A Pocket Model for Aluminum Agglomeration in Composite Propellants

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A simplified model is derived to estimate the fraction of aluminum powder that will form agglomerates at the surface of deflagrating composite propellants. The basic idea is that the fraction agglomerated depends upon the amount of aluminum that melts within effective binder pocket volumes. The thrust of the analysis is the determination of such volumes as a function of ingredient particle sizes. The objective is to reduce pocket sizes as a means to reduce the extent of agglomeration. The effective pocket depends upon the ability of oxidizer particles to segregate aluminum particles and provide a local temperature environment sufficient to ignite the aluminum. Model results are discussed in the light of data showing effects of propellant formulation variables and pressure.

Nomenclature

A = fraction of aluminum agglomeration
c = specific heat
D = particle diameter
f = fraction of aluminum melted
K = constant (1/150 for α a fraction and V_p in μm^3)
k = thermal conductivity
N = number of particles per unit volume of defined propellant
N' = number of particles situated within a defined pocket volume
(OF) = ratio of AP/binder weight fractions in a defined propellant
r = burn rate
T = temperature
V = pocket volume
V_p = effective pocket volume
α = weight fraction
α' = weight fraction in a defined propellant
Δ = particle spacing
κ = thermal diffusivity
ρ = density

Subscripts

1 = fine oxidizer component
2 = intermediate oxidizer component
3 = coarse oxidizer component
0 = standard condition
Ag = agglomerate
Al = aluminum
f = binder
j = oxidizer component (general)
m = melting of aluminum
ox = oxidizer in general
s = propellant surface

Introduction

IT is well known that aluminum powder has a tendency to agglomerate at the burning surface of solid propellants.[1] Potential problems created by excessive agglomeration in the operation of solid rocket motors include reductions in combustion, nozzle and expulsion efficiencies, variations in the burn rate of spin-stabilized motors, and a reduction in the particulate damping of acoustic waves. Several experimental programs have been conducted to learn propellant formulation effects upon aluminum agglomeration.[2-4] As a result, it is known that formulating polybutadiene propellants to achieve higher energy and lower burn rate has the tendency to promote more extensive agglomeration [for example, increased aluminum content and coarse ammonium perchlorate (AP) content]. An analytical model that would explain these effects would be useful to guide propellant development and tradeoff studies for energy/burn rate optimization.

Aluminum agglomeration has also been a subject of more fundamental research.[5-7] It is observed to involve a sequence of competing dynamic processes that are so complex and frought with unknowns as to defy analytical modeling on that level of detail. Such will not be attempted here. On the other hand, a relatively simple concept has provided the basis for rudimentary modeling[8,9] which has been useful to describe gross effects of AP particle size. Given the present physical situation but the need for propellant guidance, it is this sort of modeling which will be developed further in this paper.

The underlying concept has been referred to as the "pocket model." The spaces between AP particles are visualized as isolated pockets surrounded by the AP. These pockets consist of binder and contained aluminum. The basic idea is that agglomerates form from the clusters of aluminum particles residing within these pockets. How they are formed is not addressed, but the isolation is viewed as a limitation on the extent of agglomeration. It follows that the smaller the pockets, the less the size and extent of agglomerates. Since pocket volume decreases with decreasing AP particle size, a principal conclusion is that reducing AP size reduces agglomeration. This trend is in complete agreement with data for simple monomodal propellants in which the AP size is larger than the aluminum size. Such propellants best approximate the physical picture of this concept. However, in the case of practical propellants, there are additional dimensions to the particle size effect and the trends in the data are less systematic. Practical propellants employ blends of particle sizes, with a portion of the AP often smaller than the aluminum, and thereby complicate the physical picture of a pocket. The modeling must somehow address this complication. An approach for doing so is the subject of this paper.

Summary: The Effective Pocket

The "effective pocket" is defined as that volume which

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ultimately operates to limit the formation of an agglomerate. It is hypothesized that the fraction agglomerated is proportional to the amount of aluminum that melts within the effective pocket volume. It is assumed that metal particles emerging from the propellant unmelted do not participate in the agglomeration, whereas all other metal does participate. The fraction melted is determined by a particle heating analysis in the environment of the passing thermal wave, as described later in the paper. The effective pocket is then determined from geometric pocket volumes which are described as follows.

