

Thin Film Soft X-Ray Absorption Filters

Diploma Paper by

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Abstract

This report discusses the composition, preparation and performance of soft x-ray transmission filters for a water window soft x-ray microscope. Unbacked thin films of aluminum, silver and vanadium/aluminum were made by evaporation on a substrate from which they were released. Measured transmittances agree reasonably well with calculations. The report also includes some related theory and discussions about film preparation methods, film contamination and evaluation methods.

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1. Introduction

This is a diploma paper on thin metal films as soft x-ray filters, their preparation and use in soft x-ray microscopy. In the first sections, the diploma project is put into a context, why soft x-ray filters are needed and what filters are needed. There is an overview of basic theory in Sect. 3 and then there is a short discussion of what filter characteristics can be obtained in Sect 4. This report also contains a description of how a couple of soft x-ray filters were prepared. The performance of these filters are presented and discussed on basis of their application.

1.1 Overview

Conventional visible light microscopes¹ are quite useful. They are cheap, handy and living biological objects can be studied. However, the resolution is limited by diffraction. In order to achieve higher resolution the wavelength of the light in the microscope has to be shortened. X-rays have shorter wavelengths than visible light, hence x-ray microscopes have less diffraction and you get higher resolution than traditional optical microscopes have^{2,3}. Accelerated electrons also have quite short wavelength, which is made use of in Scanning and Transmission Electron Microscopes (SEM:s and TEM:s)¹. A different approach is to avoid the limit imposed by far field diffraction. In Scanning Tunnel Microscopes (STM:s) and Atomic Force Microscopes (AFM:s), a tiny tip is swept over the object examined, and the resolution is not limited by diffraction².

1.2 X-Ray Microscopes, Possibilities and Limits

If you are to study biological objects, it is an advantage if the x-ray microscope you have operate at wavelengths in the so called water window, i.e., $\lambda=23$ to 44 \AA . At these wavelengths water is relatively transparent compared to protein, which give biological objects a natural contrast. Some of the current synchrotron based x-ray microscopes make it possible to study wet biological objects in a non-destructive way with a resolution many times higher than that of a conventional light microscope. Other high resolution imaging methods are not quite suitable to study wet biological objects, even though they are extensively used. Electron microscope specimen needs careful preparations, which are destructive (dehydration, staining and slicing), and the imaging takes place in vacuum^{3,4,5}. With AFM:s and STM:s only the surface structure of the sample is studied.

When living biological objects are studied in an x-ray microscope, the resolution is limited by the x-ray damage to the specimen. Visible light microscopes have a resolution of $0.2\text{-}0.3 \mu\text{m}$ at best due to diffraction⁵, while x-ray microscopes with biological objects have a maximum resolution of $10\text{-}20 \text{ nm}$ ⁴. Structural damage in tissue starts to appear at an x-ray dose that corresponds to a resolution between 10 and 50 nm (with $\text{SNR}\approx 6 \text{ dB}$)⁵.

1.3 X-Ray Microscopes

The image from an x-ray microscope can be formed in a number of different ways. The currently existing main types of x-ray microscopes are contact, full field, scanning and holographic^{3,4}.

In contact x-ray microscopy the specimen is put in contact with a photoresist and then exposed, i.e., irradiated with x-rays. After development, the resist has a relief pattern that reflects the x-ray transmittance of the specimen, and the developed resist can be studied in a SEM. This imaging method has given the highest resolved pictures of the x-ray microscopes³, but the resolution of the specimen depend on its thickness. "Thick" objects are imaged with a considerably lower resolution due to diffraction. If a photocathod is used instead of the resist and is followed by an emission electron microscope, the process of imaging becomes easier to the price of degraded resolution^{4,5}.

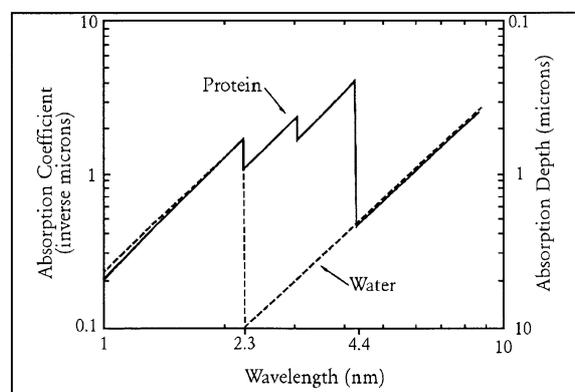


Figure 1. The water window. $I=I_0e^{-\mu x}$, where μ is the absorption coefficient. From Ref. 5.

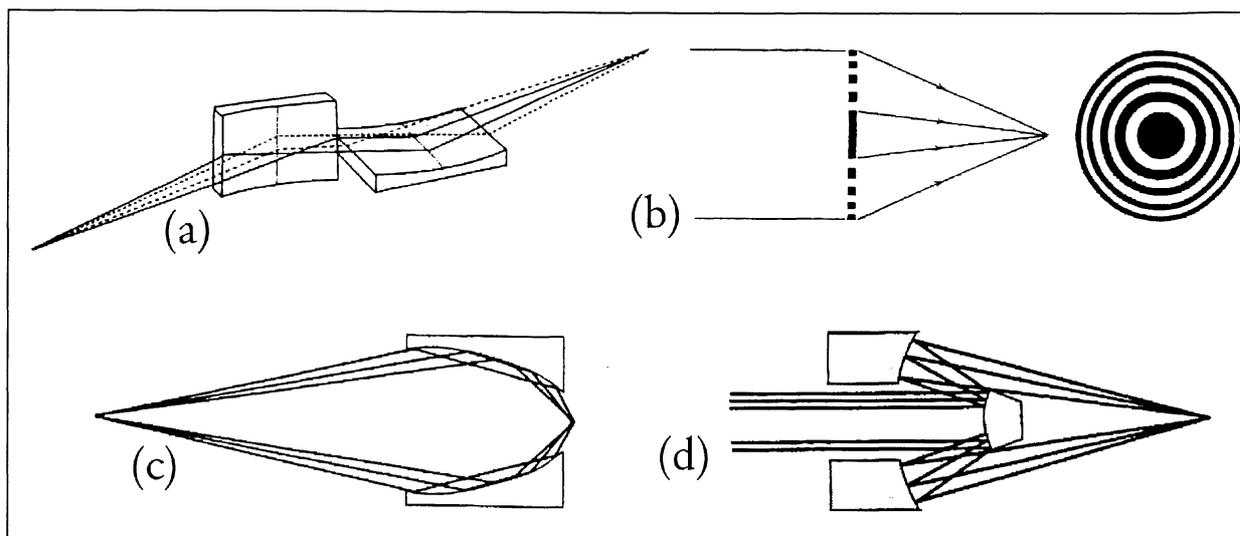


Figure 2. X-ray optics:objectives. (a) Grazing incidence. Two cylindrical (or spherical) mirrors. Kirkpatrick-Baez. (b) Grazing incidence. A combination of a hyperboloid and an ellipsoid. Wollter. (c) Diffraction grating. The circular zones are alternating transparent and opaque. "Zone plate". (d) Multilayer mirrors. Two nearly concentric spherical mirrors. Schwarzschild. From Ref. 5.

Holographic x-ray microscopy² is somewhat similar to contact x-ray microscopy, but the photoresist record the far field instead, and a reference beam is also included. How the reference beam is recorded depend on what geometry is used. Reconstruction can be numerical or optical. A resolution of less than 100 nm has been achieved, but a resolution of 10 nm is regarded as the ultimate limit². The ultimate limit is determined of the radiation damage to the biological object and of the resist resolution. Coherence requirements, exposure times, insufficient resolution and a complicated image process are drawbacks which currently make other imaging method more attractive. It is hard to say how holographic imaging with x-rays will perform in the future since development in several of areas (x-ray sources, computing) have impact on holographic imaging.

The full field x-ray microscope work in the same way as a conventional visible light microscope does, i.e., a full picture is recorded instantaneously. With a scanning x-ray microscope, a single spot of the specimen is irradiated at a time and the transmittance is measured. To obtain a picture, the sample is scanned. When a sample is scanned with a focused x-ray beam, fluorescent x-rays are produced and photoelectrons are emitted. These x-rays and electrons can be detected and can give information about the composition of the sample, but the electrons only about the surface. With full field and scanning x-ray microscopy, the depth of focus can be chosen, in contrast to contact imaging. To scan a picture takes time, which is a disadvantage if the object is moving or if you want to catch a dynamic process in progress. But the brighter the source is, the faster the scan can be made. For a picture with a given resolution, a specimen imaged in a scanning microscope is recorded with a lower x-ray dose than a specimen imaged in a full field microscope. Hence, the scanning x-ray microscope has a higher theoretical resolution for biological objects than the full field x-ray microscope⁵.

The scanning and full field x-ray microscopes with zone plates (Figs. 2 and 5) have proven to be successful since zone plates presently give the best resolution of existing x-ray objectives. Best resolution achieved is 50 nm^{3,5,6}. Unfortunately, zone plates also have severe chromatic aberration and requires monochromatic x-rays in order to be useful, which is a disadvantage⁶. Other types of x-ray optics (Fig. 2) do not have as much chromatic aberration and also have higher theoretical transmittance, but currently existing x-ray microscopes with these types of optics also suffer from lower resolution^{5,6}. Manufacturing problems and difficulties in alignment result in the lower resolution, which disqualify the alternatives.

1.4 X-Ray Sources

Currently existing x-ray microscopes with a resolution to speak of, except for contact x-ray microscopes, all have synchrotron rings as their x-ray source. This radiation source can be very bright and can be made

even brighter with a wiggler or an undulator (essentially the same thing). The brightness of synchrotron light is one explanation behind recent successes in x-ray microscopy, since high resolution x-ray optics has very low efficiency - 10% percent of transmission is considered normal or even good - and thus requires a large flux of photons.

A synchrotron is basically an evacuated path where electrons move in orbit at high speed, kept in place with magnetic fields. Generally, accelerated electrons emit radiation and electrons in circular orbit, close to the speed of light, emit radiation in a certain manner. The spectra is continuous, and a monochromator has to be used if monochromatic light is needed. A combination of a grazing incidence monochromator and a zone plate is detrimental to photon flux, but when the synchrotrons were built that problem was overcome.

The x-ray microscope that is being developed at Lund Institute of Technology will have a Laser Produced Plasma (LPP) as x-ray source. There are many different x-ray sources, but next to synchrotrons, LPP:s are the brightest⁵. The Lund microscope will be compact ("table top") and fairly cheap and therefore more accessible to biologists. A synchrotron able to generate x-rays is a huge staffed factory-sized installation for an enormous amount of money, which has to be booked long in advance. An x-ray microscope with a LPP x-ray source will be much cheaper and smaller, i.e., more accessible.

1.5 Scope

Just like synchrotron x-rays, LPP x-rays are far from monochromatic. The Lund microscope will - among other things - use a zone plate for focusing and a scanning mechanism for the specimen, a combination proven to function well. The source will probably not be bright enough for full field imaging. If a zone plate is to be used, something has to be done to the spectra. The spectra generated of the LPP is not continuous in the water window and a filter might be more suitable than a grazing incidence monochromator. For x-ray filtering, thin metal films can be used. The primary scope of the diploma work presented in this diploma paper, is to analyze the x-ray system and to design and prepare x-ray filters for the system in shape of thin metal films. Other uses has also been found for thin metal films and they are described below.

1.6 Thin Metal Films

Unbacked metal films are routinely made at many laboratories and at a few companies, but in general they are expensive to buy and have limited lifetime. The ability to customize and make thin metal films is therefore a valuable resource - also since thin metal films have many uses. In short they can serve as filters, contamination barriers and pressure barriers.

How to make thin metal films is well known, and unbacked metal films has also been made for quite some time. The metal is evaporated or sputtered onto a substrate, then the film is parted from the substrate or the substrate is dissolved. Some kind of support may be used, like a fine metal mesh or a thin plastic film. The film is mounted on a holder with a small aperture or a narrow slit - what ever you want, as long as the film does not break. How to prepare a thin metal film is described in Sections 5 through 7.

Thin film transmittance depend on the wavelength of the light, unless the wavelength is not too short. So called hard x-rays need more than a thin film to be absorbed (cm of lead instead of nm of aluminum), while soft x-rays are easily absorbed in air. Accordingly, thin films can work as filters for soft x-rays and for light of longer wavelengths. Soft x-ray interaction with matter is described more in detail in Sect. 3.

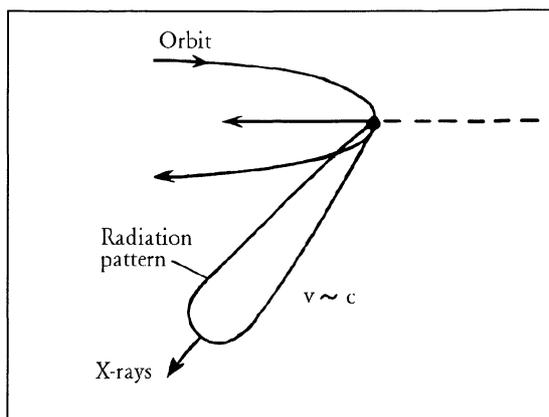


Figure 3. The radiation pattern from a high energy electron in circular orbit. From Ref. 7

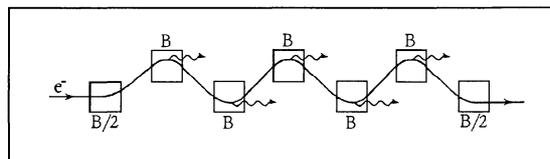


Figure 4. A multipole wiggler. The magnetic fields, B, accelerate the moving electrons. From Ref. 7.

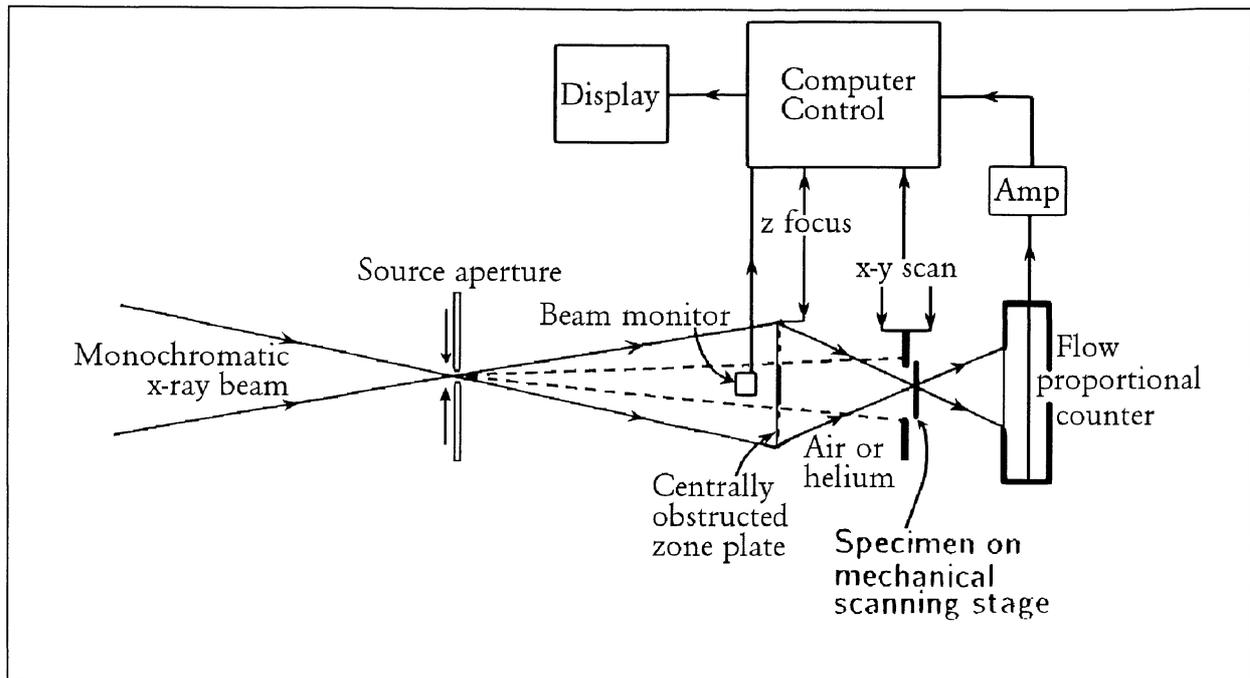


Figure 5. A scanning x-ray microscope does not have any inefficient x-ray optics between the specimen and the detector as a full field x-ray microscope has. This minimizes the exposure time. Hence, a not so bright x-ray source may be used. From Ref. 6.

Soft x-rays may loosely be defined as electromagnetic waves with wavelengths between 0.5 and 10 nm, i.e., the water window falls into the soft x-ray region. At wavelengths between soft x-rays and visible light you also have extreme ultra violet (XUV), vacuum ultra violet (VUV) and ultra violet light (UV). All these regions overlap and are loosely defined. Most detectors are sensitive to a broad range of wavelengths, and thin films may be used to pick the signal of interest. For example, thin aluminum films are used in satellites to filter the spectra of the sun to extract helium emission lines and thin films can be used for sorting out different orders in spectrographs.

