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Effect of Catalyst Addition on Subatmospheric Burning Surface Temperature of Composite Propellants

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The subatmospheric burning with its higher catalytic effectiveness, lower temperature gradient, and slower combustion wave offers a fitting environment to study the effect of catalysts on the burning surface temperature of composite propellants. Using platinum and platinum-13% rhodium 7.5-μm thermocouples in uncatalyzed as well as copper–chromite-catalyzed ammonium perchlorate/hydroxyl-terminated polybutadiene composite propellants, the subatmospheric-burning surface temperatures were measured. The results of the present experimental study are in close agreement with the established trend; the surface temperature increases with the increase in pressure. Some experimental studies of others failed to give an observable change in surface temperature with pressure. This is argued to be because of the dimensional inadequacy of detectors in the very high-temperature-gradient environment. The measured surface temperature of the catalyzed propellant is significantly higher than that of the uncatalyzed one. The study shows that the increased surface and subsurface heat release caused by catalyst addition causes this temperature enhancement.

Nomenclature

c = specific heat, J/(kg K)
p = pressure, bar
Q_l = heat loss, J/kg
Q_s = heat release, J/kg
r = steady burning rate, m/s
T = temperature, K
T_{amb} = ambient temperature, K
T_s = burning surface temperature, K
t = time, s
x = length or distance, m
α = thermal diffusivity, W/(m^2 K)
λ = thermal conductivity, W/(m K)
ρ = density, kg/m^3

Subscripts
amb = ambient
g = gas phase
p = propellant
s = surface or solid phase
tra = transition

Introduction

THE present study forms a part of our continuing effort to better comprehend the mechanism of burning-rate enhancement in catalyzed composite solid propellants. The accurate calculation of burning-surface temperature under different operating conditions is vital in the combustion modeling[1–5] and, hence, the understanding of the effect of catalyst addition on this temperature is important. The present study examines this effect by adding a catalyst, copper–chromite catalyst (CC), to an ammonium perchlorate (AP)/hydroxyl-terminated polybutadiene (HTPB) composite propellant. The study is conducted under subatmospheric pressures for the following two reasons. Firstly, for such a propellant the burning-rate enhancement caused by catalyst addition is significantly higher at subatmospheric pressures than at rocket operating pressures.[6,7] Therefore, the catalyst effect, if any, on the surface temperature will be more significant at subatmospheric pressures. Secondly, at such low-pressure conditions, with a larger flame standoff distance—less severe temperature gradient and thicker thermal wave—and a lower regression rate, the combustion offers better spatial and temporal resolutions for temperature measurements.

It is appropriate at this juncture to review the various studies conducted on the burning surface temperature of composite propellants. The experimental methods, adopted to determine this temperature, include 1) the technique using infrared emission from the burning surface,[8–11] 2) the backcalculation from the thickness over which the AP crystal lattice changes from orthorhombic into cubic,[12] and 3) the fine-thermocouple technique.[13–17]

Powling and Smith,[8–10] using the infrared emission technique, measured from subatmospheric pressures (≥0.033 bar) to higher atmospheric pressures (≤20.40 bar) the surface temperatures of 1) fuel-lean as well as fuel-rich mixtures of AP and some readily gasified fuels such as paraformaldehyde (from subatmospheric to above-atmospheric pressure ranges),[8–10] 2) a fuel rich mixture of AP/polystyrene and fuel-rich propellants of AP/polyisobutene (subatmospheric to atmospheric pressure ranges),[8,10] and 3) fuel-rich propellants of AP/polyurethane (PU) (at atmospheric pressure only).[8] The burning rates of these compositions were widely varied more than fivefold by the changes in pressure, AP particle size, and fuel/oxidizer ratio, and also by the addition of some catalysts such as copper chromate. For these compositions, the surface temperature of 495 ± 15°C at atmospheric pressure and its increase with increase in pressure were reported (370°C at 0.033 bar and 650°C at 20.40 bar). At subatmospheric and atmospheric pressures, the surface temperature of AP/paraformaldehyde mixtures had little dependence on burning rate. However, the temperature of the porous plugs of AP burning in a flow of gaseous fuels (methane, hydrogen, or ammonia) showed a significant increase with an increase in burning rates.[11] Collating his results with those obtained by many others under a wide variety of conditions and comparing the two plots, $\rho_p r$ vs $1/T_s$ against p vs $1/T_s$, Powling[11] concluded that a kinetic-decomposition law generally better represented the dissociation at the burning surface than an equilibrium one.

