

Results of a Round Robin
measurement of spectral emittance
in the mid-infrared

Ronald R. Willey

Opto Mechanik, Incorporated
P. O. Box 640, Melbourne, Florida 32901, USA

Abstract

There has been interest in infrared emittance and reflectance over the years due to space applications, solar energy, thermal imaging, etc. Several laboratories have built instruments to measure the emittance or reflectance of a variety of types of samples in some or all of the region from 1 to 25 micrometers. However, it appears that there are still no traceable standards available to calibrate these instruments. As a result of discussions among interested parties, a Round Robin was organized to circulate a few representative samples to as many laboratories as practical for comparative measurements. The various instruments represent different geometries and approaches to measuring what is expected to be the same physical property. The intent of the endeavor has been to assess the state-of-the-art and gain whatever understanding is possible from differences which are found in the results. It is hoped that: this will allow future improvements in the instruments and techniques, that it will add confidence in the data for the users, and that it can lead to future standards of infrared reflectance and emittance. Many samples were suggested. The final choice was influenced by availability, durability, reproducibility, and ease of measurement by most instruments. Some samples were chosen to be specular and therefore measurable in an absolute sense. Diffuse samples were chosen to test possible variations in the angular response of different instruments. The results are summarized and compared. The contributors were encouraged to describe their instruments and methods in separate papers in order to have a more adequate opportunity to show those details. We believe that some progress has been made as a result of the cooperation, synergism, and serendipity of the project.

Introduction

As a result of discussions at SPIE meetings where the author, Keith Snail, and Gindele, Kohl, and Mast presented papers on the measurement of emittance, the author decided to organize a "Round Robin" measurement of selected samples to assess the state of the art in emittance measurements. All of the laboratories that we were then or later became aware of were invited to participate and also suggest appropriate samples. Table I lists those who actively participated and whose results are included in this paper. A few other laboratories around the world are known to have some related measurement capabilities, but were not able to participate for various reasons. There may also be a few laboratories of which we are not yet aware who could have made some of the measurements. The principle thrust was to measure hemispheric-directional or directional-hemispheric spectral emittance from 1 to 25 micrometers or any part thereof. Supporting measurements of bidirectional reflectance and integrated emissivity were also provided as will be discussed below. There are no commonly accepted standards of diffuse reflectance or emittance over this spectral range, so we have chosen samples to compare as broad a range of conditions as practical in emittance/reflectance and specular versus diffuse reflectance. Since the instruments are all quite different in construction, the degree of agreement on results tends to indicate where the true value is likely to be. It is unlikely that all instruments have made the same systematic errors or erroneous assumptions. Where we find one instrument deviating significantly from the average of the others, it may be cause for examination of the characteristics of that particular instrument. We hope that the work will not be construed as critical of any instrument or user, but serve as a touchstone by which everyone can improve their systems and the state-of-the-art. The process of doing the Round Robin has already proved to be stimulating and enlightening for many of the participants. The extent of the data obtained is too great to be totally dealt with in this one paper. We will concentrate on presenting the results in a form which we hope will allow analysis for cause of errors and possible improvement in future papers by this author and the other participants.

Instruments Used

The instruments used by Gindele(1), Snail(2), and Neu(3) are described in their papers in this same symposium. The first two are integrating sphere types while the third is of the Coblentz hemisphere design. The basic geometry of three more integrating sphere type instruments used in the Round Robin are shown in Figure 1 (Willey), Figure 2 (Tardy), and

Figure 3 (Richter). Figure 4 shows the geometry of the heated cavity or Hohlraum used by Mirtich. Figure 5 shows schematically The COLLECTOR(TM) by SpectraTech which was used by Salisbury. This later is not actually D-H or H-D but a biconical reflectance attachment. We have therefore shown the results separately for their high signal to noise, but not necessarily photometric comparabilty. Figure 6 shows Gindele's configuration. Richter's system is described in more detail in a previous paper(4) as is the Willey instrument(5). We assume that the reader is generally familiar with the concepts of the integrating sphere, Coblentz sphere (ellipsoid), Hohlraum, and biconical ellipsoidal mirror reflectance attachments.

Samples Measured

We appreciate the help of Kevin Carr and Labsphere who provided diffuse gold samples which we understand represent their latest integrating sphere coating for the mid-infrared. We also thank J. Ternay Neu and Surface Optics who provided flame sprayed stainless steel samples which are quite diffuse and approximately a 50% reflector or emitter. However, they are not easily reproducible as we found significant variations from sample to sample. We are indebted to Kieth Snail and the Naval Research Laboratories who prepared ruled gold surfaces, and to Opto Mechanik, Inc. who prepared the balance of the samples.

