

Deflagration Rate, Surface Structure, and Subsurface Profile of Self-Deflagrating Single Crystals of Ammonium Perchlorate

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The self-deflagration of single crystals of pure ammonium perchlorate (AP) is described. This deflagration behavior is divided into four phenomenological regimes that are apparent when the data are examined. These data include 1) the deflagration rate as a function of pressure for the AP crystals, 2) the combustion characteristics as seen using cinephotomicrography and 3) the surface and subsurface characteristics of quenched samples as observed using the scanning electron microscope (SEM). From these data the energy transfer mechanism for each regime is inferred.

Introduction

THE decomposition of AP has been the subject of many investigations because AP is the oxidizer and chief ingredient in most modern composite propellants. Its behavior in the combustion zone greatly influences the overall combustion of the propellant; therefore, an understanding of the deflagration behavior of AP is an important step in understanding the complex processes of the combustion of composite propellants containing this oxidizer.

Although AP is such an important ingredient, very little knowledge has been available regarding the decomposition processes involved in the deflagration of this material. This study provides experimental data obtained for the combustion of single crystals of AP at pressures between 300 and 6200 psia.

The data obtained include several hundred SEM micrographs and many high-speed motion pictures showing the combustion of single crystals of AP. The micrographs presented in this paper are representative of the several hundred which have been taken. A more complete set has been presented in Refs. 1 and 2. Similarly, space and format considerations preclude the presentation of the cinephotomicrography results. Edited films^{3,4} are available.

Experimental Procedure and Discussion

The crystals used in this study are grown from an aqueous solution of American Potash and Chemical Corporation ultrahigh-purity AP and have a density of 1.95 g/cm³ and a purity of greater than 99.9%. All inclusions of mother liquor, cracks and veiling are carefully cleaved from the crystals so that the resulting samples are without inclusions or other visible flaws. The uninhibited crystal is placed in a small combustion bomb (Fig. 1) which is equipped with windows and pressurized with dry nitrogen gas entering from the bottom of the bomb. Electrically heated 10-mil Nichrome

wires ignite the crystal and the low-velocity stream of nitrogen gas flowing past the sample removes the combustion products. A xenon arc lamp illuminates the sample in order that pictures can be taken with a high-speed motion picture camera.

In this study, the deflagration rate was measured from the high-speed motion pictures. Advantages in determining the rate by using cinephotomicrography rather than some other method include: 1) the elimination of discontinuities within the sample, such as thermocouples or fuse wires; 2) the measurement of surface regression to within $\pm 100 \mu$; and 3) the possibility of rejecting a run if anomalous behavior is noted in the visual record.

Deflagrating AP crystals can be quenched in various manners, the most common being rapid depressurization of the combustion bomb⁵ or some type of thermal quench¹. The thermal quench technique used in this study was made possible by cleaving the crystal into thin (about 1-mm.-thick) samples, which were placed between the faces of a

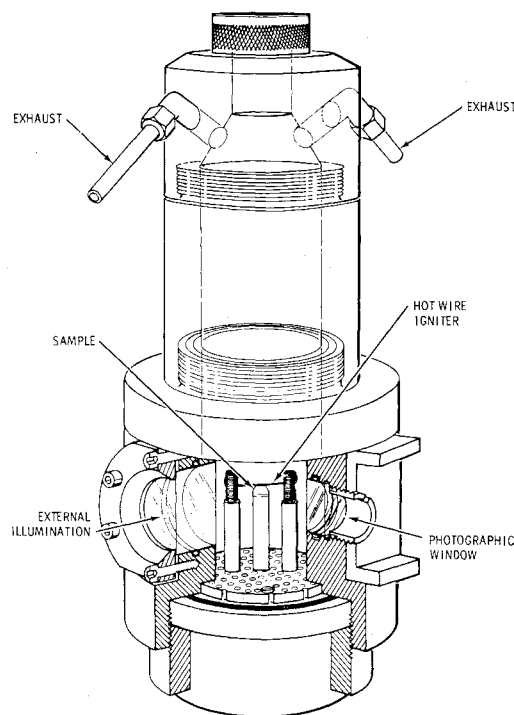


Fig. 1 Combustion bomb used for cinephotomicrography.

