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CaF2 LASER WINDOW STUDY

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Single crystal calcium fluoride material has been polished, antireflection coated and characterized. The objective of this effort was to obtain the necessary surface preparation and antireflection coating information for single crystal calcium fluoride substrates for use as high energy/power windows operating in the 3-5 μm wavelength region.

CaF_2 LASER WINDOW STUDY
FINAL REPORT

November 1974

Prepared by

S. J. Holmes, P. Kraatz, and A. Klugman

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1.0 INTRODUCTION

This report describes the results of a three-month program to polish, coat, and characterize several single crystal calcium fluoride samples. The work was undertaken at the Northrop Research and Technology Center from 15 July 1974 to 15 October 1974 and was supported by funds from the Naval Research Laboratory through the Office of Naval Research under Contract N00014-75-C-0009.

The objectives of the program were directed toward a fundamental understanding of the problems associated with the polishing and coating of calcium fluoride blanks for use as high energy laser windows in the 3.0 to 5.0 μm spectral region. In order to achieve this understanding, samples of single crystal calcium fluoride material were polished with an effort toward avoiding surface irregularities, such as pits and scratches. Surface roughness of 20 Å rms and flatness of $\lambda/8$ in the visible was set as a goal. The polished samples were thin film coated on both surfaces with two-layer antireflection coatings at the 5.0 μm, 3.8 μm, and 2.8 μm laser wavelengths to demonstrate the state-of-the-art coatings. Maximum transmission was the primary goal. Consideration was given to the choice of coating materials that would have good adhesion to the substrate surface with minimum absorption and scattering within the deposited layers.

Section 2.0 outlines the details of the surface preparation. Procedures used for blocking, grinding and polishing the calcium fluoride samples are discussed in this section. The polished surfaces were inspected and characterized by a variety of techniques including optical microscopy, polariscope evaluation, Nomarski microscopy and interferometry. The results of these characterizations are given in Section 3.0. Finally, in Section 4.0 the designs, the coating materials, and deposition techniques for fabricating two layer antireflection coatings are discussed. Spectrophotometer transmission scans of the experimental coatings are shown to demonstrate the spectral performance.

2.0 SURFACE PREPARATION

A total of four blocks of 49 mm CaF_2 window blanks were polished. Three blocks consisted of three blanks and one consisted of seven blanks. The window substrates were polished on both sides, requiring re-blocking after completion of the first side. Procedures used for blocking, grinding and polishing are discussed in this section.

2.1 Blocking. Pitch is an essential material used as an adhesive and supporting medium for maintaining the position of optical blanks on a block during surface finishing. Pitch is also the material most commonly used to fabricate the polishing lap itself. Pitch is classified qualitatively by hardness (e.g., thumbnail penetration tests) and more quantitatively by melting point. Hard pitch has a melting point between 160 and 180°F and does not allow thumbnail penetration, even under strong pressure. Medium pitch has a melting point between 145° and 160°F and allows thumbnail penetration with moderate pressure. Soft pitch melts between 130° and 145°F and allows immediate and deep thumbnail penetration.

In mounting the blanks three or seven to a block, a flat aluminum plate is used as the backing tool. The tool and blanks are heated to the same temperature on an electric hot plate at 165°F. Pitch or wax is rubbed on the tool and is spread over the tool as it melts (melting point 160° - 165°F). The warm CaF_2 is placed on the pitch and the whole block is then allowed to cool slowly to room temperature. The use of a pitch having a lower temperature melting point is not advisable becuase the lower hardness will allow the blanks to shift out of plane from each other during the grinding and polishing steps. This would cause sleeking, scratching, and preclude figure test control. After the first side is polished, the block is placed on a hot plate. When the pitch starts melting, the blanks are slid from the block. The second side requires more consideration in blocking as we now have a highly polished surface to mount against the tool. To protect this surface from scratching, a piece of lens tissue is placed

on the melted pitch, more pitch is added to the paper, and then the CaF_2 blank is placed on the pitch, polished side down. This is a standard optical procedure which has yielded excellent results with glass.

This blocking procedure was used on the first three blocks which were polished using a modified bowl feed technique. While polishing the first block, all the symptoms of a bad blocking job were observed, i.e., poor figure test control and sleeking. The windows were therefore reblocked using a harder pitch. The symptoms continued, suggesting it was a materials problem with either one or two blanks having a different hardness and/or differing in composition from the others.

The second block of samples polished quickly and reasonably smooth on both sides. The third block, containing seven blanks, polished very well on the first side. The problem encountered on the first block recurred on the second side of this block (almost obvious shifting, excessive sleeking, and poor figure control).

Side one of a fourth block of three samples was polished using the brush feed method. The surface appeared to be as good as any prepared using a bowl feed technique. The second side was blocked without any paper under it and stayed in plane throughout the entire polishing cycle. Considering these results, we must reconsider our earlier conclusion of the cause of the problem in polishing the second side of the first and third blocks as being one of blocking rather than of materials (see monthly progress report, 30 September 1974). This, of course, raises the question of how to mount the first finished side without scratching it.

2.2 Grinding. All CaF_2 samples were ground with 9 μm Al_2O_3 abrasive in water on a cast iron lap for 1 to 2 hours per side. The purpose of this operation is to remove gross defects resulting from sawing or rough grinding and reduce all surfaces to a common level, assuring full

contact with the lap in subsequent polishing operations. No problems were encountered with this procedure for any of the four blocks, so the process was not altered.

2.3 Rough Polishing. To remove the light scratches and pits remaining from the grinding operation, the ground blanks were polished with 1 μm Al_2O_3 (Linde C) in water on a pitch lap for approximately 16 hours per side. Blanks are considered ready for final polish when visual inspection with a 7X loupe reveals that all grinding marks have been removed.

In an attempt to speed up the rough polishing process, a lead lap was used with alumina powder in various vehicles such as water, oil, and grease. The concept was to rapidly remove grinding marks such as pits and scratches and then proceed with the final polishing operation. The results were disastrous; although the procedure did start to shine the surface, it introduced scattered scratches and pits deeper than the original ground surface. Hence the technique was abandoned in favor of the pitch lap. Different pitches of varying hardnesses were tried but they did not make much difference in rough polishing.

2.4 Final Polish. Following rough polishing, the CaF_2 samples were polished with 0.3 μm Al_2O_3 (Linde A) in deionized water on a medium pitch lap using either a recirculating slurry system (a modified bowl feed technique) or a brush feed technique (fourth block of the series) to achieve the final low scatter surface. The pH of the abrasive slurry is ~ 5.8 for either technique. The only substantive changes made in the procedure comprised increasing the concentration of abrasive and softening the pitch by addition of oil in the polishing of the third block (seven samples). Samples are considered finished when examination using the Nomarski microscope reveals no sleeks (at 110X) and the optical figure is satisfactory when examined with the Davidson Interferometer.

