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AN INVESTIGATION OF INFRARED DIFFUSER SURFACES

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A study of several possible infrared diffuser surfaces has been carried out for near-normal (ψ = 10 deg) incident radiation. Gold-coated grit surfaces, chemically etched aluminum, powdered sodium chloride, sulfur, magnesium oxide, and beaded surfaces were investigated using infrared bandpass filters. Angular distribution measurements of reflected radiation were made for the following infrared wavelength intervals: λ < 1.4, 7.5 to 14.5, 11.8 to 15.5,

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14.0 to 23, and 19.1 to 22.5 μm. Of all the surfaces investigated, it appears that powdered salt coatings are the most uniform and come closest to being a Lambert surface for infrared radiation. The gold-coated grit surfaces and the chemically etched aluminum also diffuse the infrared radiation somewhat but are nonuniform when viewed on a small scale.

PREFACE

The research reported herein was conducted by the Arnold Engineering Development Center (AEDC), Air Force Systems Command (AFSC), Arnold Air Force Station, Tennessee, under Program Element 65802F. The results presented were obtained by ARO, Inc. (a subsidiary of Sverdrup & Parcel and Associates, Inc.), contract operator of AEDC, AFSC, under ARO Project Numbers VW5202 and VF202. The manuscript (ARO Control No. ARO-VKF-TR-74-9) was submitted for publication on January 15, 1974.

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1.0 INTRODUCTION

Bidirectional reflectance data for diffusing surfaces in the infrared wavelengths are scarce at best. Extension of the operating range of integrating spheres into the infrared range (Refs. 1-4) has increased interest in determining the diffusing properties of integrating sphere coatings. More recently, the most appropriate design for a multiple mobile target system for infrared sensor testing at AEDC has made necessary an infrared diffuser surface for near-normal incident radiation ($\psi < 15$ deg). The requirements for the diffuser are as follows: (1) it must diffuse infrared radiation of wavelengths between 7 and 25 μm, (2) material must withstand active cooling to 20°K, (3) material must be solid, so that no dusting is encountered when the chamber is being pressurized, and (4) the surface must be composed of a low vapor pressure material, as it must remain stable under high vacuum conditions.

Some of the candidate surfaces have been previously investigated to determine their diffusing capabilities, but these measurements were made for the wavelength range in which the classical integrating sphere is usable ($\lambda < 2.5$ μm). At these wavelengths, the measurements represent a "best case" condition, since generally a surface will scatter short wavelength radiation much more that it will the longer wavelength radiation. However, there have been a few investigations made into the infrared range. Agnew and McQuistan (Ref. 5) present angular distribution measurements for radiation that is in the wavelength interval of $0.5 \leqslant \lambda \leqslant 4$ μm and in the interval of $\lambda \geqslant 4$ μm for several metallic and nonmetallic surfaces. These intervals were established using a quartz filter in conjunction with a globar source. Zentner, et al. (Ref. 6) present reflectance distribution data for gold-coated sandpaper for several wavelengths up to and including 10 μm. Several distribution curves were presented for 400-grit sandpaper for angles of incidence from 15 to 75 deg with the result that a specular component, which increased with increasing incidence angle, was observed. Distribution data were also presented by Kneissl in Ref. 4 for sodium chloride (NaCl) surfaces at the three helium-neon (He-Ne) laser wavelengths of 0.63, 1.15, and 3.39 μm. For these wavelengths, the sodium chloride coating, as expected, was a good diffuser.

For wavelengths beyond 10 μm there is almost a complete absence of spectral reflectance distribution data. This is understandable since infrared distribution measurements are difficult to obtain because small irradiance and collection solid angles are desired. Additionally, small irradiated areas and the relatively inefficient infrared detectors quickly reach the energy limits of the spectrometer as the longer wavelength measurements are attempted. In this study the problem of being energy limited was reduced by (1) using bandpass filters rather than spectral radiation and (2) using a relatively large collection solid angle, as determined by the detector, of 0.113 sr. With this experimental setup, angular distribution data of infrared radiation reflected from diffuser surfaces was accomplished.

2.0 APPARATUS

A schematic of the apparatus used in obtaining the distribution measurements is shown in Fig. 1. The radiation source was a Nernst glower approximately 3 cm long and 1 mm in diameter (for spectral output, see Fig. 2). The small size made it ideal for irradiating a small area. Radiation from the glower was chopped at 13 Hz, reflected from a small plane mirror, and focused on the test surface by an off-axis paraboloidal mirror. The sample was located at the center of a rotary table. The diffused radiation from the sample was collected by a 4-in. (101.60 mm)-diam mirror (focal length = 5.9 in. (149.86 mm)) located 20 in. (508 mm) away from the sample. The collected radiation was then focused on a pyroelectric detector located 8.5 in. (215.90 mm) away from the mirror after passing through a bandpass filter located in front of the detector. The location of the mirror and detector caused the image of the Nernst glower to be reduced by a factor of about 2.4 at the detector. The output of the pyroelectric detector was amplified, synchronously rectified, and displayed on a strip chart recorder. Transmission curves for the bandpass

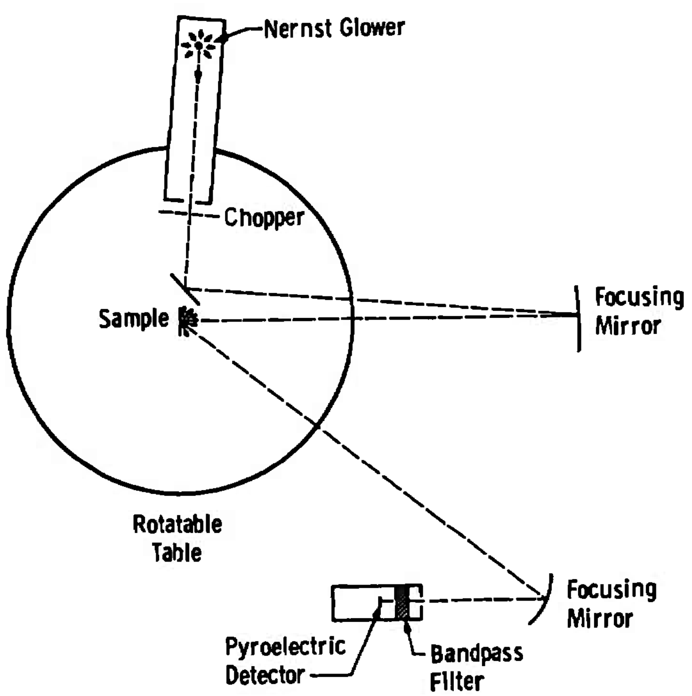


Figure 1. Schematic of biangular distribution apparatus.

