Design of High Performance Soft X-Ray Windows

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ABSTRACT

Methods of designing strong, high transmission soft x-ray windows are discussed. A material which contains several elements, most notably O, N, and C, produce the most spectrally neutral window. It is noted that a predominantly single element material such as diamond is in reality an edge filter. A structure to support very thin films and to provide exceptional mechanical strength is discussed. Pressure cycling data for such a supported window are presented.

1. INTRODUCTION

X-ray windows play a pivotal role in x-ray technology. X-ray windows are used in sources and detectors to separate the neighborhood of the x-ray generation or detection from the use environment. While each use has its own requirements, there are some principles that should be used in designing an x-ray window. Because x rays are absorbed to some extent by all materials, minimizing absorption is one criterion in preparing windows. Also, for most uses there is a pressure difference across the window so that insuring pinhole free structure and sufficient mechanical strength to support the differential is another criterion for window design.

Absorption can be minimized by the proper choice of material and by using very thin films. Generally speaking, the materials of lowest absorption are composed of the lower atomic number (low Z) elements such as Be, B or C.

Over the years many different materials have been used as windows for soft x-rays. A primary use has been for proportional counters. More recently interest has developed for

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Si(Li) detectors. The materials used to date for proportional counters include: polypropylene, lexan, and formvar[1-4]. Materials used for Si(Li) detectors have been Be, BN_x, formvar, a-C:H (diamond-like carbon), and mylar[5]. The purpose of this paper is to discuss the design of x-ray windows with particular emphasis on windows for detectors.

2. METHODOLOGY OF STUDY

For this study, two primary criteria were chosen for window study: film transmission and material integrity. Evaluation of the first is amenable to computer simulation and this is the approach taken in this study. The second is more subjective and will be treated in a descriptive fashion. While materials can be ranked according to bulk moduli and yield strengths, the integrity and mechanical properties of thin films are very dependent in preparation techniques and microstructure.

3. COMPUTER SIMULATION

The calculations were done using a computer code called UM which was designed and written by us. It is written in FORTRAN 77 and was designed to do a wide variety of calculations involving x-rays, thin films, and crystals. It accesses a data base of atomic scattering factors. For the problem at hand these are used to calculate optical constants.

The atomic scattering factors were obtained from Henke's tables and expanded at BYU and Lawrence Livermore National Laboratory. Beer's law is used in optical calculations. This is justifiable since the films are always much thicker than the wavelength of soft x rays and hence interference effects are negligible. In addition, front and back surface reflectivity is calculated to be much smaller than 10^{-4} except at grazing incidence and thus negligible over the whole range.

Calculations were made over the range 10Å to 70Å (170 eV to 1200 eV). This energy range cannot be seen with beryllium windows used in typical Si(Li) detectors and includes the K_α radiation of all elements with atomic numbers from Z = 5 to 11 (boron to sodium).

4. RESULTS

In figure 1 and figure 2 is shown the computed transmission of windows which have typically been used for x-ray applications. Typical thicknesses were used in the computations and are noted in the figure. The bottom curve is that of $8\mu m$ (0.3 mil) beryllium (this is the thinnest rolled Be foil used for Si(Li) detectors). Such a thickness allows only about 45% of the sodium K_α radiation to be transmitted and a negligible fraction of x rays less energetic than that. (In practice the transmission of sodium K_α radiation is much less than 45%. This is due in part to at least two causes. First, the Be is typically overcoated

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with a ploymer to plug the holes that are usually found in such thin rolled Be. Second, since it is rolled, there will be a high percentage of beryllium oxide.)

Another common window is $6\mu m$ ($\frac{1}{4}$ mil) polypropylene, metallized with aluminum ($\sim$ 200 Å thick). This is used in proportional counters and is the next most transmissive material/thickness combination. Polypropylene windows $2\mu m$ and $1\mu m$ thick are also used. Curves for all three are shown in the figures.

