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Gas Phase Reactions near the Solid-Gas Interface of a Deflagrating Double-Base Propellant Strand

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The abrupt changes in the burning rate-pressure curve of M-2 double base propellant at low pressures were experimentally investigated and were found to be caused by the existence of a series of gas reactions in the "fizz" zone very near the solid-gas interface. The distance between these gas reactions and the solid-gas interface decreases with increase in pressure. The gas reaction zones are at temperatures higher than the surface temperature of the solid-gas interface. Using a model that assumes that the gas reactions only affect the heat transfer rate from the gas to the solid, the change in the burning rate of the propellant as a function of the initial temperature of the propellant was predicted. The agreement between the predictions of the theoretical model and the experimental results is good.

Nomenclature

A = area
c = specific heat of the solid propellant
d = distance from the solid-gas interface to regime R
E = activation energy
F = frequency factor
H = heat released by the reactions in the solid per unit volume
k = thermal conductivity of the solid propellant
m = reaction order
P = pressure
Q = heat transfer
R = universal gas constant
R_f = radiant flux
t = time
T = temperature
U = velocity of propagation of a regime of gas reactions
V = velocity of solid propellant (burning rate)
V_g = velocity of gas
W = constant of proportionality
Y = fraction of reactants available to react
Z = distance
α = parameter defined in text
β = radiation absorption coefficient
γ = dimensionless parameter defined in text
λ = parameter defined in text
ρ = density of solid propellant
τ = dimensionless time
Υ = parameter defined in the text

Subscripts

d = pressure coefficient
E = end
f = flux
g = gas
i = intercept
1 = for specified conditions
0 = initial
P = pressure
R = a regime
S = surface
T = total
v = volume

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Introduction

IN the late 1940's and early 1950's Crawford et al.,[1] Heller and Gordon[2] and Klein et. al.[3] reported the experimental data and ideas which led to a combustion model of strand burning double-base propellant (herein referred to as Model I). The model and mathematical analyses based on it were presented by Parr and Crawford[4] and Rice and Ginell.[5] The major features of Model I are foam, fizz, dark, and flame zones.

The foam zone is the region of transition from a solid to a gas and appears to be a layer of foaming viscous liquid and bubbles. In the mathematical analysis Parr and Crawford[4] assume the net chemical reaction to be exothermic, to occur over a finite region, to be continuous, to be a first-order reaction, and to be described by an Arrhenius expression. Rice and Ginell[5] assume the net chemical reaction to be exothermic, to occur in a plane at (or just within) the solid, to be a zero-order reaction, and to be described by an Arrhenius expression evaluated at the surface.

The fizz zone is the region of gas close to deflagrating solid in which there occur exothermic chemical reactions. Parr and Crawford[4] assume the net chemical reaction to occur over a finite region which extends from the surface of the solid to the beginning of the flame reactions, to be continuous, to be a second-order reaction, and to be described by an Arrhenius expression. Rice and Ginell[5] assumed the net chemical reaction to occur over a finite region which extended from the surface of the solid to the dark zone, to be continuous, to be second order, and to be described by an Arrhenius expression. The separation of the fizz zone from the flame zone by a nonreacting zone is substantiated by the species data of Heller and Gordon.[2]

The dark zone is the region of nonreacting gas which exists between the fizz and flame zones. The length of this zone decreases with increasing pressure. The measurements of chemical species in the gas by Heller and Gordon[2] were made in this zone and in the flame zone. The existence of this dark zone was further verified by the pictures and spectroscopic data recorded by Heath and Hirst.[6]

The flame zone is the final region of chemical reactions and emits radiation in the visible band. Parr and Crawford[4] have not analyzed the flame zone but have stated that their analysis and assumptions of this zone would be identical to their analysis of the fizz zone, except the order of the reaction would be about three. Rice and Ginell[5] have analyzed the flame as they did the fizz zone, except the reaction order is taken to be two and a half. Both Heller and Gordon[2] and Heath and Hirst[6] have reported the distance of the flame from the solid, but only Heath and Hirst[6] have presented the thickness of the flame zone and have measured the spectrum emitted by the flame. The analysis of the spectrum data shows that the flame radiates as a graybody and the temperature of the flame is about 2400°K. Sottor[7] using the data of Heller and Gordon[2] for initial concentrations of species has presented a chemical kinetic analysis of the flame zone.

