

# Infrared opaque heat shield with high thermal conductance for use in changing magnetic fields\*

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Spectral data on the transmittance, reflectance, and absorption of far-infrared radiation are presented for "coil foil," Stycast 1266 epoxy, a Stycast 1266/charcoal powder composite, Stycast 2850 GT epoxy, Epibond 100 A adhesive, an Epibond 100 A/copper powder composite, silicone vacuum grease, and a vacuum grease/charcoal composite. These data show that a superior heat shield for low-temperature applications can be constructed with coil foil coated with a Stycast 1266/charcoal powder composite. This shield is opaque to all thermal radiation, has a high thermal conductivity to a heat sink, is not susceptible to eddy current heating, is light, strong, rigid, and yet easy to construct.

## I. INTRODUCTION

Recent advances in nuclear refrigeration<sup>1</sup> allow experiments to be cooled to the submillikelvin regime. At these temperatures heat shields must be designed with increased care. Except for the work of Anderson, Folinsbee, and Johnson,<sup>2</sup> very few quantitative data have been available regarding the materials used for the construction of such shields. The purpose of this paper is to present spectral data on the transmission, reflection, and absorption of radiation, at frequencies corresponding to low-temperature blackbody radiation, for various materials which have traditionally been used for heat shield fabrication in low-temperature apparatus. A shield which is both easily constructed and extremely effective is then described.

## II. DESIGN PHILOSOPHY

A good heat shield should be light in weight, self-supporting, and yet thin so as not to occupy valuable space, such as in the bore of a high-field magnet. It should not be susceptible to eddy-current heating, yet it must be able to conduct any absorbed heat to a heat sink which is usually attached to one of its ends. Finally, it should transmit no radiation, particularly at far-infrared frequencies. These frequencies are near the peak of the blackbody emission spectrum, given by Wien's law

$$\hbar\omega = 5kT. \quad (1)$$

The radiator temperatures of interest are between 0.6 and 4.2 K and give maximum energy at frequencies between 2 and 15  $\text{cm}^{-1}$ , where the frequency is measured in units of reciprocal wavelength.

Except for eddy-current effects, a copper tube satisfies all of the above requirements. A rather short shield which sees only slowly varying magnetic fields might be constructed from an alloy tube, such as stainless steel. However, a shield attached to the mixing chamber of a dilution refrigerator ( $T = 20$  mK) and extending far ( $l = 50$  cm) into the bore of a high-field magnet

( $H = 80$  kG), as is typical for the adiabatic demagnetization of copper nuclei, is more involved. The following section describes materials which are candidates for constructing such a shield.

## III. MATERIALS INVESTIGATED

"Coil foil" (described below) has frequently<sup>2</sup> been used as the basic material for fabricating heat shields. It has excellent thermal conductance along its length, and yet is not susceptible to large eddy-current heating. However, coil foil alone is lacking in rigidity and, we have found (see below), transmits up to 50% of the incident radiation. Consequently, some sort of additional layer or coating must be added to compensate for these shortcomings.

The coating materials we investigated included Epibond<sup>3</sup> 100A, Stycast 1266, Stycast 2850 GT,<sup>4</sup> Epibond Epibond 100A impregnated with copper powder,<sup>5</sup> and Stycast 1266 blended with charcoal powder. One additional material tested for comparison and possible non-permanent applications was a mixture of silicon vacuum grease<sup>6</sup> and charcoal powder.

### A. Coil foil

The coil foil was prepared in the following manner. A large-diameter (28 cm) cylindrical aluminum drum was wrapped with a sheet of 0.013-cm-thick Mylar. This was mounted on a coil winder and then wrapped in a back-and-forth manner with ten layers of No. 44HF copper magnet wire. The pitch of the winding was adjusted so that there was a space of  $\sim 0.05$  cm between individual turns. By the end of the tenth layer, the mandrel had a fairly uniform appearance, but still contained regions where small gaps had occurred in the winding. These gaps were removed by pressing a soft cloth against the wire while slowly turning the drum. Then the coil was painted with a mixture of GE7031 varnish,<sup>7</sup> toluene, and isopropyl alcohol (2:1:1) and left rotating until dry. A total of four varnish layers were applied.