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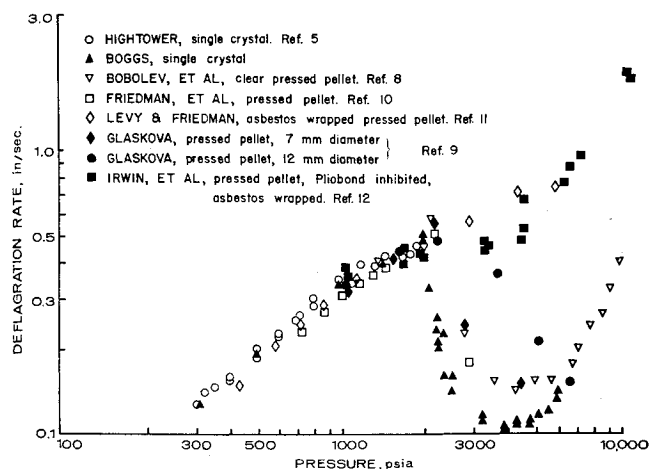


Fig. 2 Deflagration rate of ammonium perchlorate in a nitrogen atmosphere.

copper vise and then ignited. The copper vise served as a heat sink which quenched the thin samples: as the deflagration wave approached the vise, the sample lost energy to the metal and was extinguished (usually 1–2 mm above the jaws of the vise). By examining quenched samples, which were coated with approximately 100 Å of vacuum-deposited gold-palladium to reduce electrostatic charging,^{6,7} with an SEM, the details of the surface structures that were apparent in the high-speed motion pictures could be determined. After the surface had been examined the sample was removed from the SEM and cleaved perpendicularly to the quenched surface. By cleaving the sample in this manner the profile beneath the surface of the sample could be determined.

Whenever the microstructure of a deflagrating sample is to be inferred from the microstructure of a quenched sample the possibility of artifacts due to the quench technique must be considered. In order to put the results obtained from the SEM examination in perspective, these data have been compared with other data: the features seen on the deflagrating crystal surface are also seen in the SEM micrographs; the surface features seen on samples quenched by depressurization (the micrographs of Ref. 5 which were obtained for pressures between 400 and 1200 psia) possess the same structure as samples obtained by thermal quench.

Experimental Results

Deflagration Rate

The burning rate versus pressure results of this investigation, together with results of other investigations, are presented in Fig. 2. The burning rates obtained in this study agree with those obtained by the other investigators for pressures between 275 and 2000 psia. Above 2000 psia the data obtained by using uninhibited single crystals of pure AP agree qualitatively with the data of Bobolev et al.,⁸ Glaskova,⁹ and Friedman et al.,¹⁰ all of whom used pressed pellets.

The single-crystal data define a curve which can be naturally divided into four regimes (Fig. 3). Regime I is that portion of the curve, between 300 psia and approximately 800 psia, at which the curve has an equation of the form $r = Cp^n$ with a burning rate exponent of approximately 0.77. The portion of the curve between 1000 and 2000 psia, at which the slope is positive but decreasing, defines regime II. Regime III is that portion which has a negative slope, in the pressure range between 2000 and 4000 psia. Above 4000 psia is the domain of regime IV. As will be seen later, the detailed nature of the combustion zone is also qualitatively different in each of these regimes.

The combustion which occurred in regime III was unsteady with an areawise pulsation. Because the surface regression

was not a steady planar regression, a mean regression rate was determined as follows. When the film was reduced, three to six lines were drawn across the image perpendicular to the regressing surface. Instantaneous rates were calculated in $\frac{1}{16}$ -sec intervals and an average rate for the total regression along a line was determined. The over-all average regression rate for a run was determined by averaging the average rate for each line. The instantaneous rate differed from the average rate within $\approx \pm 10\%$; the average rate for each line differed from the average rate of another line on the same sample by $\approx \pm 4\%$; and the overall average rate of one run differed from the over-all average of another run at the same pressure by ≈ 4 –6%.

Although the actual magnitude of the data defining the deflagration rate curve between 2000 and ≈ 5000 psia may be subject to discussion, the trend of data is correct. Because some investigators^{11,12} have assumed that the decrease in the burning rate curve was due to convective cooling, this possibility was also explored. This was done in three ways, 1) by varying the sample size, 2) by altering the convective system of the combustion bomb and 3) by critically examining the motion picture films. Other investigators have reported difficulty in getting burning rate data in the pressure range corresponding to regime III, with results being dependent on size of sample, mode of inhibition, etc.⁹ In order to assure that the results obtained in the present study were indeed indicative of deflagration behavior of AP, several variations of technique were used. When the sample size was varied a difference of rate was observed until $\approx 5\text{ mm} \times 5\text{ mm}$ single crystals were used. In order to avoid, as much as possible, any effects of heat loss from a small sample, the crystals burned in regime III were larger than 5×5 mm cross section. It may also be noted that sample size had no measurable effect on the deflagration of regimes I, II and IV, as might be expected if convective cooling were the sole cause of the decrease of rate seen in regime III.

The combustion bomb was altered so that two runs could be made under identical conditions except that one run used the nitrogen purge flowing along the sample side while the other run used no flow; the only convection along the sample side would be free convection. No measurable difference in burning rate was observed between the flow and the no-flow conditions.