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Using the second experimental method for the surface temperature measurement in AP/PU thin propellant slices, Selzer[12] adopted the change of AP crystal lattice from orthorhombic into cubic as an indicator of temperature penetration into the solid. The calculated surface temperatures showed a wide scatter of 330–910°C at the atmospheric pressure. However, he noted a trend of increase in surface temperature with pressure (1–180 bar).

Sabadell et al.[13] were the first to use the fine thermocouple technique to measure the surface temperature of an AP/polybutadiene acrylic acid composite propellant. Embedding platinum and platinum-10% rhodium (Pt and Pt-10% Rh) 7.5- or 2.5-μm thermocouples into the propellants at the uncured stage, Sabadell et al.[13] obtained the temperature records at different pressures from 2 to 70 bar. From these records, they estimated the burning-surface temperature to be in the range of 550–650°C. The variations of pressure and AP particle size as well as the addition of a catalyst (1% CC or 1% ferric oxide) failed to give any observable change in the surface temperature. By embedding Pt and Pt-10% Rh 12.5-μm thermocouples into the oxidizer-rich AP/(HTPB + isophorone diisocyanate) pressed propellant pellets, Yano et al.[14] estimated the surface temperature to be around 330°C in a subatmospheric pressure range of 0.33–0.87 bar; and found it to be relatively independent of pressure in the test range. Using 12.5-μm thermocouples, Miyata and Kubota[15] estimated the burning surface temperature of an AP/HTPB propellant to be about 430°C in the range of 3–15 bar. They found that the addition of $SrCO_3$, a burning rate inhibitor, increased the surface temperature to 700°C. Using Pt and Pt-10% Rh 2.5-μm thermocouples, Kuwahara and Kubota[16] measured the burning surface temperatures of an AP/HTPB (16%) propellant and an AP/hydroxyl-terminated polyethylene (HTPE) (16%) propellant to be equal at 500°C at 0.5 atm. The propellants' adiabatic flame temperatures were found to be essentially the same (2597 and 2595 K, respectively). Furthermore, for the former propellant, the gas-phase temperature gradient was less, but the burning rate was faster. With these values, the overall heat balance at the burning surface gave higher surface heat release for AP/HTPB propellant. Hence, they concluded that HTPB acted more energetically than HTPE at the burning surface. Using Pt and Pt-10% Rh (2.5- to 10-μm) thermocouples, Zanotti et al.[17] estimated the surface temperature of an AP/HTPB propellant to be in the range of 430–680°C for the variation from 0.1–41 bar. They found the surface temperature to increase with the increase in pressure.

It is also pertinent to discuss the shortcomings of the temperature measurement techniques detailed previously. In the infrared emission technique the measurements are carried out by choosing a suitable wavelength for which the reflectivity of the burning surface is low, but its absorption coefficient is high. Also, the technique requires a special provision to draw the flame gases away from the burning surface to reduce the radiation from the hot gaseous envelope. However, at a high pressure ($\geq$15 bar), 1) the increase of gas emission masking the radiation emission from the surface, 2) the decrease of the effective emissivity of the surface because of the increased temperature gradient within the surface, 3) the difficulty in sucking away the flame gases without disturbing the effective combustion environment, and 4) the uncertainty in the estimation of a possible contribution of chemiluminescence from the gaseous reaction zone, all affect the measured value of the surface temperature. Selzer,[12] in his phase-change-layer thickness technique, could see AP crystal only when the crystal size was above 48 μm. Below this size, he could observe only some broad zones. Moreover, as Selzer[12] increased the pressure, he observed a black char covering the surface. He had difficulty interpreting his results and could obtain the average burning surface temperature only with a wide scatter. The fine-thermocouple technique has two flaws: firstly, the thermocouple embedded in the propellant surface may sense different temperatures for fuel and oxidizer; and secondly, the thermal-lag and the lead-losses exist at high pressures because of steep temperature gradient and these losses affect the measured temperature. However, the thermocouple technique is relatively more simple and in the subatmospheric combustion, as discussed previously, it offers relatively accurate temperature measurements.

