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## A TECHNIQUE FOR MEASURING THE THICKNESS OF A THIN CONTAMINANT LAYER

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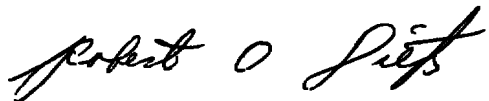
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approach to a thickness measurement, and an experimental program is outlined.

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## PREFACE

The work reported herein was conducted by the Arnold Engineering Development Center (AEDC), Air Force Systems Command (AFSC). The results were obtained by ARO, Inc. (a subsidiary of Sverdrup & Parcel and Associates, Inc.), contract operator of AEDC, AFSC, Arnold Air Force Station, Tennessee. The research was conducted under ARO Project Number VF215. The manuscript (ARO Control No. ARO-VKF-TR-74-4) was submitted for publication on January 10, 1974.

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## 1.0 INTRODUCTION

Cryogenically cooled infrared optical systems are susceptible to contamination from the environment or the transporting vehicle while operating at altitudes that vary from that of the high flying aircraft (35,000 ft) to that of outer space. The functional impairment of these systems from contamination must be evaluated in ground test facilities. Techniques for quantitatively depositing contamination or for characterizing the contamination after deposition must be developed and integrated with the test facility. In this way the optical degradation may be experimentally investigated as a function of the amount of contamination. A means of utilizing the second approach will be investigated in this report.

The general approach will be to see if the analysis of a single collimated monochromatic infrared beam reflected from the contaminated optics will yield adequate information to determine the optical constants. A simple measurement of the reflecting power at the same angle of incidence will provide sufficient information to calculate the thickness of the contaminant once the optical constants are known, provided there is negligible scattering of the light beam in traveling through the contaminant. Recent unpublished results have shown that there is negligible scattering by two of the major contaminants, water and carbon dioxide, in the 10- to 12- $\mu$  wavelength region. This will be the initial wavelength region for evaluating this technique.

Many workers, including Avery, Heavens, Ruiz-Urbieta, Humphreys-Owen, Prishivalko, Beattie, Conn and Simon (Refs. 1 through 8), have made contributions to the determination of the optical constants of materials. The works of Conn, Beattie, and Prishivalko are more applicable to the present problem. The theoretical relationship for the reflecting power of a single layer coating has been adequately covered by Born and Wolf, Berning, and Caballero (Refs. 9 through 11) and permits the calculation of the contaminant thickness once the optical constants have been determined.

## 2.0 THEORY

The physical phenomenon that must be described theoretically is that of a monochromatic infrared beam reflected from a finite contaminant deposit on an optical surface. The reflection of monochromatic

radiation from such a surface is shown in Fig. 1 where  $n$  is the index of refraction,  $k$  is the attenuation index or extinction coefficient,  $h$  is the contaminant thickness,  $\theta$  is the angle of incidence,  $I_0$  is the intensity of the incident beam, ray 1 is the initial reflected beam, and rays 2, 3, . . . ,  $N$  are the result of multiple reflection from the interfaces. The reflectivity,  $R_s$ , of such a surface for a light beam polarized perpendicular to the plane of incidence can be written (Ref. 9) as follows:

$$R_s = \frac{\rho_{12}^2 + \rho_{23}^2 e^{-4V_2mh} + 2\rho_{12}\rho_{23}e^{-2V_2mh} \cos[\phi_{23} - \phi_{12} + 2\mu_2mh]}{1 + \rho_{12}^2\rho_{23}^2 e^{-4V_2mh} + 2\rho_{12}\rho_{23}e^{-2V_2mh} \cos[\phi_{12} + \phi_{23} + 2\mu_2mh]} \quad (1)$$

where  $\rho_{ij}$  is the Fresnel coefficient for the  $ij$  interface,  $m = 2\pi/\lambda$ ,  $\lambda$  is the wavelength,  $\phi_{ij}$  is the phase change at the  $ij$  interface, and  $\mu_i$  and  $V_i$  are functions of  $n_i$ ,  $K_i$ ,  $\theta$ , and  $n_1$ .