The preceding analyses that yield a relation giving the pressure dependency of strand burning rate were in agreement with the interpretation that the experimental data could be described by

$$V = V_i + V_d P^m \tag{1}$$

However, for low pressures, Model I explains neither the abrupt change in burning rate vs pressure for constant initial temperature reported by Suh and Clary,[8] nor the abrupt change in burning rate vs initial temperature for constant pressure reported by Aleksandrov et al.[9]

Experimental investigations were conducted to determine the causes of the aforementioned discrepancies between the model and the observations made at low pressures. The double-base propellant used was M-2 and some of its characteristics are given in Appendix A. The experimental investigations were performed using high-speed photography for observing physical phenomena, high-speed oscillography for recording temperature of an embedded fine thermocouple, and fuse wire for measuring the burning rate.

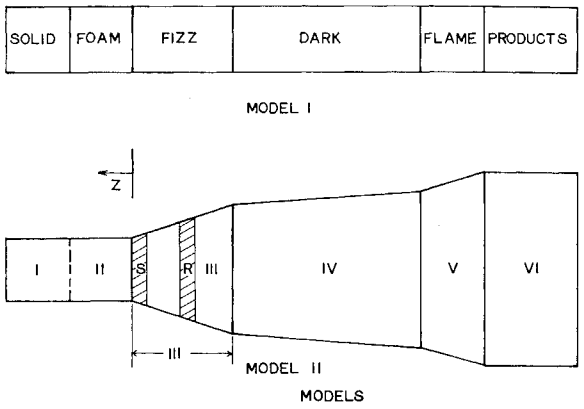

Fig. 1 Models.

The present experimental work shows that there is a series of abrupt changes in the burning rate pressure relations and that there exist distinct gas-phase reactions very near the gas-solid interface. The changes in the burning rate pressure relations occur as the distinct regimes of gas reaction reach the solid-gas interface. These gas phase reactions generally occur in what has previously been called the fizz zone.

The aforementioned observations require that Model I be modified such that the results of the mathematical analysis agree with the measured data. The predictions of the analysis based on the modified strand combustion model (herein referred to as Model II) agree with the experimental results presented in this paper and is consistent with the behavior reported by Aleksandrov et al.[9]

The basic differences between the models are in the gas zone next to the gas-solid interface, i.e., Zone III of Model II and the fizz zone of Model I, as shown in Fig. 1. Whereas in Model I the fizz zone is assumed to be of approximately constant cross-sectional area, in Model II it is shown to be divergent. The implication of the divergence is that the gas velocity decreases as the gas moves away from the gas-solid interface. Furthermore, in Zone III of Model II there exists a series of gas reactions, which were discovered by the experiments described in this paper.

It should be noted that both models describe the "cigarette" burning of double-base propellant strands. Neither the combustion of composite strands nor the combustion of double-base propellant in a rocket chamber can be determined from Model II. The extension of Model II to the case of combustion of double-base propellant in a rocket chamber requires much additional detailed information about the distinct regimes of reactions and about effects of the flowfields caused by the rocket chamber geometry.

Experimental Apparatus and Procedures

Experiments were conducted in two combustion chambers which will be referred to as the low-pressure combustion chamber (LPC) and the high-pressure combustion chamber (HPC). In the HPC chamber, data were gathered on the burning rate of the propellant as a function of the pressure for an initial propellant temperature of 25°C, and on the temperature distributions in the propellant and gas near the burning surface for steady state burning. In the LPC chamber, data were gathered on the surface temperature of the propellant for steady state burning at 1 psia and on the physical characteristics during steady-state burning.