Furthermore, the type of curative used in a propellant-binder may have an effect on the burning-surface temperature of a composite propellant. It is known that the decomposition rate of a polymeric-binder used can be changed by a factor of 2 to 3 by changing the curative of the binder. For example, the isophorone diisocyanate (IPDI) curative can give a significant enhancement to the rate when compared to the dimeryl diisocyanate (DDI) in HTPB. Also, differential scanning calorimetry studies have indicated that the IPDI-cured binder decomposes very exothermically compared to DDI in oxygenated atmospheres. These results indicate that the type of curative used in a propellant may also have an effect on burning-surface temperature. Unfortunately, the information about the curatives used in propellant binders is rarely found in literature.

Experimental Procedure

Table 1 gives the details of the two propellant formulations used for the present study. The formulations used AP with a purity of $\geq$98.6% and a weight mean diameter of ($\Sigma\, n_i d_i^4 / \Sigma\, n_i d_i^3$) = 107 μm, and CC of weight mean diameter = 15 μm and of a 33% Cu and 29% Cr composition. All of the propellants were processed using the procedure explained elsewhere.[18] The propellant strands used were 6 × 6 × 90 mm for the surface temperature measurement, and 6 × 6 × 180 mm for the burning rate measurement. The strands were inhibited at their sides with a solution of 10% polyvinyl alcohol to ensure uniform burning.

Uncoated reference-grade thermocouple wires of Pt and Pt-13% Rh, 7.5-μm diameters (Sigmund Cohn, New York) were welded together using a fine thermocouple welding technique. Approximately a 100-mm length of each wire was held on two separate micromanipulators. The micromanipulators were suitably positioned such that the ends of the wires butt with each other at an acute angle. The positioning was monitored under a low-power microscope. The butted ends were welded together using an acetylene–oxygen microtorch. The welded junction was examined under a stereo-zoom microscope. Only the thermocouples whose junction-bead diameters were less than or equal to two times the wire diameters were qualified for use. To insert the qualified thermocouple into the strand, a sharp slant cut of 3.5 mm length subtending an angle of 60 deg to the strand-surface was made (Fig. 1). By slightly bending the strand backward, a thin coat of an acetone solution of the uncured propellant was smeared on the exposed cut faces. With the thermocouple still on the micromanipulators but with its leads positioned as obtuse as possible ($\geq$120 deg), it was carefully inserted into the cut such that the bead occupied the cut end, i.e., close to the center of strand cross section. The strand-cut was then closed and held firmly for about 5 min. Thereafter, the thermocouple leads were relieved from the micromanipulators and the excess acetone solution if any was carefully wiped away. The strand-cut with the inserted thermocouple was allowed to cure for seven days at room tem-

Table 1 Propellant ingredients

Ingredients	Mass fractions[a]
AP—oxidizer	0.7300
HTPB—binder	0.2160
Di-2-ethylhexyl adipate—plasticizer	0.0405
Toluene di-isocyanate—curing agent	0.0135

[a] CC fraction in catalyzed propellant = 0.02; the balance fraction of 0.98 has the same ingredients' mass fractions as shown.

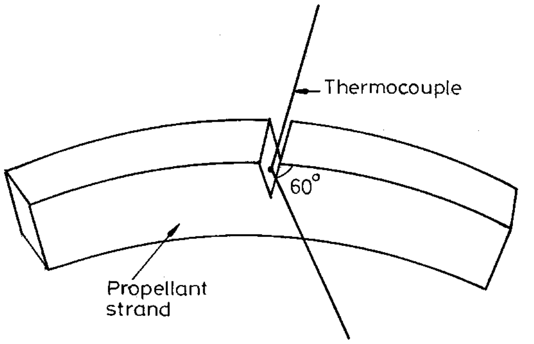

Fig. 1 Propellant sample with inserted thermocouple.

perature. The whole process of thermocouple welding and its successful insertion into the propellant was very delicate, and had to be learned with great care and patience; the present success rate is about 50%.

