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SURVEY OF MATERIAL FOR AN INFRARED-OPAQUE COATING

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ABSTRACT

More than 40 reflectance spectra in the range from 20 to 500 μm have been obtained of a variety of coatings, binders, and additives to identify promising components of an infrared-opaque coating for the Space Infrared Telescope Facility. Certain combinations of materials have a specular reflectance below 0.1 throughout the spectral range measured. In addition to estimating the optical constants of several combination coatings, this survey also supports three qualitative conclusions:

1. Promising "off-the-shelf" binders of different additives are Chemglaze Z-306, ECP-2200, and De Soto Black.

2. Carbon Black is very effective reducing far-infrared reflectance.

3. The far-infrared reflectance from coatings containing #80 SiC grit is consistently lower than that from similar coatings containing TlBr powder.

INTRODUCTION

A better infrared-opaque coating is needed to reduce stray "light" inside far-infrared telescopes such as SIRTF (Space Infrared Telescope Facility) and COBE (Cosmic Background Explorer). Previous work[1,2] has shown that most available optical-black baffle coatings become quite reflective in the far-infrared (FIR). Further, a very effective[2] visible and near-IR coating, 3M Black Velvet (Nextel), has been permanently removed from production by its manufacturer. Hence this study was undertaken to identify promising components for a new IR-opaque telescope coating which will function throughout the SIRTF wavelength range (2 to 700 μm). The work was done under NASA purchase order at the Santa Barbara Research Center, Goleta, CA.

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*This work was done while R.V.H. was at Hughes Santa Barbara Research Center, Goleta, CA.

TECHNIQUES

Spectral measurement

A Nicolet 8000 Fourier transform interferometer at the Santa Barbara Research Center was fitted with a reflectance attachment to measure specular reflectance at 17° incidence. Three beam splitters (6.25, 12.5, and 50 μm thick), two sources (Globar and mercury arc), and two detectors (TGS and germanium bolometer) were used to measure overlapping spectra from 20 to 500 μm. The detectors subtended approximately 1.2×10^{-3} sr at the sample and the spectral resolution was generally about 8 cm^{-1}.

Coating application

An attempt was made to produce a standard thickness of all materials measured. Unfortunately, dry thickness measurements were not performed until after much work had been done. Only then was it found that different evaporation volumes produced significantly different dry coating thicknesses in several cases.

1. Substrates. All substrates were smooth pieces of 6064 T-6 aluminum, 2.5 cm square and 0.32 cm thick which had been lapped optically flat. Each substrate was cleaned with Xylenes before a coating was applied. For liquids, several drops were placed on the substrate and then spread to a uniform thickness with the modified razor blade shown in Figure 1. Powdery solids were "chopped" or "fluffed" with a razor blade to help them produce a uniform layer when spread with a broad blade. The microballoons and SiC grit could be smoothly spread by the surface tension of a few drops of propahol. Approximately 13 drops of 4% General Electric #7031 Adhesive and Insulating Varnish in alcohol were used to bind solids to the substrate. A control spectrum from 100 to 500 μm showed that the resulting thickness of adhesive spread over the substrate produced no detectable absorption.

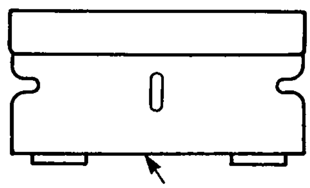

Figure 1. Coating spreading device.

2. Primers. For Chemglaze Z-306 (hereafter called Chemglaze), the substrate was primed with one coat of Chemglaze 9924 Wash Primer. ECP-2200 (hereafter called ECP) and Black Suede binders required no primer.

3. Black Suede. Combinations of this coating with other materials did not cure well and cracked when drying. A second coat was applied which caused some

bubbling of the first coat near the edges of the substrate. The Black Suede did not prove to be a hardy binder when used with the above application technique.

Coating combinations

Components were combined in the following proportions in three binders: Chemglaze, ECP-2200, and Black Suede.

<u>In the Chemglaze Binder</u>:

1. A volume of carbon black approximately 1/4 in. by 1/4 in. by 1/4 in. was mixed with a volume of Chemglaze approximately twice as large to make the Chemg/CB combination.