It is assumed that the solids in the propellant may be represented by a uniform distribution of spherical particles in a cubic array. From this arrangement, it is possible to define n geometric pocket volumes where n is the number of AP particle size groups ($n=2$, bimodal, etc.). For this purpose, it is assumed that the AP is present in discrete sizes (represented by the weight mean size for each group) such that the arrays of smaller particles fit within the arrays of larger particles. The smallest geometric pocket is defined by the array of the finest AP; the largest geometric pocket is defined by the array of the coarsest AP. For a trimodal distribution, there will be a geometric pocket of an intermediate size. The bimodal distribution is illustrated in Fig. 1. It has two geometric pocket volumes, one for the coarse AP and the other for the fine AP. The coarse pocket volume is that volume bounded by the cubic array of eight coarse particles (shown in the two-dimensional figure as shaded area within four particles). In general, it will contain many fine pocket volumes and will statistically contain representatives of all aluminum sizes present. The fine pocket volume is similarly defined; only one is shown in the illustration. It contains binder, but may or may not contain all of the aluminum sizes present. Having defined the geometric pocket volumes, the effective pocket is determined from criteria summarized as follows.

The effective pocket is determined as the minimum geometric pocket size in which the corresponding AP is able to both "encapsulate" and "locally ignite" the aluminum. By encapsulation, in its operational sense, it is meant the isolation of the aluminum contained in the pocket from aluminum in neighboring pockets. Encapsulation is determined by comparing the size distributions of the AP and aluminum, as described more fully later in the paper. To the extent that aluminum is not encapsulated by the smallest pocket, it must be allocated to the next larger pocket size where it will participate in more extensive agglomeration. By "local ignition," it is meant the ability of the AP/binder flame on the local pocket scale to provide the aluminum ignition temperature. This criterion proceeds on the theory that ignition precipitates detachment of the agglomerate from the propellant surface,[10] and from observations[3] that improved metal ignition correlates with reduced agglomeration. Determination of the local flame temperature is described later in the paper. If the local flame temperature corresponding to the smallest pocket is too low, the aluminum must be allocated to the next larger pocket. If both criteria (ignition temperature and encapsulation) are satisfied by the smallest (or "first-level") pocket, agglomeration is limited to each of these pockets from which ignited agglomerates emerge. If not, the aluminum continues across the first-level pockets to the next larger (or "second-level") pockets, etc.

The model does not consider the phenomena of particle accumulation (contact, adhesion), retention on the propellant surface, ignition, spheroidization, detachment, etc.—that which comprises the process of "agglomeration"—in any specific sense. It is taken for granted that these will occur, and it is postulated that the pocket volumes set limits on the extent of agglomeration according to the model criteria. It is assumed that the missing elements may be lumped into the proportionality between the fraction agglomerated and the effective pocket size; as a result, the quantitative aspects would be expected to change with binder ingredients and oxidizer type.

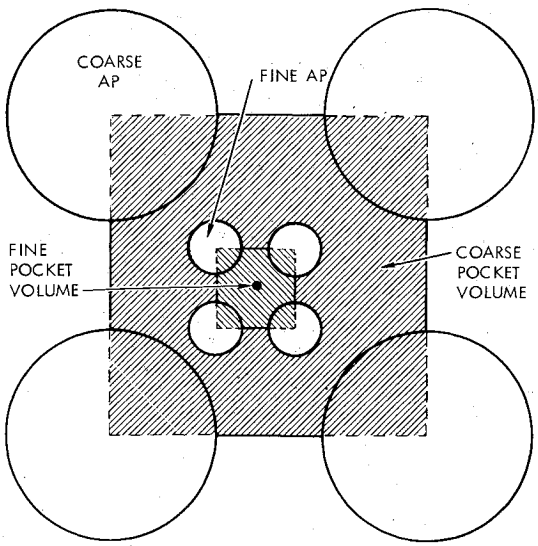

Fig. 1 Pocket model concept.