The main problem encountered in final polishing was the inherent difficulty of simultaneously achieving a sleek-free surface having a low rms roughness, and a good optical figure (e.g., $\lambda/5$ in the visible). Both problems may be related to or heavily influenced by blocking techniques and materials, as discussed previously. However, pitch lap hardness, spindle speed, overarm weight, abrasive concentration, and chemistry of the slurry all exert an influence. Further investigation is needed to isolate the effects of these parameters.

An additional problem encountered in blocking, grinding, and polishing of single crystal CaF_2 is sample cleavage. The precipitating cause of cleavage is differential stress at points of high stress concentration. The stress may arise from local thermal gradients which occur in blocking on a hot plate or in the polishing operation itself. For most of the cleavage problems encountered in the present work, the stress concentrator was a scribed identification number in the edge of a sample. These stress concentrations were revealed by polariscopic examination prior to the first blocking of the samples. Hence, indelible ink rather than scribing should be used to identify samples and thermal gradients during blocking should be reduced to a minimum by using oven heating.

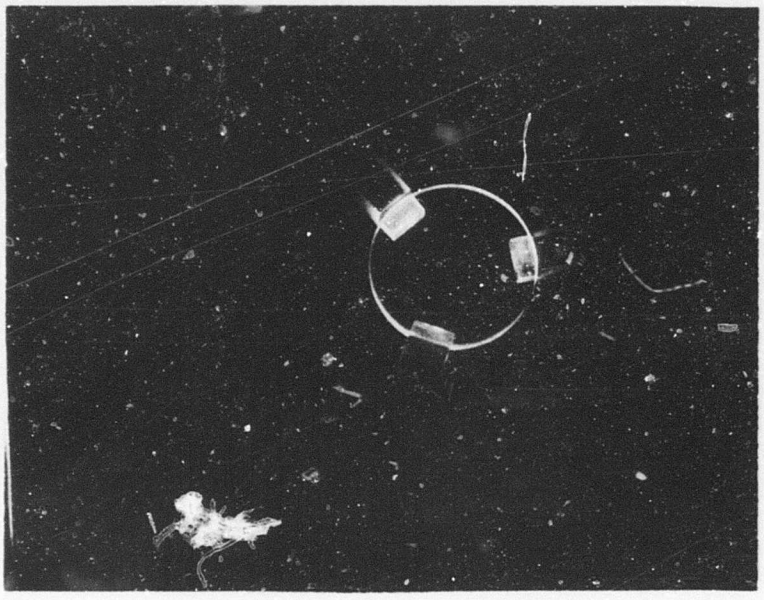
3.0 CHARACTERIZATION

All samples have been characterized with a plane polariscope, a Leitz microscope in Nomarski phase interference configuration (at 110X), and a Mach-Zehnder interferometer. The polariscope serves to reveal birefringence due to residual strain, impurity segregation, or grain boundaries. The Nomarski photomicrograph provides qualitative information on the roughness of the surface upon completion of polishing and the interferometer provides information on the optical quality of the entire sample.

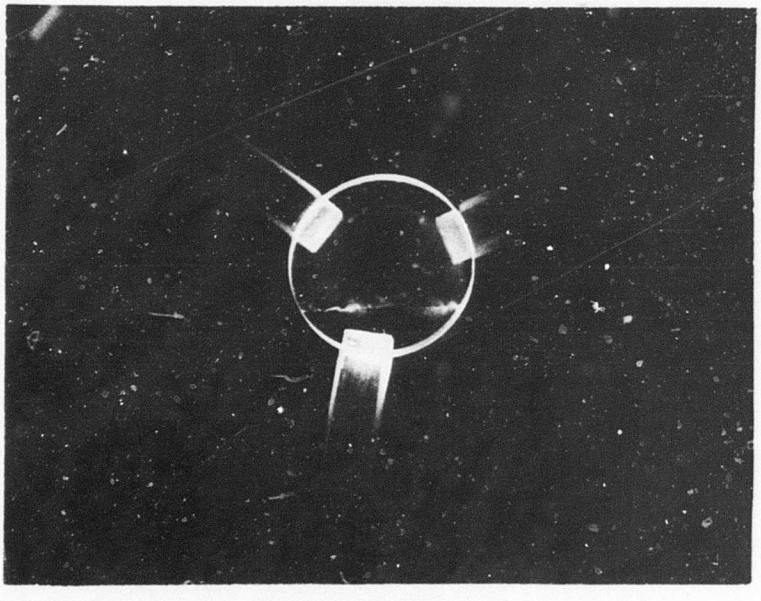
3.1 Plane Polariscope. The polariscope is a useful tool for screening optical materials prior to extensive efforts at surface finishing. A rough polish sufficient for transparency is the only preparation required. Examples of an isotropic sample and a sample containing a planar grain boundary at a high angle to the surface are shown in Figure 1. The sample (number 3) with the boundary, figure 1(b), was polished, AR coated at 3.8 μm and eventually submitted for evaluation. The boundary did not result in cleavage or other damage to the sample.

For other samples, polariscopic examination revealed concentrations of strain in material immediately adjacent to numbers scribed in sample edges. These were easily observed visually but not readily photographed. They led to cleavage of the blanks in three cases. In one case (sample number 12 polished on block number 3), a cleavage initiated at the scribed number apparently followed a grain boundary into the interior of the crystal.

3.2 Nomarski Phase Interference Microscopy. Nomarski microscopy provides an excellent qualitative means for evaluation of the cosmetic quality of optical surfaces, as affected by polishing and coating processes.



(a) Optically isotropic CaF_2 sample



(b) CaF_2 sample with grain boundary

Figure 1. CaF_2 windows in the plane polariscope.

Figure 2 shows both surfaces of a CaF_2 sample as received from the vendor. (The dark spots at bottom center and upper right are artifacts arising from dirt in the lens system of the microscope and do not represent defects in the surfaces.) This is a typical surface with which the Northrop grinding and polishing operation begins. The roughness of the sample surfaces measured at the Michelson Laboratory, Naval Weapons Center, China Lake, California, by scatter techniques, was found to average 63 Å rms. Figure 3 shows a direct comparison of Nomarski photomicrographs of the same general area of a CaF_2 sample surface as received from the vendor and as polished at Northrop. Note the multiple fine sleeks in 3(a) and their absence in 3(b).

Figure 4 provides a comparison between the cosmetic quality of a CaF_2 sample polished at Northrop and the optical figure, as illustrated by the Davidson interferometer pattern. This shows the achievement of a 22.5 Å rms surface on an optical surface flat to ~ λ at 5800 Å. This sample (number 6) was polished on the second block of 3 prepared under the contract.

Figures 5 and 6 illustrate surface quality and flatness of a sample (number 8) polished on the third block (seven samples). Note that side 1 is flat to ~ λ/5 at the visible (Figure 6 (a)) while side 2 is flat to ~ λ/2 (Figure 6 (b)).

