Optical Characterization of Absorbing Coatings for Sub-millimeter Radiation

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Abstract. Experimental results are presented on the specular and diffuse reflectance (BRDF) of a variety of surfaces that can be used to absorb sub-millimeter radiation. Although the emphasis of this study is on the development and optical characterization of black absorbing coatings, intended for use in the spectrometers aboard the FIRST satellite, also some convenient materials for laboratory use are discussed. The here presented data have been collected room temperature

Introduction. The optical properties of the coatings have been studied in the 0.1 - 0.9 mm wavelength region in order to comply with the wavelength bands covered by the HIFI and PACS spectrometers. Optical characterization has been performed using monochromatic light at selected wavelengths across the 100-900 micron wavelength region. (96.5µm, 118.8µm, 184.3µm, 496µm and 889µm) created by an optically pumped far-infrared laser.

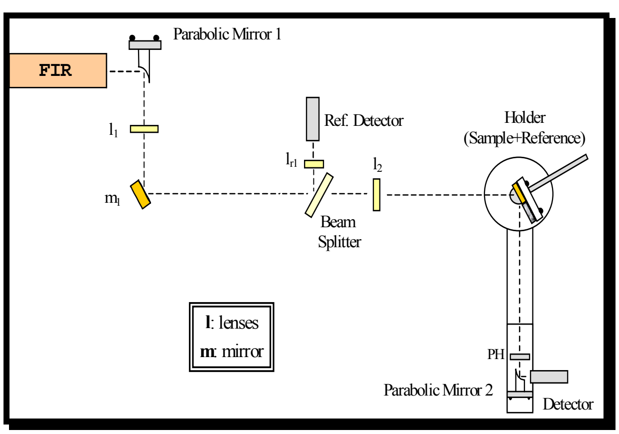

Fig.1 *Experimental set-up*

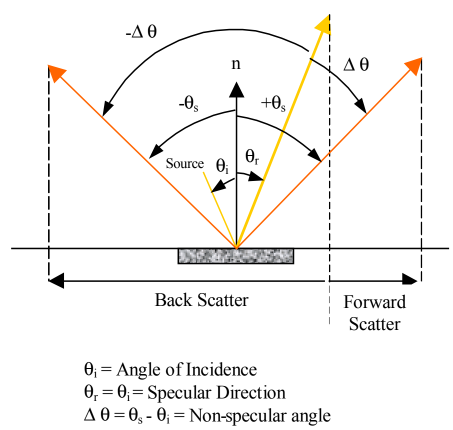

Fig.2 *Angular relations in the optical plane*

Measurements on samples for a range of directions of incident and reflected light and for different polarization directions have been done. The influence of variations in shape and intensity of the laser beam has been eliminated by comparing all samples with reference samples that have been characterized accurately. Experiments have been performed at room temperature, employing the experimental setup shown in Fig. 1. In Fig. 2 the definition of various angles in the optical plane is given. The Bi-directional Reflectance Distribution Function (BRDF), used to describe the scattering properties of surfaces, is defined as the reflectance per unit projected detector solid angle as a function of the azimuthal coordinates θ and ϕ. It is given in units of inverse steradians (sr^{-1}) and is usually measured in the plane of incidence (ϕ=180°) versus the non-specular angle Δθ,

$$BRDF(\Delta\theta,\lambda,\theta_i)=\frac{P_s(\Delta\theta,\lambda,\theta_i)}{P_o(\lambda,\theta_i)\Omega\cos\theta_s}$$

Here P_s is the power, diffusely scattered by the surface, in the direction θ_s, and P_o the total incoming optical power at the sample. The factor $\Omega\cos\theta_s$ (in general $\Omega \approx 8\times10^{-3}$ sr in our experimental set-up) is the projected detector solid angle. For a non-absorbing perfect diffuse scatterer, a so-called Lambertian surface, the BRDF is independent of direction and equals π^{-1}. A reference sample basically consists of a gold-coated rough surface, acting as a near Lambertian reflector, accurately calibrated against the total reflection of a gold-coated mirror.

The near Gaussian like sub-millimeter beam is shaped such that the waist (with a $1/e^2$ diameter of 10-15 mm) is situated at the sample surface in to assure a well-defined angle of incidence of the light. To average out the effects of the non-perfect “random roughness” of the coating surfaces, especially important for very rough surfaces, the samples are rotated around their surface normal at a speed of about 300 rpm. The scattered light is detected with a (room temperature) 2 mm diameter pyroelectric detector (Eltec) with homemade sensitive read-out electronics [1].