The samples which we settled upon were as follows

| | |
|---------------------------------------|----------------------------|
| DIFFUSE GOLD | DIFFUSE PROTECTED ALUMINUM |
| FLAME SPRAYED STAINLESS STEEL | POLISHED FUSED SILICA |
| 80 GRIT SiC PAPER (CARBORUNDUM BRAND) | DIFFUSE FUSED SILICA |
| SPECULAR PROTECTED ALUMINUM | RULED GOLD |

The protected aluminum was evaporated aluminum with an aluminum oxide overcoat of about one half wave optical thickness in the visible spectrum (E-Beam Reactively Evaporated). The diffuse fused silica was produced by a method brought to my attention by J. T. Neu. The fused silica was first coarse ground with loose #80 SiC abrasive and then etched for two hours in 50% hydrofluoric acid in water. The resulting surface looked somewhat like disturbed ocean waves on a microscopic scale. Some of the same surfaces were then vacuum coated the same as the specular protected aluminum to produce a diffuse protected aluminum. These samples cover a good range from high to low reflectance and specular to diffuse reflectance with some significant spectral character in the fused silica. I am indebted to J. T. Neu again for bringing to my attention the work of R. J. Champetier(6) on the use of polished fused silica as a reference.

We measured the samples before sending them out, and again when returned. Since our instrument is normally used in a comparative mode, we compared the samples with the diffuse gold sample as a reference. The Ruled Gold sample prepared by Snail is a very useful tool to check angular response in the instrument. It reflects a majority of the normally incident beam specularly at a 45 degree angle to the incident beam. This can be rotated about the surface normal to test the azimuthal uniformity of a system. The data obtained with this sample was too voluminous to include here and is of most value to individual user. Snail may report these results in the fall. In almost all cases, there is a measureable difference in response in the instruments as the orientation of the ruled sample is changed. This points out that we must be careful with samples that have reflectance or emittance which is not symmetric about the surface normal.

We had hoped to get help from those who could measure the absolute reflectance of specular samples but we ran into sample size limitations and availability of the measurements in time for this Round Robin. We did, however, get pertinent data from Professor William L. Wolfe and Kie B. Nahm of the University of Arizona on the BRDF (bidirectional reflectance) of three of the diffuse samples at 10.6 micrometers. We will discuss these below.

Results of Reflectance/Emittance Measurements

Figures 7 through 13 show the basic summary of results of the Round Robin emittance measurements. The data from each participant was entered into a data base and plotted in a common format for comparison. The spectral range was chosen to be in micrometers from 1 to 25 to encompass essentially all of the data ranges of the participants. The photometric scale is plotted in % reflectance which is 1-emittance for opaque samples. The fused silica samples are partially transmissive at wavelengths shorter than about 5 micrometers, and therefore the data in that region is not comparable from instrument to instrument. It would depend on the reflectance characteristics of what was behind the samples. The spectral ranges plotted for each of the participants is as follows:

Gindele 2.0 to 14.2 micrometers

| | | |
|-----------|------|------|
| Mirtich | 1.75 | 14.7 |
| Neu | 1.5 | 25.0 |
| Richter | 1.27 | 5.56 |
| Salisbury | 2.17 | 25.0 |
| Snail | 2.0 | 16.0 |
| Tardy | 2.13 | 20.0 |
| Willey | 2.0 | 19.0 |

The polished fused silica has extensive spectral structure with dramatic and sharp changes from near 0% reflectance to over 70%. The diffuse fused silica has similar features, but reflectance is quasi-Lambertian as compared to specular for the polished sample. These two illustrate good agreement among most instruments, but point out one which might bear some further investigation. The specular protected aluminum show great agreement between all of the instruments except the authors. It points out that we have a lower response for a specular sample than a diffuse sample. This is believed to be due to a lower reflectance from the specular exit port of the sphere which is removable and of apparently of somewhat different surface from the rest of the sphere.

The diffuse protected aluminum and diffuse gold show an interesting spread between instruments which points out the need for further work on the part of almost everyone to refine the state-of-the-art. The flame sprayed stainless steel sample shows somewhat better agreement in the midrange of reflectance and particularly in the shorter wavelengths. The #80 Grit SiC Sandpaper sample has high emittance and some spectral structure. It is not a good thermal conductor which may account for some of the problem which causes the one instrument to give quite different results from the consensus of the rest. For these high emittance samples, it appears that the RMS deviations from the mean are generally within less than 1% of full scale. For low emittance samples, the variations are somewhat greater as of this time.

