max-Planck-Institut für Metallforschung, Institute für Physik in Stuttgart, Germany and the National Research Institute for Metals, Tsukuba, Japan. Especially good results have been obtained in Kyoto by Kurata et al., for instance with chemical imaging using a chemical shift of the oxygen K-edge for two types of oxides(61) and the experimental demonstration of the presence of the retardation effect in the inelastic crosssection at relativistic energies(62). Theoretical analysis has shown that the filters should be able to provide the highest resolution elemental maps in any TEM(63).
Summary

The design of a post-column imaging energy filter suitable for energy-filtered transmission electron microscopy up to 1.25 MeV is described. The filter uses a single magnetic sector with curved faces and 25 cm bending radius, 5 quadrupole and 3 sextupole lenses. The filter is essentially free of second-order aberrations and distortions in both the spectrum and the image plane.

Chapter 1, "Energy-Filtered HV TEM", starts with a discussion of transmission electron microscopy and the two mechanisms of electron scattering: elastic and inelastic. It is shown that the inelastic scattering contains useful elemental and chemical information which is explored in the field of electron energy-loss spectroscopy (EELS). From a discussion of the chromatic aberration of the TEM objective lens it follows that the inelastic scattering contribution to the image is typically out of focus and causes a loss of image contrast and detail. This can be reduced by operation at high voltage where the proportion of inelastic scattering will be lower. Next the technique of electron spectroscopic imaging (ESI) is discussed. ESI allows the formation of images from narrow ranges in the energy-loss spectrum and can be used to enhance the image contrast or to generate elemental and chemical maps. An estimate is given of the angular distribution of the inelastic scattering at 1.25 MeV, showing that most of the inelastic scattering up to 3 keV energy-loss is contained within a 10 mrad semi-angle.

Chapter 2, "Instrumentation for ESI", starts with a discussion of the principle of electron spectroscopic imaging. Next the design parameters for imaging energy filters are discussed. These filters typically suffer from electron optical aberrations and their design must be carefully optimized. Important performance parameters are the filter's non-isochromaticity, or the energy variation across the energy filtered image, and the transmissivity, which is a measure for the collection efficiency of the system. The chapter closes with a brief discussion of the various types of in- and post-column filters.

Chapter 3, "Electron Optical Calculations", provides the basis for the electron optical calculations. It introduces the matrix formalism where each optical element is described by a transfer matrix. The common optical elements and their transfer matrices are discussed. The regular matrix aberration theory is adapted for the particular case of imaging energy filters allowing all the optical parameters can be calculated from the projector lens crossover plane.

Chapter 4, "Design Specifications", discusses the various boundary, performance and component specifications.

Chapter 5, "The Electron Optical Design", shows that the design process is essentially split in two parts, that of the pre- and that of the post-slit optics. The design of the pre-slit optics is discussed and evaluated. Special attention is given to the non-isochromaticity and transmissivity of the filter. Next, the design of the post-slit optics is discussed and evaluated. The design contains 3 sextupoles, that can be used to correct the about 1.5% distortion in the image, although at the expense of slightly increased field aberrations. The chapter closes with an overview of the first- and second-order alignment procedures.

Chapter 6, "The Mechanical Design", briefly covers various aspects of the mechanical design.
Chapter 7, "Experimental Results", discusses experimental results obtained from three JEOL ARM TEMs. The performance of the filter is demonstrated with spectra having an energy resolution better than 1 eV at 1.25 MeV, zero-loss filtered images with a spatial resolution of 1.1 Å at 1.25 MeV and negligible distortion, inner shell loss spectra and elemental maps.
Samenvatting

Het ontwerp van een post-column afbeeldend energie filter is beschreven. Het filter is geschikt voor gebruik op 1.25 MeV en bevat een sectormagneet met gekromde in- en uitgangsvlakken, 5 vierpolen en 3 zespolen. Het filter heeft praktisch geen tweede orde aberraties in het spectrum en het beeld.