It will be noted that the polypropylene is much more transmissive than is the typical Be window for the soft x ray region, particularly on the low energy side of the C absorption edge. One can see the effect of the carbon content by the rather dramatic absorption edge; the nitrogen K_α radiation is highly attenuated. In this respect, polypropylene resembles a carbon K-edge filter (allows carbon x rays to pass but blocks more energetic, particularly nitrogen, x rays). Indeed any material which is predominantly a single element or a single element with hydrogen will act as an edge filter. Polypropylene is not a single element but acts as such for soft x rays since the hydrogen is transparent and can be ignored. The transmission curve for 4000 Å of diamond is shown in figure 3. Like polypropylene it acts as an edge filter, extinguishing nitrogen x rays. However, because of its higher density and low hydrogen content, 0.4 μm of diamond is equivalent to about 1.9 μm of polypropylene.

There are a variety of materials which incorporate various low-Z elements into their structure. The transmission of 4000 Å of formvar (polyvinyl formal), which has been used in the past for proportional counter windows, is shown in figure 3. Another material which may be used for soft x-ray windows is a boron containing material such as BN. Also shown in figure 3 is the transmission for 4000 Å of hexagonal BN. Note that for equivalent thicknesses of 4000, Å BN windows, and especially formvar windows, are much more transparent than diamond for F, O, and N x rays. The BN falls below diamond for carbon x-ray transmission but is still useable at this thickness.

The superiority of the compound materials comes about for two reasons. First, formvar has a lower density than diamond, and second but more important than density, it is not a single element. It contains a mixture of carbon and oxygen. The importance of this second point can be seen in figure 4, which is the transmission of the same three materials for the same mass per unit area. This suggests that the best window material from the point of view of high transmission for a given mass thickness would be a mixture of C, N, and O. Hydrogen would be used as required to tie up the dangling bonds. A transmission curve for such a material is shown in figure 5. This is in contrast with single element windows like diamond which are "colored" in that they transmit the certain wavelengths better than others. Windows with 1:1:1 mixtures of the elements transmit the wavelengths which correspond to each element roughly equivalently. Such windows would be approximately neutral density for elemental soft x rays and can be termed "gray windows". Producing a "gray window" would be a noteworthy achievement. It should be noted some of the

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families of materials which contain C, N, and O are the polyimides, nylon and Kevlar.

Nonmetallic x-ray windows are normally metallized. In the case of porportional counters this is for electrical reasons. In the case of Si(Li) detectors this is to block the transmission of infrared and visible light which would otherwise generate unwanted noise. The metal almost invariably chosen for this task is aluminum. It shows about the highest ratio of visible light extinction to soft x-ray extinction of any metal and is self passivating in air. The soft x-ray transmission of various thicknesses of Al are shown in figure 6. In the visible range 400 Å of aluminum extinguishes more than 95% of the incident light. The overall transmission of the x rays is the product of the film and the aluminum transmission.

The x-ray transmission is only one issue. The window must possess mechanical properties commensurate with the task of maintaining a pressure differential of approcimately one atmosphere for long periods of time and/or under cycling conditions. The properties which go into achieving this include film mechanical strength, film perfection, and the support structure. It must have sufficient strength to survive a pressure differential of over one atmosphere and sufficient rigidity not to flow. Flow or stretching can cause cracking of the aluminum thus compromising the window.

As has been discussed above, it is desirable for transmission purposes to make a window as thin as possible. As a material is made thinner, there comes a point at which it is not mechanically robust or reliable. This will depend upon the area being spanned. The larger the area, the greater is the pracitcal limiting thickness. It was long ago realized that with some kind of support grid, effectively larger areas could be covered with thin films. Recently our group at BYU developed a method of making a very close spaced grid for the support of thin films for the purpose of soft x-ray windows for Si(Li) detectors. A patent is pending for the method and resulting structure. The method consists of micromachining a grid from silicon. Typically the structure has bars 25 μm wide separated by 100 μm, resulting in a fractional free area of 80%. Windows with such a support structure have been made using formvar, Si_3N_4, a-C:H, and other materials.