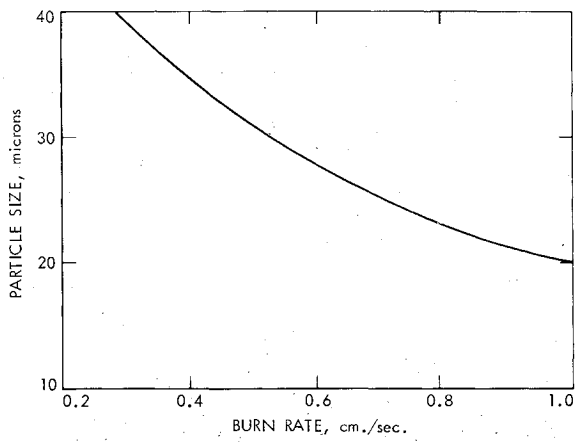

Fig. 2 Aluminum particle size that can melt as a function of burn rate.

The model has been applied to experimental conditions covering effects of AP size distribution parameters, aluminum particle size and concentration, and pressure for series of polybutadiene (HTPB) propellants. Results are in qualitative agreement with motion picture and particle collection data. Quantitative comparisons would not be meaningful because the model does not calculate agglomerate size distributions, and because the experimental definition of "agglomerates" in collected material is arbitrary and not standardized. However, the ability to predict trends is useful.

Analysis

Calculation of Geometric Pocket Volumes

The analysis is presented for a trimodal propellant. Application to a bimodal propellant will be obvious from the method. The number of coarse particles per unit volume of propellant is equal to the volume fraction of the coarse AP divided by the volume of one coarse particle. This may be expressed as

$$N_3 = \frac{6\alpha_3/\pi D_3^3}{\alpha_1+\alpha_2+\alpha_3+\alpha_{Al}(\rho_{ox}/\rho_{Al})+\alpha_f(\rho_{ox}/\rho_f)} \quad (1)$$

The spacing between the coarse particles is then

$$\Delta_3 = (1-N_3^{1/3}D_3)/N_3^{1/3} \quad (2)$$

It follows that the coarse pocket volume is

$$V_3 = (\Delta_3 + D_3)^3 - (\pi/6) D_3^3 \tag{3}$$

The first term on the right-hand side is the cube whose corners are at the centers of the eight bounding spheres, and the second term represents the eight ⅛ spherical segments of volume occupied by the bounding spheres.

The space occupied by the coarse particles is unavailable to all of the other propellant ingredients. In other words, one has to think of a new propellant which omits the coarse particles in order to determine the intermediate pocket volumes which are situated within. By analogy to Eq. (1), the number of intermediate size particles per unit volume of this fictitious propellant is

$$N_2 = \frac{6\alpha_2/\pi D_2^3}{\alpha_1 + \alpha_2 + \alpha_{Al}(\rho_{ox}/\rho_{Al}) + \alpha_f(\rho_{ox}/\rho_f)} \tag{4}$$

Thus the number of intermediate size particles within a coarse pocket volume is

$$N_2' = N_2 V_3 \tag{5}$$

The spacing between the intermediate size particles in the coarse pocket is equal to the width of the coarse pocket less the spacings occupied within by the intermediate particles. Thus the analogy to Eq. (2) is

$$\Delta_2 = \frac{(\Delta_3 + D_3) - (N_2')^{1/3} D_2}{(N_3')^{1/3}} \tag{6}$$

So the intermediate pocket volume is, analogous to Eq. (3)

$$V_2 = (\Delta_2 + D_2)^3 - (\pi/6) D_2^3 \tag{7}$$

By similar reasoning, the properties of the fine pocket volume are

$$N_1 = \frac{6\alpha_1/\pi D_1^3}{\alpha_1 + \alpha_{Al}(\rho_{ox}/\rho_{Al}) + \alpha_f(\rho_{ox}/\rho_f)} \tag{8}$$

