

Surface Effects of Satellite Material Outgassing Products

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The Air Force Wright Aeronautical Laboratories and the Arnold Engineering Development Center have initiated a program for measuring optical effects of satellite material outgassing products on cryo-optic surfaces. This paper presents infrared ($4000\text{--}700\text{ cm}^{-1}$) transmittance data for contaminant films condensed on a 77-K germanium window. From the transmittance data, the contaminant film refractive and absorptive indices (n, k) were derived using an analytical thin-film interference model with a nonlinear least-squares algorithm. Satellite materials studied include the adhesives DC93-500, DC6-1104, RTV566, and RTV560; Kapton® film; and S13G/LO paint. This program is continuing, and properties for other materials will be available in the future.

Introduction

AS satellite applications become more sophisticated and satellite lifetimes are extended, the roles of contamination prediction and control become increasingly important. Contamination can end a mission if cryogenically cooled optical systems become coated. A spacecraft designer must predict effects of this contamination with a very limited amount of data. The ASTM 595 test method has previously been the industry standard for screening materials. To be space-rated, a material must have a total mass loss (TML) of less than 1% and a collected volatile condensable material (CVCM) of less than 0.1%, as measured using the ASTM 595 criteria. Neither the time history nor the identification of outgassed species is available from the ASTM test.

The Air Force Wright Aeronautical Laboratories (AFWAL) are pursuing a plan to determine the outgassing properties of materials and the effects of condensed gases on critical surfaces such as thermal control and cryogenically cooled optical components. An improved test method for determining material outgassing characteristics has been developed by Lockheed.¹ This method utilizes quartz crystal microbalances (QCM's) maintained at 77 K to measure the mass loss by outgassing and time history of each of the outgassed species. This test method can be used to indicate the amount of a satellite material lost in space as a result of outgassing. However, the effect the outgassed material will have on an optical or thermal radiative surface must still be determined. The surface effects of these products are being studied at the Arnold Engineering Development Center (AEDC) under another program sponsored by AFWAL.

Infrared transmittance measurements of contaminants condensed from satellite material outgassing have begun in the AEDC 2-ft \times 3-ft chamber (Figs. 1 and 2). The materials were heated to 125°C under vacuum, and the outgassed products were frozen as thin films on a 77-K germanium window. A scanning Michelson-type interferometer was used to measure

the transmittance over the 4000- to 450- cm^{-1} wave-number range. These data were input into the TRNLIN computer program to determine the refractive and absorptive indices of the contaminants. Once determined, these n and k values can be used to calculate the transmittance or reflectance of other optical components contaminated with the outgassing products of the same satellite material and for any film thickness. Measurements were made for the adhesives DC6-1104, DC93-500, RTV566, and RTV560, for Kapton film, and for S13G/LO paint (a developmental coating for thermal control).

Material Test Sample Description and Requirements

At a joint meeting of government agencies and contractors, a list was compiled of the most often used satellite materials for testing for contamination potential. This list² is composed of adhesives, films, paints, fluids, lubricants, composites, and others and is being used as an initial guideline for materials being investigated by both Lockheed and AEDC. Sample material cure time, mixture ratio, batch number, and preconditioning time are all documented (see Table 1). The refractive index at 0.6328 μm is that of the condensed outgassing products and will be discussed later. In some instances, the cure time recommended was 7 days or longer. Tests on some of these materials occasionally were not successful owing to insufficient mass deposited on the germanium window to make reliable n, k measurements. Therefore, tradeoffs had to be made in that shorter cure times were used to provide a greater contaminant flux. Generally, the longer the material cures the lower is its outgassing rate. Between 50 and 100 g of material were needed to obtain a sufficiently large contaminant thickness for determining the optical constants. At least 10 film thicknesses (total film thickness of 2–3 μm) were required.

The adhesives were prepared by pouring them into an aluminum foil boat, which was 3 in. \times 1.5 in. \times 1.5 in. (7.62 cm \times 3.81 cm \times 3.81 cm), and allowing them to cure. The empty aluminum foil boat was outgassed at 125°C for 24 h before being filled with material. The TML for the foil boat and foil liner was found to be 0.03%. Even though this was relatively small compared to the material outgassing, this procedure was followed for all samples. All test materials were preconditioned by placing the boat and material in a 50% (\pm 5%) relative-humidity cell for 24 h prior to chamber installation. Where possible, the determined TML values were compared with those obtained using the ASTM 595 method for materials reported in Ref. 3. The ASTM 595 method requires that the material be heated at 125°C for 24 h in a vacuum and the mass loss determined by weighing the material before and after testing.

