Effects of Low-Temperature Ammonium Perchlorate Decomposition on the Ballistic Properties of a CTPB Propellant

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The objective of the subject work was to characterize the effect of the low-temperature condensed-phase decomposition (LTD) of ammonium perchlorate (AP) on the ballistic properties of a CTPB propellant. Propellants were prepared from three chemically altered AP oxidizers, differing widely in initial LTD rate, but having essentially identical particle size distributions. Other physiochemical properties of the propellants were maintained constant during processing. The three oxidizers were as follows, in order of increasing initial LTD rate: 1) AP cocrystallized with $(NH_4)_2$ HPO_4, 2) recrystallized control, and 3) AP cocrystallized with $KClO_3$. Experimental evidence is cited for the probable absence of phosphate-induced catalysis of the high-temperature decomposition of AP. Despite a demonstrated tenfold spread in isothermal LTD weight loss rates of the special oxidizers used in the three propellants, these differences had no measurable effects on strand burning rate, critical $\dot{P}$ extinction characteristics, low-pressure deflagration limit, burning-rate temperature sensitivity, or ignition delay time.

Introduction

ALTHOUGH the low-temperature (100°–300°C), condensed-phase decomposition (LTD) of ammonium perchlorate (AP) has been studied extensively, its influence on the transient as well as quasi-steady phases of AP and AP/propellant combustion remains quite controversial.[1–3] The main purpose of the present paper is to clarify this issue. Since the high-temperature, condensed-phase decomposition (HTD) of AP naturally follows the LTD in a rising temperature environment, the possible relevance of HTD (which is even more controversial) will be discussed briefly in the present paper where it becomes necessary to distinguish between these sequentially occurring modes of decomposition.

The primary objectives of the present work were to clarify the influence of LTD on the burning rate, ignition, and extinction characteristics of a CTPB propellant, using the following approach. Controlled sets of experiments were performed on three specially prepared propellants, made with three chemically altered lots of AP differing widely in initial LTD rate. Comparisons of the results were then used the evaluate LTD effects on the respective ballistic property tested in each set.

The prepared AP lots were shown to exhibit large differences in initial LTD weight loss rate at 158°C. These differences should prevail beyond 300°C, but converge to nearly identical HTD rate behavior in the vicinity of 380°C (Refs. 4 and 5). Although it was recognized that these temperatures were still far below the usual 550°C surface temperature associated with steady propellant combustion, subsurface reaction rates and lower temperature domains of transient phenomena such as ignition and extinction, and especially postextinction heating leading to exothermic decomposition of exposed oxidizer, were thought to be susceptible to LTD rate changes.

AP Decomposition Behavior

The following is a brief summary of the principal features of AP condensed phase decomposition (CPD) (see Refs. 1–3 for further details). The LTD of AP is characterized by formation of groups of isolated nuclei, or active decomposition centers, which form on or very near the external crystal surface about defect sites. These centers grow three dimensionally and eventually coalesce to form a "continuous reaction interface," which moves at rates, depending on crystallographic direction, toward the center of the crystal. The very early stages of nucleation and growth proceed more or less smoothly on a macroscopic scale, but give the appearance of an induction period. The rate of weight loss, due to decomposition, clearly becomes acceleratory in character after sufficient nucleation and growth, but before surface coverage is complete; the latter condition marks the beginning of the deceleratory rate period, due to geometrical considerations. Cocrystallized phosphate and chlorate, which were used in the present study to provide extremes in LTD behavior, have been shown to exert their respective LTD rate retardation and acceleration effects on weight loss during the "usual" acceleratory phase.[6,7] After about 25–35% weight loss (depending on sample and conditions) the low-temperature decomposition ceases. The highly porous residue, which ostensibly is AP, may continue to sublime at a relatively low rate but will not decompose further without rejuvenation by exposure to solvent vapors and/or grinding.

