16:19:30 OCA PAD AMENDMENT - PROJECT HEADER INFORMATION 04/25/91

Active

Project #: E-16-642 Cost share #: Rev #: 10
Center # : R6326-0A0 Center shr #: OCA file #:
Work type : RES
Contract#: 410386 Mod #: CHANGE ORDER 05 Document : PO
Prime #: F04611-86-C-0085 Contract entity: GTRC

Subprojects ? : N
Main project #:

Project unit: AERO ENGR Unit code: 02.010.110
Project director(s):
PRICE E W AERO ENGR (404)894-3063

Sponsor/division names: AEROJET PROPULSION CO /
Sponsor/division codes: 200 / 008

Award period: 870501 to 910228 (performance) 910228 (reports)

Sponsor amount	New this change	Total to date
Contract value	0.00	158,455.77
Funded	0.00	158,455.77
Cost sharing amount		0.00

Does subcontracting plan apply ?: N

Title: COMBUSTION STUDIES ON JET PROPELLANTS

PROJECT ADMINISTRATION DATA

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Security class (U,C,S,TS) : U
Defense priority rating :
Equipment title vests with: Sponsor

ONR resident rep. is ACO (Y/N): N
supplemental sheet
GIT

Administrative comments -
CHANGE ORDER #05 PROVIDES A NO-COST EXTENSION TO 2/1/91 FOR THE DRAFT FINAL REPORT AND TO 2/28/91 FOR THE FINAL REPORT.

GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION

NOTICE OF PROJECT CLOSEOUT

Closeout Notice Date 07/10/91

Project No. E-16-642 Center No. R6326-0A0

Project Director PRICE E W School/Lab AERO ENGR

Sponsor AEROJET PROPULSION CO/

Contract/Grant No. 410386 Contract Entity GTRC

Prime Contract No. F04611-86-C-0085

Title COMBUSTION STUDIES ON JET PROPELLANTS

Effective Completion Date 910228 (Performance) 910228 (Reports)

Closeout Actions Required:	Y/N	Date Submitted
Final Invoice or Copy of Final Invoice	Y	910228
Final Report of Inventions and/or Subcontracts	Y	
Government Property Inventory & Related Certificate	N	
Classified Material Certificate	N	
Release and Assignment	N	
Other	N	

Comments

Subproject Under Main Project No.

Continues Project No.

Distribution Required:

Project Director	Y
Administrative Network Representative	Y
GTRI Accounting/Grants and Contracts	Y
Procurement/Supply Services	Y
Research Property Managment	Y
Research Security Services	N
Reports Coordinator (OCA)	Y
GTRC	Y
Project File	Y
Other	N
	N

NOTE: Final Patent Questionnaire sent to PDPI.

Contract Title: COMBUSTION STUDIES OF JET PROPELLANTS

COMBUSTION OF EMULSION PROPELLANTS

E. W. Price
Georgia Institute of Technology
Atlanta, GA 30332

Monthly Progress Report No. 1
Aerojet Contract P. O. #410386
28 July 1987

I. Introduction

This is a brief summary of progress on Aerojet Contract P. O. #410386 for July 1987. Personnel involved are Dr. R. K. Sigman (Senior Research Engineer), Mr. Christos Markou (Graduate Research Assistant), and Prof. E. W. Price (Principal Investigator).

II. Objective

This research is for evaluation and improvement of the combustion characteristics of emulsion propellants presently under development by ASPC. The approach uses laboratory scale combustion tests such as the flushed window bomb to determine ignition and burning rate characteristics, nature of the burning surface, and combustion behavior of the aluminum ingredient. Actual test sequences will be determined partly by the program of formulation work at Aerojet and receipt of propellant samples. Concurrent efforts are being made to produce dry-pressed samples based on AN that can be used as an inexpensive medium for exploration and modification of aluminum combustion in AN systems.

III. Summary

Combustion tests were made on a variety of combinations of AN-wax-Al samples in N_2, methane and air environments without achieving self-sustained burning. Tests were also made on an emulsion propellant (vintage uncertain). Combustion at 1000 psi in N_2 was not achieved; combustion in 1000 psi in air was achieved. Extensive agglomeration of aluminum occurred on the burning surface, accompanied by ignition and burning after detachment from the propellant surface. Because of

the air atmosphere, progress of aluminum combustion in the propellant product flow could not be evaluated.

IV. Technical Discussion

1. Aside from a trip to a consortium meeting on May 20, 1987, this is the first month of research. Activity was concerned with expected difficulties in achieving small sample burning in the window bomb.

2. Initial tests were made on samples of ammonium nitrate formed by dry-pressing powders at 19,700 psi. Test samples were rectangular, nominally 2 mm thick by 1 cm by .5 cm, "ignited" on a 2 mm by .5 cm end. Several arrangements were tested, with photographic coverage to observe sample response. Ignition was by hot wire aided with an AP glue paste. Arrangements included:

a) An edge-burning "sandwich", consisting of a layer of BAMO-THF between a lamina of dry-pressed AP and a lamina of dry-pressed AN, at 1000 psi in an N_2 flushed chamber
b) An AN lamina at 1000 psi in a methane-flushed chamber
c) An AN lamina containing 5% carnauba wax and 10% Al, at 1000 psi in an air-flushed chamber

Combustion of the AN did not proceed appreciably in these tests. A glossy surface indicative of AN melt was evident on heated surfaces and superficial decomposition may have occurred. Efforts will continue to obtain dry-pressed samples that will burn on their own or in suitable gaseous atmospheres in order to provide a convenient means to start studies of aluminum combustion in nitrate systems.

3. Several small samples of an emulsion propellant were available, and tests were run under various conditions to achieve self-sustained combustion. Sample size used was roughly 3 mm x 1 cm x 1.5 cm. Test conditions were as follows:

a) End burning arrangement at 1000 psi in an N_2 flow
b) "Sandwiched" between two thin layers of an AP-Al-PBAN propellant, ignited on an edge, at 1000 psi in N_2
c) End burning in an air-flushed chamber at 1000 psi (several tests)

In a), the sample did not burn. In b), the AP propellant burned away but the test propellant did not self-sustain. In c), the test propellant burned.

Results are described in more detail in an attachment and in an accompany video tape. Burning rate was around 2 mm/sec.

V. Future Work

Progress to date is largely exploratory to determine suitable strategies for conduct of meaningful small scale tests on a propellant system that is expected to have "reluctant" combustion characteristics. Tests should provide enough detail of the combustion process to guide design for better experiments and future modification of formulation for improved combustion (as gauged by ignition and burning rate characteristics and by combustion efficiency). Initial efforts will continue to use the combustion window bomb with video camera coverage, and microscopic examination of quenched surfaces and recovered solid residue (particularly Al and Al_2O_3). Two parallel approaches will continue: testing of ASPC-supplied propellant samples, and formulation and test of AN-based samples at GIT. During the coming months, a series of tests will be made on AN-AP-wax-AL samples, proceeding by incremental replacement of AP by AN to determine how the burning surface and metal behavior are affected by AN (an extensive database already is available on the AP-wax-Al system). Future testing will be guided by these results and by availability of new propellant samples of ASPC.

7/28/87
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Enclosures:
1) Attachment to Monthly Progress Report No. 1
2) Scanning Electron Micrograph (magnification 30x)
3) Video Cassette (VHS)

Attachment to Monthly Progress Report No. 1

The enclosed video cassette includes three burning tests of the Aerojet propellant.

The tests were run in the combustion bomb pressurized to 1000 psi with air. There was no combustion with samples ignited at 500 psi in air or 1000 in N_2. The propellant didn't burn even when it was placed between two aluminized propellant slabs and ignited at 1000 psi in nitrogen.

All of the samples were ignited from the top using a fuse wire which ignites the ignition paste coating the top of the ignition sample. This method has been used in previous experiments in the combustion bomb and has been proved very effective.

Excess smoke was observed during the ignition phase, due to the combustion of the ignition paste. Dark smoke was observed during the extinction of the burning which came from the combustion of the epoxy used to glue the propellant sample on the propellant holder.

After the completion of the burning, the residue of the sample was shaking. This came from the gas (air) flow flushing the bomb in order to clean the exhaust piping system from any toxic gases.

Residue of the samples were observed in all cases; they were collected and examined under the microscope (optical and scanning electron).

All samples burn faster at the edges because the air flow surrounding them.

In all cases, individual Al particles coalesced to form agglomerates. Small agglomerates leave the surface faster and their white smoke trails can be seen on the video. Large agglomerates are orange in color and they become white when they leave the surface and burn in air. In some cases, those agglomerates remain on the sample and burn there. These can easily be seen as grey spheres sitting on the residue. It was noticed also that first the combustion wave passes and then the Al agglomerates and burns.

A bright orange sphere that remains on the upper part of the screen comes from the ignition wire and some Al particles that agglomerate on it. It should be ignored.

7/28/87
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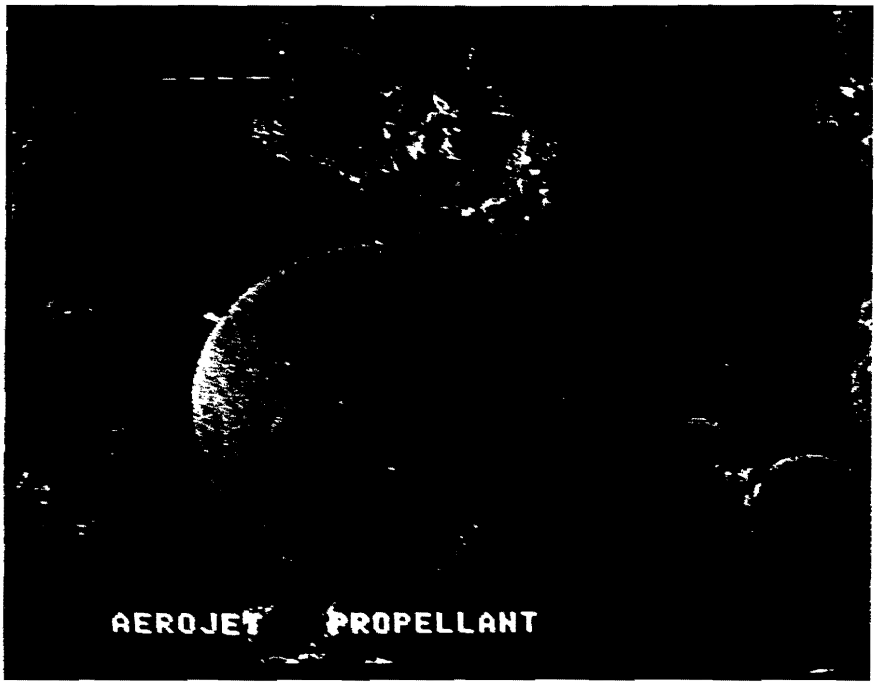

Typical Al agglomerates left in window bomb.
Large agglomerate is 1.4 mm in diameter.

Contract Title: COMBUSTION STUDIES OF JET PROPELLANTS

COMBUSTION OF EMULSION PROPELLANTS

E. W. Price
Georgia Institute of Technology
Atlanta, GA 30332

Monthly Progress Report No. 2
Aerojet Contract P. O. #410386
28 August 1987

I. Introduction

This is a brief summary of progress on Aerojet Contract P. O. #410386 for August 1987. Personnel involved are Dr. R. K. Sigman (Senior Research Engineer), Mr. Christos Markou (Graduate Research Assistant), and Prof. E. W. Price (Principal Investigator).

II. Objective

This research is for evaluation and improvement of the combustion characteristics of emulsion propellants presently under development by ASPC. The approach uses laboratory scale combustion tests such as the flushed window bomb to determine ignition and burning rate characteristics, nature of the burning surface, and combustion behavior of the aluminum ingredient. Actual test sequences will be determined partly by the program of formulation work at Aerojet and receipt of propellant samples. Concurrent efforts are being made to produce dry-pressed samples based on AN that can be used as an inexpensive medium for exploration and modification of aluminum combustion in AN systems.

III. Summary

Combustion tests were carried out on a matrix of samples consisting of AP-AN mixes and AP-AN-Al-Wax mixes in a nitrogen environment. Dry pressed mixes in various ratios of AP and AN were prepared and ignited in the high pressure window bomb at 1000 psi. Combustion behavior was observed with a video camera using external illumination of the samples. Combustion was achieved for mixes of up to 10% AN. The burning was observed to be more uneven and the measured burning rate was observed to be more erratic as the amount of AN was increased from zero to

ten percent. Similarly, AP-AN-Al-Wax mixtures were dry pressed with fixed ratios of 10% Al and 5% was. The AP-AN mixture was varied from 80/5 to 0/85 in steps of 5 percent. Uneven (but complete) combustion was achieved for a 60/25 mixture (71/29 oxidizer ratio). The 50/35 mixture self extinguished after burning 3 mm.

IV. Technical Discussions

1. Ammonium perchlorate (AP) burns as a monopropellant at pressures greater than 300 psi. Ammonium perchlorate powder was dry pressed at 19,700 psi in a steel die and samples were cut to form an orthogonal parallelpiped 2 mm thick, 1 cm long and .5 cm wide. The samples were ignited in the high pressure movie bomb flushed with nitrogen at 1000 psi. Burning rates were measured using a video replay system. The burning surface was observed to be uniform and planar with burning rates of 7.992 mm/sec and 8.07 mm/sec in two tests. The variation was less than 1 percent.

Mixtures of 95/5, 90/10, and 85/15 AP-AN were mixed and dry pressed as described above. The burning surface of the 95/5 mixture was slightly un-uniform (non planar) and burning rates of 7.6 and 7.9 mm/sec were measured (a 4% variation). The 85/15 produced a very non planar (irregularities up to 2 mm) burning surface and variations of 5% in the burning rates (7.6 and 7.22 mm/sec). The 85/15 blend did not sustain combustion.

2. Dry pressed mixtures of AP-AN-Al-Wax were prepared by mixing the powder and dry pressing as previously described. In these blends the aluminum and carnauba wax were held fixed at 10% and 5% of the total weight. These blends are used to simulate a normal propellant with the carnauba wax taking the place of the polymeric binder. Results of attempts to achieve steady state combustion in the nitrogen flushed bombs at pressures of 500 and 1000 psi are shown for the various mixtures in Table 1. As noted earlier a 60/25/10/5 mixture of AP/AN/Al/Wax burned (unevenly) while a 50/35/10/5 mixture did not sustain combustion. In all cases in which combustion was achieved, the ignition of aluminum appeared to be very good.

3. Carnauba wax has been used in this and other studies as a substitute for the polymeric binder since it is a dry powder which, when dry pressed, is

easily deformed to fill voids around oxidizer particles and forms a geometric microstructure similar to a real propellant. It has been found to be a very good fuel for igniting aluminum. In other respects it is probably a poor fuel for oxidizers such as AN since the wax has a low melting point and produces a liquid melt much as AN does. Polymeric binders such as HTPB which decompose without forming liquids would be better, but require long curving times and are difficult to blend in small laboratory batches.

4. It is known that AN is very hydroscopic and indeed, samples left overnight in a dish were found to be dissolved by atmospheric humidity. Thus, great care has been taken to reduce exposure time and keep AN in a desiccator when not in use. Advice from Russ Reed at the Naval Weapons Center at China Lake has lead to a procedure for coating AN to prevent moisture absorption. This procedure will be evaluated in the future.

