



AIAA 2001-0339
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OF COMPOSITE PROPELLANTS

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39th Aerospace Sciences
Meeting & Exhibit
January 8-11, 2001 / Reno, NV

INTERMITTENT BURNING AND ITS CONTRIBUTION TO PLATEAU BURNING OF COMPOSITE PROPELLANTS

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Abstract

The plateau burning behavior of composite solid propellants consisting of ammonium perchlorate (AP) and hydrocarbon (HC) binder with a bimodal AP particle size distribution (coarse and fine) is examined. The focus is the weak pressure dependence of the propellant burn rate (i.e., a plateau) in an intermediate range of about 2.7-6.9 MPa (~400-1000 psi). The relationship between the appearance of this mid-pressure plateau for a composite propellant and self-extinction during the burning of the corresponding fine AP/binder matrix (i.e., the propellant formulation without the coarse AP particles) is experimentally examined through the study of a compositional array of propellants, sandwiches (two-dimensional propellants) and matrixes. The burning history of the samples was captured with a high-speed digital camera, and surfaces from quenched samples (burning that was self-extinguished or intentionally interrupted) are analyzed using a scanning electron microscope. The combined results indicate the prevalence of intermittent burning of the matrixes as the pressure is varied across the boundary between continuous burning and self-extinction (burn/no-burn boundary). The burning surfaces are marked by extreme three-dimensionality coupled with a redistribution of the fine AP particles and the binder. The results point to the need for a more realistic approach to the underlying processes that contribute to plateau burning rate trends in bimodal composite propellants than has been adopted hitherto.

Introduction

Plateau (low pressure sensitivity) burning rate trends of solid propellants have always attracted rocket motor developers for several reasons: greater margin of

stability of motor operation, decreased temperature sensitivity of motor pressure to propellant initial temperature, and the possibility of reduced combustion instability of the propellant. The search for propellant formulations that exhibit plateau burning rate trends has been pursued ever since the advent of composite propellants based chiefly on ammonium perchlorate (AP) as the oxidizer in the last several decades.

Early attempts to develop plateau propellants were made by Bastress,¹ followed by others of the Princeton group led by Summerfield,² in the 1960s. These works pertain to propellants that are different from modern compositions mainly in two respects. First, they used several different hydrocarbon (HC) binders, most of which are outdated today. Second, there were several formulations that showed “anomalous” burning rate trends such as mid-pressure extinction, mesa burning (regions of decreased burn rate with increased pressure), and plateau burning, but almost all of these formulations were unacceptably fuel-rich.

Further work on propellants with plateau burning rate trends has largely been confined to company proprietary material. Some work presented by Miller and co-workers (e.g., Ref. 3) indicate the importance of the composition of the fine AP/binder matrix that forms the region between coarse AP particles in a composite propellant with a bimodal AP particle size distribution. In particular, one propellant formulation has been shown to exhibit a plateau trend in a narrow pressure range corresponding to extinction of its corresponding fine AP/binder in that pressure range, when tested alone. The propellants in this series of works were composed of a modern binder, hydroxyl-terminated polybutadiene (HTPB), but used a modest loading of total solids loading (~80%).

In the last several years, propellants with bimodal AP size distribution exhibiting “bi-plateau” burning rate trends have been developed.⁴ The rich behavior of these propellants suggests there are multiple physical mechanisms at work – mostly in concert with each

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other – in all propellant formulations, and that the interplay of these mechanisms is manifested as anomalous burning in specific propellant formulations in certain pressure ranges.^{5,6} In previous research by the authors, it was found that monomodal mixtures of fine AP particles and binder, representative of the fine AP/binder areas between coarse AP particles in bimodal propellants (and hereafter denoted as the “matrix” or “matrixes”), exhibited interesting patterns of “burn” and “no-burn” domains depending upon the AP content, AP particle size, binder type (including type of curing agent), and pressure.

The present paper focuses on the role of the matrix burn/no-burn behavior in yielding plateau burning rate trends in the corresponding propellants. Previous results indicate a link between the matrix no-burn domains and plateau burning rate trends in sandwiches (essentially two-dimensional propellant samples).⁷ The present work is a continuation of this aspect in terms of examining propellant burning rates containing such matrixes, and presents results indicating the intermittent nature of the burning process witnessed in these matrixes.

Experimental Methods

Samples

Broadly, three types of samples were tested in the present study: matrixes, propellants, and sandwiches (essentially a two-dimensional propellant system). The focus of the present study was matrixes containing 65% fine AP of either 2 μm or 10 μm size and binder made up of HTPB cured by isophorone diisocyanate (IPDI). The 2 μm AP was supplied by C. J. Hinshaw, Thiokol Corporation, in the form of a mixture with uncured HTPB and DOA (dioctyl adipate, plasticizer). The 10 μm AP was supplied by K. J. Kraeutle, Naval Air Warfare Center (Weapons Division).