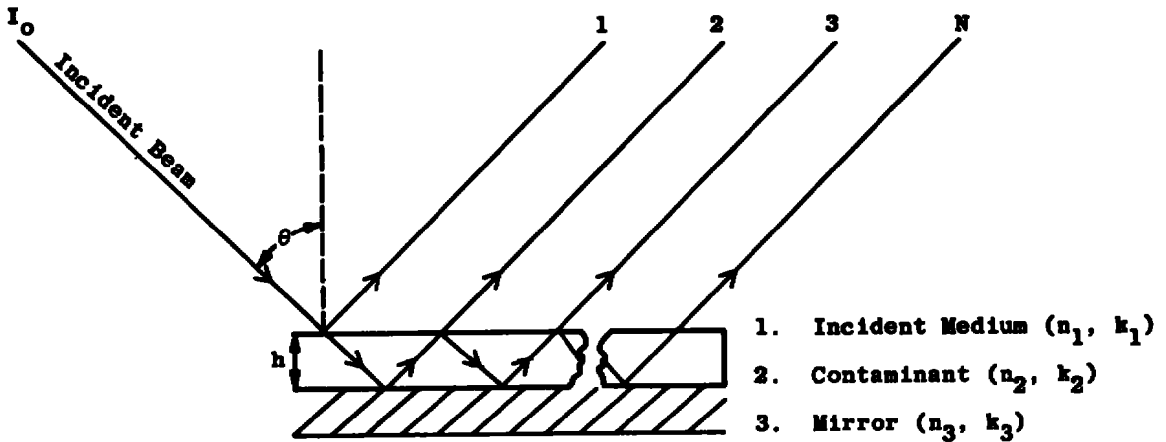


Figure 1. Reflection from finite contaminant surface.

If the following variables are introduced

$$\begin{aligned} \hat{n}_2 \cos \theta_2 &= \mu_2 + iV_2 \\ \hat{n}_3 \cos \theta_3 &= \mu_3 + iV_3 \end{aligned} \quad (2)$$

where the complex index of refractions are

$$\hat{n}_2 = n_2(1 + ik_2) \quad \hat{n}_3 = n_3(1 + ik_3) \quad (3)$$

then the Fresnel coefficient and phase change for the interfaces may be written as follows:

$$\rho_{12}^2 = \frac{(n_1 \cos \theta - \mu_2)^2 + V_2^2}{(n_1 \cos \theta + \mu_2)^2 + V_2^2}$$

$$\tan \phi_{12} = \frac{2V_2 n_1 \cos \theta}{\mu_2^2 + V_2^2 - n_1^2 \cos^2 \theta} \quad (4)$$

$$\rho_{23}^2 = \frac{(\mu_2 - V_3)^2 + (V_2 - V_3)^2}{(\mu_2 + \mu_3)^2 + (V_2 + V_3)^2}$$

$$\tan \theta_{23} = \frac{2(\mu_3 V_2 - \mu_2 V_3)}{\mu_2^2 - \mu_3^2 + V_2^2 - V_3^2} \quad (5)$$

where  $\mu_i$  and  $V_i$  are defined as follows:

$$2\mu_i^2 = a + (a^2 + b)^{1/2}$$

$$2V_i^2 = -a + (a^2 + b)^{1/2} \quad (6)$$

with

$$a = n_1^2(1 - k_i^2) - n_1^2 \sin^2 \theta$$

$$b = 4n_1^4 k_i^2 \quad (7)$$

In looking at the relationship for reflectivity, Eq. (1), it is clear that the contaminant thickness,  $h$ , may be calculated provided  $R_S$  and  $\theta$  are experimentally measured and  $n_1$ ,  $n_2$ ,  $k_2$ ,  $n_3$ ,  $k_3$ , and the wavelength,  $\lambda$ , are known. The index of refraction,  $n_1$ , of the incident medium and wavelength,  $\lambda$ , are known. Consequently, the calculation of  $h$  may be made if a means of calculating  $n_2$ ,  $k_2$ ,  $n_3$ , and  $k_3$  can be devised.

