

# Aluminum Agglomeration in Solid-Propellant Combustion

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Results of a series of tests on AP/aluminum/PBAN propellants, with particular attention to bimodal AP particle size distribution, show the effect of particle size of fine AP and the concentration of fine AP on aluminum agglomeration and ignition. Results are interpreted in terms of the distribution of aluminum in propellant microstructure and the proximity of AP-binder flamelets to precipitate ignition of the accumulating aluminum.

## Nomenclature

Al	= aluminum
AP	= ammonium perchlorate
CA	= chemical analysis
HTPB	= hydroxy terminated polybutadiene
KLLEF	= kinetically limited leading-edge flame
PBAN	= polybutadiene acrylic acid acrylonitrile

## Introduction and Theory

**A**GGLOMERATION of aluminum on the burning surface of composite propellants has been the object of a number of studies<sup>1-5</sup> because of the effect of agglomeration on a variety of practical aspects of motor performance. On the basis of these studies, it has become evident that agglomeration is a result of a complex sequence of conditions and processes. These include the initial distribution of the aluminum in the propellant matrix; its tendency to adhere and concentrate on the burning surface without immediate ignition; the sintering of the concentrated particles into an "accumulate" that will coalesce rather than disintegrate when further heated; and, finally, ignition coalescence of the accumulate, precipitated by the attainment of certain conditions of the combustion environment and heating history of the accumulate. This sequential picture of agglomeration behavior has evolved from a wide variety of experimental studies. The present report concerns an effort to test a qualitative theory of the agglomeration sequence<sup>6-9</sup> by a series of tests with AP-hydrocarbon binder propellants with bimodal oxidizer particle size distributions. Using a relatively coarse particle size (400  $\mu\text{m}$ ) for the coarse fraction of the AP, the particle size of the fine fraction was varied and the agglomeration behavior observed. The mechanistic arguments under evaluation are those in Refs. 6-10, particularized for this type of propellant as described below.

The character of the array of accumulating aluminum particles on the burning surface is dependent on the distribution of aluminum in the propellant matrix and the length of time the accumulation proceeds without interruption. When the particles of the fine fraction of the AP

are large enough to substantially fill the voids in the packing array of the coarse AP, the (small) aluminum particles are thinly dispersed in the propellant matrix and hence, if they accumulate on the burning surface, they must do so in a scattered or filigree fashion (Fig. 1). When the fine fraction of AP consists of fine particles, the voids in the packing pattern of the coarse AP, often called "pockets,"<sup>11</sup> are filled with a mixture of about equal parts of AP, Al, and binder. The nature of the surface accumulation then depends on the behavior of this mixture (referred to by Schmidt<sup>2</sup> as the "pocket" propellant) in the environment of the overall combustion zone. The emerging aluminum may either depart the surface one particle at a time (ignited or unignited) or may concentrate up to and beyond the contents of individual voids of the packing pattern (pockets) of the coarse AP.

With high-solids AP composite propellants it is observed<sup>5,6,10</sup> that agglomerates tend to form from surface accumulations on the burning surface concurrently with ignition and detachment from the surface. In view of the high temperature of the burning agglomerates, it is not surprising that they tend to detach from the burning surface. This situation places particular importance on the question of what precipitates ignition. In this respect, it is notable that aluminum does not ignite readily in combustion of binary AP/Al systems,<sup>5-10</sup> and is often observed unignited on the surface of AP particles in burning propellants<sup>4-10</sup> and on the surface of AP laminae of burning sandwiches.<sup>1-10,12</sup> From this and other evidence it has been concluded<sup>6-7</sup> that ignition of accumulated aluminum is precipitated by exposure to AP-binder flamelets, and that one must address the conditions for such flamelets in order to anticipate conditions for ignition/agglomeration of accumulated aluminum.

Recent studies of combustion of AP-hydrocarbon polymer sandwiches<sup>13</sup> have revealed a microflame stability limit that has a bearing on the proximity of the AP-binder flames that are required to ignite the accumulating aluminum. Because of its importance to the present study, it is outlined here as it pertains to AP-binder systems such as composite propellants. Figure 2 shows the flow of oxidizer vapors from three oxidizer particles and the adjoining binder. From the boundaries of each of the exposed oxidizer surfaces, there is a stoichiometric surface extending outward in the mixing flow. In the fuel-rich pocket regions of the propellants of the present studies, the stoichiometric surfaces above the small oxidizer particles will close over the oxidizer surfaces in a "stoichiometric tip" as shown in the figure. The sandwich burning results indicate that, for large particles and/or high pressures, flamelets will be established on these stoichiometric surfaces, but their presence and proximity to the surface is determined by a kinetically limited leading-edge flame (KLLEF), the location of which is pressure dependent. Either small particle size or low pressure will cause the KLLEF position to approach the