The propellant samples had a total solids loading of 87.5%, with the same fine AP/binder matrix as above. The size of the coarse AP that constituted the remainder of the solids was 355-425 μm . The propellants were processed using a unique micro-mixer and degasser,⁸ and according to procedures detailed in Ref. 8. Both matrix and propellant samples were between 7-12 mm tall, 3-6 mm wide and 2-4 mm thick.

The sandwiches used pressed pellets of AP to simulate the presence of coarse AP particles in the propellant in the combustion zone microstructure. The pellets were prepared according to details presented elsewhere.⁹ The sandwiches contained a lamina of the matrix with composition as above sandwiched by the slabs of AP. Three levels of matrix lamina thickness were

tested in the present study: “thin” (~100-150 μm), “optimum” (~250-275 μm), and “thick” (~450-500 μm). The binder composition in all the samples is HTPB – 75.73%, DOA – 18.39%, and IPDI – 5.88% as in past studies.⁷

Techniques

Two techniques were used primarily in the present study: (i) combustion photography, and (ii) scanning electron microscopy (SEM) of quenched samples. The combustion photography experiments were performed in a “window bomb”, and the combustion event was imaged using either a color video camera or a high-speed, digital camera (Redlake Imaging). Burning rates were obtained from the combustion photography experiments based on a frame-by-frame analysis of the images.

In the high-speed imaging experiments, a framing rate of 500 frames per second was employed. In these experiments, the samples were cut at 45° to the direction of flame propagation, and ignition was effected at the bottom edge of the cut surface. The line of view of the camera was, thus, oblique to the burning surface. A blue Kodak Wratten filter was fitted to the camera in most experiments (particularly when testing propellants and sandwiches) in order to filter out all radiation except that corresponding to CH chemiluminescence, which is representative of the gas-phase reaction zone.

Quenched samples were obtained by rapid depressurization under conditions when the samples burned to completion. Self-quenched samples – mostly those of matrixes in the “no-burn” domain – were also examined in the SEM. These techniques are routine, and are also detailed elsewhere.⁸

Results

Matrix Burn/No-Burn Domain

In order to form a basis for tests performed in the present study, it is important to review the pattern of burn/no-burn behavior of fine AP/binder matrixes, when tested alone.⁷ In that case, three different binder types were considered, viz., PBAN (poly-butadiene acrylonitrile acrylic acid), HTPB cured by IPDI, and HTPB cured by DDI (dimeryll diisocyanate). Further studies have expanded on this approach in terms of the fine AP/binder ratio.¹⁰

Of these, it suffices for the purpose of the present work to examine only the behavior of matrixes with 65% fine AP in a binder of HTPB cured by IPDI, at different fine AP size levels (Fig. 1). Three times of behavior are found: normal burning (the sample burns

to completion), interrupted burning (the sample burns for some time before self-quenching), and no burning at all (the sample does not continue to burn after the ignition event). It can be seen that the matrix with the fine AP size of 2 μm burns in a narrow range of 2.07-2.42 MPa (300-350 psig), while the 10 μm fine AP particle matrixes burn over a larger pressure range, with a no-burn domain for 3.45-13.78 MPa (500-2000 psig).

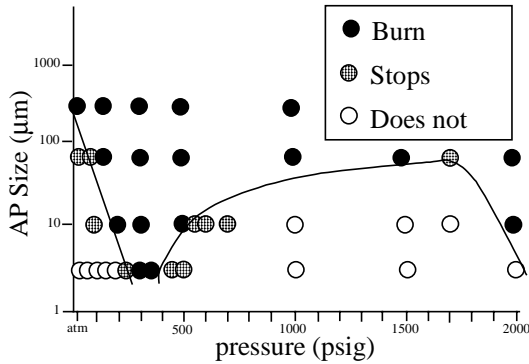


Figure 1. Pressure deflagration limits of fine AP/HTPB-IPDI = 65/35 matrixes with different particle sizes (Ref. 7).

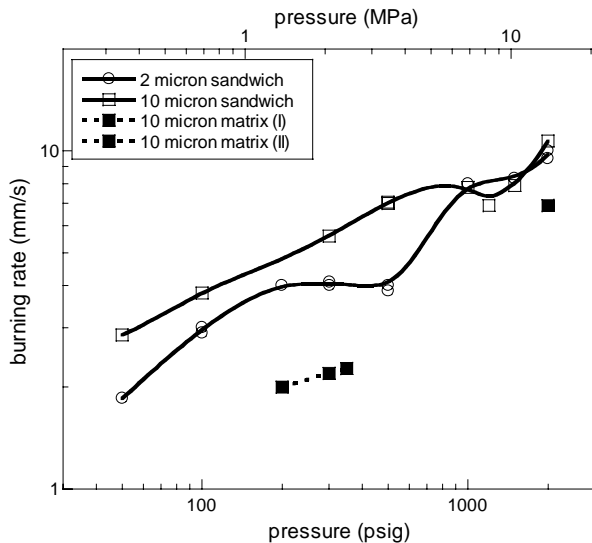


Figure 2. Burning rate versus pressure for sandwiches with optimum matrix lamina thickness (taken from Ref. 7).