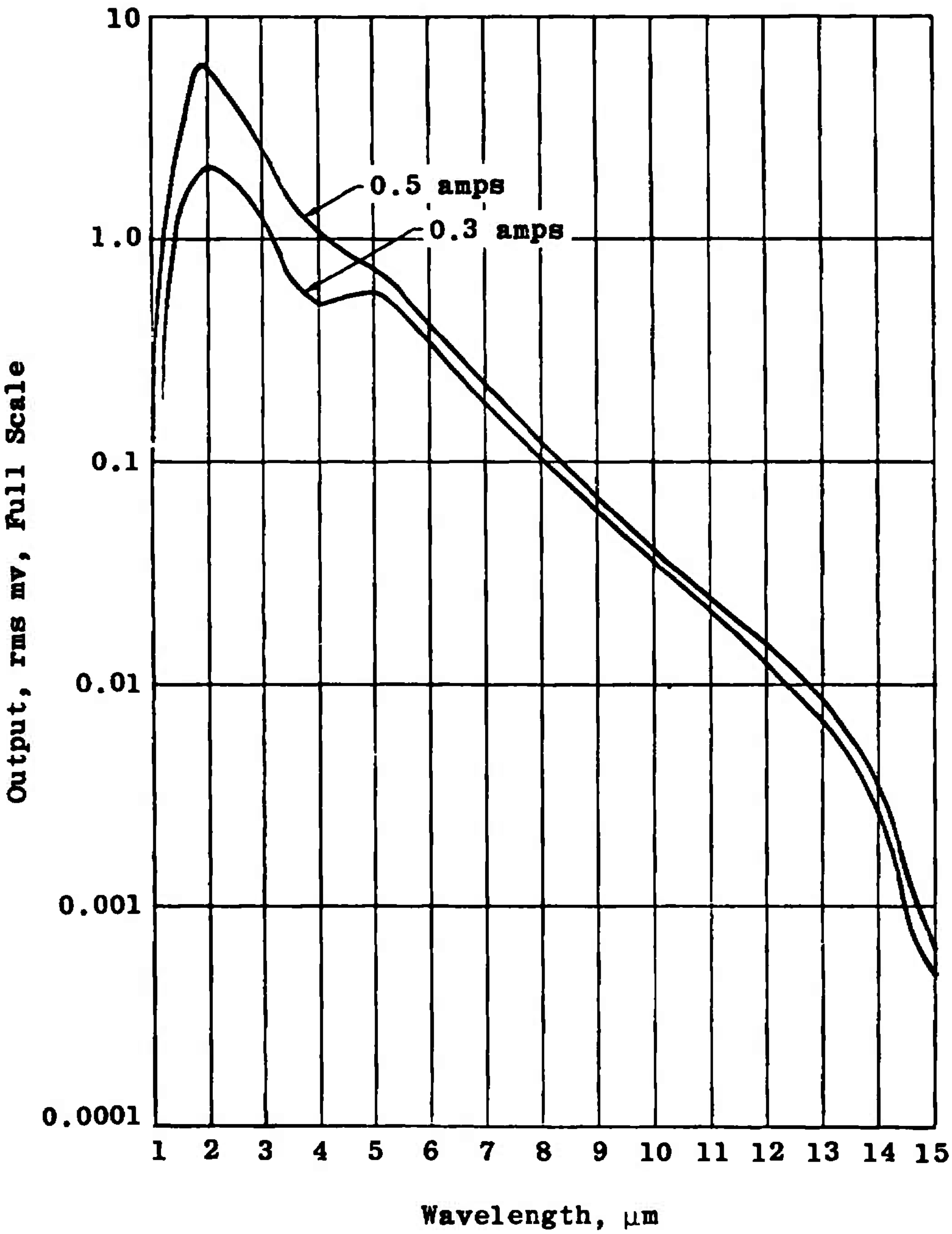


Figure 2. Spectral output of Nernst glower.

filters are shown in Fig. 3. All of the bandpass filters for wavelengths greater than 7.5 μm were obtained from Optical Coating Laboratories, Inc. (OCLI). The filter designated W-P was made using a 1/2-in. (12.70 mm)-long Plexiglas® cylinder filled with water. This provided an excellent long wavelength cutoff filter and transmitted only radiation of wavelength less than 1.4 μm.

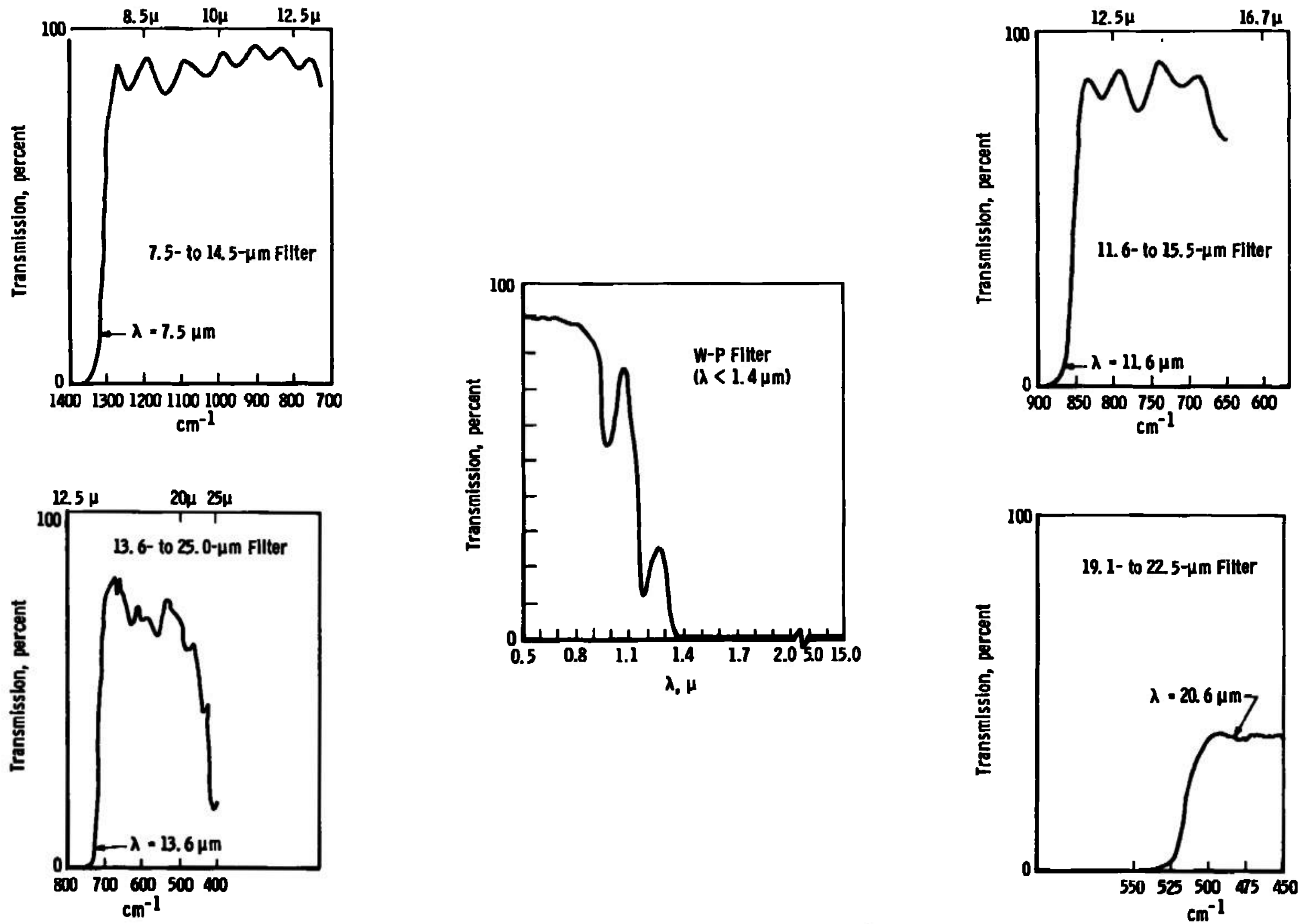


Figure 3. Transmission curves of bandpass filters.

All of the test samples were 1 in. (25.4 mm) in diameter and 1/4-in. (6.35 mm) thick. Keeping the same sample thickness was important since the focal point of the paraboloidal mirror was somewhat critical in keeping the irradiated area exactly the same for all samples. The holders for the powder-type samples such as magnesium oxide (MgO), sulfur, etc. were 1/4-in. (6.35 mm)-disks, hollowed out to a thickness of 1/16 in. (3.17 mm). Therefore, all of the powder-type samples were 3/16-in. (9.52 mm) thick to keep the total thickness of sample and holder to 1/4 in. (6.35 mm).

The Nernst glower, chopper, plane mirror, paraboloidal mirror, and sample were all mounted on the rotary table. The spherical collecting mirror, filters, and detector were mounted at fixed positions. Distribution measurements could be made as a function of incidence angle by rotating the sample with respect to the incident radiation. The angle of incidence calibration was accomplished by placing a mirror at the sample position and reflecting the image back directly onto the paraboloidal mirror, which corresponded to a zero incidence angle. Other incidence angles were measured by a protractor mounted on the test sample assembly. Calibration of the rotary table for determining the angle of reflection of the diffused radiation from the sample was done by recording the rotary table readings of the specularly reflected radiation for given angles of incidence. This calibration was required since the sample and rotary table could be rotated independently of each other.