V. Future Work

Experiments to date have been largely exploratory, seeking a reasonable range of mixture ratios for standard propellants with AN substituted for AP. Such experiments will provide experience in the special problems of AN and help develop strategies for handling this difficulty. In particular, developing the coating procedures for protecting the AN from moisture, and investigation of "dry" polymeric binder substitutes as well as "dry" binders will occupy much of the efforts until new propellant samples are available from ASPC.

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Table 1

Results for Dry Pressed AP/AN/Al/Wax Samples
at 500 and 1000 psi in Nitrogen

AN	AP	Al	Wax	500 psi *	1000 psi *
85	0	10	5	NB	NB
75	10	10	5	NB	NB
65	20	10	5	NB	NB
55	30	10	5	NB	NB
45	40	10	5	NB	NB
35	50	10	5	P	P
25	60	10	5	B (Uneven)	B (Uneven)
15	70	10	5	B	B
5	80	10	5	B	B

* NB: No burn

P: Partial (3 mm or less) burn

B: Burned to completion

Contract Title: COMBUSTION STUDIES OF JET PROPELLANTS
COMBUSTION STUDIES OF EMULSION PROPELLANTS

E. W. Price
Georgia Institute of Technology
Atlanta, GA 30332

Monthly Progress Report No. 3
Aerojet Contract P. O. #410386
28 September 1987

I. Introduction

This is a brief summary of progress on Aerojet Contract P. O. #410386 for September 1987. Personnel involved are Dr. R. K. Sigman (Senior Research Engineer), Mr. Christos Markou (Graduate Research Assistant), and Prof. E. W. Price (Principal Investigator).

II. Objective

This research is for evaluation and improvement of the combustion characteristics of emulsion propellants presently under development by ASPC. The approach uses laboratory scale combustion tests such as the flushed window bomb to determine ignition and burning rate characteristics, nature of the burning surface, and combustion behavior of the aluminum ingredient. Actual test sequences will be determined partly by the program of formulation work at Aerojet and receipt of propellant samples. Concurrent efforts are being made to produce dry-pressed samples based on AN that can be used as an inexpensive medium for exploration and modification of aluminum combustion in AN systems.

III. Summary

Efforts this month have been directed toward obtaining suitable "dry powder" binders to replace carnauba wax in dry-pressed AN-Al-binder propellant samples, and to implementing procedures for coating ammonium nitrate (AN) to reduce hydroscopic effects, and, finally, to obtaining suitable catalysts for improving combustion of AN propellants. Substitutes for carnauba wax have been obtained in the form of 1) Acrylonitrile/Butadiene/Styrene resin (a fine powder); 2)

Acrylonitrile/Butadiene Copolymer (a coarse granular mixture), and 3) Polyacrylonitrile (a coarse powder). Two methods of coating AN have been obtained from Russ Reed of the Naval Weapons Center. Materials for a silane coating and appropriate laboratory ware have been obtained and material and labware for an MgO coating are being assembled. A suitable burn rate stabilizer, sodium barbiturate, is also being sought.

IV. Technical Discussions

1. Dry pressed mixtures of oxidizer, aluminum and binder represent a fast and consistent method of preparing propellant samples. Oxidizers such as AP and AN are powders (as is the aluminum) and both are used just as in a full scale rocket motor. Preparation of small samples of polymeric binder are often difficult, trapped air bubbles are difficult to remove and curing times are often too long. Thus, it is convenient to substitute hydrocarbon in a dry powdered form which can be mixed with the oxidizer and aluminum, loaded into a steel die and dry pressed to 35,000 psi in a hydraulic press for 1 hour or longer. Previous experience with carnauba wax has shown that it is a very good binder substitute in that it deforms to fill voids between oxidizer and aluminum and is a good igniter of aluminum. Unfortunately, it has a low melting temperature which is somewhat undesirable for AN propellants. Of the three hydrocarbons listed in the summary, Acrylonitrile/butadiene/styrene has the best powdered form and is related to PBAN (polybutadieneacrylonitrile) a binder which is widely used in large rocket motors.

2. Ammonium nitrate is very hydroscopic and dissolves in relative humidities greater than 50 percent. Furthermore, since AN is a salt of a strong acid (nitric) and a weak base (ammonium hydroxide), moisture causes AN to become acidic. This combination of solubility and acidity inhibits the curing of some binders and samples prepared for viewing in the scanning electron microscope degrade in hours. Thus, much care has been taken to keep samples desiccated.

3. A more practical long range approach is to coat the AN powdered so that it is impervious to normal humidity effects. Two coating procedures have been supplied by Russ Reed of the Naval Weapons Center, one using silane and the other using MgO as coating. The preferred silane coating requires AN and .5% silane to

be mixed in a solution of Freon TF. After stirring, the mixture is filtered through a Buchner funnel and the AN is vacuum dried.

4. An additional requirement for satisfactory combustion of An propellants is the addition of 1% sodium barbiturate. Russ Reed maintains that the addition of sodium barbiturate is essential to reducing the burning rate exponent and the achievement of consistent burning rate data.

V. Future Work

Progress in improving combustion characteristics of ammonium nitrate by coating and by addition of sodium barbiturate will continue during the next month. New dry powder binder substitutes will also be evaluated. The majority of efforts will be devoted toward combustion photography of samples of a development propellant supplied by ASPC. Burning rates at 500 psi and 1000 psi will be measured and the combustion efficiency of the aluminum (tendency to form large agglomerates, reluctance to ignite, etc.) will be evaluated.

9/28/87
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Contract Title: COMBUSTION STUDIES OF JET PROPELLANTS
COMBUSTION STUDIES OF EMULSION PROPELLANTS

E. W. Price
Georgia Institute of Technology
Atlanta, GA 30332

Monthly Progress Report No. 4
Aerojet Contract P. O. #410386
29 October 1987

I. Introduction

This is a brief summary of progress on Aerojet Contract P. O. #410386 for October 1987. Personnel involved are Dr. R. K. Sigman (Senior Research Engineer), Mr. Christos Markou (Graduate Research Assistant), and Prof. E. W. Price (Principal Investigator).

II. Objective

This research is for evaluation and improvement of the combustion characteristics of emulsion propellants presently under development by ASPC. The approach uses laboratory scale combustion tests such as the flushed window bomb to determine ignition and burning rate characteristics, nature of the burning surface, and combustion behavior of the aluminum ingredient. Actual test sequences will be determined partly by the program of formulation work at Aerojet and receipt of propellant samples. Concurrent efforts are being made to produce dry-pressed samples based on AN that can be used as an inexpensive medium for exploration and modification of aluminum combustion in AN systems.

III. Summary

Primary emphasis this month has been placed on video taping combustion of an ASPC emulsion propellant (C728-46C) in the flushed window bomb. Video records of combustion in nitrogen at 500 psi (two samples) and 1000 psi (two samples) were evaluated and burning rates measured. Average burning rates were .0594 in/sec at 500 psi and .0936 in/sec at 1000 psi. Residual aluminum filigrees from incomplete combustion of aluminum were coated with a black carbonaceous residue.

A sample burned at 1000 psi in air gave a higher burning rate (.15 in/sec) and the residual aluminum filigrees did not have any evidence of the black residue. A sixth sample was prepared by pressing ammonium perchlorate into the surface of the emulsion propellant. The burning rate in nitrogen at 1000 psi was slightly higher and the aluminum formed large glowing agglomerates near the edges where the AP was located.

Progress in the development of representative "dry powder" propellants included locating sources for sodium barbiturate, ordering a supply and grinding a batch (10 gms) of AN and coating the ground AN with 6020 silane.

IV. Technical Discussions

1. An experimental emulsion propellant marked C728-46C on the aluminum foil wrapper and marked AAB-0364 on the plastic container was received from Aerojet and stored in a desiccator. Samples averaging about 11 mm tall, 6 mm wide and 2.5 mm thick were cut from the slab, mounted in the flushed window bomb, and video taped while burning at pressure. Burning rates in a nitrogen atmosphere were estimated to be .0593 in/sec and .0595 in/sec at 500 psi (2 samples) and .0951 in/sec and .0921 in/sec at 1000 psi (2 samples). The residual filigrees of unburned aluminum were fairly rigid and exhibited evidence of a black carbonaceous residue. No aluminum combustion was evident.

2. A sample burned in air at 1000 psi revealed luminous flames at the interface between the propellant gases and the flushing air flow and a substantially higher burning rate (.1513 in/sec). The aluminum did not ignite. There was no evidence of the carbonaceous residue, and the aluminum was swept away by the flushing flow.

3. A sixth sample was prepared by sprinkling 200 micron AP into a steel die, placing a thin (1.2 mm) layer of sliced emulsion propellant on the AP and sprinkling AP on the second side of the propellant. The die was sealed and pressed to 500 psi to embed the AP in the surface of the propellant. Combustion of this sample in nitrogen at 1000 psi produced a slightly elevated burning rate (.0985 in/sec). Aluminum near the AP glowed and formed large agglomerates, but did not detach and burn vigorously.

4. A sample of Titan III propellant, a standard aluminized propellant, was recorded for comparison.

5. Sodium barbiturate, an additive recommended by Russ Reed of the Naval Weapons Center as necessary to provide consistent burning rates, is not stocked by large chemical houses and must be ordered from specialist suppliers. Furthermore, it requires a letter of intent be filed before completion of the order. One hundred grams of sodium barbiturate were ordered from Eastern Chemicals, 230 Marcus Blvd., Hauppauge, NY 11788.

V. Future Work

Research into the effects of the silane coating on the ammonium nitrate, use of sodium barbiturate, and the effectiveness of the powdered binder substitutes will be resumed. Methods of improving aluminum ignition will also be explored.

10/29/87
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Contract Title: COMBUSTION STUDIES OF JET PROPELLANTS
COMBUSTION STUDIES OF EMULSION PROPELLANTS

E. W. Price
Georgia Institute of Technology
Atlanta, GA 30332

Monthly Progress Report No. 5
Aerojet Contract P. O. #410386
19 November 1987

I. Introduction

This is a brief summary of progress on Aerojet Contract P. O. #410386 for November 1987. Personnel involved are Dr. R. K. Sigman (Senior Research Engineer), Mr. Christos Markou (Graduate Research Assistant), and Prof. E. W. Price (Principal Investigator).

II. Objective

This research is for evaluation and improvement of the combustion characteristics of emulsion propellants presently under development by ASPC. The approach uses laboratory scale combustion tests such as the flushed window bomb to determine ignition and burning rate characteristics, nature of the burning surface, and combustion behavior of the aluminum ingredient. Actual test sequences will be determined partly by the program of formulation work at Aerojet and receipt of propellant samples. Concurrent efforts are being made to produce dry-pressed samples based on AN that can be used as an inexpensive medium for exploration and modification of aluminum combustion in AN systems.

III. Summary

During this shortened reporting period (due to the Thanksgiving holiday), experiments using dry pressed ingredients were resumed. A more realistic powdered binder was used, as was silane coated AN, a burning rate stabilizer and an aluminum combustion enhancer (thermite). In Report No. 2, a dry pressed propellant of 60% AN, 25% AP, 10% Al and 5% powdered carnauba wax was ignited at 1000 psi, but did not burn uniformly. Samples with increasing ratios of AP to AN burned with increasing uniformity and efficiency, while samples with higher AN

loadings did not burn. This marginal formulation was chosen as the baseline for evaluation of new binders and additives and it was found that replacement of the carnauba wax by 5% acrylonitrile/butadiene/styrene (ABS) produced stable combustion but with substantially more aluminum agglomeration than with wax. The surface profile was somewhat irregular, but it is not clear whether this is due to combustion problems or to inconsistent particle sizes. Substitution of silane coated AN (with ABS binder) appeared to produce a more even burning surface as did the addition of 1% sodium barbiturate (an AN burning rate stabilizer) and B_2O_3 (a thermite ingredient which is thought to improve aluminum combustion).

Substitution of ABS as the binder permits higher AN loadings and produces aluminum agglomeration as would be expected in a real propellant with a cured polymeric binder. The series of tests described in Report No. 2 will be repeated in order to eliminate particle size abnormalities and determine the combustion limits of ABS propellants.

IV. Technical Discussions

1. In Report No. 2, a series of tests was reported using 85% oxidizer, 10% aluminum, and 5% dry carnauba wax. The 85 percent oxidizer was varied from 85% AN and 0% AP to 5% AN and 80% AP. It was found that at about 25% AN and 60% AP samples ignited at 500 or 1000 psi burned very erratically with a highly irregular burning surface. Increasing the percentage of AP led to even more combustion. Aluminum combustion appeared to be quite good even at higher AN loadings.

Tests conducted during this period substituted powdered ABS for the carnauba wax, leading to stable combustion at 1000 psi for the borderline value of 25% AN and 60% AP. The aluminum appeared to agglomerate and produce larger burning particles leaving the surface. The burning surface was somewhat irregular.

2. Using this baseline (25% AN, 60% AP, 10% Al and 5% ABS), three additional samples were pressed substituting in turn 25% silane coated AN, 1% sodium barbiturate, and 1% B_2O_3. All samples burned, and the burning surface appeared more regular, but no definite conclusions were drawn due to the acceptable behavior of the propellants using ABS. Suspected particle size irregularities in some ingredients might account for the irregular burning surface with the ABS baseline.

3. Aluminum and carnauba wax are fairly stable powders in that they do not cake or clump during a one- or two-year period when left in a sealed vessel under atmospheric conditions. Ammonium perchlorate is known to cake when exposed to the atmosphere, but retains its individual particle size when stored in a heated oven or a desiccator. Thus, our experience has been that consistent results can be obtained using these ingredients as long as the noted precautions are followed. Ammonium nitrate tends to cake even when stored in an oven or desiccator. Original particle sizes (or smaller) can be restored using a mortar and pestle. Silane coated AN particles appear to cake so that it is not clear at this point whether the silane is effective or not. The ABS appears to be stable but was not size graded at the factory. Sieving of ABS is currently underway and has produced several large (400 micron) particles which may explain the irregular burning surface.

V. Future Work

Pending the receipt of additional propellants from ASPC for evaluation, the study of dry pressed propellants will continue as an evaluation tool for improving aluminum combustion in ammonium nitrate propellants. Combustion limits for propellants with sieved ABS and AN will be determined as a function of AN/AP ratio. The effects of sodium barbiturate and B_2O_3, as well as other additives, will be evaluated for marginal cases. The effectiveness of the silane coating will be evaluated by controlled atmospheric exposure of coated and uncoated AN.

11/19/87
prrpt.aj7

GEORGIA INSTITUTE OF TECHNOLOGY
ATLANTA, GEORGIA 30332

SCHOOL OF
AEROSPACE ENGINEERING

404-894-3000

DANIEL GUGGENHEIM SCHOOL
OF AERONAUTICS

15 December 1987

TO: Dr. Randy Peeters, Manager, Advanced Technology
Chemical Research and Development
Aerojet Solid Propulsion Company

FROM: E. W. Price EWP

SUBJECT: Progress Report for December 1987 (Contract P. O. #410386)

1. Enclosed is the subject progress report. Also enclosed is a description of studies planned for the balance of the contract and a summary sheet indicating tentative scheduling. This was prepared at the request of Mr. Joe Campbell, to be used as a starting point for discussion of reorientation of effort to conform to changes in Aerojet project plans.