Sandwich Burning Rates

The burning rate variation with pressure for sandwiches with an “optimum” matrix lamina thickness is also presented in Ref. 7. The matrixes in these sandwiches are the same as those corresponding to Fig. 1. These results are reproduced here (see Fig. 2) in order to serve as background for further results obtained in the present study. The figure shows burning rates for matrix and sandwich samples that contain 2 μm or 10 μm fine AP particles in the matrixes. The 2 μm ma-

trix did not burn in the entire test pressure range when these results were obtained. In any case, it can be seen from Fig. 2 that the sandwich with the 2 μm matrix exhibits a strong mesa in the pressure range corresponding to extinction of its matrix when tested alone, as in Fig. 1. Similarly, the sandwich with the 10 μm matrix exhibits a plateau in the pressure range corresponding to extinction of its matrix.

Propellant Burning Rates

Figure 3 shows burning rates, obtained in the present study, of a family of two propellant formulations that are identical in all respects except for the fine AP particle size being 2 μm in one, and 10 μm in the other. The fine AP/binder ratio in these formulations corresponds to the matrix compositions shown in Fig. 1.

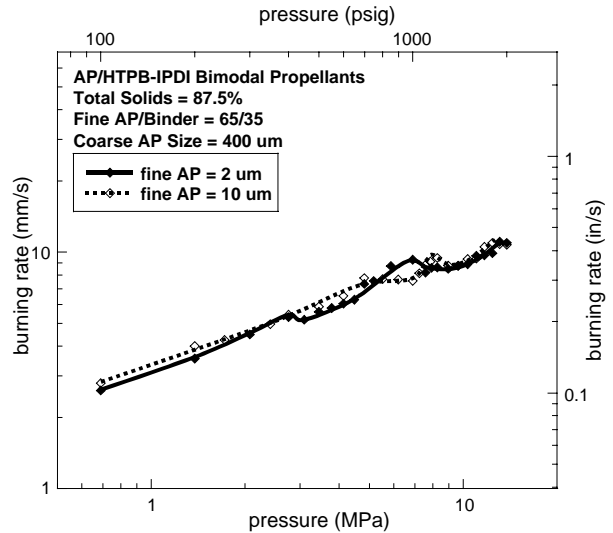


Figure 3. Burning rates of bimodal propellants with matrix formulations identical to those of Fig. 1.

Both propellant formulations clearly exhibit plateau burning in a relatively high pressure range, 6.89-13.78 MPa (1000-2000 psig). However, this aspect is not the focus of the present study, rather we concentrate on the occurrence of plateaus at an intermediate pressure range. The 2 μm fine AP formulation exhibits such a mid-pressure plateau in the range 2.76-3.10 MPa (400-450 psig). Although the plateau is small, its pressure range corresponds to the domain of onset of no-burn of the fine AP/binder matrix when tested alone (Fig. 1).

For the 10 μm fine AP propellant, a more substantial plateau in the burning rate is observed for 4.82-6.89 MPa (700-1000 psig). [Note, the high-pressure plateau in this case occurs at a higher pressure.] Again the occurrence of the mid-pressure plateau is at a pressure range where the corresponding matrix is entering its no burn domain. Also note that data was obtained at suffi-

ciently fine intervals to unambiguously resolve these trends.

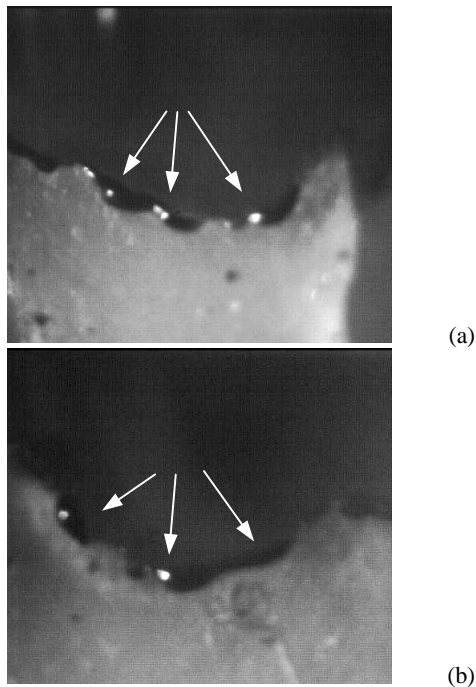


Figure 4. Frames from high-speed images of combustion of the 2 μm AP matrix at (a) 2.76 MPa (400 psig) and (b) 3.45 MPa (500 psig). Melt regions are dark (see arrows), often having bright dots from reflection of the illuminating light.