The mechanical properties of thin metal films are in the focus of interest for some applications. A film can be used to separate vessels of slightly different pressure (typically a few Torr), and to keep contamination away from detectors. Aluminum films are basically transparent in the water window and may be used to block LPP generated debris, which otherwise would destroy x-ray optics.

2. System Requirements on the X-Ray Filter

To find out what is required from the x-ray filter, the Lund x-ray microscope is analyzed more in detail below.

2.1 The Optics: a Zone Plate

A zone plate⁶ is a number of concentric transparent circles in an opaque plate. The width and the spacing of the concentric transparent circles are designed to give constructive interference at focus. The zone plate acts as a circular diffraction grating. The width of the alternating transparent and opaque circles decrease as the distance from the center increase - all the circles have the same area.

Just like a grating has several orders of diffraction, a zone plate has several focuses for a given wavelength. The first order focus is about as big as the outermost transparent circle is wide. Higher order focuses decrease in size as $1/m$, and the peak intensity decrease as $1/m^2$, where m is the diffraction order. Intensity also increase with the size of the zone plate. Because of cancellation, only odd numbers of diffraction orders appear, except for the zeroth order - undiffracted light. Hence, the first order focus has the highest peak intensity, which usually is more important than to get a slightly smaller focal spot. In

addition to the first order focus, there is a background, mainly from the zeroth order. The background noise can be reduced with a central obstruction in the zone plate and with an aperture at the focal spot.

A zone plate requires monochromatic x-rays since x-rays of different wavelengths have focuses on different depths, i.e., chromatic aberration.

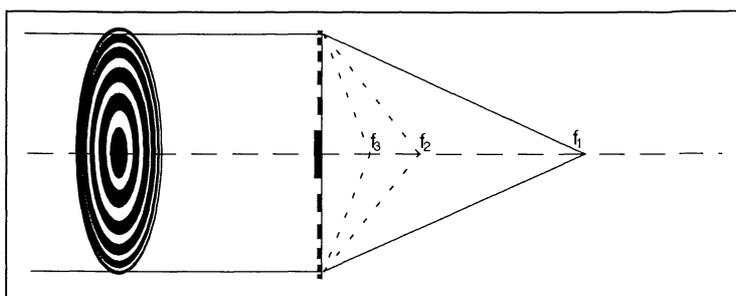


Figure 6. A zone plate. The different focuses.

Assume you have x-rays with the wavelength λ and the bandwidth $\Delta\lambda$. For a given zone plate, the focal length depend on the wavelength as $1/\lambda$ and the difference in focal length, Δf , is proportional to $\Delta\lambda/\lambda^2$. The transverse chromatic aberration is approximately proportional to $\Delta\lambda/\lambda$. If the light is not monochromatic, the performance of a zone plate is seriously degraded because of a drastical increase in background noise and/or larger focal spot. Different analyses lead to slightly different results, but both the transverse and the longitudinal chromatic aberration depend on the number of zones as $1/N$.

2.2 The Spectra from a LPP

A plasma is produced if a high peak power laser pulse is focused on a target⁵. The electric field of the laser pulse primarily accelerate the electrons, and as unbound electrons collide with atoms and ions, more electrons are torn loose. The electrons also distribute the heat through the plasma and the plasma rapidly expand - the hot negative electrons move with high velocity among the positively charged nucleuses. Since the negatively charged electrons collide with and are accelerated by the positively charged nucleuses, radiation is produced. This radiation is called bremsstrahlung and results in a continuous spectra. Continuous radiation also appear when the electrons recombine with nucleuses and ions. Later, when the excited ions and atoms decay, discrete lines appear in the spectra. Hence, the total spectra from a plasma is the sum of continuous radiation and discrete lines.

The spectra from a plasma⁵ depend on a number of parameters: laser pulse length, its intensity and its wavelength, target material and the angle between the detector and the laser beam. There are no clear rules on how to design a system to get a certain kind of radiation. However, nitrogen and carbon have strong K-shell emission lines in the water window. A single emission line is narrow and may be as monochromatic as a zone plate requires to give high resolution. Also, continuum emission can be kept low with these targets if the laser intensity is not too high (less than 10^{14} - 10^{15} W/cm²)⁵.

In Lund a C & O target has been tried, but will later a N target with O contamination will be used. The N, C and O lines expected to be most intense are listed in Table 1⁹. Any continuum radiation will be of longer wavelengths than the water window and line radiation will appear where ever nitrogen or carbon have emission lines. Just a few oxygen lines (O-VIII lines) will be below the water window. The water

Figure 7. A LPP spectra recorded with a cassette tape as target, i.e., a C & O target.⁸

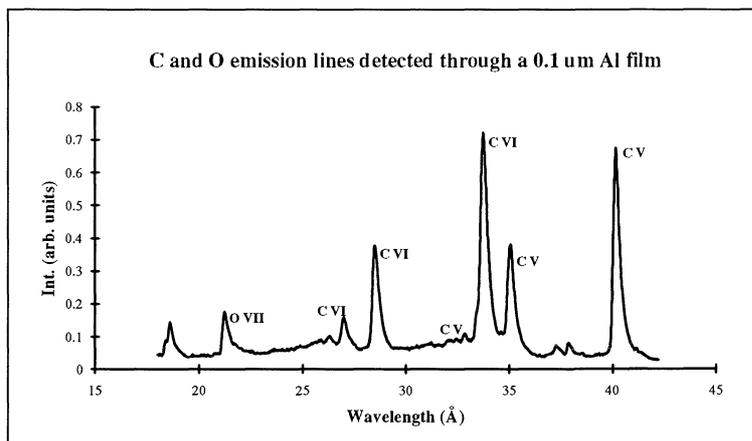


Table 1. Strong emission lines for C, N & O in the water window and below.

C	N	O
33.7Å	24.8Å	17.4Å
35.0Å	24.9Å	17.8Å
40.3Å	28.8Å	18.6Å
		19.0Å
		21.6Å
		21.8Å
		22.1Å

window spectra recorded with the C & O target is presented in Fig. 7. The monochromator does not have high enough resolution for the line widths to be determined. Note that the oxygen lines below the water window are not resolved.

2.3 The X-Ray Detector

A photon that has travelled through the specimen arrives at the detector: a high gain and low noise electron multiplier tube. The multiplier has a CsI photo cathode, with a quantum efficiency of approximately 1 in the water window. The long wavelength cut-off for CsI is about 2000 Å, where the quantum efficiency is below 0.25% (fig. 8). For example, if you expose this detector to visible light only, there will be no signal. Hence, x-ray filter transmittance above 2000 Å does not matter, there will be no signal anyway.

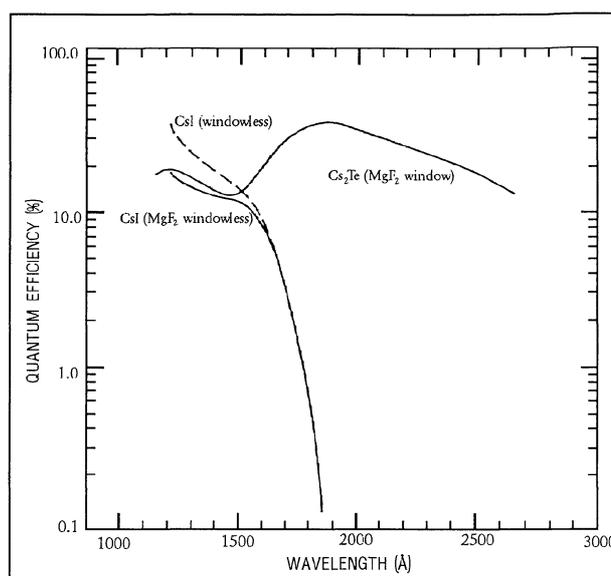


Figure 8. Long wavelength cut-off for the windowless detector. From Ref. 10.

2.4 The Ideal X-Ray Filter

How monochromatic the x-rays has to be in order not to degrade the resolution clearly depend on the design of the microscope in general and the design of the zone plate in particular. On the other hand, if source spectra is given, the zone plate design can be adjusted to give the highest possible resolution. The Lund microscope will utilize an emission line from either nitrogen or carbon. Hence, the line width in combination with the best zone plates that can be made presently, will together put the ultimate limit to the x-ray microscope resolution. At a second glance you realize that an emission line in the lower end of the water window is advantageous because of less water absorption (fig. 1), hence thicker biological objects can be studied. The filter design is then reduced to pick an emission line from the spectra of either nitrogen or carbon, and to suppress all other wavelengths, i.e., a bandpass filter is ideal.

Since nitrogen has emission lines in the water window of shorter wavelength than carbon, nitrogen is preferred. The line at 25 Å would be best and the line at 28 Å would be the second best. Especially the continuum radiation at longer wavelengths than the water window has to be removed because of the high total power in it, but also oxygen emission lines at shorter wavelengths than the water window and nitrogen lines not selected should be reduced.

3. Soft X-Ray Interaction with Matter

In order to arrive at methods on how to calculate thin foil transmittance, the interaction of soft x-rays with matter is reviewed in this Section. The material is adapted from Ref. 7 and 11.

3.1 Scattering and Absorption

Soft x-rays in matter are more or less absorbed and scattered. Absorption mainly occur via the photoelectric effect and the atom is ionized. A photon is absorbed when all of its energy is transferred to an inner shell electron and the atom is freed from one of its electron. Scattering is either elastic or inelastic. In elastic scattering, no energy is transferred from the x-rays to the matter and you speak of Thomson and Rayleigh scattering. In Thomson scattering each electron is regarded as an independent scatterer and you only take the electron density into account when you calculate scattered power. With Rayleigh scattering, the co-operative effect of all the electrons in an atom is also taken into account. Thomson and Rayleigh scattering is coherent, while inelastic, or Compton scattering is incoherent. In Compton scattering, a photon transfers a small fraction of its energy to a loosely bound electron. Of these

three processes, photoelectric absorption is by far the most important when calculating thin foil transmittance in the soft x-ray region. Attenuation due to scattering can be neglected at these photon energies.

3.2 Linear Absorption Coefficients

When x-rays of intensity I_0 travel through material with the thickness x , they are exponentially attenuated⁷:

$$I = I_0 e^{-\mu x}. \quad (1)$$

The attenuation depend on the linear absorption coefficient μ . There are several of alternative coefficients to μ , for example the mass absorption coefficient μ_m , a somewhat more useful coefficient because the density dependence is eliminated¹².

$$\mu_m \rho = \mu \quad (2)$$

Soft x-ray photons have sufficient energy to tear an inner shell electron from an atom and outer shell electrons of the atom are not involved in the absorption process. Hence, neither the phase of the material nor its chemical state influence the absorption coefficient very much and the mass absorption coefficient may be used at soft x-ray energies.

The absorption coefficient strongly depend on the wavelength in the soft x-ray region. There is no easy way to get the absorption coefficients as function of wavelength. They can be calculated from the atomic scattering factors, denoted f , for each wavelength and tables have been compiled for all the elements by for instance Henke, and they are available on disk¹⁵.

3.3 The Atomic Scattering Factor

An x-ray beam can be regarded as an electromagnetic wave. An electron in the path of the wave is mainly accelerated of the electric field. The speed the electron is accelerated to is low, and one of the approximations you can make, is to ignore the influence of the magnetic field. An accelerated charge emits radiation, and an analysis gives that the amplitude of the electric field in the emitted wave is¹²

$$E_T = -E_0 \frac{e^2}{4\pi\epsilon_0 m_e c^2 r} \cos \Phi, \quad (3)$$

where E_0 is the amplitude of the incident field, r is the distance from the charge to the place where the field is observed, and Φ is the angle between the incident x-ray beam and r . See Fig. 9. The process described is called Thomson scattering and the scattered field is denoted E_T . Notice the mass dependence; a nuclei can be regarded as stationary compared to an electron. The wave scattered in forward direction ($\Phi=0$) has a phase opposite to the incident wave.

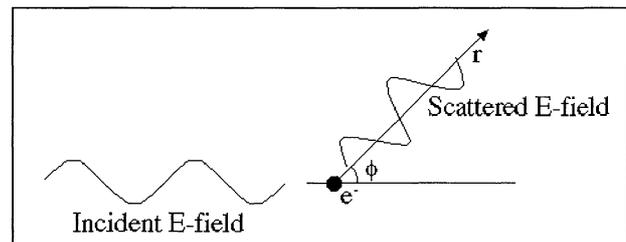


Figure 9. The scattered field of a point charge.

To get the field scattered by a whole atom, Rayleigh scattering rather than Thomson scattering should be considered. The field scattered of an atom is related to the field scattered of an electron by the atomic scattering factor⁷:

$$f = \frac{\text{amplitude scattered by an atom}}{\text{amplitude scattered by a free electron}}. \quad (4)$$

Approximately, the atomic scattering factor is angle independent since x-ray wavelengths are long in comparison to the electron distribution in an atom. You say that the electrons in the atom scatter in phase. The expression is possible because of the inherent coherent nature of the scattering. To get the phase relation between incoming and scattered wave, f is a complex number. Hence the field scattered from an atom is^{7,11}

$$E = E_T f = E_T (f_1 + if_2). \quad (5)$$

The atomic scattering factor can be calculated in relativistic quantum dispersion theory⁷. For Thomson scattering, f_1 is equal to the atomic number Z , and it agrees with reality when the photon energy is much larger than the largest ionization energy for a particular atom. But the more detailed calculation results in two more terms which are essential in the soft x-ray region. One of these two terms is an integration over photon energies of an expression containing the photoionization cross section, and the other is a relativistic correction term. The imaginary part of f , i.e. f_2 , is proportional to the photoionization cross section, the variable of interest.

One may wonder how scattering relates to absorption. The material is slightly polarized by the incident electromagnetic wave, because the electrons in an atom are displaced, while the nuclei is stationary. The scattered field is entirely generated of the time-varying electron positions, and the displacement of the electrons can be calculated from the electron positions. Hence, the electron displacement, i.e., the polarization, can be calculated from the atomic scattering factor, since f in a more or less intrinsic way describes the electron position. From the polarization, the dielectric constant and the refractive index can be calculated, and it is clear that the absorption coefficient can be derived from f . To keep the discussion short and simple, the result is presented without further motivation^{7,11}

$$\mu = \frac{e^2 \lambda}{2\pi \epsilon_0 m_e c^2} n f_2, \quad (6)$$

where n is the number of electrons per unit area. The material must not be a single crystal for Eq. 6 to be true. Interference effects might for occur in crystals, because the scattering is coherent. The relation is also more correct for small angles to the incident x-ray beam because f is not completely angle independent.

3.4 Features of the Absorption Spectra

Inner shell electron binding energies in an atom fall into the same range as soft x-ray photon energies. The shell structure of atoms influence the absorption as function of wavelength and give the soft x-ray absorption spectras a special character¹³. Absorption have sharp edges where photon energies correspond to electron binding energies. At photon energies a little less than the electron binding energy, the absorption is relatively low compared to photon energies a little higher than the electron binding energy.

Photons with higher energy than soft x-ray photons are not easily absorbed, hence they are called hard x-rays. How photons with lower energy than soft x-rays interacts with matter, very much depend on the state of the matter, chemical bonds, e.t.c. Metals, for instance, have a conducting band, which usually make them reflective at longer wavelengths, which is quite apparent at visible wavelengths.

4. Attainable X-Ray Filters

It should be clear from the previous section, that filters with bandpass characteristics are impossible to obtain in the soft x-ray region, at least as long as they work on the principle of absorption. The emission lines from nitrogen at $\lambda=25 \text{ \AA}$ or 28 \AA should be as close as possible to an absorption edge, with the absorption edge at slightly shorter wavelength so that the filter is relatively transparent where the emission line is.

4.1 Discussion

If the $\lambda=25 \text{ \AA}$ line is to be used, the absorption edge should be between 25 \AA and 22.1 \AA , where the most nearby emission line from oxygen is. The 28 \AA line should be attenuated, which is harder to do. The only way to attenuate the 28 \AA line is to have a relatively thick filter. Between absorption edges, the absorption coefficient is basically linear (there are lots of exceptions) and because the absorption depend on the absorption coefficient exponentially, a thicker filter attenuate the 28 \AA line more than the 25 \AA line. But the 25 \AA line is also seriously weakened if you want to achieve a difference between the lines of relevance. Hence, the x-rays can be made as monochromatic as desired, but at the expense of reduced intensity. Since the 28 \AA N line is weaker than the 25 \AA N-line from the beginning and since they focus on different depths with a zone plate, the problem might be overcome. The filter is made just thick enough to attenuate the oxygen lines and an axial aperture is placed at focus to reduce the background from the 28 \AA N line.