2. Also enclosed is a summary of the October JANNAF Workshop on AN propellant combustion. This is an informal summary, based on my notes and incomplete presentation handouts. The meeting was enlightening to me and I thought the summary would be useful to others.

EWP/ed
Enclosures: as stated
Prrpt.aj8

Contract Title: COMBUSTION STUDIES OF JET PROPELLANTS
COMBUSTION STUDIES OF EMULSION PROPELLANTS

E. W. Price
Georgia Institute of Technology
Atlanta, GA 30332

Monthly Progress Report No. 6
Aerojet Contract P. O. #410386
15 December 1987

I. Introduction

This is a brief summary of progress on Aerojet Contract P. O. #410386 for December 1987. Personnel involved are Dr. R. K. Sigman (Senior Research Engineer), Mr. Christos Markou (Graduate Research Assistant), and Prof. E. W. Price (Principal Investigator).

II. Objective

This research is for evaluation and improvement of the combustion characteristics of emulsion propellants presently under development by ASPC. The approach uses laboratory scale combustion tests such as the flushed window bomb to determine ignition and burning rate characteristics, nature of the burning surface, and combustion behavior of the aluminum ingredient. Actual test sequences will be determined partly by the program of formulation work at Aerojet and receipt of propellant samples. Concurrent efforts are being made to produce dry-pressed samples based on AN that can be used as an inexpensive medium for exploration and modification of aluminum combustion in AN systems.

III. Summary

Testing resumed on dry pressed samples with AN-Al mixtures and AN, Al, ABS and AP mixtures. Additional sieving supplies were procured to permit size grading of the ABS (acrylonitrile-butadiene-styrene) polymeric binder substitute so that large particles could be eliminated. Using consistently size graded materials, combustion tests were resumed on AP-Al-ABS-and AP mixtures. Based on the JANNAF-AN workshop, new mixture ratios were set at 20% binder (ABS), 15% aluminum and 65% oxidizer (AN, AP, or mixture). Dry pressed mixtures using AN as

the oxidizer did not ignite while mixtures using AP as the oxidizer burned very rapidly. Intermediate blends of AP and AN are currently being prepared to establish the minimum amount of AP which will sustain combustion with the goal of reduction of this minimum amount. Russian literature reports combustion of 59.7% AN, 40.3% Al mixture at atmospheric pressure. Attempts to obtain combustion of a similar dry pressed mixture at atmospheric pressure and 1000 psi were unsuccessful.

IV. Technical Discussions

1. Previous tests using ABS powdered binder have burned with an irregular surface. Examination of pressed samples revealed abnormally large particles which appeared to be ABS. Additional sieves in the 100-200 micron range were procured so that the ABS could be size graded in our sonic sifter. Sieving produced a substantial number of particles larger than 200 microns and future tests will use size graded particles.

2. Preliminary testing at GIT used 85% oxidizer (AN, AP), 10% Al, and 5% binder. Results from the JANNAF AN Workshop indicate that the lower density of AN requires a higher binder loading for good performance and stoichiometric considerations require slightly more aluminum. Thus the mixture ratio was adjusted to 20% binder (ABS), 15% Al, and 65% oxidizer. Samples prepared with AN as an oxidizer did not ignite at atmospheric pressure or at 1000 psi in nitrogen. Samples prepared using AP as an oxidizer burned very rapidly at 1000 psi in nitrogen. Intermediate blends using mixtures of AN and AP are undergoing evaluation.

3. Russian literature (Gorbunov, V. V., and A. A. Shidlovskii, "Combustion of Ammonium Nitrate and Transition-Metal Powder Mixtures," Fizika Goreniya i Vzryva, Vol. 21, No. 5, pp. 37-39) reports that a mixture of 59.7% AN and 40.3% Al at a charge density of 2 gms/cm^3 burns at 1.6 mm/sec at atmospheric pressure. Similar mixes were pressed in a steel die (which probably gave a higher charge density) but would not burn at atmospheric pressure or at 1000 psi in nitrogen. No information on particle size is given in this article, but the fact that such mixtures can sustain combustion would be encouraging.

4. In order to evaluate the silane coating on AN, samples of untreated AN and silane coated AN were left in petri dishes. Moisture contamination is evident from nitric acid formation which etches the petri dish and from changes in the crystaline form of the AN. After 3 1/2 weeks the untreated An appears more irregular in shape. Microscopic examination shows "particles" to be agglomerates of smaller transparent spheres. The silane treated AN appears more regular, but microscopic examination reveals agglomerates of opaque irregular particles. There is no evidence of acid etching.

5. Both treated and untreated AN are stored in a heated oven but continue to cake. While this caking is not necessarily due to moisture contamination, it is detrimental to maintaining a size grading of the particles.

V. Future Work

During January, tests on a simpler dry-pressed binder (ABS) and oxidizer system will resume using 1% aluminum. The trace amount of aluminum will be used to indicate ignition while primary emphasis will be given to maximizing AN fraction to obtain stable combustion at 1000 psi in nitrogen. Use of additives such as sodium barbiturate, CuO and others to maximize AN fraction will be investigated.

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Attachment A
12/15/87

COMBUSTION OF ALUMINIZED AMMONIUM NITRATE FORMULATIONS

E. W. Price
Aerojet Contract P. O. 410386

The focus of the GIT studies is on attainment of efficient aluminum combustion in ammonium nitrate formulations. This is a double problem in that AN formulations don't burn very well with or without aluminum, and AN formulations, even when they do burn, don't provide a very good environment for ignition and combustion of aluminum. The seriousness of these problems can be reduced by appropriate conditioning of the ingredient powders and by addition of other ingredients. It appears likely that satisfactory combustion can be achieved, but the studies to date have been too sporadic to develop the understanding of combustion needed to guide formulation studies. The following is an outline of studies planned to provide basic understanding of combustion of AN-Al-binder systems and application to optimization of formulation, particularly with regard to efficiency of aluminum combustion. This plan is aided by the information exchange at the recent JANNAF AN Workshop (October 1987 at AFAL), and a summary of information from that workshop is attached here for information.

1. Ammonium Nitrate Supplies. The material on hand is Fisher Certified Ammonium Nitrate. We can grind and sieve for other particle size, but water content is a problem. We have gone to vacuum drying before sample preparation, but believe appreciable moisture may be absorbed during sample preparation. Our success in burning samples has been dismal, and we believe the moisture problem must be decisively controlled before we go on to other efforts. We should know by mid-January whether present vacuum drying is sufficient. We are trying a procedure suggested by R. Reed involving silane 6020 treatment of dried powder samples that is reported to greatly reduce hydroscopicity. We also would like Aerojet to supply about 200 grams of NiO phase stabilized AN, which has relative low hydroscopicity and burns much better than pure AN.

2. Binders. Tests to date have been on formulations that had either Aerojet emulsion propellants (binders not specified) or carnauba wax (fuel powder in GIT-prepared dry pressed samples). Some acrylonitrile/butadiene/styrene

powder has been obtained and will be tested shortly as a replacement for carnauba wax (c. w. melts at a low temperature and is suspected of impeding combustion). The effect of the change in polymer fuel powder will be determined by mid-January. The powdered polymers are used because of ease of sample preparation by dry pressing, and we have gotten good combustion results via this route in the past. As the studies proceed and the choices of formulation variables narrow down, we will switch to wet-mix and cure of samples. The dry binder approach will be used early on to select appropriate formulations for evaluating selected binders that are reported to give good propellant combustion. In this connection, we would like to have Aerojet supply binder ingredients because of our limited background in the art of purchasing such ingredients and knowing what you are getting (quality spec. and control). Of immediate interest is silicone binder, which has yielded good combustion with gas generator propellants. We do not believe it is a suitable propellant binder, but do believe it may provide one vehicle for getting on with aluminum combustion studies and for identifying binder characteristics conducive to good combustion. Because of the importance of the binder for propellant mechanical, storage, and hazard properties, we will work closely with Aerojet on identification of binders that provide good performance in both combustion and mechanical areas. However, it is evident from the work to date that our (GIT's) primary concern at the outset is to find binders that provide good enough combustion to permit useful study of other ingredient variables, and binders that provide clues to the role of the binder in combustion. As progress is made, we can then work toward the combined goals of good combustion and good mechanical properties. The time when we can shift emphasis to the combined goal depends on how successful we are in the other strategies to achieve good combustion. Because of toxicity or hazard problems, some candidate binders may have to be excluded from GIT sample processing. For those binders, Aerojet may wish to supply test samples for GIT testing.

3. Combustion Aids. We do not expect to get uniform and reproducible burning samples (with or without aluminum) without combustion aids such as NiO, CuO or sodium barbiturate. The mechanisms by which these aids work are not known; some (like NiO) apparently catalyze the AN decomposition. It is not clear whether they "work" if they are mixed in the binder (as opposed to NiO in the AN crystal lattice in PSAN). We will evaluate those aids like sodium barbiturate

that can be coated on the AN or mixed in the propellant, and also those aids that are in the crystal lattice (like NiO PSAN) that Aerojet supplies. If necessary, appreciable amounts of other nitrates or of perchlorates will be used. Our first goal is to get a dry-pressed formulation that burns well enough to be a vehicle for study of aluminum combustion in nitrate systems.

Once we have a formulation that gives consistent, uniform burning over a reasonable pressure range (e.g., down to 400 psi), attention will be shifted to combustion of aluminum. There are 3 basic strategies to assuring good aluminum combustion. One is to minimize the extent of concentration or aggregation of aluminum on the burning surface, thus blocking the formation of slow burning agglomerates. The second is to modify the aluminum (before sample mixing) so that it either resists interparticle adhesion on the burning surface or ignites more easily on the surface (both effects minimize agglomerate growth). The third is to design the propellant in such a way that all surface concentrations will be exposed to high temperature flamelets or gas jetting (flamelets ignite Al concentrates, which, as with jetting, causes surface detachment before large pre-agglomerates have a chance to form). Our primary methods of observation of aluminum combustion involve small sample burning, where combustion efficiency is usually gauged by aluminum behavior near the burning surface. There remains some question as to how well aluminum will burn <u>if its near-surface behavior is bad by usual standards</u>. The rather poor combustion environment of AN propellants may still be adequate in very large motors. From results of motor tests reported at the JANNAF AN Workshop, combustion efficiencies were 5-10% lower than with AP propellants, but these motors were still small compared to a Shuttle booster motor. We believe that good aluminum combustion, as judged by small sample tests, may be necessary for good large motor performance, but that judgement is speculative and should be tested as soon as possible.

The specific combustion aids for improving aluminum combustion differ according to the three strategies noted above. <u>Blocking aggregation</u> involves choice of ingredient particle sizes so that the aluminum is not already in concentrated regions in the propellant matrix. <u>Modification of the aluminum</u> involves (for example) stronger oxide coating that blocks inter-particle adhesion, or aides to breakdown the oxide coating (e.g., with FeF_3) so the particles are more easily ignited. <u>Propellant design to aid ignition or surface release</u> before agglomeration involves provision of hot-burning particles (AP,

$KClO_4$) or jetting particles ($NaNO_3$, $NaClO_4$, EDDN) at the locations of aluminum concentration, so that concentrations will be blown or burned off the surface before formation of large agglomerates.

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Attachment B
12/15/87

OUTLINE OF PROPOSED STUDIES, TIME SCHEDULE

1. Resolution of moisture problems by end of January

   1.1 By drying
   1.2 By treatment of AN (silane)
   1.3 Use of NiO PSAN (contingent on supply by Aerojet)

2. Evaluation of binder effects

   2.1 Replacement of wax by acrylonitrile/butadiene/styrene (aps) powder (dry-pressed samples). Evaluate by mid-January.
   2.2 Use of silicone binder (wet mix samples) (contingent on supply of silicone binder by Aerojet). Evaluate by mid-February.
   2.3 Use of wet-mix samples with other binders (to be discussed with Arojet, contingent on outcome of tests with combustion aids). Evaluate by mid-April.

3. Evaluation of combustion aids--unaluminized formulations

   3.1 Sodium barbiturate (dry pressed samples). Evaluate with aps "binder" by end of January.
   3.2 CuO, copper chromite, other (dry-pressed samples). Evaluate with aps "binder" by end of January.
   3.3 NiO in the AN (dry-pressed samples). Evaluate as soon as NiO PSAN is received from Aerojet.
   3.4 Follow the best leads from 2 and 3 to samples with wet mix-cure preparation. Reach this point by 1 April or before.

4. Aluminum combustion

   4.1 Use 1% aluminum in all tests of 2,3 to determine by combustion photography whether particle ignition occurs.

4.2 Use the best-burning formulations from 2,3 to assess aluminum combustion in 18% Al samples without special measures. First tests (dry mix) by end of January.

4.3 Screen best candidates from 2,3 as they emerge using 18% AL; use exclusively dry mixes until about 1 March, depending on progress on 2.2, 2.3.

4.4 Evaluate addition of KNO_3, AP, $NaNO_3$, others in dry-pressed samples as aids to Al combustion (need to examine hazard of dry pressing). Tailor particle sizes for optimum effect. This will be in March, April, will go to wet mix-cure samples at time depending on results of 2,3.

4.5 Evaluate methods that act through modification of the protective oxide layer on Al particles ("preoxidized" Al, addition of FeF_3, addition of B_2O_3 or B). Primary effort in March, April with 4.4.

4.6 Combine results by early April to choose limited set of formulation variables for use in subsequent work (in consultation with Aerojet), including Aerojet supplied samples.

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Attachment C
E. W. Price
12/3/87

PRELIMINARY REMARKS ON JANNAF COMBUSTION WORKSHOP OF 18, 19 NOVEMBER 1987

1. A lot of work was reported, mostly AF funded, very little of it on combustion fundamentals (i.e., most or all contracts were development oriented). Generalizations of a basic nature are speculative because of limited common ground among formulations tested.

2. Three general applications areas were pursued, with varying degrees of success.

a) Propellants for gas generators--generally successful, using AN-binder-catalyst combinations. As I recall, the successful ones used silicone binders.

b) Propellants for big boosters with "non toxic" exhaust, meaning no HCl. The general goal was matching shuttle booster performance and safety, with corresponding propellant costs. Ingredients were AN-Al-medium energy binder. In some cases AP was used to improve combustion (rate, and Al combustion). Then HCl scavengers were also required. Combustion efficiencies and I_{sp}'s were lower than targets.

c) Minimum smoke propellants, in which a recent further emphasis on insensitive munitions was also a factor. Most formulations used GAP binder, influenced by an AF effort to exploit its efforts and progress on GAP. Combustion was relatively good compared to the booster formulations in b) above, and target burning rates were achieved. However, target rates and I_{sp} were generally approached by use of high energy binders, addition of HMX, etc., usually with a corresponding failure to reach desired low hazard goals. The more successful formulations had only moderate AN content.

