Calorimetric Measurement of Emissivity in Space Conditions

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ABSTRACT

Accurate thermal characterization of material surfaces is critical to spacecraft thermal management. A calorimetric experiment is developed to measure the total hemispherical emissivity of thermal control materials in space conditions. The space environment is simulated by a vacuum chamber with a liquid nitrogen blackbody shroud. A test sample is mounted on an electrically heated plate, which provides the energy input. The sample is thermally insulated on the sides and bottom by a heat shield. The heat shield is uniformly maintained at the sample temperature by an independent circuit of resistance heaters and temperature controllers. A steady state energy balance is applied to the test sample in order to determine the emissivity as a function of temperature and electrical power. The validity of this experiment is confirmed by testing materials with known emissivities, such as black paint and polished Error analysis of the system predicts a metals. reasonable accuracy for the emissivity measurement for sample temperatures between -50°C and 100°C.

NOMENCLATURE

As	exposed	test	sample	surface	area,	cm ²

- CTC coefficient of thermal coupling between the back plate and heat shield
- specific heat at constant volume, J/(kg*K) C_V
- radiated energy, W E
- view factor for radiation being emitted from F_{x-v} x to y
- G irradiated energy, W
- electrical current. A Ι
- Κ thermal conductivity, W/(m*K)
- LN_2 liquid nitrogen

Р	pressure, Pa
Q	heat transfer rate, W
R	universal gas constant, J/(kg*K)
Т	temperature, K
TC	thermocouple
V	voltage, V
<v></v>	average molecular speed, m/s
α	absorptivity
3	total hemispherical emissivity
λ	molecular mean free path, m
ρ	reflectivity
σ	Stefan-Boltzmann constant: W/(m ² *K ⁴)
Subscrip	ots:
В	blackbody
COND	conducted

- g gas generated by electrical power gen
- HS heat shield
- i
- in L
- loss
- 0 out S sample
- TC thermo couple

INTRODUCTION

The reliable operation of a spacecraft and its equipment requires temperatures to be maintained within specified ranges. Thermal control materials, such as passive coatings of paints and active electrochromic coatings, are very effective and lightweight solutions of spacecraft thermal management. Therefore. accurate thermal characterization of these materials is necessary to predict and control spacecraft temperatures.

Since there is no medium for convection or conduction in the void of space, radiation is the only mode of heat transfer between distant objects. Therefore, the operating temperature of a spacecraft is determined by the background temperature of

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space, the internal power generation of the spacecraft, and the total hemispherical emissivities, absorptivities, and reflectivities of the spacecraft surfaces. Total hemispherical emissivity, absorptivity, and reflectivity (now on referred to as emissivity, absorptivity, and reflectivity) are the averaged property values over all possible directions and wavelengths.

The objective of this research is to develop an experiment capable of accurately measuring the total emissivity of both active and passive thermal control materials including the recent developmental electrochromic coatings. A common method of measuring emissivity, absorptivity and reflectivity of material surfaces is with a spectrophotometer over specified wavelength and incidence angle ranges. This optical procedure is preferred for determining absorptivity and reflectivity, though it is very costly and time consuming. However, the less complicated calorimetric method is appropriate for determining the emissivity of materials in space conditions. This energy balance approach requires measuring the temperatures of the sample and its evacuated surroundings as well as power delivered to the sample. The calorimetric test is a simple and accurate way to measure the emissivity of thermal control materials in space conditions.

HEAT BALANCE ANALYSIS

By applying the law of conservation of energy to a test sample, its emissivity can be expressed in terms of the power input to the sample and the temperatures of the sample and its surroundings¹. Figure 1 is a conceptual illustration of a test sample assembly in a vacuum chamber surrounded by a blackbody enclosure.



Figure 1. Sample Energy Balance

For the sample control volume, the steady state energy balance is:

$$Q_{gen} + Q_{B-S} - Q_{S-B} - Q_L = 0 \tag{1}$$

 Q_{gen} is the electric energy supplied to the heater on the sample and Q_L is the unaccounted energy losses from the sample.

