Measurement of Temperature Profiles through Solid-Propellant Flames Using Fine Thermocouples

A. J. SABADELL,* J. WENOGRAD,† AND M. SUMMERFIELD‡
Princeton University, Princeton, N. J.

Temperature profiles in the solid preheat zone and gaseous combustion zone of burning solid propellants have been obtained by recording voltage from fine thermocouples embedded in burning solid-propellant strands. These temperature records were used to estimate the surface temperature of the burning solid. Techniques have been developed for routine fabrication and handling of fine (0.0003 in.) noble metal thermocouples. Surface temperatures were estimated by noting departure from pure conductive heat flow in a solid continuum. Surface temperatures in the range 550°–650°C at pressures between 1 and 15 atm have been observed for PBAA-AP composite propellants. A double base propellant was found to burn with a surface temperature of about 300°C. Variations of pressure and particle size and introduction of catalysts failed to give observable changes in surface temperature. These observations demonstrate important differences in combustion mechanism between composite and double base propellants.

I. Introduction

THE mechanism of flame propagation in composite propellants is quite complex and involves a number of stages. The gross features of the combustion process, as revealed by the effects of pressure and particle size on burning velocity, are rather well understood. However, the microscopic details of the combustion mechanism and the means by which the burning-rate control factors exert their influence have not been fully revealed. This experimental study was undertaken as part of a program designed to develop a more complete phenomenological description to the combustion process. This paper describes an investigation of microscopic temperature profiles through propagating flames in composite and double base propellants and the use of these profiles for the study of combustion mechanism and the estimation of surface temperatures. These profiles have been obtained by the use of fine thermocouples.

A number of theoretical models for the propagation of solid-propellant flames have been proposed which have been reviewed elsewhere.[1,2] A schematic representation of such a one-dimensional burning process is shown in Fig. 1. Below the surface at $x = -\infty$ the propellant is at T_0. As it approaches the flame, it is heated conductively to the surface temperature T_s at $x = 0$. At about this point, the solid components volatilize with some heat of reaction. These volatile products then react in a gas-phase flame giving a high temperature that supplies heat by conduction to control the surface gasification.

The one-dimensional flow of heat in a medium with heat generation $\dot{q}_x$ is given by

$$\frac{\partial}{\partial x}\left(-\lambda\frac{\partial T}{\partial x}+\dot{m}C_p(T-T_0)\right)=\dot{q}_x \tag{1}$$

If specific heat C_p and thermal conductivity λ are assumed constant, this may be integrated to give

$$\frac{d\ln(T-T_0)}{dx}=\frac{\dot{m}C_p}{\lambda}-\frac{1}{(T-T_0)\lambda}\int_{-\infty}^{x}\dot{q}_x dx \tag{2}$$

In the model just described, there is no heat generation until just below or at the surface. In the nonreactive region of the solid,

$$\frac{d\ln(T-T_0)}{dx}=\frac{\dot{m}C_p}{\lambda} \tag{3}$$

For such a medium, a plot of $\ln(T-T_0)$ against x is a straight line with a slope of $\dot{m}C_p/\lambda$. Deviations from linearity could represent the onset of energetic chemical reactions or the existence of a surface. When $\dot{q}_x$ is not zero below the surface, Eq. (2) shows that plots of $\ln(T-T_0)$ vs x are no longer linear. If heat is released, as may be the case in the foam zone of double base propellants, the integral in Eq. (2) is always positive, and the slope of $\ln(T-T_0)$ against x decreases as the surface is approached. If the reactions were endothermic, the opposite situation would prevail.

The energy balance at the surface of the propellant may be written

$$\lambda_g\left(\frac{dT}{dx}\right)_g=\lambda_s\left(\frac{dT}{dx}\right)_s-\dot{m}Q \tag{4}$$

Since $\lambda_g < \lambda_s$ by a factor of about 4, a discontinuity in the temperature gradient through a solid-propellant flame would be expected except in the rather accidental case where $\dot{m}Q = (dT/dx)(\lambda_s - \lambda_g)$. In general, if $\dot{m}Q$ is relatively small,

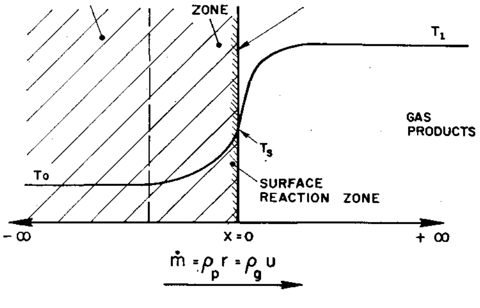

Fig. 1 Model of one-dimensional steady-state burning.