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Table 1 Summary of materials heated to 125°C under vacuum and outgassed products condensed on a 77 K germanium window

Material	Test weight, g	TML, %	Ref. index at 0.6328 μm	Cure time, h	Comment
DC93-500	66.6	0.09	1.44	46	Lot GA115893
DC6-1104	107	—	1.455	92	Lot GA016912
RTV566	95.4	0.57	1.43	44	Batch FD 239 catalyst-EA105
RTV560	78	2.0	1.42	45	Batch FD 873 catalyst-DBT
S13G/LO	53	0.16	1.34	45	Batch L-024 V-10 resin
Kapton	126	1.26	1.21	—	Obtained from Lockheed

Experimental Test Apparatus

Cryogenic Contamination Chamber

Infrared transmittance measurements were made of satellite material outgassing contamination products on cryogenic surfaces in the AEDC 2-ft \times 3-ft chamber (Fig. 1). The pumping system consisted of a turbomolecular pump with a mechanical forepump and a chamber liner cooled by liquid nitrogen (LN_2). The turbopump and the cryopanel were necessary to provide a nearly contaminant-free vacuum. Base pressures before contamination deposition were generally in the mid- 10^{-7} -Torr range.

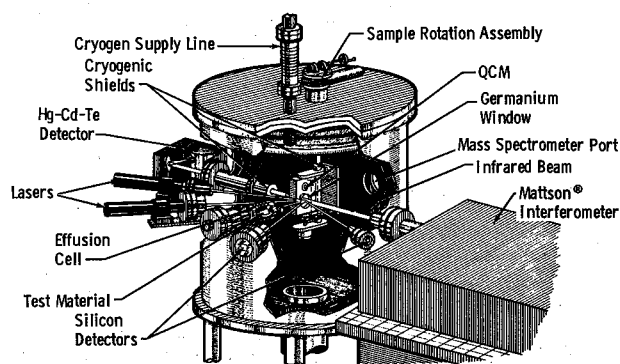
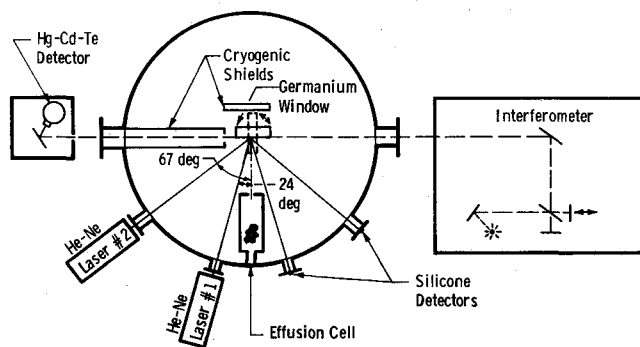
The outgassing products for contaminating the sample surface were generated using an effusion cell. It had a cylindrical aluminum body 3.5 in. (8.89 cm) long with an internal diameter of 1.75 in. (4.45 cm). The test material was placed in the closed end of the effusion cell. Heating elements covered the outside cylinder surface, and the temperature of the cell was thermostatically controlled to 125°C. The evolved gases exited through the open-end aperture, which was 1.5 in. (3.81 cm) in diameter.

The test surface was a germanium window 2.75 in. (6.99 cm) square and was 0.157 in. (4 mm) thick. It was mounted in the center of the chamber and was cooled to nearly 77 K with a constant flow of LN_2 . Germanium was picked for the deposition surface because it has good thermal conductance and a flat transmittance of 47–48% over the 700–4000- cm^{-1} (2.5–14- μm) range. The thin-film interference model on which the optical property determinations were based required that only the front surface be coated by the contaminant material. Therefore, two LN_2 -cooled baffles were strategically located near the rear germanium surface to scavenge any gases that would otherwise be incident on it. The mass flux deposited on the germanium was monitored using a quartz crystal microbalance (QCM) that was mounted adjacent to it and was also cooled with LN_2 .