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tip of the stoichiometric surface; continued reduction of the particle size or pressure will result in such fuel dilution of the oxidizer flow as to cause the KLEF to retract abruptly to a more remote location.<sup>13</sup> This in turn would be expected to result in much less favorable conditions for early ignition of the accumulating aluminum and hence more protracted accumulation and larger agglomerates. This would be particularly so in the pocket regions of the surface array, where fine oxidizer particles are present in a local matrix that is relatively fuel rich. In terms of a propellant with a bimodal oxidizer size distribution, this translates into a strong dependence of agglomerate size on the pressure and particle size of the fine AP. The available evidence on KLEF quench limits suggested that the region of strong dependence of agglomerate size would occur in conventional range of pressures and fine AP particle sizes, readily verified by a matrix of tests with suitable ranges of oxidizer size and test pressures.

In a less fuel-rich pocket propellant, attained by increasing the content of fine AP in the propellant, the fuel dilution of the oxidizer flow from the individual oxidizer particle may be reduced enough to permit particles to acquire their own kinetically limited flames under more adverse conditions.

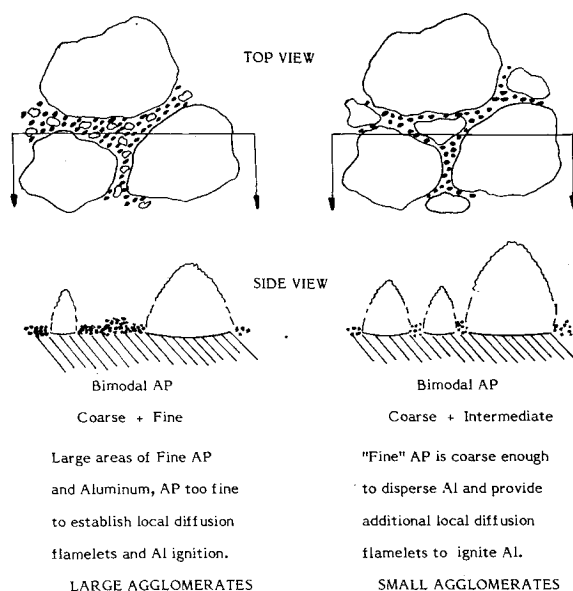


Fig. 1 Illustration of how oxidizer particle size affects dispersion of aluminum and proximity to ignition sites on the burning surface.

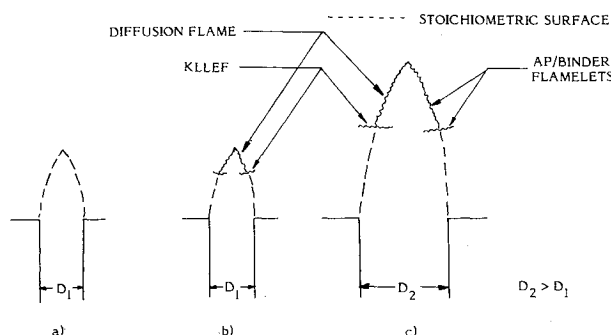


Fig. 2 Flames over fine oxidizer particles: a) fine, fine oxidizer particle at low pressure  $p_1$  (fails to establish KLEF on stoichiometric surface); b) fine, fine oxidizer particle at high pressure  $p_2$  (KLEF, hence AP/binder flame established on stoichiometric surface of particle); c) coarse, fine oxidizer particle at low pressure  $p_1$  (KLEF, hence AP/binder flame established on stoichiometric surface of the particle).

These hot flames may thus aid the ignition of aluminum close to the burning surface before prolonged accumulation, leading to less accumulation before agglomeration.

It should be stressed that the ignition of accumulated aluminum is viewed as a local inflammation induced by a local oxidizer-binder flamelet.<sup>5-10</sup> The flamelet is assumed to have regions at or near stoichiometric flame temperature as is common for diffusion flames. The investigation seeks to test the *qualitative* concept that agglomeration is delayed under conditions that are not conducive to the establishment of the near-surface flamelets associated with individual fine AP particles. Unlike the concepts utilized by Cohen,<sup>14</sup> the present argument concerns the question of the presence or absence of these particle flames, not the equilibrium temperature of the local pocket mixture.

## Experimental Procedures

### Exploratory Tests

Tests consisted of exploratory work by combustion photography and plume quench-particle collection on a series of propellants from an earlier program by Caveny and Brundige<sup>4</sup> and by combustion photography on a series of propellants made under laboratory conditions. On the basis of results of the exploratory tests, a systematic investigation was carried out by plume quench-particle collection tests on a series of propellants prepared with the facilities available at the Georgia Institute of Technology combustion laboratory.