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* Assistant in Research; now Chief, Propellants Division, Instituto Investigaciones Cientificias y Tecnicas FFAA, Buenos Aires, Argentina. Member AIAA.

† Research Engineer, Guggenheim Laboratories for the Aerospace Propulsion Sciences; now Senior Research Scientist, United Aircraft Corporation Research Laboratories, East Hartford, Conn.

‡ Professor of Jet Propulsion, Guggenheim Laboratories for the Aerospace Propulsion Sciences. Fellow Member AIAA.

Table 1 Propellant compositions

Propellant designation	Components, % wt PBAA	Epon 828	AP fine, 9μ	AP coarse unground	Copper chromite	Ferric oxide
PBAA 70F	25.7	4.3	70	...	...	...
PBAA 70F-1C	25.7	4.3	70	...	1	...
PBAA 70F-1FO	25.7	4.3	70	...	...	1
PBAA 75BM	21.4	3.6	22.5	52.5	...	...

there should be a sharp upward break in a solid-propellant temperature profile at the surface.

Temperature profiles of solid-propellant flames should be capable of yielding significant insights into the combustion mechanism. Moreover, if experiments reveal consistent sharp upward breaks in these profiles, it would be reasonable to assume that these represented the solid-propellant surface.

Several authors have reported experimental determinations of temperatures in various portions of the solid-propellant combustion wave. Klein et al.[3] used fine (12.5-μ wire diameter) thermocouples to record the over-all profile through flames in a nitrocellulose propellant. They found smooth profiles without sharp breaks. They interpreted deviations from the behavior indicated by Eq. (3) as an indication of the surface temperature. In this fashion they were able to measure surface temperatures of the order of 250°C. Similar techniques were applied by Heller and Gordon,[4] who were particularly interested in temperature profiles extending into the gaseous zone. They attempted to measure the surface temperature by pressing a rather large thermocouple (75-μ diam) against a burning surface. The surface temperature obtained in this manner was in the range 400° to 500°C, but the size of the thermocouple with respect to the thermal gradient of the field renders this result suspect. A radiometric surface temperature measurement reported by Powling and Smith[5] corroborates the low value inferred by Klein et al.[3] Other estimates of surface temperature for double base propellants have ranged as high as 1000°C at 1500 psi.[6]

Temperature profiles in burning ammonium perchlorate composite propellants have been reported by Sutherland et al.[7] and Friedman et al.,[8] but the results have been inconclusive in the present context. Powling and Smith[5] have reported infrared radiometric surface temperature measurements for mixtures of ammonium perchlorate powder with small amounts of readily vaporized fuels and for composite propellants burning at atmospheric and subatmospheric pressures. For polyisobutylene propellants burning at atmospheric pressure the reported surface temperatures were in the range 500°–550°C. By studying the dependence of T_s on pressure for ammonium perchlorate burning at subatmospheric pressures, they inferred that the burning mechanism involves, as one of the steps, the gasification of ammonium perchlorate by the reaction

$$NH_4ClO_4(s) \rightleftharpoons NH_3(g) + HClO_4(g) \qquad (5)$$

which is endothermic by 56 kcal/mole.

The existence of a unique surface temperature for a burning propellant is by no means obvious. The surface structure, especially in the case of composite propellants, is heterogeneous and rough, and it is likely that the surface is characterized by a range of temperatures. The technique used by Powling and Smith measures an average temperature over a large surface area, whereas microthermocouples measure a discrete temperature over an area corresponding to the area of an oxidizer particle. Nevertheless, it is possible that opposite sides of such thermocouples would sense different temperatures depending upon which component of the propellant contacted them. Thus, a certain amount of scatter in surface temperature values would be expected with thermocouples in composite propellant.

II. Experimental

Fine uncoated thermocouples were embedded in strands of composite and double base propellant. The thermocouples were fabricated from Wollaston wires of platinum and platinum-rhodium 10% 7.5 μ in diameter. In some experiments, 2.5-μ wires were used. The wires were mounted in micromanipulators, and the ends butted at an acute angle under a 30-power stereomicroscope. The junctions were then welded with an acetylene-oxygen microtorch. The junctions had a diameter less than twice the wire diameter.

