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Combustion of Ammonium Perchlorate-Polymer Sandwiches

E. W. Price,* J. C. Handley,† R. R. Panyam,‡ R. K. Sigman,§ and A. Ghosh‡
Georgia Institute of Technology, Atlanta, Ga.

An experimental study was made of combustion over the pressure range from 13.9 MPa to the low pressure combustion limit. Observations were made by high-speed photography and microscopic examination of quenched samples. Using sandwiches with binder laminae of graduated thickness, emphasis was placed on details in the dimensional range relevant to composite propellant microstructure. Results were qualitatively different from earlier "thick binder" results, reflecting a complex interplay of reaction sites and heat transfer not embodied in any analytical models of propellant or sandwich combustion.

Introduction

THE processes involved in combustion of heterogeneous solid rocket propellants are difficult to study because of the chemical and physical complexity of the propellant and the microscopic scale of the combustion zone. In the face of these difficulties, recourse has been taken to study of chemically and physically simpler systems. In the case of ammonium perchlorate-hydrocarbon binder propellants (AP-HC propellants), studies have been made of deflagration of the AP alone,[1-5] and of samples made by laminating a layer of binder between two layers of AP (sandwiches).[6-12] Combustion behavior of the simpler sandwiches is much easier to observe, infer, and describe than combustion of propellants. Some care must be exercised in design and interpretation of such tests if relevance to propellant combustion is desired. Studies leading up to the present work[6-8] have shown that thick binder laminae usually resulted in melt flows that encroached on the oxidizer surface, sometimes for distances of the same order as the radii of oxidizer particles in propellants. This, of course, raises doubts that the combustion of such sandwiches simulated propellants, which present very thin binder elements to the burning surface. It has also been observed in the relatively limited number of tests run on thin binder sandwiches[6-9] that the details of combustion in that same dimensional range were substantially different. Accordingly, the effect of binder thickness was evaluated systematically in the present work, and attention was directed particularly to the details of combustion in that region near the binder-oxidizer interface that is dimensionally relevant to propellant microstructure.

Experimental

Sandwiches were made from sheets of AP formed by dry-pressing ultra pure AP powder. The powder had a broad particle size distribution in the range 20-90 μm. Sheets 1.3 mm thick were pressed in a metal die at 214 MPa (31,000 psi) for 60-120 min. After cutting to desired sizes, a pair of sheets was bonded together by a binder layer (Fig. 1). Thickness of the binder was controlled by use of spacer shims during vacuum oven curing of the sample. Most samples were purposely made with a spacer on one edge only, so that the binder was shim thickness on one edge, tapered to about 10 μm on the opposite edge. Sandwiches were made with four different binders used in the propellant industry under the abbreviated names HTPB, CTPB, PBAN, and PS.¶ In addition, a few tests were run with a thin sheet of mica in place of binder.

Tests were run in nitrogen-pressurized chambers, one equipped with a burst diaphragm to permit rapid depressurization and sample quenching. The other chamber was equipped with nitrogen flushing, windows, a xenon lamp for illumination of the sample, and a high-speed (HyCam) 16-mm motion picture camera. Framing rates of 1000-2500 pictures/s were used, with image to object size typically 1:2. The samples were ignited by an electrically heated wire and a surface coating of metal-oxidant igniter paste. Experimental methods and apparatus are described in more detail in Ref. 7. Quenched samples were studied by optical and scanning electron microscopes. Table 1 lists the systematic tests that were run, the entries indicating the number of tests, and the number yielding usable results; additional tests for determining deflagration limits are listed later.

Results

General Characteristics of Sandwich Combustion

Combustion behavior has been described previously,[6-12] and results in this study confirmed those reported in Refs. 6-8. With binder lamina thickness uniform and in the range 75-300 μm (typical of earlier work), samples burned with the familiar "Christmas tree" profile in the region near the binder (Fig. 2a), extending out to a relatively flat AP surface at a pressure of 6.9 MPa (1000 psi). In low pressure tests in the present work (below the AP deflagration limit), burning was localized to that portion of the sandwich that was "supported" by the AP-binder flame, so that the surface consisted of a trough centered on the binder lamina (Fig. 2a). In the bottom of this trough, the profile still exhibited the Christmas tree profile. At high pressure (14 MPa) there was a tendency for the AP to slope upward more to the binder interface plane (Fig. 2a) giving the impression of outright retardation of AP regression. In some samples with PBAN and with PS binder, this "retardation" was so severe that plateau regions occurred locally along the interface. The binder thickness was not a critical variable until it went below about 70 μm, the main effect being less binder protrusion and melt flow with thin binder laminae (Figs. 2b and 2c). The thin-binder results are discussed in detail in the next section.