A way to get round the problem with the 25 \AA line is to make use of the 28 \AA N-line instead. There is an absorption edge somewhere between 25 \AA and 28 \AA , and the 25 \AA line is attenuated. Also the oxygen lines are suppressed, almost as much as the 25 \AA line. At wavelengths longer than the 28 \AA , there are no emission lines below 60 \AA from neither oxygen nor nitrogen, and no strong emission lines below 80 \AA . It should be possible to obtain a filter that is virtually ideal.

4.2 Which Nitrogen Line to Use

Which of the two nitrogen lines at 25 \AA and 28 \AA that will be best, depend on the final system design and what the spectra actually will look like. If it was possible to make a filter with exactly the characteristics you wanted, the 25 \AA line would be best. Even though you cannot make a perfect filter, the 25 \AA line is stronger and at shorter wavelength. But in the end the 28 \AA line might be better anyway, because the x-rays will be more monochromatic after filtering.

4.3 Elements in Question

Figure 10 depicts all the elements with an absorption edge in the water window. In Table 2 the elements with absorption edges in the region between 22.1 \AA and 28 \AA are listed^{14,15}.

A couple of these should be avoided if possible: tellurium and antimony are very toxic. Besides, tellurium - and chromium too - have their absorption edges too low to suppress the 22.1 \AA oxygen line for sure. Oxygen is not a metal and might be hard to include in an unbacked film. A gas filter is not totally out of the question, but it is beyond the scope of this work. Tin has a very smooth absorption edge and does not make as significant difference between the two nitrogen emission lines as vanadium and titanium do. Hence vanadium and titanium are the most promising candidates.

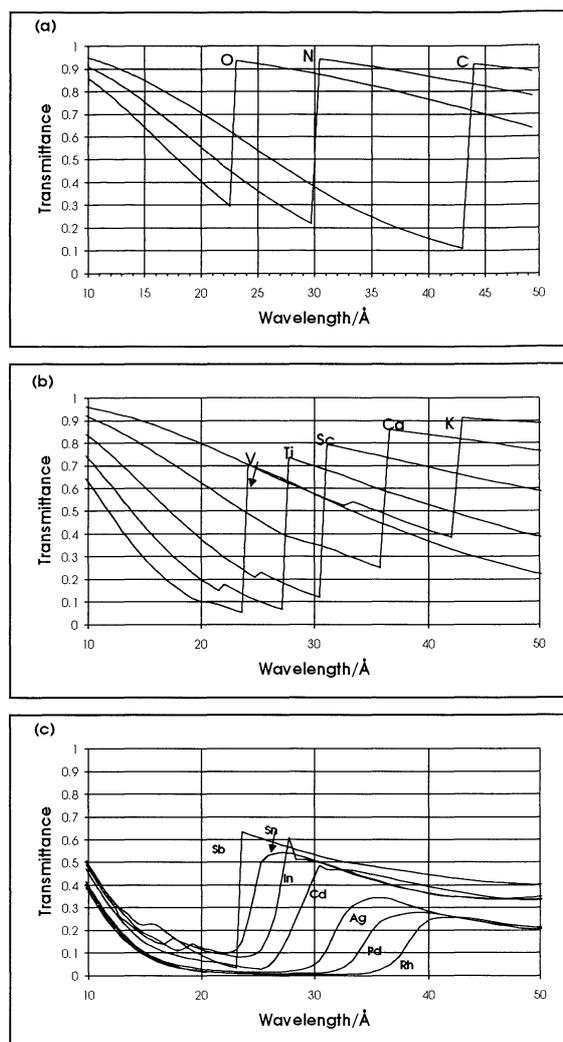


Figure 10. Absorption edges with 200 nm thickness. a. K-edge. b. L-edge. c. M-edge. Calculated with data from Ref. 15, assuming bulk density. Carbon K-edge for 200 nm of graphite. Oxygen and nitrogen K-edges with same numbers of atoms per unit area as for 200 nm of carbon.

Table 2	Element	Absorption edge wavelength/ \AA
K-edge	O	23.3
L-edge	Cr	21.6
	V	24.1
	Ti	27.2
M-edge	Te	21.5-22.0
	Sb	23.1-23.6
	Sn	22.0-27.1

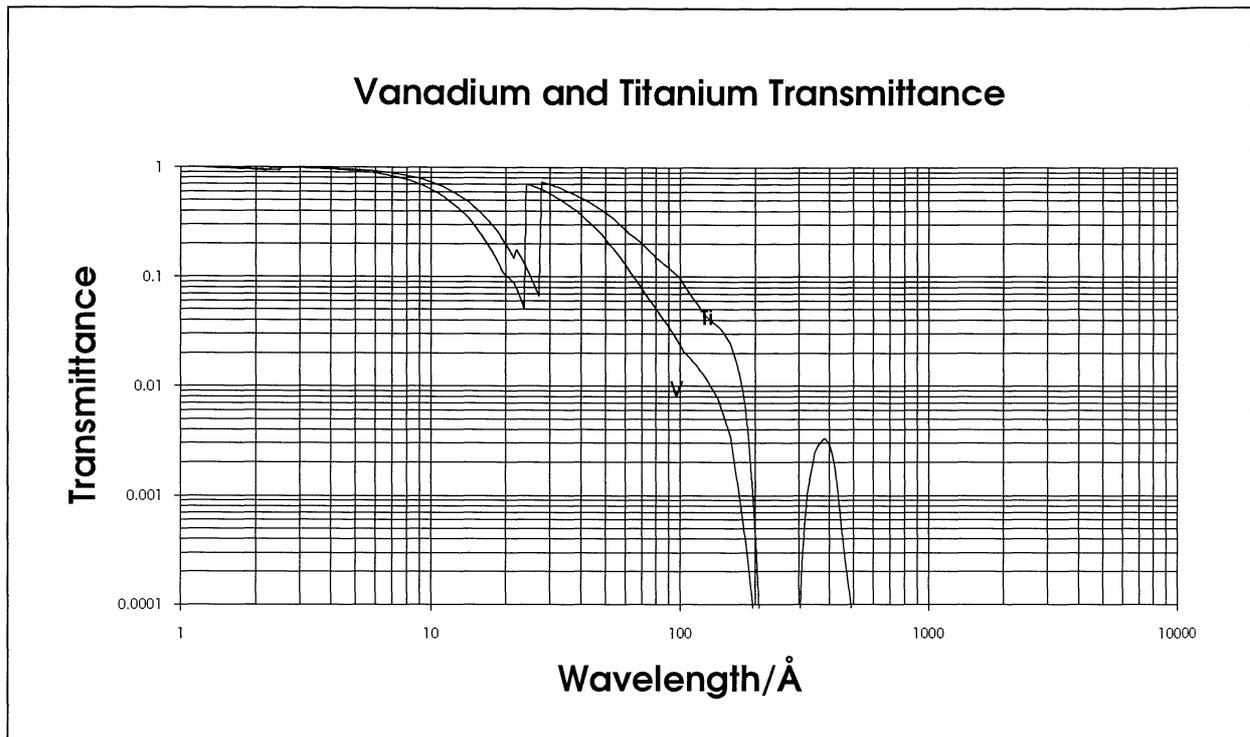


Figure 11. Transmittance for Ti and V. Calculated with data from Ref. 15.

4.4 "Long" Wavelength Considerations

Apart from the line radiation, continuous radiation will appear. The continuous radiation is expected somewhere above the water window, and will stretch all the way to and through the visible region. Below 2000 Å, the detector is sensitive and the intensity might be fairly strong, and except of a single emission line, the filter should be opaque. From the C & O target, almost no continuous radiation was detected below 2000 Å. Only line emission was observed, probably from iron too, since the target support was made of iron. The nitrogen target will be quite different from the test target, and the exact appearance of the spectra is impossible to predict.

Transmittance for a wide range of wavelengths is plotted for vanadium and titanium in Fig. 11 with data from Henke¹. Data compiled by Henke covers wavelengths less than 1200 Å and transmittance data has to be obtained from somewhere else for wavelengths 1200-2000 Å. Metals become reflective at longer wavelengths, and this wavelength can be estimated with the so called plasma frequency, denoted f_p . Tenuous plasmas and metals in some respect exhibit a similar behaviour for optical wavelengths and shorter, which make them reflective. The plasma frequency can be calculated with¹⁶

$$\omega_p = \sqrt{\frac{ne^2}{\epsilon_0 m_e}} \quad (7)$$

where n is the density of electrons taking part in the oscillations caused by the incident electromagnetic field. There is an uncertainty in n , but if you sum all the electrons in the valence bands, you get a fair

Table 3

Element	Ti	V	Al
hf_p/eV	18	22	16
$\lambda_p/\text{Å}$	689	556	775

estimation. Instead of making corrections to n , you can speak of the effective mass of the electron. If you take n as the number of electrons in the valence band per unit volume and m_e as the electron mass, you get a rough estimation for the onset of transmission. Usually f_p is below 1000 Å for metals, see Table 3 (calculated from Eq. 7).

You can argue that metals become fairly reflective at wavelengths above 1000 Å. But it is only true for a few metals. Silver, for instance, is relatively transparent between 3000 Å and 3500 Å, due to the shell structure. The discussion of a certain frequency for which a metal becomes reflective is seductive; there are no sharp boundaries. Figure 12 is a summary of transmittances, and the region from 1000 Å to 2000 Å is covered. Vanadium has a transmittance very similar to titanium and chromium.

4.5 Conclusion

Clearly, an unbacked vanadium film would be suitable as x-ray filter to emphasis the 25 Å nitrogen emission line, and a titanium film is perfect to extract the 28 Å nitrogen emission line. Vanadium and titanium films are also opaque all the way to 2000 Å, if they are not made too thin.

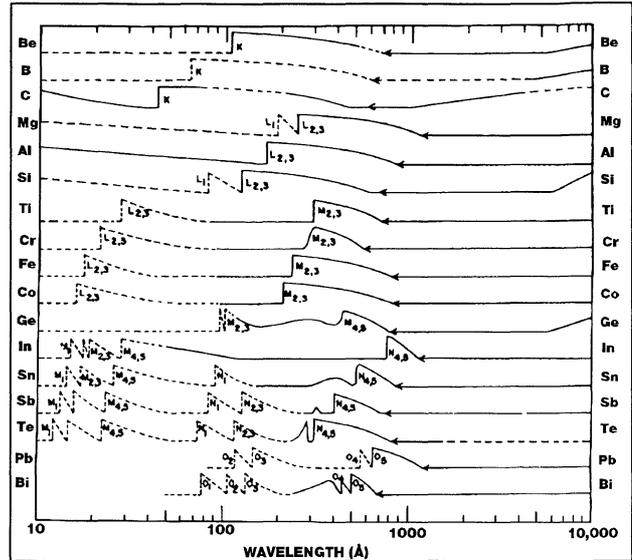


Figure 12. Transmittance for a number of elements. From Ref. 17.

5. Preparation of Unbacked Thin Films

Thin metal films are prepared in vacuum either by sputtering or by evaporation. In an evaporator, the evaporant is either resistance heated or heated with an e-beam. The resistance heating is either direct, i.e., a current is lead through the evaporant, or indirect: a current is lead through the support of the evaporant. At the Division of Atom Physics, Lund Institute of Technology, where this project was done, an evaporator was built in the late seventies. As part of the project, the evaporator was slightly modified. For instance, a thickness monitor was installed.

5.1 The Evaporator

The vacuum chamber is a glass bell jar on a steel base plate. The bell jar is lifted off when you want to reach inside the vacuum chamber. In the middle of the base plate there is a pumping port, and the pumping system consists of an oil diffusion pump and a rotary pump. Between the vacuum chamber and the diffusion pump there is a liquid nitrogen cooled baffle, which prevents oil from the diffusion pump to reach the vacuum chamber.

In the vacuum chamber there are three water cooled electrodes. A low voltage can be applied between any two of the three electrodes. If a tungsten foil is connected to the two powered electrodes, a large current will flow through the circuit, and the foil is heated. The circuit on the secondary side of the transformer can at most support 200 A. To have a flexible connection, there is a copper clamp on each electrode. The copper clamp can be adjusted to the height and the angle desired, and evaporation sources of different designs can be fitted in between the electrodes. The power can be switched between any pair of electrodes while vacuum is maintained. Substrates, where you want to deposit the material you evaporate, are mounted upside down above the evaporation source. There are a couple

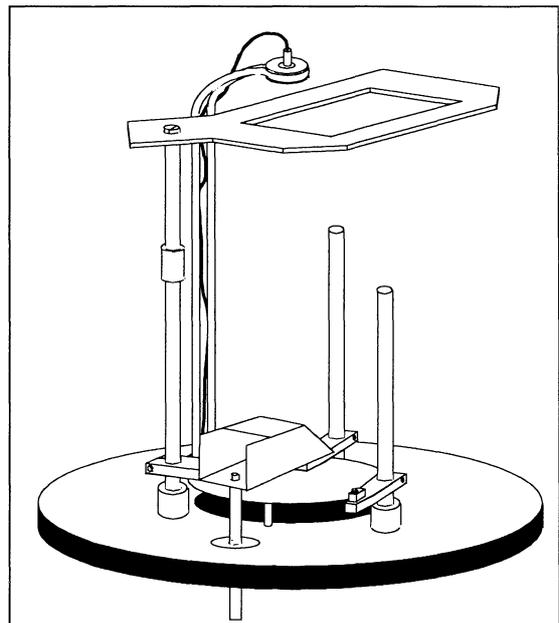


Figure 13. The evaporator without the bell jar.

of different substrate holders for different types of substrates. Right beside the substrate holder, there is a crystal thickness monitor¹⁸. The crystal is supported of a cooling water pipe, and the cooling water pipe is mounted in the base plate. Between the evaporation source and the substrate there is a shutter, with a feedthrough in the base plate.

5.2 Evaporation Sources

What source^{19,20} you choose depend on what material you are going to evaporate (see Appendix D) and the current required of the source in order to get hot enough. The resistance of the source must be low, since the transformer cannot produce a high voltage. On the other hand, if the resistance is too low, the source might not get hot enough even though largest possible current is supplied. Foils and

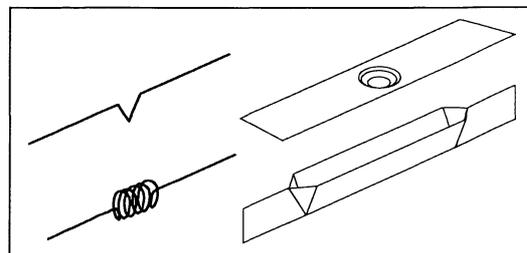


Figure 14. Some types of evaporation sources.

wires made of refractory metals are the most common sources, see Fig. 14 for examples of sources. Refractory metals (with refractory metals I mean tungsten, molybdenum, and tantalum) have the necessary qualities to be good evaporation sources, see Table 4. You can also connect wires or a rod made of the material you want to evaporate between the copper clamps and let the current flow directly through the material. Another way to avoid refractory metals, is to use a coated source or a crucible made of a refractory oxide, boron nitride or carbon. Crucibles can support a larger amount of the material to be evaporated than foils and wires, but if the crucible is made of boron nitride or a refractory oxide, the crucible is non conductive and must be heated indirectly slowly and carefully and a fair amount of power and some extra time is required. A more thorough overview can be found in Ref. 19. I have used tungsten wires and dimpled foils of tungsten and molybdenum. The sources may not have been perfect in every aspect, but - with some exception - they were good enough.

5.3 The Thickness Monitor

The thickness is measured with an oscillating crystal^{19,20,21}. As metal deposits on the crystal, its resonance frequency changes. As thickness increase the resonance frequency decrease. To a first approximation, the deposited mass is linearly proportional to the change in resonance frequency, but a more refined model gives a dependence also containing the acoustic impedance. The improved model becomes essential as deposition grows thick, since linearity decrease as the resonance frequency departure from the original resonance frequency. The control box, which contains a computer, calculates the film thickness from the difference in resonance frequency to the original resonant frequency and the difference to resonance frequency prior to evaporation. The resonant frequency of the crystal also depend on its temperature, hence the cooling diminish the impact of radiation heat from the evaporation source. It is worthwhile to notice that the computer first calculates the deposited mass, which then is converted into thickness.

5.4 Evaporation Procedure

The glass bell jar is lift off and evaporation sources are fitted in between the electrodes. The sources are loaded with the materials to be evaporated and substrates are put into the substrate holders. The glass bell jar is put back on the base plate and a pressure of 10^{-1} Torr is obtained by letting the rotary pump evacuate the chamber. The rotary pump is then connected to the outlet of the diffusion pump and the vacuum chamber is disconnected. When the diffusion pump is hot (after about 20 minutes) and after you have filled the baffle with liquid nitrogen, you open the valve between the vacuum chamber and the diffusion pump. If you have the patience to wait for hours, you can push the pressure down to 10^{-6} Torr, otherwise a pressure of 10^{-5} Torr is easy to obtain.