For determining sample uniformity, the apparatus shown schematically in Fig. 4 was used. It consisted of a He-Ne laser (λ = 0.6328 μm), a chopper, a variable aperture, the sample located on a translator table, another variable aperture, a lens, and a photomultiplier tube. The irradiated area, or spot size, was controlled by the aperture nearest the sample. Circular spot sizes of 0.025-in. (6.35 mm), 0.05-in. (1.27 mm), 0.1-in. (2.54 mm), and 0.2-in. (5.08 mm)-diam were used. Similarly, the total included view angle, β, was controlled

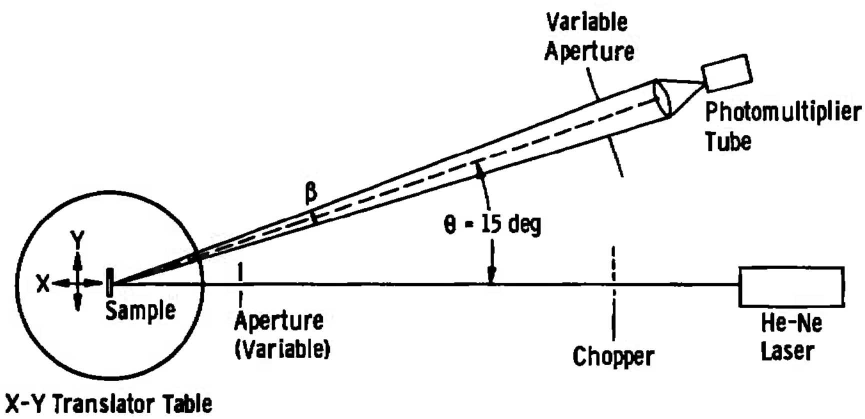


Figure 4. Schematic of uniformity-measuring apparatus.

by the variable aperture nearest the lens. These apertures were adjusted to yield included angles of 0.5 and 1.5 deg, which corresponded to included angles proposed for use in the Aerospace Chamber (7V) and in the Aerospace Environmental Chamber (Mark I), respectively.

3.0 PROCEDURE

The coordinate system used for the angular distribution measurements is depicted in Fig. 5. The angle of incidence, ψ, is measured from the test surface normal. In most of the measurements discussed, ψ was 10 deg. The angle of reflected radiation, θ, was measured from the test surface normal so that positive values of θ were observed for the quadrant in which specular reflection occurred and negative values of θ corresponded to angles located in the quadrant containing the incident radiation. For radiation scattered directly back along the incident beam for ψ = 10 deg, θ would be equal to -10 deg. Only positive values of θ were investigated in this study.

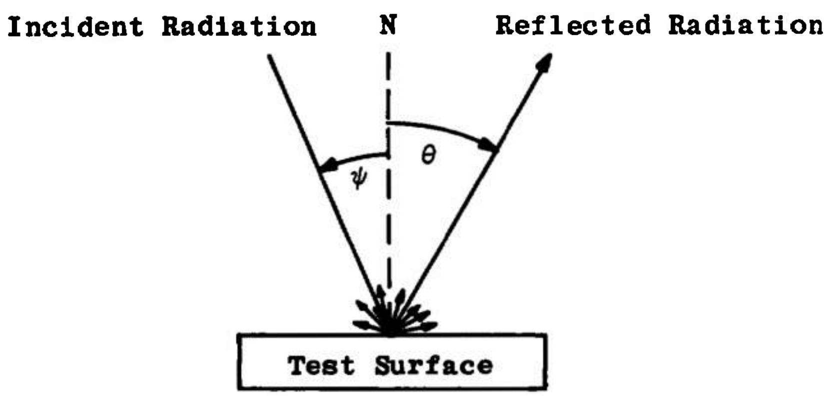


Figure 5. Spatial coordinate system.

For the angular distribution measurements, the optical alignment of the entire system shown in Fig. 1 was first established. This was done by using an MgO-coated sample and measuring the distribution of short wavelength radiation reflected. Magnesium oxide is probably the best known diffuser for visible and near-infrared wavelengths. Since the detector area is quite small, 0.5 x 0.5 mm, optical alignment and the focusing of the irradiated spot are quite critical. The spherical mirror which collected the reflected radiation had a focal length of 5.9 in. (149.86 mm) and was located approximately 20 in. (508 mm) away from the sample. This gave a magnification of slightly less than half and prevented the detector from being overfilled by the small dimension of the irradiated spot, which was essentially the same as the slit-like Nernst glower. (The long dimension of the Nernst glower overfilled the detector.) This procedure allowed a mapping of energy reflected from the surface, as this corresponds to the over-detected case in which the viewed projected irradiated area decreases as the cosine of the reflected angle from the test surface normal. The MgO test surface was used to establish that for a good diffuser surface, the detector output would have a near-cosine response. The results of these tests are shown in Fig. 6. In the figure the ordinate scale is the detector output at angle θ, $E(\theta)$, normalized by the maximum value observed, or $E(\theta)/[E(\theta)]_{max}$. For the short wavelength radiation ($\lambda < 4$ μm), the output (circled data) was in near perfect agreement with the cos θ curve (dot/dash curve). This indicates that the optical alignment of the system was satisfactory and that all of the energy reflected from the sample and collected by the mirror was being focused inside the small (0.5-mm-square) pyroelectric detector. As indicated by arrows in Fig. 6, the incidence angle was set at ψ = 10 deg. The solid curve through the triangular data points indicates similar measurements for MgO but for a 7.5- to 14.5-μm bandpass filter. The results show a strong specular peak occurring for these longer wavelengths. This peak could be caused by either (1) transparency of the MgO and the radiation transmitted by the MgO being specularly reflected from the aluminum substrate and back through the MgO or (2) specular reflection from the MgO front surface since, in general, the specular reflectance of a rough surface increases with increasing wavelength. It is felt that the latter explanation is more probable.

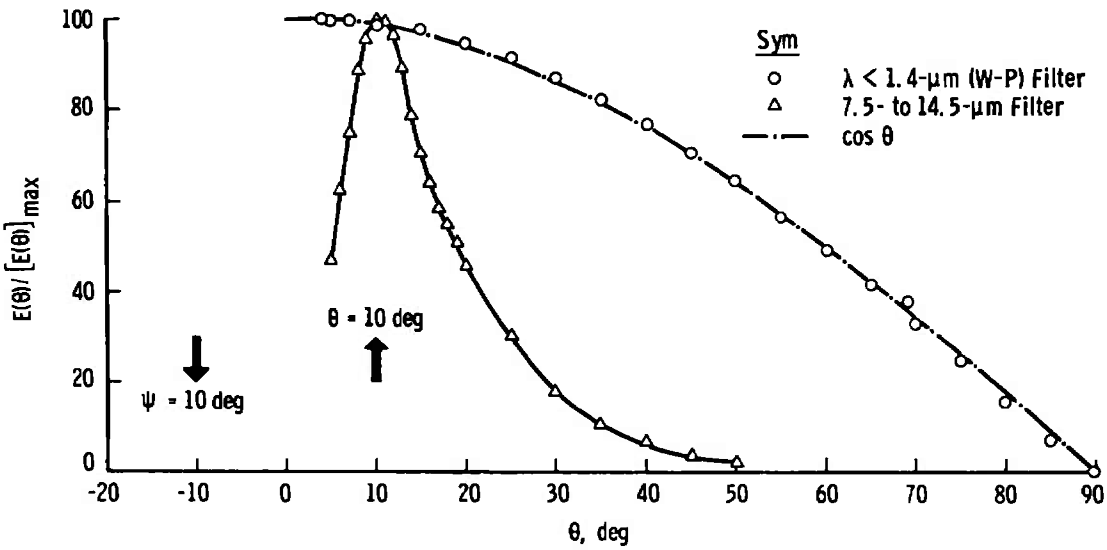


Figure 6. Angular distribution of radiation reflected from a smoked magnesium oxide surface.

Once the optical alignment of the system had been established, the other diffuser surfaces were investigated. In all cases the incidence angle, ψ, was 10 deg. The other samples were carefully positioned at the same exact locations used for the MgO sample and their angular distribution of reflected radiation measured.

In order to give some idea of the angular resolution of the angular distribution measuring system, an aluminum front surface mirror was installed and the distribution of reflected radiation measured (Fig. 7) for the wavelength range from 19.1 to 22.5 μm. The half-peak width is about 7 deg, which gives a good indication of the angular resolution obtained in these experiments.

