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A VACUUM INTEGRATING SPHERE FOR IN SITU REFLECTANCE MEASUREMENTS AT 77°K FROM 0.5 TO 10 MICRONS

B. A. McCullough, B. E. Wood, and A. M. Smith
ARO, Inc.

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AEROSPACE ENVIRONMENTAL FACILITY
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NOTICES

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FOREWORD

The research reported herein was sponsored by Arnold Engineering Development Center (AEDC), Air Force Systems Command (AFSC), Arnold Air Force Station, Tennessee, under Program Element 61445014, Project 8951, Task 895104.

The results of the research were obtained by ARO, Inc. (a subsidiary of Sverdrup & Parcel and Associates, Inc.), contract operator of AEDC, AFSC, under Contract AF 40(600)-1200. The work was performed under ARO Project No. SW5703, and the manuscript was submitted for publication on December 30, 1966.

In addition to the authors listed on the cover, R. C. Birkebak of the University of Kentucky in Lexington was also an author.

This technical report has been reviewed and is approved.

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ABSTRACT

A modified integrating sphere with a spectral measurement range from 0.5 to 10 microns is described. This integrating sphere can be used for in situ reflectance measurements of low temperature surfaces at vacuum conditions. The techniques employed in measuring the spectral reflectance of CO_2 cryodeposits are discussed. Reflectance data for CO_2 cryodeposits on stainless steel and Cat-a-Lac Black® substrates are presented as a function of wavelength and cryodeposit thickness. The reflectance characteristics of the sphere's internal coating of NaCl are also presented.

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SECTION I
INTRODUCTION

When the environment existing in a space simulation chamber is compared to the natural space environment, some significant differences are observed. For instance, the temperature of the space chamber walls is approximately 77°K, not 4°K, and the reflectance of the walls is not identically zero. In addition, the reflectance of the walls is subject to change because of cryopumping. This change in reflectance, which is a function of cryodeposit species and thickness, may alter the thermal radiative interchange between the test vehicle and its surroundings. To interpret the resulting thermal test data properly, one must account for this variation in the wall reflectance. Thus, it is necessary to measure the reflectance properties of cryodeposits formed on space chamber surfaces.

There are three basic types of instruments which are used to measure reflectance of surfaces. These are the Coblentz hemisphere (Ref. 1), the heated cavity reflectometer (Ref. 2), and the integrating sphere (Ref. 3). The hemisphere with variations has several basic limitations such as critical location of detector and sample and nonuniform detector response to both angle of incidence and image location. The heated cavity reflectometer is not operable in the visible range unless a very high temperature is maintained in the cavity. Of the three systems, the standard integrating sphere is capable of more precise measurements up to a wavelength of 2.7 microns. This limitation on the usable spectral range of the standard integrating sphere is caused by the poor reflectance properties of MgO at wavelengths greater than about 2.7 microns.

This report describes a modification of an integrating sphere which extends the spectral measurement range to a wavelength of 10 microns and makes possible in situ reflectance measurements of low temperature surfaces at vacuum conditions. The techniques employed in measuring the spectral reflectance of CO_2 cryodeposits over the wavelength range from 0.5 to 10 microns are also discussed. The data obtained are presented as a function of wavelength, cryodeposit thickness, and substrate material.

SECTION II
APPARATUS

An 8-in.-diam stainless steel integrating sphere with a center mounted sample holder was modified by changing the existing ports and flanges to vacuum type, using sodium chloride windows, and adding a vacuum pumping system (Fig. 1). The interior of the sphere was coated with powdered sodium chloride (Section 4. 2), and the sample holder was replaced with a liquid-nitrogen (LN_2)-cooled, 1- by 1-1/2-in. test surface. A gas addition system was also incorporated into the apparatus to permit CO_2 deposition on the cryosurface.

The optical system consisted of transfer mirrors and a single-pass monochromator (Fig. 2) mounted on a table which could be rotated about the vertical centerline of the view port. A tungsten-iodine lamp and a gas mantle lantern were used as energy sources, and the energy was chopped at 13 Hz before entering the integrating sphere. The electronics consisted of a 13-Hz amplifier utilizing a synchronous rectifier and a 6-in. strip recording potentiometer.

SECTION III
PROCEDURE

3.1 APPLICATION OF THE SALT COATING*

The sodium chloride (NaCl) coating was applied as follows: A saturated solution of NaCl was painted on the sphere wall. Then powdered NaCl was pressed on top of the NaCl solution using a smaller sphere. After this coating was allowed to dry, a new layer of NaCl was pressed on top of the old. The final thickness of the coating was approximately 3 mm.

3.2 ALIGNMENT

The test surface was replaced with a small sample holder having a light source mounted in the center. The optical table was then rotated to position I (Fig. 2a), and mirror adjustments were made to focus the light source on the entrance slit of the monochromator. An internal alignment check of the monochromator was then made by tracing light rays.

3.3 PUMPDOWN OF SYSTEM

After alignment was completed the NaCl windows were mounted on the sphere ports. Then the valve to the pumping system was

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*Patent application pending.

slowly opened to avoid disturbing the sphere coating. After the sphere was evacuated to approximately 20 microns, the diffusion pump was turned on. The base pressure obtained in the sphere was on the order of 10^{-6} torr.

3.4 CRYODEPOSIT BUILDUP

The system was maintained at the base pressure for several days to minimize any outgassing. The test surface was then cooled to near 77°K by LN_2. This lowered the pressure within the sphere by approximately an order of magnitude. Then the sphere was valved off from the pumping system, and the CO_2 gas addition system was turned on. With a 1.27 atm-cc/sec standard leak, the equilibrium pressure in the sphere varied from approximately 1 to 50 microns during the flow-in period. This rise in equilibrium pressure was caused by a buildup of impurities from the test gas although the gas was 99.9 percent pure. The thickness of the CO_2 cryodeposit which formed on the test surface was calculated from the time of gas flow, the ideal gas law, and the density of solid CO_2 (1.5 gm/cm^3).