3. Ammonium nitrate was from a variety of sources, different in different studies. Most studies used phase stabilized AN (PSAN), with different phase stabilizers and mfg. methods (NiO, CuO, K^+, others). Phase stabilization

(required in most applications to obtain acceptable properties over the service temperature range and life service history) seemed to be incompletely understood, and sources of material were not yet too practical. The most widely and successfully used PSAN was was NiO stabilized (e.g., 2% NiO level), and was obtained from a source in Germany at current costs much too high for service use. NiO is suspected of being a carcinogen in humans so NiO PSAN may not solve the toxicity problem. NiO in the AN is a good burning rate catalyst.

4. Ammonium nitrate does not burn on its own, and is very reluctant to burn in most binders (I don't recall any successful burns without catalysts). Linear pyrolysis tests and thermal analysis indicate that it melts at around 170^{o} C and that decomposition goes at 230^{o} C. If confined, it detonates at 260^{o} C (presumably depending on sample size, details of confinement). The state of the surface during burning seems to be an appreciable decomposing melt layer. Surface temp estimates are around 400^{o} C. The surface melt layers hypothesis is not uniformly endorsed, probably because the instances of successful burning are with mixtures with other ingredients on a microscale that is difficult to resolve, and where the other ingredients are present in such large proportions as to make surface conditions difficult to specify. The one exception is the gas generator propellants, where the mixture is primarily AN. Detailed studies of surface condition don't seem to have been made on any formulations, including the gas generator ones.

5. Formulations that burned smoothly were ones that had catalysts, or energetic binders, or substantial amounts of other oxidizers, or a combination of these. Low burning rate and high pressure exponents were persistent problems, but considerable variation in these properties was obtained. Effective catalysts included NiO (in the AN), lead and copper oxides and salicylates (double base binders) copper chromite, LC-11 lead copper resorcilate chealate (double base binder), hi surface area iron complexes, melori blue, dichromate salts, barbiturate salts. Other additives that affected burning rate included AP, KNO, KNO_3, $KClO_4$, $KCAD_4$, EDDN, $NaNO_3$; these were used in large amounts (5-25%), sometimes also to raise I_{sp}, ρ_p, improve aluminum combustion or scavange HCl ($NaNO_3$ with AP).

6. Target specific impulses were apparently not met in any programs except

where high contents of AP, HMX, EDDN, double base binder, GAP binder, TMETN, BTTN or TEGDN were used (except in gas generator propellants where I_{sp} was not an issue). The problems were:

a) the low energy inherent with AN
b) low combustion efficiency with AN
c) low density of AN
d) loss of low cost, low hazard and low toxicity exhaust benefits when other ingredients were increased to improve I_{sp}.

In those applications where aluminum was used as a fuel ingredient, aluminum agglomeration, combustion and (in some applications) slagging were severe problems, and were important contributions to low I_{sp}.

7. Aluminum combustion is a problem because burning surface conditions are conducive to concentration and agglomeration of aluminum particles, but not usually conducive to ignition of aluminum. The seriousness of this problem is probably not well assessed because it depends on how the aluminum burns in the flow field inside the motor, and there isn't much data for full size motors. Results from laboratory combustors and small motor tests are terrible in most cases, but good aluminum combustion was indicated with some formulations. Some conditions conducive to good Al combustion were:

a) Anything that gave higher burning rate (high pressure, burning rate catalysts, energetic ingredients,
b) Ingredients that gave "hot spots" on the surface that apparently aided ignition of Al in the surface (high energy binder, AP, $NaNO_3$, $LiNO_3$, EDDN, $NaClO_4$, $KClO_4$),
c) Possibly effective were agents that break down the Al_2O_3 skin on the Al particles (SiO_2, FeF_3).

Contract Title: COMBUSTION STUDIES OF JET PROPELLANTS

COMBUSTION STUDIES OF EMULSION PROPELLANTS

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Monthly Progress Report No. 7
Aerojet Contract P. O. #410386
January 1988

I. Introduction

This is a brief summary of progress on Aerojet Contract P. O. #410386 for November 1987. Personnel involved are Dr. R. K. Sigman (Senior Research Engineer), Mr. Christos Markou (Graduate Research Assistant), and Prof. E. W. Price (Principal Investigator).

II. Objective

This research is for evaluation and improvement of the combustion characteristics of emulsion propellants presently under development by ASPC. The approach uses laboratory scale combustion tests such as the flushed window bomb to determine ignition and burning rate characteristics, nature of the burning surface, and combustion behavior of the aluminum ingredient. Actual test sequences will be determined partly by the program of formulation work at Aerojet and receipt of propellant samples. Concurrent efforts are being made to produce dry-pressed samples based on AN that can be used as an inexpensive medium for exploration and modification of aluminum combustion in AN systems.

III. Summary

Following the strategy outlined in Report #6, dry-pressed samples were prepared with 1% aluminum in order to establish methods for burning ammonium nitrate and polymeric fuels. The light aluminum loading will give an indication of aluminum behavior but will not be a significant heat sink which would retard combustion. Once methods for burning dry-pressed propellants with high AN loadings are established, methods of improving aluminum combustion will be explored. Using a fixed basic mixture of 1% aluminum and 9% carnauba wax binder,

the AN/AP ratio was varied until combustion was obtained at 20% AN, 70% AP (1000 psi). Burning was slow with a thick black melt layer and several very large glowing agglomerates were formed. Substitution of acrylonitrile-butadiene--styrene (ABS) for wax in this system gave a faster, smoother burning surface with a green flame.

Next a base mixture of 1% AL, 1% sodium barbiturate (an AN burning rate enhancer), and 9% wax was used with various AN/AP blends until a mixture of 40% AN, 49% AP was found to give an even burn with a black melt. The aluminum sintered, but did not ignite or agglomerate.

Powdered binders were compared using a base mixture of 50% AN, 39% AP, 1% Al and 1% sodium barbiturate and 3 powdered polymeric binders. Samples with carnauba wax would not burn in this formulation, ABS burned about 1/8 inch, and PEG (polyethylene glycol) burned about 1/2 inch before self extinguishing.

Finally, upon receipt of 200 grams of NiO phase-stabilized ammonium nitrate, a sample with 55% AN, 36% AP, 8% wax and 1% Al was pressed and burned at 1000 psi in nitrogen. Although there was a substantial amount of black melt, the burning was quite uniform with burning rate of 3.6 mm/sec (about half the rate of pure AP) and aluminum ignition was good.

In summary, tests with silane coated AN and AP demonstrated modest improvements in the useable AN/AP ratio with the addition of sodium barbiturate and substitution of polyethylene glycol. Aluminum ignition was not observed in any tests. Substantial improvement was observed by the use of NiO phase stabilized AN. Mixtures which would not burn with silane coated AN burned quite well with AN (Ni) and produced good aluminum ignition. NiO phase stabilized AN appears to offer great promise in improving the combustion of AN propellants. The full potential cannot be foreseen without further tests.

IV. Technical Discussions

A. Dry powdered mixtures of silane coated AN, AP, polymeric fuel and aluminum were pressed in a stainless steel die, cut into rectangular samples, and burned in a window bomb filled and flushed with nitrogen at 1000 psi. In all cases, 1% aluminum was used as an indicator of aluminum combustion with a minimal heat sink effect.

B. Mixes were prepared using 9% carnauba wax and AN/AP ratios of 60/30, 40/50, and 20/70. The first two mixtures did not burn, while the 20/70 mixture

burned very slowly with a thick black melt layer which flowed down the side of the sample. Several large agglomerates were formed, increased in size with time, but never left the burning surface. The bottom of the agglomerates emitted an orange glow. No ignition of the aluminum was observed. Substitution of ABS for the wax in the 20/70 mix improved combustion and produced a green flame. Smaller agglomerates were observed which glowed orange over their entire surface. Occasional ignition of single particles was observed.

C. Continuing with 1% aluminum and 9% carnauba wax, sodium barbiturate was substituted for 1% of the AP to give AN/AP ratios of 60/29, 20/49 and 20/69. The 60/29 mixture sample did not burn, but a large dark melt flowed down the side of the sample. The melt was created by the ignition paste and hot wire and flow was aided by heat from the Xenon lamp. The 40/49 mixture burned slowly with a thick black melt, but left a filigree of sintered aluminum. No ignition, agglomeration or self luminosity of the aluminum (no orange glow) was observed. The 20/69 mix burned slowly with a large black melt. The aluminum formed medium-sized self luminous agglomerates. Thus, the addition of 1% sodium barbiturate makes a modest improvement in the combustion of ammonium nitrate propellants.

D. Using a marginal base mixture of 50% AN, 39% AP, 1% sodium barbiturate and 1% aluminum; carnauba wax, ABS (acrylonitrile-butadiene-styrene) and PEG (polyethylene glycol) were used for the 9% powdered polymeric fuel. The sample with carnauba wax did not burn while the sample with ABS burned approximately 1/16 inch before extinguishing. This was not considered to be "unaided" combustion. The sample with PEG burned about one half inch and developed an irregular surface with an aluminum filigree and black melt before extinguishing. This is clearly a marginal case since burning proceeded until heat losses due to surface irregularities, melt, and aluminum filigrees caused extinguishment. Thus, of the three powdered polymeric fuels, ABS was previously shown to be better than carnauba wax, while present results indicate PEG to be better than ABS.

E. As a final test, NiO phase stabilized AN (supplied by ASPC) was mixed with AP in a 55/36 mix with 1% aluminum and 8% carnauba wax. Although the burning rate was about half that of pure AP, the sample burned to completion (this mixture with normal AN would not burn) and individual aluminum particles ignited. The NiO phase stabilized AN offers a significant improvement in performance over silane coated AN.

IV. Future Work:

Nickel oxide phase stabilized ammonium nitrate provides a significant improvement over pure AN or silane coated AN as a storage stabilizer and as a combustion aid. It is anticipated that combustion could be obtained for AN/AP mixture ratios of 65/16 using PEG. As higher AN loadings are reached it is anticipated that aluminum combustion efficiency would deteriorate. At this point, specially treated aluminum powder and other aluminum combustion aids could be tried.

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Aerojet Progress Report for August 1989

E. W. Price

Authorization to proceed on this project made it through the contracting and internal bottlenecks on August 1, 1989. Some time was devoted to planning and setting up for experiments, which will include:

1. Combustion photography and burning rate measurement.
2. Interrupted burning tests followed by microscopic study of quenched surfaces.
3. Quench-collection of condensed material leaving the burning surface.
4. CO_2 laser pyrolysis and interrupted pyrolysis at atmospheric pressure to obtain high rate pyrolysis under better viewing conditions than in high pressure burning.
5. DSC, DTA and hot stage microscope testing.

In order to get things moving quickly, Prof. Price visited Aerojet on August 18. The current status of the propellant development and testing at Aerojet was provided by Dr. Katzakian. A number of singular aspects of work with this class of propellants pertinent to GIT experiments have emerged as a result of discussion on that day.

1. The high oxidizer content of the propellant may require new methods of inhibiting surfaces of test samples. A change will also probably be necessary in the fluid used in the quench-collector experiment (the usual fluid, ethanol, may burn with the propellant gases).
2. Difficulty in ignition may lead to excessive pre-heating of small test samples, followed by rapid burn-up without reaching steady state.
3. Examination of quenched samples in the scanning electron microscope may not be feasible because of evaporation of ingredients in the evacuated test cell. If so, it may be possible to make and use replicas. Otherwise, optical microscope photography will be used.

4. Some speculation regarding slow reaction of aluminum in the propellant led to the proposition that this effect could be tested and possibly eliminated by use of "pre-oxidized" aluminum that has a more impervious oxide layer in the aluminum particles.

Small samples of propellant were brought back to GIT, and combustion photography tests have begun. At 1000 psi in a nitrogen-flushed bomb, uninhibited samples (nonaluminized) burned rapidly down the sides of the sample. Vacuum grease was tried as an inhibitor, and samples were observed to burn somewhat less rapidly, but still so rapidly as to make the rate suspect. The vacuum grease did not appear to burn. A wispy spire of residue was left after burn up of samples (either inhibited or uninhibited).

The tests yielded a few picture frames showing fairly clear detail of the burning surface. The surface seemed to be about 50% covered by orange spheres of about 500 μm diameter. This seems to be an important feature of the combustion, but information is limited and the possibility that the spheres are due to some other source (e.g., inhibitor) has not been eliminated. We have delayed use of more costly high framing rate photography until we obtain steady reproducible burning at rates somewhere near the rate observed at Aerojet.

Two samples of powdered aluminum were sent to Aerojet, one an untreated control sample and the other the same aluminum subjected to pre-oxidation treatment.

Contract Title: COMBUSTION STUDIES ON JET PROPELLANTS

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Monthly Progress Report for September 1989
Aerojet Contract P. O. #410386

Progress in this second month of the contract continued toward development of a high pressure residue collection bomb and photography of propellant samples with the goal of elimination of obstructions to observation of the burning surface.

The present particle collection bomb uses an "abandoned" pipe and plumbing from an acoustics experiment and is limited to 250 psi. Realizing that, with a few additional parts, the plumbing and several movie bomb components could be used as a high pressure residue collection bomb, design and manufacture of the additional parts has begun. This facility will be important in analyzing aluminum combustion efficiency at elevated pressures.

The majority of effort was expended in developing techniques for observing the combustion behavior of propellant samples supplied by Aerojet. The problems will be described below.

Three samples were brought from Aerojet by Prof. Price, consisting of: A) Control: 60% oxidizer, 20% binder, [sic]; B) 96A: 13% binder, 67% oxidizer, 20% aluminum, and C) 100C: 15% binder, 60% oxidizer, 20% aluminum, 5% ballistic additive. For combustion photography, samples are cut into a rectangular parallelepiped 3mm x 7mm x 12mm, epoxied on one 3mm x 7mm end to a post, mounted in a high pressure window bomb, and ignited on the other 3mm x 7mm end. A flushing flow of cool nitrogen normally inhibits side burning of fuel-rich ammonium perchlorate propellants, resulting in a planar burning surface. The experimental propellants from Aerojet exhibited side burning (possibly due to their oxidizer-rich nature), resulting in questionable burning rates and obscuration of the burning surface by smoke. Furthermore, a fine "ash" remained after combustion, which deflected smoke. Standard inhibitors were tried,

including vacuum grease, Devcon epoxy, Bob Smith Industries' polyamine epoxy, Epo-tek 305 epoxy and 3M amine epoxy. These inhibitors seemed to combine to retain the ash and to further obscure the surface. Additional samples were soaked in water for 1 sec, 10 sec, and 1 min. to dilute the sides. Possibly the best results were obtained by sandwiching a piece of propellant between two layers of thin glass. The ash was retained but an irregular plane of radiating aluminum was observed progressing through the sample. A burning rate of 5.5 in/sec was measured. This rate is very high compared to Aerojet results; the combustion photography was not clear enough to tell whether this was a true surface regression rate.

These samples were not properly stored in a desiccator and, following the inclement weather in the wake of hurricane Hugo, "sweated" a large amount of liquid. The liquid was found to have a pH of about 3. New samples will be stored in a desiccator and mounted in a dry box.

It should be noted that high speed photography of burning propellant surfaces has proved to be a valuable diagnostic tool for judging combustion efficiency and measuring burning rates, due to the ease of observation in the case of AP-based propellants. Difficulties encountered in observing the burning surface of these experimental propellants are diagnostic problems not necessarily reflecting propellant difficulties. Further attempts with inhibitors and flushing rates are expected to improve observation.

