Care and feeding of soft x-ray and extreme ultraviolet filters

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ABSTRACT

Ultra thin metallic foils and organic films have been used as filters for the soft x-ray (SXR) and extreme ultraviolet (EUV) portion of the electromagnetic spectrum, particularly since the advent of space astronomy in the early 1970's. Much of the experience gained from manufacturing, testing and using these filters is applicable to other applications such as microscopy, lithography, and holography. This review paper attempts to correlate data and experience from a large number of projects. Transmission data is presented along with discussion of causes of filter damage. Lessons learned are presented in the form of general guidelines for preventing filter damage.

1. INTRODUCTION

For filters to perform in the SXR and ‘EUV spectrum, the foil materials utilized are typically a few hundred to a few thousand angstroms thick, and less commonly, they are a few microns (tens of thousands of angstroms) thick. The materials are fragile, and normally the foils have a fine mesh backing. Mesh supported metallic foil filters using 70 line per inch nickel mesh that is 85 to 90% transmitting are most common. If the filters are less than about a centimeter in diameter, they can sometimes be made without the mesh, or with an organic film for support. Common examples include unsupported gold and aluminum filters 1 cm in diameter and on the order of 700 to 2000Å thick. Extreme examples include a 3 cm diameter unsupported aluminum filter 12 microns thick and a **15** cm diameter unsupported Lexan film only 700Å thick. The Lexan was so fragile that it could not be shipped; it had to be hand carried, but the filter worked in its application.

SXR and EUV filters are generally not more than about 10 cm in diameter and more often in the range from 0.5 to about 4 cm in diameter. These sizes are usually large enough for collimated optics, such as on a synchrotron beam line or for positioning near detectors (analogous to eye piece filters on optical telescopes). Usually the filters are round, but sometimes they are square or pie shaped or even annular when used as entrance filters for grazing incidence x-ray telescopes. They are generally mounted on a one-piece frame using low outgassing epoxy, but if vibration is an issue, a two-piece frame is used. Sometimes they are installed within a gate valve so that they can be moved in and out of a beam line with relative ease.

For simplicity in this paper, these devices will always be referred to as filters, even though they are used in other applications such as photocathodes or laser targets. Likewise, they will be referred to simply as filters even though they are specifically designed for SXR and EUV applications (bandpass is loosely in the 1 to 15008, range).

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2. TRANSMISSION PERFORMANCE

Filter materials are chosen for their bandpass characteristics and mechanical properties. Aluminum is one of the most commonly used metals because it has a broad bandpass from 160 to beyond 750Å. It is good for eliminating visible light, it has good mechanical properties, and it is fairly easy to produce by vacuum deposition. Tin and indium work well if narrower bandpasses within the EUV are desired. Almost every material in the periodic table has been tried with varying degrees of success. Also, some salts such as potassium bromide (KBr) are sometimes used. However, salts are difficult because they have essentially no mechanical strength, and they often react with moisture and metals in undesirable ways. Organic films such as Lexan, polypropylene, and Parylene are commonly used. Their transmission characteristics are much like carbon, but there are subtle differences because of the presence of other elements (such as oxygen) in some of them; their advantage is in their mechanical properties. Whereas carbon films are very brittle, organic films are much more ductile and durable, but there are performance lifetime questions relative to radiation damage. For this reason, organic films are generally thought to be unacceptable for use in lithography.

Figure 1 gives performance curves for seven of the materials that are most likely candidates for filters for SXR and EUV lithography. Also see Section 2.3 for comments on aging relative to some of these materials. A much more detailed review of filter materials and their transmission performance is given in Reference 1.