### Propellant Details

The Thiokol propellants tested in the exploratory investigation were trimodal AP propellants with blends of 400  $\mu\text{m}$ /200  $\mu\text{m}$ /fine fraction AP in the ratio of 60/30/10, 11% HTPB binder, and 18% H-30 aluminum. The laboratory-made propellants on which exploratory combustion photography analysis was conducted were made as described in the following section. The formulation of the propellant was the same as Thiokol propellant except the binder used was PBAN.

The formulations on which results are reported here were prepared by hand mixing of 4 g samples. The different ingredients that make up a given propellant formulation were weighed carefully into a cup and mixed thoroughly, assuring the wetting of all the solid constituents by the wet binder. The mixture was then placed under vacuum to remove the trapped air. After 15 min, the mixture was pressed into a cylindrical disk in a hydraulic press at 4.4 MPa for 10 min. The pressed sample was then placed in an oven at 72°C for seven days to cure the sample.

The oxidizer particles used in this investigation were 99.5% high-purity Kerr-McGee AP. The coarse oxidizer was of nominal diameter 390  $\mu\text{m}$ . Particles were rounded, but not spherical. The nominal sizes used for the finer oxidizer were 17.5, 49, 82.5, 107.5, 137.5, and 196  $\mu\text{m}$ . All the AP particles were prepared by grinding the as-received AP and screening as per Table 1. All samples used particular nominal size AP particles from the same batch. The nominal size refers to the median of the sieve sizes given in Table 1.

The hydrocarbon binder used was PBAN (polybutadiene acrylic acid acrylonitrile). Mass fraction in the formulation

Table 1 Screened ammonium perchlorate utilized in this program

Sample No.	Size range, $\mu\text{m}$	Nominal size, $\mu\text{m}$
1	0-37	17.5
2	45-53	49
3	75-90	82.5
4	90-125	107.5
5	125-150	137.5
6	180-212	196.0
7	355-425	390

was 11%. The aluminum particles used included Valley Met H-5, H-15, and H-30 aluminum particles with nominal sizes 5, 15, and 30  $\mu\text{m}$ , respectively. All propellants were prepared with 18% aluminum, 11% binder, and ratio of fine-to-coarse AP of 20/80, unless otherwise mentioned.

#### Sample Collection and Analysis

Test samples were cylinders of 2.54 cm diameter and 0.75 cm thickness. They were fitted in quartz tubes, mounted with the burning surface downward. The experimental setup is shown in Fig. 3. The hole in the quartz tube equalizes the pressure inside and outside the tube. Combustion products and burning agglomerates flowed downward for 1.5 cm to an ethanol pool in which the agglomerates were quenched and collected. Immediately after each test the sample was washed repeatedly to remove  $\text{Al}_2\text{O}_3$  smoke and HCl.

The collected particles were then dried and screened into particles in different size ranges using sieves. The particles collected in each size range were weighed to a tenth of a milligram. The average size of all particles larger than 49  $\mu\text{m}$  was designated  $\bar{d}_{49}$  and was calculated by the following equation for all size ranges above 49  $\mu\text{m}$ .

$$\bar{d}_{49} = \frac{\sum (\text{wt. of particles in a given size range}) \times (\text{midrange diameter})}{\sum \text{wt. of particles in given size range over all size ranges}}$$

$\bar{d}_{49}$  is then plotted against the desired variable.

The variations of formulation and test conditions for the main test series in this study are summarized in Fig. 4. The large circle represents a reference state, about which changes were made in the pressure, the particle size of the fine component of AP, the particle size of aluminum, and the ratio of coarse AP mass to fine AP mass. The two circles above the large one represent secondary reference states about which the indicated changes were made. These secondary reference states were the same as the primary one, except for size of the fine AP particles.

#### Combustion Efficiency

A measure of the combustion rate of aluminum in the combustion plume was obtained by measuring the amount of unoxidized aluminum in the collected sample. The amount of free aluminum (the term "free aluminum" denotes that aluminum which can be brought into a solution by the action

of dilute HCl) in the collected sample was quantitatively determined by a complexometric titration of disodium ethylenediamine tetraacetate (EDTA) using xylenol orange as the indicator. The analysis is based on that of A.I. Busev et al. as outlined in Ref. 15. The method is highly accurate and precise. The ratio of the amount of free aluminum thus determined to the amount of original aluminum in the unburned sample was then calculated. The test conditions investigated are denoted by a CA in Fig. 4.

## Results

#### Exploratory Investigation

##### Combustion Photography of Thiokol Propellants

Combustion photography of two propellants provided by Caveny and Brundige (propellants 1780-1 and 1780-14 of Ref. 4, containing fine components of AP of 12 and 90  $\mu\text{m}$ , respectively) revealed that agglomeration at a pressure of 1.38 MPa (200 psi) was considerably less with 90  $\mu\text{m}$  fine AP than with 12  $\mu\text{m}$  fine AP. The brightness of the field of view was also greater with 90  $\mu\text{m}$  AP, indicating a larger portion of smaller agglomerates in the gas phase.

