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Thermal Contacts Between Metal and Glass for Use at Cryogenic Temperatures

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      the glass. The thermal resistances of the rubber cement and copper-filled
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Thermal Contacts Between Metal and Glass for Use at Cryogenic Temperatures

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SUMMARY

Thermal contacts designed to cool a 50-cm diam fused-silica mirror to 5 K without serious distortion of the optical surfaces were tested. Rubber cement and copper-filled rubber cement were found to be well suited for maintaining thermal contact between the fused-silica and copper straps. A variety of other adhesives proved to be unreliable because they spalled the glass. The thermal resistances of the rubber cement and copper-filled rubber cement were determined. For the copper-filled rubber cement, the thermal resistances varied from 570 K/W at 60 K to 15,000 K/W at 10 K.

INTRODUCTION

Optical testing of glass mirrors at very low temperatures requires thermal bonding techniques that will (1) not distort the mirror figure, (2) withstand repeated temperature cycling, and (3) remain thermally conductive at the lowest temperature required. The technique described in this report was used successfully in optical tests of a 50-cm-diam, fused-silica mirror at temperatures down to 5.5 K. The results of the optical tests will be reported separately, but they are similar to earlier results (ref. 1) for fused quartz at 10.5 K. In this paper we present mechanical and thermal data in the region 60 K to 10 K on seven different bonds.

To test glass mirrors at low temperatures, thermally conductive cooling straps are bonded to the appropriate mirror surfaces. By using a large number of flexible straps (e.g., copper braid), the mirror is cooled uniformly and without external force. Because the glass has a different rate of thermal contraction than most materials, there are very few materials that strongly bond the cooling straps to glass sufficiently well to withstand thermal cycling between 293 K and 10 K. In general, there are two methods used to bond cooling straps to glass: (1) soldering the straps to metalizations on a mirror surface, and (2) gluing the straps to the glass with a thermally conductive adhesive.

The soldering method is being pursued on the German Infrared Laboratory (GIRL) program (ref. 2). The Germans solder copper wire-braid straps to Invar buttons which are then soldered to metalized spots on the rear of the Zerodur primary mirror (Zerodur is a low-expansion glassy ceramic). We have tried to solder copper straps directly to metalized spots on a fused-quartz mirror and found the technique difficult to use and not completely reliable; about 10% of the bonds failed with each thermal cycle.

A wide variety of adhesive materials, such as epoxies, urethanes, silicone rubbers, and even greases, are used at low temperatures (ref. 3). Adhesives can be selected on the basis of either of the following criteria: (1) those with low-temperature flexibility (ref. 4), and (2) those that match the thermal contraction rate of glass (ref. 5). Both were tested in this study.

APPROACH

Common, easily applied materials and adhesives were used to thermally bond copper-braid cooling straps to otherwise thermally isolated blocks of mirror material. These blocks, Heraeus T08E fused quartz, were cooled below 10 K in a Dewar to obtain thermal-resistance data on the bonds. The bonds were applied in the same way envisioned for application to actual glass mirrors. We did not determine the bulk thermal resistance of the bonding material because it was not as important for the applications of interest as the thermal and mechanical performance of the bond itself.

MATERIALS AND EQUIPMENT

The adhesives selected for study were commercial adhesives, as well as laboratory preparations. The commercial adhesives were Torr Seal (Varian Associates); RTV 112 silicone rubber and silicone metal seal (General Electric); Duco epoxy EPX-1 (Woodhill Chemical Sales); and Carters rubber cement (ref. 6). These adhesives were used according to the manufacturers' instructions. The laboratory-prepared adhesives were glass-filled Duco epoxy and copper-filled Carters rubber cement.

The use of glass-filled Duco epoxy was an attempt to match the rate of thermal contraction of the epoxy to that of the block. We mixed four parts ball-mill-powdered fused quartz with one part epoxy hardener and resin. Higher ratios of fused quartz did not adhere well.

The use of copper-filled rubber cement was an attempt to lower the thermal resistance of the rubber cement. We mixed three parts fine-copper powder with seven parts liquid-rubber cement. Deviations from this ratio had little effect on the cooling rate of the blocks. Copper-powder-to-cement ratios greater than 1-to-1 did not adhere well. The size of the copper particles also had little effect on the cooling rate of the block, although particles larger than fine filings did not mix well.

The liquid-helium Dewar incorporated a completely enclosed, internally blackened radiation shield bolted to the cold surface at the base of the liquid-helium chamber, as shown in figure 1. This radiation shield, when cooled to about 4.2 K, absorbed most of the radiation emitted from the blocks and prevented heating from external sources. Three glass blocks could be accommodated on the cold surface.