$$N_1' = N_1 V_2 \tag{9}$$

$$\Delta_1 = \frac{(\Delta_2 + D_2) - (N_1')^{1/3} D_1}{(N_1')^{1/3}} \tag{10}$$

$$V_1 = (\Delta_1 + D_1)^3 - (\pi/6) D_1^3 \tag{11}$$

Relation between Fraction Agglomerated and Pocket Volume

The basic hypothesis is that the fraction of aluminum agglomerated is proportional to the amount of aluminum that melts within an effective pocket volume. The aluminum that does not melt is assumed to leave the propellant unagglomerated. All of the melted aluminum is envisioned to accumulate and eventually be converted into agglomerates of equivalent total mass. The number of agglomerates that form, and their size distribution, are not addressed. A simple picture would envision the formation of one agglomerate from the mass of melted aluminum in each pocket, but that is probably not true. In any event, for a given size distribution function the weight mean agglomerate size will be proportional to the cube root of the mass of melted aluminum in the pocket,

$$D_{Ag} \sim (f\alpha_{Al} V_p)^{1/3} \tag{12}$$

Agglomerates, in the context of experimental determinations and potential problems, do not include all of the agglomerates in their literal sense but are limited to relatively large particles by informal definition.[3,4] Thus the "fraction agglomerated," as reported experimentally, is the fraction of aluminum that exceeds an arbitrary size as the result of agglomeration. On this basis, the fraction agglomerated has been measured to be proportional to the weight mean agglomerate size.[3] Then, from Eq. (12),

$$A = K(f\alpha_{Al} V_p)^{1/3} \tag{13}$$

The proportionality K is assumed to account for the unknowns of the problem such as the processes responsible for accumulation, the distribution of agglomerates formed initially, and the processes responsible for further growth at the propellant surface prior to ejection. For purposes of this analysis, the important thing is to achieve the qualitative dependence on the pocket size. At the same time, however, the scope of applicability will be limited to the generic type of composite propellants being considered here.

Fraction of Aluminum That Melts

The calculation is for the maximum aluminum particle size that will melt in response to heating by the passage of the

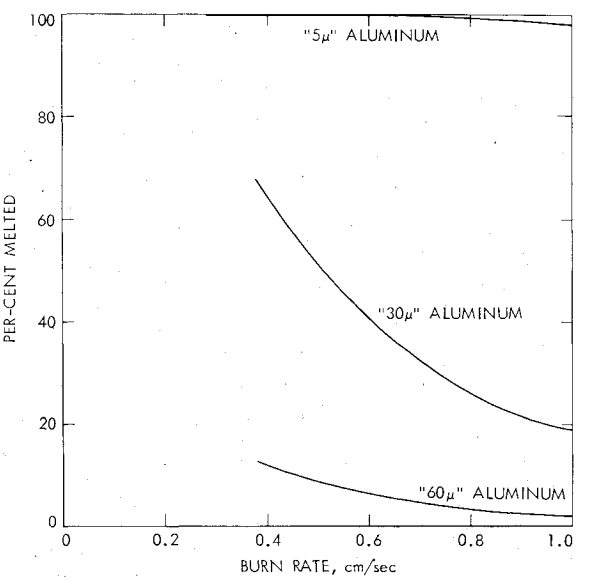

Fig. 3 Percent aluminum melted as a function of burn rate for three size distributions.

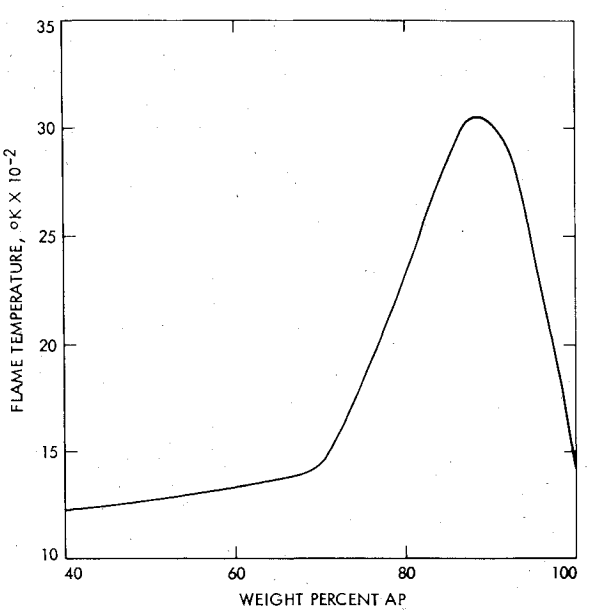

Fig. 4 Flame temperatures of AP/HTPB propellants.