The details of quenched samples are illustrated by the quenched tapered sample in Fig. 3. The pattern of the quenched AP surface is characteristic of that given in single

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*Professor, School of Aerospace Engineering. Fellow AIAA.

†Senior Research Engineer, School of Aerospace Engineering. Member AIAA.

‡Graduate Research Assistant.

§Research Engineer II. Member AIAA.

¶Hydroxy-terminated polybutadiene, carboxy-terminated polybutadiene, polybutadiene-acrylic acid-acrylonitrile, and polysulfide: ingredients were supplied by the U.S. Naval Weapons Center.

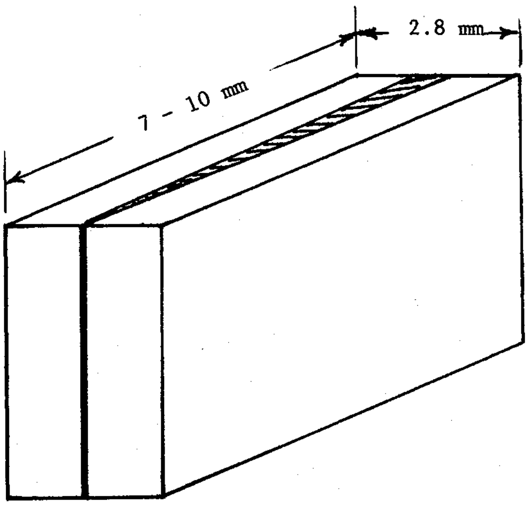

Fig. 1 Sketch of oxidizer-binder sandwich. Binder thickness is tapered to allow study of effect of thickness. Samples were burned on the tapered edge, except in deflagration-limit tests.

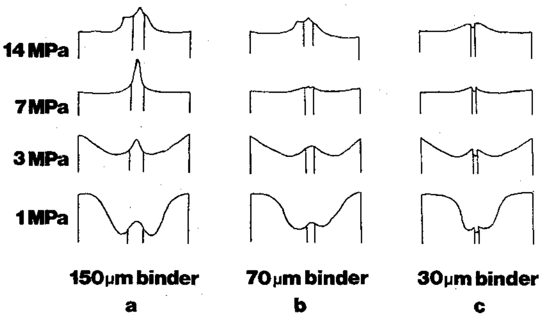

Fig. 2 Effect of pressure and binder thickness on burning surface profiles (trends, sketched; not to scale).

crystal AP deflagration tests.[1,2] The leading edge of the AP deflagration is 50-100 μm out from the AP-binder interface, and the surface at the interface slopes away from the protruding binder with evidence (most binders) of some binder melt flow over the interface onto the AP (not conspicuous with the PS binder in Fig. 3; see also Refs. 6-8). The regions of binder melt flow over the AP are recognizable by flow patterns suggestive of a viscous fluid, and by an otherwise smooth surface that obscures or prevents the usual AP surface patterns. However the smooth surface was present in virtually all tests as a smooth band of AP surface adjoining the AP-binder interface, even under conditions not conducive to melt flows (Fig. 3). The smooth band was present at all pressures, with all binders and with all binder thicknesses tested. Further, the leading edge of the AP regression was always located at the outer edge of the smooth band.

Combustion photography with viewing edge-on to the laminae showed the "bushy" flame described in Ref. 8; when viewed side-on this flame was found to be two rows of individual flamelets, one from each binder-AP interface (Fig. 4). The sandwiches with PS binder showed very little flame luminosity. Details of the flame could not be observed side-on at low pressure because the view of the flame is obstructed by the slower-burning AP. Other investigators[8,9] have reported a single, relatively steady flame at low pressure.

