SPACE ASTRONOMY
OF THE
STEWARD OBSERVATORY

THE UNIVERSITY OF ARIZONA
TUCSON, ARIZONA

RESEARCH IN FAR ULTRAVIOLET FILTERING IN SPACE OPTICAL SYSTEMS

T 69-16

RESEARCH IN FAR ULTRAVIOLET FILTERING FOR SPACE OPTICAL SYSTEMS

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INTRODUCTION

This program was initiated to investigate the design and fabrication of multilayer interference filters for the far ultraviolet. The research has been supported, in part, by NASA institutional grant NGR-03-002-091 beginning in June 1966. This document constitutes the final technical report for that grant. Most of the equipment utilized was derived through NASA contract NAS8-20651, grant ~~NSG-732~~, and general funds within the Optical Sciences Center. (NGR-03-002-032)

The main impetus for this program has been provided by a need for both reflection and transmission filters to be used in an ultraviolet sky survey camera which is being developed under NASA contract NAS8-20651 and grant NSG-732 to record ultraviolet light from stellar and nebular objects in the region from 140 nm to 300 nm. The most important filters for this project are reflection filters, which will be applied to the three mirrors of the camera. For highest system efficiency, the mirrors must be highly reflective in a moderately narrow region in the ultraviolet and have low reflectance at longer wavelengths. Such mirrors are generally referred to as "solar-blind" mirrors. Ultraviolet transmission filters are also required; these will be discussed briefly.

This report contains five sections following this introduction. First, computed theoretical properties of two types of reflection filters are presented, and a systematic design method is discussed. Second, problems associated with the vacuum system in which the filters are made are considered. The most critical part of producing interference filters is the control of the thickness of the filter layers. The optical monitor system developed for this work is described (third section), and the results are summarized (fourth section). In the final section, recommendations for further work are given.

THEORETICAL PROPERTIES OF ULTRAVIOLET FILTERS

The problem of designing and producing interference filters for the ultraviolet is basically the same as for any other region of the spectrum except that one is severely limited in the choice of materials with suitable optical properties. There are very few materials that are transparent in the ultraviolet. Rather than searching for "new" materials, we have limited ourselves to three materials: aluminum, chromium, and magnesium fluoride, which have a long history of use down to wavelengths as short as 120 nm.

Method of calculation

For purposes of calculation, we define an interference filter as an ordered series of plane parallel interfaces between media of different thicknesses and refractive indices, which is bounded by two semi-infinite media. Incident light is assumed to propagate from the medium of incidence toward the substrate medium. It is assumed that all layers are homogeneous and isotropic and hence can be characterized by a thickness and refractive index. As a result, we assume that there is a discontinuous change in the refractive index across a boundary. Previous experience indicates that this is a valid approximation.

Optical properties (reflectance, transmittance, and phase change on reflection) are calculated in a straightforward manner using a recursion relation which is a solution of Maxwell's equations with appropriate boundary conditions. The theory has previously been described by Berning.[1] The method used is called an admittance method. The essential parts of the calculation are described by Berning's Eqs. 151-156.

The computations were initially programmed for the IBM 7072 and, more recently, for the CDC 6400 computer in a program called THRUSH developed by the Optical Sciences Center of the University of Arizona. This is a general program which will compute the optical properties of any multilayer interference filter containing either metal or dielectric layers or a combination of them. The program can compute optical properties for discrete values of a variety of variables and will compute refractive indices of layers from dispersion data if needed. Fig. 1 is a block diagram of the basic program organization.

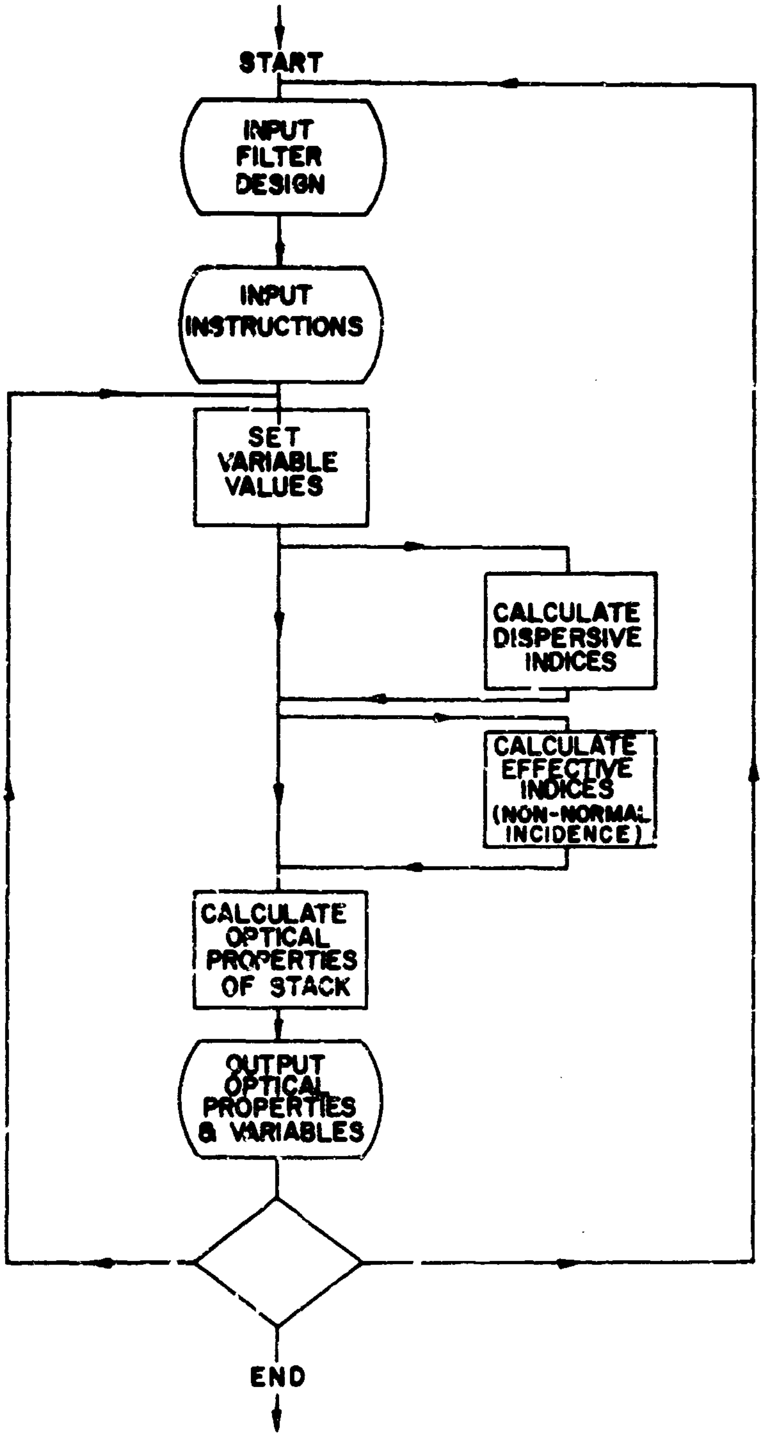


Fig. 1. Block diagram for THRUSH.

Computed properties of the mirror reflection filter

The simplest form of reflection filter is a mirror with an interference coating over it. We refer to this as a mirror reflection filter (MRF). A convenient form has been considered previously. It consists of a metal mirror coating with a dielectric layer and semitransparent metal layer over it. This configuration was considered by Hadley and Dennison.[2,3] They observed that there would be maxima and minima in the reflectance at wavelengths where the thickness of the dielectric layer was an even or odd multiple of a quarter wave.

The reflectance at a minimum depends on the thickness of the semitransparent metal layer. They considered the infrared region of the spectrum where they could make the approximation that the real and imaginary parts of the complex refractive index were approximately equal. They found it convenient to relate the thickness to the electrical resistance of the film such that, for a square film of thickness h and sides ℓ, the film resistance is inversely proportional to the thickness. For a resistance of 377 Ω (a thickness of 377 $\Omega/\Box$), they computed and produced reflection filters with zero reflection minima. These investigations were extended into the visible spectrum by Turner[4] using aluminum and magnesium fluoride. He was able to produce filters in the visible with peak reflectances of 0.90 and minima near 0. The form of the spectral reflectance, which was observed experimentally, was a number of fairly broad minimum reflection regions separated by narrow high reflection bands near the wavelengths where the dielectric film was a multiple of $\lambda/2$ optically thick. The shapes of the high reflection bands were symmetrical (somewhat like a Gaussian curve). Two problems arose in predicting the reflectance. First, the thickness of the semitransparent metal layer required for zero minumum reflection did not agree with experimental values, and second, the shapes of the computed curves were just the opposite of the measured curves (i.e., the reflection minima were narrow and the maxima were broad and asymmetrical). This same type of filter was also made in the near ultraviolet.[5] For these filters, Turner found that better stability was achieved when the semitransparent metal layer was changed from aluminum to inconel. The optical properties were essentially the same as all previous results.

Natawa[6] tried to resolve the discrepancy between the computed and measured results by changing the optical constants of the outer aluminum layer in such a way that the reflectance of the bulk material would be unchanged.

He found it necessary to change the indices from their normal value of N = 1.36 - 6.05 i to N = 8.0 - 12.0 i, a change that is hard to believe.

We also tried to resolve the differences between the well accepted theory and the experimental observations. Our approach assumes that the optical constants do not depend on the thickness of the metal film. This assumption is known to be invlaid when the films are very thin. We have tried to find the thickness for which a particular reflectance will be a minimum. A series of computations has been made for the MRF using the combination opaque Al, MgF_2, semitransparent Al. From these calculations we had hoped to find (1) the properties of such an idealized filter and (2) whether the optical constants of the semitransparent me are similar to thick film constants.

The results of the first calculations were unexpected. On checking the theory and computer program for possible errors, we found immediately that it was necessary to compute optical constants of the metal layer as a function of wavelength. Figure 2 shows how dispersion affects the optical properties of an MRF; curve A includes the variation of index with wavelength, and curve B uses a constant index. This indicates how strongly the optical constants of the metals affect the properties of an interference filter. The optical constants of aluminum used for this and all subsequent computations are shown below; the values were obtained from the *American Institute of Physics Handbook*[7] (for the region 700 - 220 nm) and from Hunter[8] (200 - 100 nm).

Optical constants of evaporated aluminum

λ (nm)	n	k
100	0.051	0.65
120	0.058	1.00
140	0.070	1.30
160	0.084	1.60
180	0.10	1.90
200	0.12	2.10
220	0.14	2.35
240	0.16	2.60
260	0.19	2.85
280	0.22	3.13
300	0.25	3.33
320	0.28	2.56
340	0.31	3.80
360	0.34	4.01
380	0.37	4.25
400	0.40	4.45
436	0.47	4.84
450	0.51	5.00
492	0.64	5.50
546	0.82	5.99
578	0.93	6.33
650	1.30	7.11
700	1.55	7.00

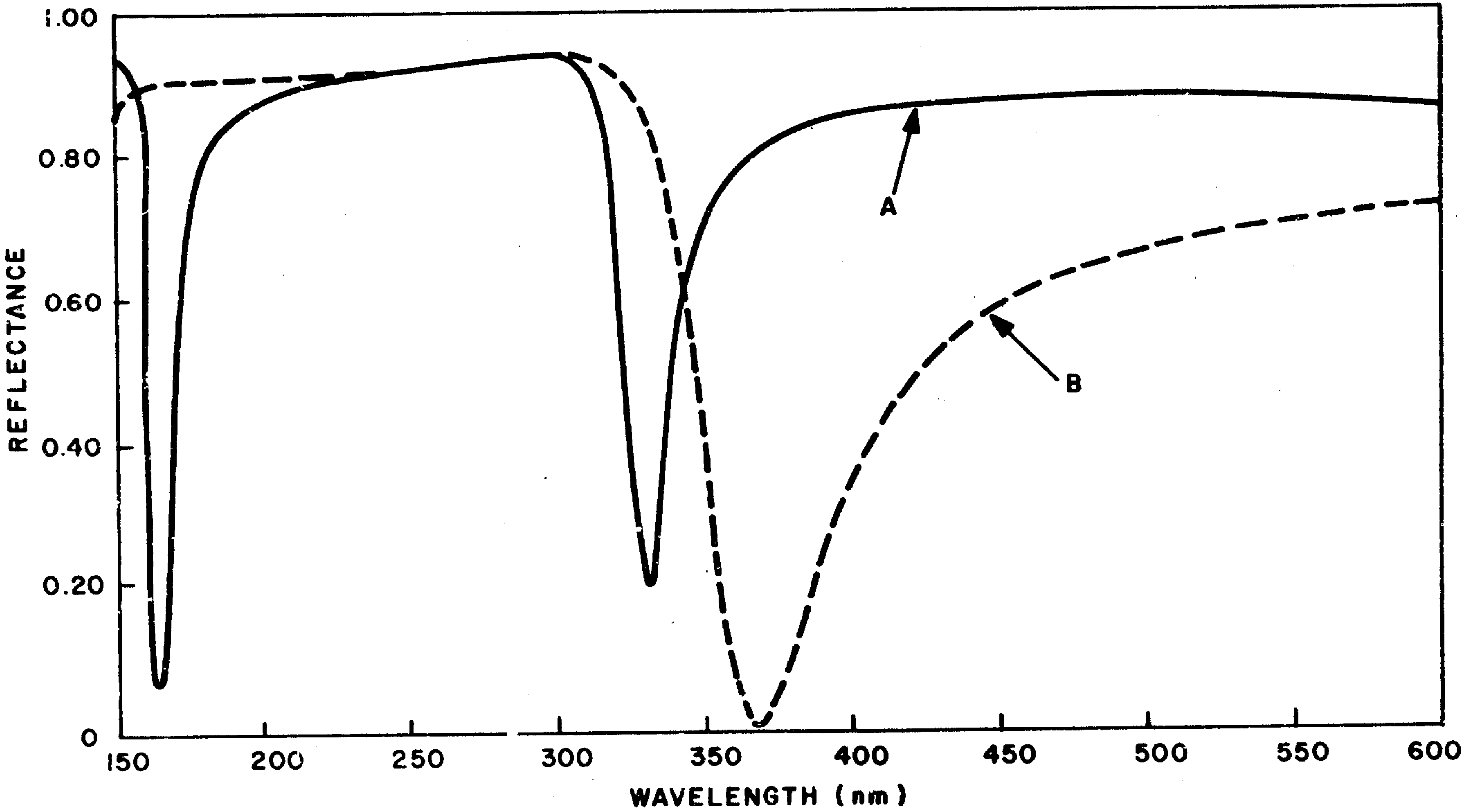


Fig. 2. The effect of the metal layer on the optical properties of a mirror reflection filter (MRF). (Curve A with dispersion, curve B without dispersion.)