##### Combustion Photography of Laboratory-Made Propellants

Since propellants with intermediate-size fine AP particles were not available from the source of Ref. 4, a series of propellants with the same formulations, except for variation in fine AP particle size, were made and tested at 1.38 MPa in the exploratory investigation. The series included propellants with 10, 49, 82.5, and 107.5  $\mu\text{m}$  nominal-size fine AP. Analysis of combustion photography of these propellants indicated that the agglomeration decreased only moderately in going from 10 to 49  $\mu\text{m}$  AP; but the reduction in agglomeration was substantial on shifting to 82.5  $\mu\text{m}$  fine AP. This shift was accompanied by a major increase in the brightness of the combustion field of view. However, further increasing the fine AP particle size to 107.5  $\mu\text{m}$  increased agglomeration somewhat.

#### Plume Quench Test Results

Exploratory work of plume quench tests on propellants 1780-1 and 1780-14 of Ref. 4 revealed that smaller agglomerates were formed with the formulation containing 90  $\mu\text{m}$  AP, confirming the observations by combustion photography. The results are tabulated in Table 2. Figure 5 gives a size distribution of the agglomerates for the two propellants at 0.69 MPa (100 psi). This clearly indicates that smaller agglomerates are formed in larger quantities with larger fine AP in the propellant.

This trend in agglomerate size with oxidizer particle size is evident in the results of Brundige,<sup>4</sup> but no systematic experimental study has been made or correlated with any detailed mechanistic explanation.

#### Results of Systematic Studies

##### Effect of Pressure

The results of the plume quench tests to evaluate the effect of pressure on agglomerate size with different formulations investigated in the present study are summarized in Fig. 6. Here the ratio of the coarse to fine AP was maintained at 8/2. The values of the mass average diameter  $\bar{d}_{49}$  of the collected particles are plotted as a function of pressure for different fine AP particle sizes. These results indicate that the mean diameter  $\bar{d}_{49}$  drops off sharply near 2.1 MPa (304 psi), when the fine component of the oxidizer blend in the propellant is 17.5  $\mu\text{m}$  AP. This dropoff in agglomerate size shifts to a lower pressure (1.38 MPa) with 49  $\mu\text{m}$  AP, and to a still lower pressure (0.4 MPa) with a coarser fine AP of 82.5  $\mu\text{m}$  AP particles in the propellant. The results also show that, in general,  $\bar{d}_{49}$  is smaller for coarser fine AP particle propellants over the entire pressure range tested. An exception resulted in

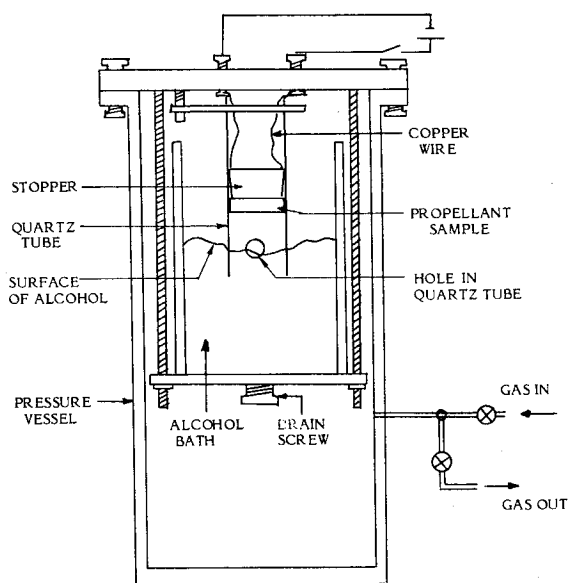


Fig. 3 Experimental setup for plume quench test.