Tapered Sandwich Burning

Earlier work[6,8,9] has shown that combustion behavior was appreciably different with sandwiches having thin binder laminae. Since the binder structure in propellants is normally thin, it was deemed important to study thin binder sandwich combustion in detail. An opportunity to do this was provided by the tapered sandwich tests listed in Table 1. A number of singular aspects of burning occurred over the range of test variables used; the more typical features were shown by the quench profile sketches in Fig. 2 and sample surfaces shown in Fig. 3. The most conspicuous effect of decreasing binder

Fig. 3 Quenched surface of an AP-HC tapered sandwich. (Ammonium perchlorate-polysulfide, burned at 6.9 MPa (1000 psi).

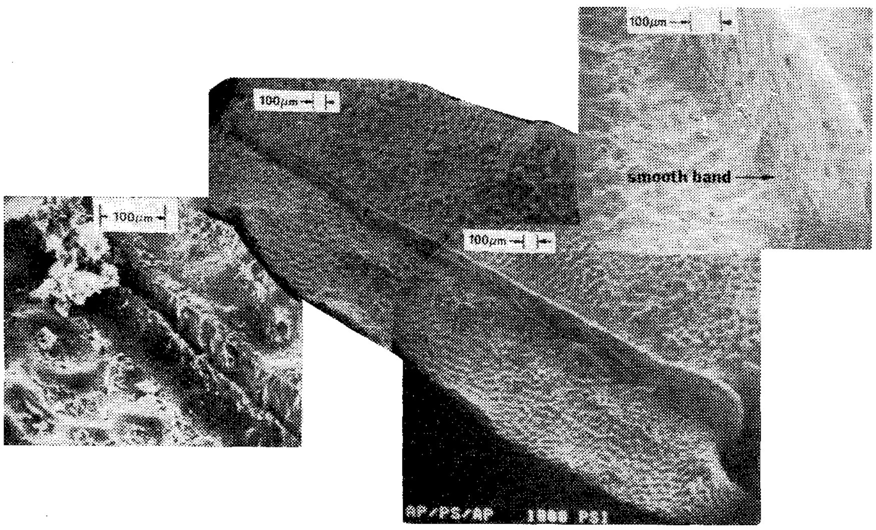

Table 1 Tests on tapered sandwiches

	1.38 MPa		4.14 MPa		6.89 MPa		13.8 MPa	
	Quench	Movie	Quench	Movie	Quench	Movie	Quench	Movie
PS	1	2/2	1	2/2	2	1/2	1	0/3*
PBAN	1	3/4	1	2/3	1	3/4	3	1/3
CTPB	1	1/1	1	1/1	1	1/1	1	0/1*
HTPB	2	4/4	2	3/3	3	3/3	4	0/1*
PBAA	1	1/1	1	0/1*	1	0/1*	1	1/1

N.B.: Numbers indicate successful tests and number of tests run. Numbers marked * indicate no successful tests.

Fig. 4 Diffusion flamelets associated with the AP-binder interfaces. Successive frames from motion picture, 0.001 s between pictures. Total sample width 2 cm: HTPB binder, tapered from 130 μm (left) to 10 μm. Pressure 6.9 MPa (1000 psi).

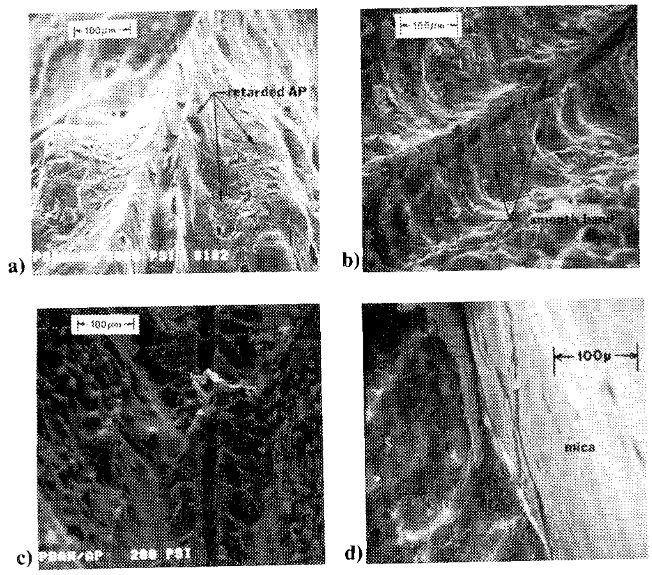

Fig. 5 Quenched surfaces, thin binder, various pressures: a) 13.9 MPa, PBAN binder; b) 4.1 MPa, HTPB binder; c) 1.4 MPa, PBAN binder; d) 6.9 MPa, mica in place of binder (mica on right, surface shows flaking).