Finally, the coil was slit parallel to the axis of the cylinder, and the resulting sheet of coil foil and its temporary Mylar backing were removed and stored on a flat surface until used.

## B. Epibond 100A

Epibond 100A is an amber colored one-component epoxy resin. Our test pieces included a pure translucent sample and a sample impregnated with No. 325 mesh (particle size  $<44\text{ }\mu\text{m}$ ) copper powder. The mixture was approximately three parts Epibond 100A to one part copper powder by weight. The volume fraction of copper in the composite is 0.04.

## C. Stycast 1266

Stycast 1266 is a clear two-component epoxy casting resin which cures at room temperature. Again our tests included a pure sample and a mixture. In this case, the Stycast was blended with powdered activated charcoal. The charcoal was powdered in a mortar and pestle, the larger pieces removed, and the remainder added to the Stycast. The charcoal showed no tendency to settle out of the epoxy. Its volume fraction was approximately 0.1.

## D. Stycast 2850 GT

Stycast 2850 GT is a black two-component epoxy resin. It can be cured at room temperature. The material contains an inert filler, small sand (quartz) particles. These make 2850 GT not machineable by ordinary techniques.

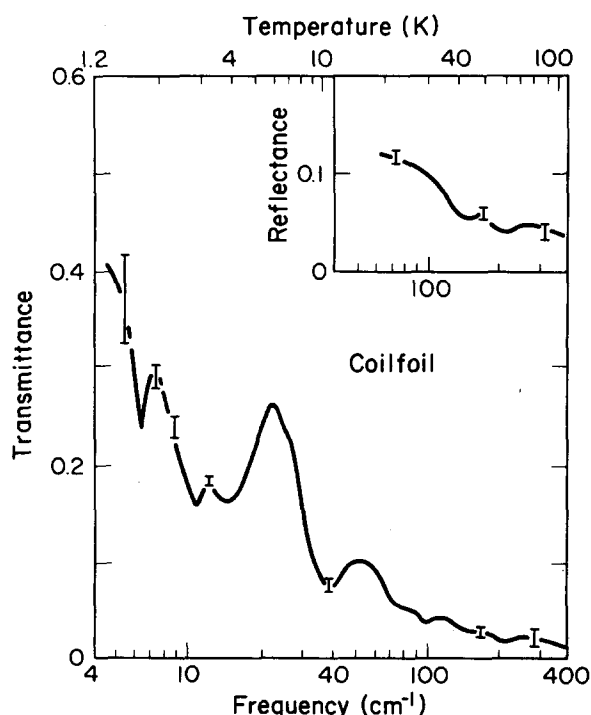


FIG. 1. Transmittance (main figure) and reflectance (inset) of coil foil for infrared frequencies between 4 and 400  $\text{cm}^{-1}$ . Notice the logarithmic frequency scale. Across the top is a temperature scale calculated from Wien's law,  $h\nu = 5kT$ .

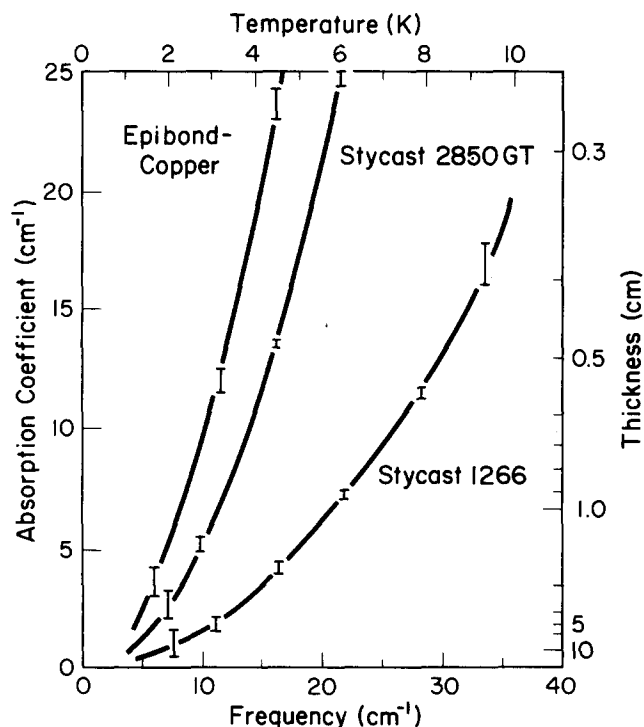


FIG. 2. Absorption coefficient versus frequency for Stycast 1266 epoxy, Stycast 2850 GT epoxy, and Epibond 100A plus copper composite. Across the top is a temperature scale calculated from Wien's law,  $h\nu = 5kT$ . The right-hand scale indicates the thickness necessary to have a transmittance of 0.001.