2. The same volume of carbon black as in combination (1) above was mixed with 0.02 g of #80 SiC grit. This combination was mixed with an approximately equal volume of Chemglaze Z-306 to make the Chemg/CB/SiC combination. This combination was replicated.

3. The same volume of carbon black as in combination (1) above was mixed with 0.03 g of TlBr powder. This combination was mixed with an approximately equal volume of Chemglaze to make the Chemg/CB/TlBr combination. This combination was replicated.

4. The replicas were given a heavy spray of Teflon Wet Lubricant (DuPont Co., Wilmington, DE) to investigate its effect as an antireflection overcoat. The results of this experiment will be reported elsewhere.[3]

<u>In the ECP-2200 binder</u>:

1. A volume of carbon black approximately 1/4 in. by 1/4 in. by 1/4 in. was mixed with approximately 10 drops of ECP to make the ECP/CB combination.

2. A volume of carbon black approximately 3/8 in. by 1/4 in. by 1/4 in. was mixed with 0.03 g of #80 SiC grit. This combination was mixed with 25 drops of ECP, and then thinned with approximately 8 drops of Xylenes to make the ECP/CB/SiC combination. This combination was replicated.

3. The same volume of carbon black as in ECP (2) above was mixed with 0.04 g of TlBr powder. This combination was mixed with 25 drops of ECP-2200 and thinned with about 8 drops of Xylenes to make the ECP/CB/TlBr combination. This combination was replicated.

4. The replicas were given a heavy spray of DuPont Teflon Wet Lubricant to investigate its antireflection effect.

In the Black Suede binder:

The Black Suede described below is the standard 3M mixture thinned 3:1 with methyl-ethyl ketone.

1. A volume of carbon black approximately equal to that put in the ECP binder (2) above was mixed with a volume of Black Suede which was approximately twice as large to make the combination BlkSu/CB.

2. A volume of carbon black approximately equal to that put in the ECP binder (2) above was mixed with 0.03 g of #80 SiC grit. This combination was mixed with an approximately equal volume of Black Suede to make the BlkSu/CB/SiC combination.

3. A volume of carbon black approximately equal to that put in the ECP binder (2) above was mixed with 0.04 g TlBr. This combination was mixed with an approximately equal volume of Black Suede to make the BlkSu/CB/TlBr combination.

Thickness and roughness measurement

Coating thickness was initially measured with a 100-power Unitron microscope near the center of each sample. Later, a few of these measurements were significantly corrected by comparator measurement at the edge of the sample. The large grain size of some additives produced very rough, nonuniform coatings where there was often a large range in measured thickness. For rms surface roughness σ less than 8 μm, roughness was measured on a METROsurf-type 180 profilometer. Greater roughness was measured by visual comparison with the GE Standard Roughness Specimens, #8651831G2. The uncertainty in thickness is estimated at ±25 μm. In roughness, the uncertainty is estimated at ±20% with the profilometer, and ±3 μm with the standards. Pertinent data from this section are given in Table 1.

Spectral errors

In replotting some of the spectra, a certain amount of smoothing has been unavoidable. The absolute reflectance levels measured have been found to differ on subsequent scans using different beam splitters by as much as 0.06 in units of absolute reflectance. Hence the measured reflectance is considered accurate to approximately ±0.05. Reflectances below 0.04 are often in the noise region of the instrument. For this reason, spectra which appear to slowly approach zero with decreasing wavelength are often displaying only the measured noise level. Independent measurements[2] of several coatings show that the reflectance at short wavelength often falls very rapidly with decreasing λ, sometimes to values as small as 10^{-5}.

SURVEY OF SELECTED COATINGS, AVAILABLE BINDERS, AND ADDITIVES

The thickness d, and rms surface roughness σ, of the coatings and additives studied here are listed in Table 1 together with the manufacturer/originator's names and a brief description of the coating construction technique. In the figures, the coating thickness and roughness in microns are given in parentheses (d, σ) following each coating name.