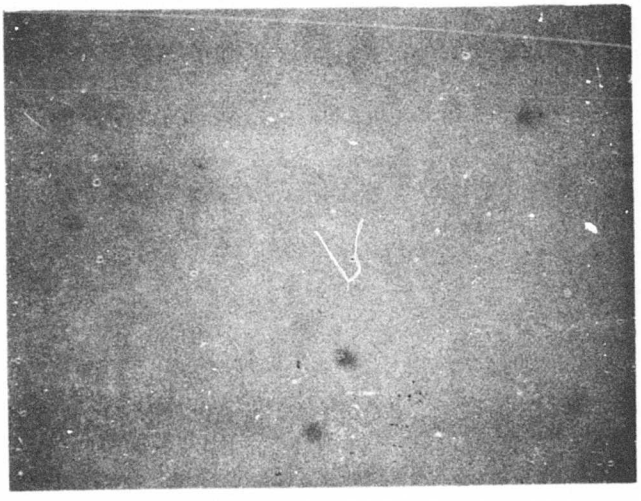
To examine the effects of AR coatings on surface quality, Nomarski micrographs of two samples were taken before and after coating. Figures 7 and 8 indicate that very little difference is apparent in the Nomarski microscope at this magnification. However, the rms roughness of Sample 5 coated with ThF_4/PbF_2, design wavelength 5 μm, showed high variability from a maximum of 71 Å to a minimum of < 19 Å, as measured by the scatter technique at NWC. This may indicate localized increases in surface roughness due to agglomeration of coating material.

(a) Side Number 1

(b) Side Number 2

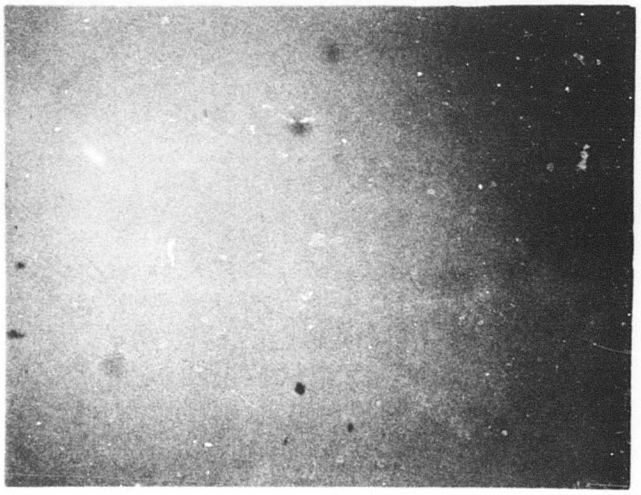
Figure 2. CaF_2 sample number 14, as received. Rms roughness 63Å as measured by scatter technique at NWC. Nomarski 110X.

(a) CaF_2 sample number 2, as received from vendor



(b) CaF_2 sample number 2, after polishing at Northrop

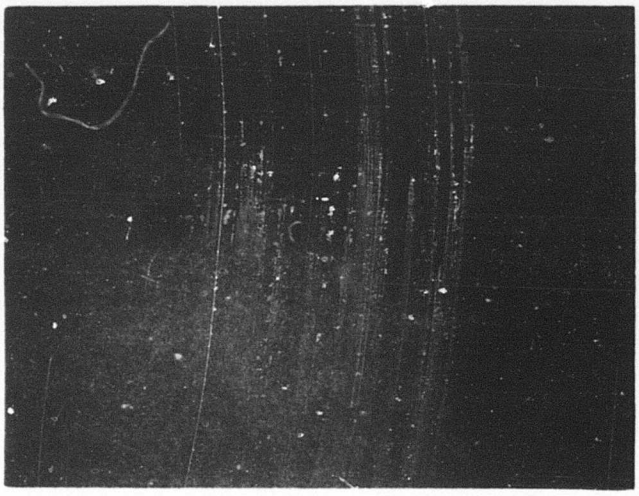
Figure 3. Comparison of CaF_2 sample surfaces before and after polishing. Nomarski 110X.



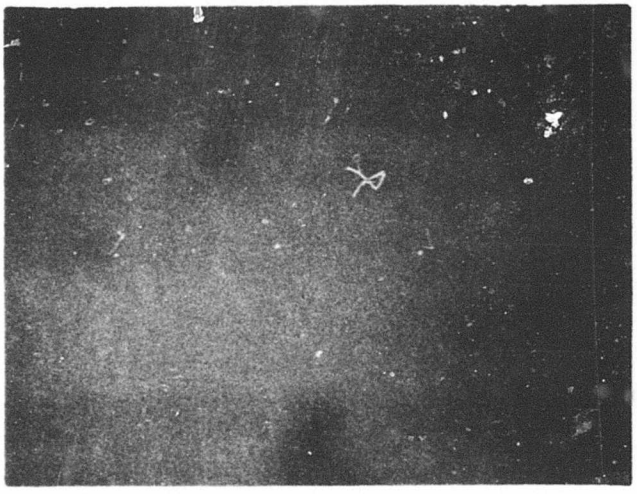
(a) CaF_2 sample number 6. Nomarski 110X

(b) CaF_2 sample number 6. Davidson Interferometer pattern

Figure 4. Comparison of surface finish (a) with figure (b) of CaF_2 sample number 6. Surface roughness 22.5Å rms (average), 19.5Å rms (minimum), measured at NWC by scatter technique.

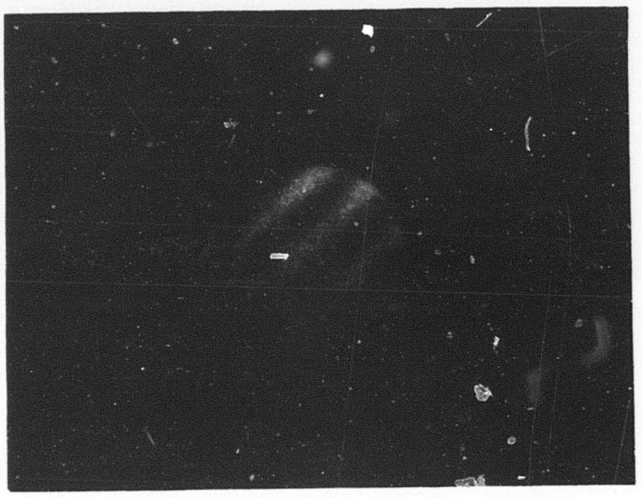


(a) CaF_2 sample number 8, side 1, Northrop polish (block of 7 samples)

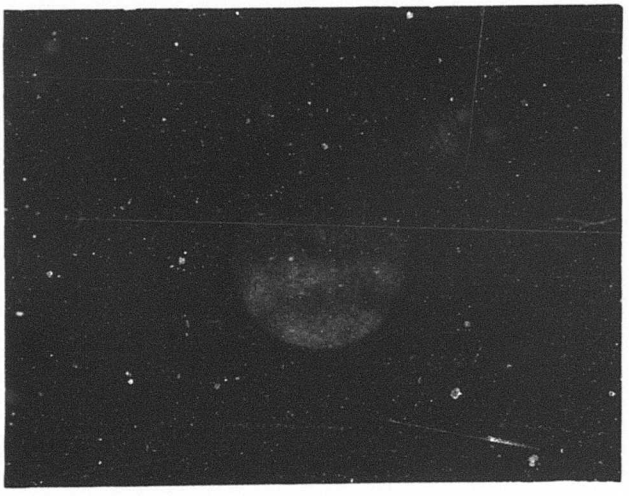


(b) CaF_2 sample number 8, side 2. Northrop polish (block of 7 samples)

Figure 5. Nomarski micrographs of sample number 8, block number 3. Nomarski 110X.