Experimental results.

Commercial absorbers. To allow for a comparison with the coatings developed for FIRST, we have also studied the scattering characteristics of two commercially available materials used as (sub-) millimeter absorbers. Marconi LAO 5, a carbon loaded soft open foam microwave absorber, and (Tessalating Terahertz) RAM from Thomas Keating Ltd., injection molded 25mm squares with sharp pyramidal surface formed in conducting plastic. In Fig. 3 the specular reflection of both samples is shown at 4 different wavelengths; the reflection increases with increasing wavelength and angle of incidence. In Fig. 4 the BRDF of LAO 5 is shown for 3 wavelengths. Its value is seen to increase again with wavelength and angle of incidence. Evidence of specular reflection is observed, that is, a peak develops for Δθ = 0. The BRDF value of 0.02-0.04 means a Total Hemispherical Reflection (THR) of 6-12% (-12- -9 dB).

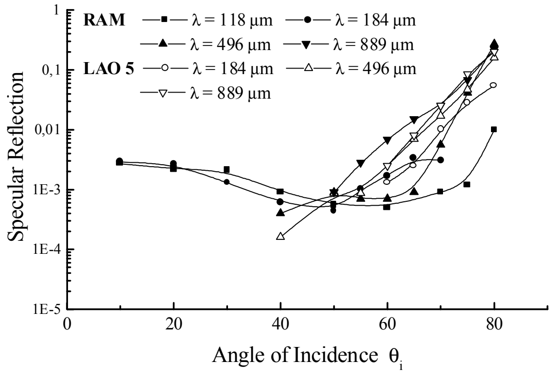

Fig. 3. *Specular reflection of RAM and LAO 5*

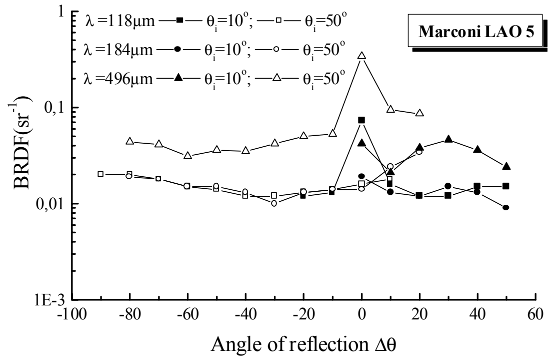

Figure 4. *BRDF of LAO 5*

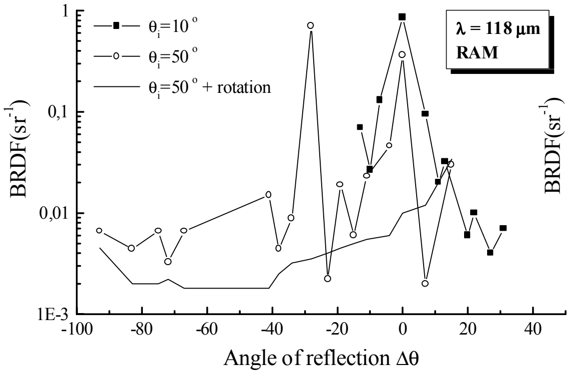

Fig. 5 *BRDF of RAM at 118μm*

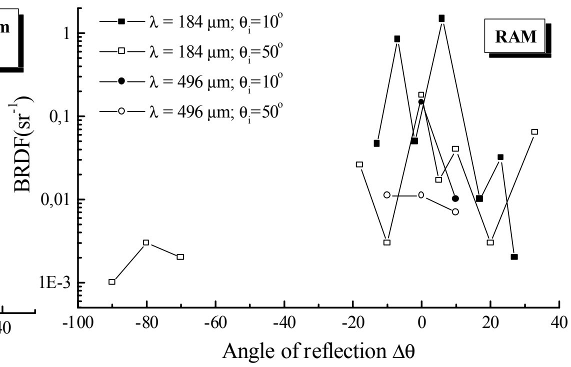

Fig. 6 *BRDF of RAM*

The BRDF data of RAM in Fig.5 and 6 show, in contrast to those of LAO 5, sharp peaks in the angular dependence. These reflections result from the regular 1x1 mm^2 structure of the sharp needles at the surface that acts as a reflection grating. These grating reflections are observed only when the optical plane is parallel to the needle rows. For other directions, or if the sample is rotated around it's surface normal, these peaks disappear, and quite a low BRDF value is observed, resulting in a THR of about –20dB. This grating effect is similar to that observed for the pyramidal structured absorber reported earlier [2].