Figure 2 shows the 20-500 μm spectra of five interesting coatings which are not available for use as a binder material. NBS Black is made by an electroless nickel plating process and is very thin. Ball Black is a similar process but somewhat thicker. LMSC Black is a multilayered, graded-index coating in which optical interference between the several different layers produces obvious fringes or "channeled spectra." Stycast-1090 is a thick epoxy potting compound filled with hollow glass microballoons. The gross effect of thickness is clearly demonstrated by these four coatings. Cornell Black is a rough, thick coating which displays many of the characteristics of a good IR-opaque coating. It uses 3M Black Velvet (Nextel 101-C10) as a binder for large #80 grit) particles of SiC. Unfortunately, the 3M Company has permanently discontinued production of its Black Velvet (Nextel) coating. These last two spectra demonstrate the noise level measurement, described above under Spectral errors, at $\lambda \leq 150$ μm.

Figure 3 shows the 20-240 μm spectra of six off-the-shelf coatings, any one of which could be used as a binder for other materials. Interference fringes of various frequency and strength are quite evident. Noise in some of the data was smoothed during replotting onto common graph scales.

Consideration of the spectra in Figure 3, and coating roughness, thickness, and composition, led to the selection of three coatings for further test as potential binders of other materials. ECP-2200 was selected because it contains silicates that absorb strongly between 20 and 50 μm and it does not already contain carbon black. Chemglaze Z-306 was selected because it is known[4] to have a low rate of outgassing. ECCOSORB CR-110 was rejected because it showed a relatively high IR reflectance for its thickness. Black Suede was selected because its reflectance was relatively low for its roughness and thickness. In hindsight, De Soto Black might have been a better choice because application problems later developed with Black Suede and its testing was discontinued. De Soto Black should be studied further.

Figure 4 displays the FIR spectra of five materials which were potential additives to the binders selected above. The three materials with the lowest reflectance are also the thinnest of the group, leading to automatic selection for further test. It may be noted that TlBr becomes relatively reflective beyond 400 μm.

SIGNIFICANT COMBINATIONS OF MATERIALS

In this section the effect on FIR reflectance of combining different additives with the selected binders is investigated. Table 2 lists the thickness and

Table 1. Commercially Available Materials

Coating Name	Manufacturer (Originator)	Sample Number	Construction Technique	d(μm)	σ(μm)
NBS Black	National Bureau of Standards (C. Johnson)		Electroless nickel plating with nitric acid etch	2	0.3
Ball Black	Ball Brothers		Electroless nickel plating	10	6
LMSC Black	Lockheed Palo Alto Research Lab (J. Grammer)		Multilayer, graded-index coating with several polyurethane layers containing increasing amounts of carbon black	200	1
Stycast 1090	Emerson & Cuming, Inc.	20	Black epoxy resin filled with glass microballoons	540	1.5
Cornell Black	Cornell University (J. Houck)		3M Black Velvet (Nextel) filled with SiC grit of 200-300 μm major dimensions	100	30
ECP-2200	3M Company (B. A. Benson)		Small jagged silica particles in a silicone binder containing proprietary black dye	55	4
Chemglaze Z-306	Hughson Chemicals		Carbon black pigmented polyurethane coating	40	2
Floquil				43	1.3
ECCOSORB CR-110	Emerson & Cuming, Inc.	19	Black epoxy resin filled with iron powder	180	1
Black Suede	3M Company	17	Black iron oxide in a polyurethane coating	100	3
De Soto Black	De Soto Inc.		Carbon black pigmented polyurethane coating	120	6.6
Nylon Flock	Cellusuede Products	11	Sprinkled onto surface and smoothed with blade	280	20
T2R-261 Micro-spheres	KMS Fusion	6	Sprinkled onto substrate, smoothed with 2 drops propanol	750	13
T2R-364 Micro-spheres	KMS Fusion	7	Sprinkled onto substrate, smoothed with 2 drops propanol	1000	13
TlBr powder	Harshaw Chemical Co.	12	Sprinkled onto surface and smoothed with blade	200	5
Carbon Black Raven 1255	Columbian Chemicals Inc.	13	Sprinkled onto surface and smoothed with blade	200	5
#80 SiC	General Abrasives, Inc.	8	Sprinkled onto surface, smoothed with 2 drops propanol	500	25
#180 SiC	General Abrasives, Inc.	9	Sprinkled onto surface, smoothed with 2 drops propanol	140	6