Figures 14 and 15 are results from the biconical reflection attachment. These show very high relative signal-to-noise as compared to the integrating sphere instruments because of the nature of the geometry. The spectral structure of the SiC Sandpaper is quite clear in Figure 14 and can be compared to that in Figure 13. Please note, however, that Figures 14 and 15 are plotted in linear wavenumbers. Figure 15 is an overlay of all of the other spectra from this instrument. One feature that should be pointed out is the spectral splitting of the 9 micrometer (1100 wavenumber) peak of fused silica. I believe this is due to the wide variety of reflectance angles of this instrument as compared to the others that are working at near normal incidence. Reference 6 goes into more detail on the reflectance of fused silica as a function of angle of incidence.

BRDF Measurements on Diffuse Samples

Professor Wolfe and Kie Nahm kindly measured the bidirectional reflectance distribution functions of three of the samples at 10.6 micrometers for 10 and 30 degree angles of incidence. Figures 16 and 17 summarize these results. For those of us with integrating spheres, there has been concern for some time over the possible tendency of diffuse gold (the interior of the sphere) to become less Lambertian and more specular at longer wavelengths. The ideal Lambertian reflector would appear to be a flat horizontal line at $1/\pi$ on Professor Wolfe's plots of BRDF. It can be seen that the surfaces are not perfectly Lambertian, but they may not be as specular as some of us feared. This area and the effects of incipient specularity need much more study and analysis. J. Ternay Neu's measurements(3) may also add some information of value.

Integrated Spectral Emissivity

There was an interest on the part of Mr. Merle Persky to see how the results of a Gier-Dunkle DB-100 emissometer would compare with the data from this Round Robin. Gindele and Willey were able to provide the integrated emissivity of the spectral emittance versus wavelength as compared to a black body at room temperature. These results are compared in Table II. This data shows an error in the Willey data for specular aluminum which is consistent with the problem described above. The table also shows that the DB-100 data is always a few percent lower than the average of the Gindele and Willey systems that are in reasonable agreement.

Conclusions

This Round Robin measurement of emittance and its supporting measurements has provided the groundwork for significant potential improvement in the state-of-the-art of this type of measurement. The degree of consensus has been demonstrated amongst a variety of instruments in a variety of laboratories around the western world. This consensus is both encouraging from its confirmation of basic agreement but perhaps surprising in that many of the participants might have believed they knew the "true" emittance of any sample

to within less than 1%. It is apparent that there is room for further advances and that some absolute and reproducible standards are needed for diffuse reflectance/emittance. We believe that every participant has gained valuable knowledge from their involvement, there has been extensive stimulating thought and discussion, and some data is now available to improve each system. We hope that this will form the basis of ongoing and expanding refinements in the field of emittance measurement.

References

1. K. Gindele, M. Kohl, "Measurement of near-normal/hemispheric reflectance and directional emittance in the mid-IR," SPIE Proc 807-22 (1987)
2. K. A. Snail, L. Hanssen, "IR diffuse reflectometer for spectral, angular, and temperature resolved measurements," SPIE Proceedings 807-21 (1987)
3. J.T. Neu, O. E. Myers, "Hemispherical directional ellipsoidal IR spectroreflectometer," SPIE Proceedings 807-23 (1987)
4. W. Richter, "Fourier transform reflectance spectroscopy between 8000cm⁻¹ and 800cm⁻¹ using an integrating sphere," Applied Spectroscopy, Vol.37, No.1, (1983)
5. R. Willey, "An instrument to measure spectral emittance from 2 to 20 micrometers," SPIE Proceedings 590-36 (1985)
6. R. J. Champetier, G. J. Friese, "Use of polished fused silica to standardize directional polished emittance and reflectance measurement in the infrared," SAMSO Report TR-74-202, SAMSO, Los Angeles, CA 90045, 9 August 1974