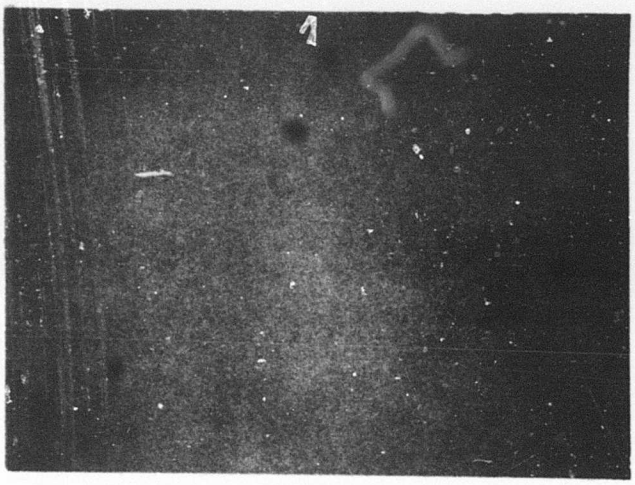


(a) CaF_2 sample number 8, side 1

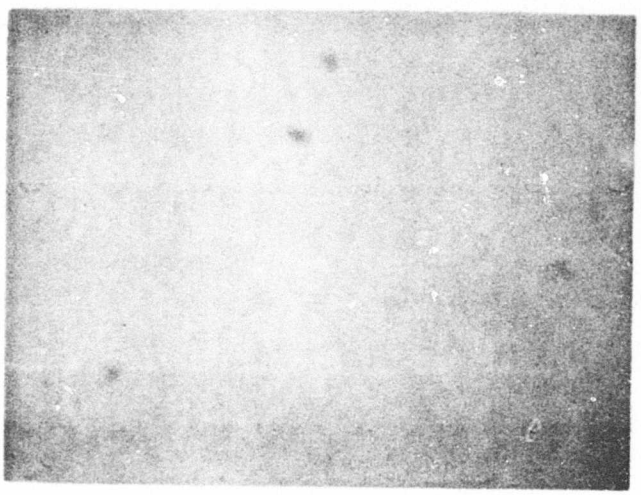


(b) CaF_2 sample number 8, side 2

Figure 6. Davidson interferometer patterns of sample number 8, block number 3 (7 samples).

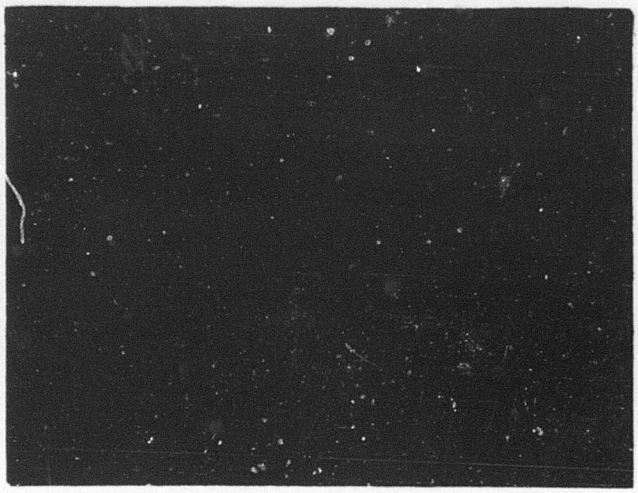


(a) CaF_2 sample number 3, as polished at Northrop

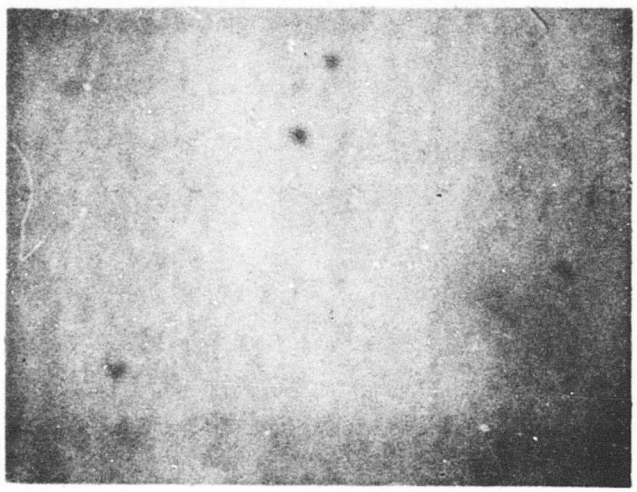


(b) CaF_2 sample number 3, after AR coating at Northrop

Figure 7. Comparison of CaF_2 sample number 3 surface before and after AR coating (3.8 μm design wavelength). Nomarski 110X.



(a) CaF_2 sample number 5, as polished at Northrop



(b) CaF_2 sample number 5, after AR coating at Northrop

Figure 8. Comparison of CaF_2 sample number 5 surface before and after AR coating (5 μm design wavelength). Nomarski 110X. Rms roughness 71Å (maximum), 19Å (minimum), measured by scatter at NWC.

4.0 ANTIREFLECTION COATINGS

Antireflection coatings are required for most laser window applications to reduce unwanted reflections to as low value as possible. The requirement of less than 0.1% reflection with absorption losses being less than 0.1% per surface is desirable. Low surface reflection is required to prevent unwanted reflections from propagating back into the laser cavity. Low absorption is required to minimize window heating and resulting beam distortion in high average power applications and to preclude damage to the window when exposed to high energy in both cw and pulsed applications. For most laser applications, highly efficient antireflection coatings are only required for a relatively narrow wavelength range. Therefore, partial antireflection coatings are usually restricted to single layer or two layer designs. This is particularly true for infrared lasers where the large physical thicknesses required for the layers might cause problems in the realization of a given design.

The work reported here was directed towards a fundamental understanding of the problems associated with the coating of calcium fluoride surfaces for use as high energy laser windows in the 3.0 to 5.0 μm region. The primary concern was the realization of maximum transmission at a prescribed wavelength. Consideration was also given to the minimization of absorption in the selection of the coating materials.