The terms Q_{B-S} and Q_{S-B} include both radiated and reflected energy exchanges between the blackbody enclosure and the sample.

$$Q_{B-S} = A_B F_{B-S} \left(G + \rho_B E \right) \tag{2}$$

$$Q_{S-B} = A_s F_{S-B} \left(E + \rho_s G \right) \tag{3}$$

Substituting for Q_{B-S} and Q_{S-B} in Eq.(1), the original energy balance yields:

$$Q_{gen} + A_B F_{B-S}(G + \rho_B E) - A_S F_{S-B}(E + \rho_S G) - Q_L = 0$$
(4)

From reciprocity, $A_SF_{S-B} = A_BF_{B-S}$.

$$Q_{gen} + A_S F_{S-B}[(1 - \rho_S)G - (1 - \rho_B)E] - Q_L = 0$$
 (5)

For an opaque surface: $1-\rho = \alpha$, and Eq.(5) becomes:

$$Q_{gen} + A_S F_{S-B}(\alpha_S G - \alpha_B E) - Q_L = 0 \tag{6}$$

Each of the terms in Eq.(6) can be expressed with fundamental parameters.

$$Q_{aan} = VI \tag{7}$$

$$G = \varepsilon_B \sigma T_B^{4} \tag{8}$$

$$E = \varepsilon_s \sigma T_s^4 \tag{9}$$

Therefore, the energy balance equation can be written as:

$$VI + \sigma A_s F_{S-B} (\alpha_S \varepsilon_B T_B^4 - \alpha_B \varepsilon_S T_S^4) - Q_L = 0$$
 (10)

Since the sample is in a blackbody enclosure, $\alpha_S = \epsilon_S$ and $F_{S-B} = 1$. Therefore,

$$VI + \varepsilon_S \sigma A_S (\varepsilon_B T_B^4 - \alpha_B T_S^4) - Q_L = 0$$
⁽¹¹⁾

Rearranging to solve for emissivity yields:

$$\varepsilon_s = \frac{VI - Q_L}{\sigma A_s(\alpha_B T_s^4 - \varepsilon_B T_B^4)}$$
(12)

Eq.(12) indicates that the emissivity of a test sample can be determined by using an evacuated, low temperature blackbody receiver, an electrical source for heat generation, and an insulating mounting assembly that eliminates heat losses from the back and sides of the test specimen.

 Q_L can be experimentally measured if samples of known emissivity are tested. If experimental Q_L is negligible (<1% of Q_{gen}) it can be ignored in Eq.(12).

EXPERIMENTAL SETUP

The test design is based on the heat transfer analysis model described in Ref 2. As shown in Figure 2, the test setup consists of the following components: the test sample, the sample mounting assembly, the vacuum chamber, the LN2 shroud, a temperature control system, and a data acquisition system.



Figure 2. Test Setup Diagram

Test Sample

This particular emissivity test is designed to use samples with a 35.72 mm x 35.72 mm square active area. Two types of calibration test samples are used for this experiment. The high emissivity sample is prepared by spraying a thin, uniform coat of ultra flat black paint on a 0.381 mm thick copper substrate. The low emissivity sample is a 0.889mm thick polished aluminum sheet.

Four thermocouples are attached directly to the back of the sample. The test specimen is clamped down on the heater and back-plate by the frame. A thin coat of thermal grease is applied to minimize contact resistance between the back-plate, heater and test sample.

Mounting Assembly

The entire test sample and mounting assembly are shown in Figure 3. The sample mounting assembly includes the back-plate, frame, and heat shield. The back-plate is a uniform temperature, but low thermal mass, 0.381 mm thick copper sheet. It has four through holes for the sample thermocouples. The test sample and its heater are clamped to the top of the back-plate by the frame and a thermocouple is soldered directly to the bottom of the backplate. The back-plate is attached to the heat shield lid -with nylon screws and ceramic washers in order to minimize conduction between the heat shield and the sample.

The heat shield is a thermal barrier designed to prevent heat transfer at the back and sides of the sample. It is a highly reflective copper box that is uniformly maintained at the back-plate temperature by an independent circuit of resistance heaters. Since TC1 matches TC6, there is no heat transfer through the back-plate and therefore the bottom of the sample is thermally insulated. At steady state, all of the sample heater energy (Q_{gen}) is transmitted directly to the test specimen. Therefore, the test sample control volume only exchanges heat with the blackbody shroud as indicated in the energy balance.





Vacuum Chamber

A 30.48 cm inner diameter vacuum chamber capable of holding a 5 x 10^{-5} Torr vacuum is used to simulate the void of outer space. Two ion gauges, one near the sample and the other near the pump, are used to measure the pressure during test runs. The vacuum chamber has feed-throughs for the LN2, power, and thermocouples. The mounting assembly is fixed to one of the chamber flanges by two low conductivity phenolic struts.