Between data points the constants are computed from a modified form of the Sellmeyer equation:

$$n(\lambda) = A_n + \frac{B_n}{\lambda^2 + C_n} \tag{1}$$

$$k(\lambda) = A_k + \frac{B_k}{\lambda^2 + C_k} \tag{2}$$

where the constants A, B, and C are computed from the three closest values in the previous table. The values of the optical constants below 200 nm are extrapolated but are thought to be reasonably close since computed filter properties agree substantially with measurement.[9]

The properties of the mirror reflection filter can be described in a rather simple qualitative manner. There are two reflecting surfaces which are separated by a dielectric spacer layer. When the amplitudes of the waves reflected from the front and back surfaces are approximately equal, there will be a maximum reflectance when the waves are in phase and a minimum when they are out of phase. One could argue that a good approximation to the thickness for the semitransparent metal layer should be such that its reflectance is less than the reflectance of the opaque layer by a factor of T_1^2, where T_1 is the transmittance of the semitransparent metal layer. This value of the thickness depends on the wavelength of the minimum because of the dispersion of the optical constants, and this, in turn, depends on both the thickness of the metal and the thickness and index of the dielectric spacer layer. Thus, the design problem is no longer simple.

The properties of the mirror reflection filter consisting of opaque Al and MgF_2 and semitransparent Al are shown in Fig. 3 for a variety of metal thicknesses but constant dielectric spacer layer thickness. It is seen that a metal layer 10 nm thick gives a good minimum on the long wavelength side of the principal maximum, but the short wavelength minimum has not developed. A value of 20 nm seems to balance the two minima as seen in Fig. 2(curve A). It is apparently impossible to get the two minima to have zero reflectance simultaneously.

The shapes of these curves are in good agreement with earlier calculated results[4] but still disagree with experimental results. It can be argued that the computed results seem correct since they correspond to a Fabry-Perot or Fizeau interferometer in reflection where narrow dark fringes (corresponding to the narrow reflection minima) are observed against a bright background.

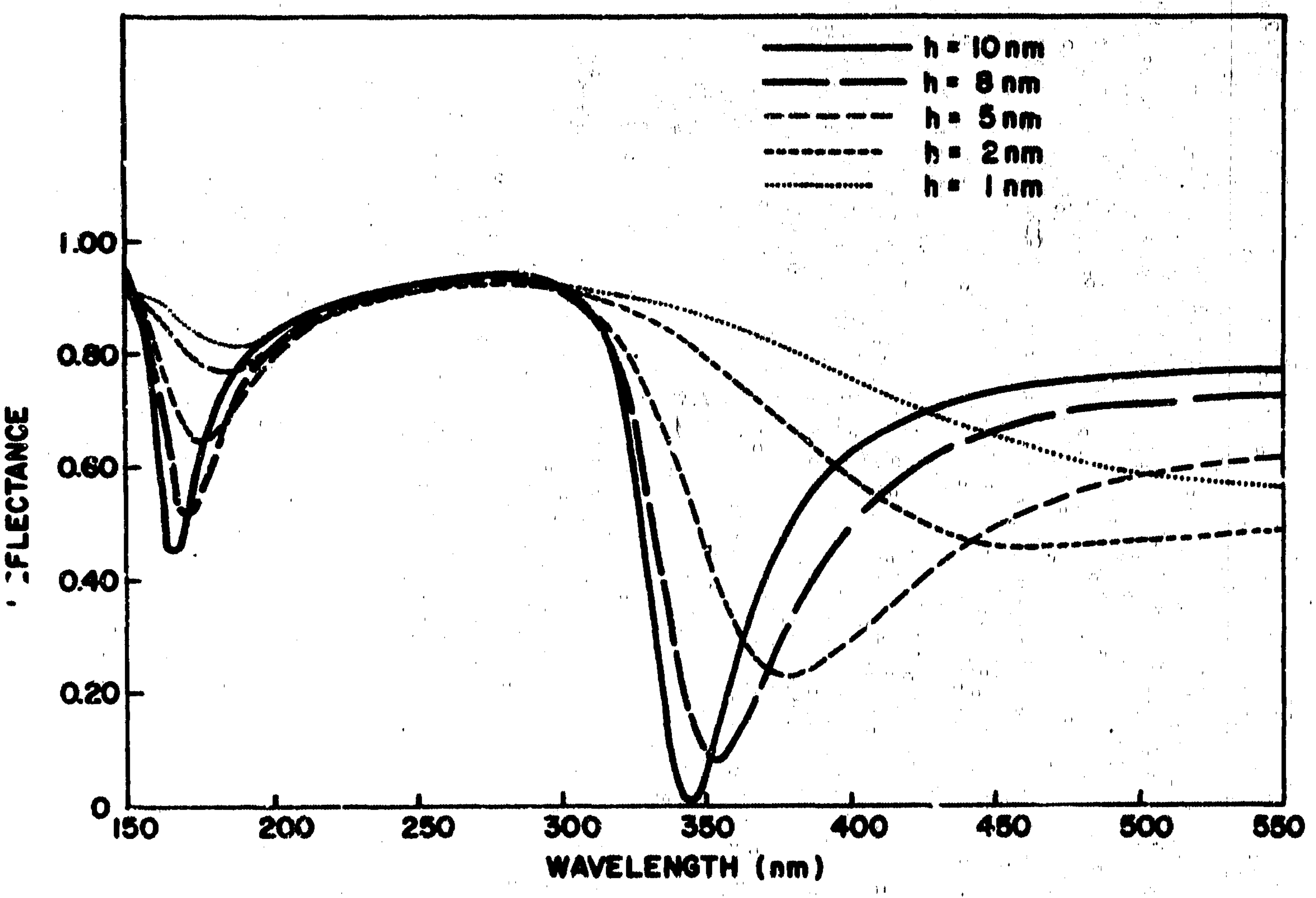


Fig. 3. Properties of a mirror reflection filter consisting of opaque Al, MgF_2, semitransparent Al, for different metal thicknesses.

For this same reason, we can argue that broader minima could be produced by using a semitransparent metal layer which has a lower reflectance. Metals with a lower ratio of k:n (imaginary:real part of the complex index) have this property. The computed reflectance of an MRF with chromium instead of aluminum as the outer layer is shown in Fig. 4. The narrower, more symmetrical reflection peak provides a better filter for broadband ultraviolet applications, and the lower background reflectance at long wavelengths provides a better solar-blind mirror. This type of filter has not been extensively investigated theoretically since the optical constants of chromium films are not well known. We have used limited data and considerable interpolation to arrive at the curve. It does not represent an "optimum" design using these materials. It may be possible to adjust the thickness of the chromium layer to balance the reflection minima and still retain the high peak reflectance. The shape of the reflection curve in Fig. 4 corresponds more closely with Turner's experimental results than when aluminum is used as the semitransparent film. The optical properties of chromium and inconel which Turner[5] used are similar. It is therefore reasonable to assume that the aluminum semitransparent films used by Turner were contaminated with aluminum oxide, which is known to increase the absorptance of aluminum. Experimental results discussed later support this assumption.

No simple means of designing MRF's has been found. In any iterative approach, the greatest amount of time is spent in finding the proper metal thickness, and it is necessary to do the computations as a function of the wavelength as previously discussed. As shown in Fig. 3, there is always a zero minimum, but its location for a given thickness of the dielectric layer would be difficult to predict without preliminary computations.

From our calculation, we conclude that a good solar-blind mirror cannot be made from three-layer combinations of aluminum and magnesium fluoride if the optical constants of aluminum are valid. The minimum is too narrow and reflectivity rises too rapidly again in the visible. It is obvious from older experimental results that there are certain conditions under which improved solar-blind properties do exist, but they are dependent upon such things as partial oxidation of the metal layers or impurities, and determining the best conditions would be a long task and would be difficult to reproduce using different coating apparatus. A more feasible approach seems

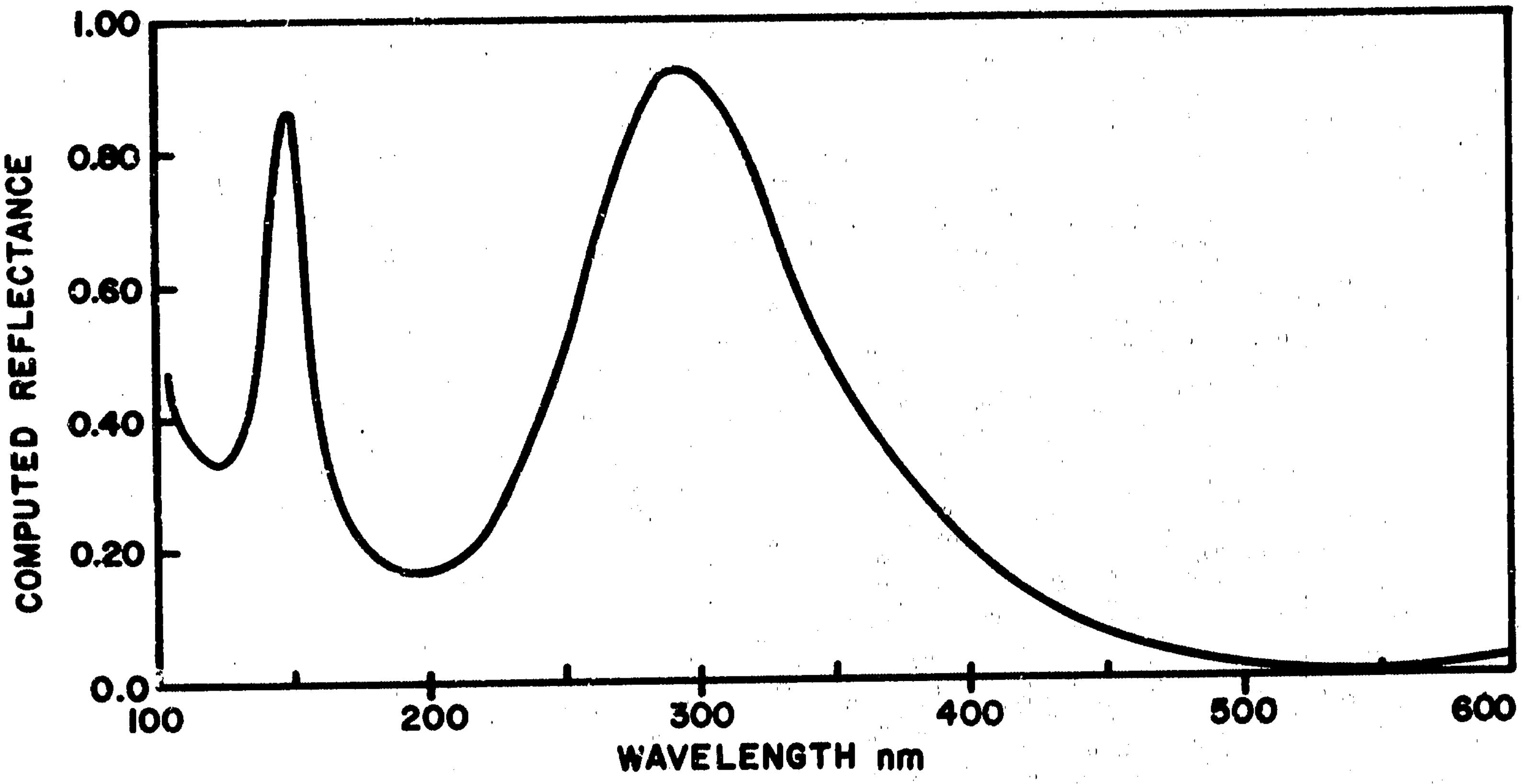


Fig. 4. The computed reflectance of a mirror reflection filter with chromium instead of aluminum as the outer layer.

to be that already pursued by Turner.[5] Either chromium or inconel will produce good three-layer solar-blind mirrors that would be durable and relatively simple to make.

One further point can be derived from the theoretical work. When monitoring the thickness of the metal layers during deposition, it is best to monitor each layer separately, in an attempt to get each layer as close to the optimum as possible. Finding the proper metal layer thickness for the semitransparent layer would otherwise be extremely difficult since a minimum reflectance is not necessarily the best thickness if there is a slight error in the dielectric thickness.

Computed properties of reflection filters with many layers

The spectral resolving power of the relatively simple filters discussed above is poor. The resolving power of any interferometer or interference phenomenon is increased as the number of coherent amplitude contributions is increased. Turner[5] demonstrated this by adding more transparent dielectric: semitransparent metal layer combinations to the three-layer mirror reflection filter. Using an empirical approach to find proper thicknesses, Turner found some good combinations. In this research, we have attempted to better understand the properties of these filters from theory to reduce the number of coating trials required and to try to find the optimum filter for a particular job.

We have begun by investigating what we call DMD stacks. The D stands for a dielectric layer which is one quarter wave optically thick and the M represents a metal layer. The three layers DMD represent a single symmetrical period, and the period may be repeated as $(DMD)^p$ or DMDDMDDM...MD. This class of filters has been found to have some interesting properties.

