EFFECTS OF DIFFUSION PUMP OIL CONTAMINATION ON THE REFLECTANCE CHARACTERISTICS OF VARIOUS SURFACES

C. E. Pinion
ARO, Inc.

July 1966

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ARO, Inc.
FOREWORD

The research presented in this report was sponsored by the Arnold Engineering Development Center (AEDC), Air Force Systems Command (AFSC), Arnold Air Force Station, Tennessee, under Program Element 65402234.

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This technical report has been reviewed and is approved.

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ABSTRACT

During thermal testing in a space simulation chamber, the extent of diffusion pump oil contamination on all important surfaces should be determined. This information and an understanding of the reflectance characteristics of the oil contaminated surfaces would contribute to a more reliable interpretation of the thermal test data. Hence, an experimental study was made of the effects of diffusion pump oil contamination on the reflectance and radiation distribution of four types of surfaces found in space simulation chambers. The total reflectance and angular distribution measurements were made in the radiation wavelength range from 0.4 to 1.2 μ. The spectral reflectance measurements were made in the radiation wavelength range from 0.35 to 10 μ. These data were obtained as a function of oil film thickness, angle of incidence, wavelength, and substrate material. Contamination of surfaces caused by diffusion pump oil backstreaming alters the reflectance characteristics of the substrate-oil film combinations.
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NOMENCLATURE

Δλ Wavelength bandwidth
θ Angle of reflection (see Fig. 5)
μ $1 \times 10^{-3}$ mm
φ Angle of reflection (see Fig. 5)
ψ Angle of incident radiation (see Fig. 5)
SECTION I
INTRODUCTION

Contamination of the interior surfaces of space environmental chambers by oil backstreaming from diffusion pumps may have a significant effect on the reflectance and distribution of radiation reflected from these surfaces. Changes in these reflective characteristics during thermal testing could introduce errors in heat-transfer parameters which would change the heat balance of the system.

A study was made to determine the effects of Dow Corning (DC) 705® diffusion pump oil contamination on the reflectance and distribution of radiation reflected from surfaces found in a space simulation chamber. The surfaces investigated were copper, front surface aluminized mirror, stainless steel, and Cat-a-lac black paint. Data were obtained on (1) total reflectance in the 0.4- to 1.2-μm wavelength range for angles of incidence from 10 to 50 deg at 10-deg intervals, (2) angular distribution for total radiation in the 0.4- to 1.2-μm wavelength range at angles of incidence of 10 and 40 deg, (3) spectral angular distribution in the 0.7- to 1.2-μm wavelength range at angles of incidence of 10 and 40 deg, and (4) spectral reflectance in the 0.35- to 10-μm wavelength range.

McCullough et al. (Ref. 1) recently reported a deviation from the fundamental reflection law when the wavelength of the incident radiation is comparable to the root-mean-square (rms) roughness of the reflecting surface. This phenomenon was of particular interest in this study because of its unusual nature and effect on thermal testing.

SECTION II
APPARATUS

2.1 7-IN. INTEGRATING SPHERE

The system used to make total reflectance measurements consisted of two 7-in.-diam, flanged, cast aluminum hemispheres joined together and coated inside with magnesium oxide (MgO) (Fig. 1). It was used to obtain data at atmospheric pressure only.

The test samples were mounted in the center of the sphere on the end of a small-diameter polished steel rod. The sample could be rotated from 0 to 360 deg from the outside, and the control was graduated in 2-deg intervals so that the angle of incidence could be adjusted to any desired value.
A tungsten-iodine light source (Fig. 1) was mounted on a cylindrical collimating system attached to the top of the sphere. The upper end of the cylinder contained a slit through which the radiation passed, and collimating plates were located at 3/4-in. intervals along the length of the cylinder. The interior of the cylinder and collimating plates were smoked with acetylene black to minimize stray radiation. The cylinder was connected to the sphere by a bellows so that it could be tilted to irradiate the wall of the sphere directly.

A silicon solar cell, having a response range from 0.4 to 1.2 μ, was used as the detector for the system. It was mounted on the wall of the sphere directly opposite the sample rotating knob and on a diameter 90 deg from the light beam entrance to the sphere.

2.2 30-IN. DISTRIBUTION SPHERE

Angular radiation distribution measurements were made with the 30-in. distribution sphere (Fig. 2), which was constructed from two 30-in.-diam stainless steel hemispheres joined with an O-ring seal and coated with Parsons flat black paint on the interior wall to minimize multiple reflections. The sphere was evacuated with a 6-in. oil diffusion pump equipped with a water-cooled cold cap and a LN2-cooled cold trap. The diffusion pump was backed with a 400-l/min mechanical pump.