thermal wave in the binder. That size, and all smaller sizes in the distribution, will melt under the conditions imposed. Given an aluminum size distribution, the fraction melted is thereby established. The analysis assumes an isothermal spherical particle and a large reservoir of heat stored in the binder. The very high thermal diffusivity of the aluminum enables it to be treated as a point heat sink in the thermal wave. The presence of the aluminum is assumed to have a negligible effect on the temperature rise and heat stored in the binder because its heat capacity is relatively low and its effect on propellant burn rate is generally small. It is also assumed that emergence of the top of the particle into the hot gas does not change the nature of the heating process. These assumptions simplify the analysis considerably. Errors introduced tend to be compensating so that the results obtained appear to be reasonable. The critical particle size is thus derived to be

$$D_{Al} = \left[\frac{12k_f\kappa_f}{\rho_{Al}c_{Al}r^2}\left(\frac{T_s - T_m}{T_m - T_0}\right)\right]^{1/2} \tag{14}$$

Equation (14) is plotted in Fig. 2. Burning rate is the key parameter determining the amount of aluminum that is melted upon arrival at the binder surface. For a burning rate of 0.5 cm/s, sizes smaller than about 30 μm will be melted. This represents half of the "30 μm" aluminum often used in propellants. The size (and amount) increase rapidly with decreasing burn rate.

Figure 3 shows the percent of aluminum that is melted vs burn rate for three aluminum size distributions of interest. Virtually all of the "5 μm" aluminum is melted at low burning rates. On the other hand, little of the "60 μm" aluminum is melted. It is interesting to note that, at very low burn rates, there is little benefit in going from 5 to 30 μm aluminum for the purpose of reducing f; and, at very high burn rates, there is little benefit in going from 30 to 60 μm aluminum. However, over the range plotted there does appear to be an advantage in using the "60 μm" aluminum.

Determination of Effective Pockets

General Criteria

Two criteria are posed for the determination of the effective pocket. The first is referred to as the "temperature criterion" and the second is referred to as the "encapsulation criterion." According to Eq. (13), it would be desirable that the effective pocket be formed by the finest AP in order to minimize agglomeration. It is postulated that both criteria must be satisfied in order for the fine AP to be able to provide an effective pocket. To the extent that the criteria are not satisfied, the aluminum must be assigned to the next larger pocket. In theory, the criteria would be successively applied to the larger pockets until one is eventually found that satisfies them. However, in cases of practical interest, it is found that the intermediate size AP will satisfy the criteria if the fine size fails to do so.

Temperature Criterion

It is postulated, as the first criterion, that the flame temperature associated with the fine AP pocket must exceed the ignition temperature of aluminum in order that the fine AP be able to form the effective pocket volume. If not, the aluminum cannot be ignited locally and so will agglomerate to the limit specified by the next larger pocket volume. It is assumed that termination of agglomeration in the pockets is contingent upon ignition, that ignition is contingent on temperature, and that this condition is satisfied when the local AP/binder flame temperature reaches an "ignition temperature."

The concept that binder is allocated among AP particle sizes in a manner other than by weight proportion was first proposed by Glick.[11] The distribution has not been ascertained, but is believed to be such that the finer AP burns in a more fuel-rich environment. Beckstead[9] has proposed an allocation based on particle surface area. Since the finer AP has more surface per unit weight it will have more binder per unit weight than the coarser AP. The concept has come to be generally accepted, but there has been difficulty in applying it to combustion models.[10] Following Beckstead, the weight fraction of a given AP size in the revised (pseudopropellant) distribution is

$$\alpha_j' = \frac{(OF)_j}{1 + (OF)_j} \tag{15}$$

where

$$(OF)_j = \frac{D_j}{\alpha_f}\sum\frac{\alpha_j}{D_j} \tag{16}$$

The significance of the fuel richness of the fine oxidizer is that the local flame temperature about the fine pocket will be less than that for the propellant as a whole. Flame temperature is plotted vs oxidizer weight fraction, for the nonaluminized AP/HTPB combination, in Fig. 4. Depending upon propellant composition, it often happens that application of Eq. (16) places the fine oxidizer system on the flat low-temperature part of the curve. As shown by the illustrative example plotted in Fig. 5, the temperature decreases as the fine size decreases.