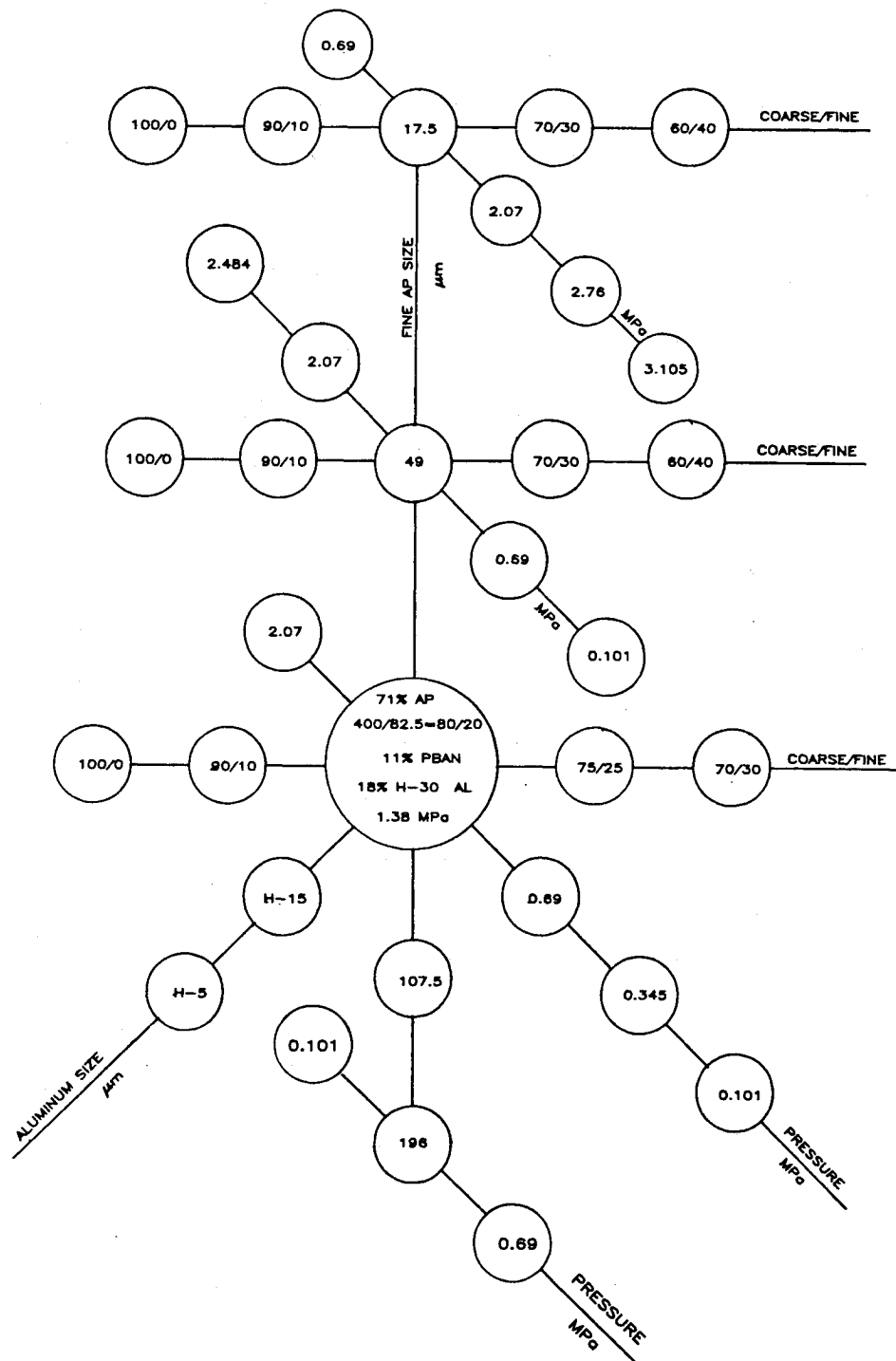


Fig. 4 Conditions for plume quench tests (CA indicates chemical analysis of the collected sample).

the replacement of the fine fraction of AP particles with the largest size AP (196  $\mu\text{m}$ ) of the series. This results in a shift in the above trends (in Fig. 6) to a larger agglomerate size.

#### Effect of Percent Fine AP in the Propellant

This effect was determined by tests at 1.38 MPa (200 psi), using three different sizes for the fine AP particles (Fig. 7). With 17.5  $\mu\text{m}$  AP, it is observed that the  $\bar{d}_{49}$  drops off sharply near 25% fine AP in the propellant. This dropoff point shifts to a lower fine AP content (15%) in propellant with 49  $\mu\text{m}$  fine AP, and it totally disappeared with 82.5  $\mu\text{m}$  AP. In

addition, it is also noted that with 82.5  $\mu\text{m}$  AP, increasing the fine AP content beyond 15% fine AP in the propellant resulted in increase in size of  $\bar{d}_{49}$ .

#### Effect of Aluminum Particle Size

The results of decrease in aluminum particle size in the formulation containing 390 and 90  $\mu\text{m}$  AP in the ratio of 8/2 are plotted in Fig. 8. The tests were run at two different pressures of 0.345 and 1.38 MPa. The results indicate that the largest agglomerate size occurred with H-15 aluminum at both the pressures.