A series of calculations were carried out for DMD stacks in which both the number of periods p and the thickness of the metal h_M were changed, always keeping the dielectric layer thickness constant (250 nm quarter wave optical thickness). The results are shown in Figs. 5 and 6. We have plotted the reflectance and 1-transmittance. This procedure shows the absorptance as well since it must account for the gap between the two points. In Fig. 5, the number of periods p is varied. It can be seen that a relatively broad high reflection band is formed in the neighborhood of 250 nm and that the band becomes sharper and approaches a limiting width as the number of periods is increased. The thickness of the metal layers is changed in Fig. 6

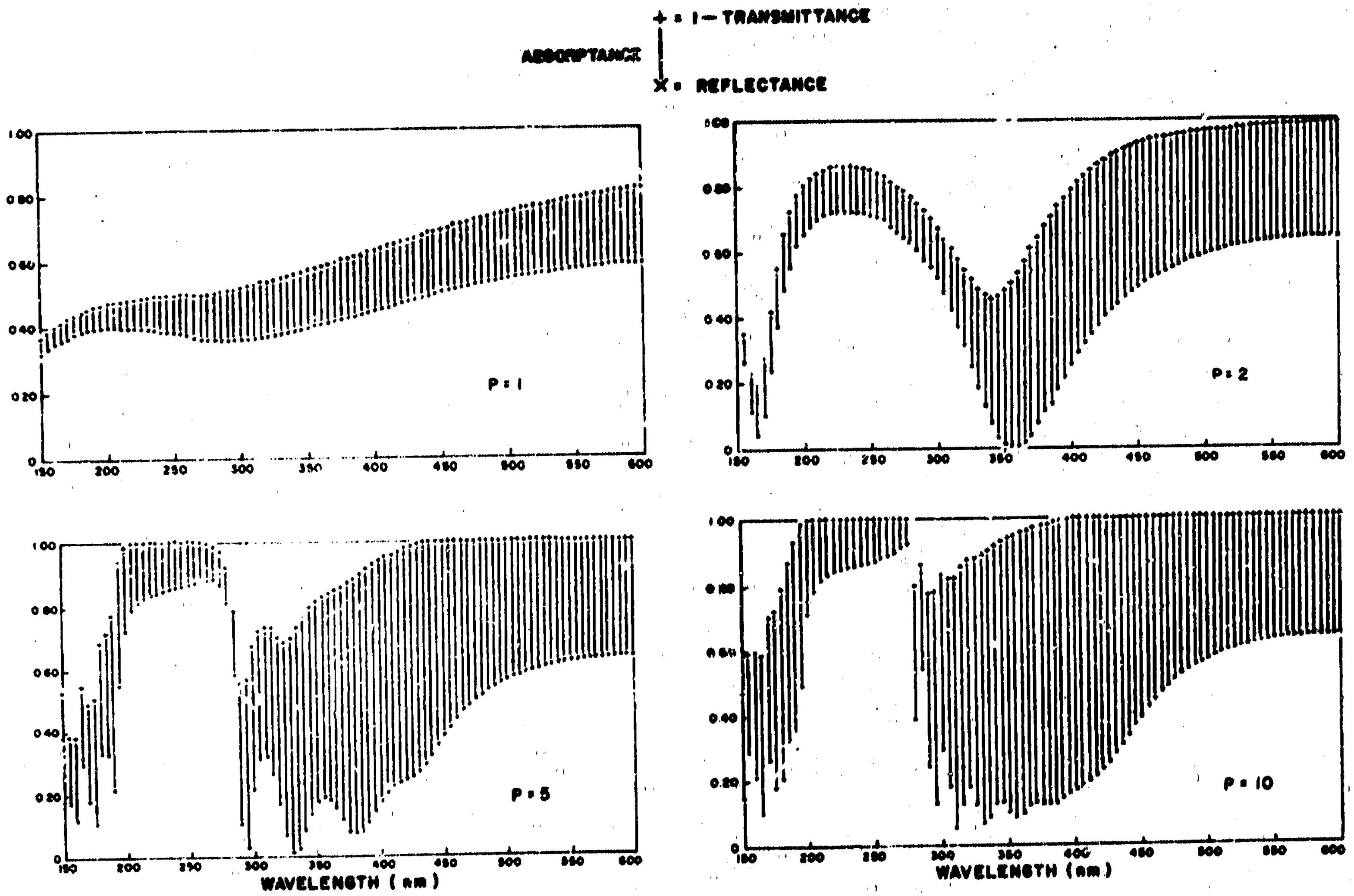


Fig. 5. DMD stacks with varied periods "p."

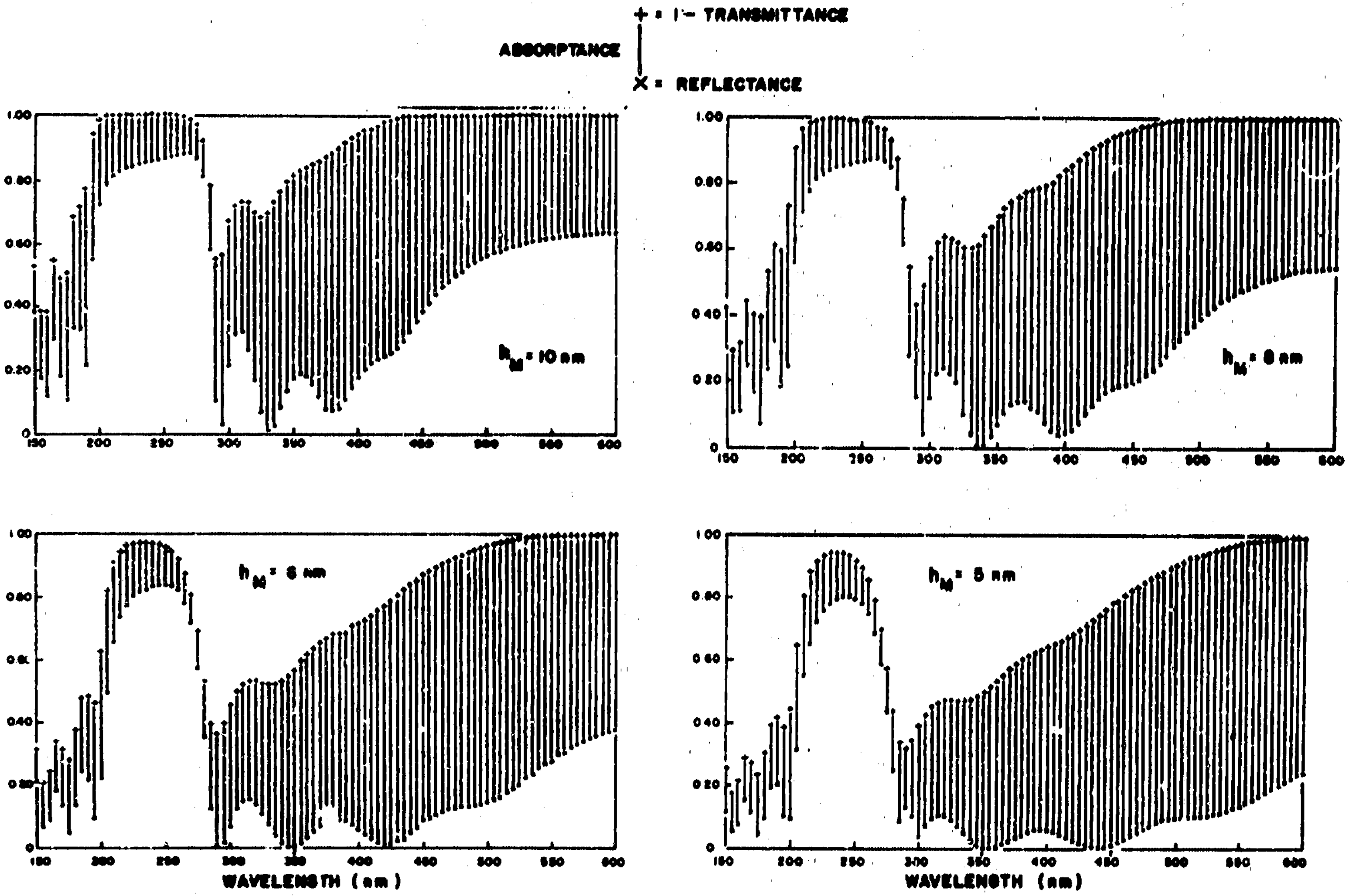


Fig. 6. DMD stacks with varied metal layer thicknesses, h_M.

for 5-period stacks. Two changes in the form of reflectance can be noted. Both the width of the high reflection band and the background reflection at longer wavelengths decrease with decreasing metal layer thickness. A good solar-blind mirror could be made with a metal thickness of ∿4.3 nm and 8 or more periods, according to our computations. The changing properties for thin metal layers may prohibit optimum filters of this type from being fully realized experimentally, however.

Some observations regarding the form of the spectral reflectance curves in Figs. 5 and 6 can be made which lend some insight into the properties of metal-dielectric interference filters without invoking detailed mathematics. First, we note that when dielectric films are $\lambda/2$ optically thick, they act just as though they were not there. When two or more DMD periods are joined, the films separating the metal layers will be $\lambda/2$ thick at 250 nm in the above examples. The reflectance and transmittance at this wavelength is thus just that of a single-period DMD stack with the appropriate metal thickness. In this case, we can imagine that the strength of the electromagnetic field, viewed as a standing wave, arrives at the next metal surface just as it left the preceding one, except with opposite sign. When the nodes in the electric field are at the metal surfaces, these surfaces will offer a minimum resistance, and the wave will pass on, giving a transmission maximum. With metal films, the phase change at metal-dielectric interfaces is important since we add amplitude with phases. The phase change on reflection for the interface between two dielectric media of different refractive indices is always 180°. The phase change on reflection from the boundary between a metal and a dielectric is always less than 180°. The phase change is dependent on the two refractive indices. This phase change causes the dielectric layers which separate two thin metal layers to act as if they were thicker than they actually are. Thus, for a given dielectric thickness, the standing wave pattern will be shifted to longer wavelengths. This can be shown in Fig. 5b, where the first transmission maximum is associated with a standing wave pattern which is of the lowest order ($\lambda/2$ optically thick). The peak is shifted from the normal half wave position at 250 nm to approximately 340 nm.

The positions of the antinodes are associated with the effective quarter wave optical thicknesses (odd multiples) of the dielectric layers. With an antinode condition, one generally expects to have a high reflectance and low transmittance. For the combination of materials shown in Fig. 5, these positions are at about 600 nm and 215 nm. For the relatively thick metal layers, there is a high reflectance. Where the thicknesses of the layers are actually $\lambda/2$ thick (250 nm) the reflectance is also high because films which are $\lambda/2$ thick act as if they were absent, and hence the reflectance is the same as for a thick metal film. The region between the actual $\lambda/2$ thickness and the effective quarter wave thickness determines the width of the high reflection band. The reflectance at the one-quarter and three-quarter wave positions is slightly different because of the difference in the optical constants and ratio of thickness to wavelength at these two wavelengths. By decreasing the thickness of the metal layers, we decrease the reflectance and increase the absorptance for each layer, giving the lower long wavelength reflectance and a narrower high reflectance band shown in Fig. 6.

From Fig. 5, it can be seen that one cannot further decrease long wavelength reflectance by adding more layers. The transmittance is very low at long wavelengths, so more layers of the same type will have little or no effect. As a general rule one should think of adding layers on the side of *emergence* where they can have an effect only when there is a transmission "window" to provide light with which to work.

A parallel between the high reflection band of the DMD stack and that of an all-dielectric stack can be drawn. Two DMD stacks separated by a spacer layer form the equivalent of an all-dielectric Fabry-Perot filter. The computed properties of such a filter are shown in Fig. 7 where, again, only aluminum and magnesium fluoride layers have been used. Although the transmission "leaks" look objectionable, such a filter used in conjunction with reflection filters of the type shown in Figs. 5 and 6 would be useful in isolating narrower spectral regions than would be possible with a reflection filter alone.

In conclusion, the DMD stack shows promise as a basis for ultraviolet filtering where a wide range of materials is unavailable. It is, theoretically, possible to control the reflectance in the ultraviolet as well as at longer wavelengths, making solar-blind mirrors possible. The greatest problem in realizing such filters experimentally is obtaining very thin metal films with good optical properties.

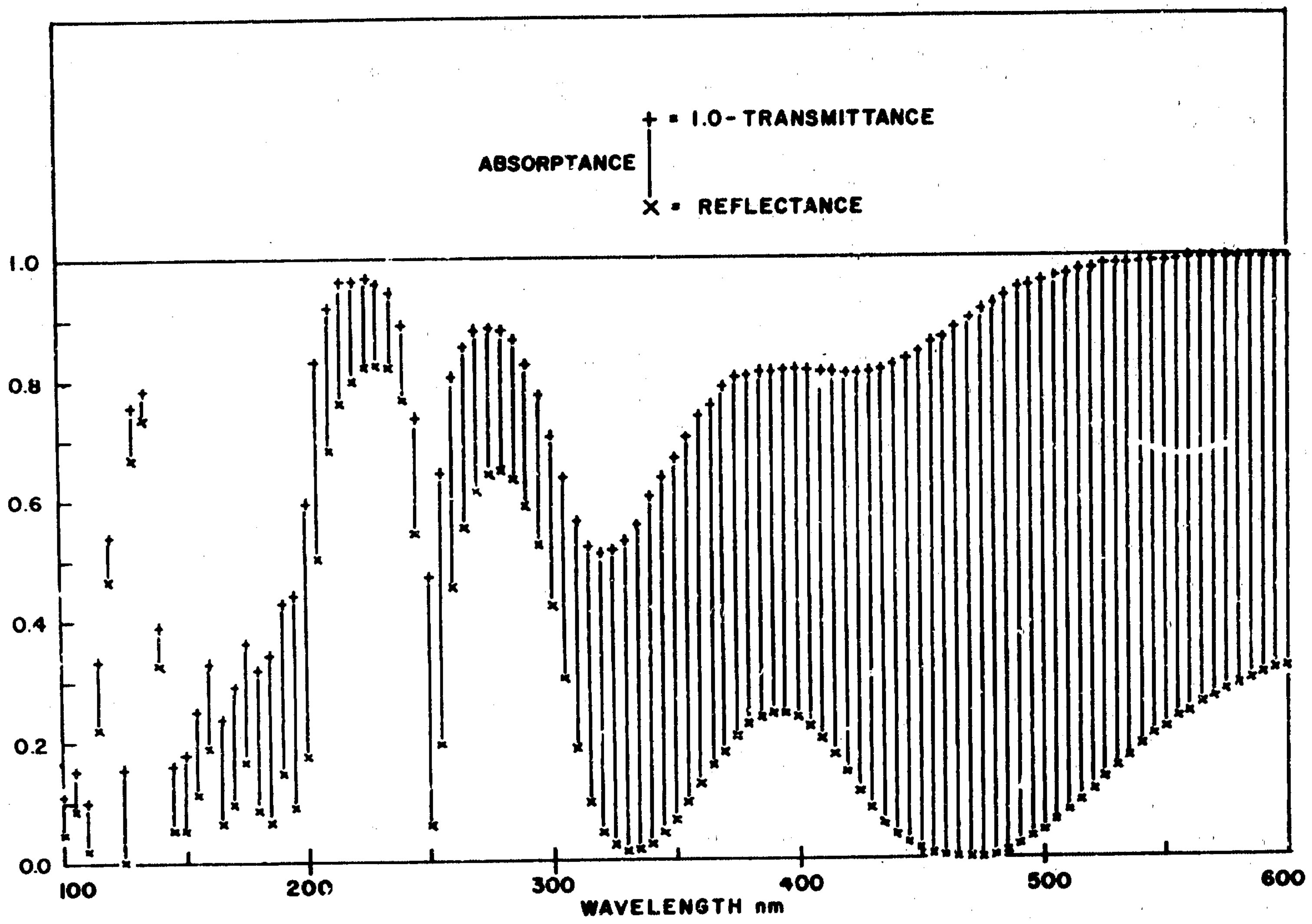


Fig. 7. Computed properties of a $(DMD)^3$ spacer $(DMD)^3$ Fabry-Perot filter.