2. MPI coatings for PACS

In the table, the different coatings developed by MPI are given. They are fabricated on an Aluminium substrate, first coated with a thin layer of Nextel Primer (5523:5524; 10:1) diluted with Nextel Thinner 8061, and then coated with either DeSoto Gunship Black (828X310:910X376; 1:1) or Nextel Suede Coating (3101:6018; 8:1). Part of the samples also contains 100µm size SiC grains that have been applied immediately after the DeSoto or Suede coating. The samples have been dried at 60°C for 1 hour. (In view of the thickness of MPI 10, possibly the last two layers are absent.)

Sample	Thickness	Layer sequence
MPI 3	0.15 mm	Primer-Suede-Suede
MPI 4	0.20 mm	Primer-Suede-Suede-Suede
MPI 9	0.50 mm	Primer-Suede-Sic grains-Suede
MPI 10*	0.55 mm	Primer-Suede-Sic grains-Suede- Suede-Suede
MPI 7	0.15 mm	Primer-DeSoto-DeSoto
MPI 8	0.20 mm	Primer-DeSoto-DeSoto-DeSoto
MPI 5	0.50 mm	Primer-DeSoto-SiCgrains-DeSoto
MPI 6	0.90 mm	Primer-DeSoto-SiCgrains-DeSoto-SiCgrains-DeSoto

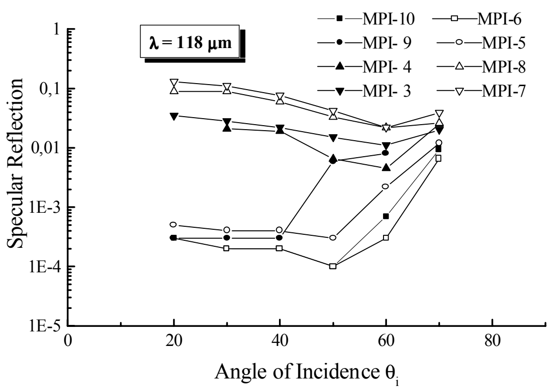

Fig. 7 *Specular reflection of MPI coatings*

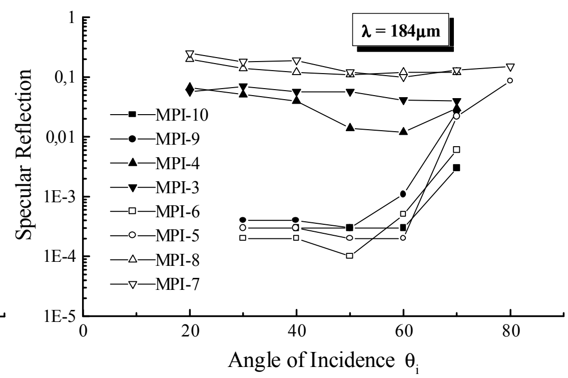

Fig. 8 *Specular reflection MPI*

The data on specular reflection in Fig.'s 7 and 8 clearly demonstrate the influence of the SiC grains on the scattering properties; their presence is crucial to suppress the specular reflection. Although the SiC itself does not contribute to the absorption, it causes multiple reflections at the rough surface and inside the layer, and thereby increases the effective absorption.