3.5 MEASUREMENTS

The reflectance is measured by the hemispherical-angular technique (Fig. 3) (Ref. 4). In this method the test surface is irradiated hemispherically by multiple reflections from the sphere wall which is coated with a material having a high uniform reflectance over the working range. The intensity of the reflected light in the differential solid angle $\Delta\omega_r$ about the polar angle θ is viewed by a monochromatic system. Then the absolute hemispherical-angular reflectance, ρ_{ha}, is determined from

$$\rho_{ha} = \frac{i_r(\theta)}{i_h}$$

where $i_r(\theta)$ is the reflected intensity at the angle θ, and i_h is the incident intensity on the surface. The method employed in measuring these quantities is as follows: The optical system was first focused on the sample as shown in Fig. 2a, and the detector output was recorded. The optical system was then rotated to view a portion of the sphere wall as shown in Fig. 2b, and the detector output was recorded again. In these measurements the same solid angle was subtended. This process was repeated for each wavelength desired.

SECTION IV
RESULTS AND DISCUSSION

4.1 ENERGY SOURCES

The energy sources used in the reflectance measurements were a tungsten-iodine lamp and a gas mantle lantern. The tungsten-iodine lamp was used in the visible and near infrared while the gas mantle lantern was used in the infrared ($\lambda > 2.5$ microns). Energy spectra for these two sources are shown in Fig. 4 where the measured intensities are relative to the maximum intensity of the tungsten-iodine lamp.

4.2 INTERIOR COATINGS FOR THE INTEGRATING SPHERE

The integrating sphere system was originally designed to have an interior coating of sulfur (Refs. 4 and 5). During the first run of the system, the LN_2-cooled test surface cryopumped a large amount of sulfur which sublimed from the sphere coating. Some of the coatings investigated as a replacement for sulfur were sandblasted aluminum, sandblasted stainless steel with vacuum deposited aluminum, plasma sprayed aluminum and copper, boron nitride, beryllium oxide, zirconium oxide, aluminum oxide, and sodium chloride. Of the coatings investigated, only sodium chloride had the reflectance properties required for the interior coating of an integrating sphere. Also, the vapor pressure of NaCl is two orders of magnitude lower than that of sulfur. The spectral reflectance properties for sodium chloride are shown in Fig. 5a, and the directional reflectance properties at a wavelength of 10 microns are presented in Fig. 5b. From these results, it was concluded that sodium chloride was a good diffuse reflector. This agrees with the conclusions of Agnew and McQuistan (Ref. 6) who found that sodium chloride was one of the best diffuse reflectance standards in the infrared region.

4.3 REFLECTANCE VERSUS WAVELENGTH MEASUREMENTS

The reflectance measurements of CO_2 cryodeposits on polished stainless steel and Cat-a-Lac Black substrates are shown, respectively, in Figs. 6 and 7. The polar angle of viewing for all measurements was 20 deg.

In general, for a thin (0.318 mm) CO_2 deposit, a decrease in the reflectance of the cryodeposit-substrate complex was noted for all wavelengths. However, for wavelengths less than 1.0 and around 3.5 microns, the CO_2-Cat-a-Lac Black complex (Fig. 7) showed an increase in reflectance. It was also observed that the reflectance of the CO_2-Cat-a-Lac Black complex increased with further deposit buildup. For the CO_2-stainless steel complex (Fig. 6), the reflectance continued to decrease with further deposit buildup except for the near-infrared and visible regions. From the reflectance data for the CO_2-stainless steel complex, possible absorption bands for CO_2 cryodeposit were noted at wavelengths of approximately 2.7, 4.0, 6.75, and 9.25 microns.

The failure of the two cryodeposit-substrate complexes in Figs. 6 and 7 to attain a common value of reflectance at long wavelengths was expected. Cunningham and Young (Ref. 7) measured total absorptance of various CO_2-substrate complexes for 350°K radiation and never attained a common value. Their experimental results indicated that for a CO_2 cryodeposit thickness less than 0.8 mm, the absorptance of the cryodeposit-substrate complex depends on the bare wall absorptivity and deposit thickness. However, for thicknesses greater than 0.8 mm the absorptance of the complex was a function of the bare wall absorptance only. They postulated from their results that the CO_2 cryodeposit absorbs infrared radiation selectively.

4.4 REFLECTANCE VERSUS THICKNESS MEASUREMENTS

In Figs. 8a and b the reflectances of CO_2-Cat-a-Lac Black and CO_2-stainless steel complexes are plotted as a function of thickness for wavelengths of 0.75 and 1.70 microns, respectively. In both cases there is a sharp decrease in reflectance for a small thickness of CO_2 cryodeposit. Then as the thickness is increased further, the reflectance attains a minimum and then increases slowly. This effect could possibly be attributed to scattering and absorption phenomena which occur in the cryodeposit. When absorption is predominant, such as in an absorption band, the reflectance decreases monotonically with deposit thickness as shown in Fig. 8c for CO_2 cryodeposit on a stainless steel substrate. From this fact and Figs. 8a and b it is postulated that absorption and internal scattering increase with thickness until some plateau is reached. Thus, for thin films, where absorption is much greater than scattering, a decrease in reflectance would be observed. However, for thicker films, where the effect of internal scattering is significant, the reflectance would reach a minimum and then increase.

It is seen from Fig. 8a that for large CO_2 cryodeposit thicknesses the reflectances of the two deposit-substrate complexes are equal. Thus, for short wavelengths and large CO_2 deposit thicknesses, the reflectance of the deposit-substrate complex is independent of the substrate reflectance. However, for longer wavelengths, the data in Figs. 6, 7, and 8b indicate that the deposit-substrate reflectance may not be independent of the value of the substrate reflectance.

SECTION V
CONCLUSIONS

The operation of the infrared integrating sphere indicates that:

1. The integrating sphere system using NaCl as an interior coating will operate satisfactorily at vacuum conditions with LN_2-cooled test surfaces.
2. In situ reflectance measurements of CO_2 cryodeposits can be made over the wavelength range from 0.5 to 10 microns.

The test results indicate that:

1. The reflectance of CO_2 frost is independent of the substrate material in the visible wavelength range for large frost thicknesses.
2. In the infrared region the reflectance of the CO_2 frost-substrate is a complex function of the CO_2 frost and substrate optical properties.
3. The reflectance for the visible and near-infrared regions decreases initially for small thicknesses and then increases with cryodeposit buildup.