In addition, when Watts and Petersen¹³ burned these same crystals in their apparatus, they measured essentially the same deflagration rate as a function of pressure. If convective cooling were truly applicable one might expect different rates when different apparatus were used. In addition, Watts and Petersen used helium as well as nitrogen for the pressurizing gas and found no effect in the deflagration rate curve. The data were also identical when the samples were burned horizontally instead of vertically. They concluded that the measured burning rates were characteristic of the AP and were not influenced by conduction to the inert environment, natural convection, or the type of apparatus.¹³

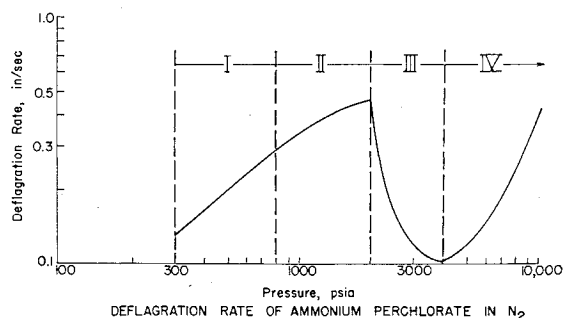


Fig. 3 Deflagration rate of single crystals of ammonium perchlorate in a nitrogen atmosphere. The curve has been divided into four naturally occurring regimes.

When the motion pictures taken of deflagration in regime III are examined, no definitive evidence for convective cooling (such as the center of the sample regressing more rapidly than the edges) is observed. In view of all this information it must be concluded that the decrease in burning rate with increased pressure between 2000 and approximately 4000 psia was characteristic of AP deflagration and not an artifact of the experiment.

Characteristics of Thermally Quenched Samples

The surfaces of pure AP single crystals which were thermally quenched while deflagrating in 300 and 600 psi (regime I) nitrogen atmospheres are shown in Fig. 4. The bubbles and froth are visible on the surfaces of these thermally quenched samples and look very similar to those observed by Hightower and Price^{5,14} on samples which were quenched by rapid depressurization from 300 to 800 psia nitrogen atmospheres.

It should be noted that the bubbles seen on these samples are not artifacts of the test technique since the samples were thermally quenched as opposed to quenching by depressurization of the test chamber. Indeed, the formation, growth, and "popping" of the bubbles were observed when the high-magnification, high-speed motion pictures taken during burning were viewed.

Examination of the profiles of cleaved samples of regime I revealed layering, with a froth covering the cubic phase of the otherwise orthorhombic AP single crystal (Fig. 5). Because the froth included two phases (liquid and entrapped gas), it was difficult to measure its thickness with any degree of accuracy. If the large bubbles (some of which were larger than $20\ \mu$ in diameter) were not included in the measurement, the froth was approximately 1 to $5\ \mu$ thick on both the 300 and 600 psia samples. The sample obtained at 800 psia showed only patches of froth as contrasted to the lower pressure samples on which froth covered the entire surface. The thickness of the cubic layer varied inversely with pressure, and the values obtained verified the earlier work of Beckstead and Hightower.¹⁵

The transition from regime I to regime II takes place between 800 and 1000 psia, as illustrated in Fig. 6. The bubbles and froth of regime I give way to a pattern of ridges and valleys first seen by Hightower and Price^{5,16} which is characteristic of regime II. The micrographs of Fig. 7 show how the surface structure evolved when the pressure was increased in regime II. The length of the ridges is an inverse function

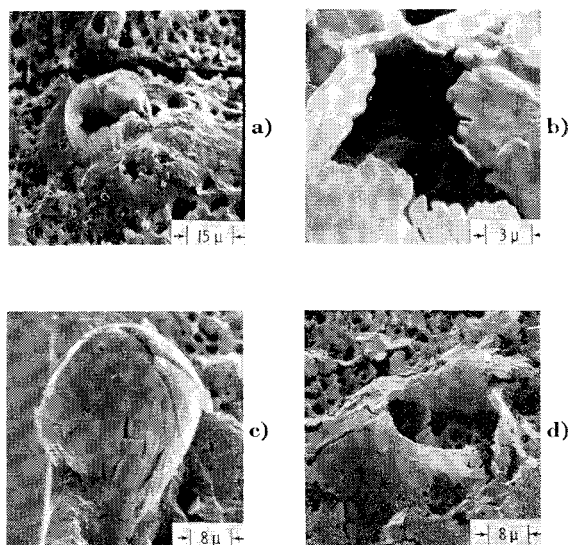


Fig. 4 Surface structure of ammonium perchlorate samples thermally quenched while deflagrating at the pressures of regime I: a)-b) 314 psia, c)-d) 600 psia.