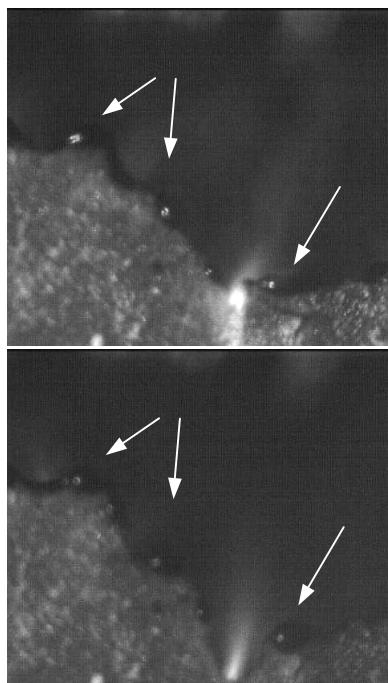


Figure 5. Two consecutive frames from a high-speed imaging sequence of the combustion of the 10 μm AP matrix at 3.79 MPa (550 psig). Melt indicated by arrows.

High-Speed Combustion Imaging

Matrix Samples

The matrixes with 2 μm fine AP were tested in the pressure range of 2.07-3.45 MPa (300-500 psig), the range that corresponds to the transition of matrix deflagration behavior from burning to a no-burn condition. Most matrix samples tested above 2.41 MPa (350 psig) quenched part way through the burn by themselves. In general, no specific event was witnessed in the high-speed images that could be considered as triggering extinction. The burning was on the whole extremely sporadic and transitory, and the burning surface regressed in an extremely non-uniform manner in the pressure range just around the burn/no-burn boundary (so much so that a sharp boundary cannot be attributed to this behavior).

The burning was also marked by formation of dark beads of what appeared like melt drops along the edges of the sample (seen along the edge facing the camera, in most cases). Figure 4 shows formation of melt blobs along the front edge of the sample at 2.76 and 3.45 MPa (400, 500 psig) during burning. These samples self-quench after the frames shown here. Although the melt drops are primarily edge effects, they point to the presence of molten material on the burning surface that accumulate along the sample edges.

While no luminous flame is visible on most of the burning surface in these samples, bright flamelets are formed in some instances along the peripheries of the melt drops and consume them. This causes a vigorous advance of the burning surface in that region subsequent to the consumption of the melt, while simultaneously another location shows formation of new melt and consequent retardation of the surface regression there. Such a sequence of events is shown in Fig. 5 for the 10 μm fine AP matrix sample burning at 3.79 MPa (550 psig). By and large, the comments made in the context of the 2 μm AP matrix are valid in the case with the 10 μm fine AP as well, except that the extent of melt drop formation in the 10 μm case is more pronounced than for the 2 μm matrix samples.

Propellant Samples

The propellant burning images were not as striking as the matrix images. Some images showed large areas of low intensity (blue flame) or appeared somewhat dark momentarily. It is not clearly discernible as a symptom of intermittent burning of the matrix, apart from the inherent intermittence associated with the coarse AP burning. For instance, Fig. 6 shows three consecutive frames that reveal momentarily poor burning (marked region). This could possibly be due burn

out of a large particle, leading to a local and temporary collapse of the matrix flame.

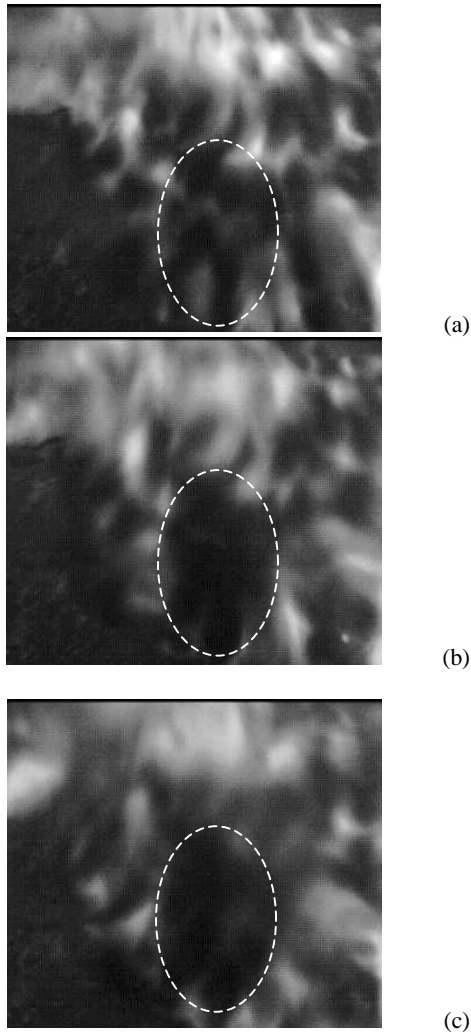


Figure 6. Sequence of consecutive frames, taken 2 ms apart, from high-speed combustion imaging of a bimodal propellant with the 2 μm AP matrix at 2.07 MPa (300 psig).