The incorporation of the thermocouples in strands of double base propellant was accomplished by placing them between two 3-mm thick by 6-mm wide sheets of a double base propellant obtained through the courtesy of Picatinny Arsenal. The two sheets were glued together with an acetone solution of the same propellant. The strands so obtained were allowed to dry at 40°C for one week before use. This technique is similar to those used by other investigators.[3]

For composite propellants, gluing did not give a strong bond representative of the bulk of the propellant. As a consequence, it was decided to develop a technique for embedding the thermocouples directly into uncured strands of propellant. A propellant with low viscosity and long curing time was selected. A propellant composition, based on polybutadiene acrylic acid fuel and containing 70% of fine unimodal ammonium perchlorate, was found to fulfill these requirements. The oxidizer particle size was characterized by a sedimentation technique described elsewhere,[9] and the mean diameter was found to be 9 μ. The same composition, with small amounts of catalysts, was also studied as was a propellant with the same fuel and a bimodal oxidizer. Table 1 gives the composition of the propellants, and Table 2 shows their burning rates at pressures of interest.

Temperature profiles are recorded as the strand with the embedded thermocouple is burned in cigarette fashion under nitrogen pressure in a strand burner described previously.[9] Propellants were ignited using Nichrome wires. The burning rates shown in Table 2 were determined in the same apparatus. The recording instrumentation consisted of a dual beam oscilloscope and an oscillographic record camera. A schematic diagram of the circuit used is shown in Fig. 2.

For data reduction, prints of the interesting portions of the runs were made from the 35-mm film. Figure 3a shows a tracing of one such film record with marks indicating the method of data reduction. Millivolt readings were made using a Gerber variable scale and were converted into temperatures using standard tables in National Bureau of Standards Circular 561. Slight temperature rises are difficult to read with accuracy, and the lower portions of the semilog plots below are doubtful.

Two criteria for the location of a surface are inherent in Eqs. (3) and (4). To find a more direct experimental technique for detection of the passage of the thermocouple through the surface, an attempt was made to employ the ionization of the flame front. Thus, in several runs, the Langmuir ionization probe circuit illustrated in Fig. 2 was hooked up to one trace of the dual beam oscilloscope. It was hoped that this second signal would record a sharp jump at some time during the temperature profile signifying passage of the thermocouple through the surface. However, the

Table 2 Average values of T_s and λ determined from temperature profiles

Propellant designation	p, psig	r, mm/sec	No. of runs	T_s, range of values	T_s av., °C	λ, range of values	λ av., cal 10^{-4}-°C-cm-sec
PBAA 70F	15	3.1	4	550–650	615	3.7–6.7	5.3
	30	3.3	15	550–670	612	3.7–13.2	7.1
	50	4.0	3	640–700	660	5.0–7.1	6.1
	100	4.8	12	490–650	583	5.4–13.7	8.4
	150	5.3	12	540–700	603	7.1–12.3	10.3
PBAA 70F-1C	15	4.4	2	600	600	10.7–11.0	10.9
	100	9.2	2	600–630	615	15.4–21.9	18.6
	200	12.3	3	530	530	18.4–19.9	19.1
	1000	22.1	5	510–550	532	25.4–39.0	31.0
PBAA 70F-1FO	15	3.8	2	550–560	555	5.2–16.0	10.6
	100	7.9	3	660–700	667	8.0–24.0	15.3
	200	10.9	2	460–560	510	17.0–21.0	19.0
PBAA 75BM	15	1.7	4	500–600	538	4.8–6.6	5.5
	30	2.0	4	450–600	540	4.5–13.7	8.9
Double base	50	2.7	8	250–350	332	4.2–10.9	7.2
	100	6.7	3	300–350	333	5.1–9.6	7.8
	150	8.4	4	250–280	268	6.4–12.8	10.7

onset of this second signal was not consistent, and, on occasion, spurious behavior was noted. In general, however, the onset of ionization occurred slightly before the main portion of the temperature rise. This behavior may have been due to microscopic porosity of the propellant.

III. Results and Discussion

Experimentally determined voltage time traces are illustrated in Fig. 3 for composite and double base propellants. Plots of log($T - T_0$) vs distance for composite and double base propellants under varying conditions are illustrated in Fig. 4.

Surface temperature in the combustion of composite and double base propellants have been determined on the basis of a large number of such log($T - T_0$) vs distance plots. In both cases the criterion for the positioning of the surface was the first significant deviation from the linear behavior predicted in Eq. (3). In the case of double base propellants, this first deviation from linearity generally occurs as a downward curvature and could also be interpreted as an onset of exothermic reaction. Moreover, there never appears to be a sharp upward break in the temperature profile which would be characteristic of an interface between a region of high thermal conductivity and one of low thermal conductivity. The temperature profiles are quite smooth, reflecting the microscopically homogeneous nature of the propellant and also, perhaps, the distributed nature of the energetic chemical reactions.