The optical constants,  $n_3$ ,  $k_3$ ,  $n_2$  and  $k_2$ , can be determined from techniques developed by various researchers (Refs. 1, 2, 4, 5, 6, 7, and 8) for absorbing substances. The constants can be calculated from experimental measurement of  $P$  and  $\Delta$  where  $P$  is the ratio of the

reflection coefficients,  $R_p$  and  $R_s$ , of the component of the reflected radiation parallel,  $p$ , and perpendicular,  $s$ , to the plane of incidence and  $\Delta$  is their relative phase difference. Functions of  $n^2 - K^2$  and  $2nK$ , the real and imaginary part of the dielectric constant, have been formulated in terms of  $P$  and  $\Delta$ . They are as follows:

$$\frac{n^2 - K^2 - \sin^2 \theta}{\tan^2 \theta \sin^2 \theta} = \frac{1 + P^2 + 2P \cos \Delta}{1 + P^2 - 2P \cos \Delta} \times \frac{(1 - P^2)^2 - (2P \sin \Delta)^2}{(1 - P^2)^2 + (2P \sin \Delta)^2} \quad (8)$$

$$\frac{-2nK}{\tan^2 \theta \sin^2 \theta} = \frac{1 + P^2 + 2P \cos \Delta}{1 + P^2 - 2P \cos \Delta} \times \frac{(4P \sin \Delta)(1 - P^2)}{(1 - P^2)^2 + (2P \sin \Delta)^2} \quad (9)$$

where  $n$  and  $K$  ( $K = nk$ ) are the optical constants of the contaminant or mirror and  $P$  and  $\Delta$  are as previously defined. From Eqs. (8) and (9)  $n$  and  $K$  are determined. In applying these equations it must be remembered that they were developed assuming a homogeneous thick film. A thick film can be defined as one having the thickness,  $h$ , and the index of absorption,  $k$ , such that multiple reflections (Fig. 1) do not occur. Consequently all measurement of reflected light involves only the initial reflection (ray 1, Fig. 1) from the principal surface. Under these conditions  $R_s \cong \rho_{12}^2$ , Eq. (1). This limits the flexibility of this technique for under these conditions  $R_s$  is not a function of the thickness of the contaminant. Consequently, a simultaneous determination of the thickness and the optical constants on the same contaminant film is not possible by this technique. The application of this technique will require a calibration run in which the optical constants of the mirror and a thick contaminant film are independently determined by Eqs. (8) and (9) so that Eq. (1) may be applied under experimental conditions to calculate the thickness.

### 3.0 EXPERIMENTAL APPARATUS

The experimental apparatus can be subdivided into the following areas: (1) research chamber, (2) infrared optics, and (3) the contaminant deposition system.

#### 3.1 RESEARCH CHAMBER

The research chamber (Fig. 2) is a cylindrical vacuum chamber of stainless steel construction, 12-3/4 in. in diameter and 15 in. in

length. The pumping system for the chamber consists of an LN<sub>2</sub>-trapped, 6-in. oil diffusion pump and a 13-ft<sup>3</sup>/min mechanical pump. The vacuum seals are standard rubber O-rings.