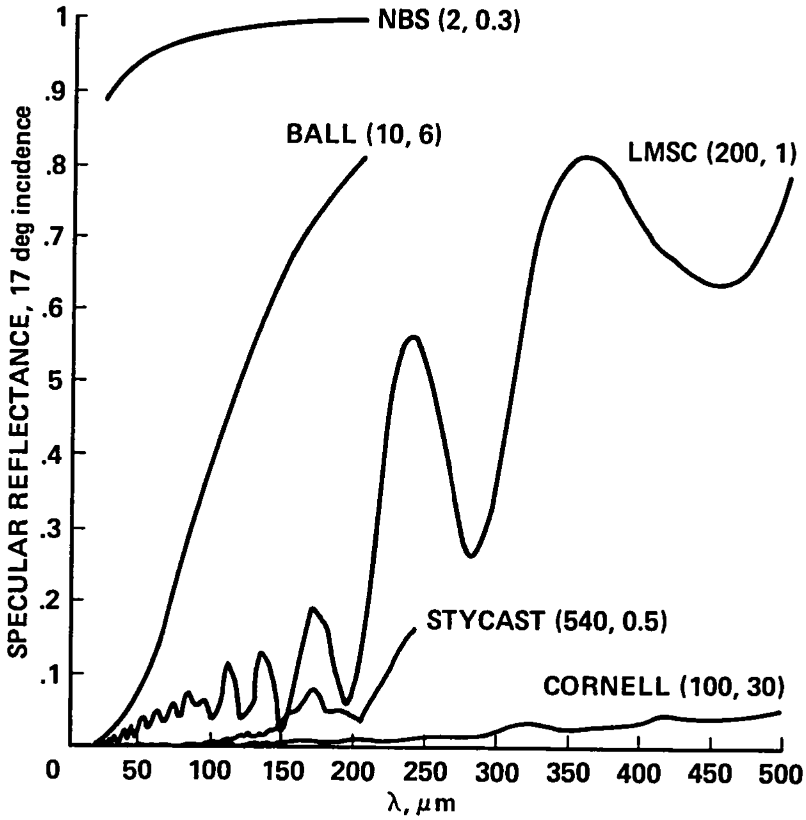

Figure 2. Reflectance spectra of several selected coatings.