With such a structure, film strength rarely is a serious issue. Calculations show that as little as 1 nm of Si_3N_4, if the material were to possess bulk properties and were free of defects, when stretched over a 200 μm opening could support a pressure differential of one atmosphere. Such a thin film is not practical since thin films rarely possess bulk properties and a single weak spot could render worthless an otherwise acceptable window. Window integrity is thus usually more important than bulk yield strength as a window selection criterion. It is well known that film perfection depends on film formation. Factors which affect the film perfection include care, cleanliness, and choice of formation techniques.

Film structure must fulfill another role with Si(Li) detectors. It must block the diffusion

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of gases into the region of the detector. This puts further constraints on the microstructure of the window membrane and particularly discriminates against polycrystalline materials such as diamond. .

Windows made of 4000 Å of formvar with 400 Å of Al have been pressure cycled. The films were on a silicon support structure with a bar width of 25 μm and a spacing of 100 μm. The active area was circular with a diameter of 6 mm. The high pressure side was pressure cycled to 15 PSIG of argon. A pressure sensor was on the other side of the window. The time to pressurize and depressurize was 1 sec. Cycle times have been 2 sec. and 6 sec. There was no discernable difference in results for the two cycle times. The results of this stress test are summarized in figure 7. The data have been fit to a lognormal curve (see Appendix). These data indicate a T_{50} (median cycles to fail) of 5800 cycles. The mean cycles to fail is approximately 5000. Similar results have been obtained for diamond windows.

5. CONCLUSION

Criteria for designing soft x-ray windows have been discussed. It was shown that a thin material containing several elements, most notably O, N, and C will create a reasonably neutral window for the 10 Å to 70 Å region. Furthermore, it was pointed out that materials such as diamond or polypropylene are essentially edge filters. A structure which allows creation of very thin, high transmission, and mechanically strong windows was disclosed. Pressure cycling data for such a window were presented.

6. APPENDIX

Lognormal Distribution

Probability density:

$$f(T) = \frac{0.4343}{T\sigma\sqrt{2\pi}} \exp\{-\frac{1}{2}(\frac{log\frac{T}{T_{50}}}{\sigma})^2\}$$

where $log = log_{10}$, T is the independent parameter, in our case it is the number of cycles to fails, and T_{50} is the number of cycles for 50% of the samples to fail. It is very difficult to obtain sufficient data to "divine" the probablity density itself, but it is easy to get the cumulative probability density.

Cumulative probability distribution:

$$F(T) = \int_{x=0}^{x=T} \frac{1}{\sqrt{2\pi}} \exp\{-\frac{1}{2}(\frac{log\frac{x}{T_{50}}}{\sigma})^2\} d(\frac{log\frac{x}{T_{50}}}{\sigma})$$

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σ is found from:

$$\sigma = logT_{50} - logT_{16}$$

where now T_{16} is the number of cycles for 16% cumulative fails.