The experimental setup used for surface-temperature and burning-rate measurements consisted of a large surge tank and a bell jar connected to a vacuum pump. All of the propellant burning was carried out under nitrogen atmosphere and in each case the pressure was held constant by continuously evacuating the bell jar that had a small supply of fresh nitrogen. Ignition was achieved using a nichrome wire. In the surface-temperature measurement, as the flame front of burning propellant approaches and crosses the thermocouple, the corresponding millivolt signals were amplified and continuously recorded in a PC/AT at a rate of 200 samples/s. The recorded millivolts--time signals were converted into the temperature–time records by employing the temperature–millivolt curve generated using standard tables. Using the same setup, the burning rates of propellants were measured separately by employing a fuse-wire cutting technique explained elsewhere.[18] The measured burning rates were used to convert the temperature–time records into temperature–distance records.

At least two reasonably repeatable temperature profiles were obtained at every chosen pressure. Figure 2 shows a typical profile at 0.049 bar for a 2% CC-catalyzed sample. The temperature trace shows a rise until the burning surface of the propellant arrives at the thermocouple junction. Thereafter it exhibits a temperature break with a steep gradient to the spatially as well as temporally dependent flame temperature fluctuations caused by the burning heterogeneous-surface.

The slope of a condensed-phase temperature profile of an AP composite propellant significantly changes at about 513 K (T_{tra}) because at this temperature the primary propellant constituent, viz., AP changes its crystalline lattice from orthorhombic into cubic. Therefore, two distinct condensed-phase regions exist, as shown in Fig. 3: one before the crystalline transition temperature and the other after it. The thermal conductivity of a propellant above T_{tra} is essentially constant and it is found to be dependent on the propellant composition—a CC-catalyzed propellant is of higher thermal conductivity than the uncatalyzed one.[13] The specific heat increases with the increase in temperature and it depends also on the propellant composition. The propellant density depends on the propellant composition, but its variation with temperature is negligible. Notwithstanding all of these, steady-state, constant material properties, and no reaction in condensed-phase are assumed to describe the temperature profiles in the ① and ② regions (Fig. 3). Such assumptions are made only to facilitate the identification of surface temperature break, though the actual temperature profile may not fully coincide with the predicted one. Hence, the equation that describes the temperature profile in the regions ① and ② is

$$\rho_p r c_p \frac{dT}{dx} = \lambda_p \frac{d^2T}{dx^2} \tag{1}$$

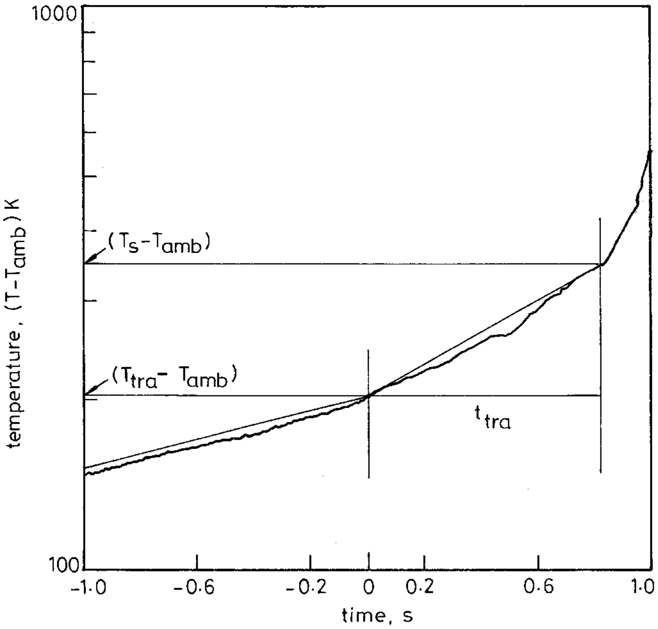

Fig. 2 Temperature profile of a CC-catalyzed propellant at a pressure of 0.049 bar.

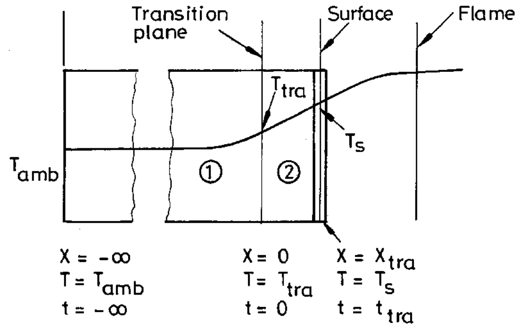

Fig. 3 Sketch of one-dimensional model used to estimate the solid phase temperature distribution and to locate surface temperature.