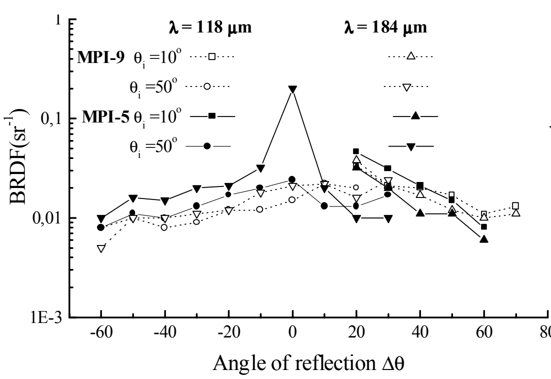

Fig. 9 *BRDF of single SiC layer MPI*

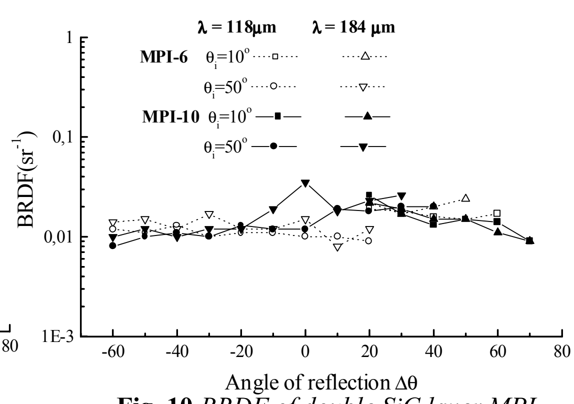

Fig. 10 *BRDF of double SiC layer MPI*

The experimental data in Fig.'s 7-10 indicate that both specular reflection and BRDF increase with wavelength, angle of incidence and diminishing surface roughness and thickness. The Suede samples show slightly better absorbing characteristics, possibly due to a larger absorption coefficient (see later). The approximate value of the BRDF of the SiC loaded coatings is 0.02 sr^{-1}, corresponding to a THR of about 6% (–12dB) Further developments of these coatings are in progress.

3. Delft/SRON coatings for HIFI

Also these coatings have been fabricated on Aluminium substrates. First a thin layer of Stycast 2850 FT + 24 LV Catalyst is applied and subsequently a mixture of Stycast 2850 FT + 24 LV Catalyst and SiC grains of various sizes. The samples are then dried for 2 hours at 60^o C. Coatings have been made with SiC grain sizes of 125µm, 250 µm, 500 µm, 750 µm and 1000 µm respectively. Because of the much longer upper wavelength of HIFI, these coatings have been studied for wavelengths up to 889 µm.