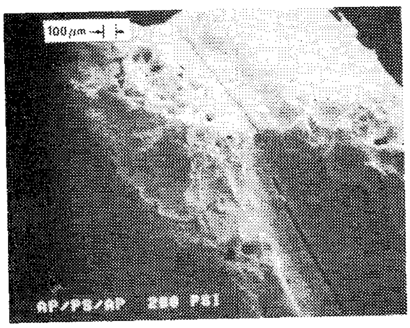

Fig. 6 Quenched sample for which the thin-binder end of the sample was below the sandwich deflagration limit. Polysulfide binder, sample burned at 1.4 MPa.

thickness below that discussed in the last section is a decrease in protrusion of the binder lamina, which became recessed below the AP surface with thicknesses less than about 30 μm (all 4 binders). The AP surface adjoining the AP-binder interface plane continues to exhibit the relatively smooth quality observed in thick-binder tests. Further, the leading edge of the AP regression remained some distance out from the interface plane (Fig. 2c), at the approximate outer edge of the "smooth band." These details are illustrated further by the example in Fig. 5, and are qualitatively typical of all four binders. Tests on sandwiches with the binder replaced by mica showed neither smooth band nor retardation of AP regression near the interface (Fig. 5d), indicating that binder-related processes are the cause of the smooth band and retardation.

At low pressure, two singular aspects of combustion developed with thin binders. The binder became conspicuously recessed (even the thick end of the sandwich), and the thin-binder end of the sample often did not burn (Fig. 6). The thin binder deflagration limit is discussed in the next section. The recessed-binder effect was present with thin binder at a test pressure of 1.4 to 13.9 MPa with each of the four binders (Fig. 5); the details at low pressure are illustrated in Fig. 7. The recess appears to have parallel walls (Figs. 5 and 7), and the surface of the binder in the recess appears to have been molten. There are some characteristic differences in appearance with different binders. Specifically, PBAN binder resulted in numerous "bridges" across the walls of the recess (Fig. 7a), initially attributed to recovery from bubble ejection from a viscous melt during depressurization quench. This explanation was later discounted, because the same features were present also on samples resulting from spontaneous quench at the deflagration limit (see later). HTPB and CTPB showed smooth surfaces with some wrinkle patterns (Fig. 7b) suggestive of a cooling liquid surface during quench. PS binder showed a profusion of microscopic flakes in (and sometimes adjoining) the recess (Fig. 7c). There was no qualitative evidence that the AP side-walls of the recesses had burned. The characteristic smooth band was present in samples, over the whole range of binder thickness that actually burned, and the AP surface regression outside the recess always appeared to be retarded in the smooth band, as at higher pressure (i.e., the leading edge of the AP burning surface was outside the smooth band).

A singular behavior observed at 13.9 MPa is shown in Fig. 8. The surface regression appears to have been delayed severely in the region of the smooth band. This behavior appears to be transitory (at this pressure, local retardation of surface regression would result in undercutting by deflagration from adjoining AP areas), and hence is not manifested as a fully developed plateau along the whole length of both interfaces. However PBAN and PS sandwiches consistently showed local plateaus, and all binders yielded