FILTER FOIL TRANSMITTANCE

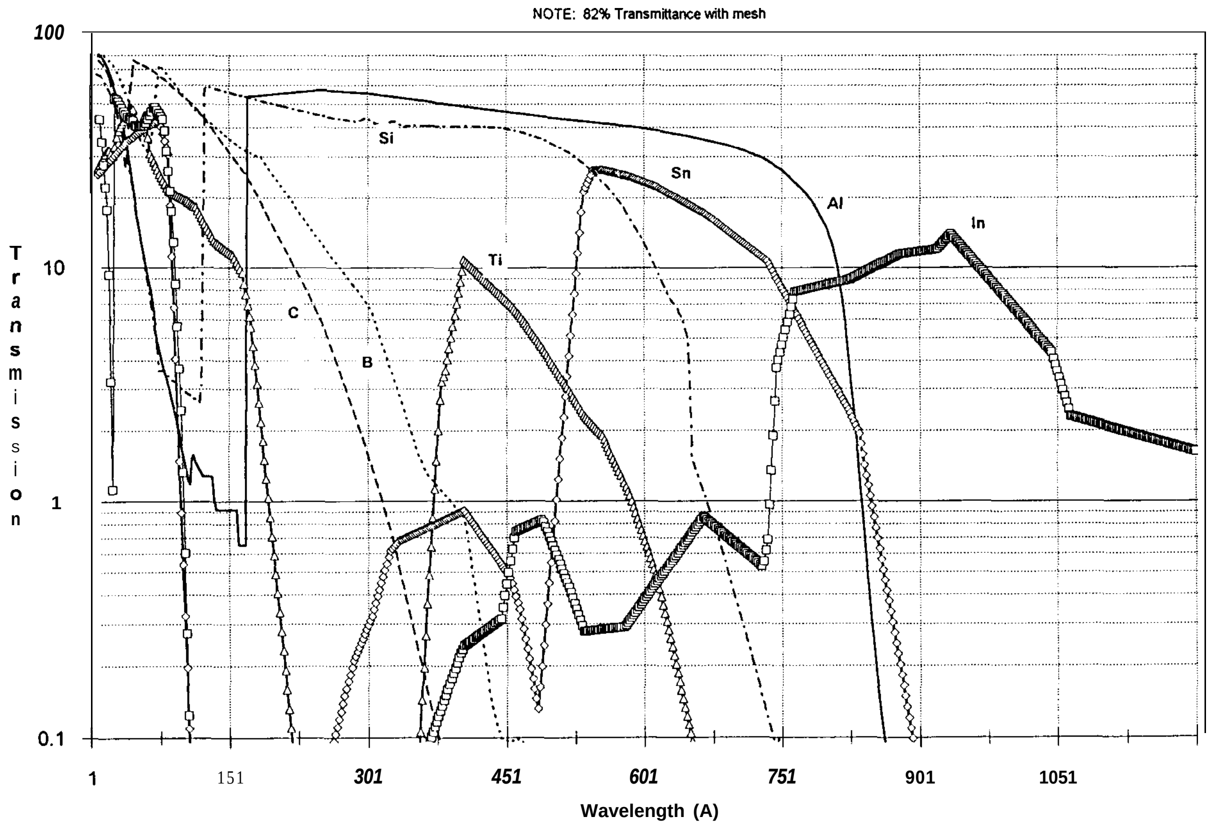

Figure 1. Transmission vs. wavelength for various materials

For the region from 130 to 150Å, silicon is generally thought to be the best candidate for thin foil filters although oxidation may be a problem. Carbon or boron are also possibilities; they show little evidence of aging or oxidation, and their transmission is stable over time. Another interesting possibility is a combination of carbon or silicon and phthalocyanine, which is an organic dye. The advantage of the phthalocyanine is that it has a higher neutral density than silicon or carbon, and therefore does a better job of rejecting any visible light that might be present, but the possible effects of high power radiation are not known. Titanium metal also shows some promise for this region of the spectrum.

For SXR lithography in the region of 8 to 15Å, aluminum, tin and titanium are all possibilities for use as filter materials.

3. FILTER DAMAGE

The enemies of filters, in approximate order of risk (starting with the ones that most often cause damage) are listed below. Each will be treated in order in more detail.