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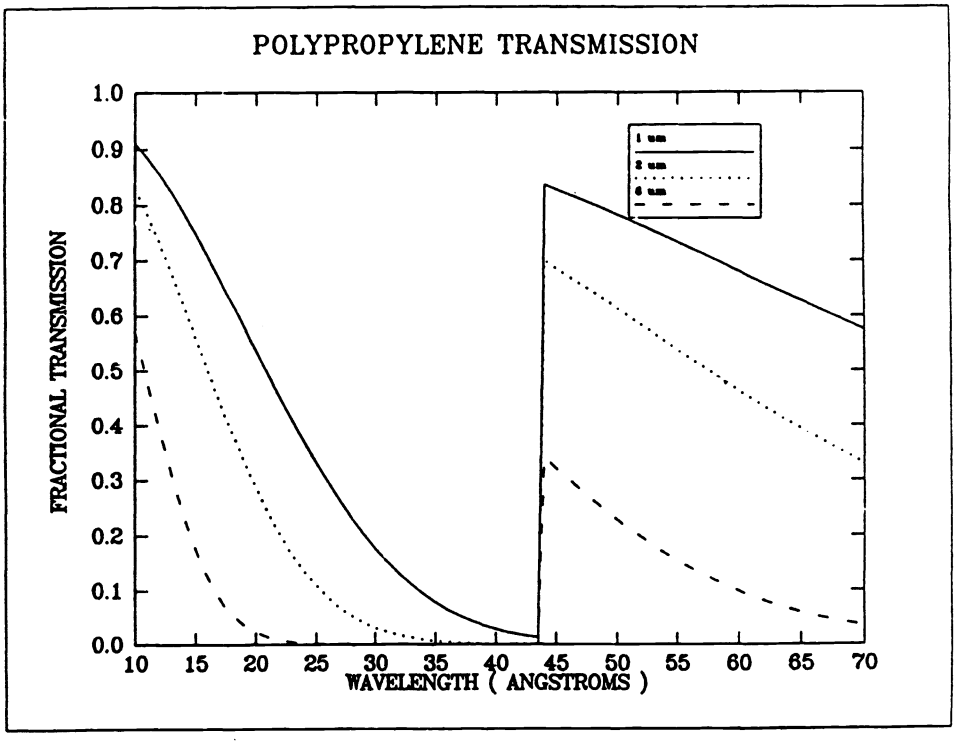


figure 1. Transmission for 1 μm, 2 μm, and 6 μm thick polypropylene.

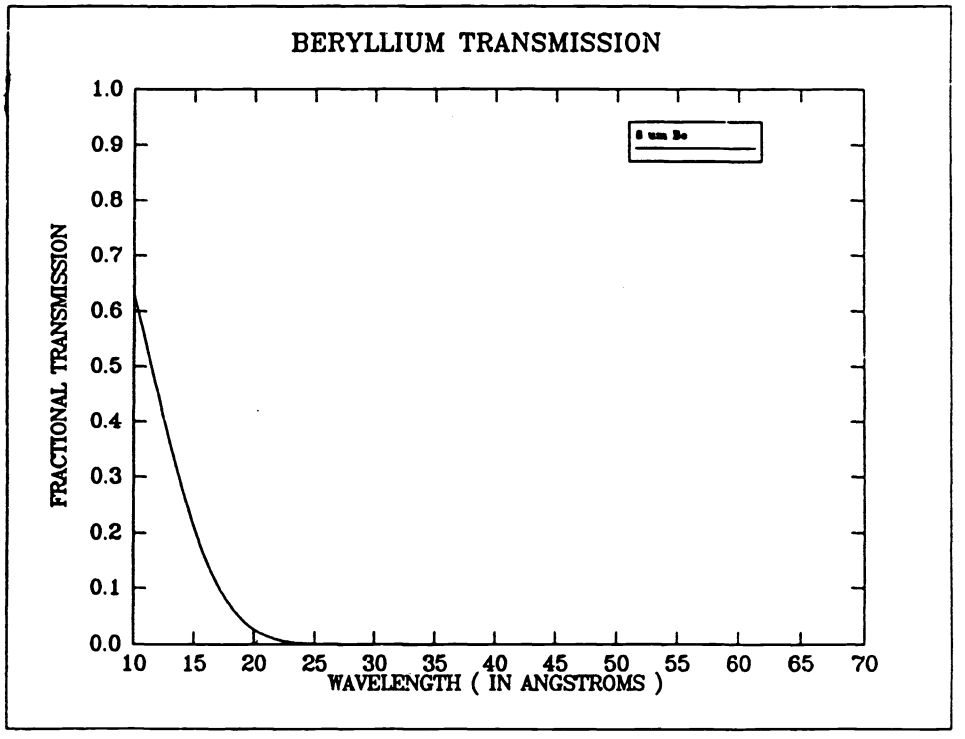


figure 2. Transmission for 8 μm thick beryllium.