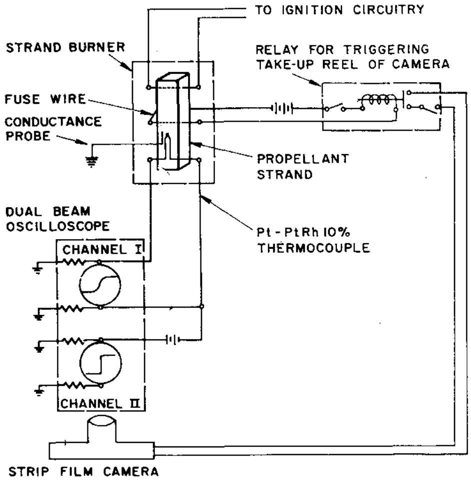

Fig. 2 Instrumental setup for thermocouple traverses; channel I measures thermocouple signal, and channel II measures onset of ionization.

In the case of composite propellants, sharp upward breaks are noted in the log($T - T_0$)-distance curves around 600°C which are associated with a significant deviation from linearity. Such breaks are illustrated in Figs. 4b, 4c, and 4d. These sharp upward breaks are preceded by an undulating, but approximately linear region. Both the deviation from linearity [Eq. (3)] and the upward break [Eq. (4)] justify the assignment of this point as the surface. It is most probable that these undulations occur as a result of the heterogeneous nature of these propellants and the differing thermal conductivities of the two components of the solid. If these points of departure from linearity indeed represent the surface temperatures of the propellants under consideration, the resulting surface temperatures obtained for a number of propellants under varying conditions are summarized in Table 2. The slopes of the linear portions of the log($T - T_0$) vs distance curves may be used to ascertain λ by Eq. (3) since $\dot{m}$ and C_p are known. The resulting values of λ are also summarized in Table 2. The values of λ_s show considerable scatter under a given set of conditions. Nonetheless, the values obtained for composite propellants at low burning rates show fairly good agreement with the value reported by Medford[10] of 9.8 × 10^{-4} cal/°C-cm-sec.

For the composite propellant designated PBAA-70F, with which the bulk of this research was performed, a surface temperature of about 600° was measured consistently. Within the sensitivity of the method, no variation of surface temperature with pressure was detected as can be seen by the average values for each pressure shown in Table 2. In some experiments, temperature profiles were recorded using thermocouples made with 2.5-μ diam wire. In this case, the subsurface rippling of the voltage time trace was much more accentuated. The length of these ripples is approximately equal to the average oxidizer particle size (Fig. 3c). A bimodal propellant composition PBAA-75BM was tested at 15 and 30 psig (Fig. 3d). The average surface temperature for this composition was 540°C, and no variation of this temperature with pressure was observed. The length of the ripples in this case are longer than in the case of the unimodal fine oxidizer composition tested.

The temperature profiles in the gas phase beyond the surface are also of interest. There is a steep initial rise with some fluctuations after which the temperature attains a constant value. This value is generally maintained until the thermocouple breaks. The initial rise occurs in 50–100 μ from the surface. The temperature fluctuations in the gas phase could be attributed to motion of the thermocouple junction in the steep temperature gradient. This effect could also be attributed to a two-dimensional flame structure in which the thermocouple might sense fuel and oxidizer-rich regions at different temperatures.

A few remarks should be made about the application of the thermocouple technique. They operate in fields with extremely steep temperature gradients. Thus, near the surface, the temperature difference may be in excess of 100° across the thermocouple. These steep gradients introduce questions of thermal lags which are not fully answered in this discussion. The magnitude of the lags can be illustrated by the very large values of the measured lambdas for the fast-burning catalyzed propellants shown in Table 2. This would be expected if the actual gradients were steeper than those measured. As burning rate decreases, the lambdas seem to level off at a value of about 6×10^{-4} cal/°C-cm-sec for burning rates below 4 mm/sec. The surface temperatures would be affected less strongly since they are given by the intersection of two curves.

Previous investigators[3,11] have noted significant temperature errors (as high as 400°C) when bare platinum metal thermocouples were used to measure the temperature of reactive gas mixtures containing such ingredients as hydrogen, oxides of nitrogen, and carbon monoxide. These errors could be avoided by coating the thermocouples with thin layers of such materials as silica, borax, or certain ceramics. Tests run during the course of this program using thermocouples coated with silica deposited by a flame fed with a fine spray of hexamethyl disiloxane showed no significant differences. The absence of temperature differences attributable to catalytic effects in these dynamic experiments could be explained on the basis of the very short contact times involved.