The shutter should be closed while the evaporation source is heated since any contamination should not reach the substrate. Unnecessary heating of the substrate is also avoided. As you turn the power up, there might be considerable temporary outgassing from the material to be evaporated. After a while, the pressure in the vacuum chamber will start to rise, because the heat from the source make the walls and the equipment in the vacuum chamber outgas more. How much the pressure will rise depend on the

temperature required to evaporate the material. In theory, the pressure should fall if the heat is maintained for a while, but in practice it takes much too long for this to happen. The correct timing of the opening of the shutter depends on the material to be evaporated. If it is a metal, it might be good to wait until the metal has molten. Then there will be a fair vapour pressure from the source.

Upon exposure to the radiation heat, the thickness displayed on the thickness monitor is smaller than the true film thickness. After the shutter is closed and the crystal has cooled for a while, the thickness monitor will show a more correct value. Some experience is needed to know when to close the shutter in order to obtain desired thickness, especially if you evaporate at a high temperature. If the shutter is used to end the film growth, the final film thickness is easier control than if you just turn off the power to the source.

If you want to evaporate some other material on top of a previously deposited film, the power supply can be switched to another pair of electrodes. You save time and you might avoid an oxide layer if you maintain vacuum. When ready, let air into the vacuum chamber, lift off the bell jar and take out the substrates. In Appendix A, there are even more detailed step-by-step instructions on how to evaporate a few materials in the evaporator used in this project.

5.5 Substrates

Several of factors should be considered when choosing a substrate^{17,19,22}. The thin film to be deposited is very thin, hence the substrate must be smooth. If the substrate is rugged the film will at best be rugged, but the film may break upon removal from the substrate. A rugged film is weaker than a smooth film since the thickness is not uniform and might be quite small at some places. The substrate must also be clean to get films with reproducible properties. Contamination can for instance lead to problems with film adhesion and pinholes. It is advantageous if the substrate has high thermal conductivity, which allows tighter control of the process conditions. With high thermal conductivity, the surface can be kept relatively cool even though the evaporation source is quite hot.

Glass is common as substrate for making thin unbacked metal films even though it has low thermal conductivity. Depending on the fabrication process the surface of glass might be quite smooth. Grinding and polishing perturb the surface and should be avoided, like for instance polished metals, even though they are good thermal conductors. Microscope slides have proven to be sufficiently smooth. If you use slides as substrates, they have to be carefully cleaned. In Appendix B cleaning procedures suitable for microscope slides are presented. To circumvent the difficulties of smoothness and cleanliness, mica can be used. Cleave a sheet of mica when you need a substrate, hence the surface is as clean as possible. The mica surface is also smooth.

In order to get an unbacked film, massive soluble substrates should also be considered. Just like mica, you cleave a sheet from a crystal and the advantages are the same as for mica: a clean and smooth surface. Potassium bromide and sodium chloride crystals can be cleaved. They are water soluble and are available on the market from instance Balzers and Agar Scientific.

5.6 Parting Agents

Unless you use massive soluble substrates, you probably need a parting agent^{17,22} to make unbacked films (there are exceptions). The parting agent is applied to the substrate prior to the deposition of the thin metal film. After the evaporation procedure, the parting agent facilitates or enables the release of the film in a suitable solvent. Depending on what parting agent is used, the technique of applying it varies. If the parting agent is evaporated on the substrate or if the substrate is dipped in a solution and then allowed to drain, the layer might be rather uniform. The alternative is application by rubbing, a technique which requires experience. When a parting agent is rubbed onto a substrate, it is easy to remove to much of it, and the layer certainly is not uniform. The most convenient method is probably application by evaporation, since the result is easy to reproduce.

The chemical activity of the parting agent should be as low as possible with respect to the thin film. Small amounts of the parting agent might remain on the film after release and degrade it as chemical reactions take place. When dissolved, a galvanic cell might be formed, where the dissolved parting agent is the electrolyte and the film with its impurities are the electrodes. If the solution of the parting agent is

non-ionic, no galvanic cell is formed, and corrosion is avoided. Also, if the parting agent is neutral, the thin film will not be degraded. Of course, the parting agent must withstand vacuum, high temperature and maybe evaporation without loosing its property as parting agent.

There is a number of parting agents. Researchers seem to have tried what ever they had on the shelf. Hence, if you start trying the chemicals you have accessible, you might find something with the proper qualities. It might be worthwhile to start with commercially available liquid detergents. Rub a drop of the detergent on a slide with a piece of cotton or dip the slide in a solution made from the detergent. Then evaporate some metal on the slide and check if the detergent work as a parting agent. As parting agents I have used Extran MA 02 neutral, a liquid detergent manufactured of Merck and a wetting agent under the trade name of victawet²³. Victawet can be bought from Agar. How to apply these chemicals and related experiences are described in Appendix C.

5.7 Removal of Films from the Substrates

If you have a metal film on an insoluble substrate prepared with a parting agent, the best method to remove the film is probably to slowly lower the substrate into the solvent^{17,22}. As the thin film is released from the substrate, the film should be floating on the surface of the solvent. The release is controlled with the rate the substrate is lowered. To minimize the number of pinholes and the risk to break the film during release, the angle between the substrate and the surface of the solvent should be small. Also the substrate should be lowered with the same speed as the film release. It should look like in Fig. 15. Instead of lowering the substrate, it is more convenient and easier to control the release if you raise the solvent level. Prior to the release you should make an incision through the film along the edges of the substrate, to prevent the film from getting stuck on irregularities on the edges²².

The technique is different if you use a massive soluble substrate²². You place this substrate in the solvent so that the thin film is parallel to and in the same level as the solvent surface. The solvent should have easy access to the substrate, for instance the substrate can rest on a coarse mesh. If the bowl where you release the film is small, the solution might become saturated and you have to renew the solvent. The stress to the thin metal film is less if you use a massive soluble substrate, since the film must not flex during release. This might be essential in some cases.

After a successful release, the film is floating on the surface of the solvent. You can rinse the film a little extra by arranging a gentle flow of the solution under the thin film. To make use of the thin film as an x-ray filter, the film has to be mounted on a frame. The frame is immersed into the solvent and is placed under the film. Let some part of the frame come in contact with the film to keep it in place. You get additional help to keep the film in place if you "squeeze" the film between the wall of the bowl and the frame. The film drapes itself over the aperture as the frame is lifted out of the solvent. The frame must be lifted in a way that avoids pressure over the aperture, hence the frame should not be parallel to the surface

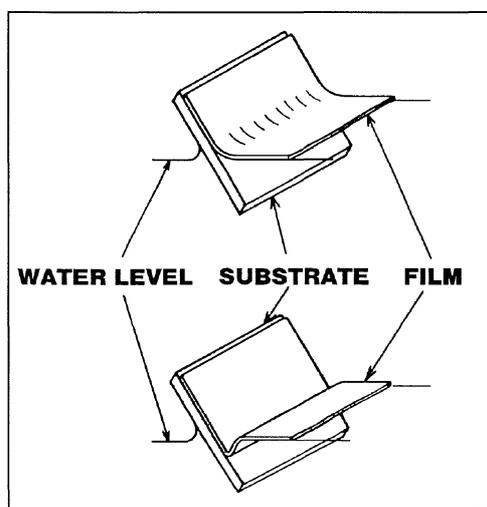


Figure 15. The release of thin film from a substrate. From Ref. 17.

when it is lifted. On the other hand the film is flexed if the frame is lifted perpendicular to the solvent surface, but optimum angle is rather 90° than 0° . Water left in the aperture can be carefully removed with a piece of blotting paper. As the frame and the film dries, the film is stuck to the frame. No glue is needed.

A different technique¹⁷ is to submerge the whole substrate together with the deposited film in the solvent. You have no control over the release and the thin film is very easy to break when it is released. This technique is probably the best if the surface tension of the solvent is very low or if you absolutely want to avoid to flex the thin film on a massive soluble substrate. When the film has come loose, you carefully manoeuvre the film to a position close to the surface with the frame. The frame is then lifted perpendicular to the solvent surface, out of the solvent, and you see to that the film covers the aperture.

5.8 Frame Design

The frame design^{17,24} depends strongly on the application of the thin film. For instance if the film is to be placed in front of an entrance slit, the aperture is made long and narrow since the film can withstand tougher handling. The aperture can be made longer the more narrow it is. Circular apertures have no corners where stress is introduced to the film. Applied pressure give a uniformly distributed stress in the film and hence you obtain an optimum in the filter area versus strength dependence if the aperture is circular.

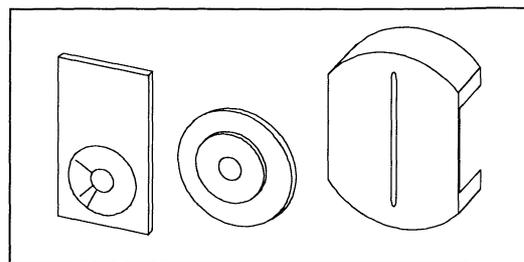


Figure 16. Examples of filter frames.

The aperture should be tapered to decrease capillary forces. The capillary forces tend to draw the thin film into the aperture as the frame is lifted out from the solvent. The tapered edge of the aperture aids to obtain the thin film flat over the aperture. If the frame has a place where you can get a good grip with for instance a pair of tweezers when you lift it out of the solvent, the process is facilitated a little more. In Fig. 16 there are a few examples on filter frame designs.

6. Thin Film Quality

Several of factors affect the thin film quality. Most properties of a thin film are hard to change once the evaporation system is given and the major film constituent is chosen, but the choice of preparation technique can make a difference²⁰. In reality you try to make a film of a certain thickness and with a certain composition under the best conditions you can provide.

If the film is too weak or too brittle, several of solutions might be considered. See Sect. 6.2 for solutions and Sect. 6.1 for a somewhat general discussion of mechanical properties of thin films.

Transmittance is the major quality of the film which has to be measured. The perfect filter is a film which consists of the element(s) you chose with the thickness(es) you wanted. Contaminations (Sect. 6.3), film thickness (Sect. 6.5) and pinholes (Sect. 6.4) affect transmittance and are hence discussed in this section.

Ultimately the filter will be used in the system, the only test that really counts. At this stage it might be to late find out what causes any problems if there is any, since you probably lack the ability to diagnose. You only have the over all system performance to look at.

6.1 Mechanical Properties

In general, the thin film has the same properties (for instance elastic properties) as the bulk material but the film has a higher strength than bulk data indicate¹⁷. Thin film strength approaches that of the bulk material as thickness increase¹⁷. Unbacked films less than approximately 200Å should not be considered. If the thickness is very small (say less than 200Å), the structure of the very thin film on the substrate is usually different from the structure of the bulk material¹⁷. A continuous film might not even have formed¹⁷.

Correlation between grain size and strength has been observed²⁴. Finer crystal structure seems to give a stronger thin film, and you can obtain smaller grains by adding a small amount of a suitable contamination²². The grain size is primarily determined of the element, but also depend on conditions during deposition^{19,20}. A film with smaller grains also seems to withstand aging better²². It has been shown that aluminum films with an inclusion of silicon withstand humidity better¹⁷. After deposition, the internal structure of the film might change spontaneously, which can make removal easier or harder¹⁷. There are also other aging effects making removal easier or harder, and hence you cannot refer this effect to a change in internal structure.

6.2 Additional Film Support

If the element, which a thin film is made of, has proven to be too weak or too brittle to survive removal from the substrate or too weak to withstand handling, some kind of support can be considered^{17,22}. Also, there might be an alternative element to make the thin film of, or it might help if the aperture size is reduced.

There are three types of mechanical support: metal mesh, plastic film and extra layer(s) of a tough metal. A fine nickel mesh with a transmittance of 70-80% is quite usual. The transmittance is good with x-ray standards and the film with a mesh can manage considerably tougher handling. When the thin film is floating on the surface of the solvent after removal from the substrate, it can be mounted on the mesh in the same way as a film is mounted on a filter frame. The only difference seems to be the angle at which you lift the support out of the solvent. A mesh should rather be parallel than perpendicular to the surface of the solvent. Some authors recommend the mesh to be cemented to the film, but the film will probably stick to the mesh anyway¹⁷. You can also choose to apply the mesh prior to removal from the substrate. Then the mesh has to be cemented to the film. Epoxy, for instance, has been used to cement meshes to films¹⁷.

A thin plastic film may support a thin metal film in many ways. Either deposit the metal on a plastic film or apply a plastic film to the thin film after deposition. The former method has been tried in various ways, but plastic films cannot resist too high temperatures. The plastic film is cooled during deposition if it is mounted on a substrate and can take higher deposition rates and higher temperature than if the plastic film is mounted over an aperture. My experience is that the former method (plastic film on substrate) might work if the material to be deposited can be evaporated at a fairly low temperature, while the latter is a reliable method to fail; at best the film get a structure far from flat and at worst the film is completely blown away. Even though a plastic film does not stick as much to a substrate as a metal film does, a layer of a parting agent is recommended. The different approach to apply a thin plastic film over the metal film on a substrate after the deposition procedure by dipping it into a solution of the plastic, can be characterized as a half failure. The films did sometimes not release at all and at other times they were only unwilling to release. A third way to apply the thin film would be to dip the frame with the unbacked film in a plastic solution. No one seems to have done it and neither have I. If you struggle to get the film to survive removal, you probably want to apply the plastic film prior to removal from the substrate.

There are a couple of ways to make thin plastic films. You can cast a film, which means that you obtain the film by dropping a solution of the plastic in its solvent on the surface of water. The solution spread, the solvent evaporate and there is a plastic film a few thousand of Å thick left on the surface. You can also dip the substrate into the solution and then let the solvent evaporate. Then there is a thin plastic film left on the substrate. You should prepare the substrate with a parting agent before you dip it.

If you can observe any interference colors, the film is too thick and you should use a more dilute solution. The plastic film has to be very thin since carbon is quite absorbing in the water window, but many plastics are tough even though they are thin. I have tried to use Formvar, but there are several of alternatives that might be better, for instance Mylar and Pioloform.

The third kind of extra support, to have one or more layers of a different metal, has been fairly successful. Aluminum is relatively transparent in the water window and is known to manage tougher handling. By trial and error you find out which order to deposit the layers, how thick the support film has to be, etc. If you let an oxide form over the aluminum before any other element is evaporated on top, the two materials will not interdiffuse since diffusion through aluminum oxide in general is very slow¹⁵. If there is no oxide layer between the two layers of metal, it is likely they will interdiffuse quite rapidly¹⁵. The change of the mechanical properties of this film over time is unpredictable, a reason to avoid interdiffusion. My experience is that a layer of aluminum work best as support if it is deposited first over the parting agent.

6.3 Contaminations

A contamination is an element not intended to be included into your filter. Soft x-ray transmittance is affected by fairly small amounts of any contamination, since every element has a considerable absorption

at some soft x-ray wavelength. Fortunately, not every element is absorbing in the water window, and hence the contamination might not matter too much if the filter is to be used in a water-window x-ray microscope. Contamination sources^{19,20} are: residual gas pressure during deposition, the evaporation source, the material to be evaporated might not be pure, oil from vacuum pumps, the substrate or the parting agent and the solvent in which the film is released from the substrate. If you know what contamination you have (from a transmittance measurement or from some other analysis), you also have a guideline on how to reduce it since you know how different contaminants might be introduced.

To get an idea of the impact the residual gas pressure have, you may compare the impingement rates on the substrate for the metal evaporated and residual gas.

In Fig. 17 there is a convenient nomogram. The impingement rate for gas molecules (in (# of molecules) $\text{cm}^{-2}\text{s}^{-1}$) is shown as function of the partial pressure for the gas (in Torr). If you know the deposition rate for the metal and assume that the film will have bulk density, the impingement rate is read in (# of atoms) $\text{cm}^{-2}\text{s}^{-1}$. Virtually every impinging atom of a metal will stick on the substrate, but fortunately not every gas molecule. You speak of a sticking coefficient, the probability that an impinging atom or molecule will get stuck. The sticking coefficient primarily depend on the element the gas molecule impinge on, but also on surface temperature and surface smoothness. Hence, if the deposition rate is very low the sticking coefficient will be affected since the surface composition is changed by other gas molecules that have already arrived on the surface. Titanium is sometimes evaporated in vacuum systems to produce lower pressure, since titanium is known to be reactive and has a high sticking coefficient. These systems are refered to as getter pumps, and the process is called cleanup. The sticking coefficient for an oxygen molecule on a titanium surface is 0.15 at most²⁵, and that number can serve as an upper limit to the sticking coefficient for any combination of a metal surface and gas molecule. The shutter in an evaporation system should be closed until you have a fair vapour pressure from your source to reduce the inclusion of gas molecules.

As mentioned above, the evaporation source can be quite hot and the pressure may raise considerably because of outgassing from the radiation heated walls of the vacuum chamber. Obviously this is undesirable. To circumvent this problem you can install a more powerful vacuum pump and increase the size of the pumping port. You can also change the material of the vacuum chamber since outgassing very much depends on the material. And third, and maybe the best improvement, is to install a water cooled or liquid nitrogen cooled shield, then you in fact have a getter pump.