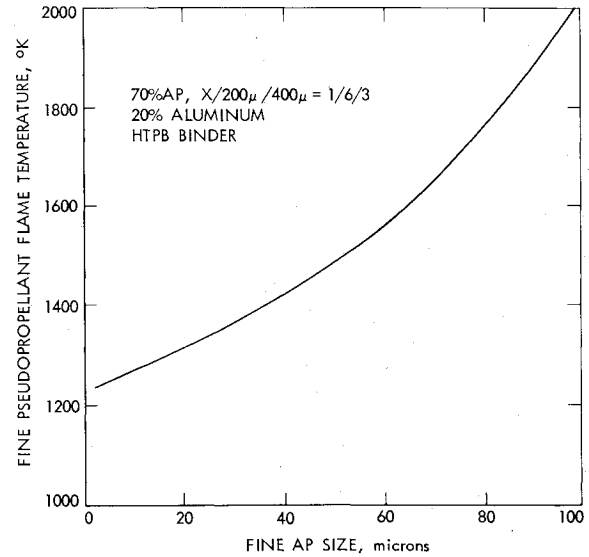

Fig. 5 Fine pseudopropellant flame temperature as a function of fine AP size for a 90% solids formulation.

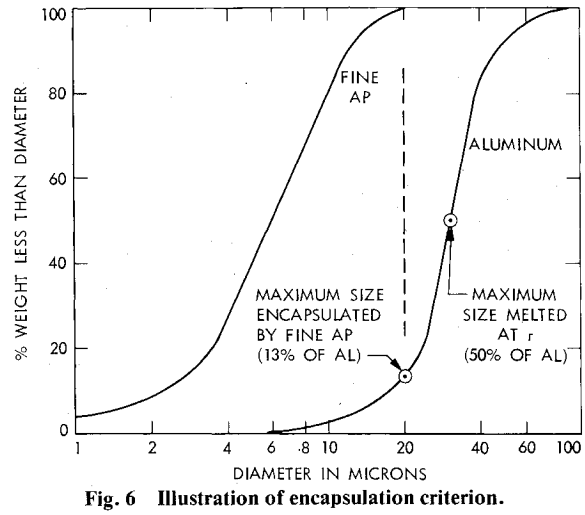

Fig. 6 Illustration of encapsulation criterion.

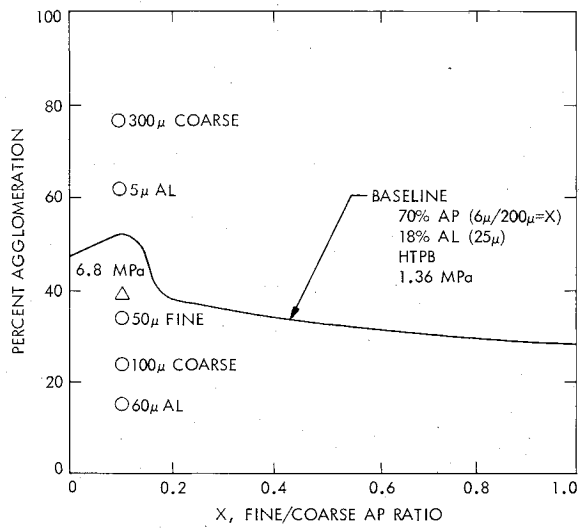

Fig. 7 Pocket model calculations for bimodal propellants.

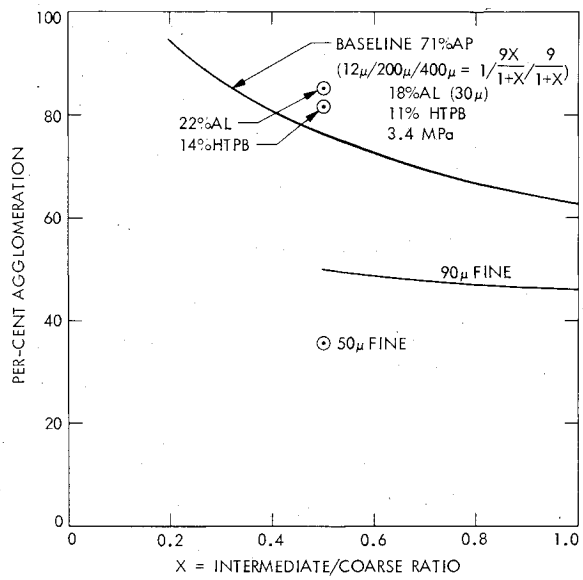

Fig. 8 Pocket model calculations for trimodal propellants.