At temperatures above the 240°C orthorhombic-to-cubic phase transition, the decomposition centers are initiated by growth of a single nucleus, rather than by aggregates of nuclei. Between 240° and, say, 300°, this mode of reaction, although complicated by the phase transition, is still considered part of the LTD phenomena; nucleation and reaction propagation still appear to be determined by the defect structure of the solid, and the decomposition stops at roughly 30% weight loss.

A transition from LTD to HTD appears to occur between 300° and 380°C, and at temperatures between 380° and 450°C the decomposition is quite clearly described as the HTD.[1–5] In general, there is no appreciable induction period τ_i with the HTD ($\tau_i <$ thermal equilibration time in TGA), the reaction is deceleratory throughout, and it results in complete gasification of the salt. The HTD appears to be relatively unaffected by the presence of cocrystallized chlorate or phosphate ions (presented later), but it can be affected by some LTD rate accelerators, for example, permanganate ion. Note that the apparent absence of an induction period in HTD must be qualified. Recent CO_2 laser pyrolyses of pure AP within the high-vacuum ion source chamber of a

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Index categories: Propellants; Thermodynamics and Combustion.

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Bendix TOF mass spectrometer[8] indicated that a short but still relatively significant (on combustion time scales) induction time existed (35 msec) for the HTD process, compared to that for dissociative evaporation (18 msec), at incident fluxes (200 cal/cm^2 sec) where recession rates of about 1.0 mm/sec were observed.[9]

Although it is still too early to describe with any certainty the topochemistry of either LTD or HTD, it seems clear that significant decomposition via purely thermal pathways (as opposed to low-energy pathways that depend upon crystal defect structure) is likely to play an increasingly important role in the transition from LTD to HTD. Recognition of the rapid increase in instability/reactivity of $HClO_4$ as temperatures approach 400°C readily suggests that thermal decomposition of adsorbed $HClO_4$, and/or gaseous $HClO_4$ just above the AP surface, followed by reaction of the resulting radicals and atoms with adsorbed NH_3, may dominate HTD[1–3] as long as AP dissociation is favored. However, recent studies of AP decomposition by Schmidt[5] suggest that other decomposition pathways may also significantly influence HTD, at least under certain conditions. Isothermal thermogravimetric analyses of dispersed granules of AP, heated to temperatures between 380° and 450°C and exposed to a flow of heated anhydrous NH_3 at 1 atm, yielded an activation energy of 52.1 ± 1.5 kcal/mole.[5] This is to be compared with an activation energy of 19.0 ± 0.5 kcal/mole for the LTD of AP granules, obtained at temperatures between 255° and 300°C in 1 atm N_2 (static) by the same TGA technique.[5] On the basis of these results (especially $\Delta E = 52$ kcal/mole), combined with measurements of $KClO_4$ decomposition rates at 440°C under both NH_3 and N_2, Schmidt suggested that HTD in the presence of NH_3, which strongly suppresses AP dissociation to adsorbed $HClO_4$ (at least up to moderately high temperatures), may proceed by a quite different mechanism; that is, one in which the perchlorate ion undergoes thermal decomposition and/or directly oxidizes the ammonium ion.

On the basis of the aforementioned results and recent reviews,[1–3] the extent of influence of HTD on AP/propellant combustion at elevated pressures still remains a highly complex and unresolved question.

Effects of Phosphate on LTD

Schmidt[4] demonstrated that 0.1% coprecipitated phosphate prepared by cocrystallizing AP with Na_3PO_4, substantially reduced the rate of LTD at 268°C, 1 atm. Mixtures of AP with 10% granular Na_3PO_4 exhibited similar behavior, but the extent of retardation was somewhat reduced.

