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Note

Emissivity measurements of solid propellants

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The application of optical radiation methods to surface temperature determinations requires the knowledge of the emissivity of the surface in question, in order to minimize the error of the measurements[1]. These methods are very convenient, especially for those processes where there is a very fast temperature variation with time.

We report here the first part of the research work we are carrying out to determine the surface temperature change as a function of time in the process which occurs during the pre-ignition period of a solid propellant under the high energy flux of a CO_2 laser beam. The selected method was total radiation pyrometry, used previously by Bouck[2] and Richardson[3]. In order to apply this technique, although with a better accuracy in the value of the temperature obtained, it was considered advantageous to know the total normal emissivity of the propellants. We did not find references on emissivity of solid propellants in the available literature, except the works by Powling and Smith[4] and Rogers and Suh[5], who used single-color pyrometry. Bouck[2] and Richardson[3] did not include the emissivity in the voltage–temperature conversion done with a black body precalibration. Therefore, they assume $\varepsilon = 1$. Thus, the results they obtained were the brightness temperatures*.

In the present work we determined the total hemispherical emissivity of double-base and composite propellants with a calorimetric method (differential scanning calorimetry) using the techniques described by Rogers and Morris[6] and Ortiz and Rogers[7].

EXPERIMENTAL

The equipment used was a Perkin-Elmer differential scanning calorimeter, Model DSC-1B.

The emissivity determinations were carried out on 6 mm diameter propellant discs. These discs were obtained slicing a propellant rod with a microtome with the

*See P. W. Kruse, L. D. McGlauchlin and R. B. McQuistan, *Elements of Infrared Technology: Generation, Transmission, and Detection*, Wiley, New York, 1963, p. 20.

desired thickness and then punching out the discs with a cork-borer. The sample thickness was measured with a dial gauge (± 10 μm).

We employed aluminum sample pans without lid, provided by Perkin-Elmer for the DSC-1B equipment. As a reference surface we used aluminum discs with a 5 μm electrolytic oxide coating, which have an emissivity of 0.7 at 400 K[7]. To apply the Rogers and Morris technique[6] we prepared an aluminum block to place it in the low-temperature cover furnished by Perkin-Elmer. The block was heated with a resistance heater disc placed on it. The temperature was measured with a Fe–constantan thermocouple and a digital voltmeter (Digital Multimeter, Model 171, Keithley Instruments Inc.). We removed the insulator material from the base of the receiver, which is the sample and reference cover, and first polished and then painted the surface with optical black paint.

To apply the Ortiz and Rogers technique[7] we used the same cover without the aluminum block, since it is convenient to avoid illumination differences between sample and reference through the window of the standard cover, which affect the measurements[8]. Tables 1 and 2 describe the double-base and composite propellants used in this work.

TABLE 1

DOUBLE-BASE PROPELLANTS TESTED

Propellant	*Heat of explosion* ($cal\ g^{-1}$)
PHE-1	835
PHE-2	920
PHE-3	1022
PHM-2	790
PHM-9	836
P-1	808
P-2	768
N-5	850

TABLE 2

COMPOSITE PROPELLANTS TESTED

PBAA = polybutadiene-acrylic acid; PBCT = polybutadiene-carboxy terminated.

Propellant	*Binder*	*Additives*
61/74	PBAA	aluminum
66/74	PBAA	—
67/74	PBAA	aluminum
68/74	PBAA	aluminum–ferric oxide
70/74	PBAA	aluminum–ferric oxide
71/74	PBAA	aluminum
90/74	PBAA	carbon
13/75	PBCT	—

RESULTS AND DISCUSSION

In order to know the transmittance of the sample discs, we ran an IR spectra of composite and double-base propellant samples (thickness 120 μm) between 2.5 and 11 μm. The samples did not transmit in the tested range.

Prior to the emissivity determinations, we obtained the propellant thermograms to verify if there was not a chemical reaction at the chosen temperature. For composite propellants we did not find reactions at 400 K, thus the emisivity was determined at that temperature. For double-base propellants, the thermograms did not show reactions at 400 K (8 K/min heating rate), but between 370 and 400 K there was a continuous displacement of the pen in the endothermic direction. At 350 K we did not observe this displacement and thus the measurements for double-base propellants were carried out at that temperature, assuming an emissivity 0.7 for the reference discs, since the total normal emissivity vs. T remains almost constant between 400 and 350 K[9].

In order to compare the Rogers and Morris[6] and Ortiz and Rogers[7] techniques we determined the emissivity of a composite propellant at 400 K, using different sample thicknesses (180 to 600 μm). We obtained an average emissivity value (6 measures) of 0.86 with the first method[6], and of 0.88 using the second one[7]. This last procedure allows the elimination of the necessary correction measurements of the first technique and, as it requires less determinations, there is a lower error affecting the calculated emissivity[10]. As the difference between both average calculated emissivities is less than 3%, and considering that the Ortiz and Rogers technique[7] is the simplest, we used it for our emissivity determinations. Table 3 gives the emissivities obtained in this study for different sample thicknesses.

TABLE 3
EMISSIVITY OF SOLID PROPELLANTS

Propellant	*Thickness (μm)*	*T (K)*	*ε*
PHE-1	120	350	0.83
	150	350	0.84
	210	350	0.84
PHE-2	160	350	0.83
	580	350	0.84
PHE-3	140	350	0.84
	310	350	0.85
PHM-2	350	350	0.84
	540	350	0.85
PHM-9	150	350	0.83
	390	350	0.84
P-1	150	350	0.83
	370	350	0.84
	410	350	0.84

(Table continued on p. 380)

TABLE 3 (*continued*)

Propellant	*Thickness (μm)*	*T (K)*	*ε*
P-2	300	350	0.84
N-5	160	350	0.83
	410	350	0.84
61/74	210	400	0.84
	390	400	0.86
66/74	220	400	0.86
	390	400	0.88
67/74	160	400	0.85
	370	400	0.86
68/74	210	400	0.84
	380	400	0.85
70/74	190	400	0.83
	370	400	0.84
71/74	150	400	0.85
	360	400	0.86
90/74	200	400	0.86
	390	400	0.87
13/75	220	400	0.84
	400	400	0.88

The emissivity values obtained for the tested propellants show small differences according to thickness and kind of propellant tested. As Table 3 shows, the emissivity may change from 0.83 to 0.88, thus we think that 0.85 is a good general approximation for solid propellant emissivity.

The calculated values are the total hemispherical emissivities, but taking into account that for rough surfaces of dielectric materials the hemispherical emissivity/normal emissivity ratio tends to unity[11] we may consider them as the total normal emissivity. Taking 0.85 as the emissivity of the solid propellant surface, the temperature error obtained (taking the surface emissivity as unity) is 5% in defect[1]. However small this percentage may seem, one should consider that optical radiation methods have several additional errors. An improvement of 5% in the accuracy of the results may be an important feature in the true temperature determination of a heated or burning surface.

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