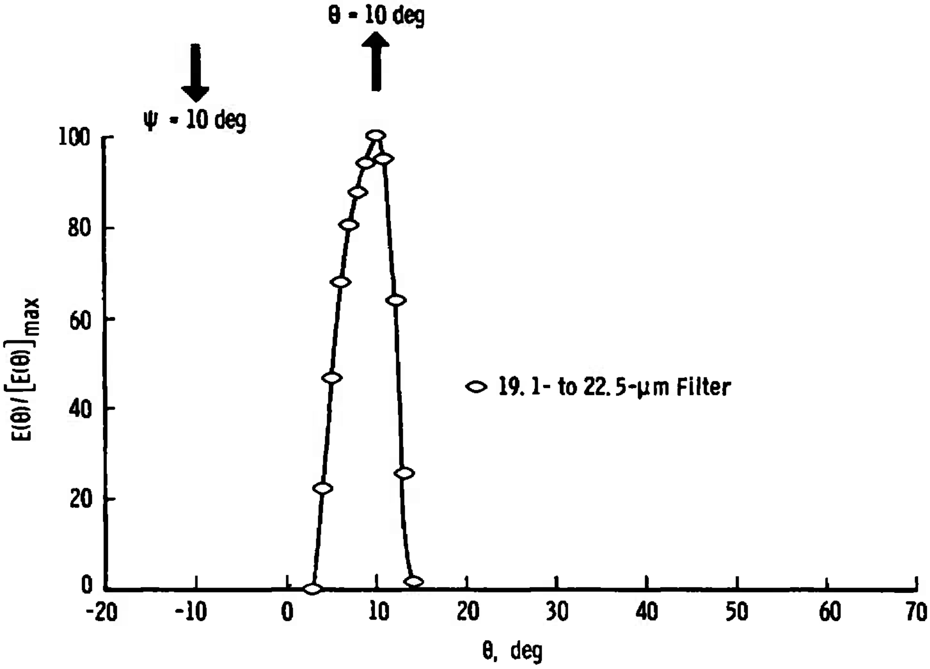


Figure 7. Distribution of radiation reflected from an aluminum mirror surface.

4.0 RESULTS

4.1 ANGULAR DISTRIBUTION MEASUREMENTS

4.1.1 Sodium Chloride

The previous use of powered sodium chloride as an infrared integrating sphere coating suggested the possible use of it as an infrared diffuser. Reagent grade sodium chloride was ground into powder form using a mixer-mill so that the fine powder obtained could be pressed to make a good sturdy coating. Angular distribution measurements were made for the wavelength ranges of 7.5 to 14.5, 11.8 to 15.5, 14.0 to 23.0, and $\lambda < 1.4$ μm. The results are presented in Fig. 8. For all these wavelength intervals, the sodium chloride is a good diffuser and at least for the wavelengths covered here would make a good infrared integrating sphere coating as far as the diffuseness criteria is concerned. With regard to the multiple target system, however, it does not appear that the salt would be clean enough for installation in a chamber which has strict cleanliness standards. Since the system is vacuum rated, repressurizing the system could cause considerable removal of salt particles resulting in a serious contamination problem within the 7V chamber. Therefore, the sodium chloride coating does not presently appear to be feasible, based on its physical characteristics.

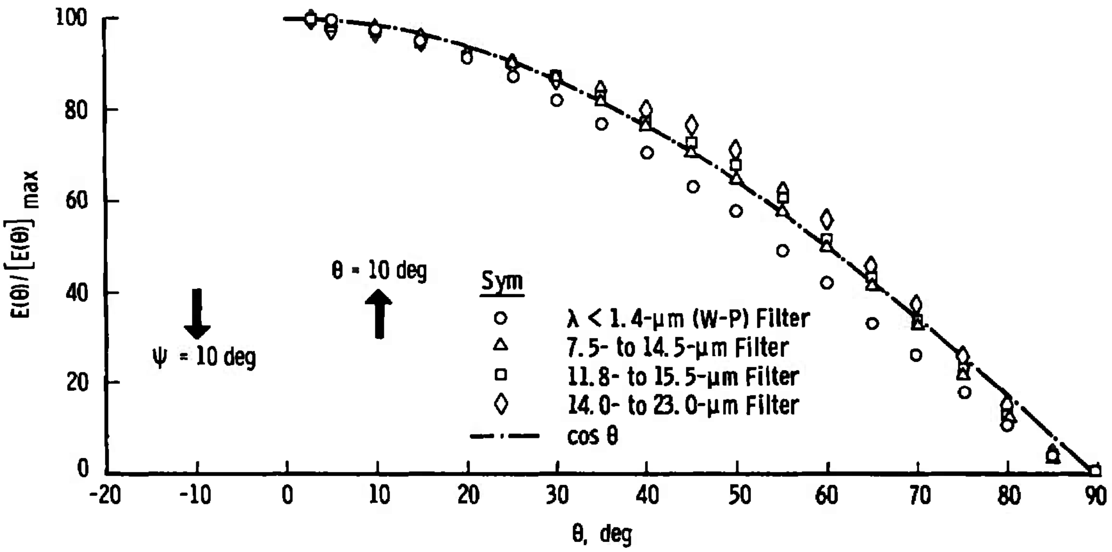


Figure 8. Angular distribution of radiation reflected from a powdered sodium chloride surface.

4.1.2 Sulfur

Sulfur is another material which, like sodium chloride, has been used previously as an infrared integrating sphere coating. A 3/16-in. (9.52 mm)-deep sample holder was filled with flowers of sulfur and irradiated at an angle of incidence of 10 deg. The results are shown in Fig. 9. For the wavelength intervals of $\lambda < 1.4$, 7.5 to 14.5, and 11.8 to 15.5 μm, the sulfur was an excellent diffuser of radiation. For the interval from 14 to 23.0 μm, a slight lobe was seen in the specular direction. Therefore, an attempt was made to investigate this sample for the longest wavelength band, 19.1 to 22.5 μm. For this wavelength band, the distribution had a strong specular peak and the scattered radiation at other angles θ was significantly less. It is believed to be a trend similar to that seen for MgO where now the sulfur front surface appears relatively smooth at the longer wavelengths. In addition to suffering dusting problems similar to those of powdered sodium chloride, flowers of sulfur is handicapped by the problem of having a fairly high vapor pressure at 300°K ($\approx 10^{-5}$ torr). This means that it sublimes under hard vacuum conditions.

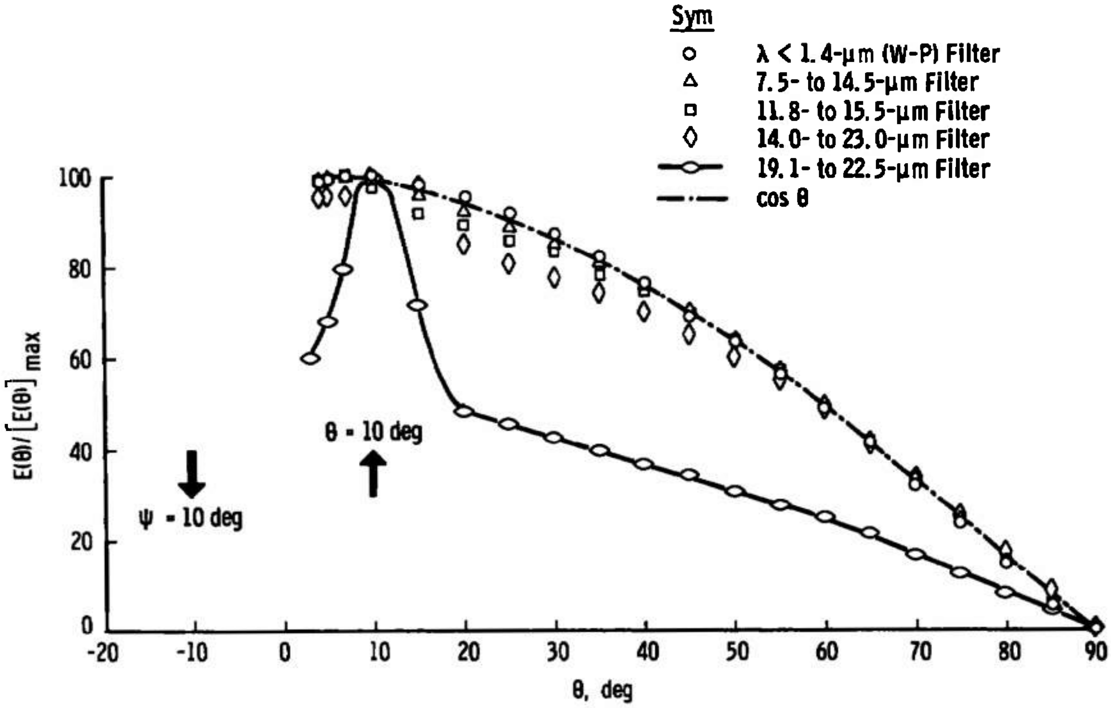


Figure 9. Angular distribution of radiation reflected from a flowers of sulfur test surface.