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APPENDIX
ILLUSTRATIONS

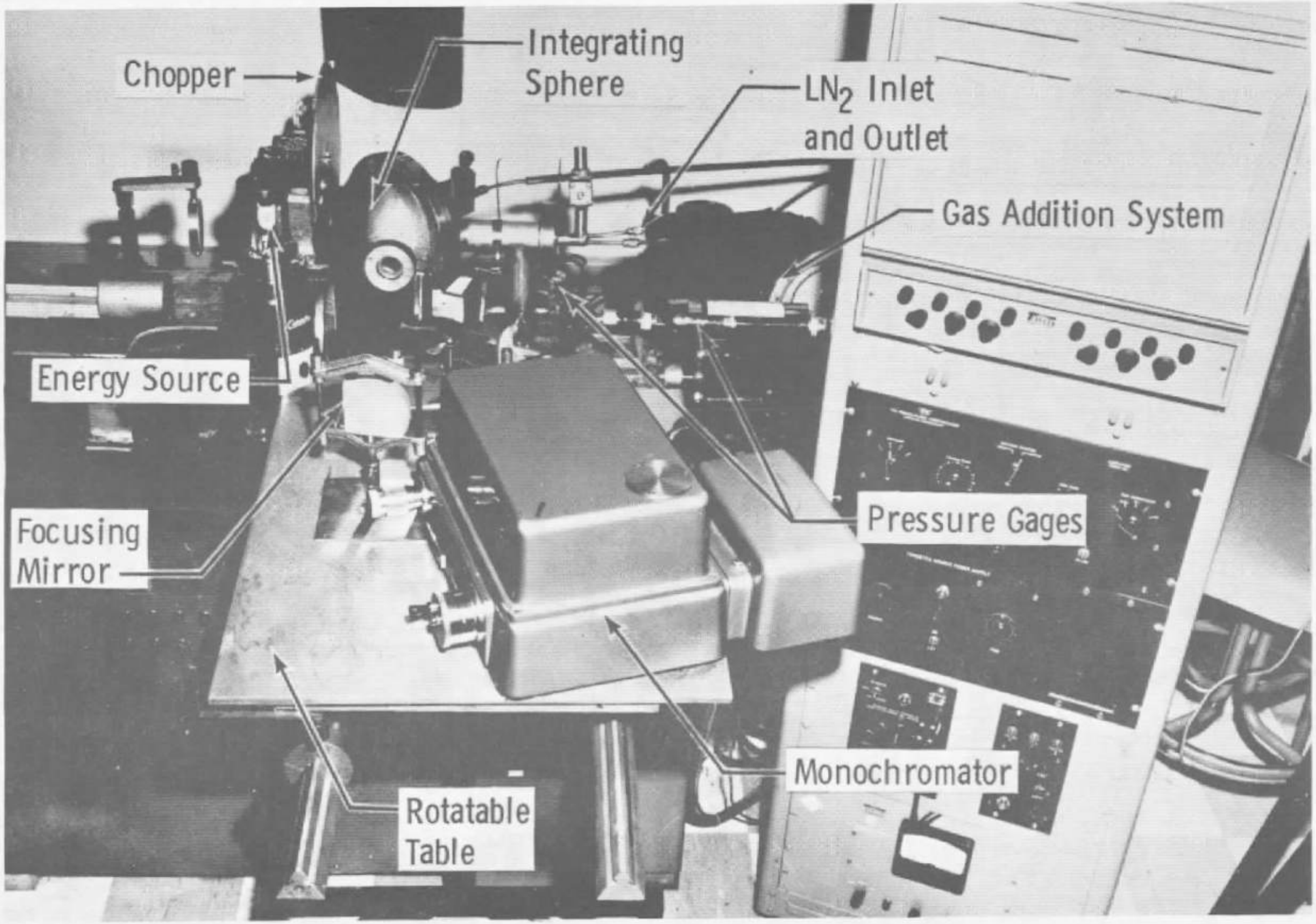