The atmosphere control system was such that the chamber's pressure could be regulated at any value from $\frac{1}{2}$ psia to

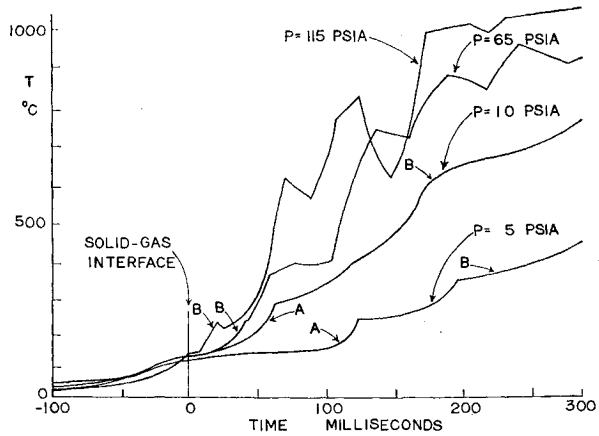

Fig. 2 Temperature distributions.

200 psia and temperature could be varied from 0°C to 200°C. In all tests air was initially used to bring the chamber to the selected temperature. Then argon was used to purge the air from the chamber and to keep the pressure and temperature in the chamber at the selected value. The initial temperature of the propellant was kept within ±1°C and the initial pressure within ±0.02 psia. During the tests the maximum variation in pressure was about 5% of the initial pressure.

The burning rate was determined in the HPC chamber by the melting of the fuse wires, which were thirty gauge Indium alloy wires with a melting temperature of about 280°C. The temperature profile was obtained by placing a platinum/platinum-10% rhodium thermocouple in the propellant. The diameter of the thermocouple bead was about three times the diameter of the leads. The emf produced by the thermocouple was amplified 150 times by a high-impedance amplifier (Honeywell Accudata 104) and recorded by a high-speed oscillograph (Honeywell Visicorder 1508, 80 in./sec). The recording galvanometer (Honeywell M-1650) used in the oscillograph had a frequency response of 1650 Hertz. The deflection trace of the galvanometer was transformed to temperature using standard tables with reference junction corrections.

Although the placement of the fuse wires was simple, the embedding of the thermocouple was difficult. In order to obtain reproducible temperature profiles, the ratio of the diameters and the physical sizes of the thermocouple bead and lead wires have to be constant and the geometric configuration of the thermocouple in the propellant has to be the same in all the experiments. In these experiments a template was used to place the bead at the apex of the angle formed by the straight leads. Further details of the process

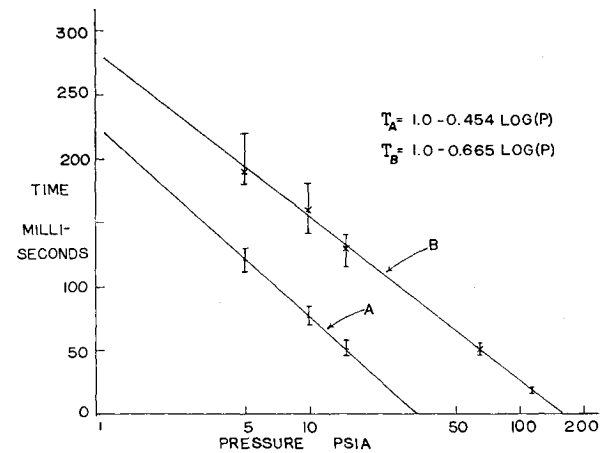

Fig. 3 Temperature "jumps."