Microscopic particles (“shrapnel” and “rocks”)
Pressure differentials and pressure shocks
Moisture, air, and aging
Shock, vibration, and mechanical stress
Fingers, tools, and other unkind objects
Reactive materials
Contamination from air, water, solvents, etc.
Radiation, including everything from infrared to x-rays
Atomic oxygen (in space)

3.1. Microscopic “shrapnel” or “rocks”

One of the most common causes of damage to filters is microscopic “shrapnel” or “rocks”. Of course, larger (i.e., visible) particles can destroy a filter, but the microscopic ones can be just as dangerous, because they can catch people unaware when they are not expecting trouble. Part of the problem is that there are so many sources of these particles or “objects.”

Damage from microscopic dust usually occurs when turbulent gas or air stirs it up. Unfortunately, even an instrument assembled in a clean room may not be as clean as one would hope, and when the instrument is backfilled after being at vacuum, the microscopic dust “comes to life”. It will become airborne, and if the filter is exposed, the filter will get bombarded and damaged. Figures 2 and 3 show what can happen. The subject filter was intended for use in a solar astronomy mission. By visual inspection, it appears to be satisfactory (Figure 2), but during backfilling after a ground test, the filter had been “peppered” with microscopic dust particles and it was, in effect, destroyed. The extent of the problem is shown in Figure 3, which was photographed with back light to show the resulting pinholes. In operation, it would have allowed so much light to pass that the detectors would have been swamped, and the mission would have been a failure.

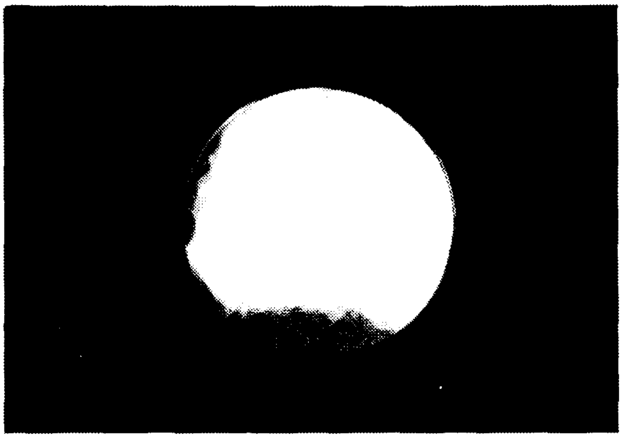

Figure 2. Front lit photo of filter

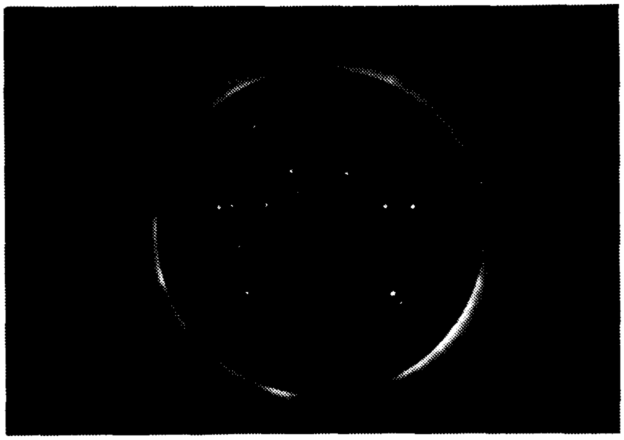

Figure 3. Back lit photo showing damage

(Filter is shown actual size)

In one case, a flight instrument was at the launch site being readied for spacecraft integration. Knowing that backfilling can cause problems, the instrument included a shutter to protect the filter; but the shutter could only be closed by remote control. The software that was supposed to send the command to close it did not, and the filter was badly damaged.

Other sources of damaging particles include devices such as ion pumps and x-ray sources which use a bombarded filament. These kinds of devices may generate debris which is not visible, but which can damage filters by making microscopic pinholes. For something like an ion pump, it is usually sufficient to make sure that there is no direct line of sight between the device and the filters. However, if the device generating the particles is a strong one, sometimes even a debris shield such as a pellicle made from an organic film is not sufficient because the particles may bounce off the chamber walls and go around a single barrier.