It is interesting to consider the magnitude of the temperature gradients across the points that are presumed to represent the surface for composite and double base propellants. For double base propellants, since the passage of the surface is approximately continuous, the gradient would be the same in the solid and in the gas at the tangential point. A value for this gradient is approximately 6°/μ. For composite propellants, where the gradient is discontinuous, there are two values. In the solid phase, the gradient at the surface runs approximately 15°/μ, whereas the value in the gas phase is at least twice this value, say 30°/μ.

In view of these experimental temperature profiles, some reassessment of the simplified phenomenological picture of solid-propellant flames just described may be necessary. For composite propellants, the existence of ripples in the profiles below the ostensible propellant surface, although they are explicable on the basis of the propellant heterogeneity, might also be suggestive of distributed exothermic reactions. However, the over-all linearity of the subsurface semilog plots in the region of the major temperature rise argues against energetically important subsurface reactions at great depth.

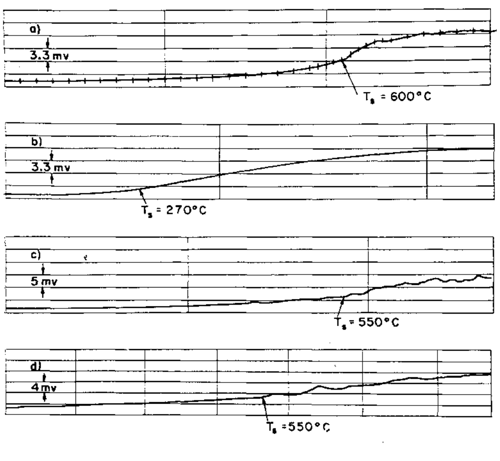

Fig. 3 Voltage time records of $Pt + PtRh$ 10% thermocouple traverses of solid-propellant flame zones (horizontal scale, 1 division = 16.67 msec): a) PBAA-70F propellant, 30 psig, 7.5-μ thermocouple (T.C.) wire; b) double base propellant, 150 psig, 7.5-μ T.C. wire; c) PBAA-70F propellant, 30 psig, 2.5-μ T.C. wire; and d) PBAA-75BM propellant, 30 psig, 7.5-μ T.C. wire.

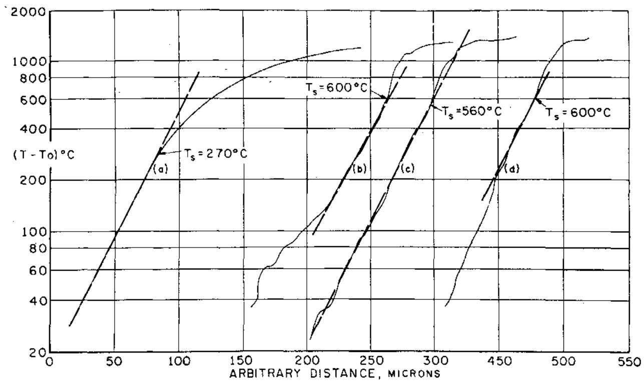

Fig. 4 Log(T-T_0) vs distance, testing exponential character of conduction zone: a) double base propellant, 150 psig, 7.5-μ T.C. wire; b) PBAA-70F propellant, 30 psig, 7.5-μ T.C. wire; c) PBAA-70F propellant, 100 psig, 7.5-μ T.C. wire; and d) PBAA-70F propellant, 150 psig, 7.5-μ T.C. wire.

The foregoing experimental temperature gradients permit some order of magnitude estimates of the quantities comprising the surface energy balance, Eq. (4). Because of lag effects, the preceding measured gradients should be regarded as lower limits. Taking the value of 30°C/μ for the gas-phase gradient, assuming a thermal conductivity of 2×10^{-4} cal/cm-sec-°C for the gases and a burning rate of 0.4 cm/sec, the heat feedback to the surface from the gas phase, $\lambda_g(dT/dx)_g$ amounts to 60 cal/cm²-sec or, with a propellant density of 1.6 g/cm³, 100 cal/g of propellant vaporized. The heat removed from the surface by conduction into the solid, $\lambda_s(dT/dx)_s$ is given by the experimental gradient, 15°C/μ times the experimental thermal conductivity 10×10^{-4} and amounts to 150 cal/cm²-sec or 230 cal/g. The difference between these two quantities, 130 cal/g, must be generated at the surface or in some surface reaction region.