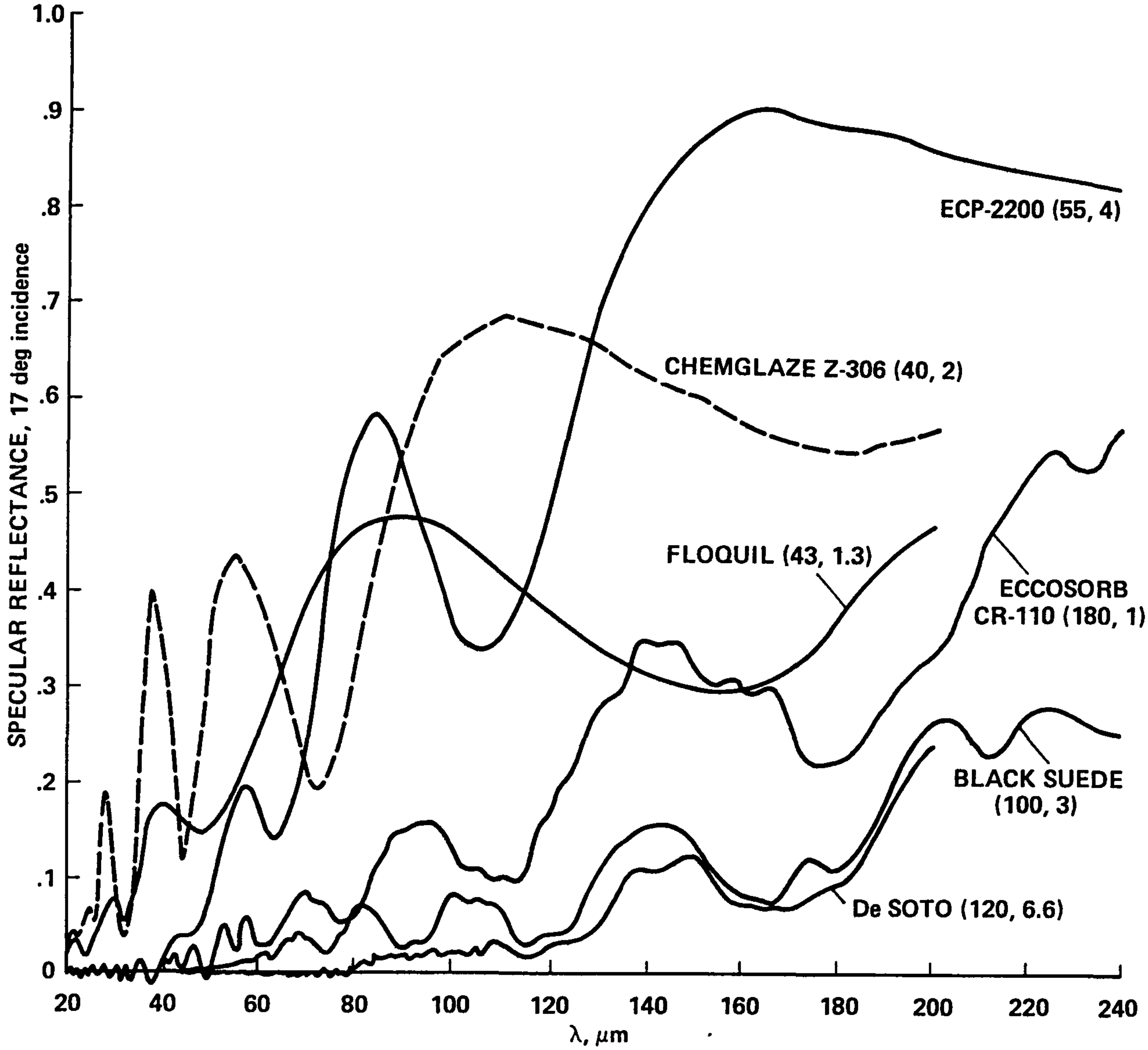

Figure 3. Reflectance spectra of available, off-the-shelf coatings.

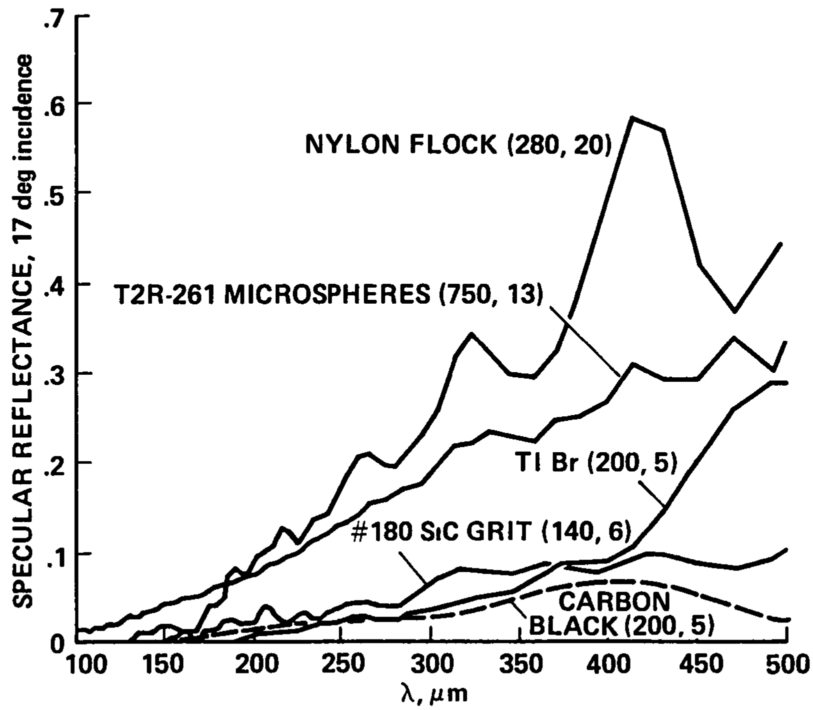

Figure 4. FIR spectra of potential additives.