THE VACUUM SYSTEM

General considerations

Most thin film interference filters have been made by vacuum evaporation. Chemical coating and vacuum sputtering can also be considered. In general, however, it is felt that evaporation is preferable because of higher film purity, more precise control of the thicknesses of the films, and better reproduceability. Simply stated, the evaporant (a solid) is heated in vacuum to form a vapor. The vapor strikes a cool surface (the substrate), and, under the proper conditions, most of the vapor sticks and forms a thin, uniform solid layer--a thin film. The essential parts of a vacuum *coater* are a means of producing a vacuum, a way of heating the evaporant, and, if the thickness of the film is important, a means of monitoring the thickness during deposition.

One of the early reasons for evaporating in vacuum was to allow the vapor to reach the substrate without hitting too many air molecules along the way. For some types of coatings, a vacuum is undesirable (for example, tin oxide coatings are prepared by evaporating stannous chloride in air.) Aluminum, however, is an active substance, especially in the molten or vapor state. To obtain "pure" aluminum coatings, the evaporation must be performed under pure conditions. Considerable work has been done to determine the best way of preparing aluminum films.[10] The current thinking is that the films should be evaporated in vacuum where the pressure is less than 10^{-5} torr and at high rates onto a cool (room temperature) substrate. The rate of evaporation is supposed to be greater than 60 nm/sec.

At such high rates, thickness control becomes a serious problem. There are basically three ways of controlling the thickness. First, one can preweigh the evaporant and evaporate all that there is. The accuracy using this technique is usually poor, since, in a violent situation, there is likely to be splashing or inconsistant distribution at the source. Also, the rate of evaporation depends on the amount of material. The second possibility is to evaporate at a predetermined rate for a certain period of time. This is inaccurate also since rate is difficult to control and the required mechanical shutters tend to be inaccurate. The third means is to monitor the thickness during evaporation. This, too, must rely on the accuracy of a mechanical shutter in addition (usually) to the uncertainty of human reaction time.

To get a feeling for the accuracies needed, consider that at the "minimum' rate of evaporation of 5-nm-thick film takes only 0.08 sec to evaporate, which for 10% accuracy would mean that the shutter would have to be better than ±8 ms. Considering that the shutter must work in vacuum, this seems nearly impossible to achieve.

We have reviewed the evaporation process to see if there might be some way to achieve the same results with a lower evaporation rate. Let us suppose that the aluminum vapor consists primarily of atoms and small molecules of aluminum which are mixed with some of the residual gas in proportion to the rate of evaporation. If the aluminum combines chemically with the residual gas, then the impure aluminum arriving at the substrate will be inversely proportional to the rate of evaporation. These contaminants will affect the entire film, as opposed to a surface contamination which occurs after the evaporation has stopped. On the basis of this argument, one should get the same results if the ratio of rate of evaporation to pressure during evaporation is constant, all other things being constant. This argument is valid over at least a few orders of magnitude and is the reason for going to an ultrahigh vacuum system for precise control of thin metal films.

The composition of residual gas in the vacuum system during and after evaporation will also have a large effect. One would not be concerned if residual gas were predominantly hydrogen or a rare inert gas, for example. Oxygen must be avoided as much as possible.

The ultrahigh vacuum system

For most of our evaporations, we have used an ultrahigh vacuum coater. Most of the system was built by Varian Associates and purchased under NASA contract NAS 8-20651. The system is completely free from oil pumps. The chamber is a cylinder 60 cm in diameter by 150 cm high. Rough pumping is accomplished with a gas aspirator pump in combination with cryogenic sorption pumps. The combination will pump the system to approximately 10^{-5} torr in 2 hours. The remainder of the pumping is accomplished by a 500 ℓ/sec sputter ion pump and cryogenic titanium sublimation pump rated at 10,000 ℓ/sec. Fig. 3 is a schematic diagram of the system. When the system is clean, it can be pumped to better than 5×10^{-11} torr in 24 hours. After two years of use without cleaning, the system can be pumped to 2×10^{-10} torr. The low pressures can be realized only after a prolonged bake-out at about 250°C.

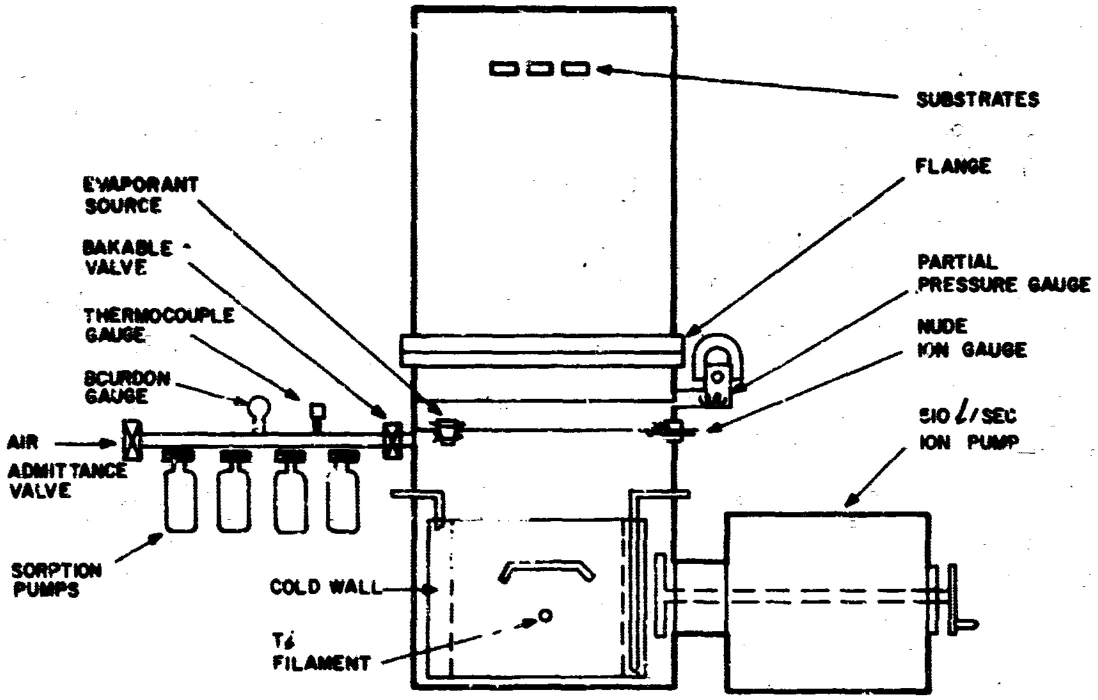


Fig. 8. The ultrahigh vacuum system.

After the bake-out, the only significant gases that can be detected with the partial pressure gauge are nitrogen and hydrogen. This seems to be a suitable environment for evaporation of aluminum films under controlled conditions. The evaporant can be heated either by directly heating a filament or metal boat by passing a high current through it, or by electron bombardment. The latter method is preferred for all materials since alloying of tungsten from the filament with aluminum could be a source of impurities, although there is no evidence this has occurred. The electron beam evaporation source focuses a stream of electrons, by crossed electric and magnetic fields, onto the evaporant which is held in a water-cooled crucible. Because only the area which is being evaporated is heated, the evaporant in effect acts as its own crucible. We have found that evaporation takes place at lower beam currents, hence lower pressures, if the aluminum is insulated from the water-cooled crucible by another crucible made from HDA (a boron nitride-titanium diboride substance produced by Union Carbide Company).

The intense heat that is required to evaporate most materials generates a large amount of gas both from the evaporant and from the walls of the system. There are very few pumps which are capable of handling this gas load. We have estimated that our pumping speed would need to be increased to 10^7 ℓ/sec to cope with our typical gas loads. The high temperature bake-out considerably reduces the problem. It is also highly desirable to outgas the evaporants with heat just below the vapor point. Aluminum is relatively easy to outgas. It can be held in its molten state and is usually outgassed sufficiently after 3 to 5 hours. The dielectric materials are a greater problem, however. Their thermal conductivity is generally lower than for metals, and because of the fixed electron beam size and position in our configuration, it is difficult to get rid of gas without evaporating the material. Degassing combined with the small available crucibles renders the system incapable of producing very many layers per change. The very low heat allowed and low thermal conductivity require an outgassing time of from 10 to 20 hours with constant attention. To achieve the lowest pressure evaporations therefore requires nearly 10 days of preparation for all five crucibles in vacuum. This has two effects. There is a relatively low yield of data and results, and one is afraid to make experimental changes for fear of losing half of the month's experimental yield.

We have been able to evaporate in the system below 10^{-9} torr, but only at very low rates. The ratio of rate to pressure is about a factor of 5 below that which is most desirable. However, even at the slowest rates (approximately 0.02 nm/sec), the resulting films are of a quality that is comparable to, if not superior to, films prepared in the conventional manner. An unexpected bonus in using the ultrahigh vacuum system is an unusually hard adherence of films to the substrate. Generally speaking, the films cannot be removed except by polishing the surface clean with abrasives. The films are apparently more compact and perhaps have a higher density.

The operation of the coater is fairly straightforward, with one exception: We take great pains to avoid the glow discharge which is commonly associated with high pressure operation of the ion pumps. This can be avoided by roughing with the cryogenic sorption pumps to a lower than normal pressure. We try not to start the ion pump until the pressure has reached $\sim 7 \times 10^{-5}$ torr. We feel that this prevents contamination of the substrate by impurity ions in the glow discharge. A good cleaning procedure before installation of the substrates, followed by a high temperature bake-out in vacuum, appears to leave the substrate surfaces clean.

THE OPTICAL FILM THICKNESS MONITOR

The major problem with any thin film deposition process is the means by which the thickness of the film is monitored during deposition so that the final multilayer stack will have the desired properties. There are many ways of monitoring film thickness.[11] It is important that the quantities of interest be directly measured. We speak, for convenience, of a film thickness in nanometers, but we mean that it must have a certain reflectance, transmittance and phase change on reflection. We have elected to measure reflectance as the basic film property to be controlled.

The optical system

Generally speaking, the optical system is used only for transmission of energy, and the quality of images need not be considered. Light used to measure film reflectance must come only from the surface of interest, and must be free of contaminant light. The system must further be stable enough to prevent vibrations from confusing the measurements.

The use of ultrahigh vacuum imposes some restrictions on the monitoring system. We have found that first-surface mirrors do not last in the system regardless of overcoating. Damage seems to occur during high temperature bakeout. The reasons for this are not presently known, but they should be investigated, as this may be important for certain types of space flights where mirrors may be subject to heating from solar radiation. Another limitation with ultrahigh vacuum is the difficulty of providing for mechanical motion inside the vacuum. Mechanical motion feedthroughs exist but are expensive, and one is limited in the number of available feedthrough ports.

Fig. 9 is a schematic diagram of the system we have used. The light source S is imaged on the pinhole D_1. This light is then chopped mechanically at 90 Hz, and the pinhole is imaged on the monitor plate G by the lens L_2 after being bent by the prism P_1. It is essential that the lignt be chopped as close to the source as possible so that it can easily be distinguished from the bright background light in the chamber which arises from ionization gauge filaments and the evaporation sources. The light reflected from the monitor plate is returned out through a feedthrough and imaged on the entrance *slit* of of a monochromator. An end-on photomultiplier senses the light at the exit slit. It is necessary to have the monochromator or filter at the photodetector to protect it from the bright light inside the chamber during evaporation.

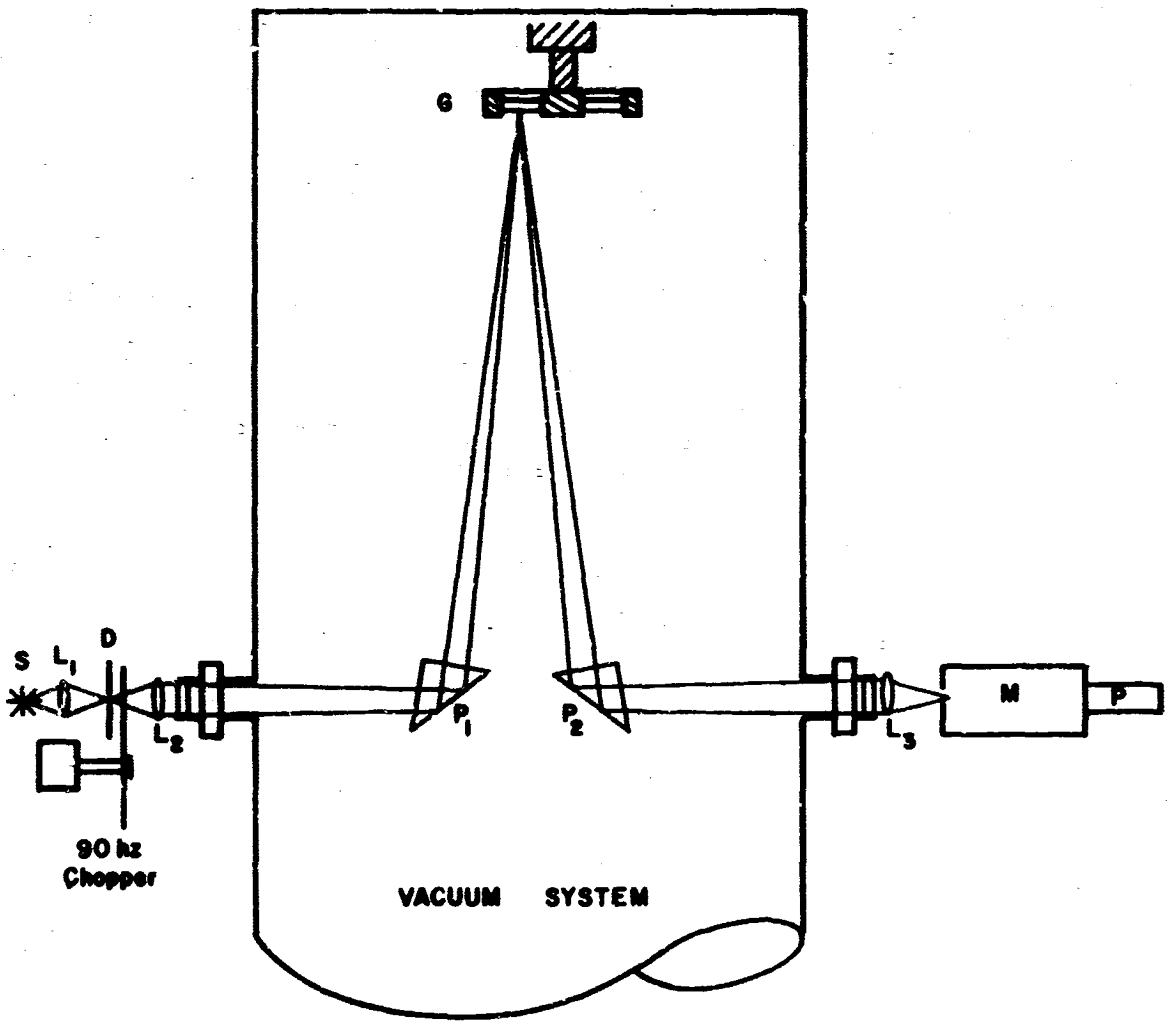


Fig. 9. Schematic diagram of the optical components of the monitor system.