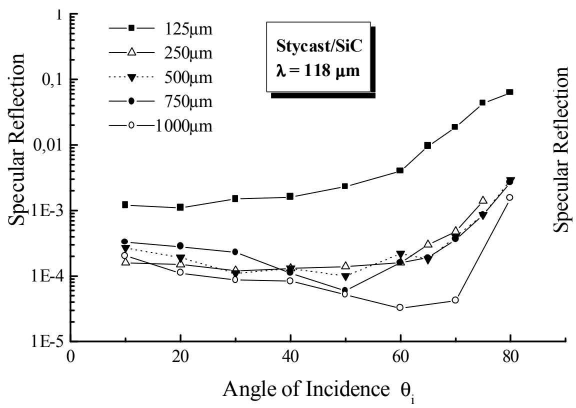

Fig. 11 *Specular reflection at 118 µm*

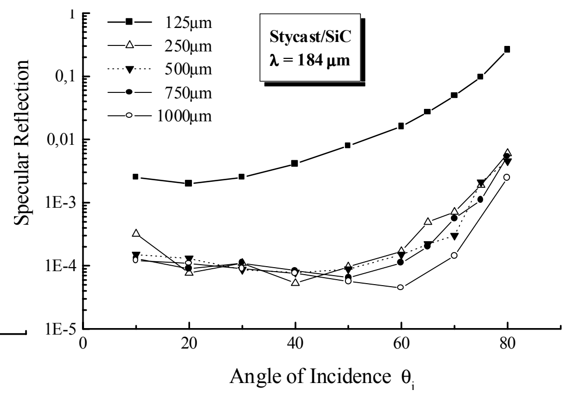

Fig. 12 *Specular reflection at 184 µm*

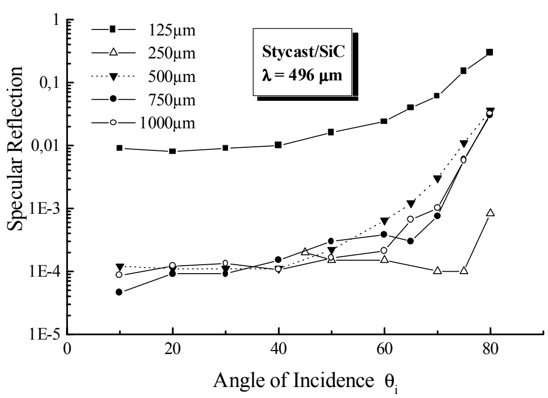

Fig. 13 *Specular reflection at 496µm*

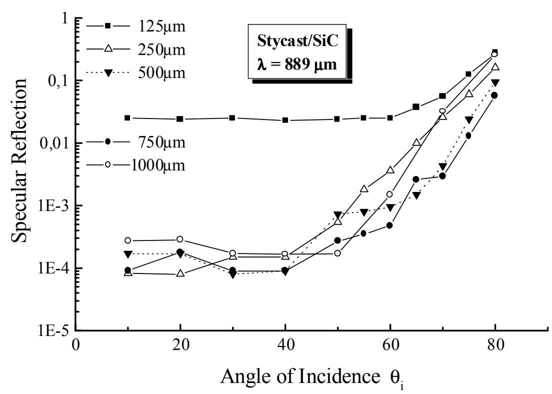

Fig. 14.*Specular reflection at 889 µm*

Similar to the findings for the MPI coatings, also for this series of coatings the specular reflection increases with increasing wavelength, angle of incidence and diminishing surface roughness, as is clearly visible in Fig.'s 11-14. Therefore, to ensure a minimum (specular) reflection at the longest wavelength, the coating containing 1000 μm SiC grains is preferable. Properties of that coating are:

Optimum weight ratio Stycast/SiC	1:4
Coating thickness	2.3 (3) mm
Mass of coating (ex substrate)	0.34 (3) gram/cm^2

This intimate relation between scattering intensity, wavelength and surface roughness is also observed in the BRDF data. A concise summary of those data is shown in Fig.'s 15-18, with emphasis on the properties of the 1000 μm SiC coating.