A commercial Michelson-type interferometer (see Figs. 1 and 2) was used in making infrared transmittance measurements of the deposited contaminant film on the germanium window. The interferometrically modulated infrared beam was collimated and passed through a KBr window on the vacuum chamber port, through the germanium test window, through another KBr window on the opposite side of the chamber, and finally to detector optics and the Hg-Cd-Te detector. The wavenumber/wavelength range sensed was 4000–450 cm^{-1} (2–22 μm). Typically, 32 scans were coadded for both the sample and reference measurements with a resolution of 2 cm^{-1} . A reference measurement was made before each sample measurement. Transmittance data were initially stored on the system hard disk and later transferred to flexible disks.

Experimental Test Techniques

In order to make accurate n, k measurements, the transmittance must be measured for carefully determined film thick-

**Fig. 1.** Cryogenic optics degradation chamber, 2 \times 3 ft.**Fig. 2.** Schematic of 2 ft \times 3 ft chamber components.

nesses. The thin-film interference technique provides a method for calculating these discrete thicknesses. This technique has been described previously^{4,5} and will only be reviewed here. As a thin film forms on a reflecting substrate, the intensity of the reflected beam of radiation will vary sinusoidally. From thin interference equations, the maxima and minima locations can be used to calculate the film thickness accurately.² For these calculations, the film refractive index n must be known for the incident wavelength. Two He-Ne laser beams were incident on the germanium window at two angles (24.0 and 67.5 deg), and interference fringes were observed as the contaminant film was deposited. The refractive index at 0.6328 μm was determined from the interference patterns observed on a strip-chart recorder trace for the two laser-solar cell outputs. This was done by using the fringe (interference maxima or minima) counting technique described in Ref. 6. Once the refractive index was known to be at 0.6328 μm , the film thicknesses at the interference maxima and minima peaks were calculated. The refractive indices determined at 0.6328 μm for outgassing products of each material tested are shown in Table 1.

Cryogenic Contamination Chamber Test Procedures

After the test material had been preconditioned, the boat containing the sample material was inserted in the effusion cell and installed in the 2-ft \times 3-ft chamber. The centers of the germanium window and the QCM were aligned equidistant from the effusion cell centerline so that the two surfaces would see the same flux rate. After the chamber pressure had reached 10^{-5} Torr, LN₂ cooling of the chamber liner, germanium window, and QCM began. Both the QCM and germanium window reached thermal equilibrium before testing was initiated. At this point, the chamber pressure was in the mid to high 10^{-7} Torr range. The effusion cell was heated to 125°C, and the laser-solar cell outputs were observed with time on a strip-chart recorder. Outgassed components were condensed on the germanium window and the QCM. As the outgassed products condensed on the germanium window, the thin-film interference caused the laser-solar cell outputs to exhibit sinusoidally varying signals. Deposition continued until the first interference minimum (quarter wavelength) occurred. The transmittance of the germanium window with the deposited film was then measured. The QCM change in frequency with time was also recorded during deposition.

Once the transmittance measurements were completed, the germanium was rotated back into the original deposition position and the film buildup and transmittance measurements continued. This procedure was repeated for as many thicknesses as could be obtained before the deposition rate decreased to a minimal value. For some materials, films up to 25 interference maxima thick were obtained. Transmittance measurements were made for as many thicknesses as possible to maximize the accuracy of the n, k calculations. In some cases, transmittance measurements were made during warmup of the germanium window. This helped to determine the temperature where individual contaminant species were re-evaporated and to aid in their identification.²

After transmittance measurements were completed, the effusion cell heater was maintained at 125°C until a total time of 24 h had been reached. This allowed a direct comparison of TML values determined from this study with values given in Ref. 3 using the ASTM 595 method and with the values obtained with the new Lockheed technique (QCM method). The TML (percent) value was determined by dividing the mass lost due to outgassing for 24 h (at 125°C under vacuum) by the original mass.

The germanium window was observed after warmup and a transmittance spectrum was taken. Often, for tests involving silicone materials, there was a film left on the germanium. It was easily removed using alcohol, Freon,[®] or acetone. Transmittance measurements of the remaining film usually showed evidence of hydrocarbons and silicones. The QCM also had a contaminant film left on it, evidenced by the fact that the frequency was sometimes 5–10,000 Hz higher than that observed prior to the beginning of the test.

Experimental Data

The housekeeping data were monitored and stored by a computer. A typical set of data included the QCM parameters (frequency, mass, mass rate, and temperature), the laser-solar cell outputs, the effusion cell temperature, and the germanium window temperature. Typical plots of these parameters are contained in Ref. 2.