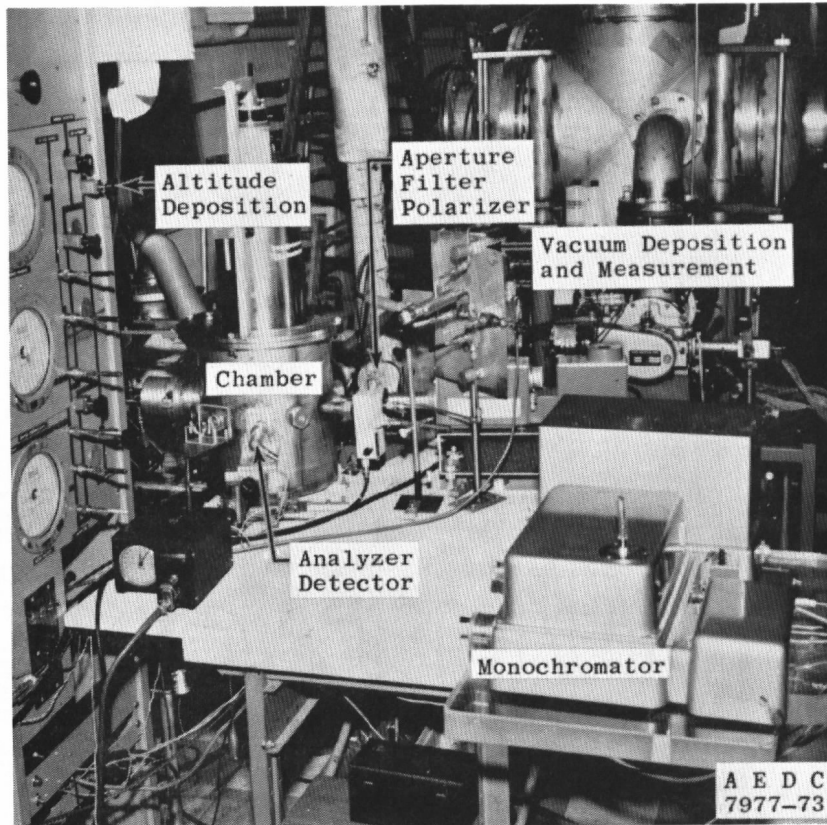


Figure 2. Infrared contaminant diagnostic research chamber.

The top of the chamber may be removed and contains a cylindrical insert to accept a test mirror holder. The holder rests on a bearing so that it may rotate freely. The holder position can be read by an indicator to one minute of arc. The holder is a cylindrical vacuum insulated dewar. An uninsulated 2-in.-diam tube extends from the bottom of the dewar into the test chamber and serves as a mount for the test mirror.

The test mirror is made of 2-in.-diam copper. The central section of the mirror, 1-3/8 in. in diameter and 3/16 in. in thickness, is gold coated and forms the reflecting surface of the mirror. The mirror area outside this central portion is 1/16 in. thick and is used for fastening the mirror to the bottom of the dewar.

There are 10 ports around the circumference of the chamber. The centers of these ports are in the horizontal plane normal to the center of the test mirror surface. Eight of these ports are designed to accept KBr windows. The two remaining ports are 4 in. in diameter. One of these connects the chamber to the pumping system, and the other accepts the altitude water vapor injector. A 3/8-in. copper tube is installed into the chamber so that it is normal to the center of the test mirror surface. This tube is connected to a controllable source of carbon dioxide or water.

### 3.2 INFRARED OPTICS

The infrared radiation is produced by a filament heated to approximately 1000°C. The radiation from this source (Fig. 3) is chopped at 13 cps and collimated. This beam is transmitted to the chamber through an aperture wheel (3-, 5-, 7-, 10-, 16-, and 24-mm-diam apertures), filter (11.6  $\mu\text{m}$  with bandwidth  $\cong 1 \mu\text{m}$ ), and polarizer. The azimuth of the polarizer can be set to 0.1 deg. The polarized beam passes through a KBr chamber window at normal incidence and is reflected from the test mirror in the center of the chamber. The reflected beam leaves the chamber at normal incidence through another KBr window. This beam passes through an analyzer to either a detector or monochromator. The analyzer is graduated in 5-deg intervals. This is adequate since in a symmetrical experiment such as this the polarizer may be used as the analyzer. The analyzer will remain in a fixed position.