(.a13)

Contract Title: COMBUSTION STUDIES ON JET PROPELLANTS

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Monthly Progress Report for October 1989
Aerojet Contract P. O. #410386

Progress through 15 October has been concerned mostly with resolution of technical problems stemming from unique features of this propellant family. Results since 15 October suggest that most previously reported results were doubtful, due to technical problems. Specifically, the propellant samples burn very rapidly down the sides of uninhibited samples (even in a counter flow of nitrogen), and effective inhibiting of the side surfaces of samples has been difficult. In addition, the samples appear to burn with a red-brown smoke that often obscures combustion features normally visible in combustion photography.

A new supply of propellant samples was received early in the month, and Aerojet also supplied an epoxy inhibitor that seems to work fairly well. Combustion video photography has been run on all the samples in the latest shipment, at pressures from 500 to 1500 psi.

Burning rates were determined, and generally confirm rate data supplied by Aerojet (some deviant results appear to be due to unwanted effects of the inhibitor coating on the samples). Resolution of features of the combustion zone in video photography are not particularly good because of smoke obscuration and limited resolution (time and spatial). However, the samples burn down smoothly. The aluminum is generally visible as a bright diffuse luminosity. While the exploratory nature of tests to date precludes quantitative statements, the brightness of aluminum combustion does to seem to be critically dependent on pressure or sample composition. The size of luminous objects indicates that aluminum is agglomerating. This results (with other propellants) from concentration of aluminum particles on the surface to form sintered aggregates that ignite and agglomerate wherever the temperature is high enough to break down the protective oxide on the aluminum. In the present case, at least some of the

agglomerates are igniting close to the surface (judging from luminosity), however some aggregates are found in the bottom of the window bomb after tests, indicating that aggregates are leaving the burning surface unignited (failure to burn thereafter may be due to proximity to the cool nitrogen flushing flow, but the amount of aggregates is much larger than with other propellants tested).

Preliminary quench tests have been made (i.e., sample quench by abrupt depressurization). The two samples to date show evidence of a liquid or frozen surface film (optical microscopy). After optical examination, the samples are gold-coated by vacuum sputter coating, and are examined at higher magnification in a scanning electron microscope. The liquid layer appears to have evaporated in the vacuum of the sputter coater, leaving a rather nondescript (but irregular) surface with areas of high concentration of aluminum particles. Sintered aggregates are not evident, and may have been stripped from the surface during quenching.

A series of tests is now planned using higher speed microcinephotography. These tests are appreciably more costly, and have been delayed until now by difficulty of assuring predictable sample burning. These new pictures should reveal much more detail regarding the metal combustion and its dependence on pressure and propellant formulation.

Contract Title: COMBUSTION STUDIES ON JET PROPELLANTS

E. W. Price and R. K. Sigman
Georgia Institute of Technology
Atlanta, GA 30332

Monthly Progress Report for November 1989
Aerojet Contract P. O. #410386

Efforts during this fourth month of the contract were directed toward refinement of our video recording techniques and initial high speed motion picture photography of Aerojet propellants. Several burning Aerojet propellants also were extinguished by rapid depressurization of the high pressure bomb and the "burning surfaces" were examined in the scanning electron microscope.

Although high speed cinematography is still the best available method for observing the burning surface of propellants, increasing delays in processing and costs forced us to turn to video photography with a high speed shutter. Our video camera records at 30 frames per second, but employs a built-in 1/1000th of a second shutter (1/5000 sec for the Hycam movie camera) to reduce smearing and increase definition during an exposure. The resolution is also less for the video system, but the advantages of low cost and immediate results give an advantage to the video system for burning rate measurements and other exploratory tests where high resolution and definition are not important.

Our experience with the video system with unaluminized and low aluminum loadings has been favorable. The high aluminum loading of Aerojet propellants makes video photography difficult. If the exposure is set for the solid propellant, the burning aluminum in the gas phase saturates the video tube. If the exposure is reduced to unsaturate the aluminum cloud, the unburned solid propellant is not visible. We have arrived at a compromise which permits measurement of the burning rate but does not allow observation of the burning surface. Although the video results are not as unambiguous as results for unaluminized samples, we are in a better position to verify burning rates if required.

High speed cinematography was used on six samples of Aerojet propellant

using the Locam (500 frames/second) and Hycam (2000 frames/second) cameras. Unfortunately, our convenient local film processors have ceased operation and we have resorted to mail order processing. We are currently seeking alternatives in film or processing to eliminate this bottleneck.

"Quenched samples" are obtained by mounting a sample in a special high pressure bomb, pressurizing the bomb and igniting the sample. After a suitable delay (adjustable) to permit steady state burning to be achieved, a Mylar diaphragm is ruptured by a heated wire, the bomb is rapidly depressurized, and the propellant is quenched. Samples can then be examined in the scanning electron microscope to obtain details of the surface microstructure.

Several Aerojet propellants were quenched by rapid depressurization from 500 psi and examined in the optical and scanning electron microscopes. The slides will be shown at the December 12 meeting. Due to the homogeneous nature of the oxidizer and binder, the surface does not display the normal heterogeneous microstructure of an AP-binder propellant. No aluminum is seen on the surface (possibly removed by the rapid depressurization). Some clusters of aluminum are visible just below the surface.

We are currently constructing a copper vise for the pressure bomb which quenches burning propellants by acting as a large heat sink. This procedure is not as abrupt as rapid depressurization, but is less traumatic and might retain any surface agglomerates or filigrees.

The test work to date has provided a lot of experimental difficulties unique to this propellant class, and not a great deal in the way of understanding of combustion. Some of the observations that are probably significant regarding combustion behavior are the following:

1. Nitrogen flushing was not initially enough to prevent end-burning samples from rapid burning down the sides. Various inhibitors were tried; those that worked obstructed the view of the burning surface. We have observed that fresh cut sample surfaces become moist, an effect that seems to aid side burning and impair inhibitor adhesion. We find that the nitrogen flush alone usually does prevent side burning of the

sample if we limit exposure to humidity and/or blot the surface dry before the test.

2. The propellant burns with a dense cloud of brown "smoke" that obscures viewing of the burning surface. Some of the smokiness may have been associated with inhibitors, but the smoke is present with uninhibited samples as well. It seems likely that this smoke is a cloud of undecomposed oxidizer aerosol.

3. Inhibitor and smoke problems have impeded observation of details of aluminum combustion, which is usually manifested as a diffuse, intense glare in the vicinity of the burning surface. Details above the surface are obscured by smoke. Due to the small sample size and cold nitrogen flow, observations more than a few millimeters above the sample surface are interpreted with reservations because of flushing flow effects (which presumably block complete combustion of the smoke).

4. Efforts to confirm burning rates provided by Aerojet have been indecisive because of erratic side burning and inhibitor and smoke obscuration of surface location.

During the month of December, we hope to improve resolution of the behavior of aluminum of the burning surface through cinephotography and quench testing.

Contract Title: COMBUSTION STUDIES ON JET PROPELLANTS

E. W. Price
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Monthy Progress Report for June 1990
Aerojet Contract P. O. #410386

Authorization to resume work on this project reached the "working level" at GIT on about 20 June; hence progress is somewhat limited.

The first laser pyrolysis test was run successfully (1 atm nitrogen flushed test chamber). Estimated flux was 500 watts on a 7 mm diameter sample surface. The sample (nonaluminized) surface receded smoothly at about 0.5 mm/sec. The camera angle was edge on, so that only limited information on the surface behavior was revealed. The cloud of aerosol that obscures burning at 1000 psi was not evident in this test, which has a nitrogen cross flush over the surface. A periodic buildup and detachment of surface residue was evident. We suspect this may have been present only on the far side of the end surface (far from the camera), and caused by low laser flux on that part of the surface. In future tests the sample will be cut to provide a slight tilt of the surface towards the camera. Tests will be run on all the samples currently available until new samples are received.

Shop work has continued on a particle collection test device, which will enable us to quench burning aluminum at different distances from the burning surface. Quenched particles will be analyzed vs quench distance, propellant formulation, etc. Such information will help to predict and control combustion efficiency and 2-phase flow effects.

A video tape was sent to Dr. Katzakian showing combustion photography of samples burning at 1000 psi. Included were similar sequences for propellants with AP oxidizer for comparison. The tape will illustrate the aerosol obscuration that has impeded observation of combustion of Al3 propellants. This aerosol is more than an experimental inconvenience. It is a basic and novel attribute of the combustion of these propellants, still to be elucidated.

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Contract Title: COMBUSTION STUDIES ON JET PROPELLANTS

E. W. Price
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Atlanta, GA 30332

Monthly Progress Report for July 1990
Aerojet Contract P. O. #410386

During this month of the contract, construction of the new particle collection bomb neared completion and laser heating of samples continued. Laser heating of aluminized samples of different composition appeared to show differences in surface behavior and aluminum ignition/detachment.

Construction of the major components of the high pressure particle collection bomb was delayed several weeks due to a loss of materials in transit. Although construction of the components is nearing completion and we hope to have preliminary results available before the end of the program, we have diverted the majority of our efforts toward laser heating tests in order to generate a more complete array of results.

Laser tests have been proceeding with reasonable success. It should be noted that in addition to our particular problems (marginal cooling system, secondary pump overheating, etc.) the proper delivery and conditioning of a high power CO_2 laser beam is a major difficulty for all systems. We have attempted to use a neon laser to bore sight the system, but insertion of the laser disturbs the alignment. Since the alignment varies constantly with building movement (thermal expansion, building vibration) some tests are regarded as suspect due to unusual behavior and subsequent alignment checks.

A single laser heating test of an un-aluminized sample was reported in last month's progress report. In this test, the sample was a rectangular parallelipiped with the camera mounted perpendicular to one surface and perpendicular to the laser flux. On the side of the sample, a thin (approximately 0.2 mm thick) black, carbonaceous layer forms on the "burning" surface. The interface between the unmelted propellant and the carbonaceous residue appears to be a boiling liquid layer while the upper portion appears solid although subsequent inspection reveals a semi-rigid ash. Toward the center of the sample, the carbonaceous material thickens (to about 2.0 mm), is swept

away by a nitrogen cross flow and re-accumulates. It should be noted that while it is often easier to observe detailed behavior on the side of the sample, the side is usually cooler than the center and may give misleading results.

Thus, in the present series of tests, the heated surface of the sample is cut at an oblique angle so that the actual irradiated surface can be seen during the test. All samples were heated at about 700 Watts and a nitrogen flushing flow was used. Since radiation from reacting aluminum saturates the video camera, all tests were interrupted, allowed to cool slightly and then reheated. In several cases, neutral density filters were fitted to the video camera before beginning the reheating process.

1) AL3: When the surface is exposed to the laser radiation, the surface appears to boil. Bright spots (aluminum agglomerates held by the residue) form and radiation from their reaction quickly saturates the picture. Some single ignited aluminum particles are seen to leave the surface but most are trapped in the residue and remain on or near the surface.

2) C478-89: Due to misalignment of the beam, only the rear half of this sample was heated and the surface was not visible. However, the picture was not saturated and numerous single ignited particles were seen leaving the surface.

3) C478-74-1: In this test, the beam was centered on the sample, but the unusual results prompted a realignment of the upper delivery system, where it was found that the beam was being clipped by a section of the lens holder. Thus the intensity was probably somewhat less than 700 Watts. In this case, the surface boiled, a solid residue forms and floats on the boiling liquid but the aluminum does not ignite.

4) C478-74-2: This sample appeared to have more single ignited aluminum particles leaving the surface. With filters installed there appeared to be several smaller, bright residual lumps, rather than a single large residue.

5) C478-74-6: This sample saturated the camera and left a single large residue.

We have noticed that scraps removed during sample preparation and left on a sheet of paper leave a pink residue on the paper. Comparison of the burning characteristics of stained and as received paper revealed no difference. The stain was found to vaporize in advance of the burning front.

During August, the remainder of the available samples will be pyrolized by laser heating and any new samples will also be tested.

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Monthly Progress Report for August 1990

GEORGIA INSTITUTE OF TECHNOLOGY
ATLANTA, GEORGIA 30332

SCHOOL OF
AEROSPACE ENGINEERING

404-894-3000

DANIEL GUGGENHEIM SCHOOL
OF AERONAUTICS

2 March 1988

Dr. R. L. Lou
Building 05025, Dept. 2131
Aerojet Solid Propulsion Co.
P. O. Box 15699C
Sacramento, CA 95813

Dear Dick:

I am writing to you to give a brief summary of our work on combustion related to the emulsion propellant project. My intent is to help you decide whether this line of investigation has relevance to the "companion" Aerojet program, to which the emulsion propellant project funds are reportedly being redirected. While the information available to me regarding that companion program is minimal, I have the impression that the Georgia Tech goal of assisting in achieving high combustion efficiency is equally critical and important in both programs.

The main objective of the Georgia Tech work was to achieve good aluminum combustion in propellants with nitrate oxidizers, something that has not been achieved in any previous work (except with energetic binders). Georgia Tech has conducted studies of aluminum combustion in other propellants for many years; we anticipated serious troubles with nitrate propellants, and felt we were perhaps better qualified than anyone else to evaluate and apply past strategies and develop new ones for improved aluminum combustion. We anticipated difficulties in obtaining propellant samples in the present program and in getting modifications prepared (usual practical problems), and proposed that we would supplement propellant testing by studies of combustion of model propellant samples prepared by dry-pressing mixtures of powdered ingredients. Progress in our work was not spectacular up to the stop work order, partly because our contract arrangements were not completed until around 1 July 1987, and partly because of usual start-up problems and the expected difficulties in obtaining respectable burning of samples. However, the outlook was very optimistic by the time work was suspended (we are still running a few tests on our own).

A limited supply of Aerojet samples were received and tested in a window bomb at 1000 psi (two formulations). All samples burned, but with very limited aluminum combustion. The aluminum concentrated on the burning surface, often leaving sintered accumulation containing most of the aluminum in an unreacted state.

Tests on dry-pressed samples started with an oxidizer blend of AP and AN, with a powdered wax binder and 15% aluminum. We could not get self-sustained

burning in early tests unless the AP/AN weight ratio was greater than 60/25, and aluminum accumulated on the burning surface and showed poor escape from the surface and little sign of combustion.

We explored means of assuring low water content in the AN (including vacuum drying at 50° C, and silane coating). Resulting improvement in combustion was marginal. We tried three different fuel powders (carnauba wax, acrylonitrile-butadiene-styrene and polyethylene glycol). The PEG fuel seemed to give the best combustion, but improvement was only moderate (in all cases the early goal was to obtain uniform burning with the lowest ratios of AP/AN). We also tested sodium barbiturate as an aid to burning (suggested by Russ Reed) and judged that some improvement in uniformity of burning resulted.