A calibrated 1/8 W, carbon resistance thermometer was bonded with rubber cement to the end of each block to monitor the temperature throughout the experiment. High-thermal-resistance electrical leads for the resistance thermometers were brought out of the Dewar through an electrical feedthrough. Current for the resistance thermometers was supplied with a constant-current source set for 10 μA to minimize electrical heating. The voltage drop across the resistance thermometer was measured with a digital voltmeter. The electrical resistance of the lead wires, connectors, and joints was a constant 47 ohms over all temperatures. This value was subtracted from all electrical resistance measurements.

The fused quartz blocks were isolated from the cold surface by 0.125-in.-thick, 0.5-in.-square nylon pads. The pads were grooved to further reduce the thermal conductivity by minimizing the area of contact. The blocks were secured to the cold surface with L-shaped nylon clamps, as shown in figure 1.

The thermal straps consisted of 16 strands of 0.005-in. diam, tinned copper wire. One end of each strap was bonded on the block, opposite the resistance thermometer. We bolted the other end of each strap to the cold surface, as shown in figure 1.

CALIBRATION AND CONTROL

The resistance thermometers were calibrated at three temperatures: 293 K, 77 K, and 4.2 K to determine the constants A, B, and K in the following equation:

$$T = \frac{B}{\log R_e + \frac{K}{\log R_e} - A} \tag{1}$$

where R_e was the measured electrical resistance at temperature T (ref. 7).

We calibrated each of the blocks and their clamps by cooling the blocks below 10 K and measuring their unstrapped cooling rates. The cooling was due to both radiation and conduction through the clamp, pad, and lead wires. After the calibration run, the bonding of the thermal strap was the only change made.

To estimate the error in the measurements, data were taken on the third block which was left unchanged and unstrapped during each series of experimental runs.

EXPERIMENTAL TECHNIQUE

Each of the thermal bonds between the cooling strap and the block was made in the same way. We wiped the strap and block clean with acetone. The cooling strap was bent so that the last 0.25 in. of the strap touched the top edge of the block. The strap was temporarily moved aside while we placed a 0.25-in.-diam spot of liquid adhesive on the top edge of the block. We then pressed the cooling strap into the spot of liquid adhesive. The adhesive dried or cured before we assembled and evacuated the Dewar. We chose 0.25-in.-diam bonds because smaller bonds did not cool the block as well, and larger bonds greatly increase the risk of producing spallation of the glass.

Measurements were made to produce a plot of the block temperature versus time in the range 60 K to 10 K. After cooling to 77 K with liquid nitrogen, the Dewar was emptied and filled with liquid helium. Starting with the liquid-helium fill and after every 5 min (15 min for the unstrapped calibration runs) of elapsed time, we connected each resistance thermometer to the current source and measured the voltage drop. The electrical resistance, calculated using Ohm's law, was converted into temperature, using equation (1) with the appropriate constants. The temperature was recorded as a function of elapsed time. After the experimental run was completed and the Dewar warmed, we opened the Dewar and examined the bonds.

RESULTS AND ANALYSIS

With the exception of rubber cement and copper-filled rubber cement, all other adhesives produced spallation of the fused quartz under the bond during the first thermal cycle. During the sixth thermal cycle, the rubber cement used to bond the resistance thermometers produced spallation of the fused quartz on two of the three blocks. However, the rubber cement used to bond the resistance thermometers was applied in a very thick layer over a 0.5 in. diam area. None of the 0.25-in. diam rubber-cement bonds used for the thermal straps produced spallation of the fused quartz, after two thermal cycles.

We wish to calculate the thermal resistance of each thermal bond, R_B. The thermal circuit for the experiment is shown in figure 2. The thermal resistance of the block itself was found to be small enough to be ignored. The thermal resistance R is given by

$$\frac{1}{R} = \frac{1}{R_B + R_S} + \frac{1}{R_C} \tag{2}$$

where R_C is the thermal resistance of the clamp, pad, and lead wires; and R_S is the thermal resistance of the copper cooling strap. Solving for R_B we obtain

$$R_B = (R_S R_C - RR_S - RR_C)/(R - R_C) \tag{3}$$

To find the bond thermal resistance R_B we must first determine R, R_C, and R_S.