A hot source also contaminates the film and a heated vacuum chamber also contaminates the film, but if you use refractory metals there should be no problems (see Table 4¹⁹). Besides from outgassing, atoms

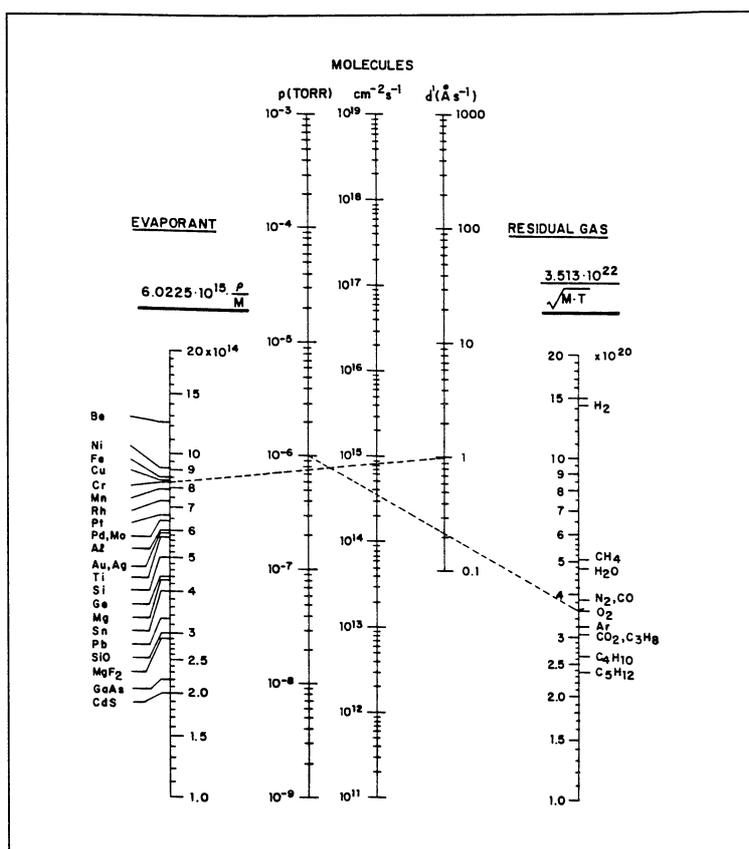


Figure 17. Nomogram to compare impingement rates. From Ref 19.

	W	Mo	Ta
Melting point/ $^{\circ}\text{C}$	3380	2610	3000
T/ $^{\circ}\text{C}$ for partial pressure of 10^{-6} Torr	2410	1820	2240

are emitted from the walls of the chamber. They can also be ignored unless you evaporate at a very high temperature and with a very low rate.

Most metals have to melt before they start to evaporate with an acceptable rate and the question arise how much of the source is alloyed into the melt and how much of it evaporates. If the liquid alloy is an ideal solution, the partial vapour pressure of the contamination is governed by Rault's law²⁶:

$$p = xp^* \tag{8}$$

where p^* is the pressure of the pure element and x is the mole fraction of the element in the alloy. Liquid alloys are rarely ideal solutions and the activity²⁶, denoted a , is introduced. To relate x and a , the activity coefficient²⁶, denoted f , is defined:

$$a = \frac{p}{p^*} \tag{9}$$

$$a = fx \tag{10}$$

Except for molybdenum, refractory metals have very low vapour pressures (Table 4) and even if the activity coefficient would be quite high and a substantial part of the melt consist of metal from the source, it is a good guess to ignore this effect. There is a limit on how far the liquid alloy deviate from an ideal solution.

From the discussion above you can also draw the conclusion that pure elements must be used in order to obtain a pure film. This is another reason why it is a good practice to keep the shutter closed until the charge is quite hot. Then the pollutant might have left before deposition starts.

Oil from vacuum pumps is disastrous if allowed to contaminate a water window x-ray filter, since carbon is next to opaque in the water window. If oil contaminates the surface of the substrate, the metal film might peel off before the evaporation is over (poor adhesion). Either you use a vacuum system where the vacuum pumps are free from oil or you have a cooled baffle between the pumps and the chamber.

The thin film is next to a parting agent or a massive soluble substrate during the deposition and until the release. Just like two adjacent metal films interdiffuse, the parting agent and the metal might interdiffuse too, especially as the temperature is high during deposition. If it is a problem, you can always use a different parting agent, but the concept remains the same: there has to be something for the metal to deposit on. All you can do is to thoroughly rinse the film during release and then observe the result. It is much more important to choose a parting agent that does not react with the film. The solvent used to release the film should be as pure as possible. For instance, if water is used as solvent, the water should be deionized. It has been shown that thin aluminum films which has been released in deionized water have a longer lifetime¹⁷.

6.4 Pinholes

Pinholes¹⁷ quite small can be seen with an unaided eyes. Traditional microscopes can be used to estimate how big they are and to see how many there are. If you want to know the total pinhole area, you can measure the transmittance for visible light and multiply it with the frame area. This is correct when the thin metal film is opaque to visible light.

Pinholes can be reduced in number by cleanliness since dust and lint cause them by shading during deposition. Metallic impurities may also cause pinholes during release because a galvanic cell can be formed. After the release, small amounts left of the parting agent might react with the film and cause pinholes as the film ages. Pinholes might also arise from a careless release from the substrate. If the total pinhole area is too large, two filters can be used in tandem.

6.5 Film Thickness

The metal film should have a uniform thickness, and the thickness should agree with the thickness displayed on the thickness monitor. Film thickness is probably not uniform since an evaporation source might not evaporate as much in every direction and every spot of the surface of a large flat substrate is not perpendicular to the source. A typical evaporation geometry is sketched in Fig. 18. The geometry dependence of the thickness, t , if you have a surface source can be expressed as²⁷:

$$t \propto \frac{\cos \phi \cos \theta}{r^2}. \quad (11)$$

If the substrate is flat and parallel to the source, then $\phi = \theta$, and $r = r_0 / \cos \theta$. To get the thickness dependence for a point source instead of a surface source, you set $\cos \phi = 1$. Hence, the worst case is a surface source with no extension.

To get the deposited thickness, the thickness monitor must be calibrated. One method is presented here: Load a tungsten hairpin with aluminum wire with $\phi=0.5$ mm and the length l . By assuming bulk density, the thickness monitor displays the thickness t_m after evaporation. Also assume that the tungsten source homogeneously emit aluminum into 4π steradians. Let A denote the area of a sphere with the source in the centre and the detector at the surface and let V denote the volume of the aluminum evaporated. Then $At_c=V$ where t_c is the thickness of the aluminum film at the surface of the sphere, assuming that the thickness is small compared to the sphere radius. Theoretically and approximately you have

$$t_c = \frac{V}{A} = \frac{\pi(\phi/2)^2 l}{4\pi R^2} \quad (12)$$

There are several of error sources. You assume a point source, which is not exactly what you have. Sources with the characteristics of a point source usually cannot be loaded very much and you are limited of the thickness monitor resolution. You can hardly know how much oxygen or nitrogen is included into the film, and there might build up an oxide layer after deposition even though you maintain vacuum. These errors add up, and you get no warning if you are too far off. All these errors act in the same direction; they make the detector show a thickness to large.

The transmittance and reflectance as function of film thickness can be used to give a rough calibration. In Table 6²⁸ you find aluminum transmittance and reflectance as function of film thickness. You can for instance use a HeNe laser and a power meter, and then you compare thickness indicated by reflection with thickness displayed on the monitor. You can also use soft x-ray transmittance to get the film thickness and to calibrate the thickness monitor.

Figure 18. Evaporation geometry and variable definitions.

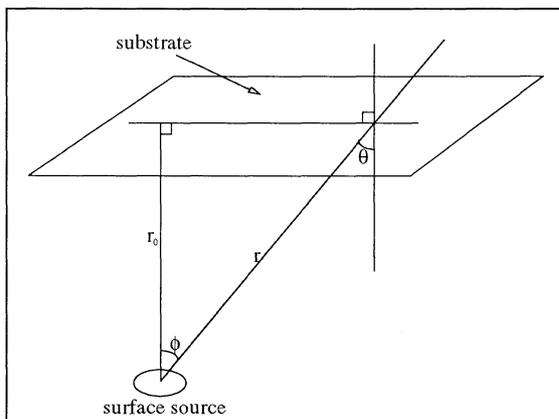


Table 6

Film thickness/Å	Wavelength/nm			
	546		650	
	R%	T%	R%	T%
40	33	51	38	42
80	60	24	63	18
120	74	12	75	9
160	81	7	82	5
200	85.6	3.5	85.4	2.6
240	88.1	2.0	87.5	1.4
280	89.5	1.1	88.8	0.8
320	90.4	0.5	89.6	0.4
360	90.9	0.4	90.0	0.3
400	91.2	0.2	90.3	0.2

7. Experimental

Before discussing the preparation and evaluation of individual filters, the measurement methods involved in the evaluation will be discussed.

7.1 Measurement and Calculation Methods, Calibrations

7.1.1 Water Window Transmittance

The x-ray source was a LPP in a vacuum chamber and the target was a running cassette tape, i.e., a carbon and oxygen target. The laser gave 35 mJ pulses with 10 Hz rate at the wavelength 532 nm. Between the x-ray source and the detector, an x-ray filter could be switched in and out. The detector was an x-ray monochromator followed by a CsI cathode electron multipliertube. The monochromator let virtually no stray light through. First the spectra was recorded without the filter, and then with the filter in place. From the spectras, the transmittance can be calculated at the emission lines. In Fig. 19 there is a scheme of the set-up.

Potential sources of error are numerous and must be considered. Their influence is determined in Sect. 7.2. Laser intensity was quite unstable. X-ray intensity increase with laser pulse energy, but how is next to impossible to calculate. As spectra was recorded, debris from the LPP punctured the filter. Hence, transmittance was not constant. The set up was not ideal and the filter quite hard to centre in front of the entrance slit. Transmittance data would be more reliable if the transmittance was measured peak by peak. To reduce the intensity fluctuation, every measurement is an average over 30 laser pulses.

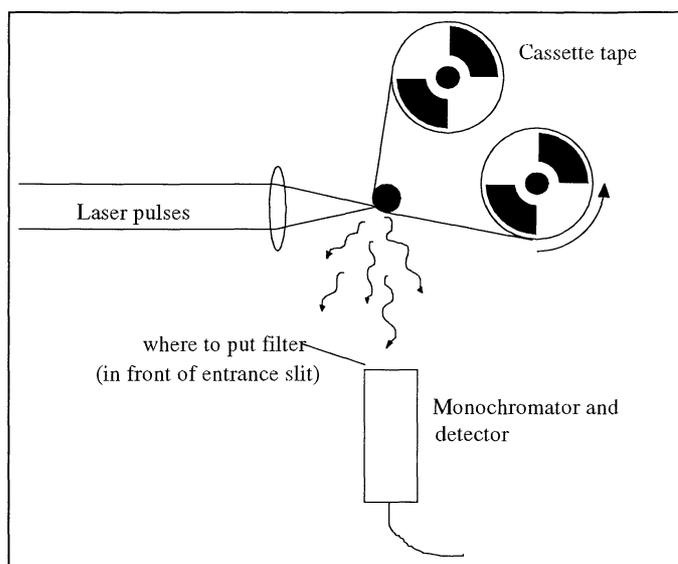


Figure 19. The set-up with which spectras were taken.

7.1.2 PIXE

In PIXE²⁹ (Proton Induced X-ray Emission), protons are shot through a thin film. The accelerated protons tear electrons from the atoms and as the atomic shells are filled, you get x-ray emission. The x-ray wavelength emitted from each element is unique, hence the element can be determined. From the x-ray intensity, you can calculate the atomic density per unit area very accurately for very small concentrations of elements with the atomic number of aluminum and higher. Sources of errors are unknown to me.

You get the results from PIXE in $\rho_2 = \text{g} / \text{cm}^2$. If you assume a density, ρ_3 in g/cm^3 , the results can be converted into thicknesses.

$$m = \rho_3 V = \rho_2 A, \quad V = At \quad \Rightarrow \quad t = \frac{\rho_2}{\rho_3} \quad (13)$$

7.1.3 The Thickness Monitor

7.1.3.1 Function

The Intellemetrics IL101 thickness monitor actually measures the deposited mass and then converts it into a thickness with the density you entered. Since transmittance is a function of number of atoms per unit area, i.e., mass per unit area, the function of the thickness monitor is ideal. The actual density of the thin film does not matter.

7.1.3.2 Calibration

Since the thickness of the film is one of the most important factors for a well defined soft x-ray filter, the x-tal thickness monitor was calibrated by two methods.

Table 5

l/mm	R/mm	measured, t_m /nm	calculated, t_c /nm	t_m difference from t_c
100	237	23.7	22.3	6%
50	181	21.3	22.6	-6%

Calibration 1: Evaporation of a known amount of aluminum with a known geometry according to Sect. 6.5. Four tungsten hairpins served as point sources and were used simultaneously to increase source capacity. Increased source capacity reduce the effect of a final oxide layer and reduce the effect of limited detector resolution. In Table 5 two calibrations, made at two different occasions, are presented. Calculations are made according to Eq. 12. Calibrations of this type was also made on several of other occasions, but with fewer evaporation sources and hence lower accuracy. The results from these calibrations are essentially the same as those in Table 5. The calculated thickness is in good agreement with the results of the x-tal thickness monitor, but since the sources of error all act in the same direction, the thickness monitor might show a thickness somewhat too small.

Calibration 2: Aluminum was evaporated on a microscope slide and the optical transmittance and reflectance was measured. The thickness monitor displayed 11 nm and the transmittance was about 10% with a HeNe laser, which roughly agree with Table 6. However, the sources of error are large. Multiple reflections in the microscope slide, problems to detect low light intensities, oxide layer etc. are a few factors that degrade the accuracy. This calibration, however, shows that the thickness monitor is about right in a totally unrelated measurement.

Since the sources of error in calibration 1 are hard to control, the final error estimation is based on calibration 2. I only dare to say that the x-tal monitor display a thickness with an error less than 20%.

In addition there is a variation in deposited thickness due to evaporation geometry. If the substrate is positioned 24 cm from the evaporation source and the substrate is 10 cm wide, then there is a variation in the thickness of the film of slightly less than 10% due to the evaporation geometry.

7.1.4 Henke data

A film thickness together with Henke data¹⁵ give x-ray transmittance curves as discussed in Sect. 3. Absorption coefficients were calculated from Henke data (fortran 77 program in Appendix E) according to Eq. 6, and transmittance curves were calculated in a spread sheet programs (Borland Quattro Pro and Microsoft Excel).

7.2 Filters

7.2.1 The Vanadium Filter

7.2.1.1 Preparation

Several vanadium filters were made. Vanadium seems to be quite brittle, hence aluminum was used as support. Microscope slides cleaned according to Appendix B were used as substrates. The non-organic part of victawet was evaporated on the substrate according to Appendix C, with the exception that vacuum was maintained afterwards. On top of the parting agent, aluminum was deposited: 95 nm was registered of the thickness monitor. Method according to Appendix A1. Vanadium was deposited on top of the aluminum according to Appendix A3 with a couple of exceptions. The source was different (a little smaller) since I temporarily did not have the usual type in stock and the evaporation geometry was modified to compensate for the smaller capacity of the sources; The distance between the source and the substrate was 14 cm instead of the usual 24 cm. First two tungsten sources were loaded with vanadium and emptied by evaporation in one run (vacuum was maintained) and then another two. In the last run a load of vanadium was deposited and then another 95 nm of aluminum. In all 240 nm of vanadium and 190 nm of aluminum was deposited.

The pressure in the vacuum chamber during deposition of the first aluminum layer was $2 \cdot 10^{-5}$ Torr, during the vanadium deposition $3 \cdot 10^{-5} - 9 \cdot 10^{-5}$ Torr and during the deposition of the final layer of aluminum, the pressure was $1 \cdot 10^{-5}$ Torr.

If the organic part of victawet is allowed to reach the substrate, the film flakes off during deposition. It might be a large internal stress in the deposited vanadium film in combination with poor adhesion to the organic part of victawet which cause the film to flake off. A vanadium film without aluminum falls into small flakes during release, which also happens if the first layer of aluminum is too thin or if the layer of vanadium is too thick. It seems as if the second layer of aluminum does not strengthen the filter very much.