Pellett and Cofer[6,7] confirmed the phosphate retarding effect on LTD at 158°C, 1 atm, and then characterized this effect using several samples prepared from reagent AP cocrystallized with variable amounts of $(NH_4)_2HPO_4$. They also characterized accelerated LTD decompositions of AP's cocrystallized with $KClO_3$, and compared the results with those from recrystallized control samples. A recent extension of the study, under very carefully controlled crystallization conditions, indicated a significant phosphate retarding effect down to 1 ppm phosphate in the AP crystal.[7]

Effects of LTD Retardants at Higher Temperatures

Schmidt[4] found that the respective rates of HTD at 382°C, leading to 100% weight loss, were identical for AP/Na_3PO_4 mixtures, AP containing 0.1% PO_4 derived from an AP/Na_3PO_4 cocrystallization, and an AP control. However, from a separate series of isothermal decomposition experiments, where Schmidt made simultaneous weight loss and sublimate-collection analyses after several minutes heating at 330° to 378°C, both CPD and sublimation were found to be affected (especially at the lower temperatures) by the presence of 10% Na_3PO_4 admixed with AP.[4] Compared to pure AP, the percent weight loss due to sublimation increased substantially, while that due to CPD decreased. The total weight loss of the treated oxidizer was always less than that for AP, but convergence toward similar weight loss histories at 378°C was evident near the usual onset (380°C) of HTD.

Effects of LTD Retardants on Combustion

Mayer and Schieler[10] presented DTA curves for AP mixtures containing NH_4PF_6, NH_4BF_4, $(NH_4)_2TiF_6$, and $(NH_4)_2HPO_4$. The hexafluorophosphate was found to be most effective, followed by $(NH_4)_2HPO_4$, in suppressing the first exotherm in both AP and AP/fluorocarbon binder systems. The suppression of LTD through the use of NH_4PF_6 in a fluorocarbon composite propellant was said to result in improved extinction characteristics and some modification of the burning-rate characteristics.

Schmidt[4] found that PBAA propellant strand burning rates (600–2500 psia) were unaffected (within ±5%) by the presence of 10% admixtures of Na_2SO_4 or Na_2HPO_4 with 65% AP, compared to a similar 10% admixture of NaCl as control. Since Schmidt did not examine the effects of AP cocrystallized with phosphate on propellant burn rate, it seemed judicious to reserve opinion on the possibility of a small LTD effect during propellant combustion. Some indications of cocrystallized AP combustion behavior were evident in recent follow-on work,[5] where Schmidt demonstrated that cocrystallized $(NH_4)_2HPO_4$ (0.13 and 0.64% in crystal as H_2PO_4) and $KClO_3$ (0.10%, as ClO_3) had: 1) variable but significant retarding and accelerating effects (about −6%, −18%, +3%, respectively) on the burn rates of pressed AP strands over the range 900–1500 psi, and 2) somewhat smaller burn-rate effects (about −5%, −12%, +2%, respectively) over the range 350–900 psi.

Munson and Reed[11] introduced a number of additives into AP by spray-drying concentrated solutions. $(NH_4)_2HPO_4$ and $NH_4H_2PO_4$, at the 10% solids concentration level, were highly effective in reducing and/or shifting the low-temperature DTA exotherm to higher temperatures. Burning rates of 18% aluminized PBAA propellants, containing 12.5% of the above spray-dried AP (69% total AP), decreased by 14% at 1000 psi compared to the control. The burning rate was also reduced by essentially the same amount when an equivalent quantity of additive was admixed in the control formulation. A comparison of these two cases with Schmidt's results with PBAA[4] suggests that 1) added aluminum in Ref. 11 may have imposed a synergistic effect, 2) the reported differences in Ref. 11 particle size distributions (from the control) may have been operative, and/or 3) the higher additive concentration level in Ref. 11 may have rendered the additive more effective.

Maycock and Payne[12] cocrystallized $(NH_4)_2PO_4$ with AP and found from TGA's (15°C/min) that the temperature for 50% weight loss decreased from 407°C for "pure AP" to 369°C for AP containing 10^{-3} mole percent SO_4. This apparent acceleration in weight loss may have been due to enhanced sublimation at higher temperatures, similar to the effect demonstrated by Schmidt for AP/Na_3PO_4 admixtures,[4] but further "enhanced" by a helium purge in their apparatus. Although Maycock and Payne found an apparent increase in propellant burn rate of about 20% at 1000 psi, with 10^{-3} mole percent SO_4 dopant, their burning-rate data were sparse and showed considerable scatter. Also, their cocrystallized AP samples, due to moisture pickup, had to be "deagglomerated" prior to mixing, raising the question of particle size rather than dopant effects.