Temperature and oxidation requirements for the ignition of aluminum are not well established. It is reported that the ignition temperature in a propellant combustion environment ranges between 1300-1500 K.[5] On this basis, the fine AP in the illustrative example in Fig. 5 should not be smaller than 20-50 μm. It is difficult to select a single temperature of demarcation that would be valid in all circumstances; for purposes of applying the model, a value of 1400 K is used.

Equation (16) suggests the following propellant formulation trends to help the fine oxidizer satisfy the temperature criterion to form the effective pocket:

1) Increase the fine AP particle size and concentration.

2) Increase the concentration of the intermediate size AP at the expense of the coarse.

3) Reduce the concentration of binder (increase total solids).

4) Reduce the particle size of the intermediate size and coarse AP.

Encapsulation Criterion

The second criterion is that the AP must encapsulate, or contain, the aluminum. Simply stated, aluminum that is larger than the fine AP is not contained. However, the only aluminum of interest is that portion of the distribution that melts. The encapsulation criterion is applied by comparing the AP size distribution with that of the portion of the aluminum that will melt. To the extent that there is no overlap in these distributions because the aluminum is larger, that portion of the aluminum must be assigned to the next larger pocket. It is considered that merely comparing mean sizes on an all-or-nothing basis would be too crude.

Application of the criterion is illustrated by the example of Fig. 6. A hypothetical 6 μm AP size distribution is plotted together with a hypothetical 30 μm aluminum. A hypothetical burning rate establishes the maximum aluminum size that will melt, as discussed earlier. In the example, this size is 30 μm so that half of the aluminum will melt. The maximum AP size is 20 μm. Hence aluminum sizes which are 20 μm or less can be encapsulated. This represents 13% of the aluminum, or 26% of the melted aluminum. The fraction encapsulated is assigned to the fine pocket volume ($f=0.13$) and the remainder is assigned to the next larger pocket volume ($f=0.37$). The faction agglomerated is then a weighted sum of the two contributions.

Model Results

Bimodal Propellants

Calculated results for a series of bimodal propellants[3] are shown in Fig. 7. The curve shows the effect of varying the fine/coarse ratio of a baseline propellant tested at one pressure. The curve is qualitatively correct. At low fine/coarse ratio, increasing the fine fraction increases agglomeration. What is happening is that the reduced volume fraction of the coarse AP is causing the coarse pocket volume to increase. The 6 μm AP is doing nothing because it does not meet the temperature criterion. As soon as the 6 μm AP meets the temperature criterion, it is able to encapsulate about 30% of the 25 μm aluminum. This causes the agglomeration to drop suddenly. As the fine fraction is increased further, there is a tradeoff between the decreasing size of the fine pocket and the increasing size of the coarse pocket; the net result is a decrease in the agglomeration. Where the fine size satisfies both criteria fully, a larger reduction in agglomeration with increasing fine fraction would be predicted.

Calculated results for other changes in variables are shown at one fine/coarse ratio, which is sufficient to illustrate the points that need to be made. First, the particle size of the coarse AP is a dominant variable. The size of the coarse AP determines the secondary pocket volume, which governs if the fine AP does not meet the criteria. It also influences the size of the primary pocket [see Eq. (9)] when the fine AP does meet the criteria. Second, increasing the size of the fine AP reduces agglomeration. There are two reasons for this: increasing the size of the fine AP raises the fine pseudopropellant O/F ratio at a given fine/coarse ratio [see Eq. (16)], so it is more likely to satisfy the temperature criterion; and the 50 μm AP can encapsulate all of the 25 μm aluminum whereas the 6 μm AP can encapsulate only a part of it, so the fine pocket is completely effective even though nominally larger. Third, increasing aluminum particle size reduces agglomeration. The reason is that a lesser portion of the aluminum melts at a given propellant burn rate. Fourth, increasing pressure reduces agglomeration. This, also, is a burn rate effect related to the amount of aluminum melted. It can be expected that pressure will have a stronger effect with fine aluminum than with coarse aluminum because all of the 5 μm aluminum is melted at 1.36 MPa whereas little of the 60 μm aluminum is melted at that pressure. All of these trends are in agreement with data.