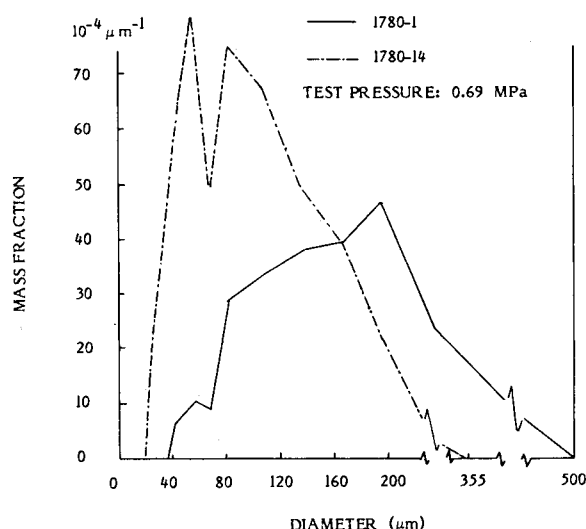


Fig. 5 Comparison of two Thiokol propellants for agglomerate distribution.

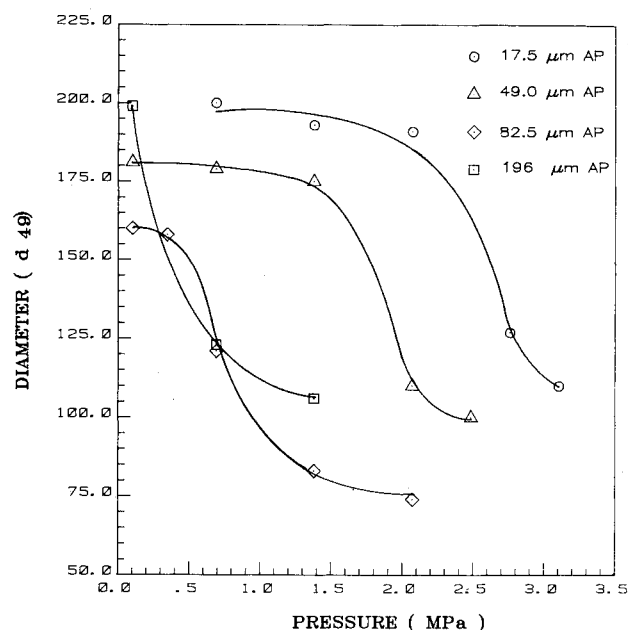


Fig. 6 Effect of pressure on mass average agglomerate size  $\bar{d}_{49}$  for propellants with coarse/fine AP of ratio 8/2.

Table 2 Average diameter of agglomerates from quench tests on Thiokol propellants (mass average of particles  $> 49 \mu\text{m}$ )

Pressure	Propellant	
	1780-1 (12 $\mu\text{m}$ AP)	1780-14 (90 $\mu\text{m}$ AP)
0.69 MPa	183	100
Atm.	195	109

#### Relation of Agglomerate Size to Combustion Efficiency

The ratio of free aluminum in the sample collected in the plume quench tests to the original aluminum in the unburned sample was plotted against the average diameter  $\bar{d}_{49}$  of the corresponding quenched sample in Fig. 9. The illustration indicates that there is a one-to-one correspondence between the size and the fraction of free aluminum. The fraction of free aluminum in the propellant increased linearly with increase in average diameter  $\bar{d}_{49}$ .

#### Discussion

The results in Figs. 6 and 7 show the dependence of average agglomerate size on pressure, fine AP particle size, and percent of fine AP in the propellant that was projected on the basis of the mechanistic argument in the Introduction. Specifically, it was argued that:

1) At low pressure, the fine AP particles of the pocket propellant fail to be established near the surface flamelets, resulting in unfavorable conditions for the ignition of the accumulating aluminum and hence formation of large agglomerates. The results in Fig. 6 (low pressure) show the relatively large agglomerate sizes.

2) As pressure increased, the fine AP particles would establish individual flamelets conducive to aluminum ignition, with a corresponding reduction in accumulation and agglomerate size. This trend in agglomerate size with pressure is conspicuous in Fig. 6.

3) When the fine AP particle size is small, establishment of individual AP-binder flamelets occurs only at high pressures, so that the drop in agglomerate size with increasing pressure is delayed to a higher pressure (Fig. 6).

4) Increasing the fine AP content in the propellant reduces the fuel excess in the environment of the fine AP particles in the pocket propellant and draws the diffusion flamelets closer to the burning surface. This increases the opportunity for

ignition of the aluminum accumulates, with correspondingly decreased agglomeration, as indicated by the results in Fig. 7. This trend in agglomerate size is not only manifested, but also depends upon AP particle size in a manner consistent with the mechanistic argument in the Introduction and the section on Theory.

While it is tempting to describe the trends of agglomerate size simply in terms of degree of accumulation in each pocket, it should be noted that the agglomerate sizes were often larger than would result from combinations of all the aluminum in one pocket. This observation draws attention to the fact that the concept of "pocket" breaks down in foreseeable ways, which are applicable to most practical propellants. Thus, the pockets defined by the packing array of the coarse AP are well connected, i.e., the combined volume of the fine AP, aluminum, and binder is large enough so that the coarse AP particles are not closely packed on each other and packing voids are hence well connected. In this respect, an opportunity is provided for "superagglomerates," if fine AP-binder flamelets are not present in the fuel-rich pocket propellant to prevent prolonged accumulation.