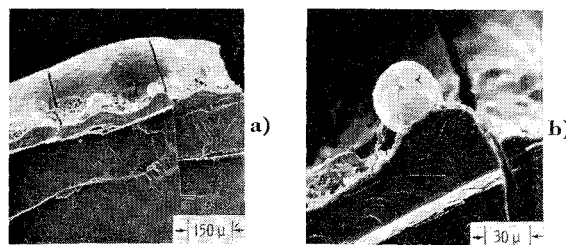


Fig. 5 Profiles of ammonium perchlorate sample which was thermally quenched while self-deflagrating in a 300 psia nitrogen atmosphere.

of pressure; the ridges of the 1200 psia sample (Fig. 7a) are well over $300\ \mu$ in length, while at 1800 psia (Fig. 7e) few are longer than $100\ \mu$. All of the samples show geometrically complex reaction sites at the bottom of valleys. The samples quenched at 1500 and 1800 psia displayed a few isolated pockets of needles as seen in Fig. 8.

The profiles of regime II samples (Fig. 9) indicated that the activity sites of the valley extended below the surface for a depth almost equivalent to the cubic phase thickness. Figure 9a shows that the activity sites extend about $10\ \mu$ into the sample. Figure 9b shows that 1) the cubic phase ranged between $10\ \mu$, valley-to-cubic-orthorhombic interface, and $20\ \mu$ ridge-to-cubic-orthorhombic interface†; and 2) the ridge is undercut (which partially explains why the high-magnification, high-speed motion pictures showed that often unburned ridges peeled away from the surface of the sample before complete burning).

The sample obtained at 1500 psia is presented in Fig. 9c and d. It should be noted that the surface of this sample is

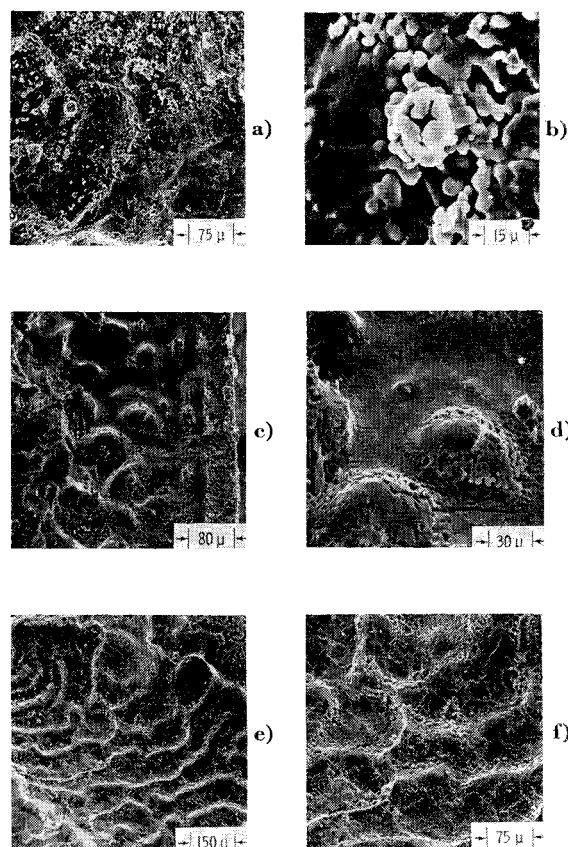


Fig. 6 Surface of ammonium perchlorate samples thermally quenched while self-deflagrating at: a)-b) 300 psia, c)-d) 900 psia, e)-f) 1000 psia.

† It must be noted that often details which are unambiguously evident on original micrographs are not plainly discernible when they are reprinted in a journal article.

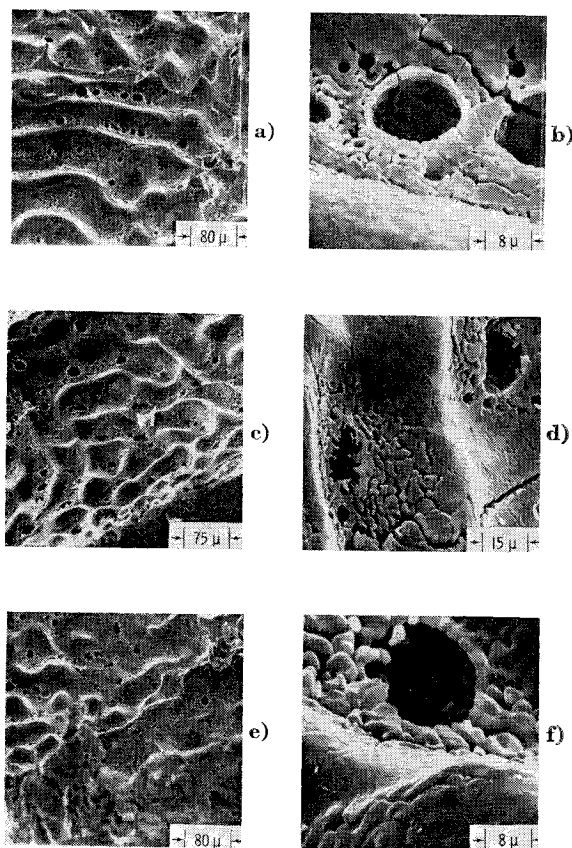


Fig. 7 Surface of ammonium perchlorate samples thermally quenched while self-deflagrating at the pressures of regime II: a)-b) 1200 psia, c)-d) 1500 psia, e)-f) 1800 psia.