The monitor system electronics

The ability to monitor and control the film thickness during evaporation is limited in accuracy by a combination of the inherent quality of the photometer electronics and the skill of the operator. It is desirable to let the human element be the limiting factor.

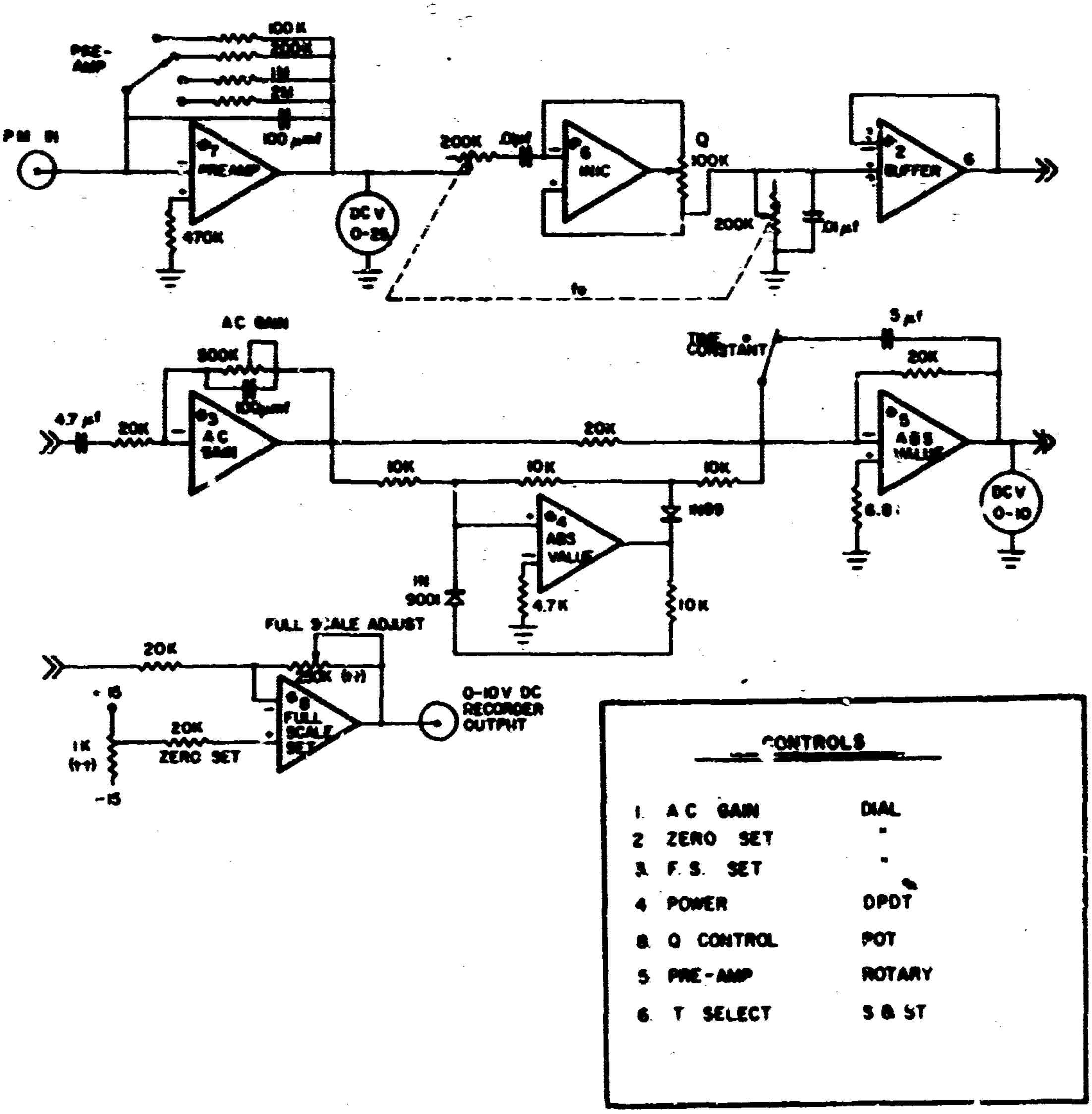
Change in reflectance with film thickness may take two forms. For dielectric layers, the reflectance changes approximately as a sine wave. For metals, the reflectance generally continously increases in a nonlinear manner. In some instances, in multilayer work, the reflectance of a stack will decrease to a minimum before increasing. When working with all-dielectric stacks that involve only quarter-wave layers, one evaporates to maxima or minima in the reflectance. As discussed above, with metals, the maxima and minima occur at the "effective" quarter-wave positions. Thus, for metal-dielectric multilayers, one must evaporate at one time or another to specific reflectance values rather than maxima or minima. For this reason, the monitor electronics must be linear and accurate over a wide dynamic range.

A typical requirement for a DMD evaporation might be to evaporate from a reflectance of 0.082 to a minimum of 0.053 and then evaporate from 0.053 to 0.620. One must be able to amplify the small reflectance changes so that they can be seen easily.

The electronic circuit, developed with support from grant NSG-732, is shown in Fig. 10. The circuit is built upon an assemblage of seven operational amplifiers. The signal from the photomultiplier is a negative current: A small dc current in phase with the 90 Hz signal combined with the noise background. The current signal is converted to a voltage which is the product of the input current and the resistor chosen at the preamplifier (#7). We select the 90 Hz signal by the active filter (#6) for which the central frequency and quiescence are initially adjusted. The signal is inverted by the buffer circuit (#2) and amplified by the ac amplifier (#3). The amount of amplification is set by adjustment of the 500 kΩ 10-turn potentiometer. Absolute value circuits (#4 and #5) convert the ac signal to a dc voltage which is proportional to the peak-to-peak difference in the ac signal. The dc signal passes through an amplifier (#8) and is added to a positive or negative zero bias signal for presentation on a strip chart recorder. By proper adjustment of the full scale, zero level, and gain potentiometers, a wide range of reflectance levels and reflectance changes can be recorded on a

10-inch chart. The preamplifier resistance is selected to provide a small enough signal so that later stages of amplification will not saturate.

The system has been shown to be linear to ±1% over a range of better than 1000:1 and has the versatility required for our monitoring work. Monitor noise is generally a serious problem, but for this system less than 0.1% noise is observed.



CONTROLS

1	A C GAIN	DIAL
2	ZERO SET	"
3	F. S. SET	"
4	POWER	DPDT
8	Q CONTROL	POT
5	PRE-AMP	ROTARY
6	T SELECT	S & ST

Fig. 10. Amplifier electronics for optical film thickness monitor.

EXPERIMENTAL RESULTS

Three basic questions must be answered from observations of the experimental results. First, there is the possibility that optical properties (optical constants in particular) of films which are evaporated in ultrahigh vacuum are different from those of films prepared in more conventional vacuum systems. Second, we know that optical constants of very thin metal films are not the same as those measured for thick films or for bulk materials: what is the thinnest film that can be used which will have the same optical constants as those used for calculations? Finally, the filter calculations discussed above should be shown to agree with actual results, and filter production must be feasible for use in actual applications.

Properties of single films

Several films of several different materials have been prepared in the ultrahigh vacuum chamber under a variety of conditions. The two main parameters which we varied were the evaporation rate and the pressure. Because of pumping limitations of our chamber, we were not able to vary these parameters independently. The highest rates of evaporation were achieved only at higher pressures.

The properties of the aluminum films are of the greatest concern. We have needed to reduce the rate of evaporation to a point where the film thicknesses can be accurately monitored and controlled. The deposition rates we investigated ranged from about 0.01 nm/sec to 10 nm/sec. The rates are inferred from the measurements of film thicknesses and the total evaporation time. It is possible that the rates were nonuniform; however, by keeping the power on the electron beam source constant, the rate should be nominally constant. Pressures during evaporation ranged from 10^{-9} torr to 10^{-5} torr.

The observations we made were as follows: general observations of the film and its durability, reflectance and transmittance, and visual inspection of the films for scattering or roughness. The scattering observations and durability are only subjective comparisons.

Generally speaking, all aluminum films evaporated in the ultrahigh vacuum chamber were durable and free from excessive scattering. The films were not readily removed by washing in a sodium hydroxide solution, even after being exposed to air for several months. This indicates that the films are of relatively high density and that any spontaneous formation of aluminum oxide is limited to a thin surface layer.

The reflectance of an aluminum film evaporated at the start of the program was measured in the vacuum ultraviolet. Measuring difficulties at that time caused minute-to-minute changes in measured reflectivity at Lyman α (121.6 nm), so final data were not recorded. The sample had been exposed to air for about 2 hours and had been in the vacuum system for 3 days after evaporation. The initial reflectance which we observed, however, may have been as high as 0.80. These observations should be confirmed. A slow time decay of ultraviolet reflectance could be useful in some situations. After the reflectance had stabilized, the measured reflectance is as shown in Fig. 11 in the vacuum ultraviolet. At wavelengths longer than 200 nm, the reflectance is the same as films prepared under older "ideal" conditions (oil-pumped system at 10^{-5} torr at high rates). Our measurements of reflectance are limited to an accuracy of about ±2%, so detailed comparisons are not possible. We do observe that the reflectance of the ultrahigh vacuum aluminum films is slightly higher than those we prepare under normal conditions.

The best material for the dielectric layers in ultraviolet interference filters is magnesium fluoride. Although, in crystal form, lithium fluoride is transparent at shorter wavelengths, it was not considered here because it is slightly water soluble and less durable. The transmittance of magnesium fluoride films prepared in ultrahigh vacuum has been measured and can be considered to be free from absorption for all wavelengths from the infrared to 120 nm. The optical properties which we observed were essentially the same as those observed for films prepared under other conditions. Since we wish to form filters consisting of alternating layers of aluminum and magnesium fluoride, we avoid heating the substrates in order to obtain the best optical properties of the aluminum. All of our evaporations were on substrates at room temperature ($\sim$ 20°C). These films were durable and relatively free from scattering. Although it is generally recommended that the best magnesium fluoride films are prepared by evaporation onto warm ($\sim$ 300°C) substrates, we have not found that this is necessary to obtain durable films.

One problem encountered was that properties of combined films often indicated that they were more absorbing than they should have been according to single film observations and the theory. This is ascribed to impurities.

We have experienced difficulties in obtaining pure films when two materials are evaporated during the same vacuum cycle. Both aluminum and

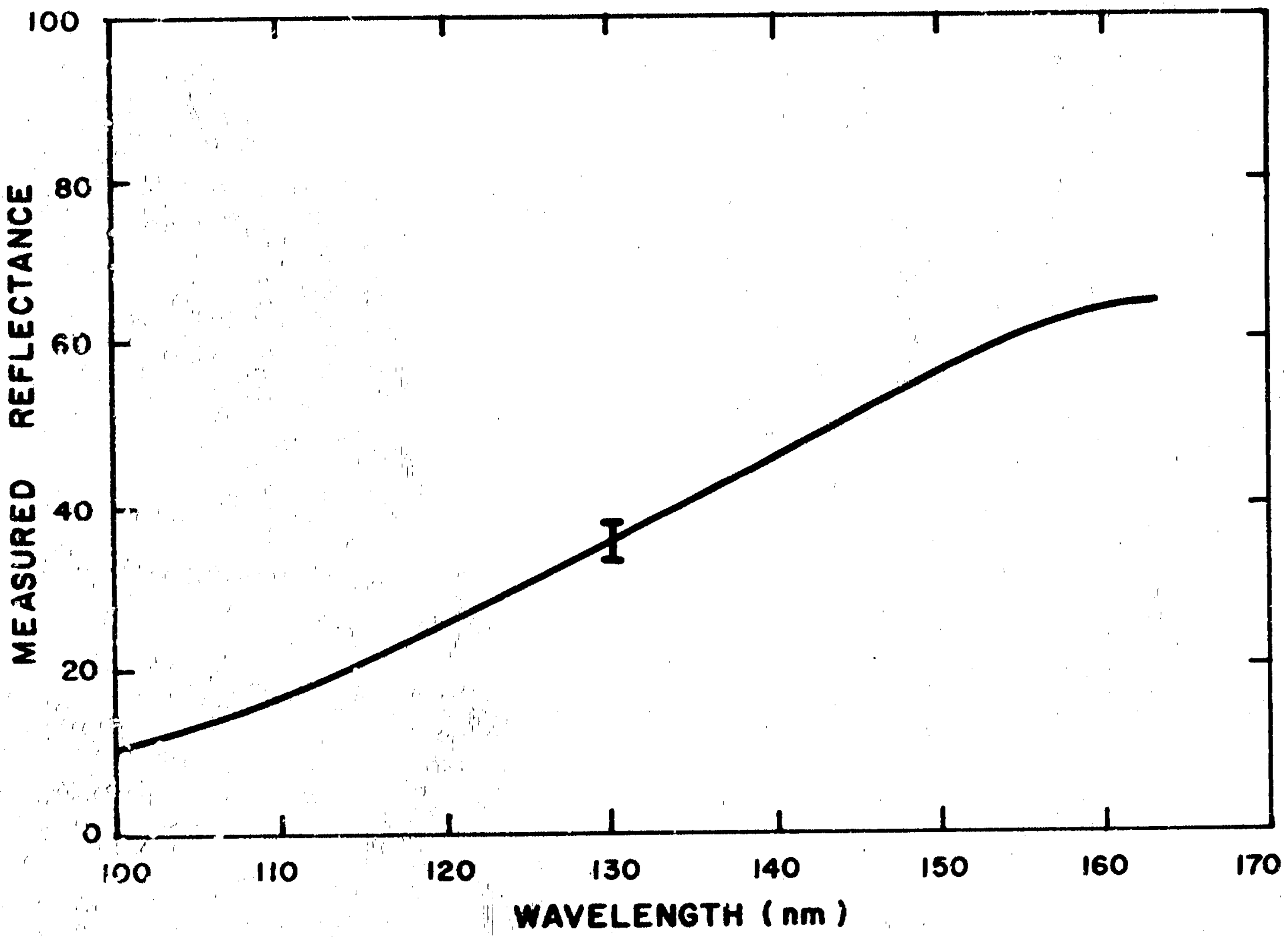


Fig. 11. Reflectance of an aluminum film measured in the vacuum ultraviolet.

magnesium fluoride films appear to be more absorbing than when they are evaporated during separate cycles. The apparent difference is due to a mixing of the materials on the hearth of the electron beam gun. We normally assume that materials in vapor form will travel in straight lines, sticking to the first surface that they hit. It is possible that some of the molecules are reflected from surfaces and appear not to travel in straight lines. This is thought to be only a minor source of contamination, however. A more important problem appears to arise from charged molecules which are deflected by the electric and magnetic fields near the vapor source. The effect is most evident for aluminum, where, after some time, we can observe a heavy deposit of aluminum near the bottom of the chamber where it is "impossible" for the atoms to condense. Apparently, some molecules migrate from one crucible to another during evaporation. This effect is lessened by greatly enlarging the shields between the crucibles. We have been able to control this effect to a large extent in this manner.