Table 2. Combination Coatings

Sample Number	Combination	d(μm)	σ(μm)
15	ECP/CB	160	7.5
24	ECP/CB/#80 SiC	200	30
25	ECP/CB/#80 SiC/Teflon	215	25
26	ECP/CB/TlBr	85	6
45	ECP/CB/TlBr/Teflon	40	3
28	Chemg/CB	200	7.5
29	Chemg/CB/#80 SiC	180	20
30	Chemg/CB/#80 SiC/Teflon	160	30
31	Chemg/CB/TlBr	130	10
32	Chemg/CB/TlBr/Teflon	210	7.5
42	BlkSu/CB/#80 SiC	300	15
44	BlkSu/CB/TlBr	200	5

roughness of the various combinations which were applied with the special razor blade shown in Figure 1. As noted earlier, the result of the Teflon antireflection experiment will be reported elsewhere.[3]

The addition of carbon black to both ECP and Chemglaze not only thickens the coatings significantly, but also reduces the reflectance dramatically. This can be easily seen by comparing Figure 5 with Figure 3. Since the increased thickness in this case is known to be due solely to carbon black, it is not surprising that the reflectance of these combinations approaches that of an approximately equal amount of pure carbon black, as shown by the lower spectrum in Figure 5. Since equal

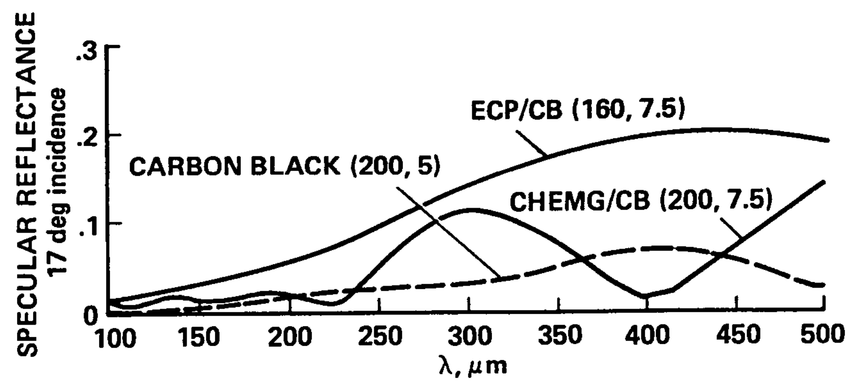

Figure 5. FIR spectra of two selected combinations compared to carbon black.

volumes were added to the two commercial coatings, the lower reflectance of the Chemglaze combination is attributed to the initial presence of carbon black in the commercial product, as indicated in Table 1.

Figures 6a-c present spectra of three-component coatings and are intended to help assess the effect of TlBr versus #80 SiC grit as the third antireflection component. In each figure the coating containing SiC grit has significantly lower FIR reflectance. It is clear that the coatings containing SiC are predictably rougher (by more than 3 times) than those with TlBr powder and that this correlates positively in all three cases with lower FIR reflectance. Unfortunately, the greater roughness also correlates with greater thickness in two out of the three cases, and this alone would qualitatively explain the lower reflectance. One case, Figure 6b, where the SiC coating is rougher yet slightly thinner than the TlBr combination, argues strongly for a greater FIR opacity of the #80 grit on some basis other than simple absorption.

Coatings containing SiC grit may be expected to have lower FIR reflectance on the basis of the following considerations. At wavelengths beyond its strong restrahlen peak near 140 μm,[5] TlBr has a large complex index of refraction (see Table 4 in the next section). Since it is in a powder form, it mixes homogeneously with the other components of the coating and thus increases the average refractive index (by approximately 25%). SiC grit on the other hand, is composed of large (~100-300 μm) particles which do <u>not</u> mix homogeneously with the other parts of the coating and so do not increase the average index of refraction. Hence the TlBr combination may be expected to have a greater upper surface reflectance because of its higher index. In addition, the large SiC particles distributed throughout the volume of the absorbing binder act as randomly reflecting facets because their refractive index (see next section) is somewhat larger than that of the carbon black loaded binder and their size is about the same as the wavelength. Thus the large particles disperse the specular beam within the volume of the absorbing binder which