convolute, as were others from regime II. It might also be expected (if the one-dimensional heat-transfer assumption used in many combustion models were correct) that the boundary between the material which had been cubic phase and the unaffected orthorhombic AP would also be convoluted to roughly the same contour. This was not the case as is clearly shown in Fig. 9c. The thickness of the cubic phase beneath the valley for this pressure was $5-7\ \mu$, while under the ridge the thickness was 25 to $35\ \mu$. It may be seen in Fig. 9d that although the cleavage plane did not cut directly into an activity site there is evidence that some reaction took place beneath the surface. Samples obtained at 1800 psia indicated that the activity sites extended through what had been the cubic layer which was $2-3\ \mu$ thick under the valleys. The thickness of the cubic phase relic was approximately $25-35\ \mu$ under the ridges.

The motion pictures taken of the deflagration at regime III pressures showed an areawise pulsation in the sample regression due to unsteady flamelets. The flamelets would "stand off" from portions of the surface and those portions would rapidly regress while those areas having no adjacent flamelet were momentarily stationary. The flamelets would shift location over the surface and areas which had been stationary would regress when contacted by the flamelets.

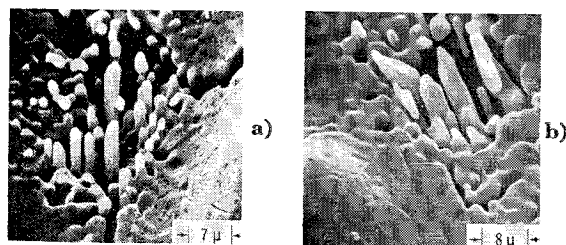


Fig. 8 Isolated pockets of needles seen on regime II samples: a) 1500 psia; b) 1800 psia.

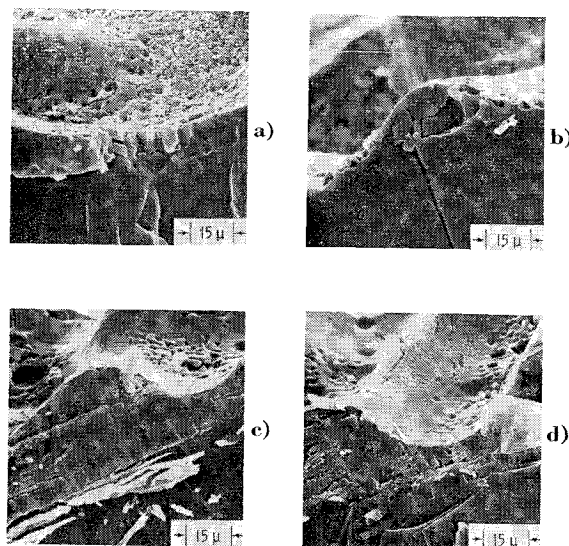


Fig. 9 Profile of ammonium perchlorate samples which were thermally quenched while self-deflagrating at: a)-b) 1200 psia, c)-d) 1500 psia.

Self-illumination motion pictures (i.e., no external illumination) were made and these motion pictures showed orange flamelets which moved from area to area as the burn progressed. The intermittency of the flame is probably the cause of the pulsations of flame temperature reported by Bobolev et al.¹⁷

A sample quenched at 3300 psig is presented in Fig. 10a-f. The area of maximum regression (Fig. 10c) possesses a needle structure on the surface (Fig. 10e, f). Figure 10e shows the structure found at the bottom of the depression and Fig. 10f shows the structure found on the side of the de-