## IV. FAR-INFRARED TECHNIQUES

Far-infrared measurements were made between 4 and 400  $\text{cm}^{-1}$  (photon energies between 0.5 and 50 meV). Over the range 4–40  $\text{cm}^{-1}$  measurements were made on samples at 4.2 K using a lamellar grating interferometer<sup>8</sup> and a germanium bolometer-detector operating at 1.2 K. Between 40 and 400  $\text{cm}^{-1}$  a Michelson interferometer<sup>9</sup> and Golay detector were used; the samples were at room temperature. The far-infrared radiation traveled in hollow brass light pipes with inside diameter 1.27 cm. Typical thickness for the epoxy and composite samples was between 0.1 and 0.2 cm. For the transmission measurements the samples were inserted into gaps in the light pipe. For reflection measurements, a light pipe optics reflectance insert, described earlier,<sup>10</sup> was employed. This insert uses a beam splitter to pass part of the incident beam to the sample and then to direct part of the reflected beam to the detector.

## V. FAR-INFRARED RESULTS

The main part of Fig. 1 shows the measured transmittance of the coil foil samples. Measurements on two different samples (from different batches) at two different temperatures match up well enough so these data may be regarded as typical for this material. Notice the logarithmic frequency scale. Across the top is a temperature scale calculated from Eq. (1). At low frequencies the transmittance is in excess of 0.4. It rolls off with considerable structure to below 0.01 at 400  $\text{cm}^{-1}$ . The inset shows the reflectance at high fre-

TABLE I. Infrared transmittance and reflectance (80–350 cm<sup>-1</sup>).

Sample	Thickness (cm)	Transmittance	Reflectance
Stycast 1266	0.27	0.0014	0.18
Stycast 1266 and charcoal	0.17	0.0003 <sup>a</sup>	0.20
Stycast 2850 GT	0.074	0.005	—
Epibond 100A	0.15	0.0013	0.21
Epibond 100A and copper	0.18	—	~0.20 <sup>b</sup>
Vacuum grease	0.13	0.04	—
Vacuum grease and charcoal	0.13	0.0005	0.21

<sup>a</sup> Transmittance remains low to 4 cm<sup>-1</sup>.<sup>b</sup> Reflectance increases smoothly from 0.15 at 80 cm<sup>-1</sup> to 0.25 at 350 cm<sup>-1</sup>.

quencies. This falls from 0.12 at 60 cm<sup>-1</sup> to 0.04 at 400 cm<sup>-1</sup>. A parallel grid of wires acts as a polarizer at frequencies low enough that the wavelength is large compared to the wire spacing. Incident radiation with the electric field parallel to the wire direction is reflected while that polarized perpendicular to the wire direction is transmitted; one would therefore expect a low-frequency transmittance of 0.50 for coil foil. When the wavelength becomes shorter than the wire periodicity, diffraction effects are observed. In principle, the transmission of the grating is still 0.50. However, some of the energy will be diffracted to high angles. The detector optics are such that only radiation in a cone of half-angle 15° will reach the detector. At frequencies such that all orders are diffracted to angles greater than 15° [i.e., for  $d > \lambda > d \sin(15^\circ)$ , where  $d$  is the grid spacing and  $\lambda$  the wavelength] only the zero-order radiation will reach the detector, and our measurements should show a low transmittance. At high frequencies the diffracted radiation will be within 15° and the signal should increase in steps as the various orders are accepted by the detector.