4.1.3 Glass-Beaded Surface

The next surface investigated was that prepared by sprinkling small spherical glass beads (1- to 30-μm diam) on a freshly prepared Devcon® aluminum-coated surface. After the aluminum had hardened, the entire surface was overcoated with a vacuum-deposited thin layer of gold. The results of the distribution measurements are shown in Fig. 10. For this surface, measurements were made for the following wavelength intervals: $\lambda <$ 1.4, 7.5 to 14.5, 11.8 to 15.5, 14.0 to 23, and 19.1 to 22.5 μm. The surface did not approach being diffuse for any of the wavelengths investigated. A broad specular lobe was observed in the specular direction for all wavelengths. For the longest wavelength interval, 19.1 to 22.5 μm, the radiation was diffused less than the shorter wavelengths, which was to be expected. It would appear that if this particular type of surface were to be used, a much wider range of glass bead sizes would have to be investigated to determine which, if any, would meet the suitable diffuser requirements.

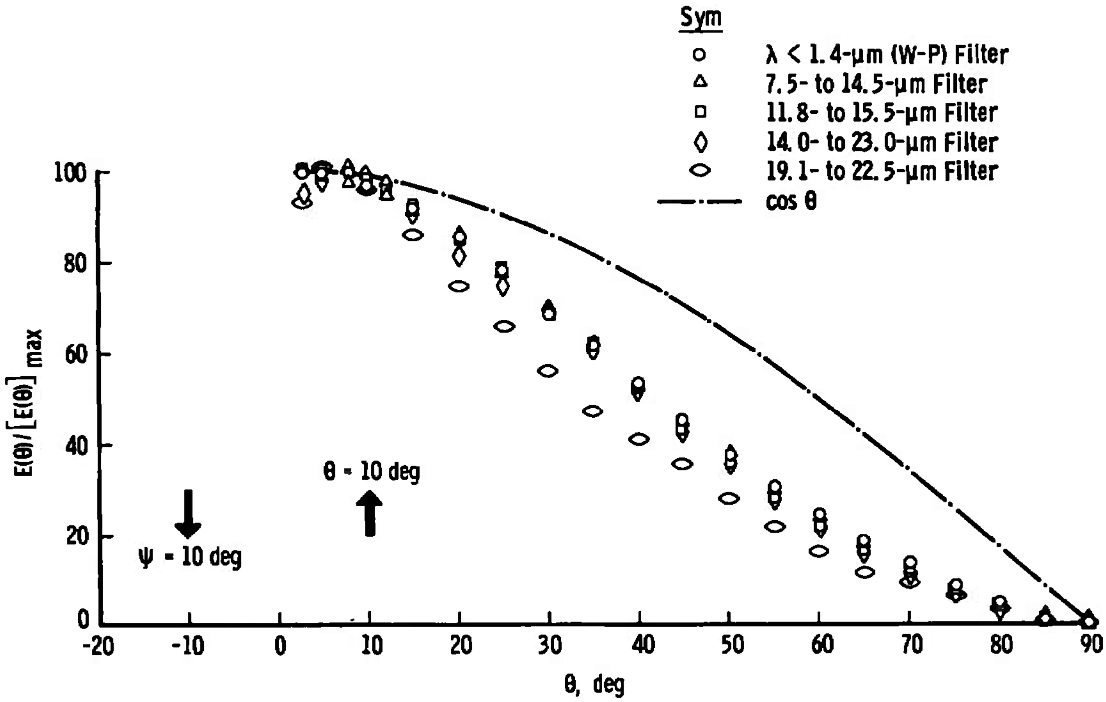


Figure 10. Angular distribution of radiation reflected from a glass bead surface overcoated with evaporated gold.

4.1.4 Chemically Etched Surfaces

Chemically etched aluminum surfaces were prepared using a sodium hydroxide (NaOH) solution at a temperature of 100°F for an etch time of 45 min. After being removed from the NaOH, the sample was normalized using a 50-percent nitric acid solution. This changed the appearance of the sample from black to an aluminum color. Visually, the etched surfaces appeared to be roughened but uniform. The reflection distribution for this type of surface showed evidence of strong backscattering. This can be observed in Fig. 11 where all the curves have a strong increasing slope near the test surface normal as θ is decreased. It is believed that if measurements in the back quadrant could have been made the curves would have peaked along the direction of the incoming radiation. For this particular surface, there was no appreciable difference for the various wavelength bands. In all cases, however, the departure from cosine was considerable. It is seen that no specular reflection was observed; thus the surface did diffuse the radiation, but in a somewhat irregular pattern. It is believed that this surface could be used as a diffuser if it were only required to diffuse the radiation since no problem would be encountered by specular reflection.

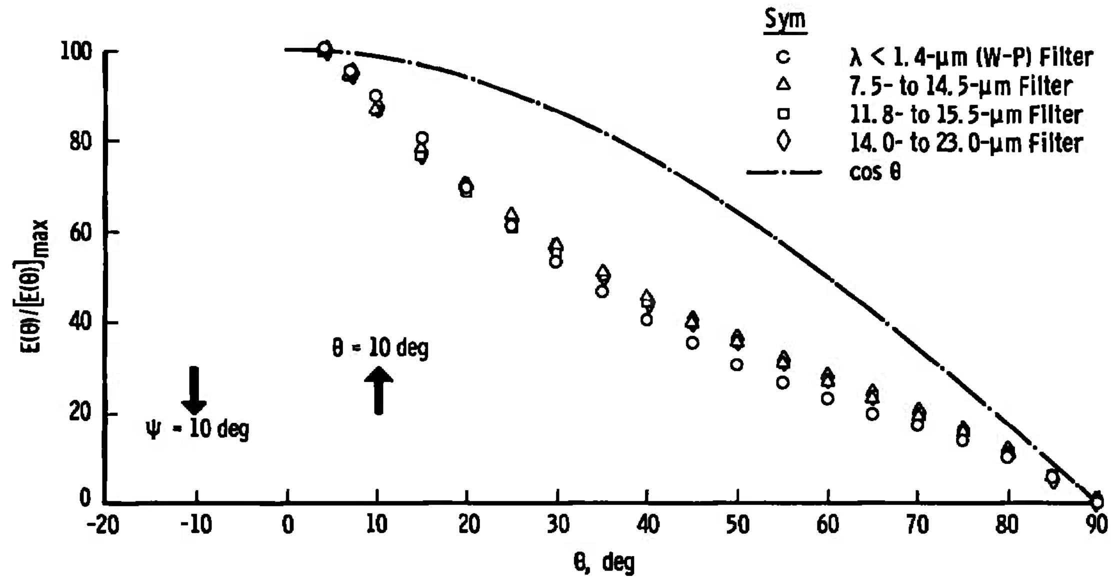


Figure 11. Angular distribution of radiation reflected from an etched aluminum surface.

4.1.5 Gold-Coated Grit Surfaces

The last type of diffuser surface tested was prepared by F. G. Sherrell (Ref. 7) in the following way: (1) a silicon carbide grit was sprinkled on a freshly prepared epoxy-coated surface until the epoxy was completely covered by the grit; (2) after the epoxy hardened and the excess grit was removed, another very thin layer of epoxy was used to overcoat the grit, with all excess epoxy being removed; (3) after the final epoxy coating hardened, the surface was coated with vacuum-deposited gold. Sherrell (Ref. 7) states that this coating has an infrared reflectance of approximately 94 percent and because of its roughness is a good diffuser although no angular distribution measurements were made.

After these surfaces were obtained, a surface roughness measurement was made using a profilometer to determine the mechanical surface roughness. When 180 grit was used, the rms surface roughness was found to be between 8 and 10 μm. The distribution of the reflected radiation from this surface is shown in Fig. 12 and, as can be seen, is essentially diffuse for all of the wavelength intervals investigated. No definite trend is seen with regard to wavelength effects as the surface seemed to diffuse all wavelengths about equally at least for an incidence angle of 10 deg. From Fig. 12 it would appear that based on these measurements, the gold-coated grit surface would be an excellent choice. However, it was found from other similar-type surfaces that the final coat of epoxy before gold coating