Detailed transmittance data were obtained for four adhesives (Dow Corning DC93500, Dow Corning DC6-1104, General Electric RTV560, and General Electric RTV566), for Kapton film, and for S13G/LO thermal control paint. Experimental transmittance measurements are presented in this paper for one of the adhesives, RTV566, for S13G/LO paint, and for Kapton film. Transmittance data for the other adhesives are contained in Ref. 2. The bare germanium window infrared transmittance is essentially flat over the wave number region from 4000–700 cm⁻¹ (see Ref. 2). It is nonabsorbing in this

range but transmits only about 47–48% owing to its high refractive index ($n \approx 4.0$).

RTV566 Adhesive

RTV566 is a low-volatile-content RTV compound, based on a phenyl silicone polymer, and it cures to a tough silicone rubber. It is designed to meet the ASTM standards of less than 1% TML and less than 0.1% CVCM. The material was prepared according to the manufacturer's suggested mixture ratio of 0.7% catalyst (RTV566B) to silicone base (RTV566A) by weight.

The RTV566 outgassing products were condensed on the germanium for films up to 25 interference maxima thick. The refractive index at 0.6328 μ m was determined to be 1.43. Transmittance curves are shown in Fig. 3 for film thicknesses of 0.23, 2.29, and 4.59 μ m, corresponding to 1.0, 10.0, and 20.0 interference maxima. Data are presented in Ref. 2 for the silicone base RTV566A and for the catalyst RTV566B measured at 56°C. The spectra shown in Fig. 3 most closely resemble those of the silicone base material RTV566A. The silicone absorption band locations and the molecular groups associated with them are discussed in Ref. 7.

The TML was found to be 0.57%, which was a little higher than the 0.08–0.36 values reported in Ref. 3. This was considered to be in good agreement since the cure time was only 44 h for this test as compared to seven days for the data in Ref. 3.

S13G/LO Paint

S13G/LO is a developmental coating developed for AFWAL by the Illinois Institute of Technology Research Institute. The paint was mixed using 0.6% catalyst by weight to the V-10 resin binder. After mixing, the coating was applied to seven aluminum foil strips. Transmittance data of the condensed outgassed products were obtained for 14 film thicknesses up through the twelfth interference maxima. The results are shown in Fig. 4 for film thicknesses of 0.25, 1.78, and 2.96 μ m (corresponding to 1.0, 7.0, and 12.0 maxima). Only for the thickest film was there evidence of anything other than H₂O or CO₂. Transmittance spectra for the individual paint binder and catalyst are contained in Ref. 2. No evidence of the catalyst was seen in the spectra of Fig. 4. Therefore, essentially all of the outgassing came from the S13G/LO base. This was not unexpected because the ratio of catalyst to base was very small. A TML of 0.16% was obtained after test completion, which is considerably less than the values of 0.4–0.6 given in Ref. 3. The refractive index at 0.6328 μ m was 1.34.

Kapton

A roll of 5-mil Kapton film weighing 126 g was used as test material. Transmittance measurements for 15 thicknesses were

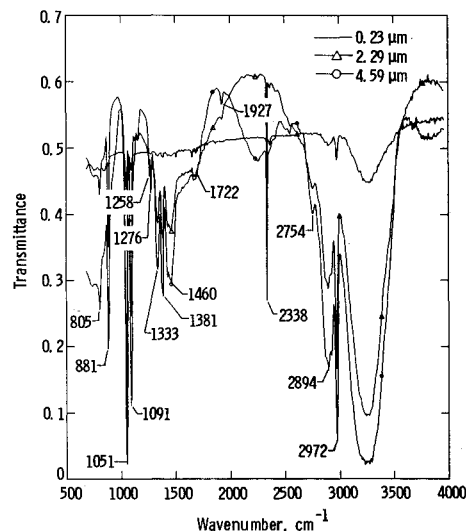


Fig. 3. Transmittance of condensed RTV566 components, 0.23, 2.29, and 4.59 μ m thick.