LN2 Shroud

The LN_2 shroud is a 27.94 cm diameter, 30.48 cm long blackbody receiver. It is held concentrically inside the vacuum by phenolic standoffs. By definition, a blackbody enclosure is a cavity whose inner surface is at a uniform temperature. The shroud is a 0.762 mm thick walled copper cylinder with one end capped. In order to ensure a uniform blackbody temperature distribution, a 6.35 mm diameter copper tube is wrapped and soldered around the outside of the shroud with a spacing of approximately one inch between coils. During the tests, the LN2 shroud is maintained at approximately -180° C.

The inside of the shroud is coated with a flat black solar absorbing paint that has an emissivity of 0.88 and an absorptivity of 0.95 according to the Advanced Materials and Process Engineering Laboratory of the Illinois Institute of Technology Research Institute.³ The effective emissivity and absorptivity of the shroud opening, based on its cylindrical geometry and surface emissivity, are 0.97 and 0.99 respectively.⁴

Temperature Control System

The temperature control system for this experiment consists of 16 thermocouples, two direct current power supply units, two resistance heater circuits and two temperature controllers. The entire test setup is instrumented with T-type thermocouples. The thermocouples are distributed as follows: five on the LN2 shroud, four on the test specimen, one on the back-plate, and six on the heat shield. The thermocouples are calibrated to \pm 0.1°C in an oil bath from 0 to 100°C and they are all soldered to their respective surfaces.

One of the DC units is used to supply power (Q_{gen}) to the sample heater. The voltage and current provided to the test specimen are measured by the data acquisition system. The other DC unit is used to power the heat shield resistance heater circuit. Figure 4 shows the two independent resistance heater circuits required for this temperature control system. The first is a simple circuit that includes the sample heater and its DC power supply. The second circuit is more complex and is designed to provide a nearly uniform temperature throughout the heat shield in order to minimize heat exchange between the backplate and heat shield. This circuit includes three heaters (one for the bottom, perimeter and lid) that are connected in parallel. External resistors, kept outside the vacuum chamber, are in series with the bottom and the lid in order to balance the power density of the heaters.

Two temperature controllers are used to match the heat shield temperature with the back-plate temperature. One controller reads the temperature of the thermocouple attached to the bottom of the back-plate (TC1) and converts it into a 4-20 mA signal. This current is transmitted to the second controller, which uses the signal as a remote temperature set point. This controller uses a relay to regulate the electrical current to the heat shield in order to maintain a time-averaged temperature within 0.1°C of the back-plate.



Figure 4. Heater Schematic.

Data Acquisition System

The data acquisition system for this experiment is the HP34970A. This system records the necessary data to determine the emissivity of the test specimen. Calibration curves were generated applied to all the temperature, voltage and current measurement channels. The accuracy of these measurements is addressed in the uncertainty error analysis. During the test runs, the data acquisition system scans its channels every 30 seconds, displays the data on a strip chart, and records it in a spreadsheet.

TEST PROCEDURE

The emissivity of the test sample is determined by achieving steady state for a given power input. This is accomplished by the following procedure. First, the entire setup is assembled and all of the instrumentation is checked and operating. Next, the vacuum chamber is evacuated. When the pressure in the chamber is below 1×10^{-4} Torr, the LN2 is supplied and the blackbody shroud reaches a uniform temperature of approximately -180°C. Note that if the LN2 is turned on before the vacuum reaches 0.1 mTorr, air moisture will condense on the shroud. Finally, the power supplies to the test sample and the heat shield are turned on and set to the desired wattage.

The experiment is carried out until the test sample stabilizes at the steady state temperature. A stability criterion for steady state is established by limiting the variation of temperature to remain within 0.5°C over a period of no less than 45 minutes. This is determined by examining strip chart plot of the sample temperature versus time (Figure 5). Once the test is terminated, the data are saved and exported to a spreadsheet. The emissivity of the sample is calculated by applying Eq.(12) to the data at steady state values. The test data is also used to examine other factors such as the temperature distribution of the heat shield and the change in sample temperature with respect to time.