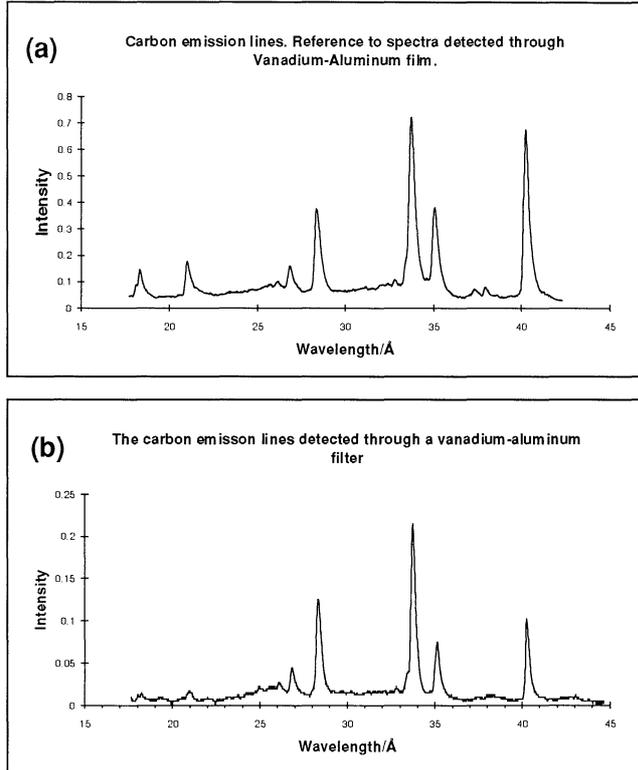


Figure 20. a. Spectra with empty filter frame. b. Spectra with vanadium/aluminum filter.

7.2.1.2 Evaluation

One spectra was recorded with the filter and one without the filter. The spectras are presented in Fig. 20. Table 7 displays the calculated transmittances. A third spectra from 80-720 Å was taken with a different gratin in the monochromator. Where aluminum is transparent (above 110 Å), we could see that vanadium is opaque.

Even though every measurement is an average over 30 shots, there are some fluctuations left. The intensity fluctuation of the 33.7 Å line was about 0.03, hence there is an error of $0.03/0.685 \approx 4\%$. When calculating the transmittance error, the fluctuation was taken as proportional to line intensity and applied to all lines, since the lines origin from the same number of carbon ions (not quite true: there are C-V and C-VI ions). A constant noise error of ± 0.048 was added to the (weak) filtered signal (± 2 quantization steps).

The results from the PIXE evaluation of the filter and corresponding thicknesses together with assumed densities are summarized in Table 8. The original results can be found in Appendix F.

Table 7

emission line/Å	intensity without filter/arbitrary units	intensity with filter/arbitrary units	Transmittance
18	0.108	0.0072	0.069 ± 0.050
22	0.138	0.0096	0.071 ± 0.040
27.0	0.121	0.038	0.317 ± 0.065
28.5	0.338	0.119	0.354 ± 0.042
33.7	0.685	0.209	0.306 ± 0.031
35.0	0.346	0.070	0.204 ± 0.030
40.5	0.637	0.097	0.153 ± 0.020

Table 8

	ρ_2/ngcm^{-2}	error	ρ_3/gcm^{-3}	$d=\rho_2/\rho_3$	# of atoms per cm^2
V	$0.385 \cdot 10^5$	7.1%	6.11	63 nm	$4.55 \cdot 10^{17}$
Al	$0.192 \cdot 10^5$	8.3%	2.7	71 nm	$4.28 \cdot 10^{17}$
Fe	356	7.9%	7.87	0.5 nm	$3.84 \cdot 10^{15}$
W	123	9.6%	19.3	0.06 nm	$4.03 \cdot 10^{14}$

As you can see, there is a negligible tungsten contamination from the source. The origin of the largest contamination, iron, is unknown, but can be neglected too.

To compare different results, they are plotted in the same graph. Transmittance is calculated from Henke data with the thickness measured of the crystal monitor (240 nm vanadium +

190 nm aluminum) and with the thickness obtained from the PIXE analysis. The density for the different elements is just a conversion factor and does not matter. Transmittance obtained from the spectras is also plotted in the same graph, Fig. 21.

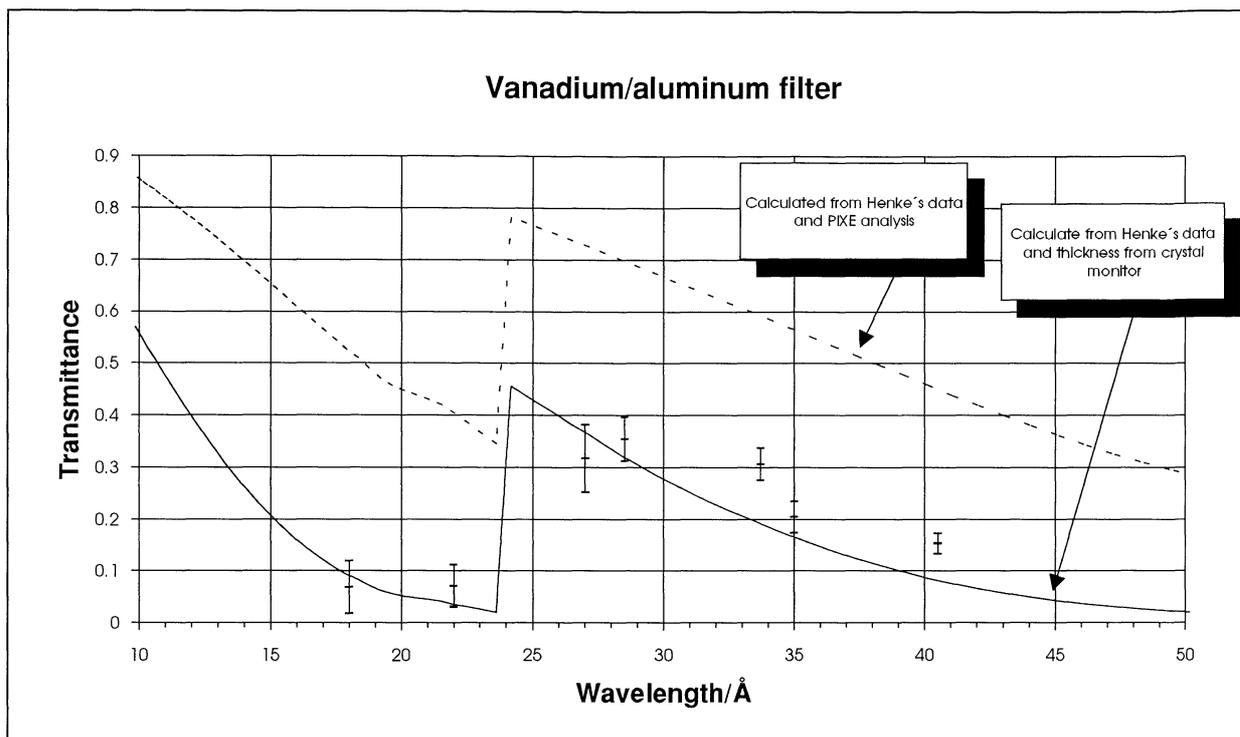


Figure 21. Vanadium/aluminum transmittance, measured or derived from other measurements.

The PIXE analysis gave a significantly different result. The only way to get the PIXE measurements to agree with the transmittance measurements, is to assume an oxygen contamination that cannot be detected in PIXE but that affects transmittance. If you assume an oxygen contamination, you need more than $5 \cdot 10^{18}$ oxygen atoms/cm² to get the same x-ray transmittance as transmittance measurements yield. Since the films have a metallic appearance, this seems improbable. If you assume a carbon contamination, you cannot explain how the oxygen emission lines at 18 Å and 22.1 Å are suppressed in the spectra with the vanadium filter inserted.

From the calibration of the crystal detector, Section 7.2.3, we know that the measured thickness is about right (with an error less than 30%). Hence, carbon or oxygen contaminations can hardly explain the discrepancy of the PIXE results. Most of the difference remain unexplained.

Pinholes were very few and small. Debris generated holes overcame any effect the pinholes from the preparation process might have. The holes in the film have no apparent impact on the appearance of the spectra. This is since the monochromator does not let any stray light pass through. The oxygen lines are severely attenuated. They would be more intense if the holes had a "large" total area.

7.2.2 The Titanium Filter

7.2.2.1 Preparation, Comment

According to Ref. 17, see Appendix D, titanium reacts with refractory metals. On the other hand, researchers have had success when evaporating titanium¹⁷ from the same source as vanadium is evaporated from in Appendix A3³⁰. Hence it should be possible to evaporate titanium from a tungsten source, even though e-beam heating is recommended.

I have tried to evaporated titanium from a tungsten source. The metal melted, but a short while afterwards the source broke in a little cascade of beautiful colors. The way the source is loaded is crucial I was told. Above all, there should not be too much titanium in the bowl. I do not know if those who had success let the titanium sublime or if they let the titanium melt, but compared to vanadium, titanium melts at a lower temperature. The current at which they made their evaporation was larger than the current at which vanadium melts, which indicate that they also let titanium melt.

7.2.3 The Silver Filter

7.2.3.1 Application

To measure emission line intensity with the carbon test target, another filter was also needed. A photo diode sensible to a very wide range of wavelengths together with a silver/aluminum filter was used to measure the intensity in the water window. A combination of tin and aluminum is a more general water window filter³¹ than silver/aluminum. To keep the discussion short, notice that silver with its absorption edge at about 30 Å and its virtually flat absorption curve above 30 Å also can do together with the carbon target. In Fig. 22a you see the silver transmittance characteristics for a wider range of wavelengths. Aluminum in combination with silver block wavelengths from somewhat above the water window and beyond 2000 Å. Aluminum transmittance characteristics is in Fig. 22b and the combination aluminum and silver in figure 22c.

7.2.3.2 Preparation

Silver does not need any extra support. In fact, silver seems to be a little tougher than aluminum. There is no description on how to evaporate silver in Appendix A since the procedure is very similar to that of aluminum. With silver you can use a molybdenum source, too.

Only one batch of silver films was made. Microscope slides, never used before and not cleaned at all, were

used as substrates. The parting agent was victawet, which was evaporated onto the substrate according to Appendix C (all of the victawet was allowed to reach the substrate). High purity silver wire was cut into small pieces and then put into the source. The evaporation procedure was according to Appendix A1, except that a molybdenum source was used. The geometry was different from the usual: distance from source to substrate was 14 cm. After deposition the thickness monitor displayed 170 nm of silver. The release was exceptionally smooth and easy. Pressure during deposition was $3 \cdot 10^{-5}$ Torr.

7.2.3.3 Evaluation

Soft x-ray spectras with and without silver filter were taken, Fig. 23. The error estimation is similar to that of the vanadium filter. No PIXE analysis was made with the silver filter. The measured transmittance is plotted in the same graph as transmittance calculated from the thickness measurement (170 nm) and Henke data, see Fig. 24.

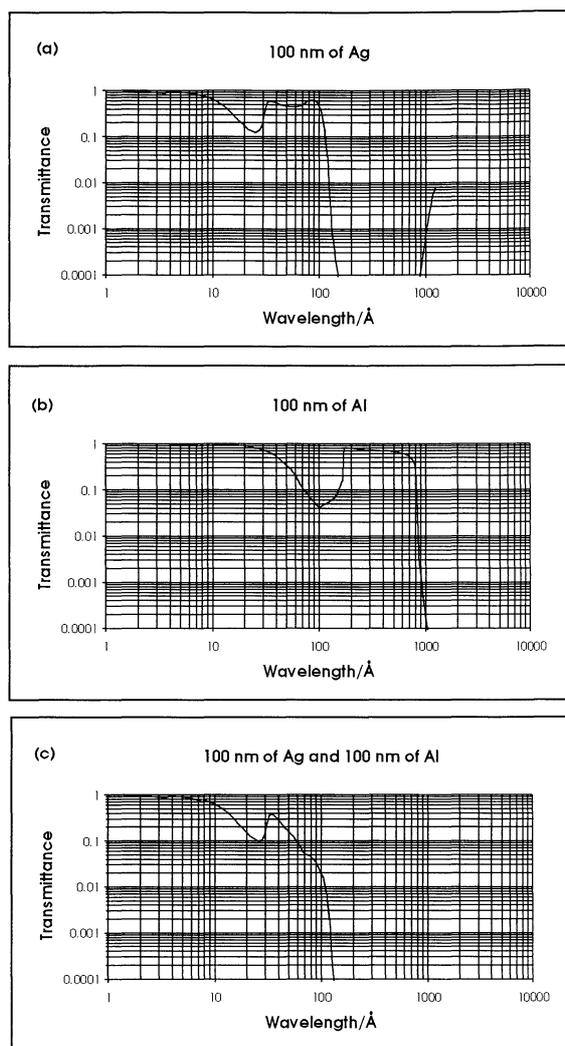


Figure 22. Transmittance for 100 nm of a. silver, b. aluminum, and c. 100 nm of aluminum and 100 nm of silver. Calculated with data from Ref. 13.

Table 9

emission line/Å	intensity without filter/arbitrary units	intensity with filter/arbitrary units	Transmittance
22	0.100	0.010	0.102±0.058
27.0	0.073	0.005	0.071±0.074
28.5	0.251	0.022	0.089±0.027
33.7	0.653	0.314	0.498±0.048
35.0	0.312	0.152	0.489±0.055
40.5	0.613	0.302	0.495±0.048

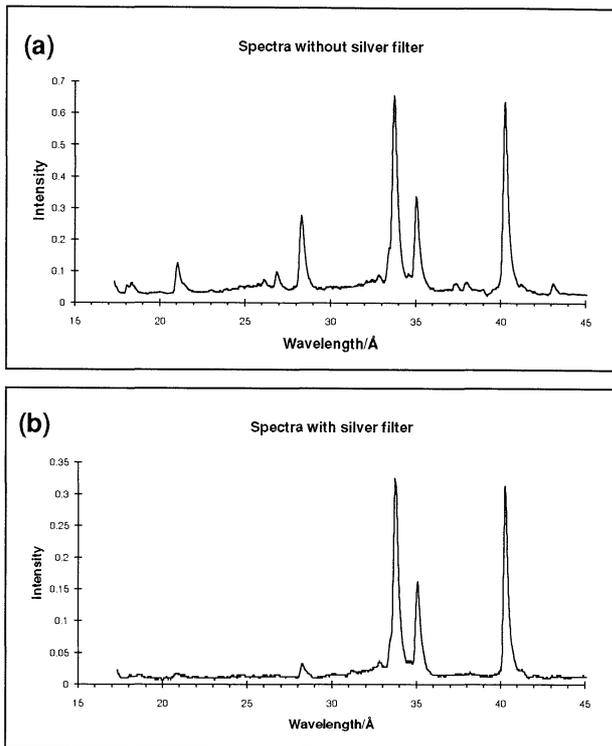


Figure 23. a. Spectra with empty filter frame. b. Spectra with silver filter.

The filter is probably thinner than the thickness monitor indicate. This is possible because of the evaporation geometry used and because of inaccuracy in thickness monitor calibration. The thickness might be up to 20% less than the monitor measured, due to the (different) geometry. The thickness monitor was never calibrated with silver. There were quite a few pinholes (since the substrates were not cleaned) and during the measurements even more holes were generated. Since oxygen and carbon lines below 30 Å were attenuated, the effect of these holes can be neglected.

7.2.4 Aluminum Filters/Debris Protection

7.2.4.1 Preparation

Aluminum is the metal with which different preparation methods has been tried. One of the aluminum films was more carefully analyzed. In the preparation of this film, microscope slides cleaned according to Appendix C was used and victawet was used as parting agent. Victawet was

evaporated, but only the non-organic part was allowed to reach the substrate. The crystal thickness monitor gave that the film was 107 nm. The release was somewhat uneven.

Most aluminum filters I have made are not quite smooth. What parameter that affects this is unknown. No other quality seems to depend on the different appearances and hence I have not bothered to do anything about it.