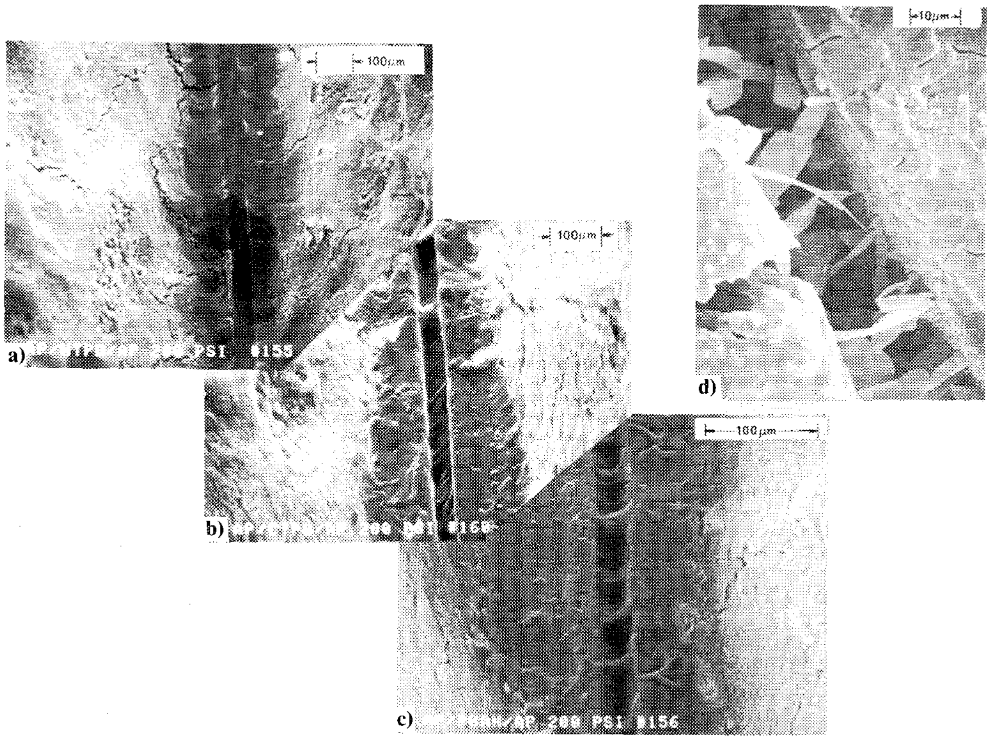

Fig. 7 Quenched samples at low pressure, thin binder, illustrating recessed binder, four different binders: a) PBAN binder; b) CTPB binder; c) HTPB binder; d) PS binder.

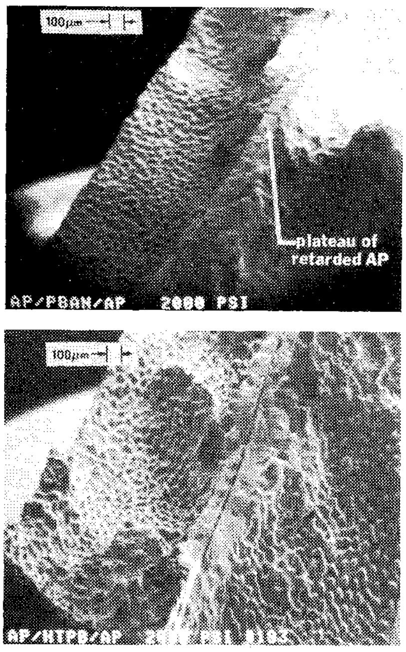

Fig. 8 Apparent retardation of surface regression of the oxidizer in the smooth band at high pressure: a) PBAN binder; b) HTPB binder.

unusually large up-slope of the AP in the profile adjoining the interface plane (Figs. 2a, 5a, and 8).

Diffusion Flame

Tapered sandwiches gave a convenient opportunity to view the nature of the AP-fuel flame vs binder thickness, as illustrated in Fig. 4. Such pictures show the luminous combustion to consist of a row of flamelets along each interface. These flamelets are individually transitory, most not being identifiable in successive pictures taken at 2200 frames/s. The flamelets at the thick-binder end of the burning surface are larger, more widely spaced, and survive for 1-3 frames. The flamelets at the thin-binder end are small, closely spaced, and short-lived (the distance scale is noted in the figure). This particular figure shows the two separate rows of flamelets particularly clearly because the front side of the sandwich has burned down ahead of the back side. To this point it has not been determined where the flame tips originate, i.e., relative to the burning surface profile. It is noted in the Discussion that this information is important to understanding or modeling burning properly, but observation is uncommonly difficult. The information provided here and elsewhere (Refs. 6-12) establishes the general dimensions of the flame zone and provokes questions about the possibly statistical nature of the flame, but not the needed space-time resolution of details. In this connection it should be noted that the orange luminosity seen in the motion pictures is probably largely radiation from hot carbon particles. As such, the luminosity is an indication of particle content and temperature, but of flame behavior only by inference.

Most of the photographic results were obtained at 6.9 MPa; at low pressure a side view of the flamelet array was not obtainable because of obstruction by the slower-burning AP in the line of viewing. Some tests were made with diagonal viewing angle, and showed nonsteady flamelets down to the lowest pressure tested (1.4 MPa). However the present observations are based on only fragmentary results because viewing of the full interfacial region was not achieved.