When a laser produced plasma is used as an x-ray source, much debris is generated. Greg Shimkaveg of the Lawrence Livermore National Laboratory (LLNL) .has reported that, because of problems during the calibration of expensive filters, they designed and built a baffle enclosure as shown schematically in Figure 4. At 150 to 16081, the two multilayer mirrors pass energy with about 20% efficiency and prevent filter damage, whereas a polypropylene pellicle which only transmitted about 10% in the band of interest did not prevent damage because debris got around it. Granted, the baffle system was elaborate and expensive, but in their case it was worth it.

If filter damage is a potential problem, tests using inexpensive witness filters to check out the situation are suggested. The researcher should err on the side of assuming that filter damage is a potential problem if there is even the slightest clue that it might be. At the very least, baffles placed in front of potential particle producing devices will reduce line of sight exposure for the filters.

If particle damage is unavoidable, a system design which places a small expendable filter where the beam is narrow is recommended. Often filter material is mounted unsupported on stainless steel washers with an inside diameter of only a few millimeters for this purpose.

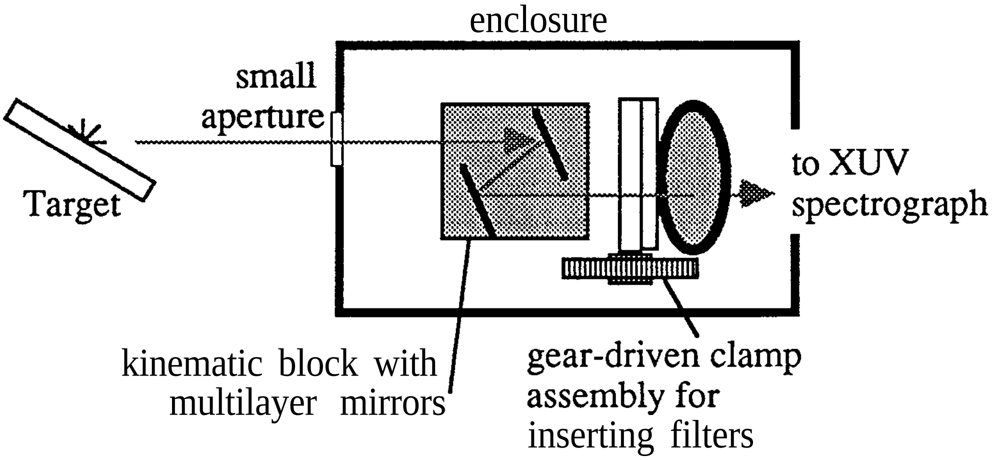

Figure 4. Schematic of baffle enclosure

In short, use covers, shutters, pellicles, baffles, convoluted paths and/or expendable filters to protect indispensable filters, particularly when they are not in use. If possible, do tests in advance to learn the extent of any potential problems.

3.2. Pressure differentials

Another common cause of filter damage is inadvertent exposure to pressure differentials. Whenever a vacuum chamber or any closed space is evacuated or filled, and at any time there is a pressure differential present, or a valve or door is opened or closed, or gas is moved from one area to another, or there is an acoustic shock for whatever reason, there is a potential problem. Many projects have reported serious troubles, including the total loss of filters.

The space science team at the UC Berkeley Center for EUV Astrophysics accomplished an extensive design and development project for the set of filters successfully launched this year on the Extreme Ultraviolet Explorer (EUVE) satellite mission. The spacecraft payload contains 20 separate thin film filters, the result of a development and optimization program lasting ten years. Two recent papers summarize this development.[2-3] On filter damage, Vallerga et al.[2] state, “Of all the times we have inadvertently damaged filters, it is fair to say the majority occurred during a pumpdown or backfill operation (excluding vibration [testing] damage).” They learned that all impulsive events such as valve openings and pumps starting could cause damage, and they developed careful procedures for equalizing pressures. When they backfilled, they used a dry nitrogen gas bottle to pressurize slightly above ambient and then bled back to ambient in order to avoid the “pop” when the chamber was opened.

Depending on the materials, most thin foil filters can only withstand a few torr of pressure, and it is easy to exceed their strength limits, especially when employed in systems with large volumes. Reports have been received of some of the more robust filters withstanding as much as 50 torr under static conditions, but the pressure differential from transient or acoustic shocks is essentially impossible to predict, and almost always more than expected.