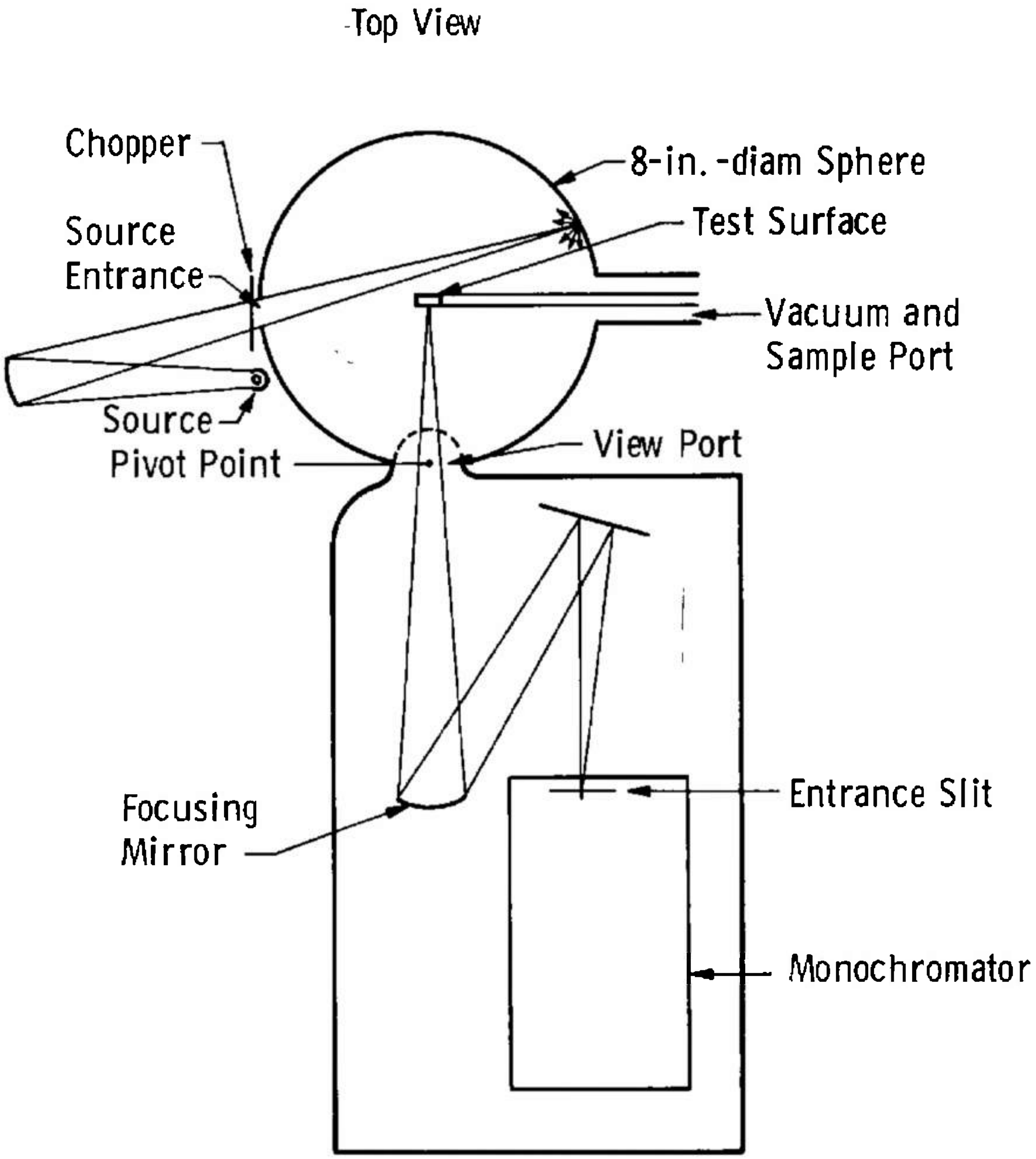
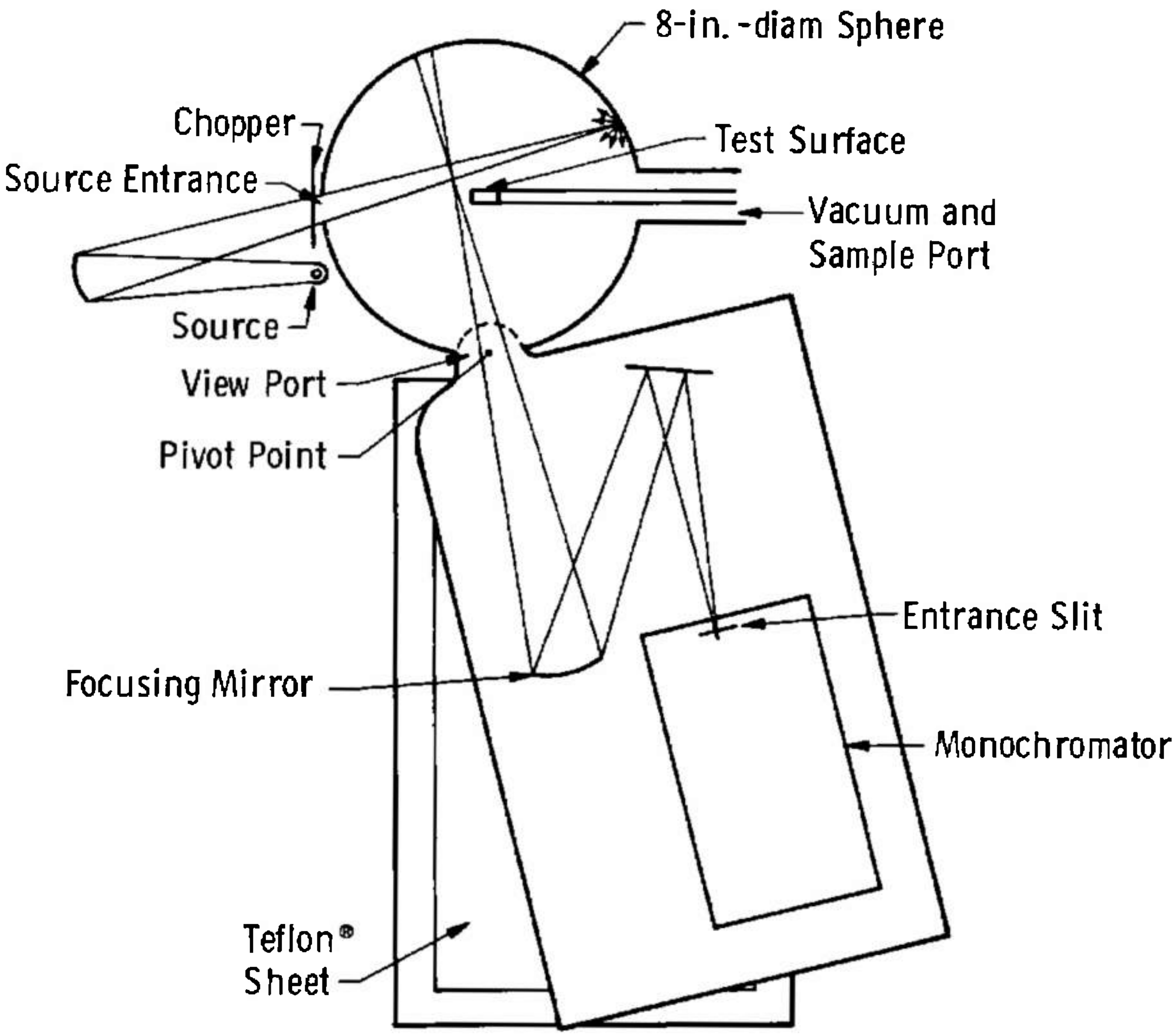