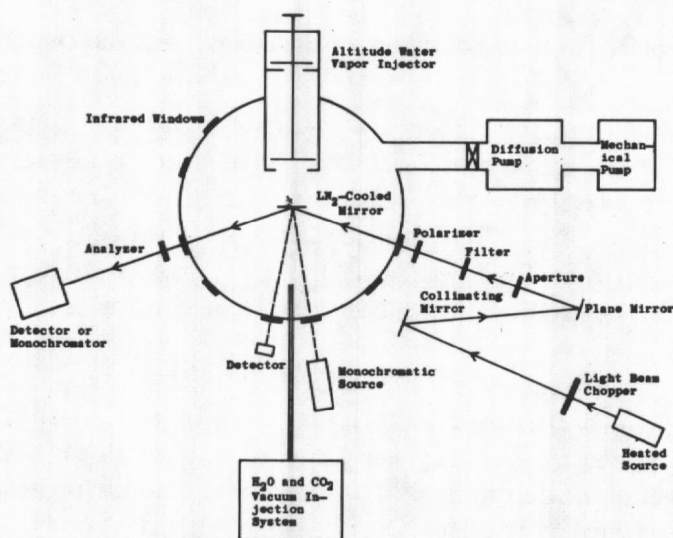


Figure 3. Infrared contaminant diagnostic optical system.

The signal from the detector or monochromator is amplified by a synchronous amplifier and displayed on a strip chart recorder.

This system was designed to operate using a 11.6- $\mu$ m filter and detector, or by eliminating the filter and detector and substituting a monochromator. The filter and detector mode would provide a larger experimental signal; consequently, they would be the initial method of operation since the reflected signal from a thick contaminant layer is expected to be extremely small. Measurements would be made to determine the optical constants at various wavelengths if the initial evaluation experiment indicates that there is sufficient signal available.

### 3.3 CONTAMINANT DEPOSITION SYSTEM

The primary means of depositing a water or carbon dioxide contaminant on an LN<sub>2</sub>-cooled mirror is by vacuum deposition. Also available for water is altitude deposition, in which the water is deposited at a finite pressure or altitude. The basic difference in these techniques is in the structure of the deposit. Vacuum deposition produces an amorphous, dense frost while the altitude deposition produces a crystalline porous frost. Thus, the optical constants and thickness measurement technique must be evaluated for water frosts of highly different structure.

#### 3.3.1 Vacuum Deposition

The supply tank for this system (Figs. 2 and 4) is of stainless steel construction, 6 in. in both diameter and length. The tank is maintained at a constant temperature by wrap-around heaters controlled by a tank thermocouple and associated electronics. The rest of the system is maintained slightly above this temperature to prevent condensation when water is the contaminant. Water vapor is obtained by filling this tank to a depth of about 2 in. with distilled water. Carbon dioxide is supplied to this tank from a regulated supply bottle.

The gas or vapor flow goes from the supply tank to a fine control valve. A flow variation of approximately 500 can be achieved with this valve. The gas flow passes through the control valve and is diverted by system valving to an LN<sub>2</sub> cold trap and mechanical pump until the flow is stabilized. The gas flow is diverted back to the chamber after a steady flow has been achieved. A 3/8-in.-diam copper tube extends into the chamber and directs the flow to the LN<sub>2</sub>-cooled mirror where it condenses.