4.1 Practical Coating Designs. The single layer antireflection coating is the simplest design both thecretically and experimentally. However, the single layer as an antireflection coating is a quarterwave optical thickness of a material whose refractive index is equal to the square root of the product of the refractive indices of the substrate material and the incident medium[1]. From this, it is clear that the relatively low refractive index of calcium fluoride ($n \simeq 1.4$) and the lack of a suitable coating material of the required refractive index precludes the use of a single layer antireflection coating design.

When two layers are used in the coating design, the added freedom permits one to choose many different combinations of refractive indices and layer thicknesses to produce zero reflectance at a prescribed wavelength. The theory and preparation of two layer antireflection coatings have been discussed by Cox and Haas[2]. The layer thicknesses necessary to produce zero reflectance with a two layer antireflection on a substrate can be calculated from the following equations[3]:

$$\tan^2 \varphi_1 = \frac{n_1^2 (n_s - n_o)(n_2^2 - n_o n_s)}{(n_1^2 n_s - n_2^2 n_o)(n_o n_s - n_1^2)} \quad (1)$$

and

$$\tan^2 \varphi_2 = \frac{n_2^2 (n_s - n_o)(n_o n_s - n_1^2)}{(n_1^2 n_s - n_2^2 n_o)(n_2^2 - n_o n_s)} \quad (2)$$

where

n_o = refractive index of the incident medium

n_1 = refractive index of the outside layer

n_2 = refractive index of the inside layer

n_s = refractive index of the substrate

$\varphi_1 = n_1 t_1 / \lambda o$ (3)

$\varphi_2 = 2 n_2 t_2 / \lambda o$ (4)

t_1 = physical thickness of the outside layer

t_2 = physical thickness of the inside layer

λo = design wavelength.

The solution of equations (1) and (2) leads to a series of choices of φ_1 and φ_2 and a considerable flexibility in the choice of coating materials. From all the combinations of n_1 and n_2 compatible with equations (1) and (2), the ones with the lowest values are preferred because they yield the broadest reflectance minimum[4].

For the calcium fluoride windows we have chosen an antireflection coating design having two layers of equal optical thickness. Figure 9 shows the notation used in discussing the parameters of the two layer antireflection coating design. The refractive indices of the incident medium and substrate material are n_o and n_s, respectively, and the layers are numbered from the outside towards the substrate. The optical thicknesses of the two layers are quarterwaves at the design wavelength. This requires, for a perfect two layer antireflection coating, that the refractive indices fulfill the following equation[5]:

$$n_1^2 n_s = n_2^2 n_o. \tag{5}$$

If the index condition of equation (5) is not fulfilled, the reflectance minimum of the antireflection coating design is not zero. The reflectance at this minimum can be calculated from the following equation[6]:

$$R_{min} = \left[\frac{n_1^2 n_s - n_2^2 n_o}{n_1^2 n_s + n_2^2 n_o} \right] \tag{6}$$

4.2 Coating Materials. While a number of thin film coating materials were under consideration for use in the fabrication of the antireflection coatings, it was decided to put the emphasis on the fluoride compounds. They are compatible with the properties of the calcium fluoride windows and have the possibility of being deposited with similar crystalline structures to that of the surface of the substrate[7]. The nominal refractive

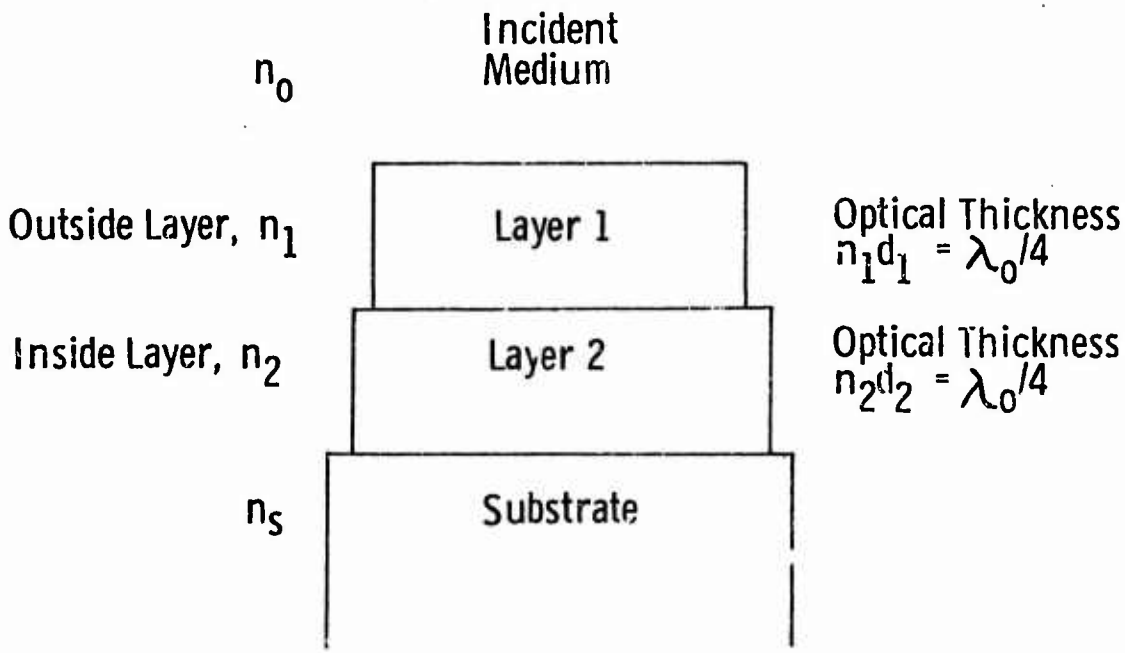


Figure 9. Notation used for the refractive indices and optical thicknesses of the antireflective coating design.

indices of the calcium fluoride window and the fluoride compounds used in the design calculations are listed in Table 1. The fluoride compounds are chemically stable and show little decomposition during the evaporation. They are easily evaporated and deposited in high vacuum using electron beam heating. This enables films to be deposited which are free from source contamination and are harder than those evaporated from a conventional evaporation source using resistance heating.

All of the coating materials used to fabricate the antireflection coatings were procured from the Balzers High Vacuum Corporation. They were of the highest purity available (99.9%) and in granular form. The powder forms are not recommended because they permit the absorption of water and other contaminants which are released upon heating, causing localized exploding or spitting from the material.

4.3 Experimental Techniques. All of the antireflection coatings described in this report were done in a commercial coating system (Balzers 710) which was supplied with an oil diffusion pump and a liquid nitrogen trap. Before placing the windows into the system for coating, they were cleaned by washing with a detergent and warm water, then rinsed with distilled water and alcohol and blown dry with nitrogen gas. The system was evacuated to a pressure of less than 10^{-6} Torr and a pressure of less than 5×10^{-6} Torr was maintained during the deposition. The windows were heated to approximately 200°C and maintained at this temperature during the coating depositions. Just prior to the deposition of the coatings, the windows were subjected to a glow discharge cleaning for three minutes to remove any remaining contaminants from the surfaces. Deposition of the coating materials was accomplished by evaporation from an electron beam source having a variability of power to avoid dissociation and to obtain the desired deposition rate. The deposition rate was approximately 2500 Å/min, and the layer thicknesses were controlled by optical monitoring techniques using a 1 μm control filter.