Sandwich Samples

As noted above, three lamina thicknesses were used for the matrix. The 2 μm AP matrix sandwiches were quenched at 2.07 and 3.45 MPa (300 and 500 psig), whereas the 10 μm AP matrix sandwiches were quenched at 4.13 and 5.51 MPa (600 and 800 psig). These pressure levels were selected to observe any contrasts in the burning behavior at pressure levels corresponding to a matrix burning condition and that corresponding to a matrix non-burning condition.

No significant differences were observed for sandwiches with either thin or optimal thickness matrix lamina. This is likely a result of the poor spatial resolution of the imaging system compared to the lamina

thickness. However, a remarkable distinction was revealed for sandwiches with thick matrix lamina at the two pressure levels that lay across the matrix burn/no-burn boundary. For the pressures where the matrix alone burned, the matrix lamina burned normally. For a small pressure change, sufficient to cross into the matrix no-burn domains, however, the matrix lamina protruded above the AP. This is shown in Fig. 7 for the case of sandwiches with thick lamina containing the 10 μm AP.

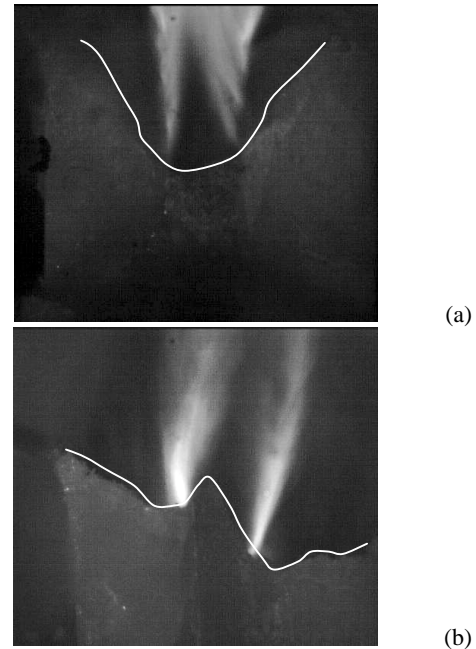


Figure 7. Thick matrix lamina sandwiches containing 10 μm fine AP, burning at (a) 4.13 MPa (600 psig) and (b) 5.51 MPa (800 psig) (surface edge outlined in white).

Quenched Samples

Matrix Samples

Figure 8 shows surface features of the 2 μm AP matrix samples quenched (by rapid depressurization) at 300 psig (normal burning) and 400 psig (corresponding to self-quenching). Figure 9 shows similar images for the 10 μm AP matrix, obtained at 500 and 600 psig. There are some significant differences in the large scale surface features of the matrix samples for the normal burning (Figs. 8-9 (a)) compared to cases where the matrix can not sustain a deflagration (Figs. 8-9 (b)). For the self-quenching conditions, the surface is quite irregular, with large depressions on a spatial scale much larger than the size of the fine AP. As there is no coarse AP in the matrix samples, these depressions represent large-scale variations in the matrix regression rate. Based on previous comparisons of thermal and depres-

surization quenching, it is unlikely that the results are an artifact of the different quenching processes.

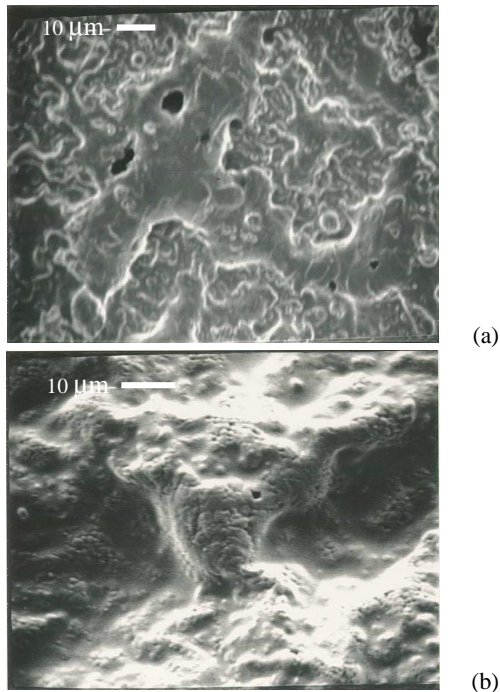


Figure 8. Surface features of 2 μm AP matrix (a) quenched at 2.07 MPa (300 psig) by rapid depressurization, and (b) self-quenched at 2.76 MPa (400 psig).