The measured cooling rates include cooling by conduction through the clamp, strap, and bond, when present, and cooling by emission of radiation from the block:

$$\left(\frac{dT_B}{dt}\right)_{\text{measured}} = \left(\frac{dT_B}{dt}\right)_{\text{conduction}} + \left(\frac{dT_B}{dt}\right)_{\text{radiation}} \tag{4}$$

where T_B is the temperature of the block. The cooling rate owing to emission of radiation is calculated from Stefans law:

$$\left(\frac{dT_B}{dt}\right)_{\text{radiation}} = \frac{\varepsilon\sigma A_B}{CM_B}\left(T_B^4 - T_W^4\right) \tag{5}$$

where ε is the emissivity of the fused quartz, taken to be approximately 1 over much of the thermal infrared range; σ is Stefans constant; 5.7×10^{-8} $\mathrm{Wm^{-2}\,K^{-4}}$; A_B is the total surface area of the block, 2.7×10^{-3} $\mathrm{m^2}$; M_B is the mass of each block, 16 g; C is the specific heat of fused quartz and is a function of the block temperature; and T_W is the temperature of the cold surface, 4.3 K. We define the thermal resistance used to determine R_C and R as

$$\left(\frac{dT_B}{dt}\right)_{\text{conduction}} = \frac{1}{RCM_B}\left(T_B - T_W\right) \tag{6}$$

Solving for R and substituting from equation (4), we obtain

$$R = \frac{1/(CM_B)(T_B - T_W)}{\left(\frac{dT_B}{dt}\right)_{measured} - \left(\frac{dT_B}{dt}\right)_{radiation}} \tag{7}$$

Using equation (7), we determined the thermal resistance R from 5-min averages of the cooling rates of the blocks with bonds and straps applied. Sample data are shown in figure 3. The temperature T_B was the mean of the block temperature over the 5-min interval. We obtained the specific heat of quartz (SiO_2) from Anderson (ref. 8) and Westrum (ref. 9). Again, using equation (7), we determined the thermal resistance of the clamp, pad, and lead wires, R_C, from 15-min averages of the cooling rates of the unstrapped blocks ($R_S = \infty$) during the calibration runs. A sample of these data is also shown in figure 3. The temperature T_B was the mean of the block temperature over the 15-min interval.

The thermal resistance of the strap R_S is given by

$$R_S = \frac{L}{A_S K} \tag{8}$$

where L is the length of the strap 0.024 m; A_S is the total cross-sectional area of the strap 1.7×10^{-8} m^2; and K is the thermal conductivity of the strap, a function of the strap temperature which is taken to be T_B. The values for the thermal conductivity of electrolytic tough-pitch 99.9+% copper were obtained from Powell et al. (ref. 10). The value of the thermal conductivity of the strap is uncertain, because the exact composition of the strap is unknown and the thermal conductivity of copper varies greatly with composition. However, the strap thermal resistance is small compared with the bond thermal resistance for any likely copper composition.

Once R_T, R_C, and R_S were determined at T_B, equation (3) was used to calculate the bond thermal resistance. The results are summarized in table 1. The bond thermal resistances quoted for the adhesives that produced spallation are typical values. The error quoted is the 1σ relative error.

CONCLUSION

Since Torr Seal, Duco epoxy, RTV silicone rubber, silicone metal seal, and glass-filled Duco Epoxy all produced spallation of the fused quartz on the first thermal cycle, they appear to be unacceptable thermal adhesives for bonding cooling straps to glass mirrors by the methods we tried in this study. Even though the fused quartz was spalled, some thermal contact was maintained and these adhesives cooled the blocks at a rate less than the rate for rubber cement. Rubber cement proved to be a strong and flexible low-temperature adhesive, but the thermal resistance of the rubber cement bond was too high. Adding copper filings or copper dust lowered the thermal resistance of the bond to an acceptable value without changing the desirable properties of the adhesive. The optimum ratio of copper to liquid-rubber cement appears to be about 3:7, but the exact ratio is unimportant. The exact size of the copper particles also appears to be unimportant. Bond diameters of about 0.25 in. are recommended, because smaller bonds have a higher thermal resistance and larger bonds increase the risk of producing spallation. We feel that copper-filled rubber cement is an ideal adhesive for bonding cooling straps to glass mirrors since the adhesive is simple to make and apply and can be readily removed at the conclusion of the experiment. More work is needed to determine the properties of copper-filled rubber cement after a large number of thermal cycles before permanent applications can be considered.