Because the presence of concentrated non-burning aluminum on the surface was deemed to hinder burning of the samples, it was decided to strive first for good burning with the lowest AP content possible without aluminum. The idea was to get a nonaluminized formulation with respectable burning, and then work for aluminum combustion from that starting point. We continued to use 1% aluminum because we felt it would not impede burning, and the combustion photography would show whether the aluminum was igniting and burning or not. The low-aluminum testing actually started back in the test series to compare fuel powders (binder substitute) noted earlier. Reproducible burning at about .435 mm/sec was achieved with AP/AN ratios down to 43/40, but aluminum was observed to accumulate in a surface melt with minimal ignition. A baseline formulation for studies of methods to improve aluminum combustion was chosen as

AP/AN/PEG/sodium barbiturate/Al in
mass fractions 49/40/9/1/1

This was not a very encouraging baseline situation, but we had a lot of ideas on how to improve aluminum participation in combustion.

The results reported at the recent JANNAF Workshop on combustion of AN propellants indicated that in almost all cases of "good" aluminum combustion, an energetic binder was used and phase stabilized AN was used. Aerojet supplied us with some NiO stabilized AN shortly before suspension of work, and we ran tests with a formulation similar to the baseline except for reduction of the AP content (in favor of PSAN)

AP/PSAN/Wax/Al
36/55/8/1

This sample exhibited smooth burning and effective ignition and burning of the 1% aluminum particles, a very encouraging result. Following the order to stop work, the aluminum loading in the baseline formulation was increased to 15 percent (giving AP/PSAN/Wax/Al ratios of 32/48/7/13). Once again the sample exhibited smooth burning and effective ignition and combustion of the aluminum. Finally, some BAMO-THF with 20% catocene prepared for an ONR project was mixed with NiOPSAN and formed into a rectangular sample. An attempt was made to obtain

NiOPSAN/BAMO-CATOCENE/Al formulation of 80/5/15, but difficulties with mixing the very viscous and rapidly curing BAMO left a mix of uncertain mass fraction. In any event, this formulation burned evenly with good ignition of the aluminum. Although BAMO is not a realistic binder candidate for the class of propellants under consideration, it is clear that satisfactory aluminum combustion can be achieved in propellants without ammonium perchlorate.

As I said at the outset, I do not have full information on the "companion" program, but from the little I know, aluminum combustion will continue to be marginal. We would like to work with you on that problem.

Sincerely,

Edward W. Price
Regents' Professor
School of Aerospace Engineering

EWP/ed
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cc: Dr. Campbell

COMBUSTION STUDIES OF JET PROPELLANTS

E. W. Price
Georgia Institute of Technology
Atlanta, GA 30332

Preliminary Draft of Final Report
Aerojet Contract P. O. #410386

Objectives:

The general goal of this research was to achieve high combustion efficiency and improved burning rate characteristics of aluminized propellants with nitrate oxidizers, particularly of solution propellants under development by Gen Corp Aerojet.

Approach:

The approach was to study combustion of small samples in laboratory scale burners, including combustion window, quench, and particle collection "bombs" and CO_2 laser pyrolysis, along with optical and SE microscope studies of quenched samples. Such studies, in combination with systematic modifications of propellant formulation, provide a quick route to qualitative understanding of combustion and propellant optimization.

Summary of Research:

The research is described in the body of the report in three phases, which emerged as a result of two extended interruptions in the study due to discontinuities and reorientations of the parent Gen Corp Aerojet project.

In the first phase (July 1987 to February 1988), studies were made of model propellants prepared by dry-pressing mixtures of AN, AP, polymer and aluminum powders (no Aerojet propellants were available yet). It was found that AN:AP ratios higher than 25:60 gave poor combustion behavior (1000 psi), with irregular burning and minimal aluminum ignition. Use of combustion aids and optimum binder resulted in AN/AP ratios greater than 25:60 with regular burning and fair aluminum ignition. Work was suspended before optimization of combined aids was attempted.

In the second phase (August 1989 to December 1989), studies were made of combustion of propellant samples supplied by Gen Corp Aerojet (solution propellants). This phase of the studies was relatively nonproductive because of several features of combustion that impeded photographic observation (samples that would not burn, burning down the sides of end-burning samples, accumulation of residues (char) on the surface, and clouds of aerosol from the surface). These are all significant findings regarding combustion of this novel propellant, but singular, unexpected, and not particularly helpful in getting to the target of controlling burning rate and aluminum combustion. In particular, observation of the aluminum combustion was obstructed by residue and smoke obscuration. The propellant apparently burns with substantial release of undecomposed oxidizer from the surface, formation of char on the surface, and intense combustion of accumulated aluminum in the char layer. In most samples, a column of residue was left after the sample was consumed, with some unburned aluminum remaining. The practical significance of these novel features of the combustion, or their relevance to combustion in a motor environment remain uncertain.

In the third phase (June 1990 to September 1990), recourse was taken to atmospheric pressure laser pyrolysis tests in order to simulate combustion under conditions where less obscuration would be present. Samples were observed by video photography in a nitrogen flushed test chamber with incident infrared surface heating at about 320 watts/cm^2 by a CO_2 gas laser. Tests were run on all the propellant in the first shipment from Aerojet (Table 1) and all samples of a second shipment (Table 2). Full information on formulation was not supplied; however, each set of samples consisted of an unaluminized sample and a range of aluminum concentrations. In the laser pyrolysis tests, the details of surface behavior and aluminum combustion were relatively observable.

Experimental Methods

Evaluation of the performance of nitrate based propellants was conducted using facilities in the combustion and microscopy laboratories in the School of Aerospace Engineering. The combustion lab contains: (1) a high pressure window bomb with lighting, video, and motion picture cameras; (2) a high pressure quench bomb; (3) decomposition equipment (DTA, DSC, TGA); (4) a 1000 watt CO_2 laser with beam optics and optical table; and (5) sample preparation, handling, and storage

facilities. The adjacent microscopy laboratory consists of scanning electron and optical microscopes with associated heating stages.

These laboratories have been used extensively for studying propellant (primarily ammonium perchlorate based) combustion as well as the high temperature decomposition of individual ingredients. These studies of nitrate based propellants concentrated on the well established tools of propellant research: combustion photography and microscopic examination of quenched surfaces, as well as a relatively new procedure: laser assisted heating.

I. Video and Cine Photography

The most powerful and convincing available method of evaluating the combustion of solid propellants is cinephotography. All of the old saws: "seeing is believing" and "a picture is worth a thousand words" apply to this method. Burning rates as well as the uniformity of burning rate can be measured and qualitative features such as the combustion behavior of aluminum (accumulation, agglomeration) can be determined. The ability to witness the event and observe unanticipated processes is a valuable research tool.

The normal combustion facility photographs a small parallelepiped of propellant ignited on one end. In an actual rocket motor, the burning surface will experience a cross flow of propellant gases ranging from low in the head end to high at the aft end. This cross flow leads to higher burning rates (erosive burning) and is dependent on the actual location within the motor. Thus, it is important to note that standard combustion photography provides "non-erosive" or end burning. Thus, in an actual motor the local burning rate and aluminum behavior will depend on the character of the cross flow and thus will vary from point to point within the motor. The end burning results provide only a baseline.

Photography of propellants has traditionally been done by high speed (1000 to 10,000 frames/sec) 16 mm motion picture (cine-) photography. Unfortunately, the convenience of video has all but eliminated the amateur motion picture industry. Film processors are limited to a handful of national services with attendant shipping delays. High speed and high resolution have given way to

instant replay and low cost. Useful videos are limited to exposure times of 1/1000 sec but are adequate for measuring burning rate and observation of gross behavior. High magnification is still accomplished by lens extension tubes and close-up (macro-) lenses. Resolution is limited with the current generation of television monitors.

Propellant samples are mounted in a stainless steel chamber with quartz windows for external illumination and photography. Illumination is provided by a 2500 watt Xenon lamp. Nitrogen is used to pressurize the chamber and to provide a flushing flow parallel to the sides of the sample for removal of the smoke. As previously noted, this flushing flow is not parallel to the burning surface and does not provide a cross flow to affect the burning surface.

II. Surfaces of Quenched Samples

Another useful tool for investigating combustion behavior is optical and electron microscopy of quenched samples. A quench bomb is similar to a window bomb except that the top is sealed with a stack of thin mylar disks trapping a nichrome wire. The chamber is pressurized, the sample is ignited, and after a brief delay the nichrome wire is heated by electrical resistance. The mylar diaphragm bursts, the chamber is depressurized in milliseconds, and the burning sample is quenched. The sample is then examined in a scanning electron microscope to obtain details on the microstructure of the burning surface.

For a heterogeneous propellant, the surface consists of partially burned ammonium perchlorate regions, divided by mounds of binder coated aluminum. Many details can be inferred by the surface structure of the AP, the size of the aluminum accumulates, and other microscopic features.

III. Laser Pyrolysis

An alternative to the high pressure window bomb is pyrolysis with a CO_2 laser. Radiation from these lasers is in the infrared (10.6 micron) region and is thus an effective heating source. High power CO_2 lasers (about 1000 watts) can produce heat fluxes on the same order as a burning propellant. The lower density at atmospheric pressure reduces the smoke density and thus the

obscuration. Since the laser flux must enter the atmospheric chamber perpendicular to the burning surface, the flushing flow is removed from the side producing a cross flow parallel to the regressing surface. Previous tests heating a solid polymer revealed a strong shearing force on the pyrolyzing surface. Thus, the laser heating tests will permit clearer observation of the burning surface and thus the behavior of aluminum on the burning surface.

The purpose of the laser heating tests is to investigate the combustion behavior of the aluminum in these propellants. This is important because unlike the oxidizer and fuel which react at the burning surface (within several hundred microns), the aluminum is ignited at the surface but normally continues burning in the gas flow through the rocket motor. The transition from a single aluminum particle in the propellant to an ignited mass in the gas flow is important in determining combustion efficiency, slag formation, acoustic damping, and other parameters. Before describing the behavior of the aluminum in each of the samples, it is worthwhile reviewing the possible scenarios for aluminum combustion in solid propellants and their efficiencies. From an analytical viewpoint, the simplest path would be for an isolated aluminum particle (in a lightly loaded composition) to arrive at the burning surface, detach from the surface, ignite and burn in the gas flow. This is a simple and efficient path, but it is not common. In practice, when an aluminum particle arrives at the burning surface it is in contact with other aluminum particles (more so as the aluminum content is increased) and the relatively high temperatures cause the particles to sinter together to form an accumulate.

The accumulate is basically an irregular chain of sintered particles which eventually begins a slow, exothermic oxidation and thus increases in brightness. The accumulate may linger on the surface feeding heat back to the surface and/or may detach to burn in the gas flow. If the surface (or gas) temperature is high enough, the oxide shells defining the original particles will collapse (possibly melt) and all of the aluminum will coalesce into a large sphere called an agglomerate. The agglomerate will usually detach at some point and burn in the gas flow. The ignited agglomerates are extremely bright and comet-shaped with a convective trail.

In summary, in video coverage of the laser sustained combustion, the

simplest (and possibly ideal) case of single particle detachment would appear as numerous small, very bright streaks above the surface. In the more realistic case of ignited agglomerates, bright spheres would move across the burning surface (as the sintered accumulate draws up into an agglomerate), detach and be convected slowly away from the propellant surface. If the aluminum does not agglomerate it will appear as less bright irregular shapes on the surface and possibly in the gas flow. Aluminum combustion by this path is less efficient since combustion may not be complete by the time the accumulate leaves the rocket motor. Finally, the aluminum may not detach at all but rather form a sintered glowing bed on the surface. This has the worst efficiency and the self heating may cause the propellant to continue "burning" even after the laser heating is removed.

Results

I. Phase I: Investigation of Dry Pressed Nitrate Propellants

The first phase of this investigation involved attempts to produce in-house propellants using ammonium nitrate, powdered binders, and aluminum. Due to the poor performance of ammonium nitrate, ammonium perchlorate was added to produce a mixture of AP and AN capable of sustaining combustion. Established methods of coating AN to reduce the hydroscopicity as well as burning rate stabilizers were also investigated.

A. Oxidizer Self Deflagration

Initial experiments attempted to establish a baseline for the combustion of ammonium nitrate. Ammonium perchlorate will burn as a monopropellant in nitrogen at pressures above 300 psi. Ammonium nitrate (dry pressed into a parallelepiped) would not burn at 1000 psi in nitrogen or methane (a fuel). A dry pressed mixture of 90% AP and 10% AN burned irregularly at 1000 psi in nitrogen while an 85/15 mixture did not burn.

B. Combustion of Dry-Pressed Oxidizer-Fuel Combinations

In order to avoid problems associated with small scale propellant mixing

(inaccurate mixtures, trapped air bubbles, long curing times), pressed mixtures of dry powdered ingredients were used. While the oxidizers and aluminum are dry powders, a solid powdered hydrocarbon is substituted for the normally liquid binder to produce a dry mixture. The dry powders are measured, mixed and poured into a stainless steel die. The die is hydrostatically pressed at 19,700 psi and held for several hours to produce a compact sample. The sample is mounted in the window bomb, pressurized (with nitrogen to 1000 psi except as noted) ignited and recorded on video tape.

Variation of Oxidizers

The initial mixture consisted of 85% oxidizer, 10% aluminum and 5% carnauba wax (a dry powder binder substitute). Tests at 500 psi and 1000 psi indicated that a 60% AP and 25% AN mixture burned unevenly while a 50/35 mixture would not. Results of this series of tests are shown in Table 3. Switching to a mixture of 90% oxidizer, 9% binder (with 1% aluminum just to indicate ignition performance), gave a formulation closer to stoichiometric without the heat sink associated with heavy aluminum loadings. This mixture required a 70% AP, 20% AN ratio to sustain combustion.

Variation of Fuels

Powdered carnauba wax has been used extensively as a dry binder and produces good ignition of aluminum. Substitutes including ABS (Acrylonitrile/-Butadiene/Styrene) resin, Acrylonitrile/Butadiene Copolymer and Polyacrylonitrile were also used as dry binders. ABS was available in a more desirable particle size range and the 50% AP/25% AN/10% ABS/5% Al sample burned more evenly (but with greater aluminum agglomeration) than did the equivalent sample with wax.

Variation of Ammonium Nitrate

Due to the hydroscopic nature of ammonium nitrate, it must be kept desiccated. Sample preparation is delayed by inclement weather and quenched samples often deteriorate before examination in the scanning electron microscope. In an attempt to create a moisture barrier, Silane, a standard protective coating for AN, was used to coat a batch of AN. The coated AN was marginally better at

resisting clumping and dissolving in high humidity and produced slightly more even combustion. The most notable improvement came with the use of NiO phase stabilized AN. Although only preliminary work was carried out using NIOPSAN (because it was received after the stop work notice, it showed substantial improvement with regard to AN/AP ratio for good combustion and aluminum ignition.

Burning Rate Stabilizers

Sodium barbiturate is reputed to be a necessary additive in some applications for obtaining consistent, uniform burning of AN. The addition of 1% sodium barbiturate to the formulations with marginal burning characteristics gave improved results, but this line of investigation was also terminated by the stop work order.

Results from video recording of emulsion propellants supplied by Aerojet were of acceptable quality, but revealed poor performance. Early samples did not burn (at 1000 psi) while later samples burned with a measurable burn rate but without aluminum ignition. The combustible sample (C728-46C) retained aluminum filigrees which were coated with a black carbonaceous residue. Substituting air for nitrogen as a pressurizing and flushing gas removed the carbonaceous residue and aluminum detached but did not ignite.