Even handling can cause pressure differentials. If a filter is placed on a smooth flat surface, some air will be trapped under it, and the foil will balloon up and then gradually relax as the air escapes. It's best to avoid this kind of stress by putting a screen under the filter, or at least laying it down very slowly. Foils less than about 10008, thick are often broken this way. Even a tight fitting container such as a metal can will put a pressure load on the filter if it is opened too quickly. Filters have been known to have been broken just this way.

Shipping and storage containers need to be carefully designed to provide internal venting so that any pressure changes can be equalized. Needless to say, great care must be taken at all times to avoid any pressure differentials or pressure shocks.

In short, avoid transient pressures by changing pressures very slowly and making sure venting is proper.

3.3. Moisture, air, and aging

In one sense, aging is the most common cause of filter damage, because all filters age to varying degrees with time. The problem is usually moisture or oxygen, particularly in the presence of moisture. Therefore, the exposure to moisture, oxygen and any other reactive chemicals should be minimized.

The best way to store filters is in a vacuum. But even this is not the complete answer, probably because susceptible materials will act as a getter for trace amounts oxygen and moisture. For example, Hurwitz et al.4, have shown that tin and indium will continue to age some, even in a vacuum. A practical problem is that having filters stored in a vacuum makes them generally unavailable for testing and use.

The second best way to store filters is in dry nitrogen, and the third best way is in dry air. Generally speaking, dry nitrogen is preferable to dry air, but experience shows that it is not much better. Apparently the dry oxygen in air is not particularly reactive, and most materials do not tend to oxidize very quickly unless some water vapor is also present. Both dry nitrogen and dry air are good for storage if care is taken to ensure that the nitrogen really is dry and that the desiccant is fresh.

The UC Berkeley EUVE space science team was quite concerned about the effects of filter aging. Vedder et al.3 point out that the EUVE flight filters will be between 6 to 9 years of age at the end of the nominal 4 year mission life. At the time of the launch in June 1992, the Berkeley team had accumulated 6 years of data on filter performance vs. age, based on EUV calibrations both in and out of band, visible light photometer measurements on witness test filters, and physical examinations. They concluded that "These data to date do not show any evidence of physical degradation or significant changes in the transmission properties of the filters."

The materials utilized in the EUVE filters were Lexan, boron, aluminum, titanium, carbon, tin, silicon monoxide, and antimony. (Antimony has to be layered with something else to protect it from air at all times.) The witness test filters were generally stored in dry nitrogen except when in use, and the flight filters were also stored in dry nitrogen until the last 2 years before launch during which time they were generally stored at vacuum. The flight filters ranged in age from 2 to 5 years at the time of launch. In orbit, they are performing exactly as expected. Efforts to control aging do work.

However, the EUV portion of the spectrum is a difficult region for thin film filters because oxidation and aging are a real problem under ordinary working conditions. A significant amount of data collected on aluminum filters from several projects at various stages of aging under widely varying conditions has shown that the aluminum oxide will build with time, depending on conditions, from a few tens of angstroms to as much as 150 angstroms. [1] There seems to be a natural limit to the oxide growth at about this thickness except under extreme conditions. The data shows that the transmission of a 1500Å thick aluminum filter will degrade from almost 70% to about 50% at 200Å, whereas the degradation at 700Å will be from about 35% to less than 5% when 150Å of oxide has accumulated. However, with care, aluminum filters can be used for some time, and aluminum is probably the best choice for a filter material in the 600 to 800Å region being considered for projection lithography. It would probably be best to plan on changing them fairly often to get optimum performance and through put.

The other two materials that have good bandpasses in the EUV, namely tin with a bandpass centered at about 650Å and indium with a bandpass centered about 850Å, age quickly. As can be seen from the aluminum, the effect of oxygen generally worsens with increasing wavelength in this region. As a very loose rule of thumb, a tin filter left in air will loose most of its transmission within a year, and an indium filter left in air will be essentially opaque to EUV wavelengths within 6 months. Again, much more detail data on the transmission of various materials is given in Reference 1.