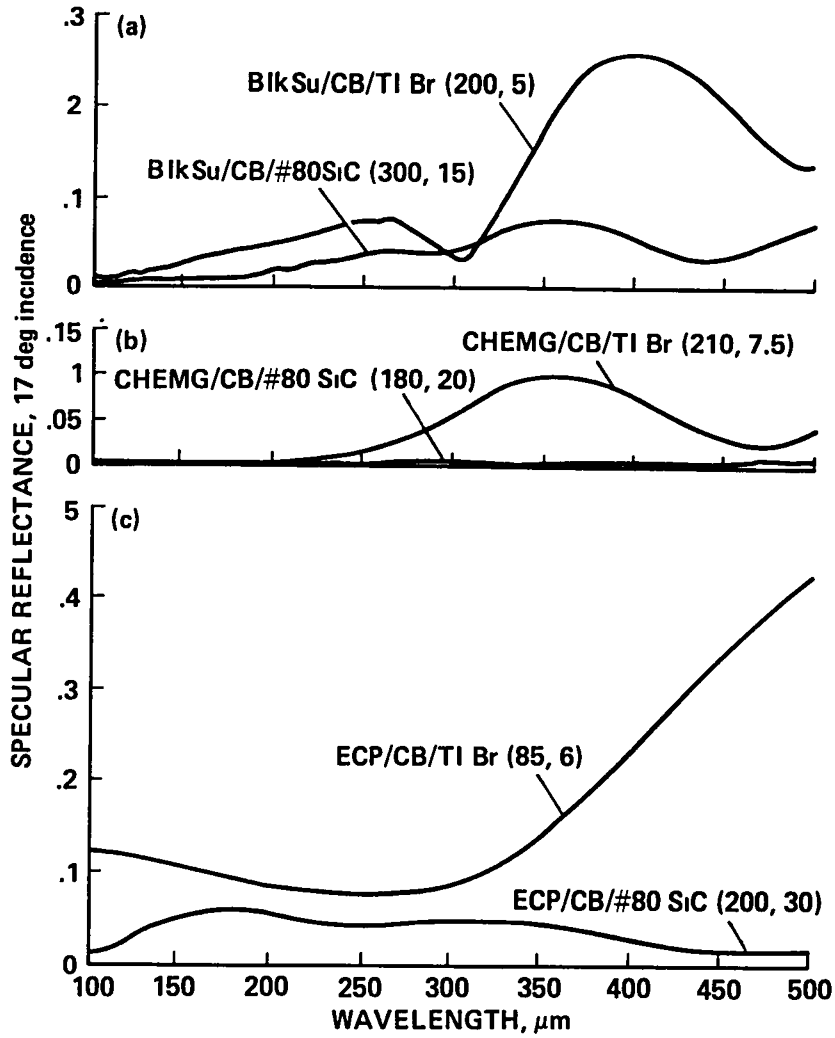

Figure 6. FIR spectra of combination coatings.

leads to a longer path length and thus greater absorption. Protrusion of the particles through the upper surface increases the surface roughness (as already noted) which also decreases the first-surface specular reflectance.[6] Finally, the large size of the grit alone often leads to a thicker coating in order to cover the SiC particles. All of these factors operating to varying extents are probably responsible for the consistently lower FIR reflectance demonstrated in Figure 6.

ESTIMATE OF OPTICAL CONSTANTS

In as much as this is primarily a survey of material and selected combinations, the optical constants of the combination coatings will be estimated rather than determined from a least-squares modeling procedure. The estimates are based on the volumes and weights of the additives given earlier, and upon the optical constants of 3M Black Velvet (Nextel) when loaded with increasing amounts of carbon black as determined by Grammer et al.[1] It was found in an earlier paper[2] that the real refractive index of 3M Black Velvet (n = 1.37) is similar to that of ECP (1.48) and Chemglaze (1.38) over a wavelength range from 66 to 262 μm. When such coatings are loaded with a considerably higher index material such as carbon black (n ≈ 2), the differences in the resulting average index become even smaller. For this reason Grammer's data with Black Velvet can be used here for the index of the carbon-black-loaded binder in place of the measured optical constants of the combinations Chemg/CB and ECP/CB.

The wet density and the nonvolatile, or dry, fraction of the wet coating weight can be found from the product literature. With the volumes given earlier for each sample, the dry weight of each binder used can be calculated. The percent additive in each dry combination coating can then be found from the dry additive weights. These data are listed in Table 3. The percentage of carbon black in just the dry