Figure 5. Sample Temperature Strip Chart

ERROR ANALYSIS

Measurement Uncertainty

The uncertainty of this experiment is based on Eq.(12). From this relation, the root mean squared uncertainty of the emissivity calculation due to its experimentally measured parameters is:

$$\Delta \varepsilon = \begin{cases} \left(\frac{\partial \varepsilon}{\partial V} * \Delta V \right)^2 + \left(\frac{\partial \varepsilon}{\partial I} * \Delta I \right)^2 + \left(\frac{\partial \varepsilon}{\partial A} * \Delta A_s \right)^2 \\ + \left(\frac{\partial \varepsilon}{\partial T_s} * \Delta T_s \right)^2 + \left(\frac{\partial \varepsilon}{\partial T_B} * \Delta T_B \right)^2 \\ + \left(\frac{\partial \varepsilon}{\partial \alpha_B} * \Delta \alpha_B \right)^2 + \left(\frac{\partial \varepsilon}{\partial \varepsilon_B} * \Delta \varepsilon_B \right)^2 \end{cases}$$
(13)

The delta values, shown in Table 1, are constant and depend on instrumentation accuracy while the partial derivatives are based on the parameters for a specific test run. Applying Eq.(13) to this experiment reveals an uncertainty of approximately +/- 3.10% for the emissivity measurement. This overall uncertainty is dominated by the random error associated with the effective sample surface area for two reasons. First, A_s is a small area. Second, ΔA_s is relatively large (about 0.425 cm²) because it is very difficult to line up the effective sample surface area exactly with the square hole in the lid. Since the uncertainty of temperature and power measurements do not contribute significantly to the overall uncertainty, $\Delta \epsilon$ is considered consistent for all test runs.

Table	1.	Measurement	Uncertainty	V

ΔV (volts)	1.00E-04	
∆l (amps)	1.00E-04	
∆A (m^2)	4.25E-05	
∆Ts (K)	0.1	
ΔT _B (K)	0.5	
$\Delta \alpha_B$	0.01	
$\Delta \epsilon_{B}$	0.01	

Systematic Error

<u>Blackbody Assumption</u>: The assumption that the shroud acts as a perfect blackbody simplifies the heat transfer analysis. Without these simplifications, Eq.(16) would be:

$$\varepsilon_{s} = \frac{VI}{\sigma A_{s} F_{s-B} (\alpha_{B} T_{s}^{4} - \frac{\alpha_{s}}{\varepsilon_{s}} \varepsilon_{B} T_{B}^{4})}$$
(14)

For this test configuration, $F_{S-B} = 1$ since the test sample is completely submerged in the shroud and therefore emits all of its energy from the front surface to the blackbody enclosure.

The blackbody assumption that $\alpha_S / \epsilon_S = 1$ is valid for cases where there is a small difference between the sample and blackbody temperatures but does not necessarily hold true for significant temperature differences.³ Error analysis of this assumption was done by varying the ratio of α_S / ϵ_S for different test conditions. It was determined that error associated with this ratio assumption is negligible except for high emissivity, low absorptivity samples (such as white coatings) at low steady state temperatures. For such cases, the emissivity calculated by Eq.(12) is overestimated by as much as 0.15%. <u>Gas Conduction</u>: Because the vacuum chamber used for this test is not a perfect void, there exists some energy exchange between the sample and the blackbody shroud via gas conduction. According to kinetic theory, the conductivity of gas is ¹ (Note that ρ is density in Eq.(15)):

$$K = \frac{1}{3} \rho \langle v \rangle \lambda C_v \tag{15}$$

Therefore the heat flux via gas conduction is:

$$Q''_{COND} = \frac{1}{3} \rho \langle v \rangle \lambda C_v \frac{dT}{dx}$$
(16)

In an unrestricted volume, the conductivity of a gas is independent of pressure.¹ This is because density is directly proportional to pressure while the mean free path is inversely proportional to pressure. However, at 5 x 10^{-5} Torr, the mean free path of air exceeds the distance that the shroud allows an air molecule to travel. Therefore, the air is rarefied and Eq.(16) is no longer valid for the situation. Rather, Eq.(17) is the appropriate expression for energy transferred between two surfaces in rarefied conditions.

$$Q_{COND} = A_{S} P \sqrt{\frac{2R}{\pi T_{g}}} (T_{S} - T_{B})$$
(17)

For this experiment, the temperature of the gas between the shroud and the sample mounting assembly (T_g) is estimated as the area averaged surface temperature. At a sample temperature of 100°C, the heat lost by means of gas conduction is approximately 3.1 mW, which leads to an emissivity overestimation 0.22%. At a sample temperature of -50°C, the heat lost by means of gas conduction is approximately 1.5 mW, which leads to an emissivity overestimation of 0.86%.