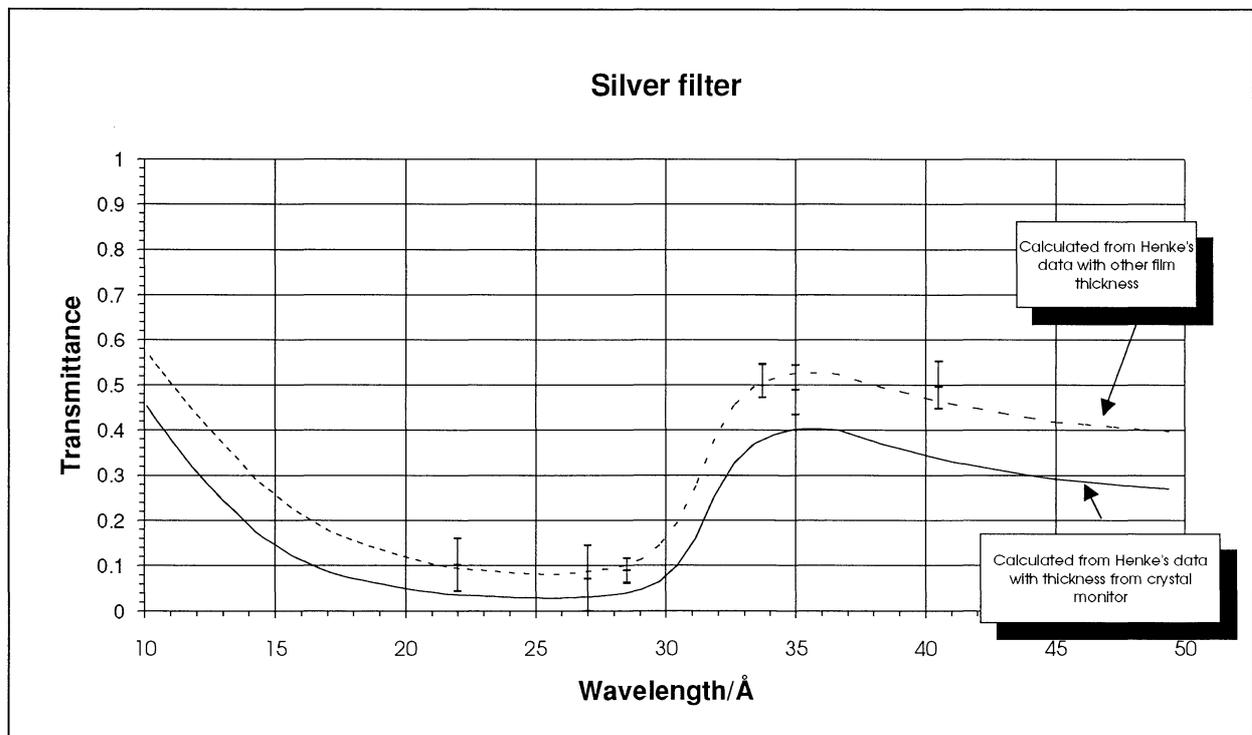


Figure 24. Silver filter transmittance, all the measurements. Other thickness is 0.12 μm and measured is 0.17 μm .)

7.2.4.2 Evaluation

The aluminum filter was analyzed with PIXE. Results are presented in Table 10. The original results are similar to those in Appendix F. These results also differ significant from those of the thickness monitor (107 nm). A thickness of 46 nm is not very likely; the film would hardly manage to be mounted over a 0.5 cm aperture. Spectras were never recorded to get aluminum transmittance, because aluminum films were also mounted as debris protection. When you scan the monochromator from 100 Å to 110 Å you suddenly get a signal according to an absorption edge of aluminum. It can only serve as an indication of the presence of aluminum, since a spectra without the aluminum debris protection film never was recorded.

The pinholes were very few, small and is of far less importance than holes from debris. It was shown with the vanadium filter that the holes does not change the spectra in an observable way.

Table 10

	ρ_2/ngcm^{-2}	error	ρ_3/gcm^{-3}	$d=\rho_2/\rho_3$	# of atoms per cm^2
Al	$0.124 \cdot 10^5$	7.1%	2.7	45.9 nm	$2.80 \cdot 10^{17}$
W	94.4	7.2%	19.3	0.05 nm	$3.09 \cdot 10^{14}$
Ca	48.3	7.4%	1.55	0.32 nm	$7.26 \cdot 10^{14}$
P	29.9	9.0%	1.82	0.16 nm	$5.81 \cdot 10^{14}$

8. Summary, Conclusion

I have developed methods and modified some equipment to prepare thin unbacked metal films. With these methods and the equipment, I have made thin unbacked films of aluminum, vanadium/aluminum and silver. These films were also evaluated. Their compositions were studied with PIXE and their water window transmittances were also measured. The pinholes were never quantified, but can virtually be eliminated by cleanliness. The transmittance characteristics of the films were as expected, but PIXE results disagree. I reject the quantitative results from the PIXE analysis at the same time as I accept qualitative results. The results show that certain sources of contamination can be neglected. There might be other contaminations that cannot be detected with PIXE, but then there is too little of these contaminations to affect water window transmittance.

The vanadium/aluminum film was designed to work as transmission filter to emphasize the 25 Å emission line from nitrogen, but no transmittance measurements were made right about $\lambda=25$ Å. If the filter is good enough to be used in a water window x-ray microscope with zone plates remains to be seen, since the microscope is not built yet. Aluminum films are used as debris protection and an aluminum film in combination with a silver film has been used to measure detector linearity and water window photon flux.

The preparation technique of thin unbacked films can be changed in an infinite number of ways. I recommend to try mica and solid solubles as substrates instead of microscope slides. A few other parting agents should also be evaluated to see if there is an even better alternative. The evaporator can be improved in several ways. A lower pressure during evaporation is desirable, even though no negative effects of residual gas has been observed. If an e-beam heater was possible to use, much trouble could probably be avoided.

9. Acknowledgement

I am grateful for the help and the support I have got from those who work at the Division of Atomic Physics, Lund Institute of Technology. My instructor Hans Hertz, in particular, has provided inspiration, guidance and different kinds of help. The project gave valuable experiences, especially since it was carried out in a free and generous atmosphere. Also, the spectras recorded of L. Rymell and initiatives from L. Malmqvist have been of special importance. Tomas Starczewski and Åke Bergquist have been most helpful in overcoming certain problems.

I also acknowledge the people outside the division from whom I have got help, help for which I am also grateful. Without advises and know-how from Eric Carleman, Lund University Hospital, the project would never have got going, and the co-operation of Jan Hultkvist, at the engineering workshop, made the project run smooth. Measurements made of Eryk Swietlicki, the Division of Nuclear Physics and support from Lena Timby, Division of Solid State Physics, are small contributions but never the less valuable. Karin Löwkvist and Peder Christensen gave elementary guidance within the area of chemistry.

APPENDIX A

There are several of different sources available from different manufacturers. Basically, the source only decides what current you need in order to reach a certain temperature. Of course, the source has to fit into your evaporator.

1. Evaporation of aluminum from a dimpled tungsten foil

The evaporation source is called ME6B-0.005W and is manufactured of R.D. Mathis, USA.

1. If you have used the source before, see to that there is no hole in the foil. It is very likely that the foil will break if there is a single tiny hole. Why these holes occur is not clear. In Appendix D you can see that aluminum alloy with tungsten and maybe substantial amounts of tungsten is alloyed into the melt where tungsten is as hottest.
2. Fit the tungsten foil into the evaporator. Be careful to get good electrical contact. You should clean the copper surface the current is to pass through if it is dirty. If the electrical contact is not perfect, say there is a voltage drop over the contact of 2 V at 200 A. Then the power generated is 0.4 kW.
3. Fill the bowl in the foil with aluminum. I have used fine aluminum granules and it has worked well. When the source is filled, be careful not to bump into the evaporator since the granules tend to "jump" out of the bowl. You should protect the pumping port so that no dirt can fall into it.
4. Put the substrates into their holder, place the bell jar over the base plate and pump down the system. It takes a while.
5. When the pressure is low enough to begin evaporation (I usually wait for $1 \cdot 10^{-5}$ to $2 \cdot 10^{-5}$ Torr), turn on the cooling water to the electrodes and the crystal detector. If you turn the cooling water on too early, water will condense on the crystal. Pump down will then take longer and the crystal detector might show an incorrect value.
6. Zero the thickness monitor and see to that the shutter is closed.
7. Turn the power supply on and slowly increase the power to the source. A source that has been used before needs more current to get as hot.
 - a. You can increase current to about 100 A fairly fast. As the source gets hot and its resistance increase, the voltage over the source will raise and hence the current will decrease. At some moment you will get outgassing from the source material, but you can ignore it since it is so little (aluminum granules in this case).
 - b. Increase the current in steps of 10-20 A. At some point the aluminum will start to melt and wet the source, and at some later point there will be a vortex (or two) in the melt. I use to make the next increase when the situation has "stabilized": current does not drop

any more due to increasing source temperature and nothing more seems to happen in the source. The current will increase when the aluminum melts, since the resistance decreases.

- c. When the surface of the melt is "clean", you cannot see the vortex any longer since there is nothing on the surface of the melt. Then I open the shutter. At this point the current usually is 150-160 A. Keep your eyes on the thickness monitor and adjust the power to give the deposition rate you want. I usually have a deposition rate of 4-5 nm/s. The higher deposition rate, the less contamination from residual gas will be included into the film. Now the deposition rate rather than the current is the parameter you control, but I have had a current of 170-180 A during the deposition of aluminum. Pressure is usually $5 \cdot 10^{-5}$ Torr during deposition.
 - d. Close the shutter a little before you have the film thickness you desire. With a deposition rate of 5 nm/s I close the shutter at a thickness 10-15 nm less than the desired thickness.
 - e. As far as I know it does not matter very much whether you empty the source or not before you turn the power off. If there is metal left till the next evaporation, a larger current is needed to heat the source next time since the resistance is lower.
8. Read the thickness from the thickness monitor and turn the cooling water off.
 9. Let air into the vacuum chamber.
 10. Now you might notice aluminum build-up on the tungsten foil next to where the copper clamps are. Aluminum tends to creep out of its container, and when it gets closer to the cold copper clamps, it solidifies.

2. Evaporation of aluminum from a tungsten "hairpin"

The source was bought from Balzers (BU 007-102T) and had $\phi = 0.5$ mm. A similar source was made of twisted wire with $3 \times \phi$ cross section. Manufacturer unknown.

1. Take a piece of aluminum wire and form it into a suitable shape. You can for instance wind it round the tip of a pair of tweezers. Thread the aluminum wire onto the tungsten hairpin. A piece of aluminum wire with $\phi = 0.5$ mm and the length 2.5 cm does work and maybe the source can be even heavier loaded.
2. Fit the hairpin source into the evaporator. See too that the electrical contact is good.
3. Put the substrate into the holder, place the bell jar over the base plate and pump down the system.
4. When you have obtained a pressure of $\approx 1 \cdot 10^{-5}$ Torr, turn the cooling water on.

5. Zero the thickness monitor.
6. Turn the power supply on and slowly increase the power to the source. The tungsten wire starts to glow and at some point the aluminum melts and form a drop in the bend.
7. Open the shutter.
8. Increase power and you can see that the drop decrease in size as the aluminum evaporates. You can also watch the thickness, but since the source cannot be loaded too heavy, deposition rate does not say very much.
9. Turn the power off when there is no aluminum left or terminate the process by closing the shutter when you have the thickness you want.
10. Read the thickness. The thickness is always small with this source and since aluminum density is relatively low, the deposited mass is small. You must see too that the crystal has about the same temperature when you zero it as when you read it, which is difficult. If you want relatively good accuracy, it takes time.
11. Let air into the vacuum chamber and lift off the bell jar. The tungsten hairpin can be used many times, but is quite brittle when it is used once.

If you are to deposit a precise thickness a repeated number of times, you can figure out the exact length of aluminum wire needed by trial and error, and then keep the shutter open during the whole evaporation.

3. Evaporation of vanadium from a resistance heated dimpled tungsten foil.

The source is called ME6B-0.005W and is manufactured of R.D. Mathis.

1. If you have used the source before, see to that there is no hole in the foil. The foil will break for sure during deposition if there is a single tiny hole or the foil might not get hot enough. You can also take a look at the bottom side of the dimpled foil. If there is a tiny area that looks corroded, then there is a substantial risk that the source will break during evaporation.
2. Fit the foil into the evaporator. Be careful to get good electrical contact.
3. Put vanadium into the bowl of the foil. I have used fine vanadium powder, but small granules would probably be better. Granules are easier to handle and does not outgas as much as powder during heating. How many times the same source can be used clearly depend on how heavily loaded it is. I have

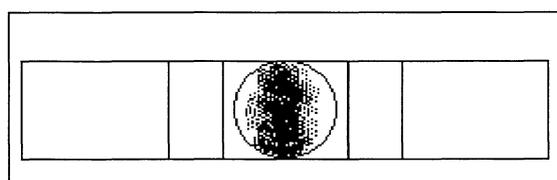


Figure 25. The evaporation source from above. How to load it to make it last longer. Do not overload.

managed to use the same source 3 times, but I do not know what load gives the best economy. When the source is loaded with caution, you usually get a film thickness of 80 nm and if you load the source as much as possible, you get a film thickness of 160-200 nm (with the usual geometry).

4. Put substrates into the holder, place the bell jar over the base plate and evacuate the vacuum chamber.
5. Wait until you have a pressure less than $1 \cdot 10^{-4}$ Torr or less and until the diffusion pump is really hot (work at maximum capacity). Turn on the cooling water to the crystal detector and the electrodes. Zero the thickness monitor.
6. Turn the power supply on and lowly increase the power.
 - a. Between 80-150 A there will be considerable outgassing and I use to increase the power in a pace that keep the outgassing relatively constant and the pressure at least below $1 \cdot 10^{-3}$ Torr.
 - b. Wait at 150 A until outgassing stops. From now on you probably need welding goggles to observe the source. You might observe that something happens to the vanadium powder at about 150 A.
 - c. Increase the current to about 170-180 A. If the source has never been used before, the vanadium powder should start to melt at this current. You might not get all the vanadium powder to melt.
 - d. Wait until nothing more happens in the tungsten source and then open the shutter. The temperature shock will make the thickness monitor show negative growth.
 - e. When you have a stable signal, increase the current to 200-205 A. If it is the first time the source is used, the deposition rate should be about 5 nm/s or even faster. If the source has been used before, the vanadium powder might not melt until the current is 210 A, and to start evaporation you might increase the current to about 250 A for a couple of seconds. Empirically, the maximum current is somewhat more than 200 A. Usually evaporation starts after this "shock treatment".

When the current is over 170 A you must be very attentive. Once the evaporation starts, the source is empty in a few seconds. The pressure is quite high during deposition, usually $1 \cdot 10^{-4} - 2 \cdot 10^{-4}$ Torr, or even higher if you make the process too lengthy.

- f. When you want to obtain a certain thickness, you have to keep in mind that the heat from the source make the thickness monitor show a thickness 25-35 nm less than it really is and add the usual 10-15 nm offset to get the thickness that signal the moment at which to close the shutter.
7. After deposition, wait a little for the crystal to cool in order to read the correct film thickness, and turn the cooling water off.
8. Let air into the vacuum chamber. Be careful not to burn your fingers. The source is slightly deformed after it has been used once, which can you see when you release it from the copper clamps.

APPENDIX B

The method described below is sufficient to clean microscope slides. If there is any metal left from before I recommend you to discard the slide. Methods to remove metals deteriorate the surface. If the slide has never been used before or if the slide does not seem to dirty, you can simplify the procedure significantly. Which steps to ignore I leave to your own judgement.

1. Wash the slide in water with a detergent and then thoroughly rinse it.

2. Using ultrasonic agitation, rinse the substrate 2-5 minutes in each bath of
 - a. trichlorethylene.
 - b. acetone.
 - c. acetone.
 - d. methanol.
 - e. methanol.
 - f. ethanol.
 - g. ethanol.

3. Blow the last chemical from the substrate with for instance nitrogen.

1 and 3 are the most essential steps.

APPENDIX C

Evaporation of victawet from a resistance heated molybdenum foil.

The evaporation source is called ME6B-0.005Mo and is manufactured of R.D. Mathis.

1. Fit the molybdenum source in to the evaporator.
2. Put a tiny bit of victawet into the bowl of the foil. The amount of victawet is not critical at all. I have tried to use 5-50 mg with the ordinary evaporation geometry. Victawet successfully acted as a parting agent at every occasion. Usually I have taken 10 mg of victawet.
3. Put the substrates into the holder, place the bell jar over the base plate and evacuate the vacuum chamber.
4. Wait for the pressure to drop. I have patience to wait for $2 \cdot 10^{-5}$ Torr, but the pressure is not that crucial during deposition of a parting agent. Turn on the cooling water, but it is no catastrophe if you would forget it.
5. You may choose to open the shutter now.
6.
 - a. Turn the supply on and feed the source with 50 A. Increase the current in steps of 2 A and watch the pressure. Victawet is disastrous to the pressure when it is evaporated, and you should be prepared to decrease the current if you think the pressure is too high. I try to keep the pressure below $1 \cdot 10^{-3}$ Torr, since the pressure gauge does not work above this pressure. If you deliver too much power, the little piece of victawet might jump out of the bowl. During this stage victawet does not melt and only the organic component is evaporated.
 - b. When the pressure falls and you drive 60-70 A through the source, the organic component of victawet has left. If you chose to keep the shutter closed before, you should open it now.
 - c. Keep increasing the power. There is still a risk that what is left might splutter from the source.
 - d. When the source is empty you have usually brought the source to white heat. Turn the power off when the source is empty.
7. Turn the cooling water off.
8. Let air into the vacuum chamber.

It seems possible to use the same source an infinite number of times, even though it gets more brittle the more it is used. From what is said above it should be clear that victawet is far from ideal to evaporate. The residue left through the evaporation is identical to Graham's salt, which also can be purchased and then used as parting agent. When victawet is used in combination with vanadium, the organic part of victawet should not reach the substrate. This is since the organic part act as a contamination. If you try anyway, you will see that the vanadium film starts to flake off before the deposition is over.

Application of victawet by rubbing

A tiny piece of victawet is put on the substrate and then rubbed with a piece of cotton. The substrate is ready to be used when there is no trace of victawet visible to the unaided eye. I have tried this method only once and the layer was obviously uneven since the film release was very uneven. There is a substantial risk that you remove too much of the parting agent and experimentation and experience is needed to get a perfect result. To avoid lint on the substrate from the piece of cotton to stick, you can for instance hold it tight with a pair of tweezers.