Sometimes salts such as potassium bromide (KBr) are used for filters or photocathodes. Salts, of course, are extremely sensitive to moisture, and extraordinary measures must be taken to protect them from moisture and air during manufacture and use. If left exposed, they will degrade within a matter of hours or days. Some excellent calibration and test data on KBr photocathode performance for an x-ray streak camera is presented in a test report by Cote et al. [5]. They show that KBr has a useful life as a photocathode material of 1 to 6 months if care is taken to avoid water vapor in storage and in use.

Luxel shipping containers are evacuated and backfilled with dry nitrogen to reduce aging effects during shipping and prior to first use.

3.4. Shock, vibration, and mechanical stress

In laboratory applications, shock and vibration are generally not too much of a problem. Reasonable care to avoid vibration from machinery and shock loads from banging things around is all that is required.

On the other hand, all spacecraft filters must pass rigorous shock and vibration testing. The launch environment is severe, and the boosters with large strap-on solid propellant engines (such as Shuttle) are the worst. Vibration levels will vary within a payload depending on the resonance characteristics of the particular mounting structure. The EUVE vibration specifications are typical. Generally the levels fall within the range of 10 to 20 g_{rms} for qualification and 5 to 15 g_{rms} for acceptance.[2] Generally,-acoustic loads put the most stress on the filters, and should be avoided if at all possible by launching with the payload evacuated so that the filters are in a vacuum. Lexan and aluminum filters do the best, and as might be expected, brittle materials tend to have problems.

For general use filters, shipping usually presents the worst shock and vibration loads. The filters in their shipping containers should be packed with care in shock-absorbent packaging. UPS and Federal Express have specifications for proper packaging and their guidelines should be followed. Drop tests on packaged filters have been conducted at heights up to 8 feet with some success, and with high success rates at lesser heights. Damage on fragile filters does occur in shipping, and sometimes having them hand carried is the only way to go if they are very expensive or particularly delicate. Again, brittle materials are the most likely to have problems. Shipping by air can also induce pressure cycling.

If vibration causes damage, it will make pinholes or tears around the edge of the filter. Two part frames provide better support and can be designed such that the edge region is covered to minimize light leaks in the event of problems. Pinholes are most commonly seen, but if the loads are severe enough to stretch the support mesh, tears can result. Enlarged pictures of a damaged aluminum filter are shown in Figures 5 & 6. This sort of damage is typical for a ductile material such as aluminum. If necessary, stainless steel mesh, which is stronger than the more commonly used nickel mesh, can be selected.

Figure 5. Front lit photo of damaged filter

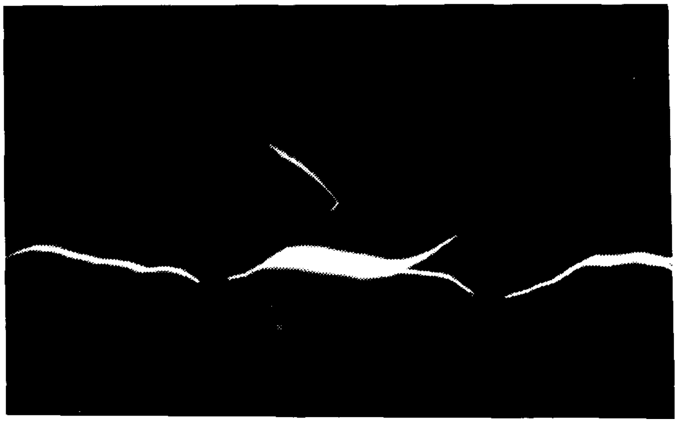

Figure 6. Back lit photo of filter

(Mesh "pane" size is 0.0143 in.)