<u>Heat Loss (Q_1) from Sample</u>: A small quantity of heat may be transferred between the back-plate (sample) and heat shield because of slight temperature differences between the two components during a test, as shown in Figures 6 and 7. The reasons for this heat loss are radiation, gas conduction, and conduction through the mounting nylon screws, ceramic washers, and thermocouple leads.

A test was conducted to determine Q_L in Eq. (12) by defining a thermal coupling coefficient between the back-plate and heat shield at near room temperatures. For this test, both the back-plate and the heat shield

are initially set at 30° C and the power to the backplate heater is recorded. Then, the heat shield temperature is lowered to 20° C while the back-plate is maintained at 30° C by increasing the power to its heater. This test was repeated by maintaining the back-plate temperature at 30° C while varying the change in heat shield temperature from 30° C. The thermal coupling coefficient was calculated as:

$$C_{TC} = \frac{\text{change in power to the backplate heater}}{\text{change in heat shield temperature}}$$
(18)

The thermal coupling coefficient for back-plate and the heat shield at near room temperature is approximately $0.02 \text{ W/}^{\circ}\text{C}$. The amount of heat transferred between the backplate to the heat shield can be defined as:

$$Q_{L} = C_{TC}(T_{BP} - T_{HS})$$
(19)

Figure 7 shows that the average temperature difference between the back-plate and the heat shield is approximately 0.07° C for any particular test run. Therefore, the unaccounted heat loss from the back-plate to the heat shield is 1.4 mW. By neglecting this energy loss, Eq. (16) overestimates emissivity by about 0.24% for this test run at room temperature. Since the time averaged temperature difference between the back-plate and heat shield is always within +/- 0.1°C, the emissivity inaccuracy due to thermal coupling is +/- 0.35%.



Figure 6. Back-plate and Heat Shield Temperature Profiles

<u>Overall Test Error</u>: The overall error for the calorimetric test is a combination of both measurement uncertainties and systematic errors. Taking these factors into account, the overall error of the system is +3.23/-3.67% for sample temperatures around 100° C and +2.59/-4.31% for sample temperatures near -50° C.



Figure 7. Temperature Difference between the Backplate and Heat Shield

RESULTS AND DISCUSSION

The validity of this experiment is confirmed by testing materials with known emissivities at near room temperatures. Krylon Ultra-Flat Black paint (hereinafter called black paint) is used as the high emissivity reference and polished aluminum is used as the low emissivity reference. The results of these preliminary tests are presented in Table 2 and Figure 8.

Table 2. Test Results Matrix

Sample	Run	Power (mW) +/-0.5 mW	Steady State Temp (C)	Measured Emissivity	Ref Std Emissivity ^{4,5}
Flat Black	1	546.4	26.65	0.055	0.052
Paint 2	1	546.4	26.65	0.955	0.953
Flat Black Paint 2	2	666.6	42.70	0.944	0.953
Polished					
Aluminum	1	96.0	38.25	0.144	0.09 - 0.12
Polished					
Aluminum	2	91.9	38.74	0.137	0.09 - 0.12

According to published data, black paint has an emissivity of approximately 0.953 within the thermal radiation wavelength range at near room temperatures^{4, 5}. The black paint on a copper substrate sample was successfully tested twice. The average measured emissivity for the black paint test runs is 0.950.

Typically, polished aluminum plate has an emissivity of 0.09 to 0.12 near room temperature⁴. Since the sample prepared in the lab is not perfectly flat and has some slight surface blemishes, the higher emissivity value is used as the standard. The polished aluminum sample was tested twice with an average emissivity value of 0.141. All four of the initial tests produced measured emissivity values that lie within the predicted error limits. These measurements and results provide confidence to test other samples such as variable emissivity electrochromic devices. An important lesson learned is that extra care has to be taken in measuring the electric power supplied to the sample heater.



Figure 8. Emissivity Experimental Results

CONCLUSIONS

A calorimetric type of experimental setup is developed to measure the total hemispherical emissivity of planar opaque samples. The sample enclosure size and shape are appropriately chosen to apply the radiation laws and blackbody assumptions. The space conditions are simulated by an LN₂ shroud and vacuum chamber. This system is considered adequate for total emissivity measurement or verification of radiative surfaces that do not require solar absorptivity data. The experimental accuracies are within ±5% from -50°C to 100°C. The low emissivity samples such as polished aluminum are more difficult than high emissivity samples for experimentally controlling the heater power and testsample temperature. The present setup is easily adaptable for variable emittance test samples.

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