While the general description of 6.9 MPa results given above is qualitatively applicable, there were discernible differences with different binders. The description itself is typical of HTPB binder, illustrated in Fig. 4. The samples with PS binder showed almost no visible flame luminosity. CTPB binder gave more smoke, and more contiguous and steady flamelets. PBAN and PBAA binders gave results similar to HTPB.

Low Pressure Deflagration Limits

In the tapered sandwich experiments, ignition difficulties were encountered at low pressures and, as noted above, it was often found after rapid depressurization quenching that the thin-binder end of the sandwich had not burned. A preliminary series of low pressure tests established that, at 1.4 MPa, burning of tapered samples proceeded only in parts of a)

b)

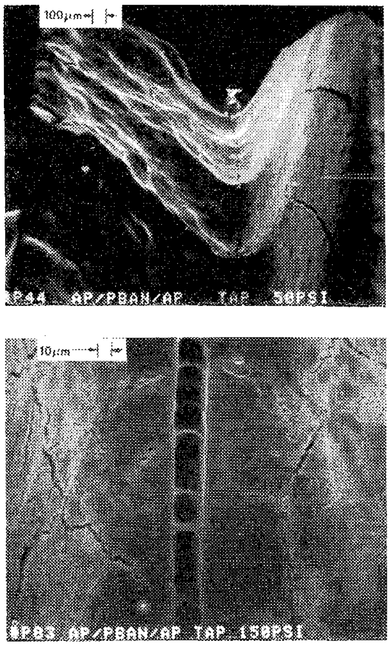

Fig. 9 Samples that quenched spontaneously when burned from the thick edge of the sample towards the thin edge (PBAN binder): a) general view—0.35 MPa test; b) surface detail—1.04 MPa test.

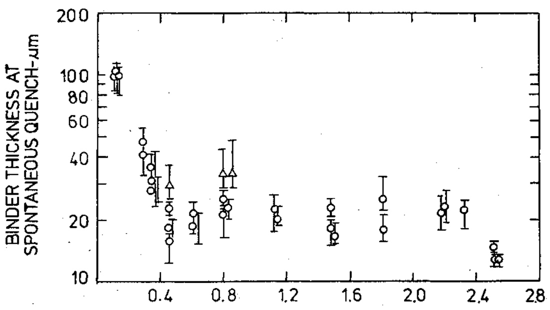

Fig. 10 Spontaneous quench conditions (circles PBAN binder, triangles polysulfide binder).

the sandwich with binder thickness > 20 µm. It was anticipated that this limit would be sensitive to pressure, type of binder, and possibly other variables important to propellant combustion. Accordingly, a series of tests was run on tapered sandwiches that were burned from the thick-binder edge of the sandwich towards the thin-binder edge. Samples burned in this way in low pressure tests quenched spontaneously, so that several measurements of binder thickness could be made along the quenched edge (using scanning electron micrograms). Examples of spontaneous quench samples are shown in Fig. 9. Detailed examination of the profile showed no qualitative difference from samples quenched from moderate pressures above the deflagration limit by rapid depressurization.

Detailed measurements of the binder thickness of spontaneously quenched samples are shown in Fig. 10. The error bars on the data points represent the range of measured thickness on each sample, with the data point being the average of 5 or more measurements. The binder used in these tests was PBAN. A limited number of tests were also run with PS binder (also shown in Fig. 10).

Discussion of Results

The principal features of combustion of AP-HC binder sandwiches are illustrated in Fig. 11. These include 1) a monopropellant flame close to the AP surface, 2) an AP-

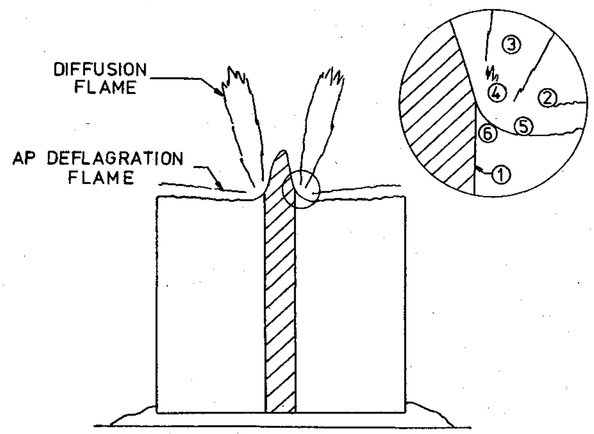

Fig. 11 Structure of the sandwich combustion zone. 1) AP-binder interface; 2) AP self-deflagration flame; 3) diffusion flamelet array; 4) collision rate-limited "phalanx" flame; 5) leading edge of the combustion front; 6) smooth band-melt flow region.