Propellant Preparation

Three CTPB propellants were prepared with treated oxidizers differing widely in AP initial LTD rate, but having essentially identical particle size distributions. Other physiochemical properties of the propellants were maintained constant during processing.

The three lots of prepared AP were as follows, in order of increasing initial LTD rate: 1) cocrystallized with $(NH_4)_2HPO_4$ (hereafter PAP); 2) recrystallized control containing no additive

(RAP); and 3) cocrystallized with $KClO_3$ (CAP). The treated oxidizers were prepared in a 25-gallon vertical mixer by dissolving standard propellant grade AP and the desired additive§ in distilled water at 71°C. The solution was then cooled to 21°C and the crystals filtered from the cooled mother liquor, distributed on trays, and dried at 68°C in an air-circulating oven. The dried material was delumped and the larger particles removed by screening. The crystals passing through a 420-μm screen were classified and used as the coarse fraction in the propellants. The particles retained were ground in a micropulverizer to obtain the fine fraction used.

The particle size distributions for the coarse oxidizer (sieve analysis) are shown in Fig. 1 for the three screened oxidizers. The size distributions for the fine materials (micromerograph) were nearly log-normal with distribution statistics as given in Table 1.

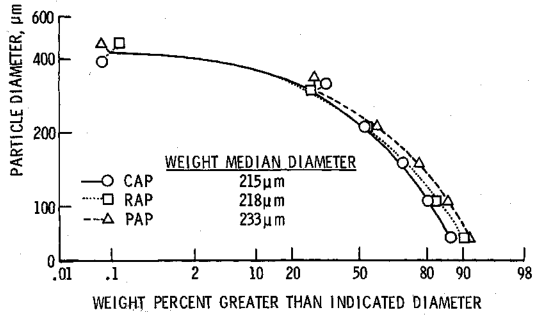

Fig. 1 Coarse oxidizer particle size distributions.

The particle size distributions of the prepared oxidizers were purposely matched in order that changes in ballistics could be evaluated without the influence of particle size effects. All other controllable propellant processing variables were maintained constant to minimize batch variations.

Table 1 Fine oxidizer size characteristics

Weight % less than indicated diameter	Diameter, μm CAP	RAP	PAP
84.1	45	44	39
50	20.6	20.5	20.0
15.8	9.6	9.0	9.7
Log-normal standard deviation	2.17	2.21	2.01

The moisture of all three oxidizers was high due to the lack of adequate drying. The total moisture in the fine materials was approximately 0.044% with about 0.12% in the coarse oxidizers. It was felt that the moisture should have little effect on the differences in propellant ballistic properties' tests.

The LTD rates of the three prepared lots of oxidizers were characterized by isothermal gravimetric analysis (TGA) at 158°C, 760 torr. These test results are shown in Fig. 2 for the coarse and fine fraction of the oxidizers as used in the three propellants. There was a tenfold difference in LTD weight loss rate between the CAP and PAP oxidizers. The RAP oxidizer that was used as a control had a LTD rate between PAP and CAP. Figure 2 also shows the decomposition of a PAP coarse sample that was ground in the laboratory to detect differences in composition that may have arisen due to initial oxidizer classification. Chemical analysis of the oxidizers gave 0.15 and 0.18% PO_4 by weight for PAP and 0.032 and 0.029% ClO_3 in CAP, for the coarse and fine fractions, respectively. The concentration of potassium ion in CAP was not measured, and thus it was not possible to determine whether or not measurable secondary burning-rate effects might be present, based on the K^+ doped single AP crystal burning-rate data of Boggs, Price, and Zurn.[13]