TABLE I
ACTIVE PARTICIPANTS IN THE ROUND ROBIN

| | |
|---|-----------------------|
| K. Gindele Universitat Stuttgart Institut Fur Theorie Der Elektrotechnik Pfaffenwaldring 47 7000 Stuttgart 80 WEST GERMANY | 011-49-711-685-7278# |
| Keith Snail, Code 6520 Naval Research Laboratory Washington, DC 20375-5000 | 202/767-3069 or -1360 |
| Herb Tardy, Div. 1824 Sandia National Laboratories Albuquerque, NM 87185 | 505/844-2109 or -7038 |
| Prof. William L. Wolfe Optical Sciences Center The University of Arizona Tucson, Arizona 85721 | 602/621-3034 |
| J. Ternay Neu Surface Optics Corp. 9929 Hibert, Suite C San Diego, CA 92131 | 619/578-8910 |
| Michael Mirtich, MS 16-1 NASA Lewis Research Center Cleveland, Ohio 44135 | 216/433-5616 |
| Dr. John W. Salisbury, MS-927 US Department of the Interior Geological Survey Reston, VA 22060 | 703/648-6382 |
| Dr. W. Richter Physikalisch-Technische Bundesanstalt Post Fach 33 45 3300 Braunschweig West Germany | 011-49-531-592-3300 |
| Merle J. Persky Massachusetts Institute of Technology Lincoln Laboratory Lexington, Massachusetts 02173-0073 | 617/863-5500 x4904 |

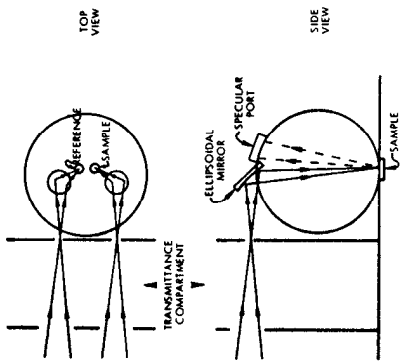


Figure 1. Willey integrating sphere geometry

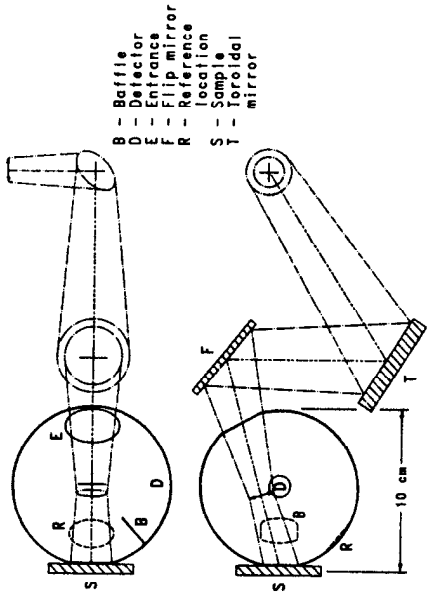


Figure 2. Tardy integrating sphere configuration

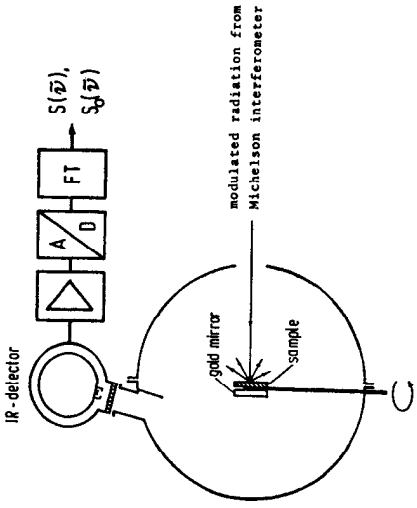


Figure 3. Richter integrating sphere system

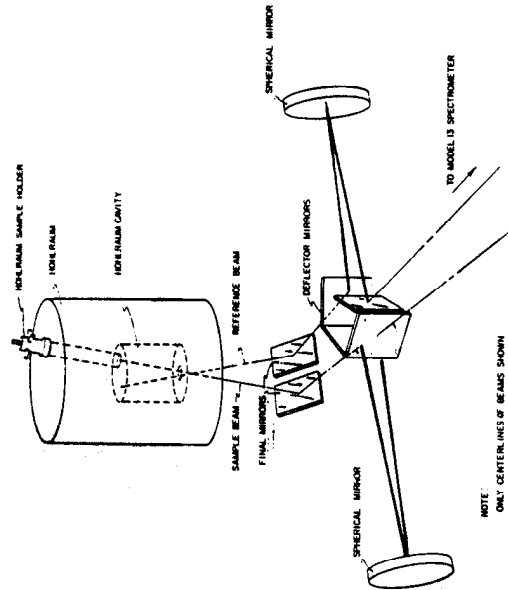


Figure 4. Hohraum used by Mirtich

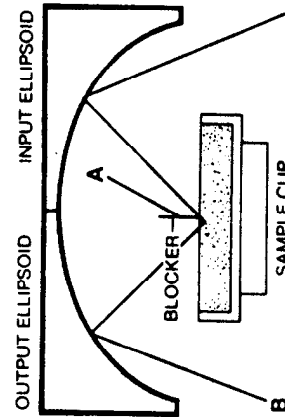


Figure 5. Geometry of biconical reflectance attachment, used without blocker by Salisbury