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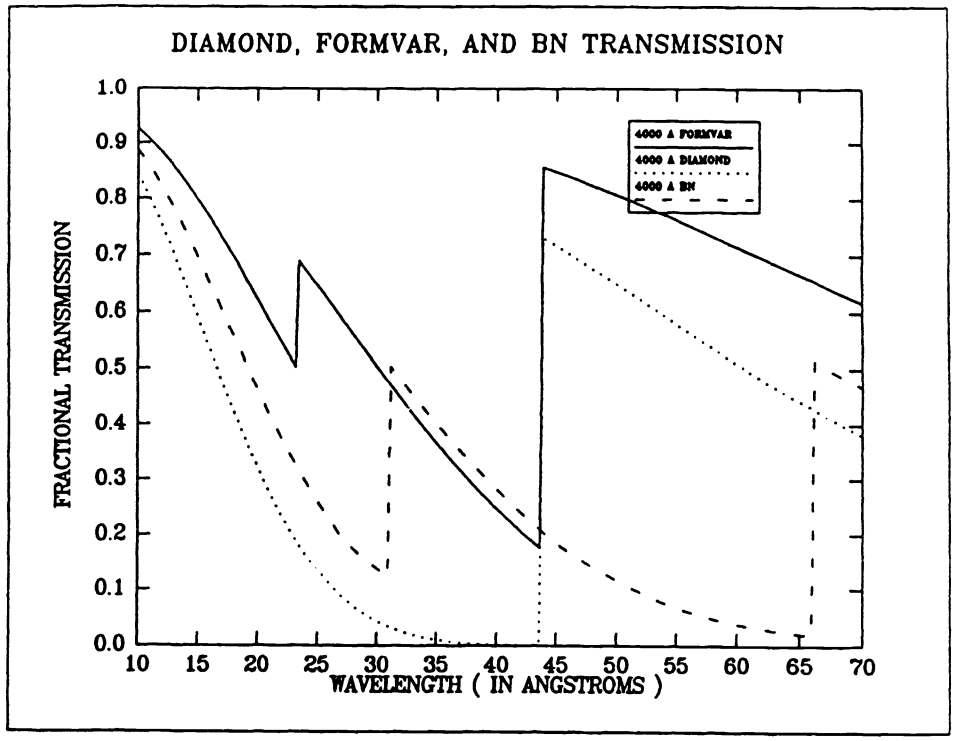


figure 3. Transmission for 4000 Å of formvar, diamond, and BN.