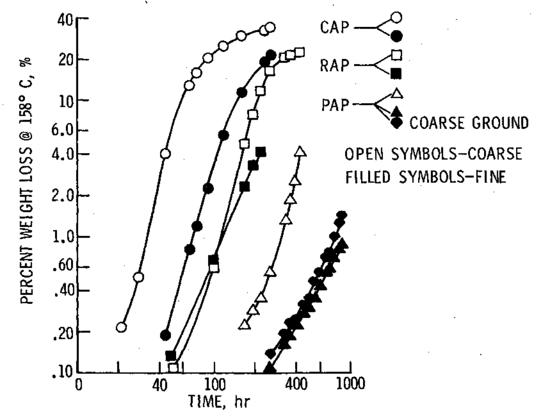

Fig. 2 Isothermal low-temperature decomposition of the three treated oxidizers at 158°C.

The propellant composition for the 82% solids nonaluminized CTPB/MAPO/EPOXY propellant used is given in Table 2.

Table 2 Propellant composition

Ingredient	Weight %
HC-434 polymer	14.57
MAPO	0.39
ERLA 0510	0.34
DOA	2.70
Carbon	0.20
Fine oxidizer	28.63
Coarse oxidizer	53.17

Results and Discussion

The primary objective of this investigation was to clarify the role of AP LTD rate on the ballistics of a nonaluminized CTPB propellant. Ballistic tests were made to determine strand burning rates, $\dot{P}$ extinction characteristics, low-pressure deflagration limit, temperature sensitivity of burning rate, and the ignition characteristics of the three propellants.

Ballistic Tests

Strand burning rates

The strand burning rates at 75°F of the propellants made from the three specially prepared oxidizers were measured at pressures from 5–1815 psia using a Crawford bomb. These data were taken to determine, for these propellants, the possible effect of AP LTD rate on propellant regression. A comparison of the burning rate for the three formulations is shown in Fig. 3. The data points

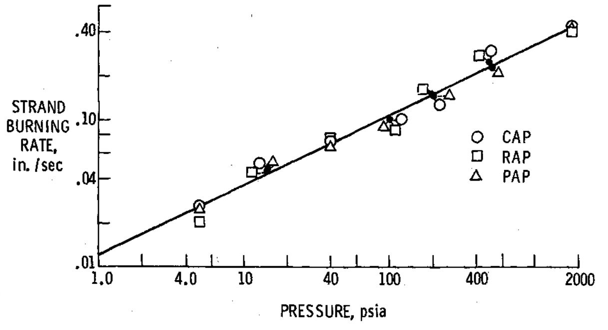

Fig. 3 Strand burning-rate data. Small filled circles indicate actual locations of coincident data points.

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§ The weight percents of phosphate and chlorate added to the origional AP solutions, on a total solids basis, were 3.846 and 0.552%, respectively.

represent the average from two or more burns at a given pressure level, with 10 measurements for each propellant at 500 psia. The line shown is the least-squares line through all the data. The burning-rate equation $r = 0.0118\,p^{0.477}$ described the grouped burning-rate data for all the propellants with a correlation coefficient of 0.99907. The only data not correlating well were an unexplained reduction in the RAP propellant rate at 5 psia. Thus, an order-of-magnitude difference in the LTD rate of the oxidizer had no effect on the strand burning rate of the three CTPB propellants made with AP having closely controlled particle size distributions.

$\dot{P}$ extinction characterization

One of the primary objectives of this work was to determine the role of the LTD in the depressurization extinction ($\dot{P}$) process. It was thought that thermal relaxation in the solid leading to variable exothermic decomposition of these oxidizers might have a significant effect on extinction as determined by critical initial depressurization rates. Or even more likely, reducing the LTD should minimize the tendency for reignition.