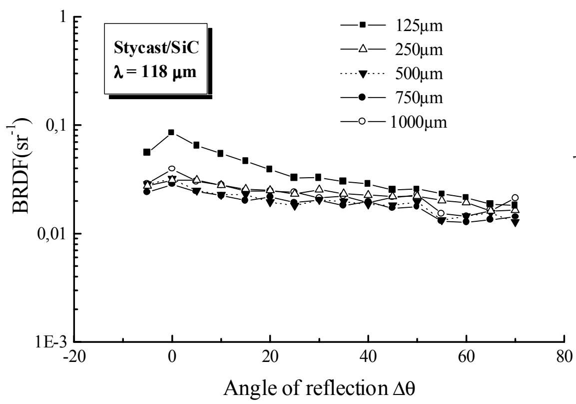

Fig. 15 *BRDF of Stycast/SiC samples at 118μm*

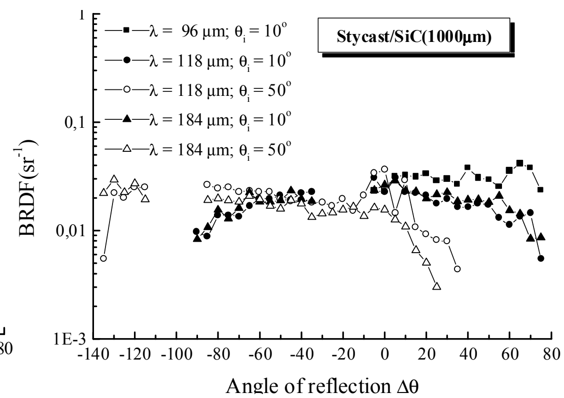

Fig. 16. *BRDF of 1000 μm sample at 3 wavelengths*

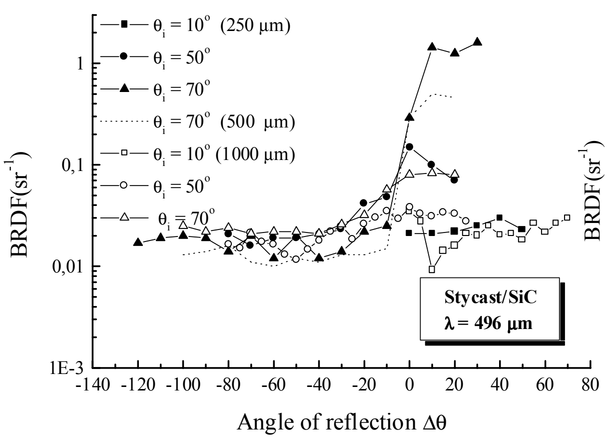

Fig. 17 *BRDF of Stycast/SiC samples at 496 μm*

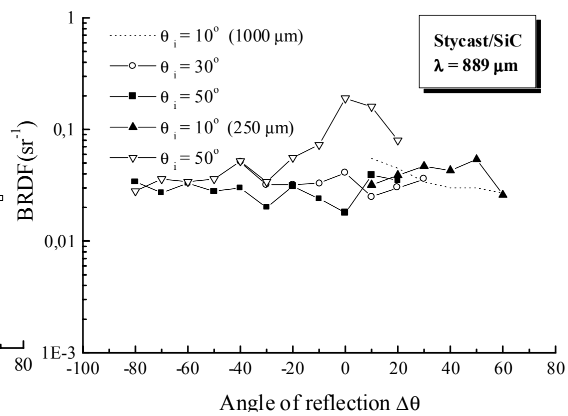

Fig. 18 *BRDF of Stycast/SiC at 889 μm*

The nearly wavelength, polarization [2] and directional independent - value of the BRDF (also for out of plane scattering [2]) of this coating is about 0.02. That means a THR of 6%, i.e. –12dB. This number agrees very well with the measured emissivity e = 0.93(2) at λ = 435 μm and T = 70K [2].

The disadvantage of this very rough surface however is two-fold. First of all, because of the large grain size, the coating layer is rather thick. Secondly, in those cases where the optical beam has a relatively small waist at the coating surface, the scattering is observed to be not random anymore, due to the limited number of SiC grains (size ≈ 1mm!) within the illuminated area. Strongly angular dependent variations, up to a factor 5, in the reflected intensity have been observed. In order to measure a representative and reproducible value for the BRDF, the samples have to be rotated around their surface normal to measure an "averaged" reflection signal. For critical applications, such as beam dumps, these unpredictable variations may be harmful. Therefore, in the near future it will be investigated whether the coating with 500µm SiC grains, which shows this effect much less, can serve, after further optimization, as an alternative.

Absorption coefficients. The wavelength dependent absorption coefficients of some of the materials used for the coatings discussed in this paper have been derived from transmission through thin layers, not loaded with SiC grains. In the table the values are given in cm^{-1}. Those for DeSoto and Suede have been estimated from reflection data on unloaded samples.

Material	**118 µm**	**184 µm**	**496 µm**	**889 µm**
Stycast 2850FT	89	41	11.5	7.8
DeSoto	65	45		
Suede	100	70		
RAM	100	62	22	19

Table: Absorption coefficients(cm^{-1})

The increase of absorption coefficient with decreasing wavelength is in accordance with data reported by Halpern et al [3] on Stycast 2850FT for wavelengths above 300 µm , although their values are somewhat lower than ours. It is interesting to observe that the large decrease of the absorption coefficient from 118 µm towards 889 µm does not lead to a similar increase of the BRDF values of the Stycast/SiC (1000µm) sample. It should be mentioned that the SiC crystalline grains only show an absorption band near a wavelength of 12 µm; in the sub-millimeter range no absorption occurs. The nearly wavelength independent BRDF value shows that the absorption processes in such a rough layer is due to a complicated interplay between absorption of the binder material and scattering of the grains. In the very near future the scattering properties of these coatings at cryogenic temperatures will be studied. In view of the small sensitivity of the BRDF for changes in the absorption coefficient, it is expected that the decrease of the absorption coefficient towards low temperature, as reported in [3], will not strongly influence the BRDF values.

Qualification test. First qualification tests of the 1000µm SiC coating have been performed at SRON; these included:

- Thermal shock: cool-down from 294 K to 88 K in 15 min + subsequent heating to 300 K
- Thermal cycling: 5x cool-down from 290 K to 88 K in 60 min + increase of temperature gradient by attaching conducting straps to the sample
- Extended bake out: T = 80 C at p < 1mbar for 48 hours.
- Vibration test: T = 70 K perpendicular to surface; random and sinus up to g=30

In all cases, no flakes or loose grains, no blistering coating, no bending of sample and no colour changes were observed. The adhesion test gives an averaged tensile strength of 14.6N/mm^2, not influenced by the above mentioned treatments.

Still to be performed: out gassing and influence of treatments on optical properties.

Possible surfaces for a black body calibration source. Requirements for a black body calibration source for HIFI are: 2 cm^2 emission area at T = 100K with a maximum electric power of 2 mW. The emitting surface must have an optimum emissivity and a minimum temperature gradient.

Following the results of study of the optical properties of various types of absorbing surfaces, so far three types of surfaces could qualify.