Fig. 1 Infrared Integrating Sphere System



a. Position I

Fig. 2 Infrared System Diagram



b. Position II

Fig. 2 Concluded

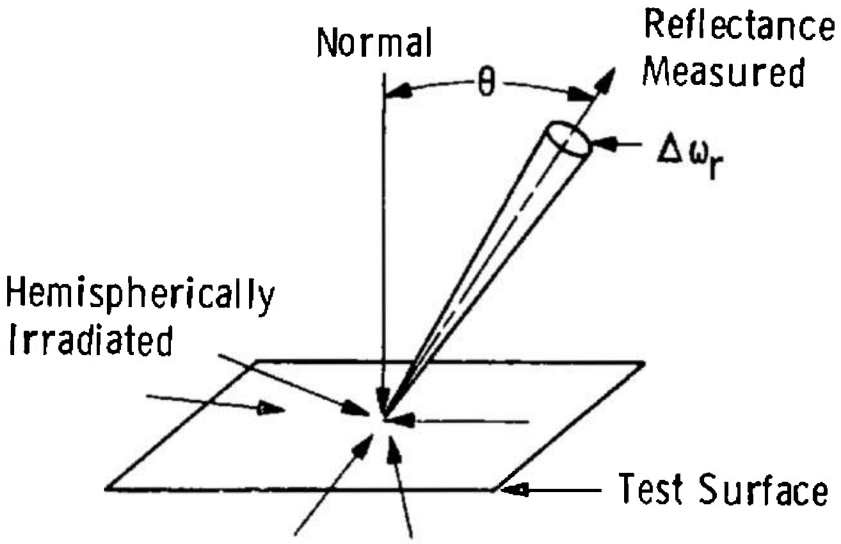


Fig. 3 Hemispherical-Angular Reflectance Measuring Technique

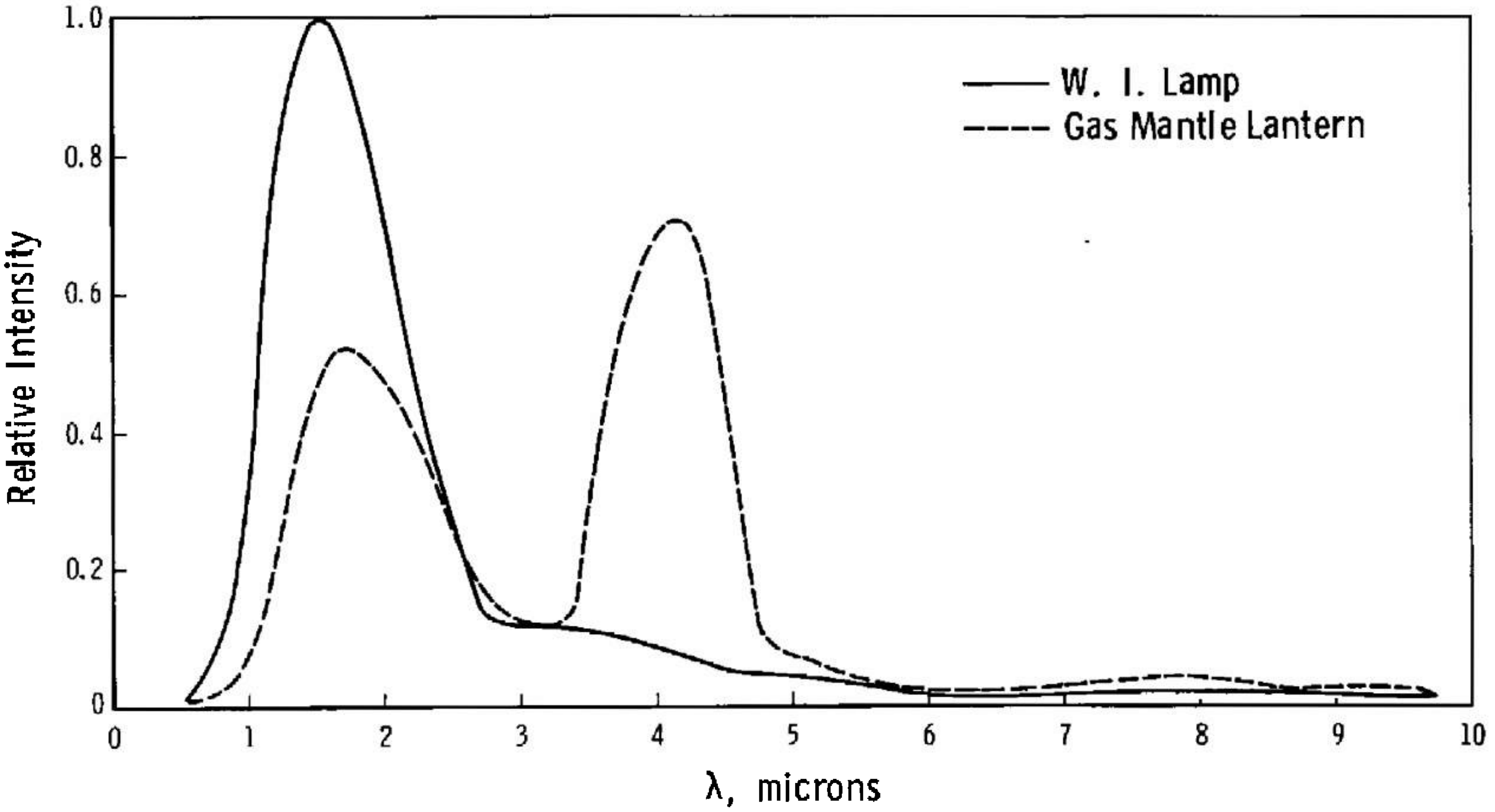
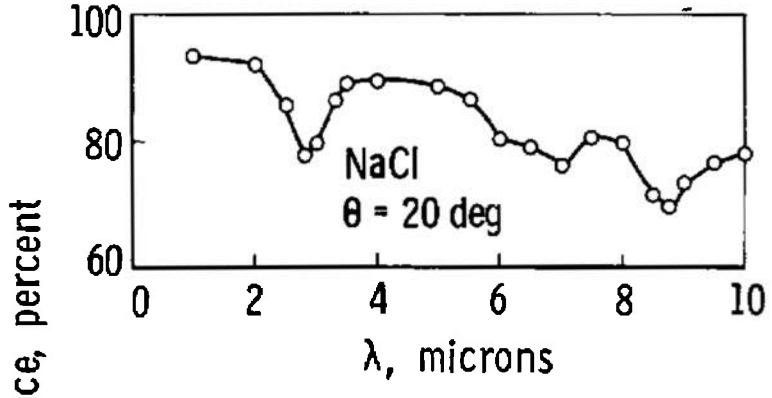
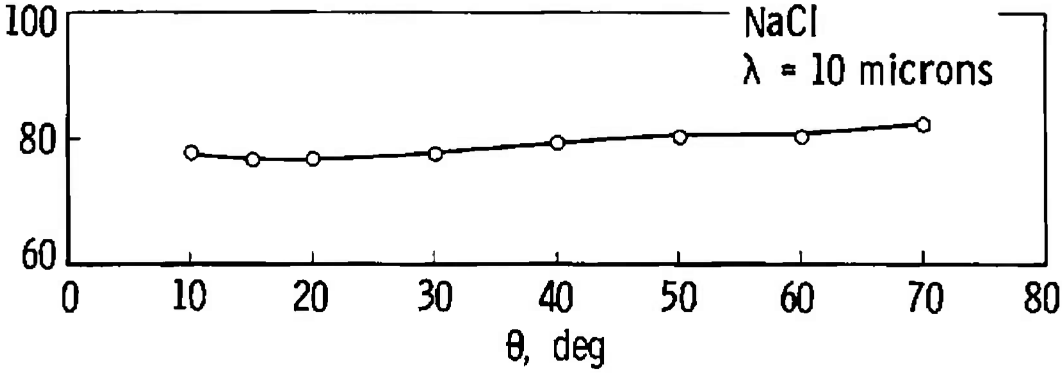