Table 3. Percent of Dry Weight

Combination	#15	#25	#45	#28	#30	#32
	ECP/CB	ECP/CB/ SiC	ECP/CB/ TlBr	Chem/CB	Chem/CB/ SiC	Chem/CB/ TlBr
Volume of binder used, cc	0.11	0.27	0.24	0.40	0.35	0.40
Wet density, g/cc	1.21	1.21	1.21	.953	.953	.953
Dry fraction	.48	.48	.48	.275	.275	.275
WEIGHTS:						
Dry weight binder, g	.064	.157	.157	.105	.092	.105
Dry weight carbon black	.056	.056	.056	.042	.042	.042
Dry weight additive	---	.03	.04	---	.02	.03
PERCENTAGES:						
% CB in binder only	47	26	26	29	31	29
% CB in the combination	---	23	22	---	27	24
% Additive in the combination	---	12	16	---	13	17

combination binder coating is needed in order to enter Grammer's Figure 4 to obtain the optical constants of carbon-black-loaded Black Velvet. Then the optical constants $\binom{n}{k}$ of the combination coatings made with homogeneously dispersed TlBr powder can be estimated by the weighted mean

$$\binom{n}{k} = \left(\begin{matrix}\%\ \text{dry}\\ \text{additive}\end{matrix}\right)\binom{n}{k}_{\text{additive}} + \left(1 - \begin{matrix}\%\ \text{dry}\\ \text{additive}\end{matrix}\right)\binom{n}{k}_{\text{CB loaded binder}} \quad (1)$$

where of course the complex index of refraction is $(n \pm ik)$.

Thalium bromide has a very strong absorption (restrahlen) band[5] centered near 140 μm which may explain why its IR and FIR optical constants are not well known. For the IR we take McCarthy's 24-μm value[7] of $n = 2.3$, and the statement in Armstrong and Low (Table 1, Ref. 8) that the transmission of a one-third millimeter thick sample is less than 1% beyond 80 μm. From this statement, exponential absorption leads to a value for $k \approx 0.09$. In the FIR (350-400 μm), the square-root of the dielectric constant at 30 m is 5.5. With that as a lower bound on n, Hadni's[5] room temperature reflectance spectrum and the Fresnel reflectance formula

$$R = \frac{(n - 1)^2 + k^2}{(n + 1)^2 + k^2} \qquad (2)$$

then lead to an estimate of the FIR optical constants of TlBr of $n \approx 6.2$ and $k \approx 1$.

As noted earlier, the large grains of #80 SiC grit do not disperse homogeneously throughout the binder coating, hence Equation 1 cannot be used when SiC is the additive. The only alternative is to use the optical constants of the appropriate carbon-black-loaded binder and assume that the inhomogeneous facets of SiC can be handled in some other manner. For comparison, the optical constants of SiC given by Spitzer[9] are $n \approx 3.3$ and $k \approx 0$.

With the percentages given in Table 3, the optical constants of TlBr and of carbon-black-loaded Black Velvet, Equation 1 can then be used to estimate the optical constants of the combination coatings. The resulting values are given at two representative wavelengths in Table 4. It may be noted that the larger FIR values are generally due to increased values of both the additive (TlBr) and the binder.

Table 4. Estimated Optical Constants

Sample No.	Combination	~75 μm		350-400 μm	
		n	k	n	k
	SiC	3.5	0	3.2	0
	TlBr	2.3	.09	6.2	1
15	ECP/CB (47% CB)	1.8	.9	3.35	1
25	ECP/CB/SiC	1.3	.35	2.25	1.1
		(SiC is not homogeneously dispersed)			
45	ECP/CB/TlBr	1.45	.3	2.9	1.1
28	Chemg/CB (29% CB)	1.55	.8	2.95	1.1
30	Chemg/CB/SiC	1.55	.8	2.95	1.1
		(SiC is not homogeneously dispersed)			
32	Chemg/CB/TlBr	1.7	.5	3.5	1.1

CONCLUSIONS

Specular reflectance spectra have been obtained of a variety of coatings, binders, and additives in the spectral range from 20 to 500 μm. Throughout this range certain combinations have a reflectance below 10%. The optical constants of several combination coatings were also estimated. In addition, this survey supports the following three qualitative conclusions:

1. Promising "off-the-shelf" binders of different additives are Chemglaze Z-306, ECP-2200, and De Soto Black.

2. Carbon black is a very effective additive for reducing FIR reflectance.

3. The FIR reflectance from coatings containing #80 SiC grit is consistently lower than that from similar coatings containing TlBr powder. Several identifiable factors mostly associated with the large particle size of the grit are probably responsible for this result.

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16 Abstract

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