Trimodal Propellants

Calculated results for a series of trimodal propellants[4] are shown in Fig. 8. These results are discussed more fully as follows. All are in qualitative agreement with trends shown by data.

Effect of Aluminum Content

The model predicts an increase in agglomeration as aluminum is substituted for AP. There are three grounds: the slightly reduced burn rate (more of the aluminum melts), the increased size of the effective pocket (the AP is spaced further apart), and the increased amount of aluminum.

Effect of Solids Loading (Binder Content)

Substituting binder for AP increases agglomeration. The model predicts this effect in these formulations on two grounds: the reduced burn rate and the increased size of the effective pocket. The fine AP (12 μm) is not forming the effective pocket in these formulations. If it did, another important effect of increasing binder content would be an eventual shifting of the effective pocket to the next larger AP size; the fine AP system would no longer be able to satisfy the temperature criterion [see Eq. (16)]. This shift would be brought about more readily by increasing binder content than by increasing aluminum content.

AP Modal Variations: Fine AP Size

As fine AP size is increased from 12 to 50 to 90, μm, the model predicts decreasing agglomeration in the order 12, 90 and 50 μm. The optimum fine AP size comes about because of a tradeoff between pocket volume and the ability to satisfy the temperature criterion. Since the 12 μm AP does not satisfy the temperature criterion, the effective pocket is formed by the next larger (200 μm) size. The 90 μm AP does satisfy the criterion, so the effective pocket is smaller than with 12 μm AP. The 50 μm AP also satisfies the criterion, although more marginally, and so provides the smallest effective pockets of the three fine sizes considered. The burn rates are not changing enough to be an important factor here.

The same trends were computed for analogous bimodal formulations (fine/400 μm = 3/7), but the variability in the fraction agglomerated was greater. These results are not included in Fig. 8 for the sake of clarity. The main reason why the variability was more pronounced in the bimodal formulations was the absence of the intermediate size (200 μm) AP. The presence of the intermediate size AP in the trimodal formulations mitigates the sensitivity of effective pocket size to fine AP size. It follows that a bimodal propellant containing a marginal fine size might show great sensitivity to formulation changes or lot changes.

AP Modal Variations: Intermediate/Coarse Ratio

Whether the fine size is 12 or 90 μm, increasing the intermediate/coarse ratio reduces agglomeration. With 12 μm fine AP, the intermediate size is forming the effective pocket; increasing the content of the intermediate size serves to reduce the volume of that pocket. With 90 μm fine AP, the effect is more subtle because the 90 μm AP forms the effective pocket. In that case, increasing the content of the intermediate size serves to reduce the fine pocket volume by causing the fine particles to be closer together.

These effects of the intermediate size are additional advantages of trimodals relative to bimodals. Another possible effect of increasing intermediate/coarse ratio, not seen here, would be to help the fine AP size satisfy the temperature criterion in marginal cases [from Eq. (15)].

Conclusions

A pocket model has been developed which explains qualitative effects of propellant formulation and pressure on the extent of aluminum agglomeration in AP/HTPB propellants. Variables which tend to reduce agglomeration are summarized as follows:

1) Increased concentration of AP as opposed to binder or aluminum.
2) Increased aluminum particle size.
3) There is an optimum particle size of fine AP, depending upon the other ingredients; this optimum appears to be in the range of 20-90 μm.
4) Increased concentration of intermediate-sized AP in trimodal distributions (substitute for anything but the fine AP).
5) Reduced particle size of the intermediate AP.
6) Reduced particle size and concentration of coarse AP (substitute with finer AP).
7) Increased burn rate.

In addition, trimodal distributions appear to offer greater flexibility for minimizing agglomeration than bimodals. Although the model furnishes insight into several important agglomeration influences, it is limited by lack of attention to the complex details of the agglomeration process itself.

Acknowledgments

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