Phase II.

The primary objective of Phase II was burning rate measurement and evaluation of aluminum ignition behavior of the AL3 series of propellants from Aerojet General Corp. using combustion photography. The composition of these samples is listed in Table I. Unfortunately, observation of all samples photographed in the window bomb was limited due to "smoke" produced by these propellants. Combustion was superior to the original emulsion propellants, but the AL3 leaves a fine ash residue which accumulates on the burning surface, deflecting the products of combustion and filling the volume between the window and the sample with a dense smoke. Sides of the sample were coated with various inhibitors in an effort to eliminate side burning, but the surface was usually concealed by the inhibitor. The aluminum did not ignite and leave the surface

but remained trapped in the residue, glowing due to a modest amount of exothermic oxidation.

While viewing the details of combustion of AL3 propellants was impossible due to smoke and residue, it was often possible to measure the rate of advance of aluminum self luminosity through the sample. These rates were close to the measured burning rates supplied by ASPC. Unfortunately, the residue did not permit the identification of a clearly definable surface (such as the gas-solid interface normally seen in AP-based solid propellants) and subsequent measurement of the regression rate of this surface. Thus, while measurement from most cinephotography is unambiguous, the actual surface is subject to speculation in the AL3 combustion photograph and thus its regression rate is uncertain. High speed motion pictures of the combustion in place of video photography increased both time and spatial resolution of clear areas. The actual burning surface was still obscured, but the dense smoke was observed to be emitted in small unsteady jets. It is probable that this residue would be swept off by the cross flow in a real motor and would not be a significant factor in propellant combustion. It is simply an impediment to observation. The effect of the residue on aluminum behavior could not be evaluated in these tests.

In summary, the visual recording processes, both cinematography and video, which were effective in evaluating the combustion of dry pressed propellants with AN and AP propellants were not capable of producing unambiguous burning rates or evaluation of the accumulation and agglomeration characteristics of aluminum. They did reveal a tendency for the AL3 propellants to form a surface residue in an end burning or cigarette configuration and to emit dense "smoke" from the surface, possibly oxidizer aerosol.

Quench testing of the AL3 samples was not hindered by the smoke or residue (which was stripped by the rapid depressurization) but was limited by the near homogeniety of the samples. The Aerojet propellants are only heterogeneous because of the aluminum. During the rapid depressurization, the surface residue and accumulated aluminum was stripped off. Optical examination of the burning surface revealed a liquid or glazed surface with patches of aluminum that appeared to be below the glazed surface. After coating with gold and examining in a scanning electron microscope, the liquid layer seems to have evaporated

leaving a fairly nondescript (but irregular) surface with areas of high concentration of aluminum. Whereas accumulates of aluminum are normally seen on the surface of a heterogeneous AP propellant, the aluminum is buried in the Aerojet propellants with only slight bumps to indicate their presence. Although a comprehensive SEM study of quenched nitrate propellants might reveal subtle but important characteristics, the lack of any obvious distinct features at this point forced our efforts toward methods with a higher guarantee of success.

Phase III.

The objective of the final phase remained the evaluation of the combustion efficiency of the aluminum in the AL3 series. Due to the lack of success with video and cine photography in the high pressure window bomb, video recording of laser assisted heating in an atmospheric chamber was pursued. Measurement of the regression rate of samples irradiated with 550 watts of power (700 watts at the laser) produced a regression rate value about 40% of the burning rate (as supplied by Aerojet) at 500 psi. Smoke and residue ash were often found in the exhaust, but did not obscure the observation of the pyrolyzing surface.

Laser heating tests were carried out in a thin walled test chamber with a nitrogen atmosphere and purge. The chamber is scavenged through a side port which creates a cross flow of nitrogen across the pyrolyzing surface of the sample. The laser flux enters through a Zinc Selenide window on the top while quartz side windows permit illumination and video. Laser power was set at 700 watts at the shutter for all tests, resulting in 550 watts over a 1.5 cm circle at the sample.

Multiple runs on each of the eight propellants in the C478-74-X series and the two propellants in the 652-8X series have been analyzed and the results are listed below:

C478-74-1: Some small, fast burning particles (single particles or small agglomerates) convect with the gas flow. Some large agglomerates form and detach. Some glowing spherical agglomerates remain on the surface near the edges.

C478-74-2: A few small, ignited particles are seen in the gas flow but most aluminum is retained on the surface forming a large glowing bed of accumulated aluminum. This sample had the poorest efficiency.

C478-74-3: Small surface accumulates form and detach to burn as agglomerates.

C478-74-4: More surface accumulation. Detached aluminum does not appear to spheroidize, implying only limited aluminum reaction. Burning accumulates/agglomerates are larger than -3.

C478-74-5: Substantial surface accumulation. Accumulates ignite to form large agglomerates. Burning agglomerates leave the surface.

C478-74-6: Bright surface ignition, a few single particles or small accumulates are seen in the gas flow. Numerous large agglomerates ignite and detach.

C478-74-7: Large surface accumulates are formed. Accumulates change to large agglomerates on the surface and detach reluctantly. After the laser was switched off, a large surface accumulate continued glowing, feeding heat back to the surface and continuing to pyrolize the solid. The sample continued burning several seconds before finally extinguishing.

C478-74-8: Basically good ignition with large agglomerates which detach and are convected away.

An initial series of tests on two new propellants, 652-81 and 652-82, were also carried out.

652-81: Large surface accumulates are formed. Some form large agglomerates.

652-82: More surface accumulates are formed. A large amount of ash is formed with this sample. All samples burned to completion after the laser was turned off. It would appear that this formulation will burn at atmospheric pressure after a thermal wave and a glowing aluminum bed are established.

Table 1

Composition of C478-74-X Series of Propellants

Component	1	2	3	4	5	6	7	8
AL MDX-65	15.0	7.5	18.0	9.0	22.0	11.0	26.0	13.0
H-60	--	7.5	---	9.0	---	11.0	---	13.0
PVA	13.0	16.0	13.0	16.0	16.0	13.0	16.0	13.0
Burning Rate Add.	2.0	3.5	3.5	2.0	2.0	3.5	3.5	2.0
Oxidizer	70.0	65.6	65.5	64.0	60.0	61.5	54.5	54.0

Table 2

Composition of C494-29 and 652-81 Series of Propellants

	C494-29A	29B	29C	29D	29E	29F	29G	652-81	652-82
S-HAN-5	65.3	62.0	65.0	65.0	81.0	70.0	75.0	62.0	65.3
GNX	3.7	4.0	--	--	--	--	--	4.0	3.7
Al MDX-65	18.0	15.0	15.0	10.0	--	5.0	2.5	15.0	18.0
Al H-60	--	5.0	5.0	10.0	--	5.0	2.5	5.0	--
PVA HT	12.0	13.0	15.0	7.5	9.5	18.0	18.0	13.0*	12.0*
PVA	1.0	1.0	--	7.5	9.5	2.0	2.0	1.0	1.0

* PVA HT from Cone Dryer

Table 3

Results for Dry Pressed AP/AN/Al/Wax Samples
at 500 and 1000 psi in Nitrogen

AN	AP	Al	Wax	500 psi *	1000 psi *
85	0	10	5	NB	NB
75	10	10	5	NB	NB
65	20	10	5	NB	NB
55	30	10	5	NB	NB
45	40	10	5	NB	NB
35	50	10	5	P	P
25	60	10	5	B (Uneven)	B (Uneven)
15	70	10	5	B	B
5	80	10	5	B	B

* NB: No burn

P: Partial (3 mm or less) burn

B: Burned to completion

COMBUSTION STUDIES OF JET PROPELLANTS

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Final Report on Aerojet Contract P. O. #410386
June 1991

Objectives:

The general goal of this research was to achieve high combustion efficiency and improved burning rate characteristics of aluminized propellants with nitrate oxidizers, particularly of solution propellants under development by Gen Corp Aerojet.

Approach:

The approach was to study combustion of small samples in laboratory scale burners, including combustion window, quench, and particle collection "bombs" and CO_2 laser pyrolysis, along with optical and SE microscope studies of quenched samples. Such studies, in combination with systematic modifications of propellant formulation, provide a quick route to qualitative understanding of combustion and propellant optimization.

Summary of Research:

The research is described in the body of the report in three phases, which emerged as a result of two extended interruptions in the study due to discontinuities and reorientations of the parent Gen Corp Aerojet project.

In the first phase (July 1987 to February 1988), studies were made of model propellants prepared by dry-pressing mixtures of AN, AP, polymer and aluminum powders (no Aerojet propellants were available yet). It was found that AN:AP ratios higher than 25:60 gave poor combustion behavior (1000 psi), with irregular burning and minimal aluminum ignition. Use of combustion aids and optimum binder resulted in AN/AP ratios greater than 25:60 with regular burning and fair aluminum ignition. Work was suspended before optimization of combined aids was attempted.

In the second phase (August 1989 to December 1989), studies were made of combustion of propellant samples supplied by Gen Corp Aerojet (solution propellants). This phase of the studies was relatively nonproductive because of several features of combustion that impeded photographic observation (samples that would not burn, burning down the sides of end-burning samples, accumulation of residues (char) on the surface, and clouds of aerosol from the surface). These are all significant findings regarding combustion of this novel propellant, but singular, unexpected, and not particularly helpful in getting to the target of controlling burning rate and aluminum combustion. In particular, observation of the aluminum combustion was obstructed by residue and smoke obscuration. The propellant apparently burns with substantial release of undecomposed oxidizer from the surface, formation of char on the surface, and intense combustion of accumulated aluminum in the char layer. In most samples, a column of residue was left after the sample was consumed, with some unburned aluminum remaining. The practical significance of these novel features of the combustion, or their relevance to combustion in a motor environment remain uncertain.

In the third phase (June 1990 to September 1990), recourse was taken to atmospheric pressure laser pyrolysis tests in order to simulate combustion under conditions where less obscuration would be present. Samples were observed by video photography in a nitrogen flushed test chamber with incident infrared surface heating at about 320 watts/cm^2 by a CO_2 gas laser. Tests were run on all the propellant in the first shipment from Aerojet (Table 1) and all samples of a second shipment (Table 2). Full information on formulation was not supplied; however, each set of samples consisted of an unaluminized sample and a range of aluminum concentrations. In the laser pyrolysis tests, the details of surface behavior and aluminum combustion were relatively observable.

Experimental Methods

Evaluation of the performance of nitrate based propellants was conducted using facilities in the combustion and microscopy laboratories in the School of Aerospace Engineering. The combustion lab contains: (1) a high pressure window bomb with lighting, video, and motion picture cameras; (2) a high pressure quench bomb; (3) decomposition equipment (DTA, DSC, TGA); (4) a 1000 watt CO_2 laser with beam optics and optical table; and (5) sample preparation, handling, and storage

facilities. The adjacent microscopy laboratory consists of scanning electron and optical microscopes with associated heating stages.

These laboratories have been used extensively for studying propellant (primarily ammonium perchlorate based) combustion as well as the high temperature decomposition of individual ingredients. These studies of nitrate based propellants concentrated on the well established tools of propellant research: combustion photography and microscopic examination of quenched surfaces, as well as a relatively new procedure: laser assisted heating.

I. Video and Cine Photography

The most powerful and convincing available method of evaluating the combustion of solid propellants is cinephotography. All of the old saws: "seeing is believing" and "a picture is worth a thousand words" apply to this method. Burning rates as well as the uniformity of burning rate can be measured and qualitative features such as the combustion behavior of aluminum (accumulation, agglomeration) can be determined. The ability to witness the event and observe unanticipated processes is a valuable research tool.

The normal combustion facility photographs a small parallelepiped of propellant ignited on one end. In an actual rocket motor, the burning surface will experience a cross flow of propellant gases ranging from low in the head end to high at the aft end. This cross flow leads to higher burning rates (erosive burning) and is dependent on the actual location within the motor. Thus, it is important to note that standard combustion photography provides "non-erosive" or end burning. Thus, in an actual motor the local burning rate and aluminum behavior will depend on the character of the cross flow and thus will vary from point to point within the motor. The end burning results provide only a baseline.

Photography of propellants has traditionally been done by high speed (1000 to 10,000 frames/sec) 16 mm motion picture (cine-) photography. Unfortunately, the convenience of video has all but eliminated the amateur motion picture industry. Film processors are limited to a handful of national services with attendant shipping delays. High speed and high resolution have given way to

instant replay and low cost. Useful videos are limited to exposure times of 1/1000 sec but are adequate for measuring burning rate and observation of gross behavior. High magnification is still accomplished by lens extension tubes and close-up (macro-) lenses. Resolution is limited with the current generation of television monitors.

Propellant samples are mounted in a stainless steel chamber with quartz windows for external illumination and photography. Illumination is provided by a 2500 watt Xenon lamp. Nitrogen is used to pressurize the chamber and to provide a flushing flow parallel to the sides of the sample for removal of the smoke. As previously noted, this flushing flow is not parallel to the burning surface and does not provide a cross flow to affect the burning surface.

II. Surfaces of Quenched Samples

Another useful tool for investigating combustion behavior is optical and electron microscopy of quenched samples. A quench bomb is similar to a window bomb except that the top is sealed with a stack of thin mylar disks trapping a nichrome wire. The chamber is pressurized, the sample is ignited, and after a brief delay the nichrome wire is heated by electrical resistance. The mylar diaphragm bursts, the chamber is depressurized in milliseconds, and the burning sample is quenched. The sample is then examined in a scanning electron microscope to obtain details on the microstructure of the burning surface.

For a heterogeneous propellant, the surface consists of partially burned ammonium perchlorate regions, divided by mounds of binder coated aluminum. Many details can be inferred by the surface structure of the AP, the size of the aluminum accumulates, and other microscopic features.

III. Laser Pyrolysis

An alternative to the high pressure window bomb is pyrolysis with a CO_2 laser. Radiation from these lasers is in the infrared (10.6 micron) region and is thus an effective heating source. High power CO_2 lasers (about 1000 watts) can produce heat fluxes on the same order as a burning propellant. The lower density at atmospheric pressure reduces the smoke density and thus the

obscuration. Since the laser flux must enter the atmospheric chamber perpendicular to the burning surface, the flushing flow is removed from the side producing a cross flow parallel to the regressing surface. Previous tests heating a solid polymer revealed a strong shearing force on the pyrolyzing surface. Thus, the laser heating tests will permit clearer observation of the burning surface and thus the behavior of aluminum on the burning surface.

The purpose of the laser heating tests is to investigate the combustion behavior of the aluminum in these propellants. This is important because unlike the oxidizer and fuel which react at the burning surface (within several hundred microns), the aluminum is ignited at the surface but normally continues burning in the gas flow through the rocket motor. The transition from a single aluminum particle in the propellant to an ignited mass in the gas flow is important in determining combustion efficiency, slag formation, acoustic damping, and other parameters. Before describing the behavior of the aluminum in each of the samples, it is worthwhile reviewing the possible scenarios for aluminum combustion in solid propellants and their efficiencies. From an analytical viewpoint, the simplest path would be for an isolated aluminum particle (in a lightly loaded composition) to arrive at the burning surface, detach from the surface, ignite and burn in the gas flow. This is a simple and efficient path, but it is not common. In practice, when an aluminum particle arrives at the burning surface it is in contact with other aluminum particles (more so as the aluminum content is increased) and the relatively high temperatures cause the particles to sinter together to form an accumulate.