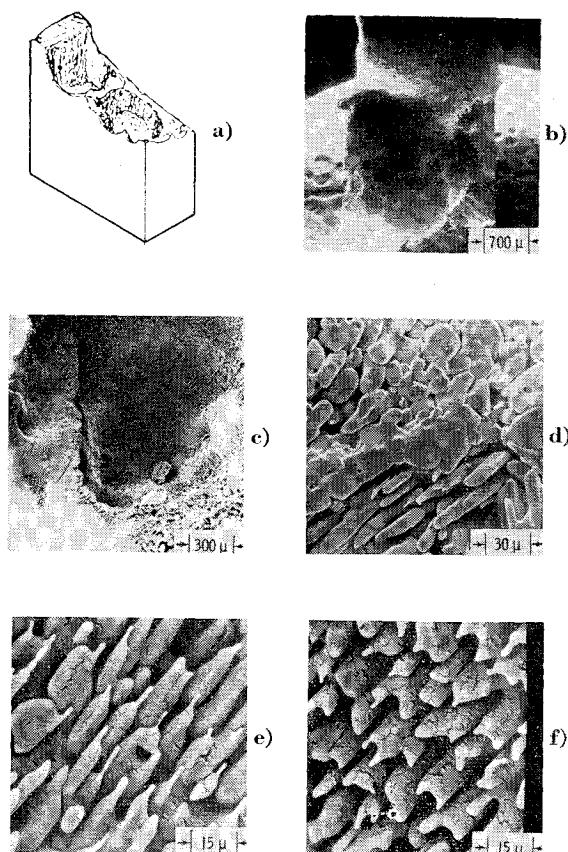


Fig. 10 Surface of ammonium perchlorate sample which was thermally quenched while self-deflagrating at 3300 psia.

Table 1 The deflagration rate, surface structure, and subsurface profile of self-deflagrating single crystals of ammonium perchlorate

Re-gime	Pres-sure, psia	Rate, in./sec	Rate as a function of press. increase	Regression	Surface		Subsurface		Energy transfer
					Characteristic	Function of press. increase	Characteristic	Function of press. increase	
I	300-800	0.13-0.30	$r = cp^n$ where $n = 0.77$	Steady planar	Gas entrapped in liquid resulting in a froth	Thickness of liquid layer decreases, amount of gas increases	Froth on cubic phase, cubic phase on orthorhombic phase	Cubic phase thickness decreases, froth appears to decrease	"Exothermic" froth
II	1000-2000	0.34-0.48	Decreasing positive dr/dp	Steady macroscopically planar	Ridges & valleys with activity sites in valleys, surface pattern spatially invariant with time	Length of ridges decreases, activity sites go from froth to vigorous gas reaction sites, a few needles	Ridges & valleys composed of cubic phase, activity sites extend depth of cubic phase	Cubic layer under valleys decreases, height between ridges & valleys increases	Condensed phase and gas-phase coupled
III	2000-4000	0.48-0.10	Negative dr/dp	Intermittent nonplanar	Needles in areas of max. regression		Thickness of layer of needles $\approx 200-300 \mu$, no needles at areas of min. regression		Intermittent flame, localized decomposition in needle array
IV	>4000	>0.10	Positive dr/dp	Steady macroscopically planar	Entirely covered by needles		Surface layer of needles on solid		Steady flame with uniform array of needles

pression. Because the flame was observed to be in the depression, the "lip" seen in Fig. 10b provides a chance to view a portion of the sample (Fig. 10d) which was partly accessible to the flame and at the same time view a portion which was shielded from the flame. That portion which the flame contacted possesses the needle structure previously seen while the shielded portion has a different surface structure, indicating that the needle structure is characteristic of proximity to the flame.

The profiles of regime III samples differed quite significantly from those of regimes I and II. No longer was there any froth or measurable cubic phase; rather, the structure seen in Fig. 11 was observed. The thickness of this layer of needles varied from zero at those points that were not exposed to the flame to 200 to 300 μ where the flame existed.

Although the samples of regime III had pockets of needles concentrated at the points of maximum regression, the needles entirely covered the surface of regime IV samples (Fig. 12). This is consistent with the observation that the flame displayed an area-type pulsation in regime III and was in contact with the entire surface in regime IV.

As was inferred from a comparison of the surfaces of samples from regimes III and IV, the subsurface profile of regime IV samples should closely resemble the profiles found under the depressions of regime III samples (see Fig. 13 which shows the needles covering the entire surface of the sample).

Discussion

The deflagration-rate curve, the high-speed motion pictures, and the SEM micrographs all indicate that the deflagration of AP between 300 and 6200 psia (and probably up to at least 10,000 psia if Bobolev's data are considered) can be characterized by four pressure-dependent regimes (described in Table 1). Such a division is important in that it allows the correlation of observations from many sources, thus permitting predictions of crystal behavior during deflagration to be made from a knowledge of pressure.