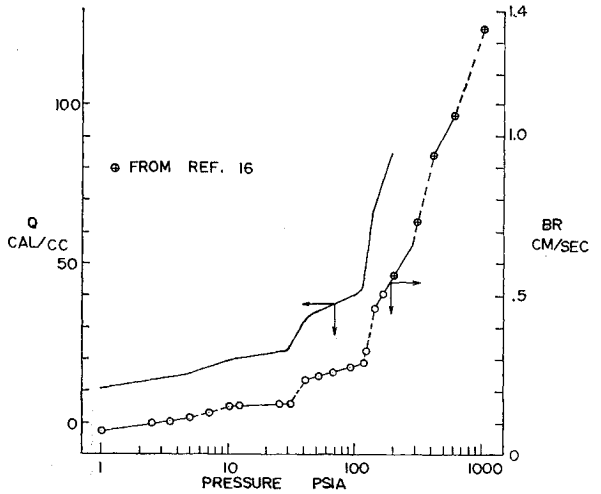

Fig. 4 Burning rates for constant initial temperature.

of embedding thermocouples in this manner are reported by Suh and Tsai.[10]

In the LPC chamber, the physical phenomena were recorded by a high-speed camera (Fastax, Category II, 500-9000 frames/sec.) and the surface temperature was measured by the photo-electric-magnetic infrared sensor (Honeywell PEM cell) and/or by an Alumel/Chromel thermocouple restricted to the deflagrating surface. The diameter of the thermocouple was 25.4 microns.

In the HPC chamber ignition was accomplished by passing a current through a spiral of wire pressed against one end of the specimen. In the LPC chamber ignition was accomplished by heating the specimen with uniform radiant flux on one end of the specimen. The radiation was produced by a 1.25 cm. square plate of thin metal (Kanthal alloy) which was at a temperature of 1400°K. Further details of experimental apparatus and procedures are reported in Ref. 11.

Experimental Results

Although the earlier investigators[1–5] implied that Zone III of Model II was a foaming viscous material, the high-speed movies taken at 1000 to 4000 frames/sec showed that it is not. The pictures showed a solid surface with small solid particles moving around on it. In addition to the small moving particles on the surface, the pictures also showed a random distribution of larger particles, which appeared to be black, that remained stationary on the surface until they left the surface. When the propellant receded past one of the black particles, it flew off into the gas. Comparison of the particles with the thermocouple bead indicated that the moving particles were of the order of 10μ in size and the black particles were of the order of a hundred microns.

Typical temperature distributions in the solid and gas near the deflagrating solid as functions of time are shown in Fig. 2. Since the time in both Figs. 2 and 3 is the axial distance from a point to the solid-gas interface divided by the burning rate, it is identical to the time for the embedded thermocouple to traverse the distance. Negative time represents points in the solid, positive time represents points in the gas, and zero time indicates the solid-gas interface. The position of the solid-gas interface was located at subatmospheric pressures by careful matching of high-speed pictures of the thermocouple emergence from the solid with the recorded thermocouple emf.

The positions of the temperature "jumps" A and B were observed to change with pressure, as indicated in Fig. 3. The pressure at which the temperature jump reached the solid-gas interface was determined by extrapolating the measured data.

The systematic measurement of the burning rate for an initial propellant temperature of 25°C for selected pressures from 1 to 165 psia yielded the data plotted in Fig. 4. The pressure ranges in which the changes in the burning rate-pressure relations occurred were observed to coincide with the pressures at which regimes A and B reached the solid-gas interface. The theoretically predicted heat transfer from the gas to the solid is also included in Fig. 4.

Theoretical Model

The theoretical investigation of steady-state "cigarette" burning of a double-base solid propellant is based upon the application of the conservation of mass and energy principles to the deflagrating solid. The diameter is assumed sufficiently large so that edge effects are negligible and that a one-dimensional model is adequate. The absorption of radiation is included by a volumetric absorption description as reported by Rogers and Suh.[12] The heat generation rate in the solid is assumed to be proportional to the solid gasification rate as described by an Arrhenius expression.

The determination of the parameters in the Arrhenius expression was reported by Thompson and Suh.[13] Briefly, the order of the reactions in the solid was taken to be zero, since Kirby[14] found from the heat of reaction data that the reactions during the early stages of combustion could be predicted best by this order. The heat of reactions, the frequency factor, and the activation energy were determined by matching the theoretical values of ignition delay time and surface temperature with the experimental values measured for several levels of radiant flux. The values of these parameters given in Appendix A are used in the present analysis.