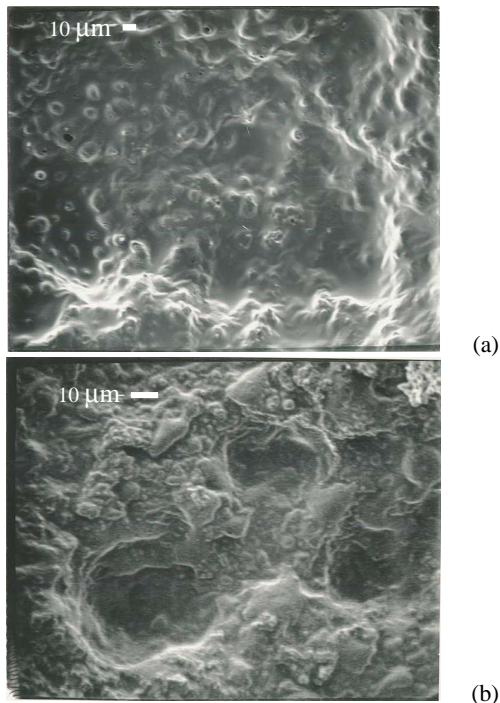


Figure 9. Surface features of 10 μm AP matrix (a) quenched at 3.45 MPa (500 psig) by rapid depressurization, and (b) self-quenched at 4.13 MPa (600 psig).

On a smaller spatial scale, both burn and self-quench cases exhibit a burning surface that is locally non-planar with noticeable undulations. The undulations are more evident in the normal burning cases, and it is likely that they intensify and grow into the large-scale depressions seen in the self-quench cases. Moreover, the spatial distribution of the fine AP particle appears to be non-uniform in both cases, with the fine AP particles more likely to be found along the undulations. For example, the left and central regions of Fig. 9(a) exhibit a lower AP concentration than the ridge region along the right and bottom sides of the image. This latter feature is even more prominent with the 2 μm fine AP particles than with the 10 μm fine AP particles. In addition, some samples show smooth layers of melted binder along the periphery of the matrix sample, corroborating the formation of melt regions seen in the high-speed images.

Propellant Samples

The surface features of propellants containing the matrixes with 2 μm fine AP particles quenched by rapid depressurization at 2.07, 2.76, and 3.45 MPa (300, 400, and 500 psig) are shown in Fig. 10. Similarly, Fig. 11 shows the corresponding images for the propellant with 10 μm fine AP, but quenched at 4.13, 4.82, and 5.51 MPa (600, 700, and 800 psig) – also by rapid depressurization. The pressure levels are chosen to match the matrix burn/no-burn boundaries.

At the lowest pressures for each propellant, Figs. 10-11(a) (where the matrix burns better on its own), the burning surface is relatively flat in the matrix areas, when compared to the higher pressures (Figs. 10-11(b) and (c)), where the matrix-alone burning progressively worsens. Furthermore, this is manifested in undulations in the matrix areas as the pressure is increased across the matrix burn/no-burn boundary, with the undulations now aligned along the layers between the coarse AP particles. Where a coarse AP particle has just finished burning, the underlying matrix area is large enough for some “natural” undulations to occur similar to the surface features of a matrix undergoing self-quenching, when tested alone.

Sandwich Samples

Figure 12 shows SEM images of quenched samples of the 10 μm fine AP matrix lamina at a high pressure (800 psig) and two thicknesses (thin, Fig. 12(a), and thick, Fig. 12(b)) and a low pressure (600 psig) for one thickness (thick, Fig. 12(c)). Also shown is one SEM for the 2 μm case (Fig. 12(d), thick lamina, 500 psig).

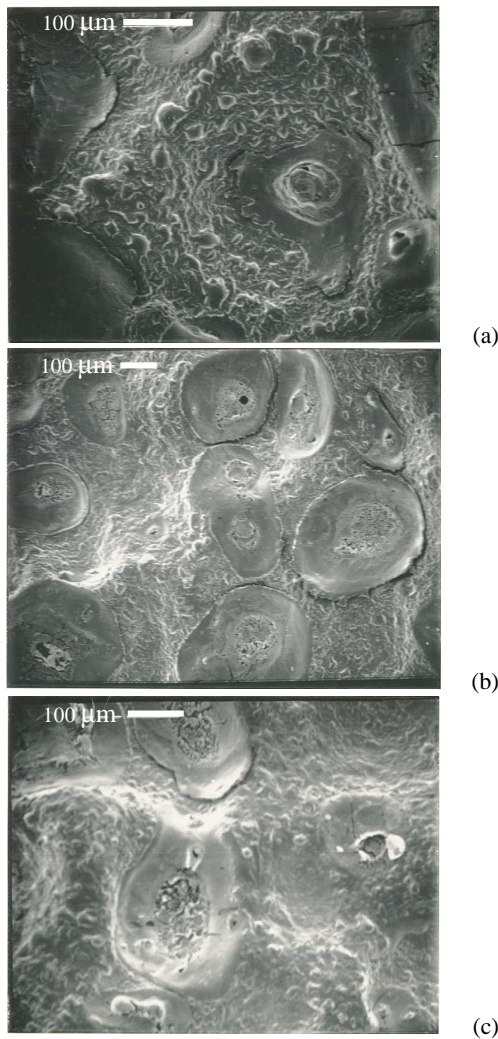


Figure 10. Surface features of a bimodal propellant containing the 2 μm AP matrix quenched at (a) 2.07 MPa (300 psig – a matrix burn case), (b) 2.76 MPa (400 psig – a matrix extinction case), and (c) 3.45 MPa (500 psig – a matrix extinction case).