The results of changes in aluminum particle size (Fig. 8) highlighted the importance of the distribution of aluminum in the packing array of the propellant. In general, if an aluminum particle is large enough, it will take its place in the oxidizer packing array, isolated from other aluminum particles. Under such conditions the aluminum particles have little opportunity to concentrate before ignition and hence do not agglomerate (although such particles may be comparable in size to conventional agglomerates). When the aluminum particle size is reduced sufficiently, it will form a relatively continuous array around and among the larger oxidizer particles. Surface concentration can then form a fairly connected filigree. When the particle size of the fine component of the AP blend is small, this gives the "superagglomerate" situation described above, with pocket concentrations interconnected by filigree (when the ignition conditions are unfavorable). When the particle size of the fine component of the AP is large enough, as in the samples corresponding to the results in Fig. 8, the aluminum array is limited to filigree. Large agglomerates may be formed with these formulations under unfavorable ignition conditions (e.g., low pressure). Otherwise, small agglomerates are

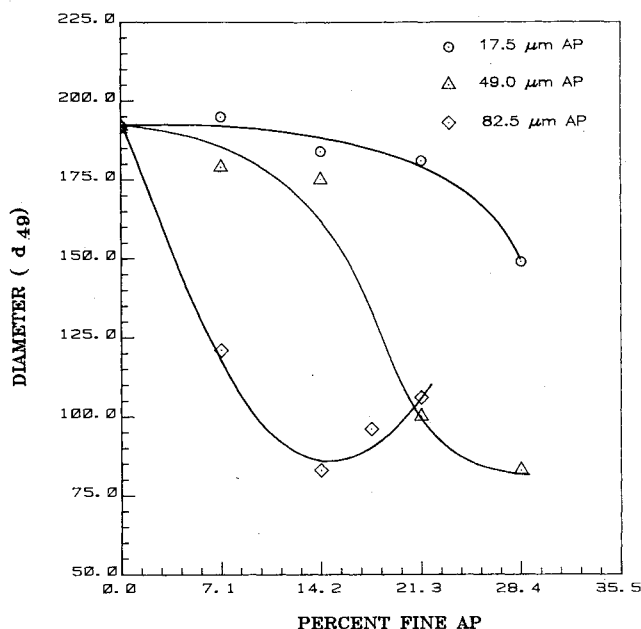


Fig. 7 Effect of percent fine AP in propellant on  $\bar{d}_{49}$  at 1.38 MPa.

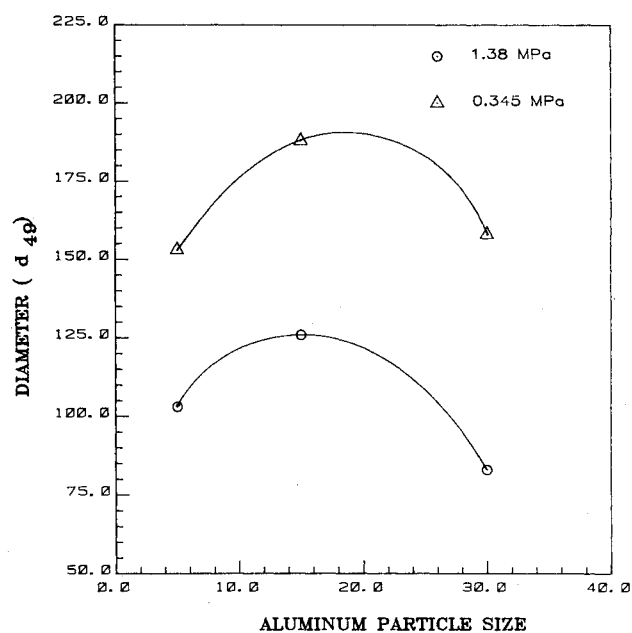


Fig. 8 Effect of aluminum particle size on  $\bar{d}_{49}$  at 1.38 MPa. The coarse (400  $\mu\text{m}$ ) and fine (82.5  $\mu\text{m}$ ) AP particles were of ratio 8/2 in all formulations.

formed since the aluminum particles are well situated for exposure to high-temperature flamelets. This is particularly so if the aluminum particles are very fine, because the resulting filigrees have very high surface areas (more easily heated and more reactive). These effects are reflected in the trends of agglomerate size vs aluminum particle size in Fig. 8. Large aluminum particles yield relatively small agglomerates because of the isolation of the aluminum particles from each other (with the relatively coarse fine fraction of AP used for the tests in Fig. 8). Intermediate-size aluminum yields relatively large agglomerates because interconnected filigrees are possible; the more favorable ignition conditions at higher pressure limit the size of the agglomerates. Fine aluminum behaves similarly to the intermediate size, but forms smaller agglomerates because it ignites more easily.