Mechanical stress on a filter usually comes from the filter frame not being stiff enough, allowing tortional stresses to be transferred to the foil surface. It could be said that it is almost impossible to make a filter frame stiff enough, because the slightest flexing of the frame will usually cause wrinkles in the filter material. Wrinkles may not be a problem other than for cosmetics, but they can cause stress concentrations which can cause failures and thus should be avoided. Filters which are not round have the most problems in this regard. Sometimes grazing incidence x-ray telescopes have entrance filters which have an annular shape, and these are particularly difficult. This problem is discussed by Jurcevich et al.[6] in their paper on the structural design of Lockheed's Solar-A soft x-ray telescope. It is stated that the entrance filters were "one of the most challenging design problems" of the telescope in spite of how simple they look.

Even shipping containers must be designed with care relative to mechanical stress. To serve their purpose, they should be much stiffer than the filter frame. It may be possible to accidentally apply uneven force to the filter frame when installing it in the container, thus causing wrinkles and potential problems, including permanent set in the filter foil or frame.

3.5. Fingers, tools. and other unkind objects

Almost anything that touches a thin foil filter will damage, if not destroy it. If any tools are going to get close to a filter, it may be best to make a cover to protect the filter from possible damage. One researcher, very proud of his new filter set, laid them out on a pretty velvet fabric to get their picture. He then turned them over, face down on the soft fabric to get a picture of the back side to show the frames. The filters were gone! As soft as the nap on the velvet was, it riddled the foils with countless holes. He had no idea that the filter foil was that fragile, but it is. Almost without exception, nothing can be allowed to touch them. Period.

The production crew at Luxel calls mishaps with tools and fingers "yield" problems, as in microelectronics For any given order or production run, a number of filters are produced, and the yield of good filters is almost always less than 100%. (If we hated ourselves for every accident that happened, we couldn't work in this field.) Thin foil filters are surprisingly strong, (just like a soap bubble is very strong for what it is) but as has been made obvious by now, they are also very fragile. The researcher should plan on some attrition, and if at all possible, have spare filters to replace those that fall victim to "yield" or other problems.

3.6. Reactive materials.

There is enormous variation in the properties of materials utilized for thin film filters. Some of them are strange, such as the ability of tin to grow whiskers when under stress[2], or the fact that some materials can be in either an amorphous or crystalline state, and this can sometimes effect the transmission of the filter. Even more complications can arise from combinations of materials that are either co-mixed or layered. Some combinations can form unexpected compounds including intermetallic compounds. Again, the transmission characteristics of the filter can be changed by such interactions. Knowledge and testing provide the only sure answers for any particular application.

Combinations of salts and metals almost always cause problems. Figure 7 is an enlarged photo of a foil x-ray laser target composed of aluminum and sodium fluoride. (The actual target area is 0.3 mm x 1 .O mm.) The materials had been co-mixed during deposition, and originally the materials were uniformly distributed, but they obviously did not stay that way, particularly in the presence of moisture in the air. Often there is no known solution to this kind of a problem. In this particular case, nature provided a unique solution in the form of a naturally occurring compound, cryolite, which has a formula Na_3AlF_6. This compound can be uniformly deposited, as shown in Figure 8. (Paul Springer of LLNL provided these two photos.) Some knowledge and pure serendipity came into play on this one!


Figure 7. Al and NaF target

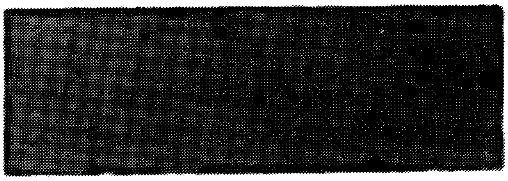
Figure 8. Cryolite target

3.7. Contamination

Contamination of filters after manufacture usually comes from dirty air. It is best to handle filters in a clean room or at least under a clean hood. Generally, they should not be handled with bare hands, and if one is going to get close to them, it is best to wear a face mask.

Generally speaking, filters cannot be cleaned, particularly if they are less than about 1000Å thick. A speck of lint can sometimes be blown off with a gentle puff of clean air from a hand-held air syringe or "puffer". Rarely, a more robust filter can be given a gentle rinse with isopropyl achohol, but it usually doesn't work, and should only be tried as a last resort. Plan on routine replacement if exposure to contamination cannot be avoided.