1. A standard SiC/Stycast coating on an Aluminium substrate.

The heater and temperature sensor(s) can be placed on the back side of the Al substrate.

For the 1000µm SiC coating surface at T = 77K and λ = 435 µm, the emissivity e=0.93; emissivities of the 250 µm and 500 µm coating are expected to be about the same.

Further specifications for the 1000 µm coating at T = 100K are:

Mass (excluding the Al substrate) :		0.34 gram/cm^2.
Heat capacity:		43 mJ/cm^{2}K
Thermal conductivity:	(low purity) SiC :	1 W/cmK
	Aluminium:	3 W/cmK
	Stycast 2850 FT:	0.014W/cmK @300K

2. Aluminium foam (ERG Materials and Aerospace Corp., Oakland, CA, USA).
40 pores per inch (ppi) coated with Stycast 2850 FT + 24 LV Catalyst.

In Figure 19 the BRDF data of this surface at λ = 496 µm are given. From the average BRDF of about 0.06 an emissivity of approximately 0.80 is calculated. The advantage of this material is that only 1 cm^2 is needed because both sides will emit equally well. Heater and temperature monitors could be attached to an aluminium ring around the (approximately 4 mm thick) outer contour of the surface; the excellent heat conduction of the aluminium grid will establish a good temperature homogeneity across the sample.

Moreover a finite sub-millimetre transmission through the sample is present. Transmission data at different wavelengths show that the overall transmission of a 4 mm thick sample is in the range of 1%.
This finite transmission might be profitable in establishing a homogeneous optical field distribution, if the emitter is placed inside an integrating sphere.

General properties of such a surface:
Density of foam: 8-10 %
Al filament diameter: 0.11 mm
Mass: 0.29 gram/cm^2
Mass of foam + Stycast: 0.87gram/cm^2
e ≈ 0.80 @300K

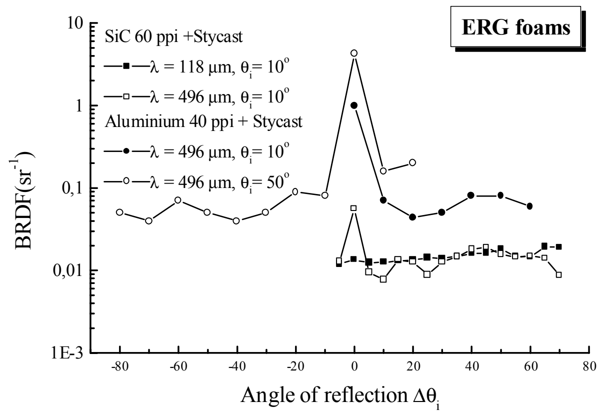

Fig. 19 *BRDF of Stycast coated foams*

3. SiC foam (ERG), 60 ppi, with relative density of 12-14%, coated with Stycast 2850 FT.
As can be seen from the BRDF data in Figure 19, the emissivity of this material will be quite the same as found for the SiC/Stycast home made coatings, i.e. e ≈ 0.93. These samples with 3.2 mm thickness, show an sub-millimeter transmission of the order of ≤ 10^{-3}. The incorporation of heater and temperature sensors in such a material however, might cause some problems.
The properties at 100 K are:

Mass of foam	0.18 gram/cm^2
Mass of foam + Stycast	0.25 gram/cm^2
Thermal conductivity foam:	4 W/cm^{2}K
Specific heat of foam	18 mJ/cm^{2}K
Specific heat foam + Stycast:	78 mJ/cm^{2}K

Conclusions
A number of home made absorbing surfaces have been developed that are suitable as sub-millimeter absorbers for the spectrometers aboard the FIRST satellite. In order to achieve a sufficient suppression of the specular reflection, the absorbing binder materials such as DeSoto, Suede or Stycast 2850FT, have to be mixed with (SiC) grains to produce a rough surface.
The size of the SiC grains should be about equal to the wavelength region of interest. Typical BRDF values are 0.02 sr^{-1}. The corresponding Total Hemispherical Reflection equals 6%, or –12dB. Because of the low specular reflection under non-grazing incidence, the occurrence of standing wave patterns will be effectively cancelled.

These coatings might also be employed as emitting surfaces for black body radiation sources. Alternatively, some rigid foam materials, coated with for instance Stycast 2850FT, could also be useful for that purpose.

References

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