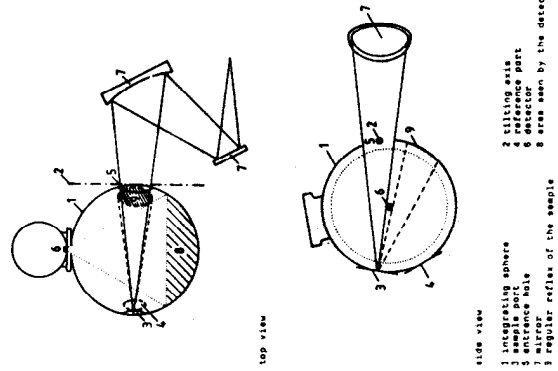


Figure 6. Gindele integrating sphere geometry

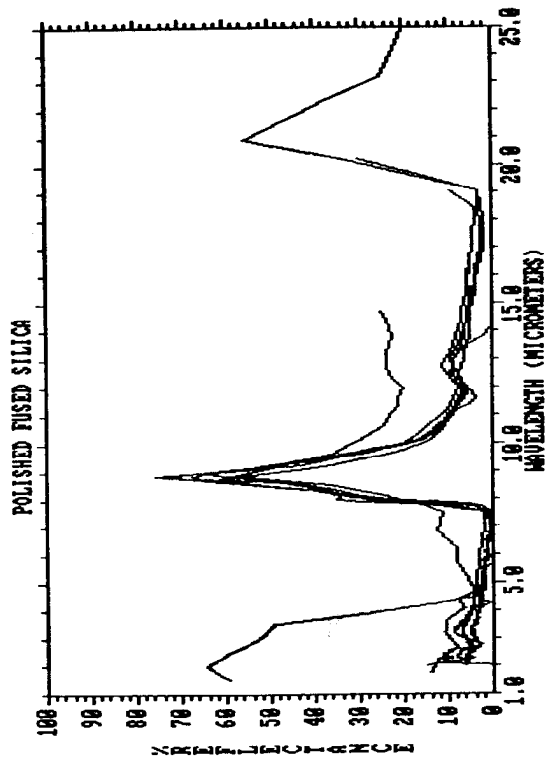


Figure 7. Overlay of all reflectance measurements of Polished Fused Silica

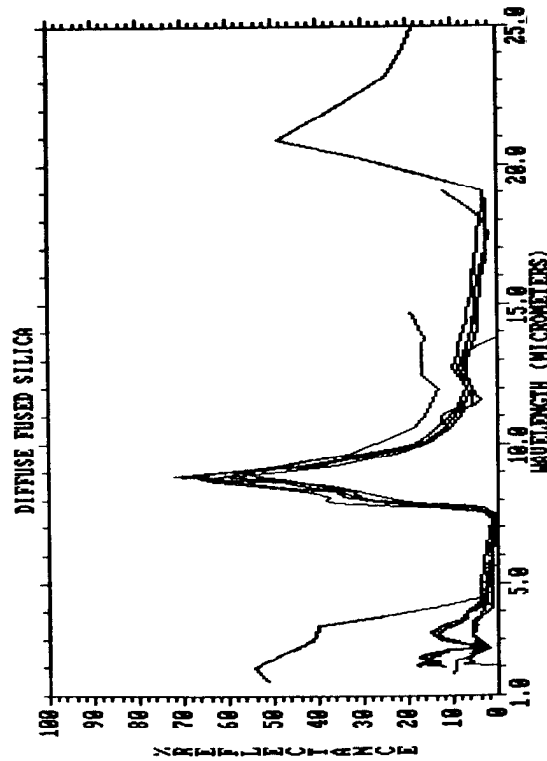


Figure 8. Overlay of all reflectance measurements of Diffuse Fused Silica

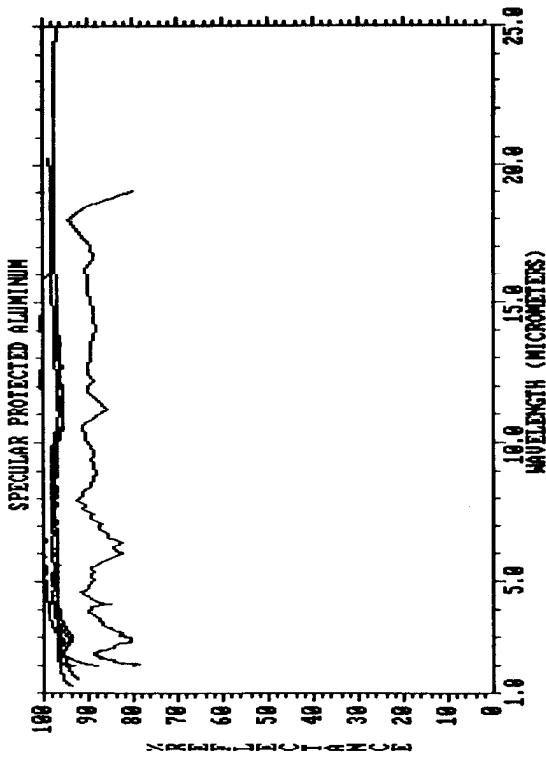