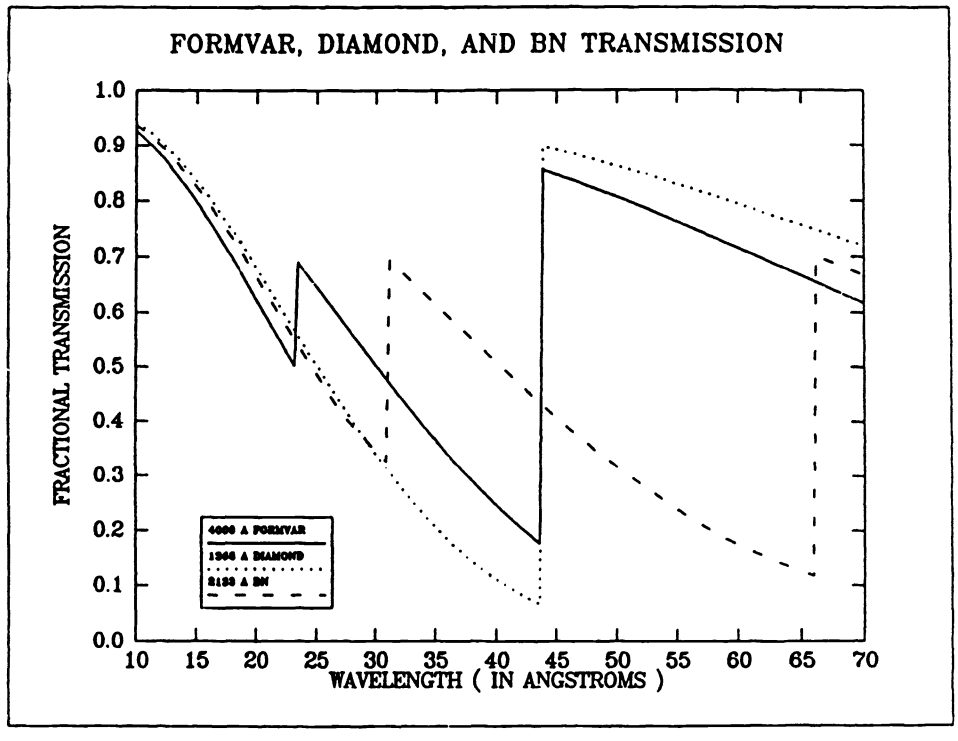


figure 4. Transmission for 4000 Å of formvar, 1388 Å of diamond, and 2133 Å of BN.

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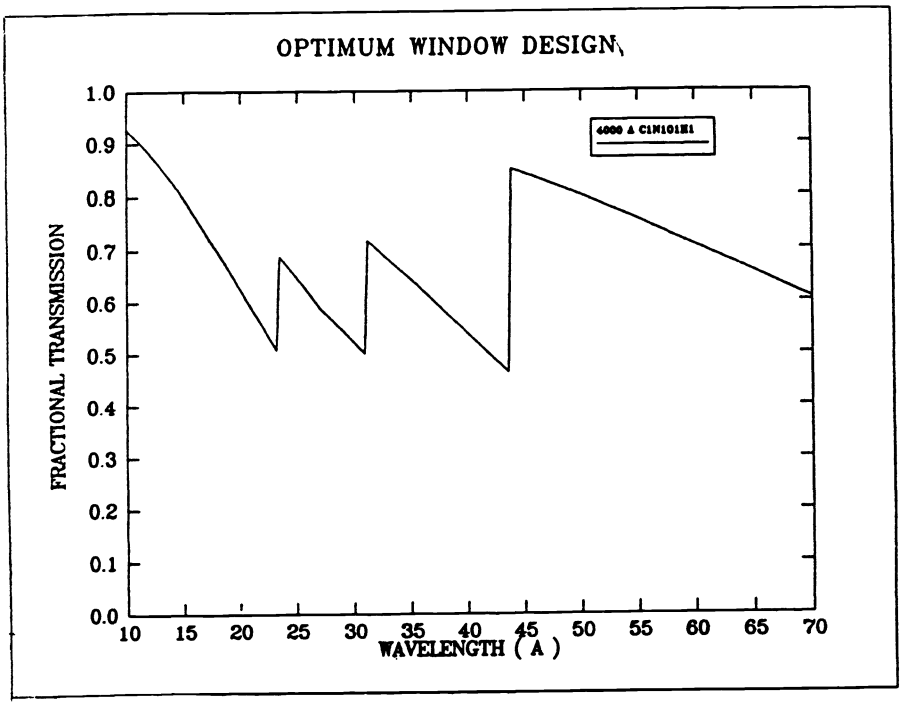


figure 5. Transmission for 4000 Å of an idealized material.