But perhaps most important, the data presented show that the deflagration of AP is much more complex than was previously imagined. No longer can it be assumed that one

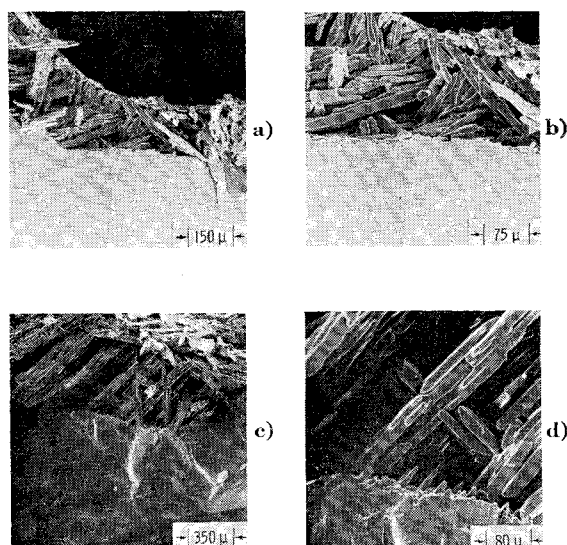


Fig. 11 Profile of ammonium perchlorate samples which were thermally quenched while self-deflagrating at pressures of regime III: a)-b) 2800 psia, c)-d) 4000 psia.

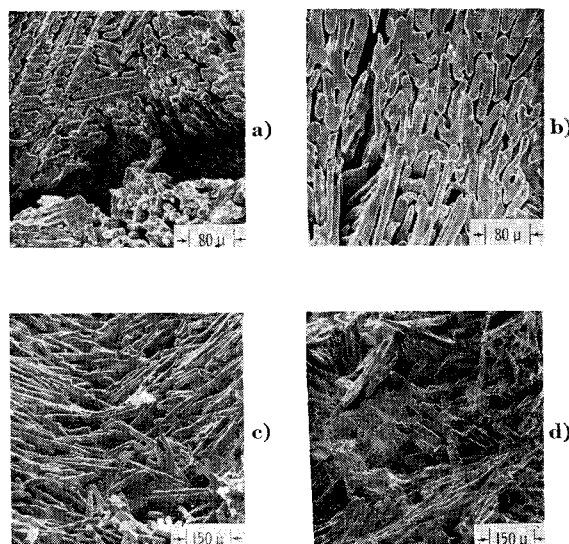


Fig. 12 Surface of ammonium perchlorate sample which was thermally quenched while self-deflagrating at pressures of regime IV: a) 4000 psia, b) 4500 psia, c) 5500 psia, d) 6200 psia.

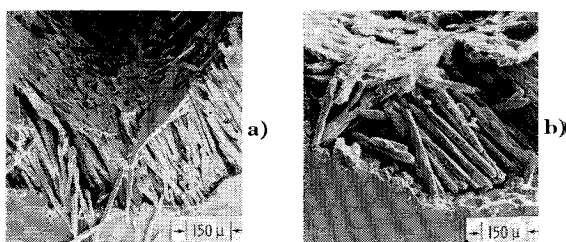


Fig. 13 Profiles of ammonium perchlorate samples which were thermally quenched while self-deflagrating at pressures of regime IV: a) 4000 psia, b) 6200 psia.

mechanism (e.g., sublimation of the crystalline AP and conduction of heat from the gas-phase reactions of ammonia and perchloric acid vapors) supplies all the energy required for self-deflagration. From the results presented in this study one must conclude that there are at least two distinct deflagration mechanisms: one responsible for the low-pressure (up to 2000 psia) deflagration and one responsible for the high-pressure (above 4000 psia) deflagration.

The SEM micrographs and the high-speed motion pictures show that for regime I a pseudo-condensed phase covers the surface of the deflagrating samples. This pseudo-condensed phase consists of a liquid layer with entrapped gas bubbles whose nucleation and growth within the liquid produce a froth. A frothy liquid on the surface of the deflagrating sample provides excellent possibility for energy transfer to the solid; liquid, heterogeneous-phase, and gas-phase reactions can take place within the froth, with a very large area for energy transfer. Thus, it seems plausible to assume that the froth is essential to the self-deflagration of AP at low pressures ($300 < p < 800$ psia): pressures where no evidence of a strong gas flame was seen when the high-speed motion pictures were viewed. When the profiles of regime I samples were examined no change in the AP was detected beneath the liquid (other than the phase transition) which implies that there was no significant contribution to the heat release during deflagration by solid phase reactions.

Regime II samples also showed evidence of condensed-phase reactions. Why the reaction should be concentrated in individual reaction sites and cause the more rapid regression of the area around the sites (thereby forming the valleys seen in the SEM micrographs) is not presently known. The consequences of these reaction sites can be seen when sample profiles are examined. The reaction sites extend several microns into the sample. The fact that the phase transition is thicker under the ridge than under the valley shows that the temperature gradient through the ridge was not as steep as under the valleys and that heat was transferred from these reaction sites in three dimensions (including conduction of heat in a direction nearly perpendicular to the direction of surface regression). A few flamelets standing over the regime II samples were also observed in the high-speed motion pictures. Flamelets standing over regime II samples could also be inferred from the few isolated pockets of needles on the quenched samples obtained at 1500 and 1800 psia. Thus, while the reactions taking place in the pseudo-condensed phase were probably the dominant reactions, a secondary contribution from the flame was also possible.