The coil foil is, of course, far from an ideal grating. At higher frequencies the sum of the reflectance and transmittance is 0.07; much of the remainder is absorbed. However, there are increases in transmission at 14, 40, 70, and 100 cm<sup>-1</sup>. These roughly correspond to odd orders from a spacing of 0.27 cm. Unfortunately, the basic coarse periodicity in the sample is 0.05 cm. Nonetheless, much of the structure seen in Fig. 1 must be diffraction effects. Our data then give a lower limit for the transmission of the coil foil.

Figure 2 shows the absorption coefficient versus frequency of Stycast 1266 and 2850 GT and the composite of Epibond 100A and copper. Across the top is a temperature scale, calculated from Eq. (1). The absorption coefficient is given by

$$\alpha = -\frac{1}{x} \ln \frac{T}{(1-R)^2}, \quad (2)$$

where  $T$  is the measured transmittance,  $R$  the reflectance, and  $x$  the thickness of the sample in centimeters. In all samples the absorption coefficient rises as  $\omega^2$  as the frequency increases. This same frequency

dependence is seen in infrared studies of other amorphous systems.<sup>11</sup> The copper–Epibond 100A composite has the highest absorption coefficient of the three samples shown. This absorption is probably due to eddy-current losses in the copper small particles. The behavior of this system is qualitatively different than has been observed in much smaller particles.<sup>12</sup> All of these materials are too transparent at low frequencies to serve as efficient radiation shields.

In this frequency region the absorption coefficient of the mixture of charcoal and Stycast 1266 is so high that the frequency dependence of the transmission could not be measured. Over the frequency range below 40 cm<sup>-1</sup>, the transmittance is 0.0005. This corresponds to an absorption coefficient larger than 70 cm<sup>-1</sup> averaged over the spectral region covered. At higher frequencies the absorption coefficients continue to increase. The transmittance and reflectance between 80 and 350 cm<sup>-1</sup> are shown in Table I. The reflectance of most of the samples was relatively independent of frequency between 40 and 400 cm<sup>-1</sup>.

From the absorption coefficient [Eq. (2)] one can calculate the thickness necessary to have a given transmission at a particular frequency. On the right-hand side of Fig. 2 is the thickness necessary to have a transmittance of 0.001, taking  $R = 0.2$ . For the Stycast and charcoal mixture, a thickness of 0.1 cm is sufficient to give this transmittance at frequencies above 4 cm<sup>-1</sup>.

Infrared measurements have shown that coil foil, a traditional heat shield material, is ineffective for long-wavelength radiation, approaching 50% efficiency at frequencies of 4 cm<sup>-1</sup> and below. Therefore, on the basis of rigidity, machineability, and opacity, coil foil coated with epoxy impregnated with charcoal is clearly a superior heat shield.

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<sup>1</sup> A. I. Ahonen, P. M. Berglund, M. T. Harkala, M. Krusius, O. V. Lounasmaa, and M. A. Paalanen, *Cryogenics* **16**, 521 (1976).

<sup>2</sup> A. C. Anderson, J. T. Folinsbee, and W. L. Johnson, *J. Low Temp. Phys.* **5**, 591 (1971).

<sup>3</sup> Epibond 100A formerly produced by Furane Plastics, Inc., Rahway, NJ (not presently manufactured).

<sup>4</sup> Stycast 1266 and Stycast 2850 GT produced by Emerson and Cuming, Northbrook, IL.

<sup>5</sup> Copper electrolytic powder (99.9%) from A. D. Mackay, New York, NY.

<sup>6</sup> High vacuum grease (No. 970V) by Dow Corning Corp., Midland, MI.

<sup>7</sup> GE 7031 varnish manufactured by General Electric (Insulating Materials Dept.) in Schenectady, NY.

<sup>8</sup> R. L. Henry and D. B. Tanner (unpublished).

<sup>9</sup> R. B. Sanderson and H. E. Scott, *Appl. Opt.* **10**, 1097 (1971).

<sup>10</sup> D. B. Tanner, C. S. Jacobsen, A. F. Garito, and A. J. Heeger, *Phys. Rev. B* **13**, 3381 (1976).

<sup>11</sup> K. K. Mon, Y. J. Chabal, and A. J. Sievers, *Phys. Rev. Lett.* **35**, 1352 (1975).

<sup>12</sup> D. B. Tanner, A. J. Sievers, and R. A. Buhrman, *Phys. Rev. B* **11**, 1330 (1975).