With these assumptions the conservation of mass and energy equations may be written as

$$V = \int_0^\infty F \cdot \exp(-E/RT)dZ \tag{2}$$

$$(d^2T/dZ^2) + (\rho cV/k)(dT/dZ) = (R_f\beta/k)\exp(-\beta Z) - (HF/k)\exp(-E/RT) \quad \text{for } 0 \leqslant Z \leqslant \infty \tag{3}$$

where boundary conditions are $T = T_0$, $dT/dZ = 0$ at $Z = \infty$; $T = T_s$ at $Z = 0$. The origin of the coordinate system is fixed at the solid-gas interface with the positive Z direction being into the solid.

The boundary conditions are mathematically overspecified, but the conditions are compatible. They were used because they made the numerical calculation easier. The aforementioned equations were expressed in finite difference form, programed in Fortran IV language, and solved by an IBM 7040 Digital Computer. The solution was obtained by an implicit technique with the initial temperature and burning rate as the input variables. An iterative procedure was used to vary the surface temperature until the computed deflagration rate of the propellant was equal to the burning rate. The surface temperature, the temperature distribution in the solid, and the temperature gradient at the surface were the outputs. The heat transfer rate from the gas to the solid was computed by applying Fourier-Biot law of conduction heat transfer at the surface of the solid. Further details of the numerical solution are in Ref. 11.

Discussion

When pictures of the deflagrating surface taken at 300 to 500 frames/sec were viewed, there appeared to be a foam zone, but movies of the same scene taken at 1000 to 4000 frames/sec did not show a foaming layer. In the higher resolution pictures (1000-4000 frames/sec) the apparent foaming layer was seen to be a photographic effect produced by solid particles moving around on the surface. In addition, pictures taken of the gas evolving from the solid indicated that the flow of gas diverges. This fact was shown by the pattern of the deposits which condensed upon a thin vertical plate immediately under the burning specimen.

Since the gas diverges as it moves away from the gas-solid interface, the gas velocity must decrease, provided that the change in the density and number of moles of gas is small. From the temperature profiles it is observed that the temperature of the gas, especially at low pressures, remains practically constant from the interface to a point (about 130μ for 5 psia) in the gas where the temperature abruptly jumps. Since the pressure does not change, the velocity of the gas flowing into the regime in which the temperature jump occurs must be less than the velocity of the gas leaving the solid. Similar arguments apply to the regions between the regimes of temperature jumps.

The existence of a series of abrupt temperature jumps in the temperature profiles implies the existence of standing waves in the gas. To insure that the temperature jumps are independent of the thermocouple's response characteristics, several temperature profiles were measured with thermocouples one half the diameter (lead wires 12.7μ, bead 38μ) of those used to produce the data shown in Fig. 2. The position of the first and third jumps were the same distance from the solid surface for both size thermocouples, but the position of the second and fourth jumps were different. Since the second and fourth jumps were one bead diameter behind the first and third jumps, respectively, the second and fourth jumps were seen to be effects caused by the size of the thermocouple relative to the standing wave. The interpretation of the stationary waves (first and third temperature jumps) in the gas as regimes of chemical reactions is evident as shown in the next two paragraphs.

The existence of stationary gas reactions in Zone III and the influence of the ambient pressure on their positions relative to the gas-solid interface may be explained in terms of the velocity of reaction propagation and the velocity of the gas. Since the gas leaving the gas-solid interface is a homogeneous fuel-oxidizer mixture, the propagation velocity of gas phase reactions may be written as $U = U_0 P^{1/m}$. The direction of the propagation is toward the gas-solid interface since the reactants are generated at the interface. However, the gas velocity is in the opposite direction. The gas reactions assume a stationary position with respect to a moving coordinate system fixed at the gas-solid interface when the propagation velocity and the gas velocity are equal. The change in the position of the gas reactions with increase in pressure may be determined qualitatively in terms of the changes in the burning rate, the reaction propagation velocity, and the gas velocity. The net movement is toward the gas-solid interface with increase in pressure.