Fig. 4 Energy Distribution of Sources



a. Spectral Reflectance



b. Directional Reflectance

Fig. 5 Reflective Properties of NaCl

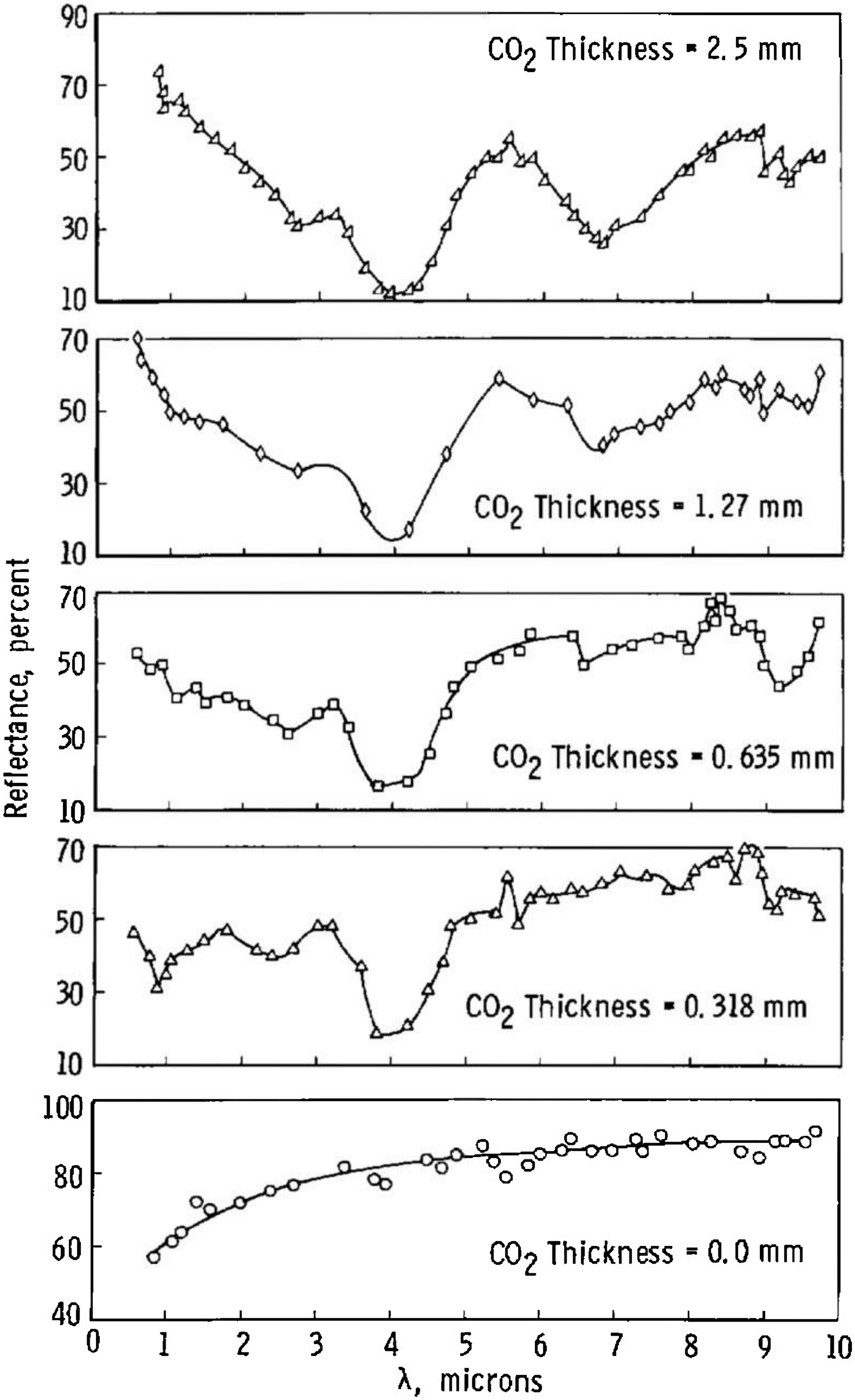