The extinction tests were conducted in a slab burning motor with a swing nozzle to allow rapid increases in throat area to a predetermined size. The motors had a chamber volume of 71 in.^{3} and used propellant slabs nominally 4 in. wide by 6 in. long with $\frac{1}{2}$ in. thick webs.

Since differences in the extinction characteristics of the three propellants were the parameter sought, $\dot{P}$ tests were conducted with the primary nozzle sized to give chamber pressures of 550 psi, and there was no attempt to characterize the dependence of extinction on initial chamber pressure. The motors were burned approximately 1.0 sec before activation of the explosive bolts that allowed the primary nozzle to swing immediately out of position, permitting the gases to be exhausted through only the larger stationary secondary nozzle. By changing the diameter of the stationary nozzle, the initial depressurization rate was varied until the critical $\dot{P}$ was established. The data presented in Fig. 4 show that there was no significant difference in the critical depressurization rate for the three propellants. All propellants required about 80 kpsi/sec for extinction. However, since there were no tests where motor reignition occurred, this technique did not aid in establishing the effect of LTD on reignition phenomena.

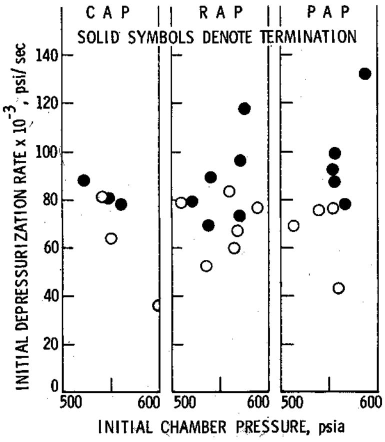

Fig. 4 Depressurization extinction test results.

The absence of reignition was probably a result of minimum hardware heating associated with the 1 sec burn time.

Low-pressure deflagration limit tests

The low-pressure deflagration limit was also investigated as part of the program to establish extinction characteristics of the treated propellants. The propellants were ignited in a small pressure vessel connected to a large evacuated tank through a regulating valve. After ignition, and when the pressure in the small vessel began to rise, a quick opening valve was activated venting the combustion gases through the regulation valve at a predetermined rate of 2 psi/sec. A photocell viewing the burning surface was used to detect extinction as the pressure was reduced. The pressure in the small chamber when the photocell output decreased and approached zero was taken to be P_{dl}.

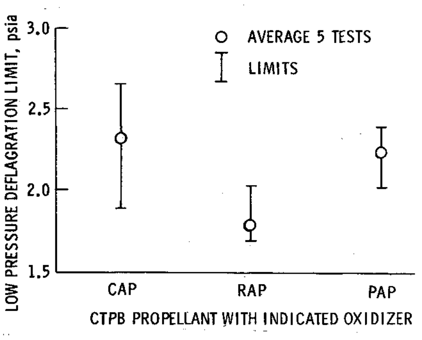

Fig. 5 Low-pressure deflagration test results.

The data from these tests for the three propellants are shown in Fig. 5, with the average of five tests and the maximum and minimum limits shown. The CAP and PAP propellants had nearly equal P_{dl}. The P_{dl} of the RAP propellant was somewhat lower, but remained nearly within the scatter of the other propellants. These tests should have exaggerated the effects of the LTD since at these low-burning rates the thermal gradient was relatively shallow, allowing maximum time under combustion conditions for the LTD to be effective. Thus, for these propellants, the LTD rate of the AP had no direct correlation with the measured P_{dl}.

Temperature sensitivity tests

The temperature sensitivity σ_p of the treated propellants was measured at 500 psia by conditioning propellant samples at 5°, 75°, and 150°F and measuring the strand burning rate. Four strands were burned at each temperature extreme, and as reported earlier, 10 were burned at 75°F. The σ_p was evaluated by fitting a least-squares linear equation through the rate vs temperature data and using the slope to evaluate the equation $\sigma_p = [\partial(\ln r)/\partial T_0]_p$.