Window Material	Wavelength			
	5.0 μm	3.8 μm	2.8 μm	Ref.
CaF_2	1.399	1.411	1.419	8
Coating Materials	**Wavelength**			
	5.0 μm	3.8 μm	2.8 μm	Ref.
PbF_2	1.73	1.74	1.74	9
ThF_4	1.49	1.50	1.50	10
MgF_2	1.33	1.35	1.36	11
BaF_2	1.45	1.45	1.46	12

Table 1. Refractive indices of the materials used in the Design Calculations

4.4 Experimental Results. The emphasis of the experimental work was placed on the fabrication of antireflection coatings for the potential high power laser wavelengths of the CO laser centered at 5.0 μm and those wavelengths of the chemical lasers, DF and HF, centered at 3.8 μm and 2.8 μm respectively. Spectral transmission scans were taken on a Beckman IR-20 spectrophotometer to check the spectral performance of the coating designs and are discussed below.

Antireflection Coating at 5.0 μm

The spectral transmission of a calcium fluoride window coated on both sides with a two layer coating for which each layer is one quarter wavelength thick at 5.0 μm is shown in Figure 10. This design uses PbF_2 with a refractive index of 1.73 as the layer next to the window and ThF_4 with a refractive index of 1.49 as the outside layer. Although the minimum reflectance of this two layer coating is nearly zero, it is restricted to a rather narrow spectral region from 5.0 to 5.3 μm.

Antireflection Coating at 3.8 μm

Ideally the same antireflection coating design should be usable to coat the calcium fluoride window at any of the required wavelengths. To test this, a calcium fluoride window was coated on both sides with the same design used at the 5.0 μm wavelength. The optical thicknesses of the layers were adjusted to correspond to a quarter wavelength at 3.8 μm. Figure 11 shows the measured spectral transmission of the coating. The width of the minimum reflectance band decreases with decreasing wavelength.

Antireflection Coating at 2.8 μm

Figure 12 shows the measured spectral transmission of calcium fluoride window coated on one side with a two layer antireflection coating designed for 2.8 μm. The coating design uses a quarterwave optical thickness of

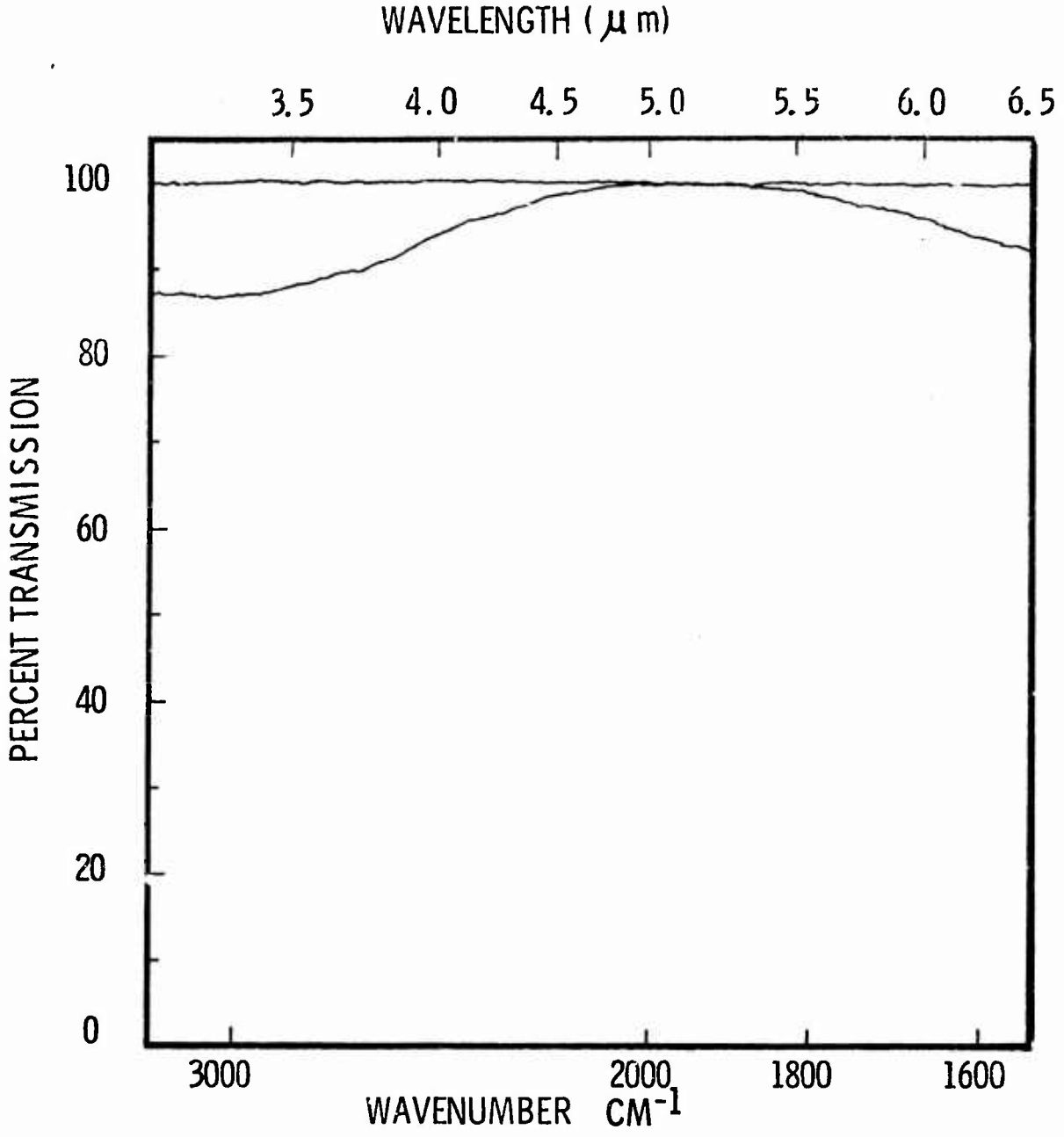


Figure 10. The measured spectral transmission of a CaF_2 window coated on both surfaces with quarterwave optical thicknesses of ThF_4 and PbF_2. Design wavelength is at 5.0 μm.