The sample to be investigated was mounted at the center of the sphere at the end of a long rod, the other end of which was fastened to a rotatable flange on top of the sphere (Fig. 2). This arrangement made it possible to adjust the angle of incidence of radiation to the desired values.

The light source for the sphere was a 1.6-kw xenon short-arc lamp mounted in a lamp house (Fig. 2). The radiation from the lamp passed through a condensing lens system and a collimating tube.

Silicon solar cells, having a response range from 0.4 to 1.2 μ, were used as the radiation detectors. These were mounted at 10-deg intervals on an arm which could be rotated in a 300-deg arc, having a 10-in. radius of curvature centered on the sample.

2.3 INFRARED INTEGRATING SPHERE SYSTEM

The sphere used to measure spectral reflectance in the 1- to 10-μ range consisted of two 7-in.-diam flanged stainless steel hemispheres,
joined together and coated on the inside with pressed flowers of sulfur (Fig. 3). This system was operated at atmospheric pressure only.

The sample was mounted on the end of a support rod at the center of the sphere and rotated by using a knob mounted on the exterior of the sphere. The sample was irradiated by multiple reflections from the hemisphere facing the sample. The radiation source was a Globar® heater, and the sphere was irradiated through a port opposite the sample rotating knob.

Reflected radiation from the sample was viewed by the transfer optics and focused on the slit of a double pass monochromator employing a short response time thermocouple detector.

2.4 RATIO RECORDING SPECTROPHOTOMETER

A ratio recording spectrophotometer (Fig. 4) was used to measure the spectral reflectance of the samples in the 0.35- to 2.7-μ wavelength range at atmospheric pressure.

It consists of an integrating sphere coated with MgO on the interior, monochromator, tungsten light source, two detectors, and a recorder. A photomultiplier detector was used for the wavelength range from 0.35 to 0.7 μ and the lead sulfide detector for the wavelength range from 0.7 to 2.7 μ. Radiation from the tungsten lamp passed through the monochromator and into the integrating sphere through two ports in the side of the sphere. The sample holders were directly opposite the entrance ports. The samples were irradiated directly with the monochromatic radiation which is incident 5 deg from the normal to the sample surfaces. On top of the sphere and 90 deg from the entrance ports and sample holders, either a lead sulfide or photomultiplier detector was located. The reflected radiation was sensed by the appropriate detector, and the signal from the detector was amplified and recorded using the MgO sample as a standard.

SECTION III
PROCEDURE

Four 1- by 1.5-in. substrates - Cat-a-lac black paint, copper, aluminized mirror, and stainless steel - were tested. The samples were used as received from the supplier, and their roughness was typical of these materials as found in a space simulation chamber.
The samples were marked to ensure proper orientation in the integrating sphere sample holder each time reflectance measurements were made. Initially the total reflectance measurements on the copper sample were made in the wavelength range between 0.4 and 1.2 μ. The sample was cleaned with methylene chloride (CH₂Cl₂) and then placed in the 7-in. integrating sphere so the angle of incidence of the radiation measured from the normal was 0 deg. The light source and collimating system were positioned to irradiate the copper. The detector output caused by light reflected from the copper was measured at angles of incidence from 0 to 50 deg at 10-deg intervals. Then the light source and collimating system were tilted so that the light was incident on the wall of the sphere in order to obtain a reference measurement. The sample was in the 0-deg position during the reference measurements. From these two measurements, the absolute reflectance of the sample could be calculated (Ref. 2). The reflectance measurements were then repeated for the copper over the same angles of incidence.

The copper sample was removed from the integrating sphere, weighed, and the diffusion pump oil was applied with an eye dropper. The oil was spread over the sample as uniformly as possible, and excess oil was removed. The sample was weighed, placed in the integrating sphere, and total reflectance measurements were made as previously described for the cleaned, bare surface. The sample was removed from the sphere, weighed, and thoroughly washed in spectral grade CH₂Cl₂ to remove the oil.

The diffusion pump oil film thickness was determined by two different methods: The gravimetric method, whereby the sample was weighed before and after application of the oil, and an ultraviolet absorbance method. In the case of the ultraviolet absorbance method, the weight of oil on the substrate was determined by measuring the concentration of oil in a known quantity of CH₂Cl₂ in which the oil contaminated substrate had been washed. The absorbance at 258 mμ of the diffusion pump oil solution in CH₂Cl₂ was measured using a spectrophotometer with a hydrogen light source. Using a calibration curve of absorbance versus concentration, the concentration of oil in the solution was determined and from this the weight of oil on the sample (Ref. 3). Then, for both methods, since the precise dimensions of the sample and the weight and density of the oil were known, the oil film thickness on the substrate could be calculated.