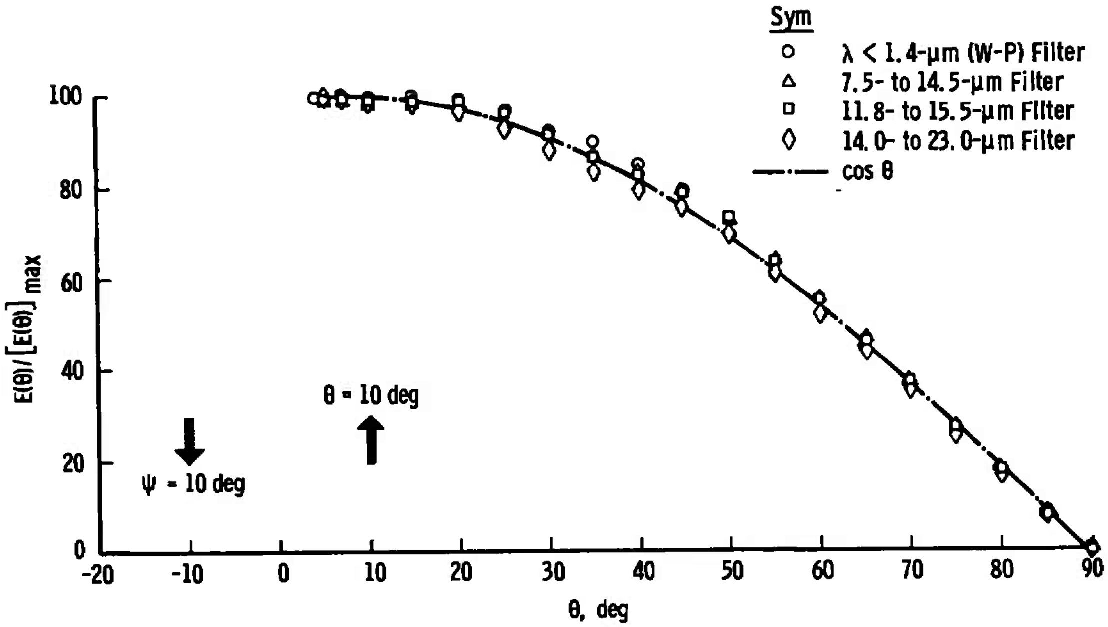


Figure 12. Angular distribution of radiation reflected from a gold-coated 180-grit surface.

could make the surface much smoother, with the result that the surface would become more specular. Thus, the preparation of these surfaces is somewhat critical as far as the actual distribution of reflected radiation is concerned. The effect of the final coating of epoxy on top of the grit before gold overcoating is shown in Fig. 13. Sample 1 was prepared as discussed previously and was essentially diffuse for the 14.0- to 23.0-μm wavelength band. Sample 2 was prepared by omitting the final thin layer of epoxy before gold coating, and as can be seen the distribution of the radiation reflected was considerably different and did not resemble a Lambert surface at all. Evidently the final layer of epoxy eliminates some of the radiation traps present prior to the application of the final epoxy coating. This effect then increased the probability that the radiation would be multiply reflected and increased the reflectance in addition to making the distribution more uniform.

Sample 3 in Fig. 13 was for a relatively thick final coat of epoxy on the grit before gold overcoating was accomplished. This in effect made a much smoother surface, with the result that the distribution is somewhat specular. It should be added that the thickness of the final coat is critical, as there was little difference in the thickness of the final epoxy coats for samples 1 and 3. Although the gold-coated 180-grit surfaces appeared to be diffuse reflectors, there were indications in Fig. 12 of some nonuniformity problems which could have been masked by the relatively large collection angle required for obtaining these measurements. The uniformity measurements for this type of surface are discussed in the following section.

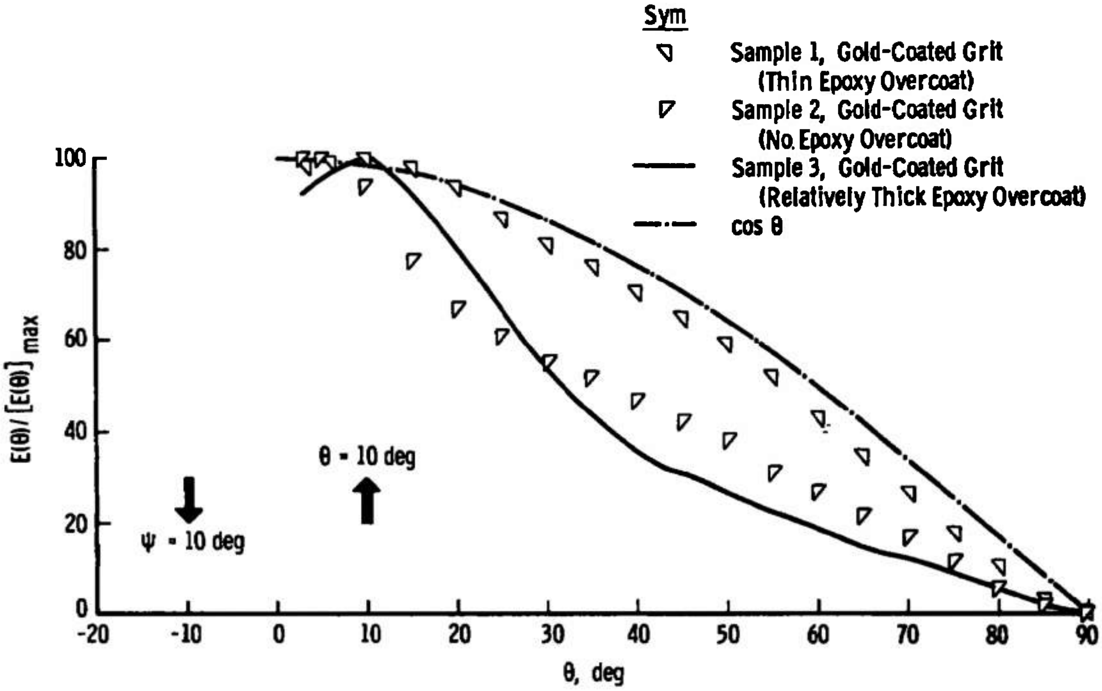


Figure 13. Effect of final epoxy coating on distribution of radiation reflected from gold-coated grit surfaces.

4.2 UNIFORMITY MEASUREMENTS

Uniformity across the diffuser surface will be important since in the mobile target system the irradiated spot will be swept across the entire diffuser surface. Since relatively large collection angles were required to achieve a workable signal level at the longer wavelengths, it was not possible to make uniformity measurements in the infrared wavelengths using the small spot sizes and collection angles required for use in the 7V and Mark I tests. Uniformity measurements were made for various samples using the test apparatus shown in Fig. 4. Rather than infrared radiation, a He-Ne laser source (0.6328 μm) irradiated the samples for spot sizes varying from 0.0125 (0.31 mm) to 0.2 in. (5.08 mm) in diameter through a variable aperture. Similarly, the angle of collection by the lens inclined at an angle of 15 deg from the test surface normal was restricted to either 0.5 or 1.5 deg total included angle. These ranges of spot sizes and collection angles correspond to that proposed for use in 7V and Mark I tests, respectively. The uniformity of a surface was determined by translating the surface in the Y-direction (see arrows in

Fig. 4) while holding the remainder of the optical and detector system fixed. For the powdered-type surfaces such as sodium chloride, sulfur, and potassium bromide (KBr), there was essentially no change in the photomultiplier tube output as the sample was translated in a direction perpendicular to the laser beam (see Fig. 14). This is the detector response that is desired for a diffuser, one independent of position on the sample. The other output shown in Fig. 14 is for the etched aluminum sample, and as can be seen the variation in the response was about 8 percent. For some applications this variation would be too great. Figure 14 presents data for a spot size of 0.025-in. (0.63 mm) diam and a collecting angle of 1.5 deg. No comparison of the signal levels for the various samples should be made in Fig. 14, since different amplifier gains were used.