Figure 9. Overlay of all reflectance measurements of Specular Protected Aluminum

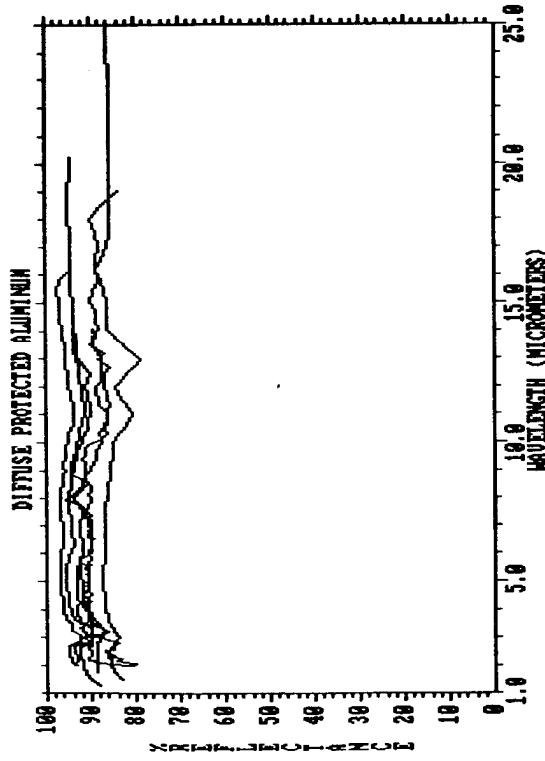


Figure 10. Overlay of all reflectance measurements of Diffuse Protected Aluminum

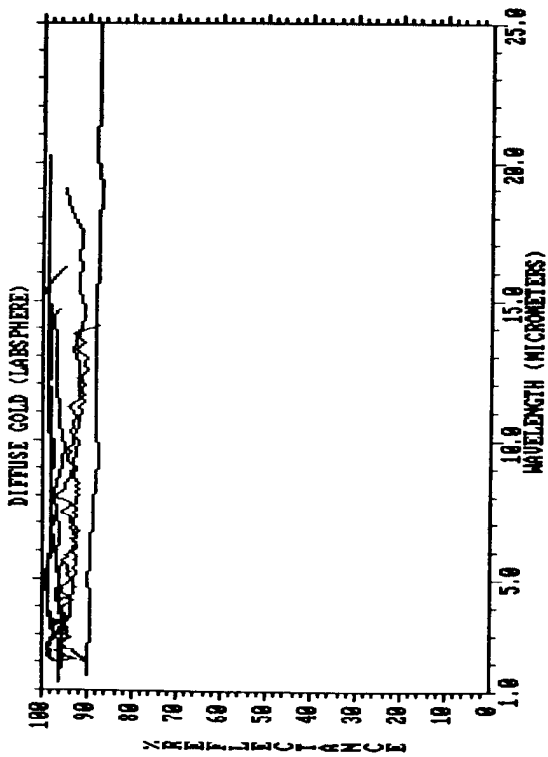


Figure 11. Overlay of all reflectance measurements of Diffuse Gold (Labsphere)