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TABLE 1.- RESULTS

<table>
<tr><th>Bond</th><th>Produced spallation?</th><th>$R_{B\ 60\ K}$, K</th><th>$R_{B\ 25K}$, K</th><th>$R_{B\ 10K}$, K</th></tr>
<tr><td>Torr seal</td><td>yes</td><td rowspan="5">1,700 ±10%</td><td rowspan="5">5,500 ±4%</td><td rowspan="5">21,000 ±4%</td></tr>
<tr><td>RTV 112</td><td>yes</td></tr>
<tr><td>Silicone metal seal</td><td>yes</td></tr>
<tr><td>Duco epoxy</td><td>yes</td></tr>
<tr><td>Glass-filled epoxy</td><td>yes</td></tr>
<tr><td>Rubber cement</td><td>no</td><td>1,500 ±10%</td><td>4,700 ±4%</td><td>17,000 ±6%</td></tr>
<tr><td>Copper-filled rubber cement</td><td>no</td><td>570 ±28%</td><td>2,700 ±8%</td><td>15,000 ±6%</td></tr>
</table>

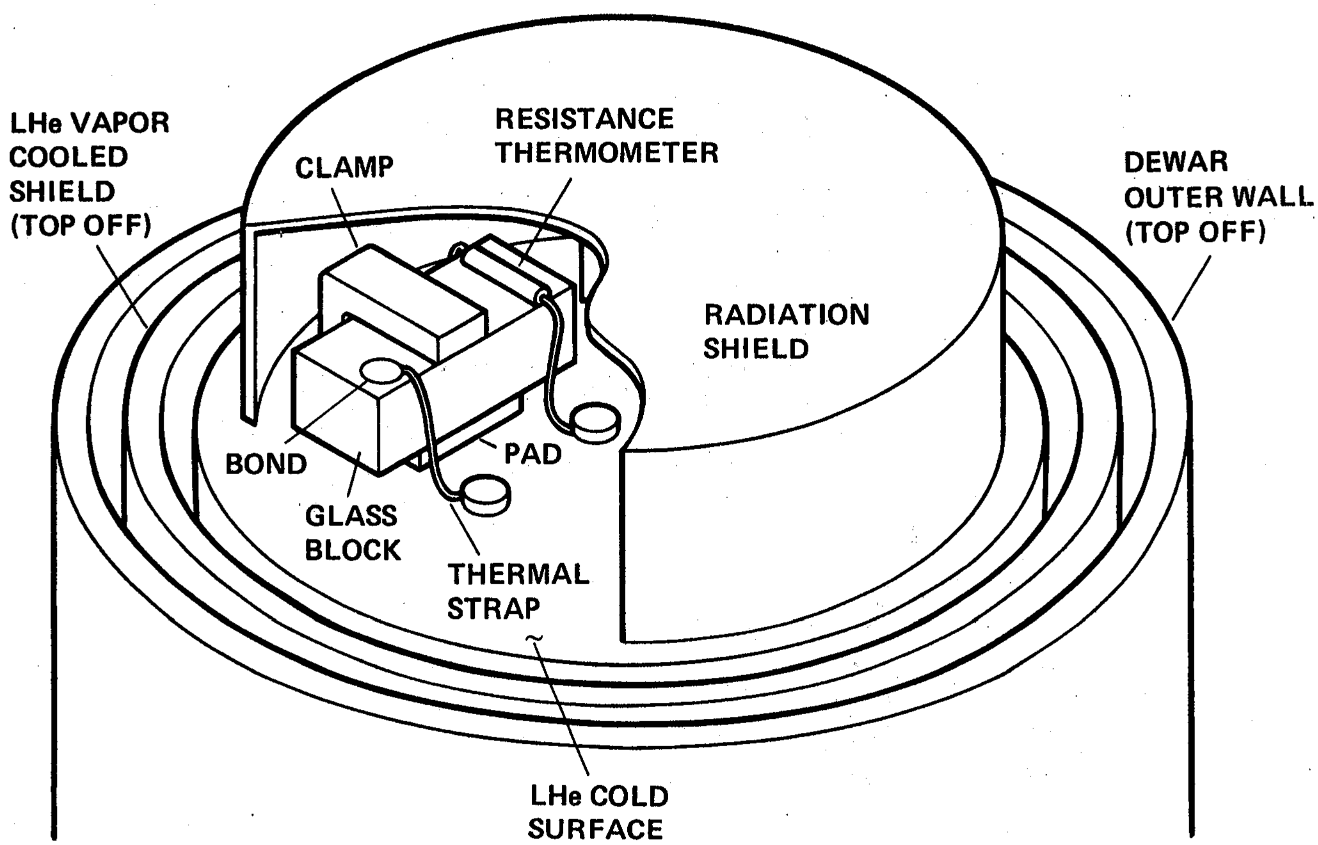

Figure 1.- A cut away diagram showing the Dewar with radiation shield, one of the three glass blocks with clamp and pad, the thermal strap, the location of the bond, and the location of the resistance thermometer.