However, since the calculation of the film thicknesses involved dividing the weight of the oil on the substrate by the area of the substrate, the error in the film thickness was impossible to estimate because the actual surface area of any particular substrate may be much larger than
the apparent geometric area because of surface roughness. Also, what may seem to be a thin film may not cover the surface uniformly and completely since all or part of the oil could be in the crevasses of the surface, and this would lead to erroneous film thickness calculations. The effect of surface roughness on film thickness accuracy diminishes with increasing film thickness. Even though an absolute film thickness measurement may be in error because of surface roughness, the errors in the measurements of that thickness by both thickness measurement techniques will be comparable permitting a comparison of the two techniques, since the error in the calculation of the quantity of oil was estimated to be the same for both techniques, about ±3 percent. Consequently, the term "oil film thickness" would be more exactly called "apparent film thickness" since no oil in the surface crevasses, complete coverage of the surface, and uniform thickness of the film were assumed.

For rough surfaces and very thin films when oil may exist in the crevasses of the surface only, the reflectance characteristics of a surface may be largely dependent on the relative exposed area of each type of exposed surface. In other words, part of the surface may have little or no oil on it, whereas part may have a thick film of oil and the reflectance of the entire surface will be a combination of the reflectances of the two surface conditions.

The procedures used to measure total reflectances for oil films on an aluminized mirror and stainless steel were the same as those for copper, and only the ultraviolet absorbance method was used to measure film thickness. To deposit very thin films, the oil was mixed with spectral grade CH₂Cl₂, the sample was dipped in this solution, and then the CH₂Cl₂ was allowed to evaporate, leaving an oil film.

Procedures used to make the total reflectance measurements from the Cat-a-lac black surface were the same as for the copper sample, but only the gravimetric method was used to determine oil film thickness since the CH₂Cl₂ dissolved the Cat-a-lac black paint.

These procedures were repeated until a sufficient number of data points could be obtained to define curves of total reflectance versus oil film thickness, and percent changes from initial values of reflectance were calculated from these data.

In making distribution and spectral reflectance measurements, the samples were first washed in reagent grade CH₂Cl₂ and coated with oil. After the measurements were made, the sample was removed from the sphere, washed in spectral grade CH₂Cl₂, and the concentration of oil in the solution and film thickness was determined as described above.
Angular distribution measurements were made in the 30-in. distribution sphere at atmospheric pressure as a function of substrate material, angle of incidence of the radiation, and wavelength. The angles of incidence (ψ) selected for investigation were 10 and 40 deg, and the angles of reflection were θ and φ (Fig. 5). The angle of reflection (θ) was scanned from 0 to 90 deg. The distribution measurements were also made in a vacuum in the tests of the aluminized mirror so that the effect of vacuum conditions on the angular distribution could be determined. The distribution data were taken for total radiation in the 0.4- to 1.2-μ wavelength range, and spectral distribution data were taken in the 0.7- to 1.2-μ range. The filters used to obtain the spectral distribution data transmitted a band of wavelengths approximately 0.2 μ wide.

Spectral reflectance measurements were made in a ratio recording spectrophotometer for the 0.35- to 2.7-μ wavelength range and in the 7-in. infrared integrating sphere system for the 1- to 10-μ wavelength range. The samples were cleaned in CH₂Cl₂ with the exception of the Cat-a-lac black surface. Cleaned samples were placed in the sample holder of the 7-in. infrared sphere, the Globar heater power was adjusted, and the desired wavelength selected. The sample was positioned so that the radiation reflected at 45 deg was measured, and the sphere wall was then viewed with the monochromator and the reading recorded. The reflectance was calculated using these two values. This was repeated for each desired wavelength in the 1- to 10-μ wavelength range. For spectral reflectance measurements in the 0.35- to 2.7-μ wavelength range on the ratio recording spectrophotometer, a scan to establish the zero reflectance line was made by closing the radiation entrance slit shutter. The 100-percent reflectance line was established by recording a ratio of the reflectances of two identical MgO samples placed in the sample ports of the integrating sphere portion of the spectrophotometer. The reflectance of the test sample was then measured, with magnesium oxide used as the standard.

Spectral reflectance measurements were made for each of the four bare samples and one oil film thickness on each sample.

SECTION IV
RESULTS AND DISCUSSION

4.1 INTRODUCTION

In some instances, the observed reflectance characteristics may have been caused by interactions taking place as a result of wavelength,
surface roughness, and oil film thickness being of comparable dimensions. However, since this work was primarily of an experimental nature no attempt will be made to give a theoretical explanation of the observed phenomena.