binder flame or flames, and 3) a complex decomposition on the AP surface that may be exothermal. The AP can deflagrate without a fuel if the pressure is above about 2.1 MPa, and in sandwiches the AP-binder flame sustains the burning to substantially lower pressure. At high pressure (e.g., 10 MPa) the AP regression is substantially independent of the AP-binder flame. The progression of experimentally observed surface profiles as a function of pressure in Fig. 2a reflects the increasing dependence of the over-all combustion on the AP-binder flame with decreasing pressure. At low pressure (e.g., 1 MPa), the combustion front progresses in a deep groove in the AP, because the AP-binder flame supports the AP decomposition only locally. The actual details of the AP-binder flame are largely unknown, although mechanistic arguments and experimental results have suggested that at high pressure the flame is primarily diffusion-limited. It has been noted[13] that the diffusion flame must possess a leading edge that is in effect premixed and kinetically limited. Further it has been noted[14] that the portion of the flame that is kinetically limited would be expected to increase with decreasing pressure, as collision rates decrease with density.

The foregoing provides a widely accepted mechanistic description of sandwich burning, and the present results conform to this description on the dimensional scale usually considered. However, in the present studies an effort was made to simulate and examine combustion more closely on the dimensional scale of the microstructure of composite rocket propellants, by going to thin-binder laminae and examining the combustion on a more microscopic scale. These studies led to a number of aspects of the combustion behavior that have not been generally recognized. While substantial effort will be required to clarify the mechanisms involved and their importance to propellant combustion, a brief summation at this point is in order.

Binder Thickness

Thin-binder sandwiches consistently exhibited different combustion behavior than thick binder (> 50 µm) sandwiches. Thick binder laminae protrude above the adjoining AP surface, exhibit extensive melt flows onto the AP surface, and burn with a flame consisting of multiple transitory flamelets. Thin binder sandwiches burn with the binder recessed below the adjoining AP surface, show melt, but usually no melt flow onto the AP surface, and they burn with a flame consisting of a larger number of smaller, more short-lived flamelets.

Smooth Band

The relatively smooth quality of the AP surface near the AP-binder interface has been evident in work reported earlier; under the thick-binder conditions of those tests, the effect was

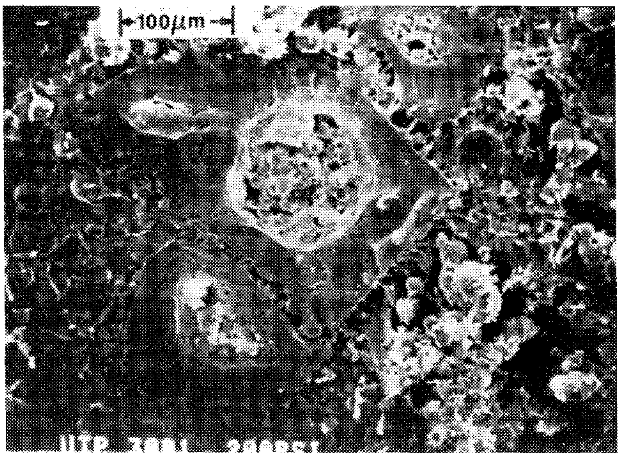

Fig. 12 Quenched AP-HTPB propellant, showing smooth surface of AP adjoining the AP-binder interfaces. Test at 1.04 MPa.

sometimes thought to be caused by melt flow. This explanation does not appear to be sufficient in all cases, since the smooth band is observed in the present work also with thin, recessed binders. It seems likely that the location of the smooth band is a reflection of conditions in the adjoining part of the gas phase diffusion field and flame. To date the smooth band seems to be more properly considered to be a condition resulting from important unidentified processes rather than an important feature in its own right. Interest in the band stems from the observation that the leading edge of the AP profile is always at the outer edge of the smooth band, giving the impression that the burning rate is retarded in the region of the smooth band. The dimensions of these features of the sandwich burning profile are of the same order as the radii of AP particles in propellants, which can exhibit similar features (Fig. 12).