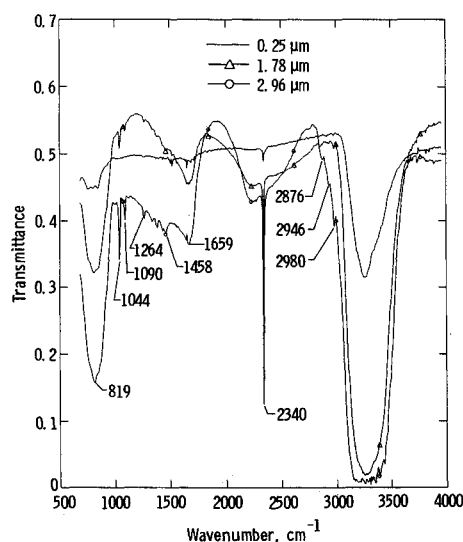


Fig. 4. Transmittance of condensed S13G/LO components, 0.25, 1.78, and 2.96 μm thick.

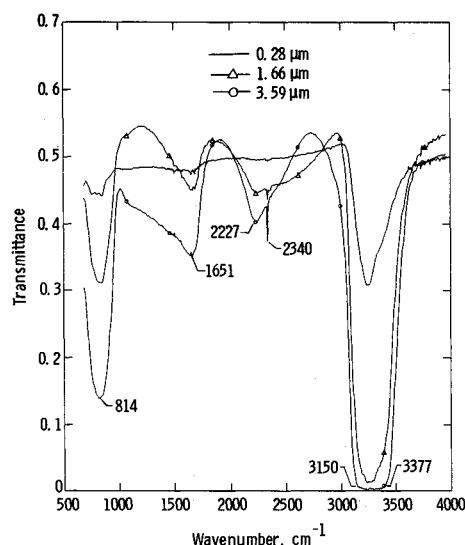


Fig. 5. Transmittance of condensed Kapton components, 0.28, 1.66, and 3.59 μm thick.

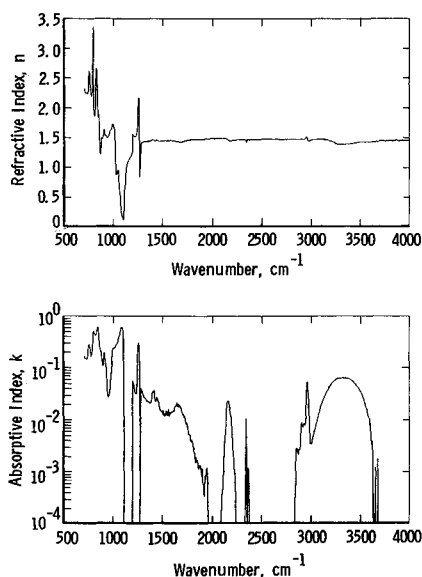


Fig. 6. Refractive and absorptive indices for DC93-500 outgassing products.

made for films up to 13 interference maxima thick. Transmittance spectra for the condensed outgassing products on the germanium window are presented in Fig. 5 for film thicknesses of 0.28, 1.66, and 3.59 μm . H_2O was the only major identifiable component. Again, CO_2 shows up as only a trace. Although the deposit appears to be predominantly H_2O , the refractive index at 0.6328 μm was found to be 1.21, which was considerably lower than for pure H_2O films (~ 1.31). The reason for the low refractive index at 0.6328 μm is currently unknown. The outgassed products condensed and measured (Fig. 5) show little resemblance to the pure Kapton spectra (see Ref. 2). After testing, the sample was removed and weighed. The TML was found to be 1.26% as compared to the 0.77 value reported in Ref. 3.

Optical Properties Determination

Most contaminant problems encountered in space involve contaminant thicknesses of a few micrometers or less. Therefore, thin-film interference equations are generally used to predict contaminant effects on the reflectance or transmittance of an optical element. These calculations require knowledge of the optical properties of the contaminant film — the refractive and absorptive indices n and k . These are components of the more general expression for the complex refractive index given by $n^* = n - ik$. In order to determine the complex refractive index of the thin solid contaminant films, an analytical model called TRNLIN has been developed.^{4,5,8,9} The model uses the expressions given in Ref. 10 for thin-film transmittance and reflectance. The model assumes that the germanium window is a thick film and that all interference occurs within the thin contaminant film. The expressions derived were for the normal transmittance of a thin film on a nonabsorbing substrate, which accurately represents the experimental conditions under which the transmittance measurements were made.