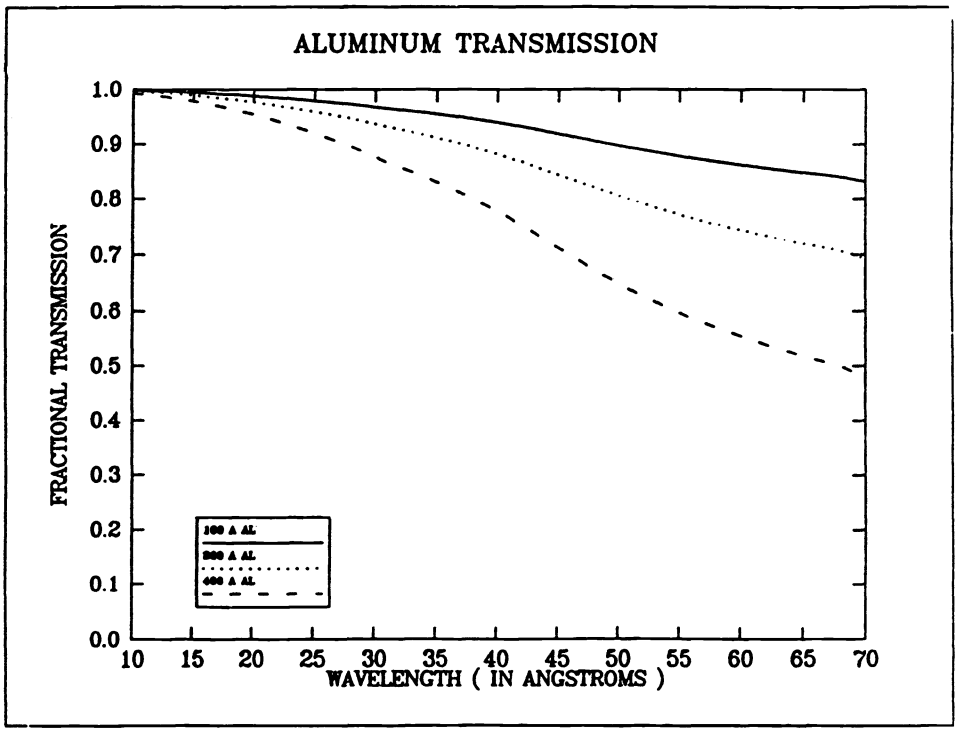


figure 6. Transmission for 100 Å, 200 Å, and 400 Å of Al.

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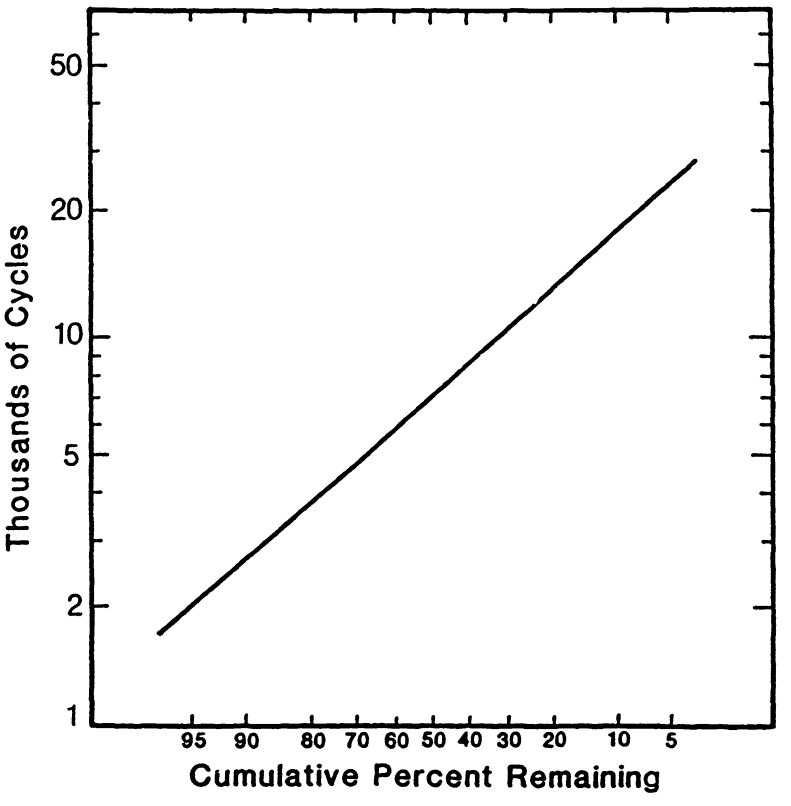


figure 7. Pressure cycle results for 4000 Å of aluminized formvar on 80% transmission grid.

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