In the case of sandwich burning, there appear to be different “modes” of undulations permitted on the burning surface of the matrix lamina, depending upon its width. For thin matrix lamina, the matrix surface has some small-scale ripples (Fig. 12(a)). For the optimally thick matrix lamina (not shown), truly large-scale undulations similar to those in the matrix burning alone, but aligned along the lamina interface edge, are seen. This gives the impression of unsymmetrical burning as well as non-two-dimensional burning of the sandwich. With very thick matrix lamina, the undulations are not confined to be along the interface edge, and so the behavior is similar to that when the matrix is tested alone (Fig. 12(b)).

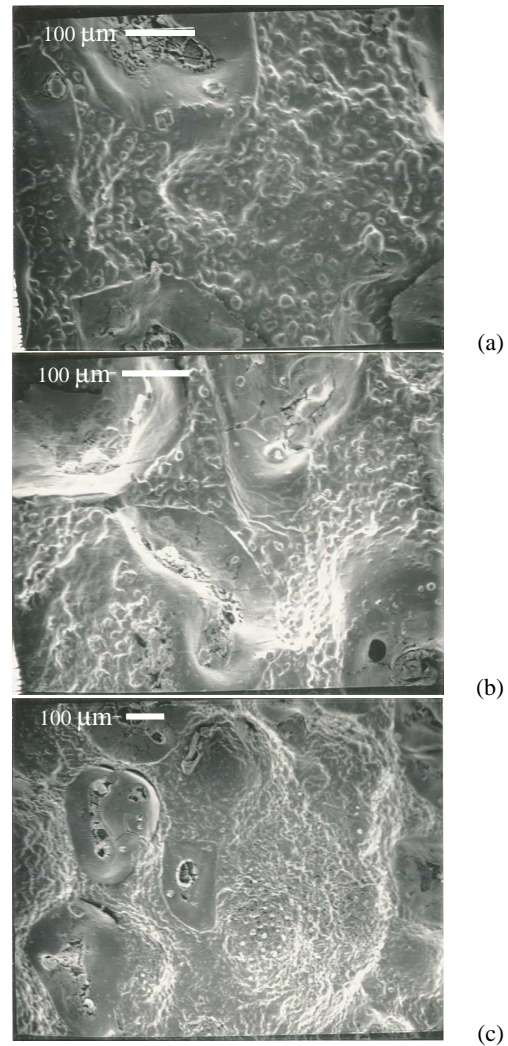


Figure 11. Surface features of a bimodal propellant containing the 10 μm AP matrix quenched at (a) 4.13 MPa (600 psig), (b) 4.82 MPa (700 psig), and (c) 5.51 MPa (800 psig).

As seen in the quenched matrix samples, there are more non-uniformities on the matrix lamina surface at pressures corresponding to matrix no-burn (Fig. 12(b)) than at pressures when the matrix burns by itself (Fig. 12(c)). These features are similar between samples with matrixes containing the 2 μm and the 10 μm fine AP, but the features are less pronounced for the finer AP lamina (Fig. 12(d)).

Discussion

The present work has achieved some success in highlighting the following:

- a) The link between matrix no-burn domains and plateaus in propellants and propellant sandwiches.