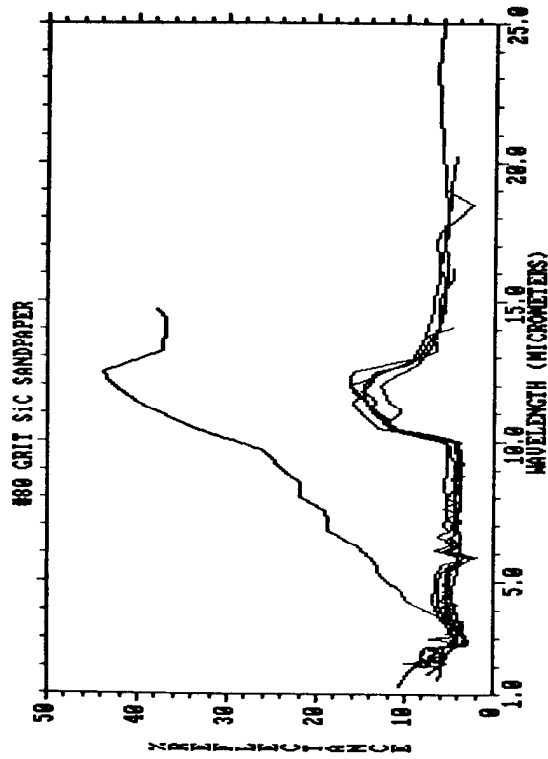


Figure 13. Overlay of all reflectance measurements of #80 Grit SiC Sandpaper

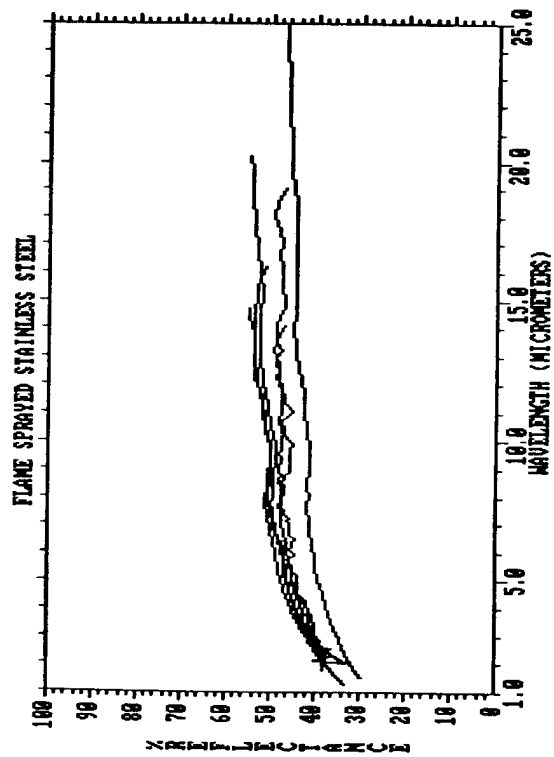


Figure 12. Overlay of all reflectance measurements of Flame Sprayed Stainless Steel