This equation has the following boundary conditions:

$$x \to -\infty, \qquad t \to -\infty, \qquad T = T_{amb}$$

$$x = 0, \qquad t = 0, \qquad T = T_{tra}$$

$$x = x_{tra}, \qquad t = t_{tra}, \qquad T = T_s$$

The solutions to Eq. (1) are, for region ①

$$(T - T_{amb}) = (T_{tra} - T_{amb})\exp[t/(\alpha/r^2)] \tag{2}$$

and for region ②

$$(T - T_{amb}) = (T_{tra} - T_{amb}) + [(T_s - T_{amb}) - (T_{tra} - T_{amb})] \times \{1 - \exp[t/(\alpha/r^2)]\}/\{1 - \exp[t_{tra}/(\alpha/r^2)]\} \tag{3}$$

For the surface-temperature estimation, the thermal conductivity and the specific heat were taken to be constant at 0.14 J/(ms K) and 1840 J/(kg K), respectively.[17] The densities of uncatalyzed and catalyzed propellants were 1490 and 1530 kg/m^3, respectively.

The surface temperature of a burning propellant at a test pressure is estimated according to the following steps. In the measured temperature–time record, Fig. 2 ($T_{tra} - T_{amb}$) is first located. Choosing a trial ($T_s - T_{amb}$), the time t_{tra} associated with the propellant thickness x_{tra} that is corresponding to the temperature rise from the known $(T_{tra} - T_{amb})$ to the chosen $(T_s - T_{amb})$ is first estimated. This trial $(T_s - T_{amb})$ is identified by the steep gradient in profile. The propellant thickness

$$x_{tra} = t_{tra} r \tag{4}$$

where r is the measured average burning rate of propellant at the test pressure. With the trial values of $(T_s - T_{amb})$ and t_{tra}, and the thermal diffusivity $[\alpha = \lambda_p/(\rho_p c_p)]$, $(T - T_{amb})$ is calculated against t [Eq.(3)] for a trial fitting. If the fitting is not satisfactory, the preceding steps are repeated until an acceptable fit is obtained and the resulting $(T_s - T_{amb})$ gives the best-fit surface temperature. Reference 17 gives detailed information on this temperature-estimation procedure.

Results and Discussion

Figure 4 depicts the burning-rate curves obtained for the uncatalyzed and CC-catalyzed propellant samples. The corresponding filled symbols on the x axis indicate the low-pressure deflagration limits (LPDLs). Figure 5 gives the plot of the measured surface temperatures at various pressures. The surface temperatures of the propellants increase with an increase in pressure. The important fact brought out by the present study is that the surface temperature of the CC-catalyzed propellant is significantly higher than that of the uncatalyzed one. The subsurface thickness values for high temperature, between $(T - T_{amb}) = 75$ K (arbitrarily chosen warm temperature) and $(T_s - T_{amb})$, show a decreasing trend with the increase of pressure (Fig. 6). However, this decrease is steeper above about 0.1 bar for the uncatalyzed propellant, indicating its thinner subsurface layer. Also, the average condensed-phase temperature slope $(\Delta T/\Delta x)_s$ between $(T - T_{amb}) = 75$ K and $(T_s - T_{amb})$ increases with the increase in pressure for both the propellant samples (Fig. 7).

Figure 8 shows the surface temperature values of the present study along with those of others, and the values are in close agreement with the established trend. Collating a large number of data from various studies, Powling[11] showed that the surface temperature of a burning composite propellant should increase with pressure. This pressure dependence has been employed in all the recent composite-propellant modeling efforts.[4,5] Powling,[11] however, cautioned that if the dimensions of temperature detectors (even the 2.5-μm diam thermocouple) were not suitable to the high-temperature gradients at intermediate and high burning pressures, the detected temperatures might not depict this pressure dependence.[11] Similar concern has also been expressed by Guirao and Williams.[19] Therefore, the failure to give observable changes in surface temperature with pressure by some experimental studies[13–15] may be attributed to the dimensional inadequacy of temperature detectors in the very high-thermal-gradient environment. For the present investigation, taking the thermocouple junction to be a sphere of diameter equal to two times the wire diameter, viz., 15 μm, and noting that the maximum burning rate at 1 bar is around 1 mm/s the time to reach 99% of the environmental temperature can be in the range of 1.5–4.5 ms.[20–22] Furthermore, in a supporting manner, the time that the burning surface requires to cross the thermocouple junction is much longer, around 15 ms, and the condensed-phase temperature gradient at 1 bar is about 1 K/μm. Hence, the temperature measurement at 1 bar is expected to be accurate and the conditions for accuracy only improve at further lower pressures.