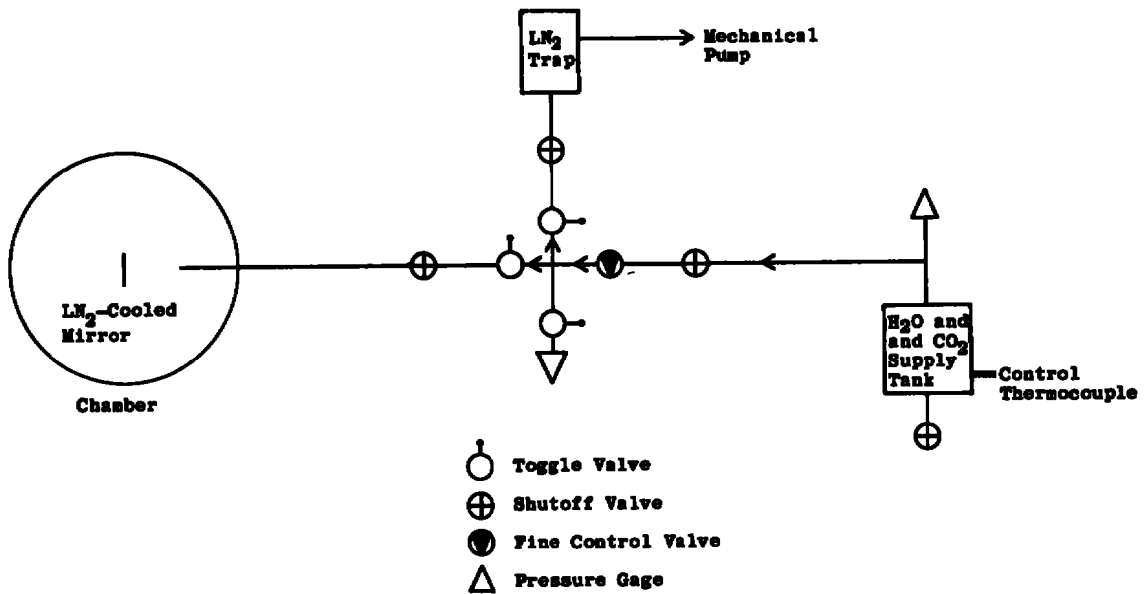


Figure 4. Schematic of water and carbon dioxide injection system.

The purpose of the deposition systems is to put on the mirror a contaminant layer of known thickness so that the thickness technique may be verified. The deposition system is not calibrated; consequently, the thickness must be determined by some other method. The interference pattern between light reflected from a mirror while deposition is in progress has been used successfully by some workers (Ref. 12) to measure the thickness when the index of refraction of the contaminant is known or measured. This method has been adapted to the deposition system for use in the visible where the index of refraction for water and carbon dioxide is known. A monochromatic beam of light ( $0.589 \mu$ ) is reflected from the mirror to a detector and the interference pattern is recorded. The angle of incidence is 10 deg and the respective refractive index for water and carbon dioxide are 1.264 and 1.428. The thickness of the contaminant layer is given by the following relation

$$\text{Thickness} = h = \frac{(q - 1/2)\lambda}{\left[ 2n_2 \left( 1 - \frac{\sin^2 \theta}{n_2^2} \right)^{1/2} \right]} \quad q = 1, 2, 3, \dots \tag{10}$$

where q is the number of minima observed.

### 3.3.2 Altitude Water Deposition

The altitude water vapor injector is approximately 15 in. in length and is inserted into the test chamber through a 4-in. port (Fig. 2). For water deposition the test mirror is rotated 180 deg from its normal position and a screw mechanism translates the injector to the mirror. Internally, the injector provides a thermostatically controlled water reservoir and a constant area diffusion path from the reservoir to the test mirror. A single acting double valve creates a buffer volume between the reservoir and the chamber when closed. The reservoir, buffer volume, and chamber have provisions for independently measuring the pressure, pressurizing with nitrogen, and evacuating. The water injector has been calibrated such that a 100-sec run, with the water reservoir at 95°F and the total pressure equalized at 150 torr, will deposit 0.00003 gm/cm<sup>2</sup>. By re-establishing the calibration conditions, any number of consecutive depositions may be completed.

## 4.0 EXPERIMENTAL PROCEDURE

Working equations must be developed relating measurable quantities to the theoretical relationship for  $n$  and  $K$  (Section 2.0, Eqs. (8 and 9)) The quantities that must be determined from experimental measurements are  $P$ ; the ratio of the reflecting coefficient for radiation parallel ( $R_p$ ) and perpendicular ( $R_s$ ) to the plane of incidence,  $\Delta$ ; the phase difference between these components of radiation; and  $\theta$ , the angle of incidence. The angle of incidence is measured directly. However,  $P$  and  $\Delta$  must be calculated from measured intensities for various orientations of the polarizer and analyzer. The intensity transmitted by such a system is as follows (Ref. 13, Fig. 5):