Another problem is that we have difficulty getting enough material into the evaporation region of the electron gun to be able to deposit as many layers as desired. This is due to the nature of the electron gun (Varian Associates Model 980-0005), which has five crucibles that can be moved in front of the filament, and uses a fixed accelerating potential and magnetic field. With the fixed field configuration, it is impossible to change the focus of the beam or the position with respect to the crucible. Shifting the crucible distorts the fields in such a manner that the electrons still hit the center of the crucible. Evaporation occurs only over a small (about 5 to 10 mm wide) area, leaving a large amount of material around the edge which cannot be evaporated. We have tried to evaporate from conducting crucibles in the manner of resistance heated sources, but the boats have evaporated before enough material has evaporated. There is a noticeable amount of contamination from the boat in these instances. We have also tried to premelt the evaporant in a vacuum furnace so that we would be evaporating from a solid piece. When this is done, the solid usually falls apart from thermal stress, leaving less material than when a powdered form is used.

The obvious solution is to use an electron gun with a beam control. This requires an electromagnet to produce the field. None of the manufacturers of this type of gun recommend their product for ultrahigh vacuum systems such as ours. Further experimentation with electrostatic deflection will have to be done before this problem can be solved. We are therefore presently limited to investigating designs that contain only a few dielectric layers.

Contamination of dielectric material is easily observed during deposition when the films are deposited directly on a glass monitor slide (without metal films). For a homogeneous nonabsorbing film, the reflectance varies nearly sinusoidally with thickness. When the film is slightly absorbing, the maxima and minima do not have constant values.

We have concluded from our observations of single films that evaporation in a clean ultrahigh vacuum system gives films which have nearly the same optical properties as those evaporated in a more conventional oil-pumped system. The optical constants are apparently close enough to published values that they may be used for computations. Good optical properties of aluminum may be obtained with very low evaporation rates and low pressures during evaporation. The physical properties of these films are at least satisfactory and quite possibly are superior to those prepared under more normal conditions.

Properties of very thin metal films

The filter designs discussed in other sections of this report require semitransparent films for most of the metal layers. For the MRF's, the film thickness should be about 20 nm. This thickness has been used for many MDM type filters with moderate success. The optical constants of films thinner than 20 nm may change with thickness, however.[12] The DMD filters require thicknesses between 4 and 5 nm to realize optimum theoretical characteristics. Whether the optical constants of aluminum are a function of the thickness for such thin films had not been determined for ultrahigh vacuum evaporations. The optical constants given in the table on page 5 are reported to be valid for films as thin as 10 nm when evaporated under normal conditions.[13] It had not been determined whether the limit was a function of the evaporation conditions, however.

There are several problems associated with working with very thin aluminum films. Probably the greatest problem is that the pure metal is very reactive. An oxide layer forms very rapidly on the surface, and this affects the optical properties of the "films." When the film is very thin, it is difficult to determine whether there is any pure material left. The thickness of the film may be affected because of the increase in the total number of atoms. Changes in density also cause uncertainty in the nature of the film.

We have investigated the properties of DMD single periods for different metal thicknesses. In this way, we can study the properties of metal films in the environment in which they will eventually be used. In principle, the optical constants of a metal film can be determined from a knowledge of three quantities. One of the ordinarily necessary parameters is the thickness of the film. Film thicknesses of 4 to 5 nm are, at best, difficult to measure with any precision. We have found that such measurements do not need to be made to determine a "limit" for the thickness of the film that will have the "thick" film optical constants. We are unable, however, to determine the optical constants as a function of film thickness; only deviation from assumed values can be determined. The method used is to evaporate a number of separate DMD periods with different metal thicknesses. The outside dielectric layer protects the aluminum from oxidation. The "thickness" of the metal layer is chosen by evaporating to a certain value of the reflectance. Computations of the reflectance for several metal thicknesses are used as a guide. The reflectance and transmittance of the DMD period are then measured and compared to all possible values computed from the thick-film optical constants (see table on page 5). Fig. 12 shows a plot of the reflectance vs transmittance of DMD periods where the quarter-wave optical thicknesses of the D layers are 400 nm and the measurements are at 400 nm. Several thicknesses are labeled along the theoretical curve, which represents all possible reflectance and transmittance values. The measured points show the variation. From the data, we can infer that thicknesses greater than 9 nm can be considered to have the optical constants given in the table. It was possible to predict the "thickness" of the film for these values before measurement. There was no indication, during the evaporation, that oxide layers were responsible for the deviations of measured and computed values. Under less-than-ideal conditions, it is possible to observe the change in reflectance with time of the unprotected aluminum layers when the films are very thin. Such changes were not observed with these films for periods up to 10 minutes in ultrahigh vacuum.

These evaporations were done in the UHV chamber using the techniques described above with the pressure and evaporation rates among the higher values. The results are relatively insensitive to small (about one order of magnitude) changes in the pressure. Typical values for the aluminum films were a total pressure of 3×10^{-6} torr and an evaporation rate of about 10 nm/min.

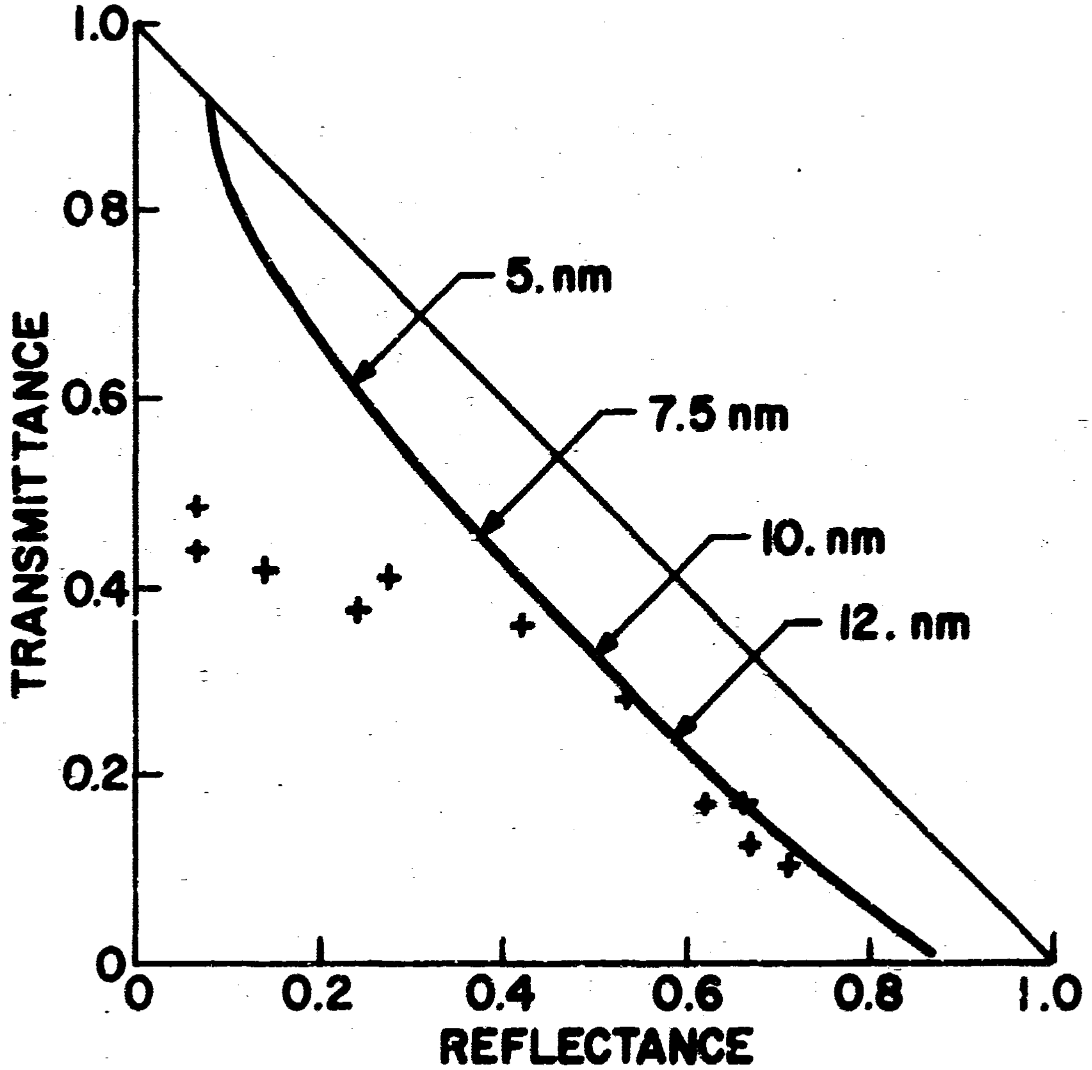


Fig. 12. Reflectance vs transmittance of dielectric-metal-dielectric filter stacks whose D layers are 400 nm thick.

Properties of mirror reflection filters

The three-layer mirror reflection filter will be the easiest type of filter to make. First, the number of layers is minimal, and second, the reflectance is the only quantity which needs to be compared with the theoretical value. As discussed earlier, however, there have been significant differences between theoretical and measured results.

One of the most difficult problems in making an MRF is the monitoring of the film thicknesses. As discussed earlier, the minimum reflectance and the wavelength of the minimum depend on many factors, and slight errors in the dielectric thickness or unexpected changes in the optical constants of the semitransparent metal layer can cause large errors in the thickness of the final layer.

We have had good success monitoring each layer on a separate piece of glass as it is deposited. The main disadvantage in this method is that one cannot compensate for small errors as the filter is formed.

An example of an MRF with a semitransparent aluminum layer that corresponds closely to the theoretical prediction (Fig. 3) is shown in Fig. 13. The evaporation conditions used to obtain this result are essentially the same as those described in the previous section. There is a 45-nm-thick protective layer of magnesium fluoride over the semitransparent metal layer. This layer has only a small effect on the properties of the filter.

The role of impurities in dielectric films is demonstrated in Fig. 14, which is essentially the same filter as above except that there has been some contamination. The increase in absorption is dramatic, even though it was only slightly observable during evaporation. The reason for the enhanced effect is that the dielectric layer is inside a resonant cavity where the electric field should build up. The absorption has two effects. In addition to decreasing the filter efficiency, there is an error in the thickness of the dielectric layer of the final filter which is caused by a change in the thickness where the monitor reflectance is a minimum, due to the fact that the phase changes on reflection are no longer 180° as required. From the shape of the curve, we can conclude that the optical constants of the aluminum layer are not seriously affected. These results still differ from previous results, which gave a high peak reflectance, symmetrically shaped reflectance band, and low background (wide reflection minima). These characteristics were not observed in this project without a change in the materials used.

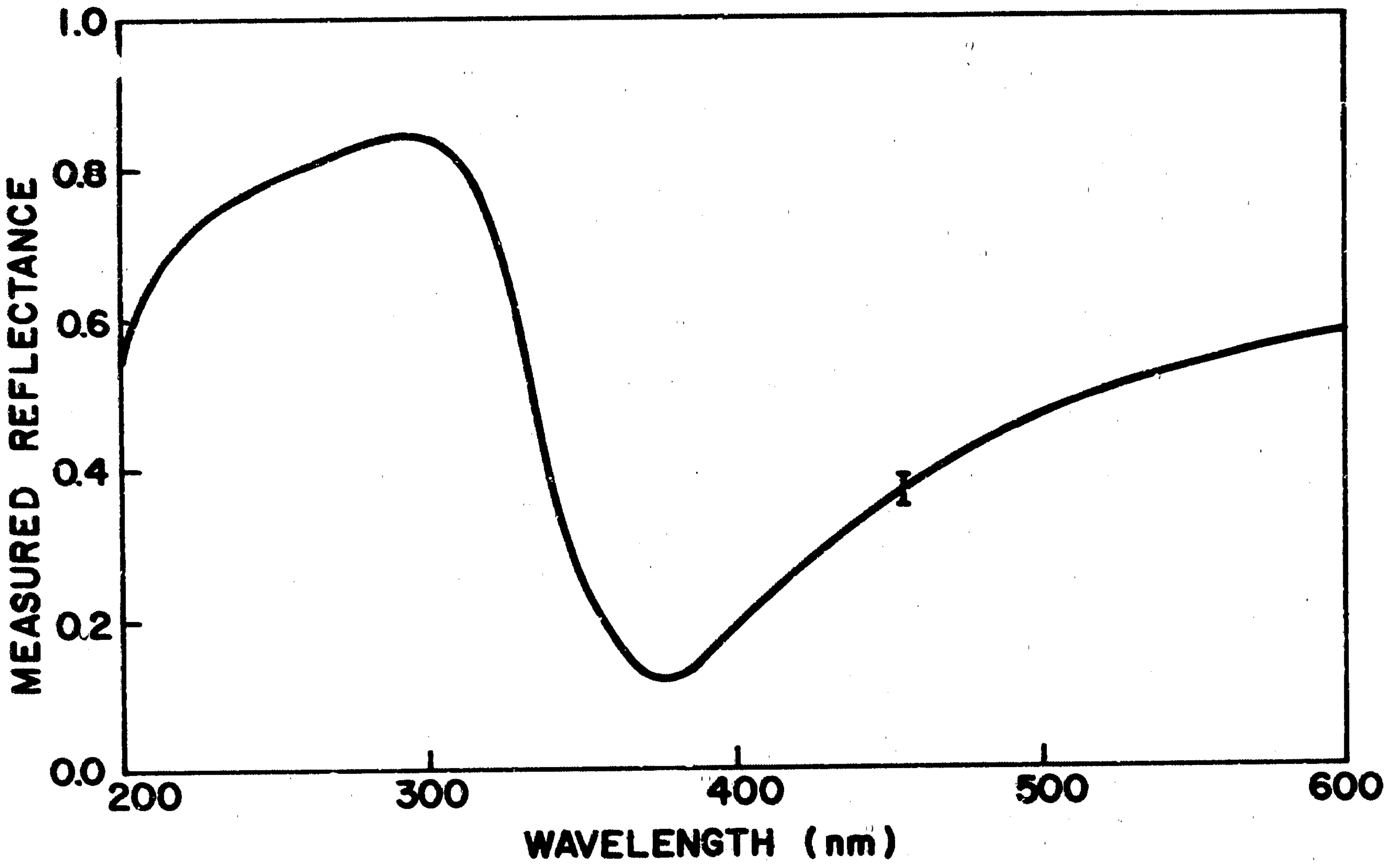


Fig. 13. Mirror reflection filter with a semitransparent aluminum layer.