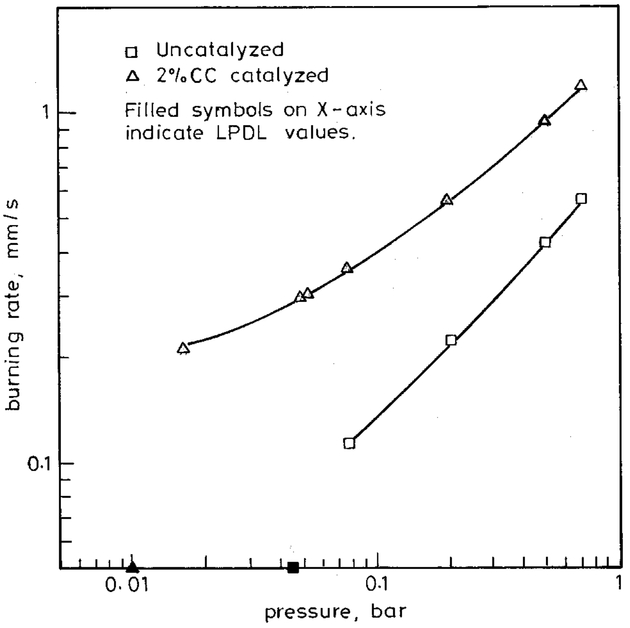

Fig. 4 Burning rates of AP/HTPB propellants.

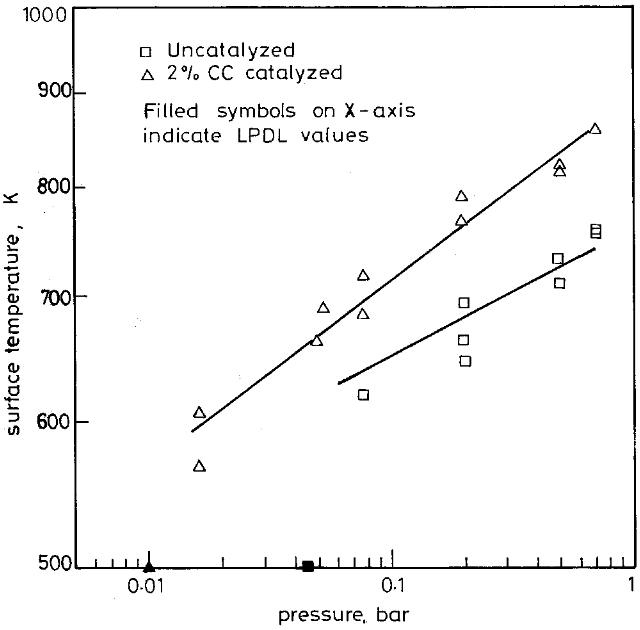

Fig. 5 Surface temperatures of AP/HTPB propellants.

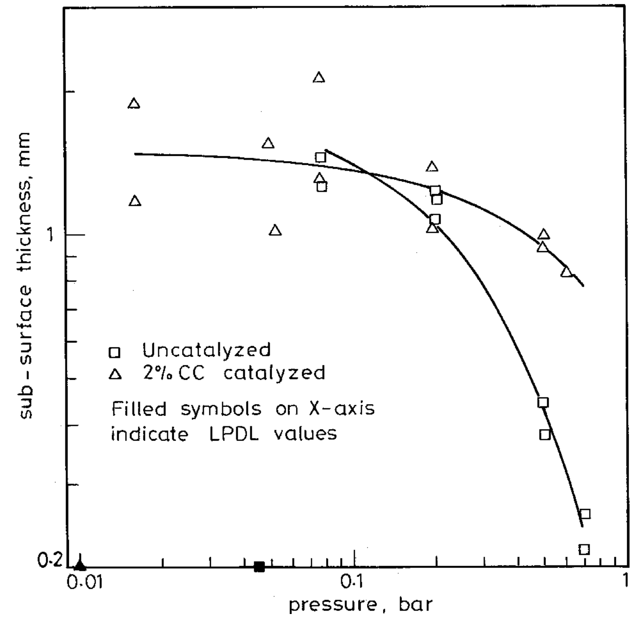

Fig. 6 Reacting subsurface thickness of AP/HTPB propellants.