A heat production term of 100 cal/g of propellant at the surface was suggested earlier[7] as a combined result of the complete exothermic decomposition of ammonium perchlorate into oxidizer and other gases plus the endothermic decomposition of fuel polymers into low-molecular-weight hydrocarbon vapors. The present experimental results tend to confirm this interfacial heat release term, and, thus, they suggest that these decomposition processes occur very close to the surface, and that the main heat generation process in the flame is the result of the subsequent oxidation reaction between the component vapor streams. The monopropellant combustion character of the ammonium perchlorate is important but does not appear to manifest itself within this distance scale as a discrete flame. Further studies of this type, using the finest possible thermocouples, might help in the establishment of these points. Finally, in the double base propellant studies there is no steep gradient in the region of interest which would be characteristic of an intense heat feedback from a flame.

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Ignition of an Evaporating Fuel in a Hot, Stagnant Gas Containing an Oxidizer

C. E. HERMANCE,[*] R. SHINNAR,[†] AND M. SUMMERFIELD[‡]
Princeton University, Princeton, N. J.

Experimental evidence indicates that the ignition of a solid propellant by conventional agents occurs in the gaseous reaction zone adjacent to the propellant surface and that, in such heterogeneous systems, the gas composition, pressure, and temperature strongly influence the ignition delay. This paper, in contrast to extensive past study of ignition in homogeneous mixtures, describes some of the key theoretical properties of ignition in heterogeneous systems through similarity theory and numerical analysis of the mathematical model. Certain common simplifying assumptions and ignition criteria, valid for homogeneous systems, were found to be invalid in heterogeneous systems. In heterogeneous systems with igniter gas temperatures near E/R ,the particular choice of ignition criterion affects the formula connecting the ignition delay and the chemical or physical parameters of the gas phase. Furthermore, a log-log plot of ignition delay vs concentration of gaseous oxidizer exhibits a slope that varies over a wide range of negative values; this slope is not a simple multiple of the controlling reaction order. These theoretical results require verification by critically designed experiments.

Nomenclature

A = dimensionless constant, characteristic of relative fuel and oxidizer abundance, $(ny_F{}^0/y_{\text{ox}}{}^\infty)$ in case 1 and $\dot{m}_F{}^0/[\rho^3(y_{\text{ox}}{}^\infty)^3ZD]^{1/2}$ in case 2
B = dimensionless constant characteristic of heat release capability of fuel, $(qR/c_PE)y_F{}^0$ in case 1 and $(qR/c_PE)(\dot{m}_F{}^0/[\rho^3y_{\text{ox}}{}^\infty ZD]^{1/2}$ in case 2
C_F = fuel vapor concentration, g/cm³
C_{ox} = oxidizer concentration, g/cm³
C_P = gas specific heat, cal/g-°K
D = mass diffusion coefficient, cm²/sec
E = activation energy of chemical reaction, cal/mole
$[F]$ = fuel
$L^*(\theta)$ = specification of ignition criterion
m = stoichiometric coefficient of combustion products
$\dot{m}_F{}^0$ = constant fuel mass flux in case 2
n = stoichiometric coefficient of oxidizer
[ox] = oxidizer
$[P]$ = products
q = heat of combustion based upon the fuel, cal/g
R = universal gas constant, cal/mole-°K
T = temperature, °K
t = time, sec
v = velocity, cm/sec
y = mole fraction
Z = reaction rate pre-exponential, cm³/g-sec
α = dimensionless constant used to characterize the ignition criterion, or thermal diffusivity, cm²/sec
η = dimensionless concentration
θ = dimensionless temperature
ξ = dimensionless length
ρ = gas density, g/cm³
τ = dimensionless time
ϕ = dimensionless variable equal to $(\theta + BN_F)$ in case 1

Superscripts

0 = $x = \xi = 0$ conditions at the wall
∞ = conditions at infinity
$*$ = denotes value of variable at ignition

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[*] Research Associate, Department of Aerospace and Mechanical Sciences, Guggenheim Laboratories for the Aerospace Propulsion Sciences. Member AIAA.

[†] Visiting Research Scientist; also Professor of Chemical Engineering, City University of New York, New York, N. Y. Member AIAA.

[‡] Professor of Jet Propulsion, Department of Aerospace and Mechanical Sciences, Guggenheim Laboratories for the Aerospace Propulsion Sciences. Fellow Member AIAA.