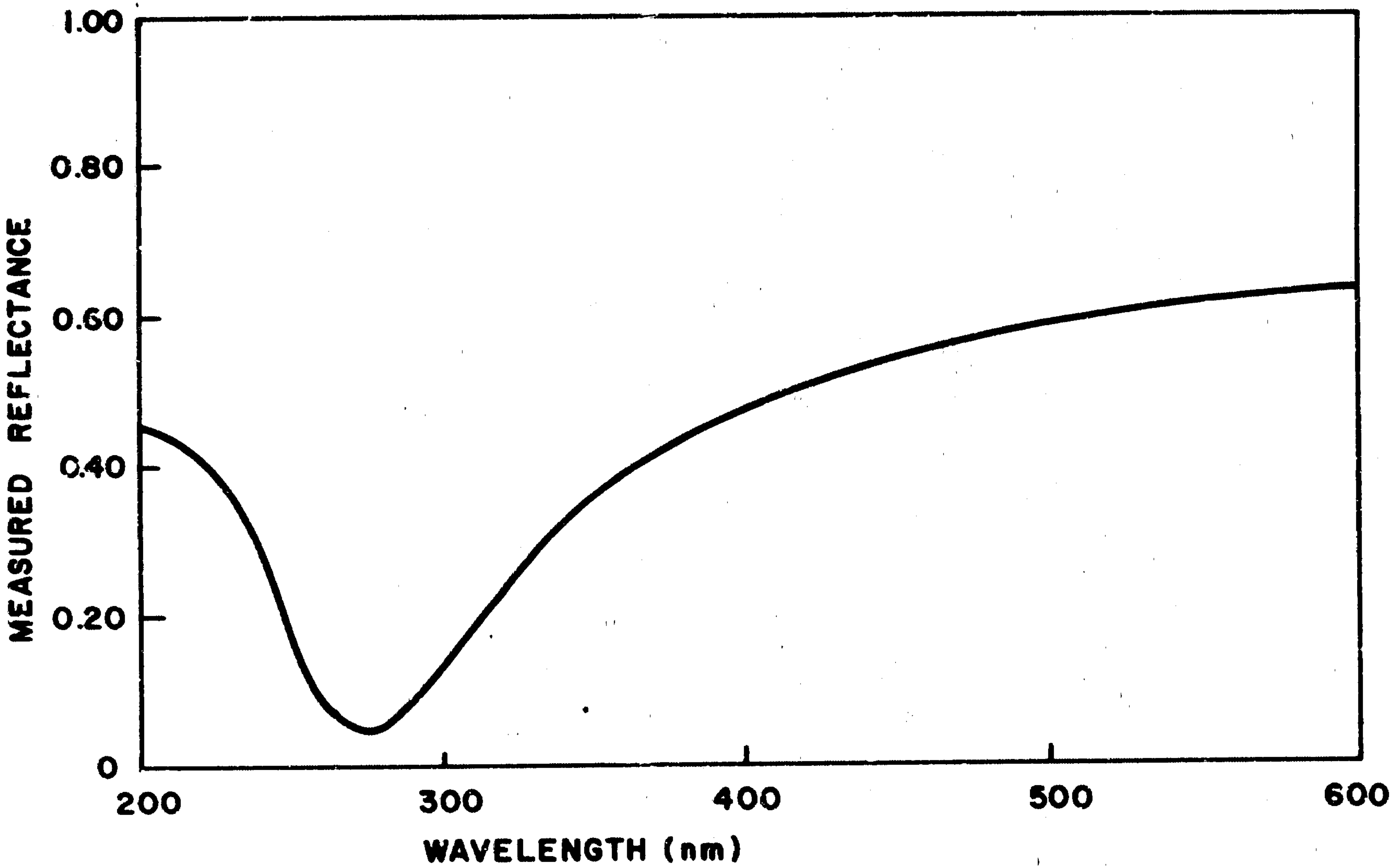


Fig. 14. Role of impurities in dielectric film.

Our earlier conclusion that solar-blind mirrors based upon only aluminum and magnesium fluoride cannot be effectively produced in our highly controlled conditions appears to be valid. It has been shown that changing the material for the semitransparent metal layer can produce a filter which would be a better solar-blind mirror. A filter with chromium, magnesium fluoride, and aluminum has been made; its reflectance is shown in Fig. 15. The result is close to that predicted although there is some evidence that the dielectric was slightly absorbing. Use of chromium would be recommended for solar-blind mirrors.

The observed decrease in the reflectance of unprotected aluminum immediately after evaporation can be explained by an increase in the real part of the complex refractive index. The aluminum thus becomes more like chromium. We can perhaps conclude that Turner's earlier experimental results were for a chromiumlike semitransparent film. That experimental results which agree with computed results can be achieved has been demonstrated by this research.

Properties of reflection filters with more than three layers

Design possiblities for ultraviolet filters with more layers based on the DMD period have been discussed. Application of these designs to solar-blind mirrors may not be optimal using aluminum and magnesium fluoride with present-day techniques and coating chamber facilities. The "minimum" allowed thickness for aluminum films is more than twice that required for the best designs. It has not been established whether the present limitations are due to techniques or basic physical properties. That the films 5 nm thick are different is not really surprising since they would be only about 10 unit cells thick (the size of the unit cell being 0.5 nm).

We were able, however, to make some of the thicker DMD stacks to compare with theoretical calculations. Such comparisons allow us to draw some definitive conclusions regarding the properties of the materials and the validity of our calculations.

Filters of DMD stacks with one to four periods have been made, with a 10 nm metal layer thickness. The properties of the single period stack were discussed above, and further measurements confirmed the predictions of Fig. 5(a). An example of a two-period stack is shown in Fig. 16. This result can be considered to be in agreeement with the computed result shown in Fig. 5(b). The absolute value of the differences between theory and observation

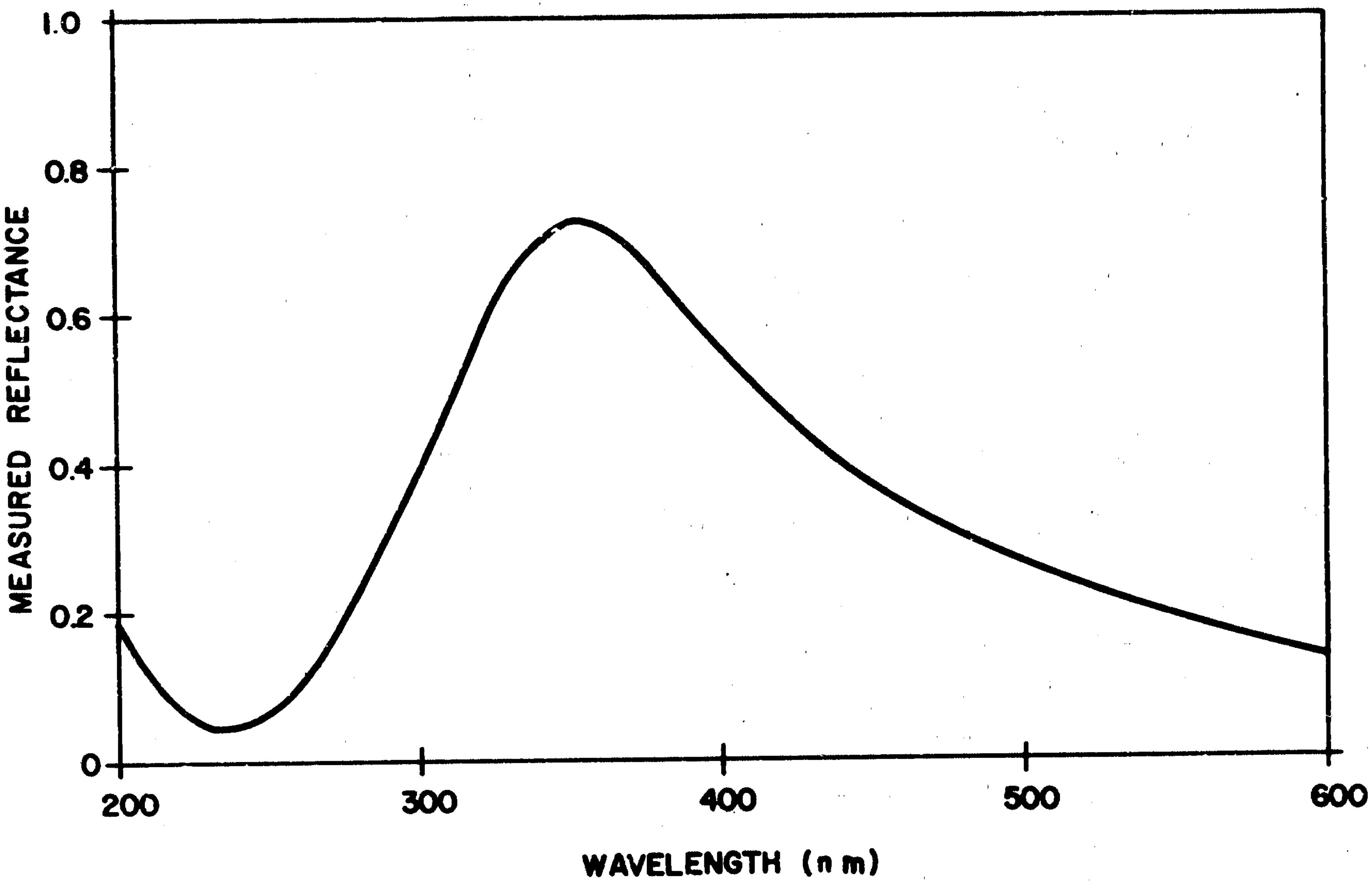


Fig. 15. Reflectance of a filter with chromium, magnesium fluoride, and aluminum.

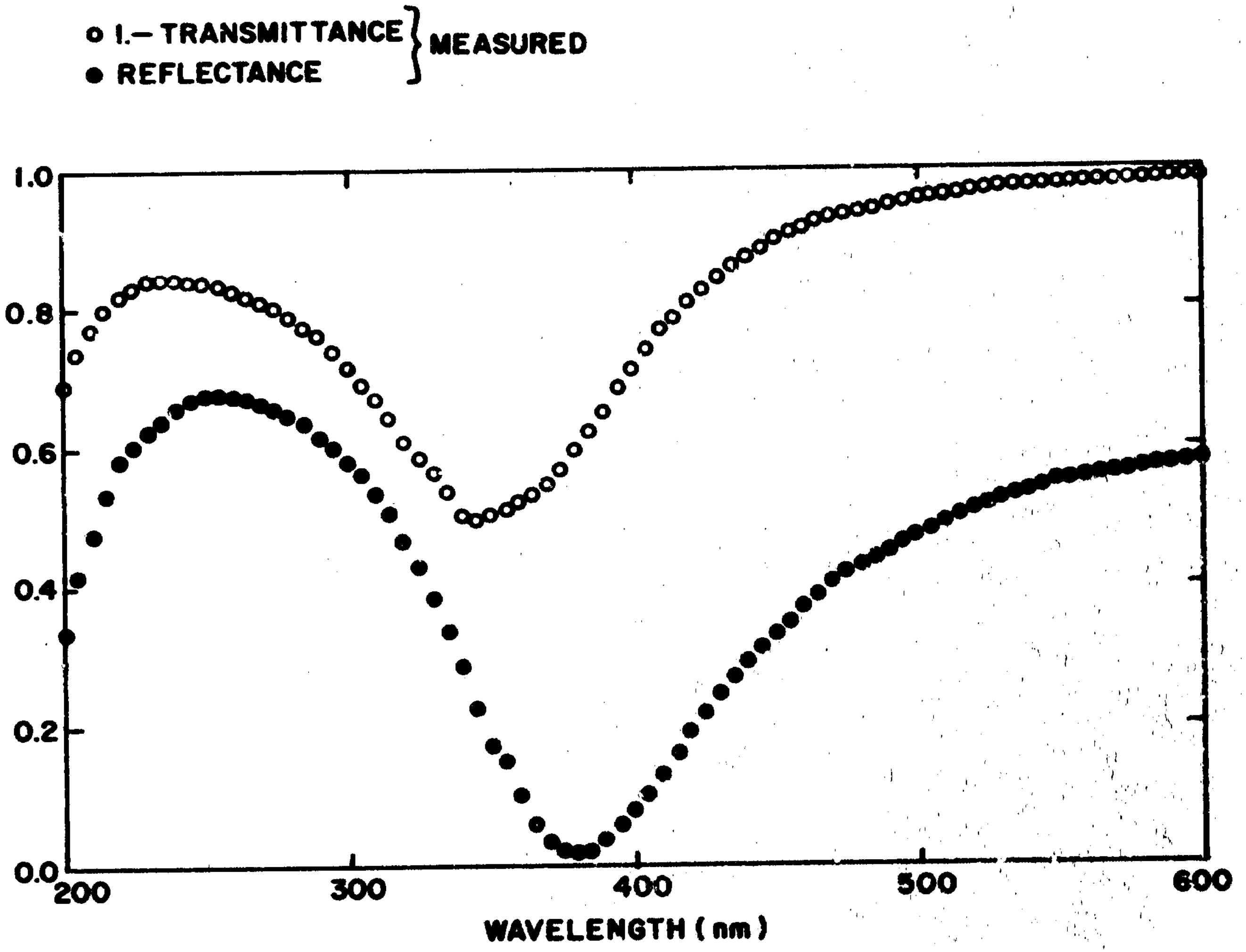


Fig. 16. Example of a $(DMD)^2$ filter.

is less than 0.03, which is only slightly greater than the uncertainty in the measurements. The differences, though slight, are thought to be real. Their source is not known, however. There may be some residual absorption in the dielectric layers. It is also possible that the aluminum-magnesium fluoride interfaces are not as abrupt as assumed. The chemically active aluminum surface may be interacting with the magnesium fluoride vapor as the dielectric film condenses. For the double period, however, such effects can be considered negligible.