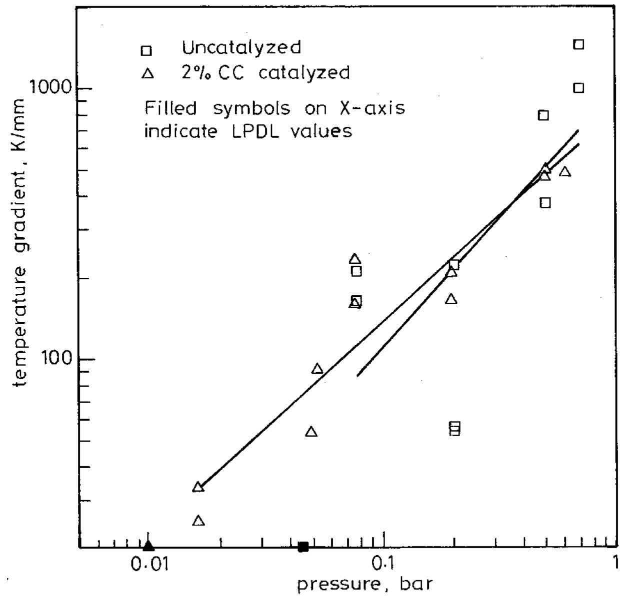

Fig. 7 Temperature gradient in the condensed phase of AP/HTPB propellants.

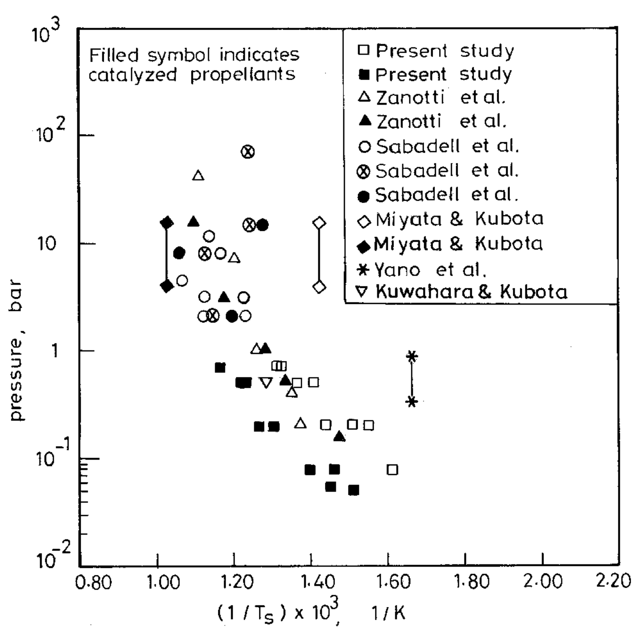

Fig. 8 Pressure vs $(1/T_s)$ from various studies (thermocouple measurements).

On the effect of addition of a catalyst (CC or Fe_2O_3), Sabadell et al.[13] did not find any observable surface temperature changes in the pressure ranges of 2–70 bar for CC and 2–15 bar for Fe_2O_3. However, on the effect of adding a burning-rate depressant $SrCO_3$, Miyata and Kubota[15] found an enhancement in surface temperature for the pressure range of 3–15 bar. They attributed this to a higher decomposition temperature of AP with $SrCO_3$. Our results in the subatmospheric pressure range indicate that the addition of CC enhances the surface temperature. This effect is because of the increased surface and subsurface heat release caused by CC. This can be shown from our experimental results as follows. Considering the one-dimensional heat balance at the burning surface

$$c_p(T_s - T_{\text{amb}}) - \left[\frac{\lambda_g}{(\rho_p r)}\right]\left(\frac{\mathrm{d}T}{\mathrm{d}x}\right)_g = Q_s - Q_l \qquad (5)$$