Fig. 6 Spectral Reflectance of CO_2 Cryodeposit on a Stainless Steel Substrate

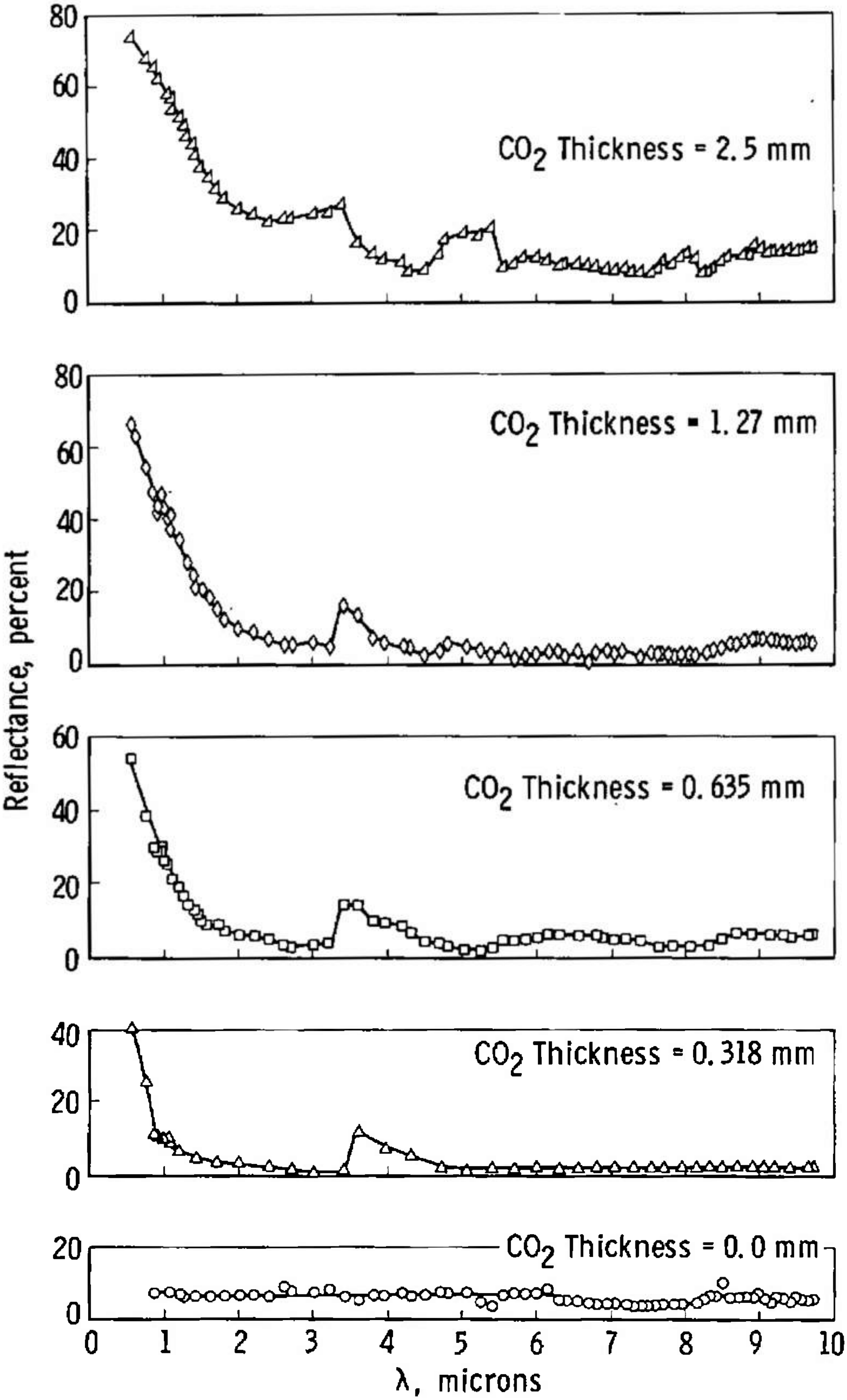
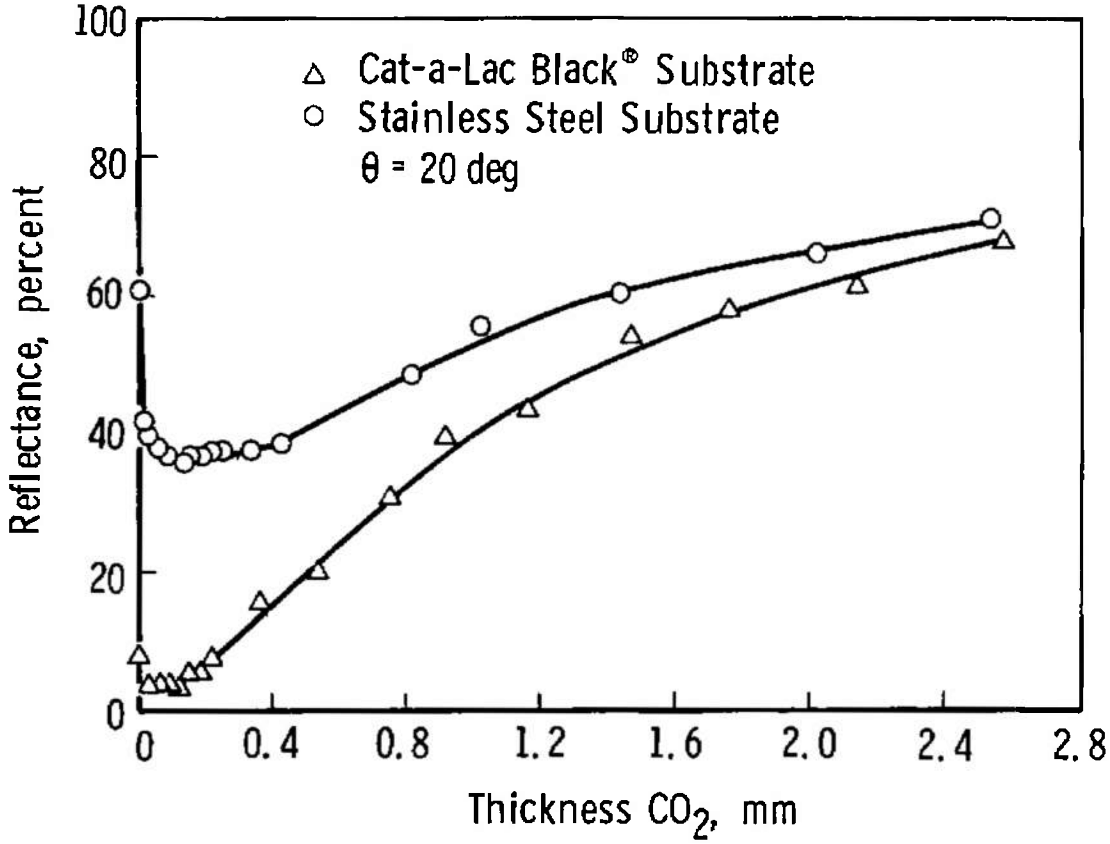
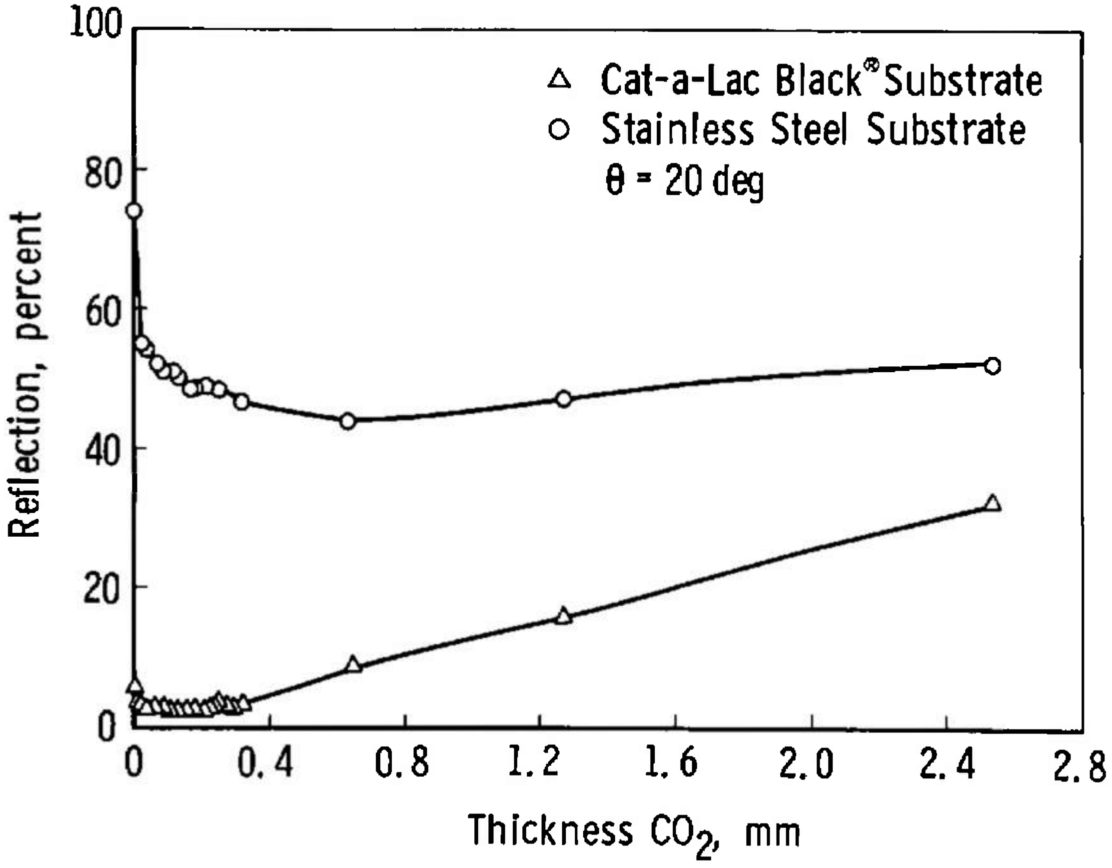