The heat-transfer rate, which is shown in Fig. 4, from the gas into the solid was theoretically computed from the temperature gradient at the surface obtained from Eqs. (2) and (3). Figure 4 shows that the "jumps" in the heat-transfer rate correspond to the "jumps" in the burning rate at the same pressure regimes. It can be shown theoretically, in addition to the experimental evidences presented earlier, that the "jumps" in the burning rate are due to the arrival of the gas reactions at the solid-gas interface. For this purpose the computed heat transfer may be approximated by $Q = Q_0 + Q_E \log(P)$. If the temperature of the regime of gas reactions and the surface temperature are only a weak function of pressure, the conduction heat transfer from the gas to the solid may be approximated by $Q = kA(T_R - T_S)/(Z_R - Z_S) = kA(T_R - T_S)/Vt$. Equating these two equations and solving for $1/t$ gives: $1/t = \alpha + \lambda \log(P)$, where $\alpha = Q_0V/kA(T_R - T_S)$ and $\lambda = Q_EV/kA(T_R - T_S)$. Or in nondimensional form $1/\Upsilon = 1 + \gamma \log(P)$ where $\Upsilon = t\alpha$ and $\gamma = \lambda/\alpha$. When $\gamma \log(P)$ is less than one, Υ can be

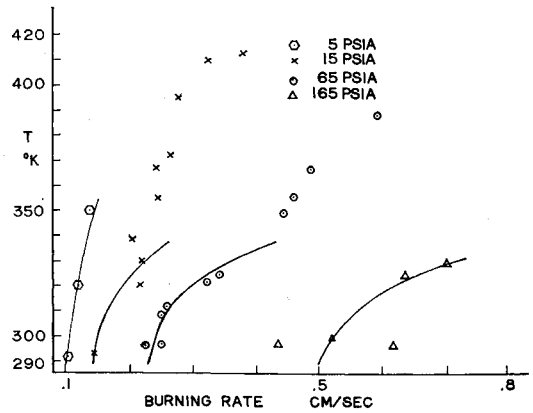

Fig. 5 Isobaric burning rates.

approximated by the following equation

$$\Upsilon = 1 - \gamma \log(P) \tag{4}$$

As indicated in Fig. 3, the aforementioned relation describes the experimentally measured movement of the regimes.

The gas regimes are not due to catalytic effects of the platinum, since Chromel-Alumel thermocouples also produced "jumps" A and B. A sliver of carbon embedded in the propellant began glowing at the tip, when the tip attained the position of regime B. Since this effect was the same as observed for the thermocouples, there was very little probability that the regimes were artifacts produced by the measurement techniques.

At five psia the distance from the surface to regime A was about three bead diameters (200μ) and the distance between regimes A and B was about the same. These distances were verified with experimental data gathered with thermocouples whose diameter was one half of those used to establish Figs. 2 and 3. At higher pressures these distances became smaller than the bead diameter and interpretation of the data was difficult. The thickness of the regimes was not determined, but dips in the thermocouple bead temperature between the regimes implies that the regimes were separated by nonreactive or very slow reacting regions.

The preceding discussion is intended to show the consistency of Model II and the measured data, and to provide confidence in the following analysis which predicts the burning rate as a function of initial temperature.