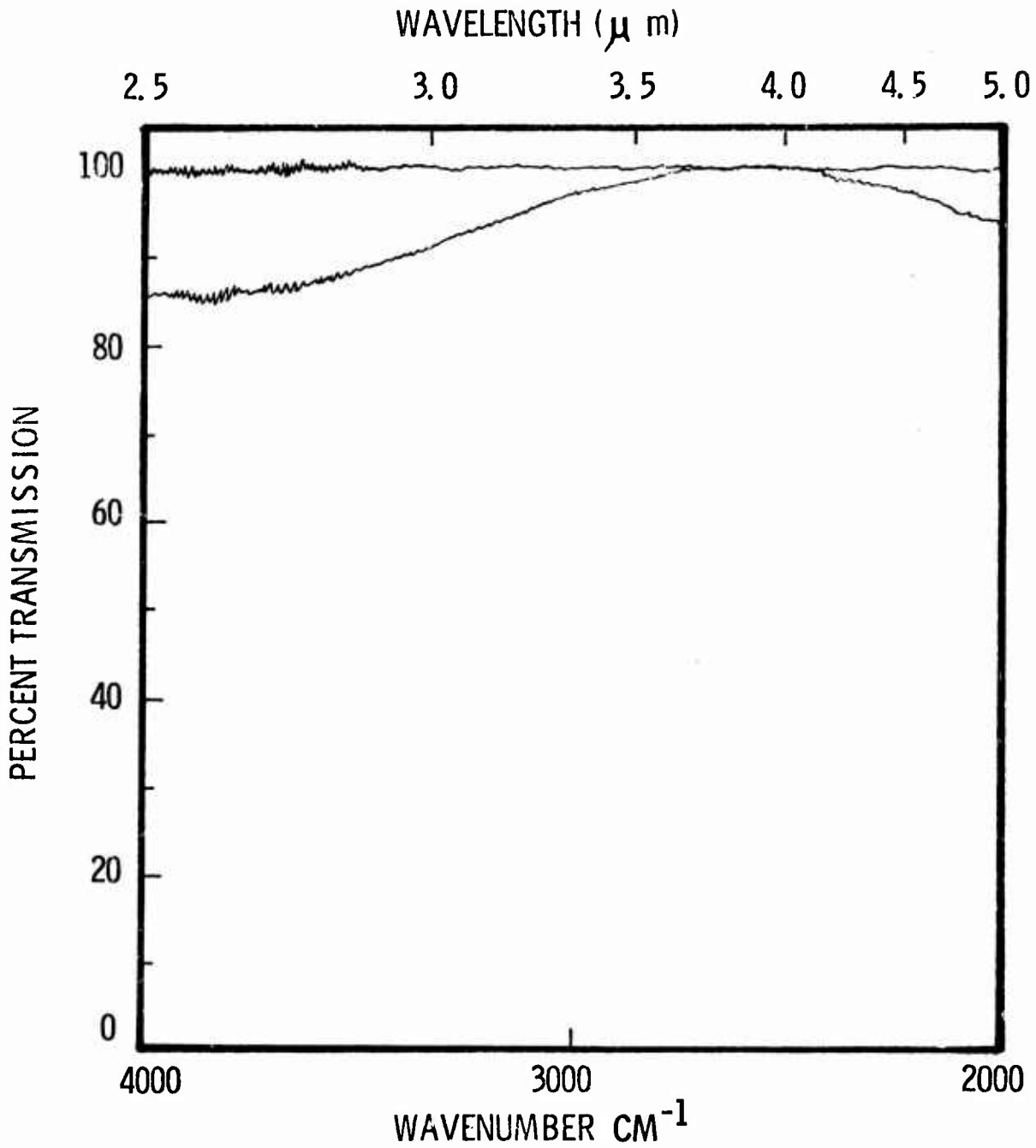


Figure 11. The measured spectral transmission of a CaF_2 window coated on both surfaces with quarterwave optical thicknesses of ThF_4 and PbF_2. Design wavelength is at 3.8 μm.

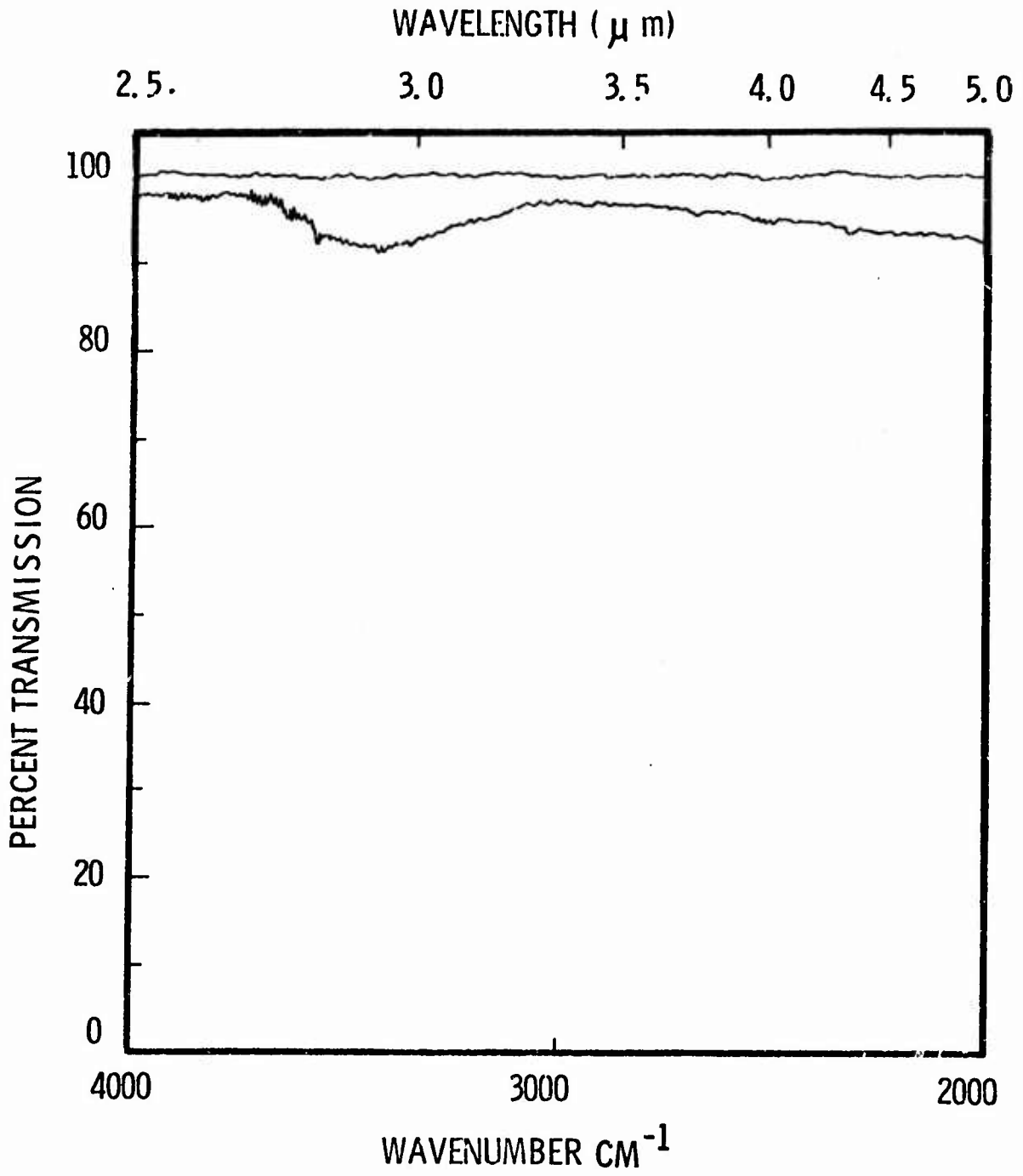


Figure 12. The effect of the OH absorbion band on the transmission of a CaF_2 window coated on one surface with quarterwave optical thicknesses of MgF_2 and PbF_2. Design wavelength is at 2.8 μm.