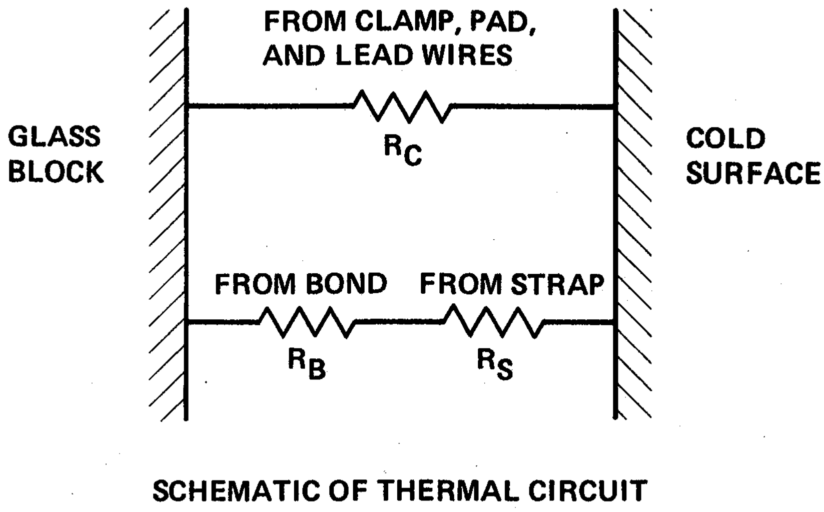

Figure 2.- A schematic diagram showing the thermal circuit used to calculate the thermal resistance of the bond.

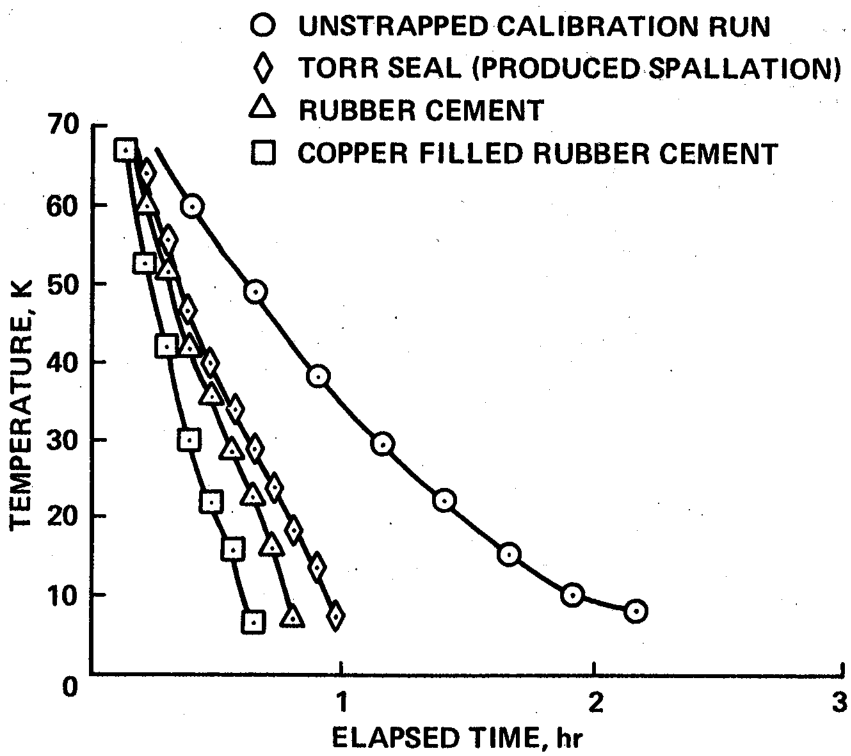

Figure 3.- A plot of the cooling rates of a strapped glass block with the following bonds applied: Torr Seal, rubber cement, and copper-filled rubber cement compared with a plot of the cooling rate of the same glass block without a thermal strap or bond.

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16. Abstract

Thermal contacts designed to cool a 50-cm diam fused-silica mirror to 5 K without serious distortion of the optical surfaces were tested. Rubber cement and copper-filled rubber cement were found to be well suited for maintaining thermal contact between the fused-silica and copper straps. A variety of other adhesives proved to be unreliable because they spalled the glass. The thermal resistances of the rubber cement and copper-filled rubber cement were determined. For the copper-filled rubber cement, the thermal resistances varied from 570 K/W at 60 K to 15,000 K/W at 10 K.

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