The optical constants of the contaminant films were determined using the experimentally determined transmittance values over the 4000- to 700-wave number range and for as many film thicknesses as possible. These were input into the analytical transmittance model TRNLIN, which uses a nonlinear least-squares convergence routine for determining n and k . The transmittance experimental data were taken using 2-cm^{-1} resolution. The n and k were then determined at every 2 cm^{-1} in the range from 700 to 4000 cm^{-1} . Tabulated n, k data, obtained for one of the materials investigated, RTV566, are given in Ref. 2. The refractive and absorptive indices determined from the transmittance data are shown in Figs. 6–11 for DC93-500, DC6-1104, RTV566, RTV560, S13G/LO, and Kapton, respectively. The refractive indices are shown at the top with the absorptive indices directly below.

The standard deviations for each wave number were calculated as part of the TRNLIN program. They generally varied with wave number but, for the most part, were on the order of 0.01 for the refractive index and 0.001 for the absorptive index. The real test was to determine how the calculated values agreed with the original data. This will be shown in a later section, where the experimental transmittance is compared with that calculated using n and k determined analytically for RTV566 material.

Tabulated n, k data for all of the materials discussed will eventually be included in an AFWAL data base. They are currently available on request to the authors.

Transmittance and Reflectance Calculations Using CALCRT

To realize the maximum utility of the n, k data generated from the experimental and analytical studies, a computer program, CALCRT, was developed. CALCRT, written in FORTRAN IV, calculates the reflectances and transmittances for a radiation beam that strikes a film and substrate system that has planar interfaces, ideally, infinite in extent. The film and substrate are sandwiched between semi-infinite vacuums, and the

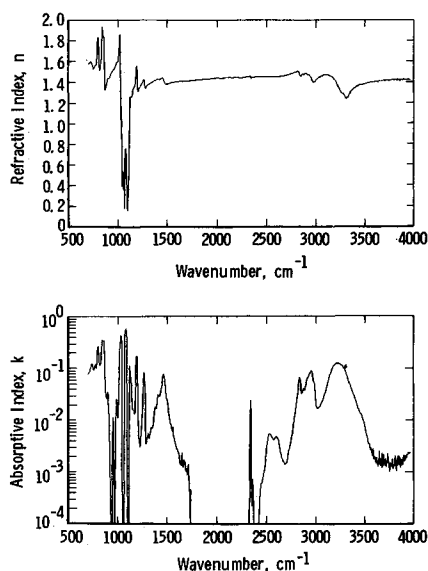


Fig. 7. Refractive and absorptive indices for DC6-1104 outgassing products.

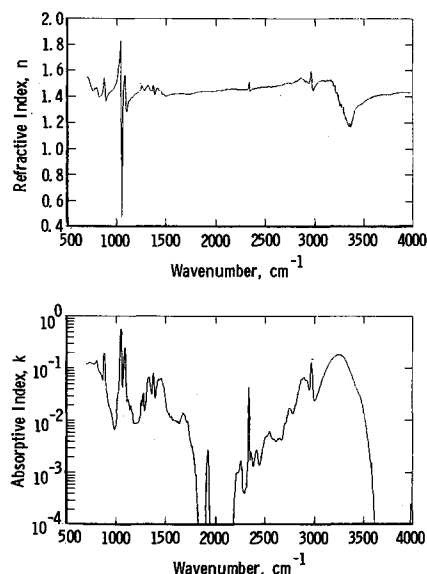


Fig. 8. Refractive and absorptive indices for RTV566 outgassing products.

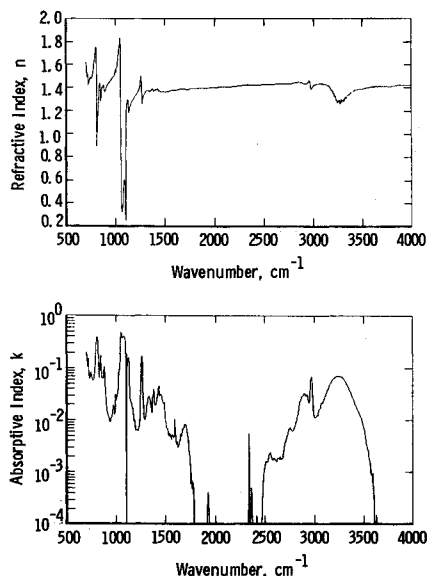


Fig. 9. Refractive and absorptive indices for RTV560 outgassing products.