$$I(\psi_P, \psi_A) = I_0(P^2 \cos^2 \psi_P \cos^2 \psi_A + \sin^2 \psi_P \sin^2 \psi_A + \frac{1}{2} P \sin 2\psi_P \sin 2\psi_A \cos \Delta) \quad (11)$$

where  $\psi_P$  and  $\psi_A$  are the azimuths of the polarizer and analyzer,  $I_0 = E^2 R_s^2$ , with  $E$  being the amplitude of the incident beam, and  $P$  and  $\Delta$  are as defined above. Now, from Eq. (11),

$$I_1 = I(\psi_P, \pi/2) = I_0 \sin^2 \psi_P$$

$$I_2 = I(\psi_P, 0) = I_0 P^2 \cos^2 \psi_P$$

$$I_3 = I(\pm\pi/4, \pm\pi/4) = \frac{I_0}{4} (1 + P^2 + 2P \cos \Delta) \tag{14}$$

$$I_4 = I(\pm\pi/4, \mp\pi/4) = \frac{I_0}{4} (1 + P^2 - 2 \cos \Delta) \tag{15}$$

Combining Eqs. (12) and (13) and Eqs. (14) and (15) gives the following expression for P and Δ:

$$P = \tan \psi_P \sqrt{I_2/I_1} \tag{16}$$

$$\begin{aligned} \cos \Delta &= 1/2 \left( P + \frac{1}{P} \right) \frac{I_3 - I_4}{I_3 + I_4} \\ &= 1/2 \left( \tan \psi_P \sqrt{I_2/I_1} + \frac{1}{\tan \psi_P \sqrt{I_2/I_1}} \right) \frac{I_3 - I_4}{I_3 + I_4} \end{aligned} \tag{17}$$

P and Δ are expressed in terms of experimentally measured quantities, and hence, the expression (Eqs. (8 and 9)) for n and K are functions of the experimental quantities I<sub>1</sub>, I<sub>2</sub>, I<sub>3</sub>, I<sub>4</sub>, and θ, the angle of incidence. The intensities I<sub>1</sub>, I<sub>2</sub>, I<sub>3</sub>, and I<sub>4</sub> need only be relative measurements,