"Leading Edge" and "Retarded" Regression

The tendency for the AP surface to slope *up* to the AP-binder interface plane (Figs. 2, 3, 5, 7-9, and 12) is as persistent as the smooth band. This feature may be due to enhancement of the AP rate by the AP-binder flame, with the effect maximizing at the location of the observed leading edge. Alternately, the feature may be viewed as retardation of AP rate in the region closer to the interface. At high pressure, one is inclined to attribute the feature to retardation, since the AP surface leads the profile *everywhere* else on the sample (i.e., flat surface). At low pressure, the AP rate must be regarded as enhanced everywhere that it burns, because it is below the AP self-deflagration limit. Then one may speculate that the leading edge is simply the point most favorably located relative to the sustaining exothermic gas phase reactions; however, one cannot accept this without doubt because it has not been shown that it is plausible for the gas phase heat release to be so distributed. Further, the possibility cannot yet be discounted that retardation effects are operative at low pressure as they appear to be at high pressure, although perhaps through different mechanisms. In the case of thin binder sandwiches where the binder is recessed, retardation of rate would presumably be produced by loss of heat from the AP thermal wave by convective transfer to the binder pyrolysis products flowing out of the adjoining recess. Present analytical models of sandwich burning[15-17] do not deal with these aspects of the process.

Combustion Limits

The initial objectives of the spontaneous quench tests were 1) to obtain a limiting condition for the low pressure combustion zone, that might help identify dominant processes, and 2) to obtain quench profiles that did not reflect possible effects of rapid depressurization. There was no doubt in advance about the existence of a thin-binder limit, since it had been encountered inadvertently in the 1.4 MPa tests burning the tapered face of the sandwiches (Fig. 6).

Regarding point 2 above, there was no notable difference between profiles of spontaneously quenched samples and ones quenched by rapid depressurization from 1.4 MPa (PBAN binder). Thus nothing dramatic seems to occur to the profile or surface details as a prelude to spontaneous quench, and the features observed on samples quenched from 1.4 MPa by depressurization were apparently unaffected by depressurization.

Regarding the nature of the limiting conditions for combustion, it is helpful to consider two burning experiments. In one, a sample of constant binder thickness is burned while the pressure is slowly reduced. At some pressure the *reaction rate in the gas phase will become* primarily *collision rate-limited*, and decreasing with pressure. This situation typically leads eventually to a condition where heat loss exceeds heat generation, and quenching occurs. This quench condition is indicated by the trend of the data at low pressure in Fig. 10 (viewed as a limit curve for decreasing pressure experiments). In the present problem it appears that the heat balance is less conducive to sustained burning when the binder thickness is less (Fig. 10), probably because of *unfavorable stoichiometry* with thin binder.

In the second of the two burning experiments, consider the experiments actually run, i.e., at constant pressure with decreasing binder thickness. As the binder thickness decreases, the flame eventually becomes so fuel lean, and the steepening sample profile becomes such a good "heat sink," that energy balance is no longer maintained. In the pressure range 0.6-2.3 MPa, this is pressure independent (lower part of the curve in Fig. 10) because the flame is diffusion limited and hence pressure independent. At higher pressure, self deflagration of the AP eliminates the binder-thickness dependence of the sample combustion, although the diffusion flame continues to affect combustion details near the interfaces. For the low pressure range ($p<0.6$ MPa), the spontaneous quench of the tapered sandwich is determined by the same arguments as at higher pressure, but with the added limitation of low collision rates. Then as test pressure is lowered, the more favorable stoichiometry of thicker binder is required for sustained burning.

This rather simplistic explanation of the trend of the combustion limit data in Fig. 10 does not explain the details of quenched sample surfaces; an explanation would require detailed analytical modeling. However the results do permit some general statements about AP/HC sandwich burning: 1) the AP flame reaction appears to be indistinguishable from the AP-binder flame below 2.3 MPa; 2) the AP-binder flame is diffusion-limited above 0.6 MPa; 3) the diffusion flame is not self-sustaining at any pressure below the AP deflagration limit when the binder lamina is below about 20 μm in thickness.

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