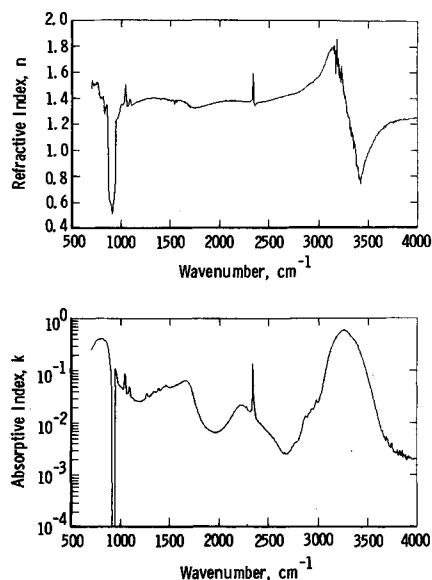


Fig. 10. Refractive and absorptive indices for S13G/LO outgassing products.

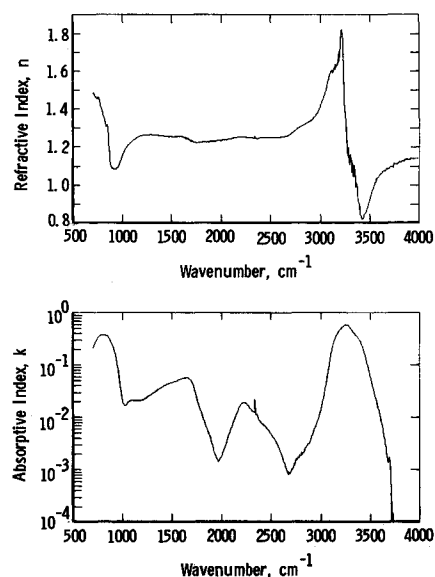


Fig. 11. Refractive and absorptive indices for Kapton outgassing products.

refractive indices on each side of the film/substrate are identically equal to one.

The user must supply the optical constants and thicknesses of the film and substrate, as well as the incidence angle of the beam. Also, the user must indicate which beam coherence case applies and must choose either 1) an output format that gives the reflectances and transmittances as functions of wave number or wavelength at a constant film thickness or 2) an output format that displays the transmittances and reflectances vs film thickness at a constant wave number or wavelength.

CALCART Transmittance Calculations

To show how CALCART can be used, examples of transmittance vs wave number and transmittance vs thickness were calculated for RTV566 material, using the n and k previously determined. Figure 12 shows curves of transmittance vs wave number for: 1) the actual experimental data obtained for condensed RTV566 outgassing products 4.59 μm thick on germanium, 2) the same film thickness but calculated using the n, k values determined from TRNLIN, and 3) the same film thickness using n and k that were determined using a subtractive Kramers-Kronig technique.² As shown in Fig. 12, there is ex-

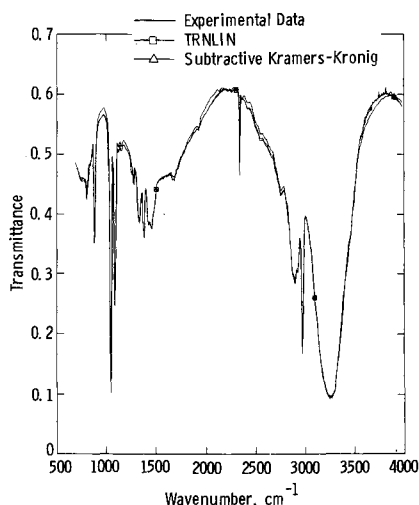


Fig. 12. Transmittance vs wave number for experimental and CALCRT calculations for a 2.30 μm -thick RTV566 contaminant film.

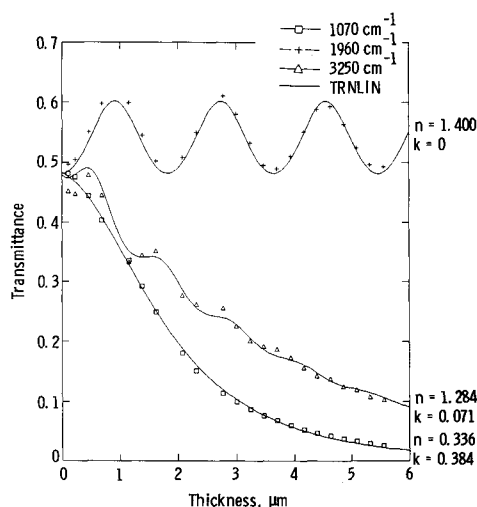


Fig. 13. Comparison of calculated and experimental transmittance values vs film thickness for RTV566 contaminant films for 1070, 1960, and 3250 cm^{-1} .