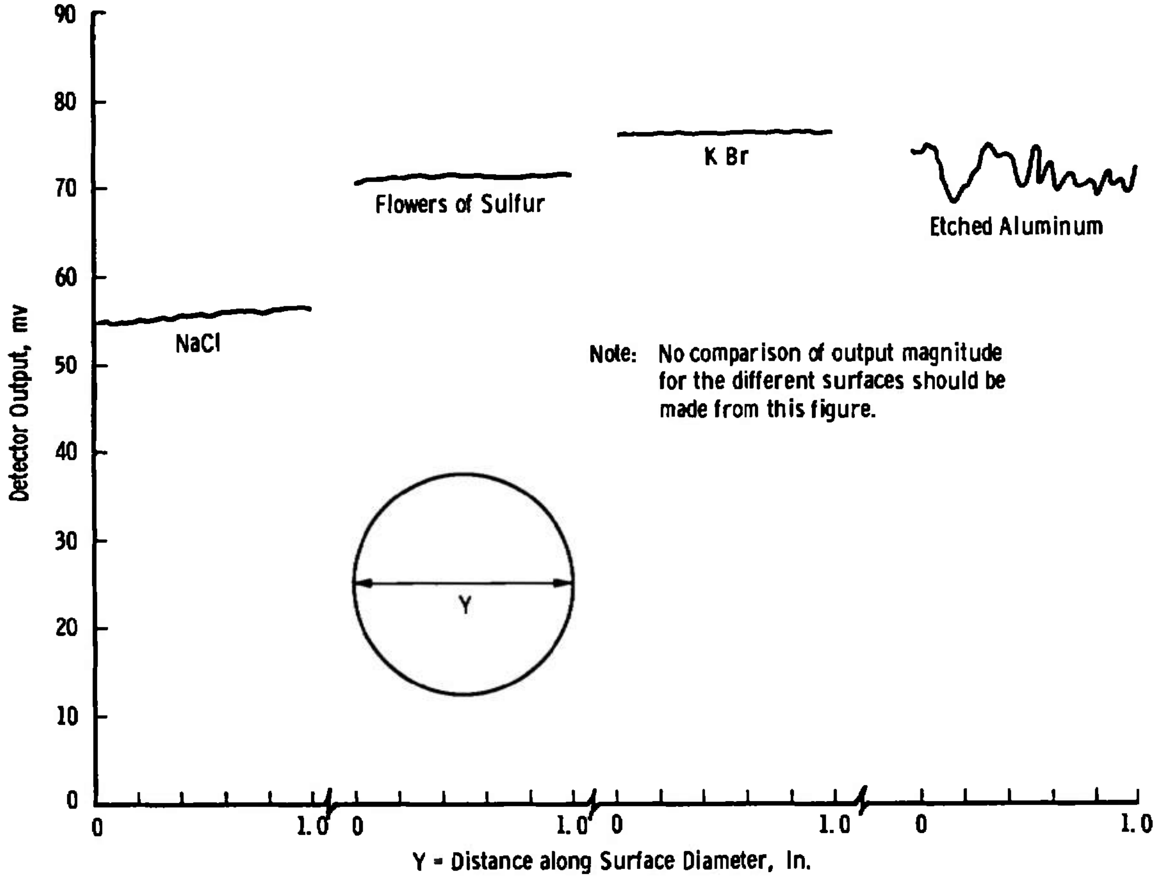
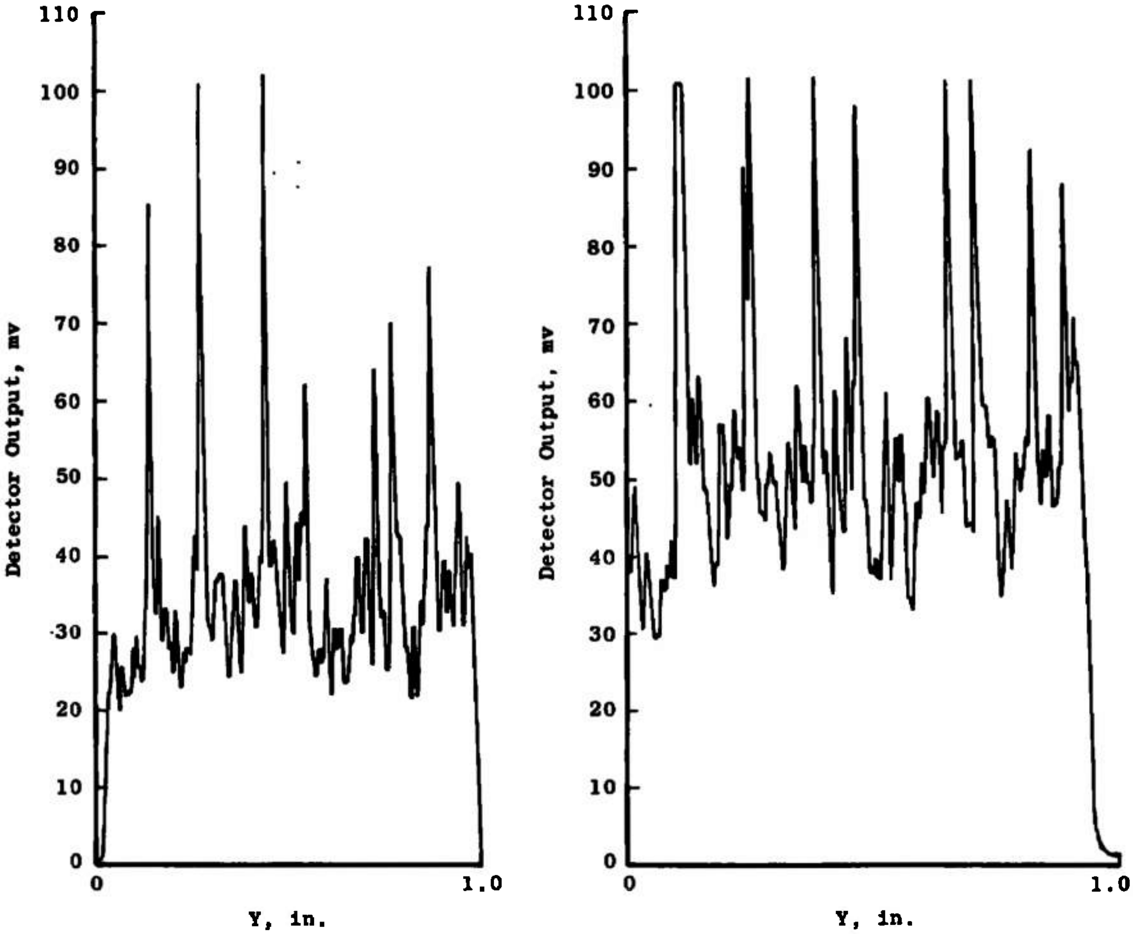


Figure. 14. Uniformity measurements for sodium chloride, flowers of sulfur, potassium bromide, and etched aluminum surfaces.

More detailed uniformity measurements were made for the gold-coated grit samples since this surface showed the most promise as a diffuser based on the angular distribution data. In Figs. 15a and b the irradiated spot size was 0.025 in. (0.63 mm) in diameter, and the collection angles were 0.5 and 1.5 deg, respectively. Similarly, Figs. 15c and d present the same collection angles but for an irradiated spot size of 0.2-in. diam. For

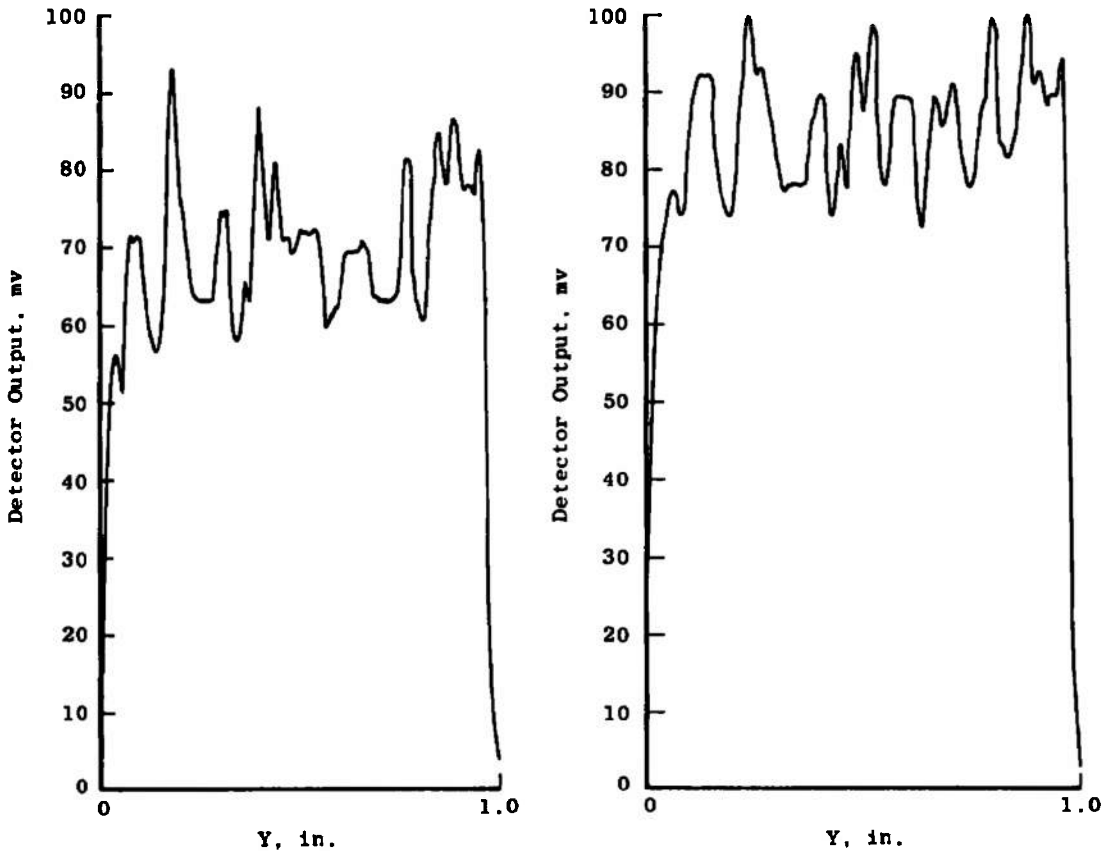


a. Spot size = 0.025 in., included angle β = 0.5 deg

b. Spot size = 0.025 in., included angle β = 1.5 deg

Figure 15. Uniformity measurements for gold-coated 180-grit surfaces.