For the isobaric case in which the initial temperature of the propellant is raised, Model II implies that the heat conducted into the solid from the gas can be expressed as

$$Q_T = Q_S + Q_R \tag{5}$$

where Q_T is total heat conducted into the solid, Q_S is the heat conducted in the solid from the regimes of gas reactions which have attained the solid-gas interface, and Q_R is the heat conducted from the first gas reaction regime R which is away from the interface. As in the previous approximation,

$$Q_R = kA(T_R - T_S)/d \tag{6}$$

Since the changes in T_R and T_S are small at a given pressure, the time for the gas to react may be assumed to remain approximately constant. Then the position of the gas reaction with respect to the interface is given by

$$d = t_R V_g \tag{7}$$

Since the velocity of the gas is related to the burning rate, Eq. (7) may be written as

$$d = W \cdot t_R \cdot V \tag{8}$$

The heat-transfer rate Q_R at a given initial temperature of the propellant may be related to that at some other initial temperature Q_{R1} under a set of otherwise identical conditions as

$$Q_R = (V_1/V)Q_{R1} \tag{9}$$

Equation (9) is a consequence of the fact that only d is a function of initial temperature in a pressure regime where there are no "jumps" in the burning rate. Equation (5) may be written using Eq. (9) as

$$Q_T = Q_S + (V_1/V)Q_{R1} \tag{10}$$

When the burning rate V_1 is known at a given initial propellant temperature and ambient pressure, Q_{R1} can be found from the temperature gradient predicted by the solution of Eqs. (2) and (3). Q_S is the value of Q_T when the reaction immediately preceding regime R first reaches the solid-gas interface. The burning rate at some other initial temperature of the propellant can be found by solving Eqs. (2) and (3) until it predicts the heat-transfer rate into the solid given by Eq. (10).

When this approximation is used, the burning rate as a function of initial temperature is produced. The comparison of the computer isobaric curves with data reported by Suh and Clary[8] is shown in Fig. 5. Logically, as the burning rate increased a condition would be achieved at which the velocity of the gas leaving the solid equals the velocity of propagation of the regime at the surface. At and after this condition these approximations would not apply. Whereas they are too simple to predict when the regime separates from the interface, they do provide a technique by which the increase in burning rate with an increase in initial temperature can be computed. Also, these results show that Model II is consistent with the measured data.

Since the surface temperature of the solid could be measured by a thermocouple restrained to the surface only if the regimes of gas reactions were at least several bead diameters from the surface, the surface temperature was measured with this technique only at 1 psia. The value of surface temperature determined at this condition was 300°C ± 25°C. This value compares favorably with the computed value of 275°C, a value of 290 ± 50°C as determined optically, and a value of 315°C as determined with embedded thermocouples by Suh et al.[15]

Conclusions

At low pressures and constant initial temperature there are "jumps" in the burning rate of a double-base solid propellant. Between the "jumps" the burning rate is described by the following equation:

$$V = V_0 + V_E \log(P) \tag{11}$$

The gas near the deflagrating solid contains a series of distinct regimes of gas reactions whose movement with respect to the solid-gas interface is described by a series of relations with the following form:

$$t = t_0 - t_E \log(P) \tag{12}$$

The "jumps" in the burning rate are a consequence of these distinct gas reactions reaching the gas-solid interface. The theoretical model presented in this paper (Model II) is consistent with the experimental results.

Appendix A

Physical Properties of Propellant

I. Double-base propellant, product of Hercules Powder Company
Type—Hivel No. 2 (Trade Mark), M-2
Solid density: 1.64 g/cc
Thermal conductivity: 5.5×10^{-4} cal/cm sec°K

Specific heat: 0.37 cal/g
Ratio of specific heats: 1.191
Moles gas/gram: 0.03876
Flame Temp: 3017°K (at constant volume)
2532°K (at constant pressure)
Molecular weight of Gas: 25.8 g/mole
Heat of explosion: 1060 cal/g
Composition (wt %)

Nitrocellulose	76.65	
Nitroglycerine	19.90	
Graphite	0.30	
Diphenylamine	0.65	
Potassium Nitrate		1.00
Barium Nitrate		1.50
		100.00

Reactions in the solid

Heat Released—100 cal/cc
Order—0
Frequency Factor 10^{17} sec^{-1}
Activation Energy 40,000 cal/mole—°K

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