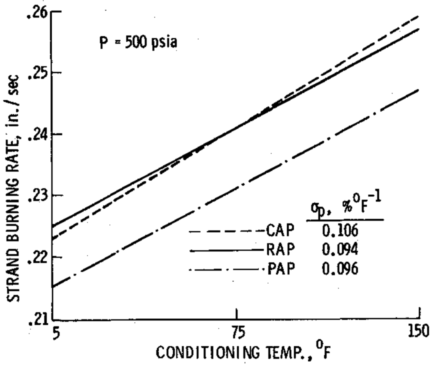

Fig. 6 Temperature sensitivity data.

The least-squares-fitted burning rate vs conditioned temperature data are plotted in Fig. 6 with the data points omitted for clarity. Also shown are the tabulated σ_p data. The effects of AP LTD on the temperature sensitivity of these CTPB propellants were relatively small. The σ_p for the CAP propellant was 13% higher than that of the RAP control, but the stabilized PAP showed only a 2% reduction in σ_p. Thus, it appears that increased LTD rate associated with the CAP propellant may have increased the temperature sensitivity slightly; however, a more thorough statistical analysis on the confidence limits of $\partial r/\partial T$ should be made due to the strong influence of rate variations and the limited number of tests.

Ignition tests

The ignition delay in air at one atmosphere for the three propellants was evaluated using a CO_2 laser ignition source. It was felt that at low heat fluxes and long ignition delay times any effect of LTD on propellant ballistics would surely be exaggerated. The unfocused 10.6-μm wavelength beam (15 mm diam) from the CO_2 laser was reflected by a wobbling plate beam scrambler to minimize hot spots on the propellant. The effective spot size and the flux distribution at the propellant plane were determined by radiating unexposed polaroid film and adjusting the beam scrambler to give nearly uniform heating, as evidenced by the film. The incident heat flux was varied from 4–14 cal/cm^2 sec.

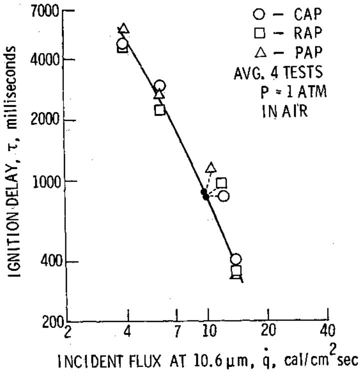

Fig. 7 CO_2 laser ignition test results.

The ignition delay time is plotted as a function of incident flux in Fig. 7. The data points shown for each propellant are the average data from four tests at that condition. The least-squares slope of the log τ vs log q data was -1.98 which corresponds well with the classical value of -2 for the asymptotic case of heterogeneous ignition at low-radiant flux, where heat conduction is dominant in determining the time to critical surface temperature. Again, the tenfold difference in the LTD rate was not reflected as variations in ignition delay, even at the low-incident heat flux of 4 cal/cm^2 sec.

Conclusions

The influences of low-temperature decomposition (LTD) on propellant burning rate, ignition, and extinction were evaluated on three propellants with oxidizers differing widely in LTD rates. The initial LTD of ammonium perchlorate (AP) oxidizer was retarded by cocrystallization with $(NH_4)_2HPO_4$ (0.16% included PO_4 by weight) and accelerated by cocrystallization with $KClO_3$ (0.03% included ClO_3). Isothermal weight loss measurements at 158°C, 760 torr, indicated a tenfold difference between the LTD weight loss rates of the treated oxidizers. It was anticipated that measurable differences in the ballistics of the nonaluminized CTPB/MAPO/EPOXY propellants should be found if the low-temperature condensed-phase decomposition of AP was a rate-determining factor in propellant deflagration.

The CTPB propellants made from these oxidizers with nearly identical particle sizes showed little effect of the wide differences in LTD rate on 1) strand burning rate, 2) critical $\dot{P}$ depressurization rate, 3) low-pressure deflagration limit, 4) temperature sensitivity of burning rate at 500 psi, and 5) ignition delay using a CO_2 laser radiation source.

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