The decrease in deflagration rate with pressure increase of regime III may be due to neither mechanism being dominant. No conclusive evidence was found for condensed-phase reactions in regime III and an area-wise pulsation of the flame was observed.

It appears that the planar surface regression of regime IV deflagration was due entirely to energy feedback from the flame which covered the entire surface, although some problem is encountered when "flame stand-off" distance is considered. The needles seen in the SEM micrographs extend from the surface 150–200 μ ; yet flame stand-off distance

calculations¹⁸ show that the distance from the unreacted solid to the flame should be significantly less than 150–200 μ . Hence, discussions of flame stand-off distances for the self-deflagration of AP, while being valuable computational schemes, may have little physical significance when applied to regime IV self-deflagration.

What causes these needles is not known but strong evidence for their existence during the deflagration is present on the high-speed motion pictures. The films of back-lighted samples deflagrating in regime IV show clear AP, 150–300 μ of opaque solid with the flame standing above the opaque solid.

Summary

1) This study has shown that four phenomenological regimes which characterize the deflagration of AP for pressures between 300 and 6000 (and probably at least to 10,000) psia may be defined. The behavior has been conveniently summarized in Table 1.

2) These results indicate that previous views regarding AP deflagration deserve re-examination because a) Ammonium perchlorate doesn't sublime while self-deflagrating at low pressures; rather, a liquid exists on the deflagrating crystals' surface. b) The decrease in deflagration rate with pressure increase between 2000 and 4000 psia is not due to "convective cooling," but instead is a characteristic of the self-deflagration of AP. c) Flame stand-off distances which have been calculated have little physical significance to the self-deflagration. d) One energy-transfer mechanism is not sufficient to describe the self-deflagration of AP. The possibility of the existence of both condensed phase and gas phase reactions must be considered. e) One dimension is not sufficient to describe the energy transfer (especially in regimes I, II and III). f) Low-temperature decomposition kinetic parameters (obtained from a situation where no melt exists, i.e., the sample undergoes partial decomposition and subsequent sublimation) cannot a priori be applied to the self-deflagration of AP.

3) Any theoretical modeling of the self-deflagration of AP must be able to describe all behavior noted in Table 1 if it is to be truly reflective of the actual combustion process.

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Further Investigations on Low-Density Hall Accelerators

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Based on theoretical considerations, the dependence of the thruster efficiency on the magnetic field and on the length of the accelerator channel has been calculated. To reduce the anomalous electron back-flow, an external ion source should be used which produces a homogeneous ion beam. This can be obtained by an annular plasma source with a slit hollow cathode. Experiments have been carried out on two different accelerator types. The plasma properties in the exhausted beam have been measured using probes and spectroscopical methods. The ion velocity distribution has been measured by a retarding potential method. For the determination of the thrust a pendulum has been used. Further experiments have been done on ion sources.

Nomenclature

A_g = weighted transition
 B = magnetic field
 E = electric field
 J = relative line intensity
 Q = cross-section for collisions
 T = temperature
 U = accelerator voltage
 W = excitation energy
 j = current density
 k = Boltzmann constant
 l = accelerator length
 m = mass
 n = particle density
 p = pressure
 q = electric charge
 r = radial coordinate
 t = time
 v = velocity
 z = axial coordinate
 η = efficiency
 λ = wave length
 σ = electric conductivity

φ = azimuthal coordinate
 ω = angular velocity

Subscripts

c = cyclotron
 e = electron
 i = ion
 o = neutral

1. Introduction

IN the near future, several missions into the near space, orbit-to-orbit transfer, and mapping of the Van Allen Belt, etc., can be realized by using electric thrusters. The electric power requirements for these missions are in the range of 1-20 kw. Typical thruster data are mass flow rates $\sim 10^{-6}$ kg/sec, exhaust velocities between 20 to 50 km/s, thrusts between 10^{-2} to $2 \cdot 10^{-1}$ N.

Of all electric thrusters the ion thrusters have reached the highest status of development up to date. Their main field of application, however, appears to be missions, where exhaust velocities between 50 km/sec and 100 km/sec are required. Good results have also been obtained with MPD-arcjets. The practical range of electric power input, however, is in general beyond that needed for the missions mentioned previously. Hence, there is a gap in the spectrum of thrusters available up to date which can be closed by Hall ion-thrusters. The power requirement of these thrusters is between 0.5 to 5 kw, the velocity range 20-50 km/sec. The principle of the Hall ion-thrusters is based on the acceleration of ions as in

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