Hoofdstuk 1, "Energie-filterd TEM" begint met een diskussie van de transmissie elektronenmicroscopie en de twee vormen van elektronenverstrooiing: elastische and inelastische verstrooiing. Het wordt aangetoond dat de inelastische verstrooiing nuttige chemische informatie bevat. Hiervan wordt gebruik gemaakt in de elektronen energieverliespectroscopie. Door middel van een diskussie van de chromatische aberratie van de objectief lens wordt aangetoond dat de inelastische contributie aan de beeldvorming typisch gedefocuseerd is wat ten koste gaat van het contrast en detail in het beeld. Dit kan verminderd worden door gebruik van hogere versnellingspanningen waar de inelastische verstrooiing in verhouding minder is. Vervolgens wordt de Electron Spectroscopic Imaging (ESI) techniek besproken. Door middel van ESI kunnen beelden opgenomen worden die overeenkomen met verschillende delen van het energieverliespectrum. Dit kan gebruikt worden om het beelcontrast te verhogen of om chemische beelden op te nemen. De hoekverdeling van de inelastische verstrooiing wordt geschat voor 1.25 MeV. Dit toont aan dat voor energieverliesen tot 3 keV praktisch alle elektronen over hoeken kleiner dan 10 mrad verstrooid worden.

Hoofdstuk 2, "Instrumentatie voor ESI", begint met het principe van electron spectroscopic imaging. Vervolgens worden de ontwerpparameters voor afbeeldende energie filters behandeld. Deze filters lijden typisch van elektronenoptische aberraties en dienen zorgvuldig geoptimaliseerd te worden. Belangrijk zijn hier de non-isochromaticiteit, oftewel de variatie van de energie in het gefilterde beeld, en de transmissivity, hetgeen een maatstaf is voor de kollektiewe efficiëntie van het systeem. Het hoofdstuk wordt afgesloten met een korte diskussie van de verscheidene filter types.


Hoofdstuk 4, "Ontwerps Specificaties", bespreekt de randvoorwaarden en de specificaties voor het gebruik en de verschillende componenten.


Hoofdstuk 6, "Het Mechanische Ontwerp", bespreekt verscheidene aspecten van het mechanische ontwerp.
Hoofdstuk 7, "Experimentele Resultaten", behandelt experimentele resultaten van 3 JEOL ARM TEMs. De kwaliteit van het filter op 1.25 MeV wordt aangetoond met spectra met 1 eV energieresolutie, met zero-loss gefilterde beelden met 1.1Å resolutie en verwaarloosbare vertekening, met energie-gefilterde beelden en spectra en chemische kaarten.
Acknowledgments

First of all I would like thank Ondrej Krivanek whom I worked for for many years, who inspired and lead the development of the Gatan Imaging Filter, and without whose guidance none of the projects I worked on would have been as successful.

I greatfully acknowledge Gatan Inc. for allowing me to publish the work performed at Gatan in this thesis. Many thanks are due to all Gatan employees, who each in their way contributed to this work. In particular I would like to thank Harold Brink, Dan Bui, Pete Burgner, Talbert Choye, Niklas Dellby, Stuart Friedman, Bernd Kraus, Mike Kundmann, Chris Meyer and Daniel Ray.

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Sander Gubbens

Walnut Creek, December, 1996
Curriculum Vitae

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1983 Graduation Atheneum, College Hageveld, Heemstede

May 1988  Ingenieur in Applied Physics

Technische Universiteit, Delft, The Netherlands

May 1988 - present  Start employment at Gatan Inc., Pleasanton, California, USA.

May 1988  Development of ion guns for Gatan specimen preparation equipment.

March 1989  Development of a High Voltage Parallel Electron Energy Loss Spectrometer for operation up to 1.3 MeV.

March 1990  Development of the Gatan Imaging Filter, a post-column imaging energy filter for use up to 400 keV.

September 1992  Development of the High Voltage Gatan Imaging Filter GIF, a post-column imaging energy filter for use up to 1.25 MeV.
## Appendix A

### Energy, momentum, velocity, dispersion

<table>
<thead>
<tr>
<th>Energy (keV)</th>
<th>Momentum (kg m/s)</th>
<th>Velocity (m/s)</th>
<th>$\beta = v/c$</th>
<th>$k$</th>
<th>Dispersion (µm/eV)</th>
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<td>100</td>
<td>1.79E-22</td>
<td>1.64E+08</td>
<td>0.548</td>
<td>5.445</td>
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Appendix B

Patents

The (High Voltage) Gatan Imaging Filter is protected by the following US patents:

Patent #:4,743,756 Parallel-Detection Electron Energy Loss Spectrometer
Patent #:4,851,670 Energy-Selected Electron Imaging Filter
Patent #:5,097,126 High Resolution Electron Energy Loss Spectrometer
References

34. S. Lanio, H. Rose, D. Krahl, Optik 73, 56-68 (1986).
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60. Software courtesy B. Kraus, Gatan GmbH.