Fig. 7 Spectral Reflectance of CO$_2$ Cryodeposit on a Cat-a-Lac Black Substrate



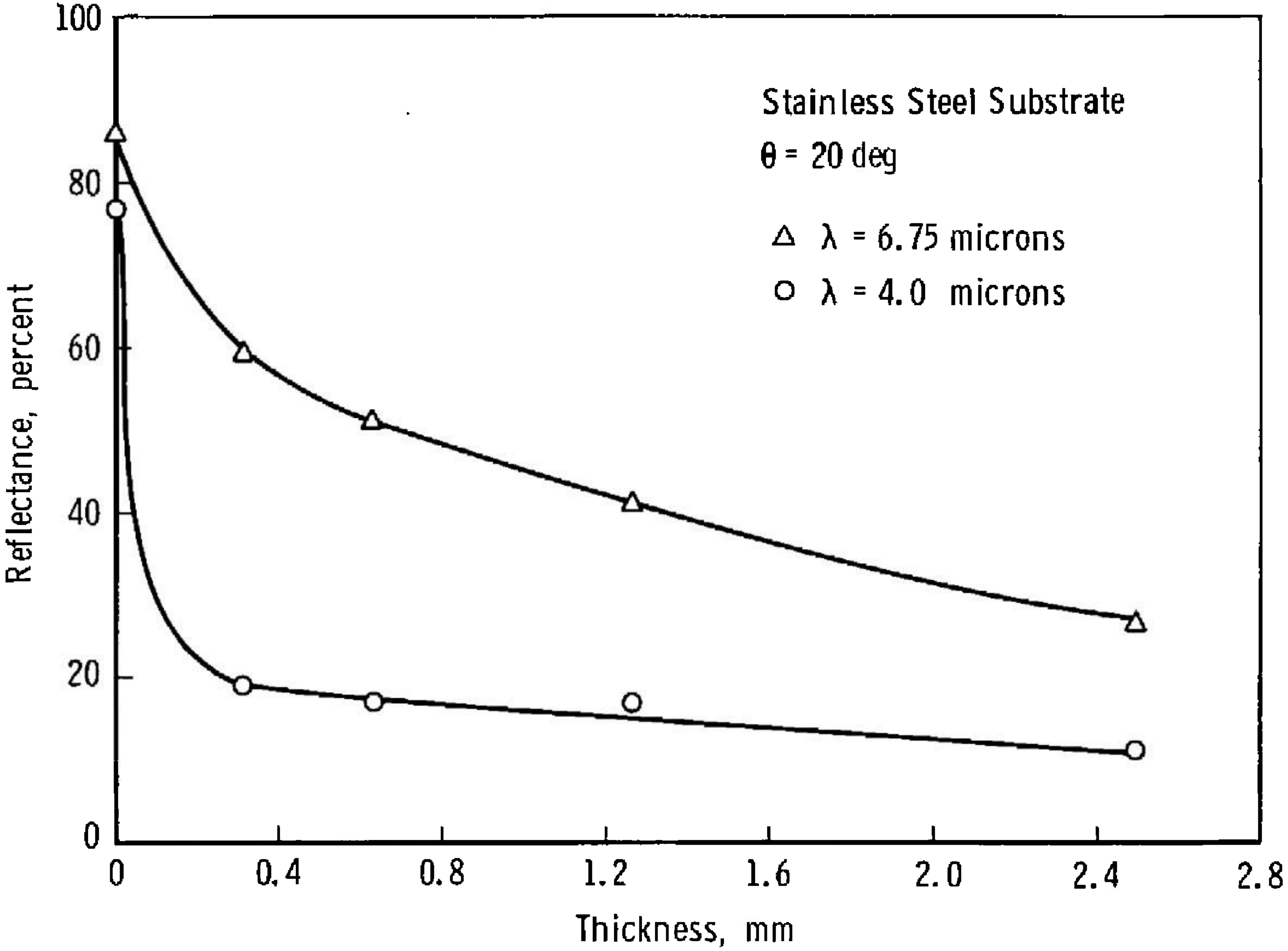
a. λ = 0.75 microns

Fig. 8 Effect of CO_2 Cryodeposit Thickness on the Cryodeposit-Substrate Reflectance



b. λ = 1.70 microns

Fig. 8 Continued



c. λ = 4.0 and 6.75 microns

Fig. 8 Concluded

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13 ABSTRACT

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