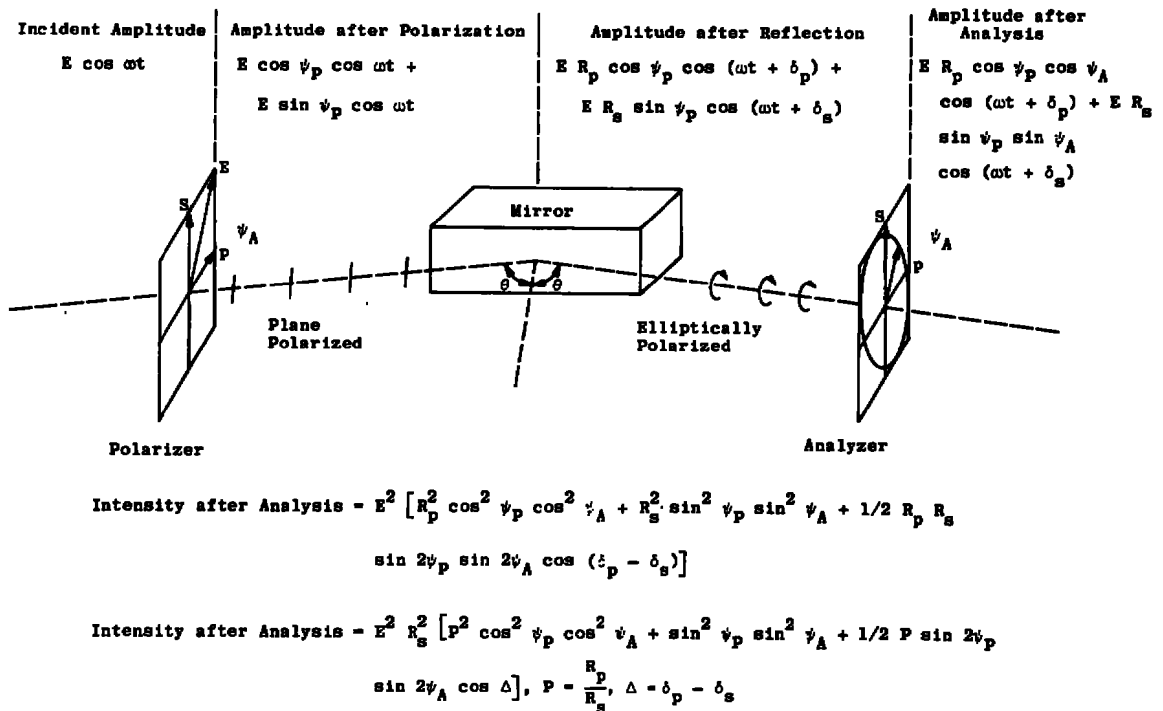


Figure 5. Analysis of reflected polarized monochromatic radiation.

provided the radiation source output is constant. This greatly simplifies the experimental procedure.

The relationship (Eq. (1), Section 2.0) for the thickness of the contaminant is a function of the angle of incidence,  $\theta$ ; the reflecting power,  $R_S$ ; index of refraction,  $n$ ; and the attenuation index,  $k$ . With the exception of  $R_S$ , all of these quantities are known from the optical constants measurements. From the system  $R_S$  may be obtained by removing the analyzer from the light path and setting the polarizer such that the light is polarized perpendicular to the plane of incidence. The measurement of  $R_S$  in this manner provides the information necessary to calculate the thickness,  $h$ , from the derived expression (Eq. (1), Section 2.0).

## 5.0 CONCLUDING REMARKS

A technique has been considered for the in situ measurements of the thickness of known contaminants on cryogenically cooled optics. This technique is an application of existing theory for the experimental determination of the optical constants of an absorbing substance to contaminants of interest and the application of these constants in conjunction with a reflectance measurement to determine the thickness of the contaminant.

An existing research chamber has been modified in order to experimentally evaluate this method with the following program:

1. Determine the optical constants of a typical metallic reflecting surface at room temperature and 77°K.
2. Determine optical constants of contaminants such as water and carbon dioxide after deposition at 77°K on a metallic reflecting surface.
3. Evaluate the thickness measurement by utilizing a water or carbon dioxide deposit of known thickness on a 77°K reflecting surface.
4. Investigate the useful wavelength region for this technique with respect to light scattering by the contaminant and the detectability of the analyzed light beam.

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### NOMENCLATURE

E	Amplitude incident beam
h	Contaminant thickness
K(k = nk)	Optical constants of the contaminant or mirror
k	Attenuation index or extinction coefficient
I <sub>0</sub>	Intensity of incident beam
$m = \frac{2\pi}{\lambda}$	Constant
n	Index of refraction
P	Ratio of the reflection coefficients, R <sub>p</sub> and R <sub>s</sub>
p	Parallel component
q	Number of minima observed
R <sub>p</sub>	Reflection coefficient for radiation parallel to the plane of incidence
R <sub>s</sub>	Reflection coefficient for radiation perpendicular to the plane of incidence
s	Perpendicular component
V <sub>i</sub>	Function of n <sub>i</sub> , K <sub>i</sub> , θ <sub>i</sub> , and n <sub>1</sub>
Δ	Relative phase difference of the reflection coefficients
δ <sub>p</sub>	Phase of parallel component of polarization
δ <sub>s</sub>	Phase of perpendicular component of polarization
θ	Angle of incidence

$\lambda$	Wavelength
$\mu_i$	Function of $n_i$ , $K_i$ , $\theta_i$ , and $n_1$
$\rho_{ij}$	Fresnel coefficient for $ij$ interface
$\phi_{ij}$	Phase change at the $ij$ interface
$\psi_A$	Azimuth of analyzer
$\psi_P$	Azimuth of polarizer