4.2 TOTAL REFLECTANCE

Curves of total reflectance versus oil film thickness for all the substrates at 10- and 40-deg angles of incidence are shown in Figs. 6 and 7. Only these data are presented since they are representative. For copper, stainless steel, and the aluminized mirror, the reflectance decreases with increasing film thickness down to a limiting reflectance. This limit occurs at a different film thickness for each surface. These characteristic thicknesses are approximately 10 μ for copper, 2 μ for the aluminized mirror, and 4 μ for the stainless steel. Percentage decreases from the bare surface values for the level portions of the curves are given in Table I for all angles of incidence and all substrates investigated. Data points for copper in Figs. 6 and 7 are shown to illustrate a typical spread of data. For the other substrates, only the curves are shown to represent the average of the data points. The estimated error in these total reflectance measurements is ±2 percent. Both methods of determining apparent oil film thickness were used on copper, and the methods were in good agreement (Figs. 6 and 7).

The apparent oil film thicknesses on copper, aluminized mirror, and stainless steel ranged from 1.6 to 59 μ, 0.076 to 9.7 μ, and 2.7 to 13.3 μ, respectively.

For the Cat-a-lac black paint surface, the reflectance changed little with increasing oil film thickness (Figs. 6 and 7). The range of apparent oil film thicknesses was 1.2 to 15.9 μ.

These results indicate that for very thin films on copper, aluminized mirror, and stainless steel, the oil films have a marked effect on the reflectances of the substrates and that the effect becomes constant as the film thickness increases. The data also indicate that if the reflectance of the substrate is relatively low, as is the case with Cat-a-lac black paint, then the application of oil films will not greatly change the reflectance. On the other hand, if the reflectance of the substrates is high - as with copper, stainless steel, and the aluminized mirror - then the application of oil films on these will reduce the reflectance.
McCullough, Wood, and Dawson report in Ref. 1 an anomaly in the fundamental reflection law - that is, they discovered an angular shift in the specular component of the reflected radiation when the incident radiation is of a wavelength approximately the same as the rms roughness of the reflecting surface. This deviation is also a function of the angle of incidence, $\psi$. Since this phenomenon is not completely understood, data were taken to determine if oil contamination of the test substrates would produce this shift. Any great change in the magnitude or the spread of the specular component is of interest because this would affect the scattering of reflected radiation.

Both the variation of the magnitude and angle of reflection of the specular component can lead to significant errors in the calculation of radiant heat exchange, whether based on the laws of specular reflection or on the cosine distribution.

A thin oil film on the aluminized mirror produced only slight changes in the distribution of the reflected radiation, as is shown by the intensities of the peaks in Figs. 8 and 9. The distribution curves (Figs. 8 through 17) are representative and do not show all the data. It was suspected that the oil might evaporate in a vacuum and significantly change the index of refraction of the surrounding medium, especially right at the surface of the oil. However, a comparison of Figs. 8 and 9 with Figs. 10 and 11 indicates there is no significant difference between the data obtained at atmospheric pressure and in a vacuum. This was assumed to be true for all substrates.

The application of thin oil films on Cat-a-lac black paint, stainless steel, and copper produces appreciable changes in intensities of the specular peaks (Figs. 12 through 17). These changes indicate changes in the diffuse character or absorption of the reflecting surfaces. It is especially interesting to note the great change in the specular component of radiation reflected from the Cat-a-lac black surface after oil is applied, whereas there is little change in the total reflectance because of oil contamination. Only the stainless steel surface showed both a change in intensity as well as an increase in the angular spread of the specular peaks (Figs. 14 and 15). The distribution measurements may have an error of as much as ±2 percent of full scale.

None of the substrates showed a significant angular shift in the specular component which would have indicated a deviation from the fundamental reflection law (Figs. 12 through 17). The small angular shifts shown in the figures are caused by instrument errors and the fact that the test sample could not be placed in the exact center of the sphere.
4.4 SPECTRAL REFLECTANCE

Percent spectral reflectance with and without oil contamination on all the substrates is plotted versus wavelength in Figs. 18 through 21. Even though data were obtained with the ratio recording spectrophotometer and the infrared integrating sphere system in the 1- to 3-μ wavelength range, only data obtained with the spectrophotometer were presented here. Data obtained with these two instruments generally agreed within 10 percent over the 1- to 3-μ wavelength range.

There is a significant change in the reflectance in the visible and near infrared regions caused by oil contamination on all substrates except the copper where the oil contamination has little effect in the near infrared region of the spectrum.