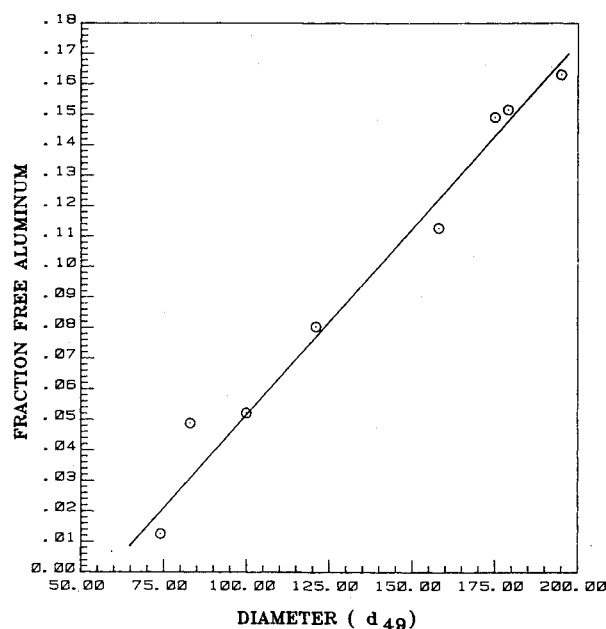


Fig. 9 Fraction of free aluminum in the collected sample vs mass average agglomerate size  $\bar{d}_{49}$ .

In the above description of the effect of aluminum particle size, it should be noted that a complete departure from the "pocket" concept of agglomeration was necessary to explain certain results. The occurrence of "superagglomerates" forces the consideration of the connective structures of aluminized binder between pockets and of the conditions under which such structures will produce filigree bridges of aluminum between pockets. This phenomenon of filigree bridging is even more important in formulations of relatively large size for the fine component. Under these conditions, the packing voids of the coarse component are substantially filled by individual "fine" AP particles. Although this effectively eliminates pockets, agglomerates continue to be substantial (Fig. 6), especially under unfavorable ignition conditions (low pressure). Thus, it is evident that filigree formation is a necessary consideration in modeling agglomerate formation and in explaining the trend of results in the present studies when either the AP particle size combination minimizes pocket concentrations of aluminum or unfavorable ignition conditions permit filigree formation between pockets. It is important to note that most commercial propellants have high-solids loading that is achieved by careful particle size blending. With such propellants the filigree formation process is necessarily dominant over the formation of pocket concentrations, because pockets have been eliminated by the particle blending process. The bimodal formulations used in the present study served well to demonstrate the mechanisms of agglomerate formation, but are otherwise of direct practical interest only in special applications requiring the unique combustion characteristics of such propellants.

In summary, the experimental results are explained in terms of:

- 1) Retention and concentration of aluminum on the burning surface in arrays dictated by the particle size combinations of ingredients and resulting distributions of aluminum in the propellant microstructure.

- 2) A limit on the concentration determined by the exposure to oxidizer-binder flamelets that induce ignition and detachment from the burning surface.

- 3) A criterion for proximity of oxidizer-binder flamelets that involves the conditions of AP particle size and pressure necessary for such flamelets to form on individual AP particles.

### Acknowledgment

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## ENTRY HEATING AND THERMAL PROTECTION—v. 69

## HEAT TRANSFER, THERMAL CONTROL, AND HEAT PIPES—v. 70

*Edited by Walter B. Olstad, NASA Headquarters*

The era of space exploration and utilization that we are witnessing today could not have become reality without a host of evolutionary and even revolutionary advances in many technical areas. Thermophysics is certainly no exception. In fact, the interdisciplinary field of thermophysics plays a significant role in the life cycle of all space missions from launch, through operation in the space environment, to entry into the atmosphere of Earth or one of Earth's planetary neighbors. Thermal control has been and remains a prime design concern for all spacecraft. Although many noteworthy advances in thermal control technology can be cited, such as advanced thermal coatings, louvered space radiators, low-temperature phase-change material packages, heat pipes and thermal diodes, and computational thermal analysis techniques, new and more challenging problems continue to arise. The prospects are for increased, not diminished, demands on the skill and ingenuity of the thermal control engineer and for continued advancement in those fundamental discipline areas upon which he relies. It is hoped that these volumes will be useful references for those working in these fields who may wish to bring themselves up-to-date in the applications to spacecraft and a guide and inspiration to those who, in the future, will be faced with new and, as yet, unknown design challenges.

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