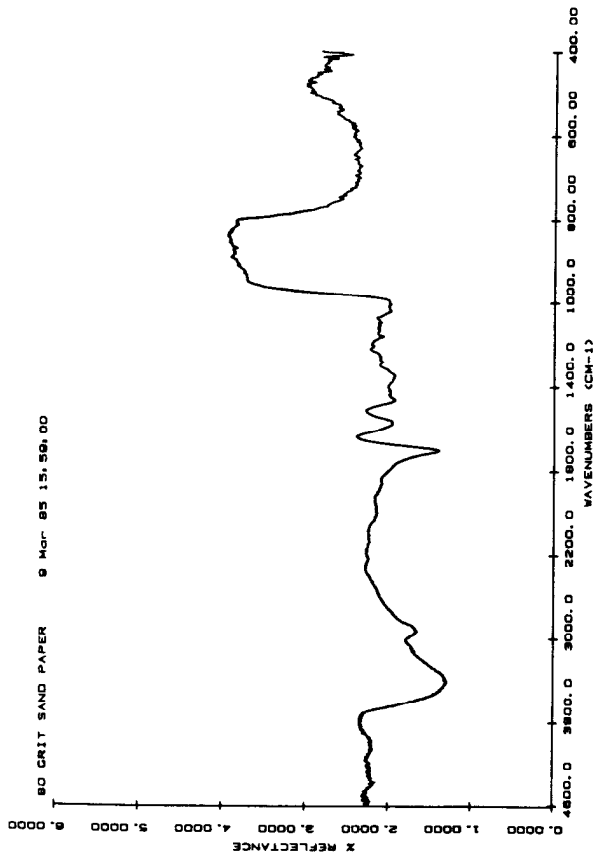


Figure 14. Biconical reflectance of #80 Grit SiC Sandpaper

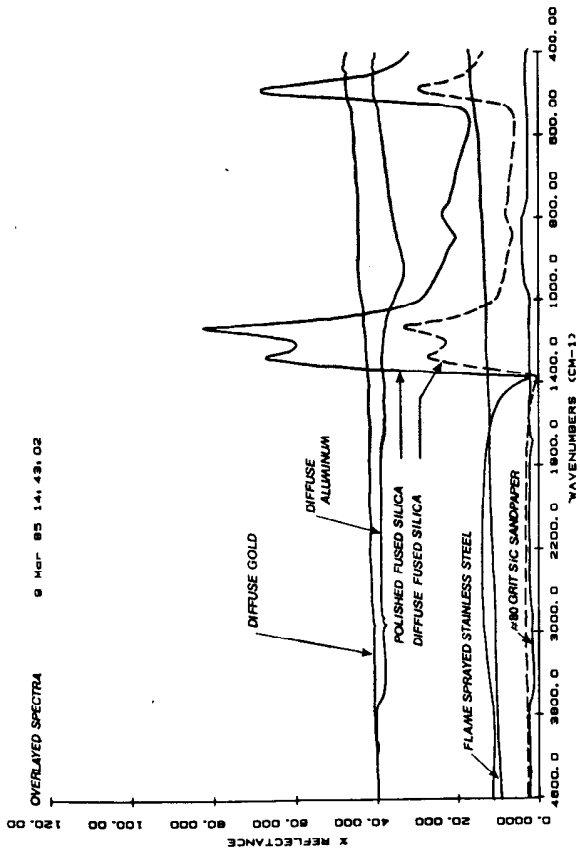


Figure 15. Overlay of biconical reflectance spectra of a variety of specular and diffuse samples

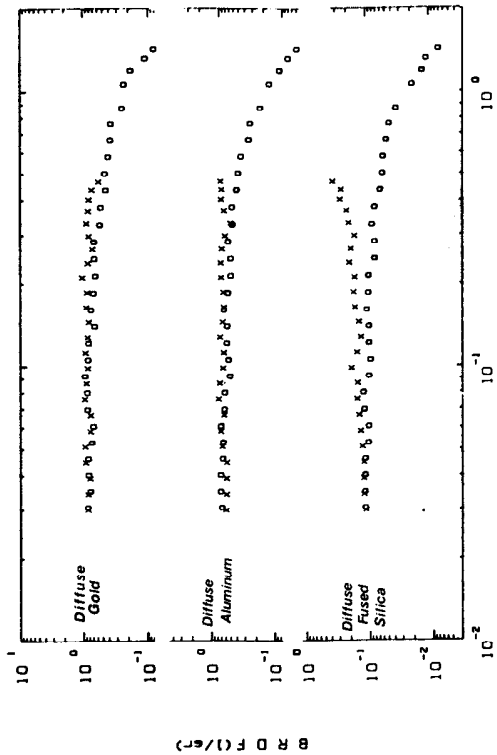


Figure 17. BRDF of three samples at 10.6 micrometers for 30 degree incident angle, x = B-B0>0, o = B-B0<0

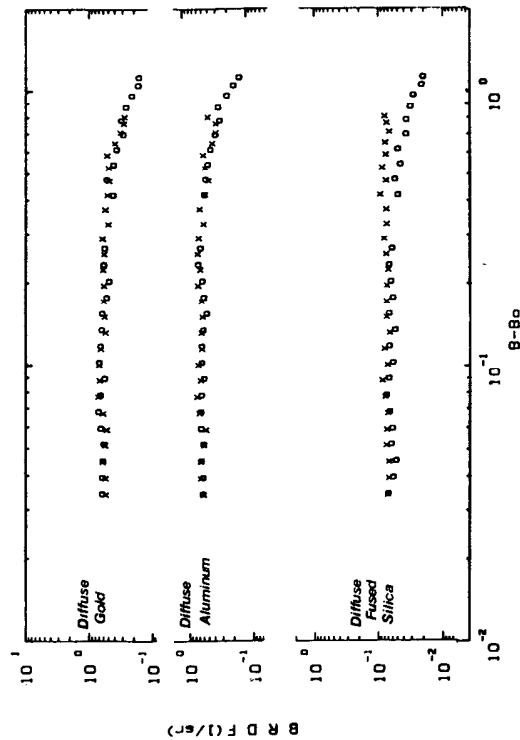


Figure 16. BRDF of three samples at 10.6 micrometers for 10 degree incident angle, x = B-B0>0, o = B-B0<0

TABLE II
THREE MEASURES OF INTEGRATED EMITTANCE

| INSTRUMENT USER | Gier-Dunkle DB-100 Persky | Bruker (Modified) Gindele | Willey 318 Willey |
|----------------------|---------------------------|---------------------------|-------------------|
| SAMPLE: Diffuse Gold | .060 | .078 | .075 |
| Diffuse Aluminum | .080 | .110 | .110 |
| Specular Aluminum | .020 | .032 | .110 |
| Flame Sprayed S.S. | .500 | .523 | .535 |
| Diff. Fused Silica | .820 | .859 | .882 |
| Polished Fused Sil. | .810 | .850 | .890 |
| #80 Grit SIC Paper | .890 | .913 | .937 |