the 0.025-in. (0.63 mm) spot, the detector output was very nonuniform with many random sharp spikes and was essentially independent of the size of the collection angle. For the 0.2-in. (5.08 mm) spot size (see Figs. 15c and d) the output was considerably more uniform, but still variations of ±20 percent were observed. This particular gold-coated grit sample was prepared using 180-grit silicon carbide particles. Several other grit sizes and combinations were prepared and investigated to try to improve the surface uniformity.

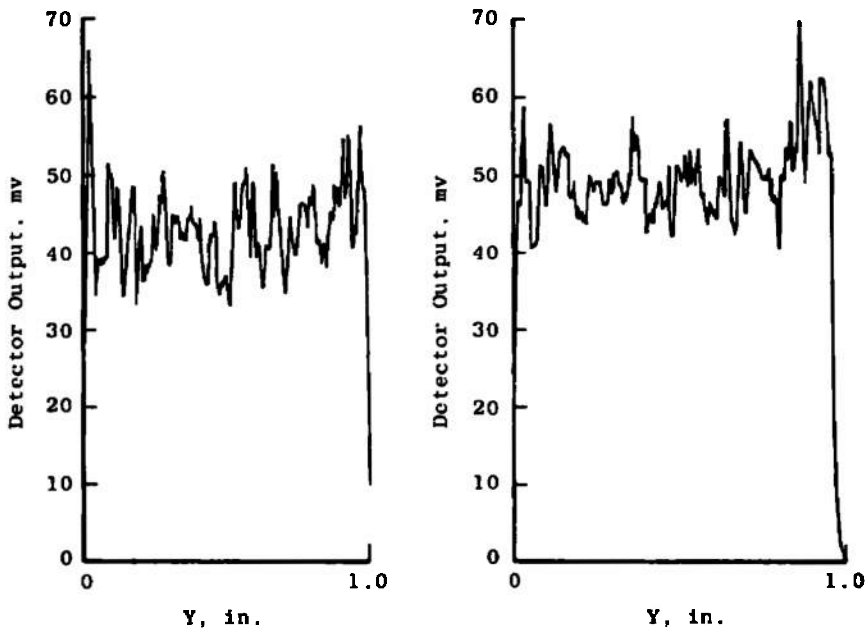


c. Spot size = 0.2 in., included angle β = 0.5 deg

d. Spot size = 0.2 in., included angle β = 1.5 deg

Figure 15. Concluded.

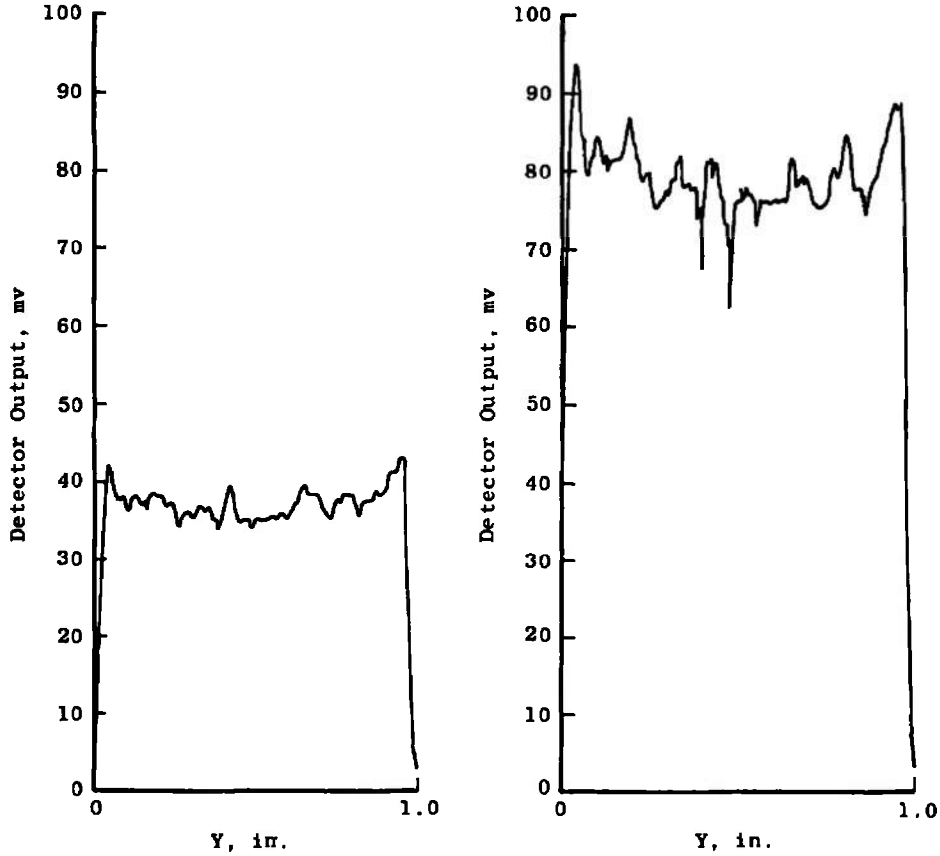
The best such surface developed was one prepared in the previous manner using 180 grit and then applying an epoxy coat, a layer of 320 grit, and a final thin layer of epoxy before the gold coating. The results for this surface are shown in Figs. 16a through d. Figures 16a and b, as previously, are for an irradiated spot size of 0.025-in. (0.63 mm) diam and collection angles of 0.5 and 1.5 deg, whereas Figs. 16c and d are for a spot size of 0.2-in. (5.08 mm) diam for the same collection angles. The results show considerable improvement over that for the previous figure and especially for the 0.2-in. (5.08 mm)-diam spot size and 0.5-deg collection angle, where the variation is approximately ±8 percent. Evidently, the large spot size effectively washes out much of the finer nonuniformities. This same effect has been observed by Voyshvillo and Blinova (Ref. 8), who found that when the beam diameter of the incident radiation was varied from 0.28 to 2.65 mm, the distribution of the reflected radiation changed from very erratic to a very smooth curve. Although the results of Fig. 16 show considerable improvement, the use of these surfaces as IR diffusers in the multiple target system is still questionable.



a. Spot size = 0.025 in., included angle β = 0.5 deg

b. Spot size = 0.025 in., included angle β = 1.5 deg

Figure 16. Uniformity measurements for gold-coated 320-grit surface.



c. Spot size = 0.2 in., included angle β = 0.5 deg

d. Spot size = 0.2 in., included angle β = 1.5 deg

Figure 16. Concluded.

5.0 CONCLUSIONS

The results of this investigation show that of all the materials investigated, the powdered salt coating is by far the most uniform and diffuse surface. Unfortunately, it would present a possible contamination problem in the sensor test chamber because of its powder-like texture. The metallic-coated surfaces diffused the radiation somewhat but either were very nonuniform across the surface or did not approach being a Lambert reflector. For the mobile target application, if a surface is only required to diffuse the radiation without regard to uniformity, either the gold-coated grit surface or the etched aluminum surface could be utilized. The criteria for which type of surface would be most beneficial would depend on the specifications of the users who would be using the target system. It would appear that for a target system such as originally proposed for the 7V chamber only a diffusing type of surface would be required. If there were sufficient energy scattered at all angles for the sensor to detect, it would seem that either the gold-coated grit or the etched aluminum surface would be adequate. For some cases, it would appear that a varying signal such as that observed for the gold-coated grit would be desirable in simulating a target moving through the atmosphere in which there was considerable cloud cover or other absorbing media.

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