Figs. 17 and 18 show the results of measurements of 3 and 4 period DMD stacks, respectively. Several of the features which were predicted above can be observed. As the number of periods is increased, the peak reflectance rises, and the high reflection band near 250 nm becomes sharper. Since there is no transmittance at long wavelengths, nothing can be done about the high long wave reflectance by adding more similar periods. The exact features of the three- and four-period stacks were not as close to the predicted values as was the case with the two-period stack. The variances can be attributed to a combination of a 1 to 5% increase in the real part of the refractive index of the metal layers, and a 5% or less error in the thicknesses of the dielectric layers. Such errors are within the expected error. To maintain an accuracy in the dielectric layer thickness of ±5% or better, one must be able to determine the minimum to an accuracy of better than ±0.0003 absolute reflectance. With the scale expansion features of the present monitor electronics, this represents a reasonable limit.

Although the properties of these filters correspond closely to the computed properties, they do not represent an optimum solar-blind mirror. According to the theory, a better result would be obtained by decreasing the thickness of the metal layers. We demonstrated above that we could not expect the computed results because of the thickness dependence of the optical constants. Since we have not measured the refractive indices, we cannot compute, with confidence, any filter properties for metal layers thinner than 10 nm. We did, however, make a DMD stack with five periods and a metal thickness of about 5 nm. The measured reflectance and transmittance are shown in Fig. 19. The most interesting feature to note is the low visible reflectance. That the peak reflectance is not high is not surprising since the metal is probably more absorbing that it would be if it were thicker.

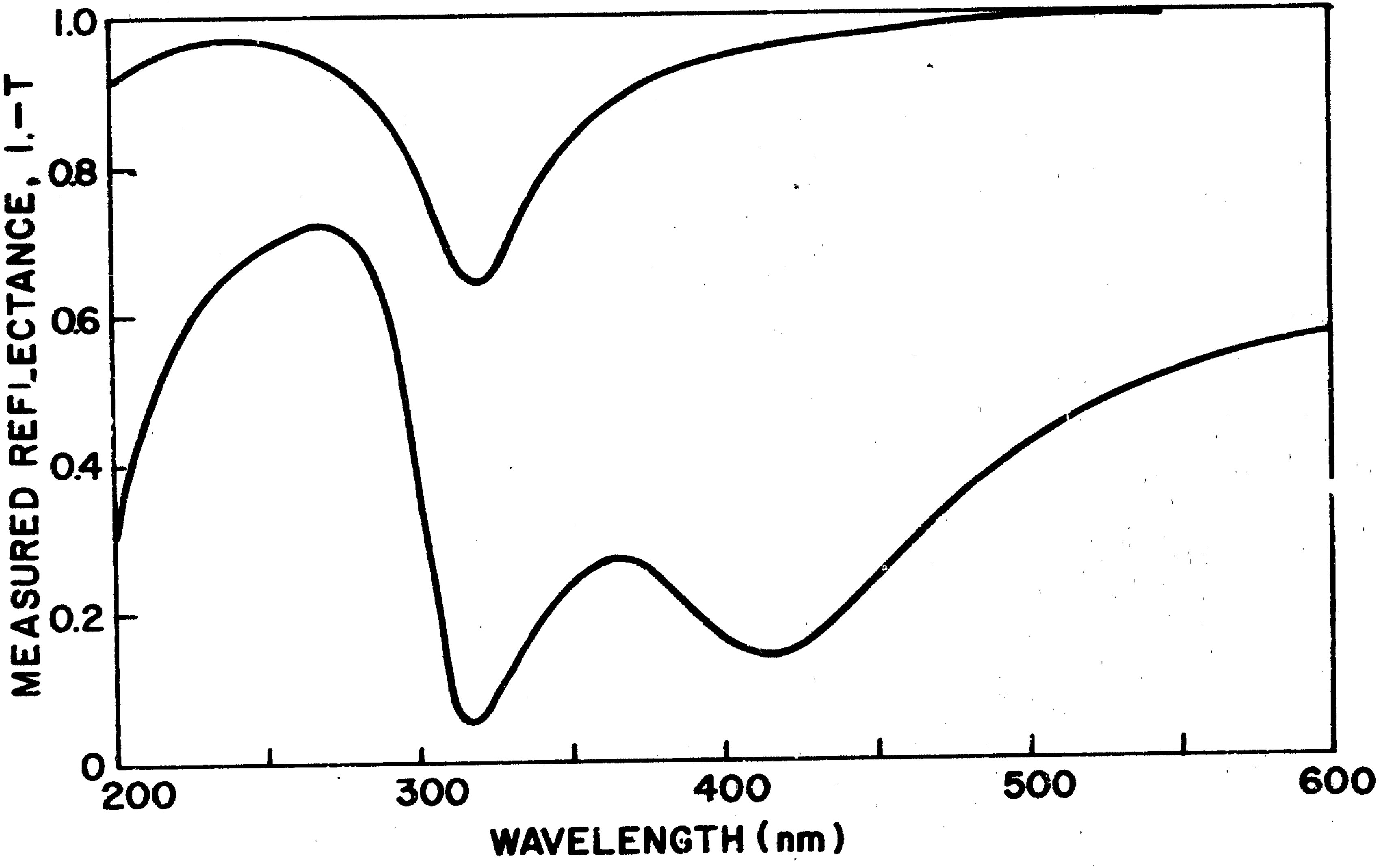


Fig. 17. Measurements of a three-period DMD stack.

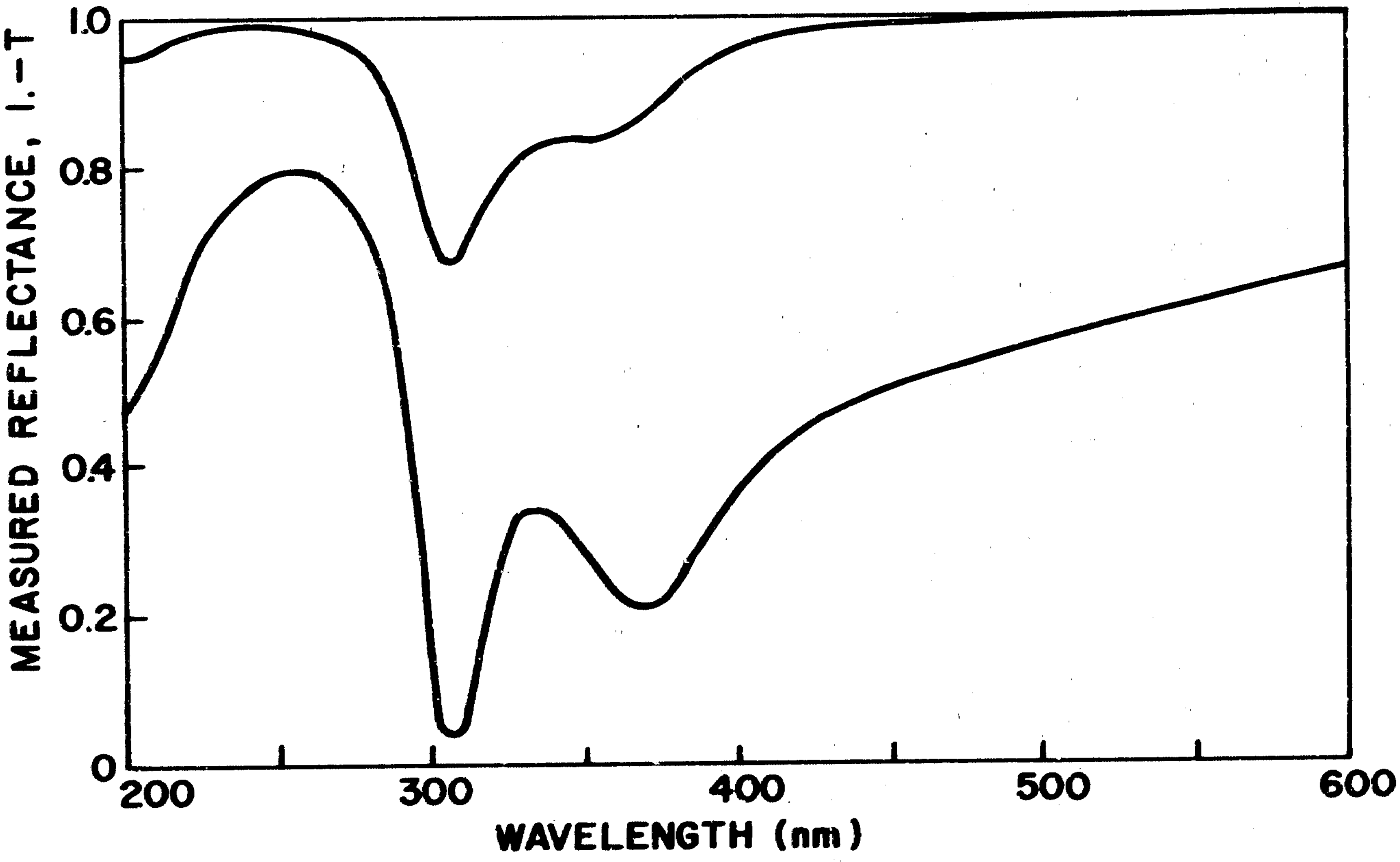


Fig. 18. Measurements of a four-period DMD stack.

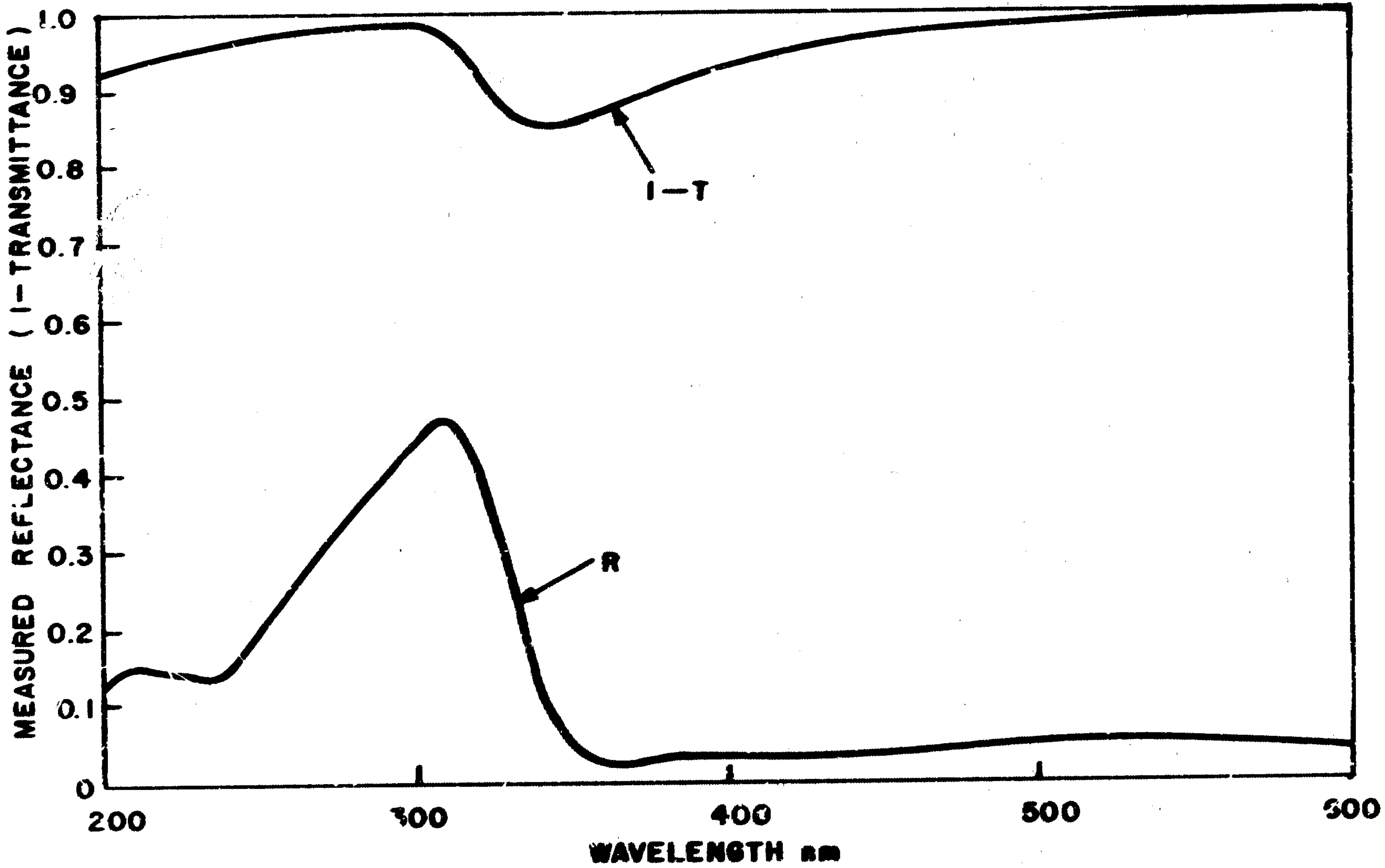


Fig. 19. Reflectance and transmittance of a DMD stack of five periods and metal thickness of about 5 nm.

DISCUSSION AND RECOMMENDATIONS FOR FURTHER WORK

We have studied the properties of two types of interference filters in this research to better understand their properties and to determine whether it might be feasible to make mirrors which reflect only ultraviolet light. Techniques for both the design and fabrication of such mirrors have been developed, and it has been established, for the first time, that under certain conditions the properties of metal-dielectric interference filters can be predicted from the simple theory of homogeneous thin films.

We have found that the thickness limit for thin aluminum films is 9 nm. Films thicker than this value will have the optical constants which have been previously published. Detailed measurements of the indices of thinner films should be made, as it appears possible to use the material to advantage to obtain solar-blind mirrors. One method that is attractive without further extensive work is to use thick (10 to 12 nm) aluminum films in a DMD stack with about five periods, and over-coat this with one or more periods using thin (~5 nm) metal layers. Such a combination should leave the desirable square high reflectance at short wavelength and rapidly kills the long wave reflection. This should be effective because the thin metal films will be located at the electric vector nodes at short wavelengths and at the antinodes at longer wavelengths. If measurement of the indices proves too difficult, an empirical determination of such a design is feasible since there are, at most, only one or two metal thicknesses as variables. Work of this sort is continuing. It should also be determined whether good solar-blind mirrors could be found by using combinations of two dissimilar metals as we did for the MRF's.

We have found that the properties of the materials utilized are very good when evaporated in ultrahigh vacuum. Further work should be undertaken to improve these properties by changing evaporation parameters in a systematic way. Evaporation rate, residual gas atmosphere, and substrate temperature effects should be considered.

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