For unmetallized AP/HTPB propellant, Zanotti et al.[17] found the condensed-phase c_p to be 1423 J/(kg K) [0.34 cal/(g K)] before T_{tra} and 1841 J/(kg K) [0.44 cal/(g K)] after T_{tra}; an average value of 1632 J/(kg K) seems appropriate. For combustion modeling calculations the values used for λ_g vary from 0.084 to 0.13 W/(m K).[4,13] However, for the propellant composition used, the NASA SP-273 code[23] calculates the λ_g value as 0.18 W/(m K) and this does not vary much with the addition of CC. The code value corresponds to an ideal combustion condition that may not be attained at subatmospheric pressures. The temperature of the gases is expected to be much lower than the adiabatic flame temperature. A value of 0.084 W/(m K) is taken for present calculations. However, the uncertainty involved in the estimation of the λ_g value is noted with caution. We tried to calculate the gas-phase-temperature-gradient $(\mathrm{d}T/\mathrm{d}x)_g$, just above the burning surface from our experimental temperature profiles. Because these profiles above T_s were having temperature fluctuations, the values were showing a large scatter. However, these slopes were plotted against pressure and they demonstrated the expected trend of the increase in slope with pressure resulting in $(\mathrm{d}T/\mathrm{d}x)_g$ for uncatalyzed propellant as

$$\left(\frac{\mathrm{d}T}{\mathrm{d}x}\right)_g = 94933(p) + 10{,}706 \text{ K/cm, with } p \text{ in bar} \qquad (6)$$

and for the CC-catalyzed propellant as

$$\left(\frac{\mathrm{d}T}{\mathrm{d}x}\right)_g = 83865(p) + 25{,}387 \text{ K/cm, with } p \text{ in bar} \qquad (7)$$

These equations give essentially same $(\mathrm{d}T/\mathrm{d}x)_g$ for both propellants. Using these gas-phase gradients and the experimental values of surface temperatures, the $Q_s - Q_l$ values for both propellants were calculated and are shown in Fig. 9. As the heat losses in both cases are expected to be essentially same, it is seen that the addition of CC results in enhanced surface and subsurface heat release.

At this juncture, it may be suitable to briefly consider what will happen to the surface and subsurface heat release at rocket operating pressures. Diverse mechanisms are proposed for the burning-rate enhancement in catalyzed composite propellants at low as well as high pressures.[6,24] As per the surface reaction model,[5] significantly higher surface temperature is to be ex-

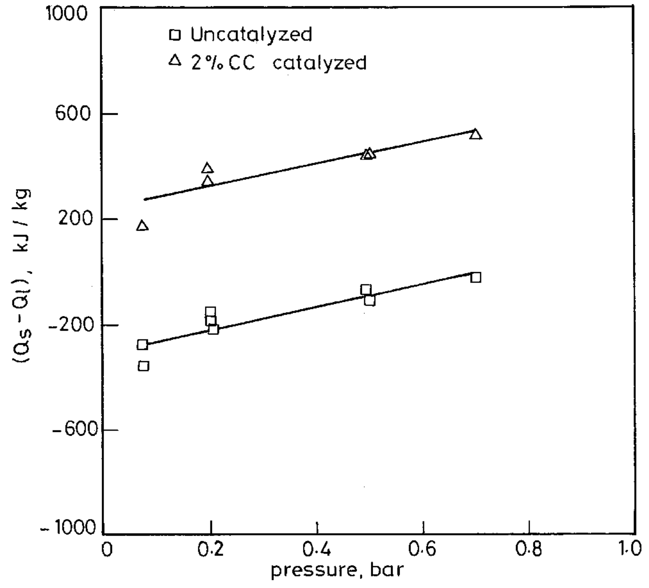

Fig. 9 Surface and subsurface heat release in AP/HTPB propellants.

pected at low pressures for the catalyzed propellant as it has been shown in the present experimental study. However, the difference between the surface temperatures of the catalyzed and the uncatalyzed propellants is expected to decline at rocket operating pressures. Applying the energy balance [Eq. (5)], at these high pressures, the surface and subsurface heat release of the catalyzed propellant is still expected to be higher than that of the uncatalyzed propellant, but not as much as at low pressures.

Conclusions

Subatmospheric burning surface temperature measurements, using 7.5-μm Pt and Pt-13% Rh thermocouples in CC catalyzed as well as uncatalyzed AP/HTPB propellants, indicate enhanced surface temperature for the catalyzed propellant. This temperature increase is shown to be because of the augmented surface and subsurface heat release that may result from condensed phase and/or heterogeneous reactions.

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