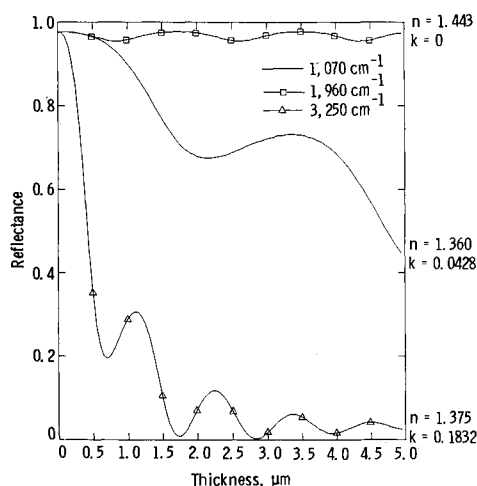


Fig. 14. Calculated mirror reflectance vs RTV566 film thickness for 1070, 1960, and 3250 cm^{-1} ; mirror optical constants are $n^* = 1.8 + 18i$.

cellent agreement between the experimental and calculated curves. Differences of about 2% were the largest seen.

CALCRT can also be used to calculate the transmittance dependence on film thickness. An example is shown in Fig. 13 for RTV566 contaminant films condensed on 77-K germanium. These represent no absorption ($n = 1.44, k = 0.00$) for 1960 cm^{-1} , strong absorption ($n = 1.375, k = 0.186$) for 3250 cm^{-1} , and intermediate absorption ($n = 1.36, k = 0.0428$) for 1070 cm^{-1} . The solid curves for each of the three show the analytical results calculated using the TRNLIN-derived n and k . The data points are for the actual experimental values. As can be seen in Fig. 13, the analytical and experimental results agree quite well.

CALCRT Reflectance Calculations

Figure 14 shows calculated reflectance vs thickness curves using CALCRT that are based on the TRNLIN-determined n and k for RTV566 contaminant films condensed near 77 K. For these calculations, the substrate is assumed to be a specular metal surface, such as a gold mirror, with a complex refractive index of $n^* = 1.8 + 18.0i$ and independent of wavelength/wave number. This yields a bare surface reflectance of 97.8% for all wavelengths/wave numbers. The symbols on the curves in Fig. 14 do not represent experimental data points but are included to facilitate curve identification. The reflectance vs film thickness curves in Fig. 14 are for the three wave numbers 1070, 1960, and 3250 cm^{-1} . Thin-film interference is vividly displayed for all three wave numbers. These curves give a good indication of how a large decrease in a mirror's reflectance may be seen for only a thin contaminant film. CALCRT was also used to calculate reflectance vs wave number for various film thicknesses. These results are contained in Ref. 2.

The important point that should be re-emphasized about the n, k data and their use is that once the contaminant n and k have been determined, transmittances and reflectances can be calculated for any desired film thickness, incidence angle, or substrate, provided the substrate refractive index is known.

Summary

The Air Force Wright Aeronautical Laboratories and the Arnold Engineering Development Center have initiated a program for measuring contaminant surface effects of satellite material outgassing products on cryo-optic surfaces. The complex refractive index components, n and k , of thin contaminant films condensed on a cryogenic surface were determined from experimental infrared transmittance measurements for the wave number range 4000–700 cm^{-1} . The materials studied were heated to 125°C and were condensed on a 77-K germanium window. The n and k were determined using a thin-film interference analytical model and a nonlinear least-squares algorithm. Materials investigated included the adhesives RTV566, DC6-1104, RTV560, and DC93-500; S13G-LO thermal control paint; and Kapton film.

A computer program, CALCRT, has been written that calculates the transmittance and reflectance values for the following parameters: substrate refractive index and thickness, contaminant refractive index and film thickness, incidence angle, wave number, and wavelength. This provides a potential optical property user with the program for utilizing the n and k generated for the materials mentioned previously. This program and tabulated n, k data for the satellite materials previously discussed may be obtained by contacting the authors.

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