PbF_2 as the inside layer and a quarterwave optical thickness of MgF_2 as the outside layer. The wide dip in transmission from 2.7 to 3.3 μm is attributed to a water absorption band in the MgF_2 coating material. Similar results were observed when ThF_4 was used as the outside layer in the coating design. The best antireflection coating fabricated on the calcium fluoride window material at 2.8 μm has been a two layer coating. The design consists of a quarterwave optical thickness of BaF_2 as the outside layer. The minimum reflectance band of this design is considerably broader than the designs for 5.0 and 3.8 μm. This is because the refractive index of BaF_2 (1.46 at 2.8 μm) very nearly fulfills the refractive index requirement of Equation (5). The measured spectral transmission is shown in Figure 13.

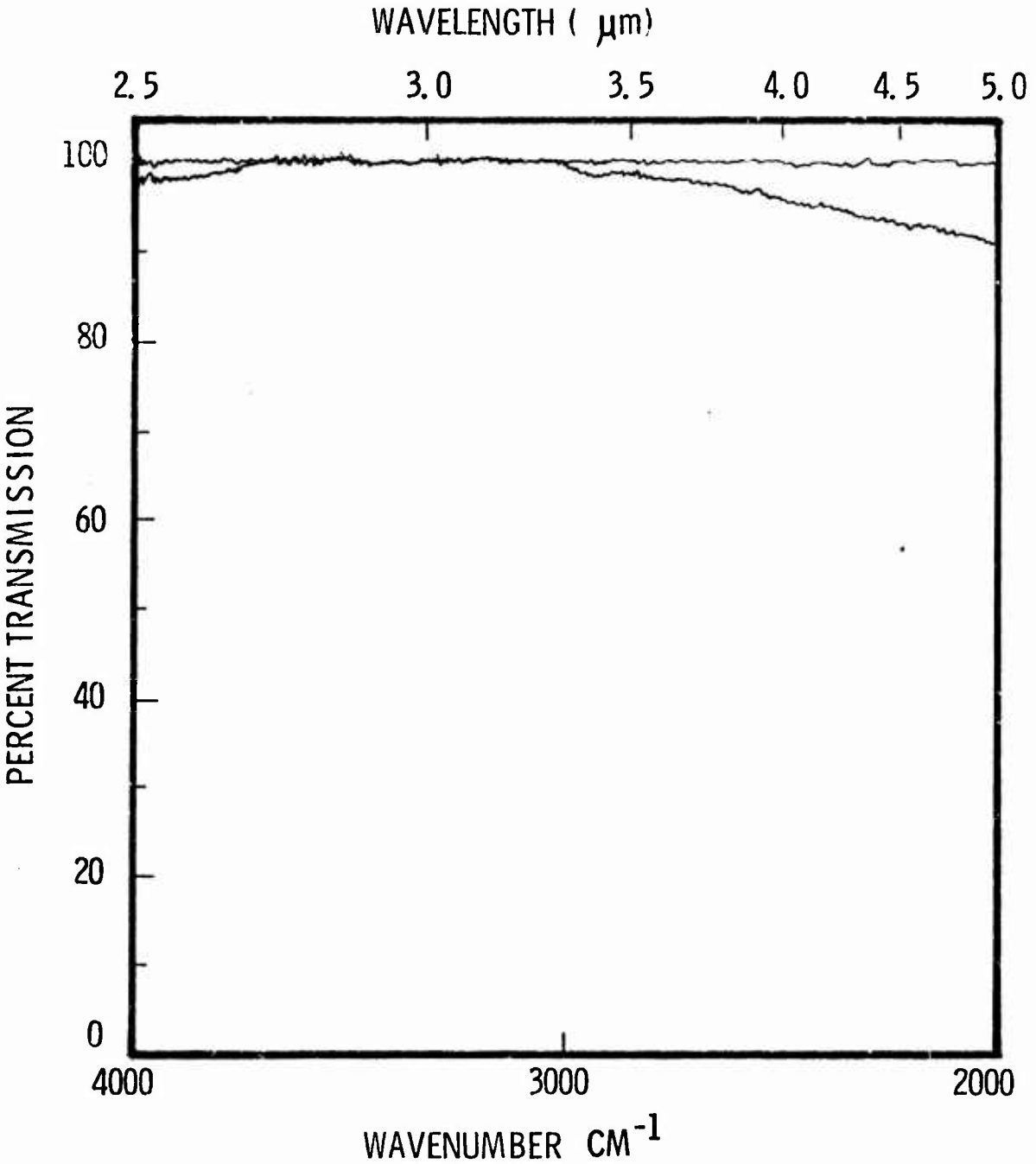


Figure 13. The measured spectral transmission of a CaF_2 window coated on both surfaces with quarterwave optical thicknesses of BaF_2 and PbF_2. Design wavelength is at 2.8 μm.

5.0 SUMMARY AND CONCLUSIONS

CaF_2 windows 49 mm in diameter can be polished to a superquality surface on multiple blocks with few sleeks and low rms. surface roughness. The surface figures achieved were flat between λ and $\lambda/5$ at 5800 Å. Both fresh feed and bowl feed polishing methods have been utilized. The polishing time varied from block to block, averaging 32 hours per side (three to seven pieces) for low scatter surfaces. This preparation time may be reduced by optimization of the polishing techniques. Further investigation is required for this optimization as well as to develop a method for blocking the second side of a multiple block so as not to damage the first side which has been finished.

Antireflection coatings for design wavelength of 5.0 μm, 3.8 μm, and 2.8 μm have been fabricated, using a two-layer (V-coat) design. All show very low reflection in narrow bands about the design wavelengths. Further work is needed to evaluate the absorption of the coated windows and to determine the absorption indices of various candidate coating materials for improvement of coating performance.

Nomarski microscopy and polariscopic evaluation have proven to be useful tools for characterizing substrate materials and surfaces. Further Nomarski work at higher magnification would be useful for analysis of particular problems occurring in polishing and coating processes.

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