Also, oil contamination has little effect on the reflectance of all substrates in the intermediate infrared region with the exception of the Cat-a-lac black paint surface. The apparent oil film thicknesses for all substrates were 3 to 6 μ in the 3- to 10-μ wavelength range and 2 to 10 μ in the 0.35- to 2.7-μ wavelength range. Errors in reflectance of ±1 percent in the 0.35- to 2.7-μ wavelength range and ±2 percent in the 3- to 10-μ wavelength range are estimated for these data. Error bands are shown on Figs. 18 through 21.

SECTION V
CONCLUSIONS

An analysis of the results of the total reflectance tests has led to the following conclusions:

1. Application of an oil film has a significant effect on the reflectance of the surfaces except the Cat-a-lac black paint, for which the effect is small.

2. At apparent oil film thicknesses greater than a characteristic thickness for each substrate-oil film combination, the total reflectance tends to level off.

The following conclusions were reached from an analysis of the angular distribution measurements:

1. Thin films of a diffusion pump oil applied to surfaces similar to those found in a space simulation chamber will probably not cause an angular shift in the specular component of the reflected radiation.
2. System evacuation has no appreciable effect on the distribution of radiation reflected from diffusion pump oil contaminated surfaces.

The spectral reflectance data indicate that:

1. The spectral reflectance of all substrates tested was altered by the application of a thin oil film.

REFERENCES


Fig. 1 7-in. Integrating Sphere
Fig. 2 30-in. Distribution Sphere
Fig. 3 Infrared Integrating Sphere System
1. Oscillating mirror
2. Sample beam mirror
3. Reference beam mirror
4. Integrating sphere
5. Entrance ports
6. Exit ports (sample and reference materials are placed here for reflectance measurements; otherwise, ports are normally covered with magnesium oxide plates)
7. Integrating sphere opening
8. Detector

Fig. 4 Optical Diagram of Ratio Recording Spectrophotometer
Fig. 5 Angular Distribution Measurement
Fig. 6 Total Reflectance ($\Delta \lambda = 0.4$ to $1.2 \mu$) versus Oil Film Thickness for Various Surfaces, $\psi = 10$ deg
Fig. 7 Total Reflectance ($\Delta \lambda = 0.4 \text{ to } 1.2 \mu \text{m}$) versus Oil Film Thickness for Various Surfaces, $\psi = 40 \text{ deg}$
Fig. 8  Angular Distribution of Total Radiation (0.4 to 1.2μ) Reflected from an Aluminized Mirror, θ = 10 and 40 deg
Fig. 9 Angular Distribution of 1-μ Radiation Reflected from an Aluminized Mirror, \( \psi = 10 \) and 40 deg
Fig. 10 Angular Distribution of Total Radiation (0.4 to 1.2 μ) Reflected from an Aluminized Mirror in a Vacuum, θ = 10 and 40 deg.
### Table

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### Figure 11

Fig. 11 Angular Distribution of 1-\( \mu \) Radiation Reflected from an Aluminized Mirror in a Vacuum, \( \psi = 10 \) and 40 deg
Fig. 12 Angular Distribution of Total Radiation (0.4 to 1.2 μ) Reflected from Cat-a-lac Black, φ = 10 and 40 deg
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Fig. 18 Spectral Reflectance of an Aluminized Mirror with and without Oil Contamination
Fig. 19 Spectral Reflectance of Copper with and without Oil Contamination
Fig. 20 Spectral Reflectance of Cat-a-lac Black with and without Oil Contamination
Fig. 21 Spectral Reflectance of Stainless Steel with and without Oil Contamination
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EFFECTS OF DIFFUSION PUMP OIL CONTAMINATION ON THE REFLECTANCE CHARACTERISTICS OF VARIOUS SURFACES

**5 AUTOMATION (Last name, first name, initial)**

Pinion, C. E., ARO, Inc.

**13 ABSTRACT**

During thermal testing in a space simulation chamber, the extent of diffusion pump oil contamination on all important surfaces should be determined. This information and an understanding of the reflectance characteristics of the oil contaminated surfaces would contribute to a more reliable interpretation of the thermal test data. Hence, an experimental study was made of the effects of diffusion pump oil contamination on the reflectance and radiation distribution of four types of surfaces found in space simulation chambers. The total reflectance and angular distribution measurements were made in the radiation wavelength range from 0.4 to 1.2 μ. The spectral reflectance measurements were made in the radiation wavelength range from 0.35 to 10 μ. These data were obtained as a function of oil film thickness, angle of incidence, wavelength, and substrate material. Contamination of surfaces caused by diffusion pump oil backstreaming alters the reflectance characteristics of the substrate-oil film combinations.
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