Application of detergent by rubbing

Put a tiny drop of the detergent (for instance Extran MA-02 neutral, Merck) on the substrate and the rub with a piece of cotton. If you rub too vigorously, the detergent will foam. You should avoid this since foam gives an uneven layer. To keep lint from sticking on the substrate you hold the piece of cotton tight with a pair of tweezers. When the detergent has "dried" there is a layer with a characteristic brush pattern. This relief pattern is transferred to the thin metal film and the film also gets a large number of pinholes due to these irregularities.

Application of a detergent by submerging the substrate into a solution of the detergent

A very dilute solution of a detergent work as parting agent for formvar on microscope slides, but not for metal films. Simply submerge the substrate in to the solution (0.5 mg detergent + 200 ml water has been used) and let it dry well protected from dust and dirt.

APPENDIX D

Evaporation Source Table for the Elements

A compilation from Ref. 19, 32, and 33. A few elements are left out.

Element	Melting Point/°C	Partial Pressure 10 ⁻⁴ Torr/°C	Wire, Foil	Crucible	Remarks
Aluminum Al	660	1010	W	C, BN, TiB ₂ -BN, ZrB ₂	Wets all materials readily and tends to creep out of containers. Alloys with W and react with carbon. Nitride crucibles preferred. Stranded wire superior to single strand.
Antimony Sb	630	425	Mo, Ta, Ni, Al ₂ O ₃ , coated boats	oxides, Al ₂ O ₃ , BN, metals, C	Requires temperatures above melting point.
Arsenic As	814	210		oxides, C, Al ₂ O ₃ , BeO	Sublimes rapidly at low temperature. Toxic.
Barium Ba	725	735	W, Mo, Ta, Ni, Fe	metals	Wets refractory metals without alloying. Reacts with ceramics.
Beryllium Be	1278	1000	W, Mo, Ta	C, BeO, refractory oxides	Wets refractory metals. Very toxic, particularly oxides.
Bismuth Bi	271	520	W, Mo, Ta, Ni	oxides, BeO, Al ₂ O ₃ , C, metals	Vapours very toxic. High resistivity.
Boron B	2300	1797	C	C	Material explodes with rapid cooling.
Cadmium Cd	321	180	W, Mo, Ta, Fe, Ni, Nb	oxides, Al ₂ O ₃ , metals, quartz	Sublimes. Cd deposition can spoil vacuum. Wets Nb.
Calcium Ca	842	459	W	Al ₂ O ₃ , quartz	Corrodes in air.
Carbon C	Subl.	2137			e-beam or arc evaporation
Cerium Ce	795	1380	W	Al ₂ O ₃ , BeO, C	
Cesium Cs	28	30		quartz	
Chromium Cr	1890	1157	W, Ta	C	High evaporation rates without melting. Evaporation from radiation heated rod or electrodeposited filament.
Cobalt Co	1459	1200	W, Nb	Al ₂ O ₃ , BeO	Alloys readily with refractory metals. Charge should not weigh more than 30% of source to limit destruction.
Copper Cu	1083	1017	W, Mo, Ta, Nb, Al ₂ O ₃ , coated boats	Mo, C, Al ₂ O ₃ , TiB ₂ -BN	Mo preferred for crucibles because it conducts heat well.
Dysprosium Dy	1409	900	Ta		
Erbium Er	1497	930	W		
Europium Eu	822	480	Ta	Al ₂ O ₃	Low Ta solubility.
Gadolinium Gd	1312	1175	Ta	Al ₂ O ₃	High Ta solubility.

Element	Melting Point/°C	Partial Pressure 10 ⁻⁴ Torr/°C	Wire, Foil	Crucible	Remarks
Gallium Ga	30	907	Al ₂ O ₃ coated boats	BeO, Al ₂ O ₃ , quartz	Alloys with refractory metals. Oxides are attacked above 1000°C. Reacts with metals.
Germanium Ge	937	1167	W, Mo, Ta	W, C, Al ₂ O ₃ , quartz	Wets refractory metals, but low solubility in W.
Gold Au	1062	1132	W, Mo, Al ₂ O ₃ coated boats	Mo, C, Al ₂ O ₃ , BN	Reacts with Ta. Wets W and Mo
Hafnium Hf	2230	3090			E-beam evaporation is good.
Holmium Ho	1470	950	W		
Indium In	157	742	W, Mo	Mo, C, Al ₂ O ₃	Mo preferred for foils.
Iron Fe	1535	1180	W	BeO, Al ₂ O ₃ , ZrO ₂	Alloys with all refractory metals, therefore charge should not weigh more than 30 % of source to limit destruction.
Lanthanum La	920	1388	Ta	Al ₂ O ₃	
Lead Pb	328	497	W, Ta, Mo, Ni, Fe	metals, Al ₂ O ₃ , quartz	Does not wet refractory metals. Toxic.
Lithium Li	179	407		Fe, Al ₂ O ₃ , BeO	Oxide destroys quartz.
Lutetium Lu	1652	1300	Ta	Al ₂ O ₃	
Magnesium Mg	651	327	W, Mo, Ta, Nb, Ni	Fe, C, Al ₂ O ₃	Sublimes. Produces high reflective coating.
Manganese Mn	1244	647	W, Mo, Ta	Al ₂ O ₃ , BeO	Wets refractory metals.
Mercury Hg	-39	-6			
Molybdenum Mo	2610	2117			E-beam evaporation preferred. Fine wires evaporated with own resistance.
Neodymium Nd	1024	1062	Ta	Al ₂ O ₃	Low Ta solubility.
Nickel Ni	1453	1262	W, W foil lined with Al ₂ O ₃	refractory oxides, Al ₂ O ₃ , BeO	Alloys with refractory metals, hence the charge should be limited or use electroplated filament where Ni mass is less than 9%. E-beam evaporation preferred.
Niobium Nb	2468	2287	W		Evaporates rapidly. Alloys with W. Fine wires will self evaporate.
Palladium Pd	1550	1192	W, W foil lined with Al ₂ O ₃	Al ₂ O ₃ , BeO	Evaporates rapidly. Alloys with refractory metals. Resistance heated fine wire with rapid evaporation suggested.
Platinum Pt	1769	1747	W	ThO ₂ , ZrO ₂ , C	Alloys with refractory metals and must be evaporated rapidly. E-beam evaporation preferred.
Polonium Po	254	244		quartz	
Potassium K	64	125	Mo	quartz	
Radium Ra	700	416			
Rhenium Re	3180	2571			E-beam evaporation preferred. Fine wires will self evaporate.
Rhodium Rh	1966	2040	W	ThO ₂ , ZrO ₂	E-beam evaporation preferred. Use very low pressure for W source.

Element	Melting Point/°C	Partial Pressure 10 ⁻⁴ Torr/°C	Wire, Foil	Crucible	Element
Rubidium Rb	38.5	111		quartz	
Samarium Sm	1072	573	Ta	Al ₂ O ₃	
Scandium Sc	1539	1002	Ta	Al ₂ O ₃ , BeO	Alloys with Ta.
Selenium, Se	217	170	Mo, Ta, W, stainless steel 304,	Mo, Ta, C, Al ₂ O ₃	Wets all support materials. Se deposition may contaminate vacuum system. Toxic.
Silicon Si	1410	1337	(W)	BeO, ZrO ₂ , ThO ₂ , C	Refractory oxides crucibles are attacked by molten Si, resulting in SiO contamination. Load with small amounts of Si. E- beam gives purest films. Alloys with W
Silver Ag	961	648	Mo, Ta, Al ₂ O ₃ , coated boats	Mo, C, Al ₂ O ₃	Does not wet W. Mo crucibles are very durable sources.
Sodium Na	97	192		quartz	
Strontium Sr	769	403	W, Mo, Ta	Mo, Ta, C	Wets all refractory metals without alloying.
Sulphur S	115	57	W, Ta, Mo	quartz	Poisons vacuum system.
Tantalum Ta	2996	2590			E-beam evaporation preferred. Getters O ₂ . Fine wire will self evaporate.
Tellurium Te	452	277	W, Mo, Ta	Mo, Ta, C, Al ₂ O ₃ , quartz	Wets all refractory metals without alloying. May contaminate vacuum system. Toxic.
Terbium Tb	1357	1150	Ta	Al ₂ O ₃	
Thallium Tl	302	470	Ta, W	Al ₂ O ₃ , quartz	Wets Ta without alloying. Very toxic.
Thorium Th	1700	1925	W, Ta, Mo		Wets W readily. Toxic.
Thulium Tm	1545	680		Al ₂ O ₃	Sublimes with a fair rate.
Tin Sn	232	997	W, Ta, Mo?	C, Al ₂ O ₃	Wets Mo readily (Ref. 32 and 33). Wets and attacks Mo (Ref. 19).
Titanium Ti	1675	1453	W, Ta	C, ThO ₂ , TiC	Reacts with refractory metals. Deposition contains W traces. E-beam evaporation preferred.
Tungsten W	3410	2757			E-beam evaporation preferred. Forms volatile oxides. Fine wires will self evaporate.
Uranium U	1132	1582	(W)		Films oxidize.
Vanadium V	1890	1547	Mo, W	Mo	Wets Mo without alloying. Alloys slightly with W.
Yttrium Y	1495	1157	Ta	Al ₂ O ₃	High Ta solubility.
Zinc Zn	419	250	W, Ta, Ni	Fe, Al ₂ O ₃ , C, Mo, quartz	High sublimation rates. Wets refractory metals without alloying. Films oxidize readily. Wall deposition may spoil vacuum system.
Zirconium Zr	1852	1987	W		Wets and slightly alloys with W. Films contain traces of W. Requires good vacuum to avoid oxidation. E-beam evaporation preferred.

APPENDIX E

Adapted from J. Trail.

Program HenkeQ

```
Real data(5,301),dens,atomvikt,atomsperAA3,energy,f1,f2
Real lambda,multfact,delta,beta,absorb,phase
Integer i,j
Character infil*24, utfil*24,in*2,line*80,line2*16, a*1
character infil2*21,subst*2

write(*,*)'This program converts Henkedata (energy (eV), F1, F2)'
write(*,*)'to wavelength, n, k, absorption coeff (inverse'
write(*,*)'micrometer) and phaseshift/micrometer. Output'
write(*,*)'is suitable for QPRO. Input files are assumed'
write(*,*)'to start with 1 line of text. Atomic weight is'
write(*,*)'read from old henkedata files'
write(*,*)
write(*,*)'The outputfile will be "inputfile.out"'
write(*,*)
write(*,*)'Phaseshift might be wrong factor 2!'
write(*,*)
100 format(A)
write(*,*)'Enter inputfile (t.ex. he, SMALL LETT!)'
write(*,*)
read(*,100)in
infil='e:\henke\newdata\' // in // '.asc'
infil2='e:\henke\a_weight.dat'
utfil='g:\' // in // '.out'
write(*,*)'inputfile= ', infil
write(*,*)'outputfile= ',utfil
write(*,*)'Enter density (g/cc)'
write(*,*)
read(*,*)dens
open(unit=9,err=998, file=infil2)
read(9,100)line
do 800 iter=1,92
read(9,101)subst,atomvikt
101 format(A2,F12.6)
if (subst.eq.in)goto 801
800 continue
801 continue
close(9)
write(*,*)'Atomvikt= ',atomvikt
atomsperAA3=.602205/(atomvikt/dens)
multfact=4.484889E-6*atomsperAA3

c multfact is the classical electron radius divided by 2 pi, times
c the number of atoms per cubic angstroem. Unit is inverse angstrom
c square

open(unit=9,err=998, file=infil)
read(9,100)line
do 20 i=1,301
read(9,*)data(1,i),data(2,i),data(3,i)
20 if(data(2,i).lt.-9000)data(2,i)=0.0
continue
close(9)
do 30 i=1,301
lambda=12398.5/data(1,i)
delta=lambda*lambda*data(2,i)*multfact
beta=lambda*lambda*data(3,i)*multfact
absorb=125663.71*beta/lambda
phase=62831.9*delta/lambda
data(1,i)=lambda
data(2,i)=delta
data(3,i)=beta
```

```

        data(4,i)=absorb
        data(5,i)=phase
30      continue

c where the refr index is  $n=1-\delta-i\beta$ , and absorb is
c in inverse microns

        open(unit=9,err=999,file=utfil)
        do 40 i=1,301
40      write(9,110)data(1,i),data(2,i),data(3,i),data(4,i),data(5,i)
110     format(' ',F8.3,',',F11.7,',',F11.7,',',F11.7,',',F11.7)
        close(9)
        write(*,*)'Press return to end program'
        read(*,*)
        stop

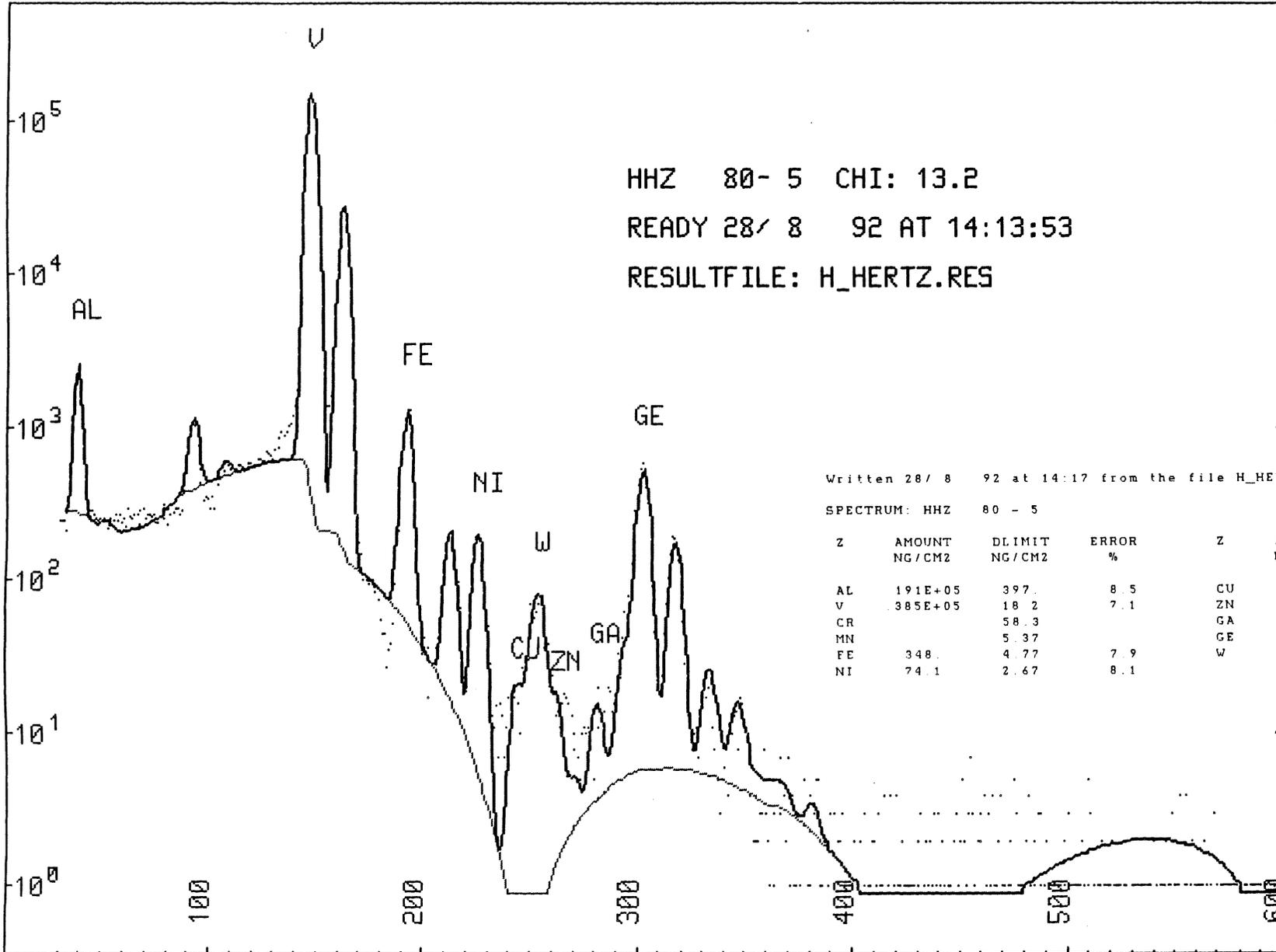
998     write(*,*)'ERROR opening henkefile'
999     write(*,*)'ERROR writing henke out file'
        end

```

APPENDIX F

PIXE data for AL/V/AI film,
discussed in Sect. 7.2.1.

HHZ 80-5 CHI: 13.2
READY 28/ 8 92 AT 14:13:53
RESULTFILE: H_HERTZ.RES



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