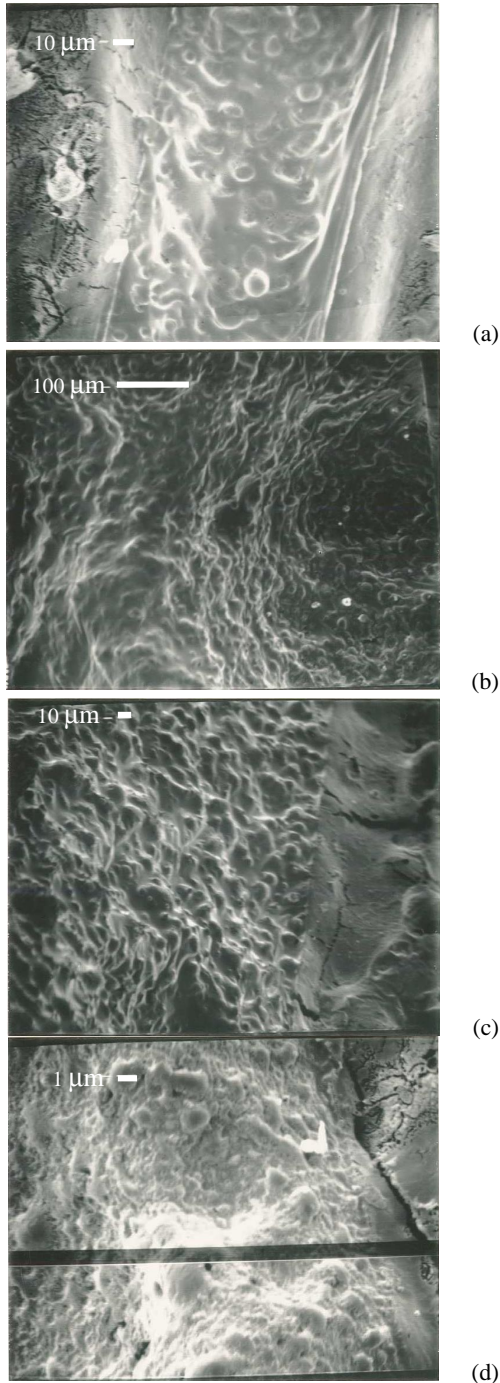


Figure 12. Surface features of sandwiches quenched under different conditions: (a) thin matrix (10 μm AP), 5.51 MPa (800 psig); (b) thick matrix (10 μm AP), 5.51 MPa (800 psig); (c) thick matrix (10 μm AP), 4.13 MPa (600 psig); (d) thick matrix (2 μm AP), 3.45 MPa (500 psig).

- b) Intermittent burning and far-from-planar burning surfaces are associated with the processes leading to matrix no-burns and propellant/sandwich plateaus.

As the pressure is raised across the burn/no-burn boundary of the fine AP-binder matrix (when tested alone), large-scale non-planar burning sets in. This suggests a situation of multi-dimensional heat feedback to the condensed phase, which triggers intense heat loss at certain sites in the reaction zone relative to others. Such sites undergo retarded regression relative to other parts of the burning surface, thus accentuating the non-planarity.

The appearance of the melt material along the edges of the burning surface is an artifact of the finite extent of the sample cross-section. However, it points to the presence of binder in molten form in a thin layer on the burning surface, and moreover, to the possibility of mobility of thick enough regions of the molten binder layer. It is not otherwise possible for the molten binder to accumulate along the edges.

This is manifested on a larger scale in the form of non-uniform surface regression in the matrix areas away from coarse AP particles in the propellants or away from AP lamina in the sandwiches in the pressure range corresponding to extinction of the matrix alone. On a local scale, this process causes the fine AP particles to accumulate and align along the surface non-uniformities. This is because the fine AP particles are small enough to be within the melt layer of the binder. Such features are borne out by the quenched samples.

It has been argued previously^{5,11} that a pyrolyzing surface of a two-component material would tend to allow one of the components, the one that pyrolyzes more slowly, to accumulate at the surface layer. This is a precondition for stable pyrolysis, and has been termed as "surface disproportionation". On the other hand, it is likely that the redistribution of the fine AP particles as part of the microflow of the binder melt on the burning surface could cause the disproportionation process to fail. This situation would lead to an unstable situation leading to a local quench event.

When the matrix areas find themselves in a propellant or a sandwich, they derive support from the near-surface leading edge parts of the oxidizer/fuel diffusion flamelets attached to peripheries of the adjacent coarse AP particles or the AP lamina. Hence, they do not undergo a no-burn condition. However, the processes encountered by the matrix when it burns alone would still exist and be more dominant in regions far away from

the coarse AP particles or the AP lamina. This results in intermittent burning of the matrix areas.

Conclusions

Burning of composite solid propellants based on ammonium perchlorate oxidizer of bimodal particle size distribution involves substantial areas of burning surface layer made up of a matrix of fine AP particles and binder. Appropriate combinations of matrix variables result in a scenario of extinction of the matrix when tested alone in an intermediate pressure range. This mid-pressure extinction of the matrix is related to the likelihood of plateau or mesa burning rate trends for propellant formulations that contain such matrix compositions.

High-speed combustion photography and examination of quenched surfaces of such matrices, and propellants and sandwiches containing those matrices, in the pressure range across the burn/no-burn boundaries of the matrices, reveal the onset of non-uniform burning as the burn/no-burn boundary is approached. The non-uniform burning is accompanied by binder melt flow and a local re-distribution of the fine AP particles along non-planar patterns on the burning surface. These processes cause the pyrolysis behavior of the matrixes to be unstable, and form the basis for intermittent burning of the matrix areas in the propellant and sandwich samples.

Acknowledgments

This work was supported by a grant from the US Office of Naval Research (N00014-99-1-1055) with Dr. Judah Goldwasser as technical monitor.

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