3.8. Radiation. including everything from infrared to x-rays

Of all the things that can happen to filters, radiation damage seems to have the least written about it. Some data is available, but more work needs to be done in this area.

Moore et a1.7, in their paper on the testing of a large variety of neutralizer foils in an accelerator beam, points out that when a foil is supported by a metal mesh, almost all the heating occurs in the metal grid, and from their work, the most durable foils were carbon on a carbon grid and free standing polycarbonate (Lexan). The Lexan was only 8308, thick. (And as an aside, and as discussed previously, they witnessed damage to foils from both pump down and bleed up which they found had to be done "extremely slow. ")

Peter Guttmann, from the Universitat Gottingen, and working at BESSY on x-ray microscopy, has reported that polypropylene and polycarbonate (Lexan) films are destroyed by synchrotron radiation that polyimide films can withstand. Specifically, he subjected a 0.25 µm thick Lexan foil to a power density of about 170 $\mathrm{mJ/(sec\cdot cm^2)}$ and it did not last, whereas a 0.3 µm polyimide window in the same beam was stable for some months. It is believed that damage is not due to melting, but rather to other effects such as the breaking of chemical bonds. Where radiation is an issue, polyimide films probably offer the best choice among organic films. Silicon, carbon and some of the metals are probably better, if acceptable transmission can be achieved.

Some people working in x-ray lithography at wavelengths in the region of 10Å have concluded that even polyimide is not suitable for long-term use. In one case, the expected radiation is on the order of 2×10^7 rad per exposure of about 5 secs. Under these conditions polyimide might be good for 100 to 1000 exposures or a total dose on the order of 10^9 rad, and that is probably not acceptable for a production system. Thus, the plan is to use a thick (20µm) beryllium window that will pass the radiation and also withstand atmospheric pressure, making it relatively easy to keep the photoresist equipment separate from the hard vacuum of the synchrotron.

Heat sources, especially radiant heaters, can cause problems for filters. In a vacuum chamber, they should be mounted such that there is no direct line of sight between the heat source and the filters.

3.9. Atomic oxygen

For completeness, in spacecraft applications, the effect of atomic oxygen must be considered. Atomic oxygen in space ablates materials to varying degrees. Carbon and especially Lexan are susceptible. The best solution is to overcoat them with a very thin layer of some other material such as boron or aluminum. This was what was done on the EUVE[2], and all indications are that the problem is solved.

4. **CONCLUSIONS**

Ultra thin foil filters are delicate and must be treated with care. Some problems are unexpected, and handling must be based on knowledge of the risks. Most of the problems by far are caused by microscopic particles and inadvertent pressure differentials. Moisture is a constant and universal concern, and accidents with fingers and tools are a cause of heartache. The general rules and guidelines are as follows:

- Avoid drafts and air or gas currents that can stir up microscopic particles and protect the filters with covers whenever they are not in use.
- Avoid pressure differentials and pressure shocks, especially during the backfilling of vacuum chambers
- Avoid moisture and moist air or gas to inhibit oxidation and aging
- Avoid excessive shock, vibration and mechanical stress, including in shipping
- Never allow anything to touch the thin foil
- If possible, avoid reactive materials or plan to accept the effects
- Exercise contamination control at all times
- Avoid excessive radiation, including everything from infrared to x-rays, or plan to accept the effects
- If necessary, design the filters to combat the effects of atomic oxygen (in space)

5. **SUMMARY**

In spite of their fragility, thin foil filters will function well when treated properly. As far as is known, and in spite of the relatively harsh environment, no Luxel filter has ever failed to perform as expected in space. The lessons learned from the space testing programs and the experience of many varied projects relate to other applications, and by applying this knowledge, it is possible to design and build filters that will perform well in most situations.

6. **ACKNOWLEDGMENTS**

The author wishes to thank all the people named in the text and others who responded to our call for help in collecting data for this paper. Luxel will continue to try and serve as a central clearinghouse for knowledge and data on experience gained in handling thin foil filters for the benefit of everyone working in this field.

7. REFERENCES

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