The accumulate is basically an irregular chain of sintered particles which eventually begins a slow, exothermic oxidation and thus increases in brightness. The accumulate may linger on the surface feeding heat back to the surface and/or may detach to burn in the gas flow. If the surface (or gas) temperature is high enough, the oxide shells defining the original particles will collapse (possibly melt) and all of the aluminum will coalesce into a large sphere called an agglomerate. The agglomerate will usually detach at some point and burn in the gas flow. The ignited agglomerates are extremely bright and comet-shaped with a convective trail.

In summary, in video coverage of the laser sustained combustion, the

simplest (and possibly ideal) case of single particle detachment would appear as numerous small, very bright streaks above the surface. In the more realistic case of ignited agglomerates, bright spheres would move across the burning surface (as the sintered accumulate draws up into an agglomerate), detach and be convected slowly away from the propellant surface. If the aluminum does not agglomerate it will appear as less bright irregular shapes on the surface and possibly in the gas flow. Aluminum combustion by this path is less efficient since combustion may not be complete by the time the accumulate leaves the rocket motor. Finally, the aluminum may not detach at all but rather form a sintered glowing bed on the surface. This has the worst efficiency and the self heating may cause the propellant to continue "burning" even after the laser heating is removed.

Results

I. Phase I: Investigation of Dry Pressed Nitrate Propellants

The first phase of this investigation involved attempts to produce in-house propellants using ammonium nitrate, powdered binders, and aluminum. Due to the poor performance of ammonium nitrate, ammonium perchlorate was added to produce a mixture of AP and AN capable of sustaining combustion. Established methods of coating AN to reduce the hydroscopicity as well as burning rate stabilizers were also investigated.

A. Oxidizer Self Deflagration

Initial experiments attempted to establish a baseline for the combustion of ammonium nitrate. Ammonium perchlorate will burn as a monopropellant in nitrogen at pressures above 300 psi. Ammonium nitrate (dry pressed into a parallelepiped) would not burn at 1000 psi in nitrogen or methane (a fuel). A dry pressed mixture of 90% AP and 10% AN burned irregularly at 1000 psi in nitrogen while an 85/15 mixture did not burn.

B. Combustion of Dry-Pressed Oxidizer-Fuel Combinations

In order to avoid problems associated with small scale propellant mixing

(inaccurate mixtures, trapped air bubbles, long curing times), pressed mixtures of dry powdered ingredients were used. While the oxidizers and aluminum are dry powders, a solid powdered hydrocarbon is substituted for the normally liquid binder to produce a dry mixture. The dry powders are measured, mixed and poured into a stainless steel die. The die is hydrostatically pressed at 19,700 psi and held for several hours to produce a compact sample. The sample is mounted in the window bomb, pressurized (with nitrogen to 1000 psi except as noted) ignited and recorded on video tape.

Variation of Oxidizers

The initial mixture consisted of 85% oxidizer, 10% aluminum and 5% carnauba wax (a dry powder binder substitute). Tests at 500 psi and 1000 psi indicated that a 60% AP and 25% AN mixture burned unevenly while a 50/35 mixture would not. Results of this series of tests are shown in Table 3. Switching to a mixture of 90% oxidizer, 9% binder (with 1% aluminum just to indicate ignition performance), gave a formulation closer to stoichiometric without the heat sink associated with heavy aluminum loadings. This mixture required a 70% AP, 20% AN ratio to sustain combustion.

Variation of Fuels

Powdered carnauba wax has been used extensively as a dry binder and produces good ignition of aluminum. Substitutes including ABS (Acrylonitrile/-Butadiene/Styrene) resin, Acrylonitrile/Butadiene Copolymer and Polyacrylonitrile were also used as dry binders. ABS was available in a more desirable particle size range and the 50% AP/25% AN/10% ABS/5% Al sample burned more evenly (but with greater aluminum agglomeration) than did the equivalent sample with wax.

Variation of Ammonium Nitrate

Due to the hydroscopic nature of ammonium nitrate, it must be kept desiccated. Sample preparation is delayed by inclement weather and quenched samples often deteriorate before examination in the scanning electron microscope. In an attempt to create a moisture barrier, Silane, a standard protective coating for AN, was used to coat a batch of AN. The coated AN was marginally better at

resisting clumping and dissolving in high humidity and produced slightly more even combustion. The most notable improvement came with the use of NiO phase stabilized AN. Although only preliminary work was carried out using NIOPSAN (because it was received after the stop work notice, it showed substantial improvement with regard to AN/AP ratio for good combustion and aluminum ignition.

Burning Rate Stabilizers

Sodium barbiturate is reputed to be a necessary additive in some applications for obtaining consistent, uniform burning of AN. The addition of 1% sodium barbiturate to the formulations with marginal burning characteristics gave improved results, but this line of investigation was also terminated by the stop work order.

Results from video recording of early emulsion propellants supplied by Aerojet were of acceptable quality, but revealed poor performance. Early samples did not burn (at 1000 psi) while later samples burned with a measurable burn rate but without aluminum ignition. The combustible sample (C728-46C) retained aluminum filigrees which were coated with a black carbonaceous residue. Substituting air for nitrogen as a pressurizing and flushing gas removed the carbonaceous residue and aluminum detached but did not ignite.

Phase II.

The primary objective of Phase II was burning rate measurement and evaluation of aluminum ignition behavior of the A3L series of propellants from Aerojet General Corp. using combustion photography. The composition of these samples is listed in Table I. Unfortunately, observation of all samples photographed in the window bomb was limited due to "smoke" produced by these propellants. Combustion was superior to the original emulsion propellants, but the A3L leaves a fine ash residue which accumulates on the burning surface, deflecting the products of combustion and filling the volume between the window and the sample with a dense smoke. Sides of the sample were coated with various inhibitors in an effort to eliminate side burning, but the surface was usually concealed by the inhibitor. The aluminum did not ignite and leave the surface

but remained trapped in the residue, glowing due to exothermic oxidation.

While viewing the details of combustion of A3L propellants was impossible due to smoke and residue, it was often possible to measure the rate of advance of aluminum self luminosity through the sample. These rates were close to the measured burning rates supplied by ASPC. Unfortunately, the residue did not permit the identification of a clearly definable surface (such as the gas-solid interface normally seen in AP-based solid propellants) and subsequent measurement of the regression rate of this surface. Thus, while measurement from most cinephotography is unambiguous, the actual surface is subject to speculation in the A3L combustion photograph and thus its regression rate is uncertain. High speed motion pictures of the combustion in place of video photography increased both time and spatial resolution of clear areas. The actual burning surface was still obscured, but the dense smoke was observed to be emitted in small unsteady jets. It is probable that this residue would be swept off by the cross flow in a real motor and would not be a significant factor in propellant combustion. It is simply an impediment to observation. The effect of the residue on aluminum behavior could not be evaluated in these tests.

In summary, the visual recording processes, both cinematography and video, which were effective in evaluating the combustion of dry pressed propellants with AN and AP propellants were not capable of producing unambiguous burning rates or evaluation of the accumulation and agglomeration characteristics of aluminum. They did reveal a tendency for the A3L propellants to form a surface residue in an end burning or cigarette configuration and to emit dense "smoke" from the surface, possibly oxidizer aerosol.

Quench testing of the A3L samples was not hindered by the smoke or residue (which was stripped by the rapid depressurization) but was limited by the near homogeneity of the samples. The Aerojet propellants are only heterogeneous because of the aluminum. During the rapid depressurization, the surface residue and accumulated aluminum was stripped off. Optical examination of the burning surface revealed a liquid or glazed surface with patches of aluminum that appeared to be below the glazed surface. After coating with gold and examining in a scanning electron microscope, the liquid layer seems to have evaporated

leaving a fairly nondescript (but irregular) surface with areas of high concentration of aluminum. Whereas accumulates of aluminum are normally seen on the surface of a heterogeneous AP propellant, the aluminum is buried in the Aerojet propellants with only slight bumps to indicate their presence. Although a comprehensive SEM study of quenched nitrate propellants might reveal subtle but important characteristics, the lack of any obvious distinct features at this point forced our efforts toward methods with a higher guarantee of success.

Phase III.

The objective of the final phase remained the evaluation of the combustion efficiency of the aluminum in the A3L series. Due to the lack of success with video and cine photography in the high pressure window bomb, video recording of laser assisted heating in an atmospheric chamber was pursued. Measurement of the regression rate of samples irradiated with 550 watts of power (700 watts at the laser) produced a regression rate value about 40% of the burning rate (as supplied by Aerojet) at 500 psi. Smoke and residue ash were often found in the exhaust, but did not obscure the observation of the pyrolyzing surface.

Laser heating tests were carried out in a thin walled test chamber with a nitrogen atmosphere and purge. The chamber is scavenged through a side port which creates a cross flow of nitrogen across the pyrolyzing surface of the sample. The laser flux enters through a Zinc Selenide window on the top while quartz side windows permit illumination and video. Laser power was set at 700 watts at the shutter for all tests, resulting in 550 watts over a 1.5 cm circle at the sample.

Multiple runs on each of the eight propellants in the C478-74-X series (Table 1) and a second shipment of samples labeled C494-29A to G and 652-81 and 652-82 (Table 2) have been analyzed and the results are listed below:

C478-74-1: Some small, fast burning particles (single particles or small agglomerates) convect with the gas flow. Some large agglomerates form and detach. Some glowing spherical agglomerates remain on the surface near the edges.

C478-74-2: A few small, ignited particles are seen in the gas flow but most aluminum is retained on the surface forming a large glowing bed of accumulated aluminum. This sample had the poorest efficiency.

C478-74-3: Small surface accumulates form and detach to burn as agglomerates.

C478-74-4: More surface accumulation. Detached aluminum does not appear to spheroidize, implying only limited aluminum reaction. Burning accumulates/agglomerates are larger than -3.

C478-74-5: Substantial surface accumulation. Accumulates ignite to form large agglomerates. Burning agglomerates leave the surface.

C478-74-6: Bright surface ignition, a few single particles or small accumulates are seen in the gas flow. Numerous large agglomerates ignite and detach.

C478-74-7: Large surface accumulates are formed. Accumulates change to large agglomerates on the surface and detach reluctantly. After the laser was switched off, a large surface accumulate continued glowing, feeding heat back to the surface and continuing to pyrolize the solid. The sample continued burning several seconds before finally extinguishing.

C478-74-8: Basically good ignition with large agglomerates which detach and are convected away.

C494-29A: Burns with surface accumulation with vigorous aluminum combustion in the accumulation. Some agglomerates appear to escape the surface (not many). Burning seems to fade only slowly after laser cut-off.

C494-29B: Burns with surface accumulation and very large and bright aluminum combustion in the accumulation, with a little spewing of aluminum outward (fine streaks). Large Al reaction sites in accumulate fade only slowly after laser cut-off.

C494-29C: Burns with some conventional outward-moving agglomerates and some aluminum combustion in a surface accumulation. The brighter burning accumulates

were slow to fade after laser cut-off.

C494-29D: Similar to 29C, but with fewer detaching agglomerates, more surface accumulation and larger bright sites in the accumulation, reluctant to go out after laser cut-off.

C494-29E: (Non-aluminized) (The beam is centered to left side of sample top surface.) Massive accumulation of irregular structures.

C494-29F: Large accumulate structures, bright areas that appear to be mobile but are probably propagating through a relatively nonmobile accumulate structure. Brightness comparable to 29D, but spews some bright streaks. Fades quickly at laser cut-off.

C494-29G: Forest of surface accumulate with aluminum combustion in the accumulate with some spewing and Al_2O_3 smoke. Fades fast on laser cut-off.

652-81: Forest of accumulates with bright aluminum reaction sites that propagate busily with some spewing and streaking. Very few agglomerates leave the surface. Bright sites fade only slowly after laser cut-off.

652-82: Similar to 652-81 except Al combustion sites in the accumulate are brighter, larger, and persist after laser cut-off (sample burns to completion).

SUMMARY COMMENTS

Limiting Conditions on Investigation and Experimental Problems

In this section, an effort is made to describe combustion of A3L propellants. This should be prefaced by a general comment on the character and limited success of the investigation and credibility of results. a) Because of the stop-start nature of the program (two extended "stop work" orders) and shifting propellant systems, there was only limited opportunity to study the final candidate propellants. b) Interpretation of results is hindered by the limitation of tests to a set of propellants provided by Aerojet, in which variation of formulation variables was chosen to meet total program goals, but

not well suited to a combustion mechanism study. c) There were a number of unique difficulties involved in testing this propellant, that frustrated the usual methods for studying combustion.

As a result of these frustrating (but unavoidable) circumstances, the picture of combustion of A3L propellant that emerged is incomplete and speculative. What is clear is that A3L propellant combustion is very different from that of other well-studied propellants.

Combustion Photography Results

In window bomb tests of A3L propellants at 1000 psi, the combustion photography shows a dense cloud of aerosol, with relatively stationary areas of intense luminosity seen diffusively through this "fog," where aluminum is present in the propellant. Unlike other propellants, no view of the burning surface is possible because of the smoke. This was further complicated by a propensity for rapid flame spreading down the sides of the samples. After burnout there was usually a substantial amount of ash, sometimes still in place as a column where the sample had been. Residual ash occurred with both aluminized and non-aluminized samples, and a major part of the aluminum combustion occurred "in place" in the ash column. The residual ash did not appear to contain unburned aluminum (no quantitative analysis made). The window bomb tests revealed completely novel aspects of A3L propellant combustion (aerosol, ash, constraint of burning aluminum in the ash). The aerosol may have been a fog of HAN. It seems likely that ash concentration on the surface would be less in the flow environment of a motor. Also, it has been suggested that the propellant may have a second mode of steady state combustion in which the "ash" material is oxidized at (near) the surface, with heat release and corresponding minimization of surface accumulation of either ash or aluminum. While this possibility cannot be ruled out, there was nothing marginal about the ignition in the window bomb tests, and the burning was usually very intense due to aluminum combustion. It is worthy of note that, in Sandia studies of liquid gun propellants with the same oxidizer, the liquid burning was dominated by the oxidizer decomposition, with oxidation of the fuel ingredient occurring as a "fog" burning some distance from the surface. This was attributed to the low exothermicity of the oxidizer decomposition, along with low activation energy for the decomposition reaction.

In the solid propellant, the fuel is less easily convected away than the liquid fuel fog, leaving a complex, flow environment-dependent fuel burning process above the surface. There is an obvious need for much more detailed study of this unique combustion process.

Laser-Assisted Combustion at One Atm.

In an effort to gain a better view of the surface processes during combustion, tests were run in an atmospheric-pressure nitrogen-flushed test chamber with infrared CO_2 laser illumination to support combustion. This allowed a pyrolysis rate comparable to that in combustion at 1000 psi, but with less smoke obscuration. In these tests, smoke obscuration was minimal and the ash concentration was revealed as an extraordinarily complex sponge-like structure. With the unaluminized propellant, the accumulation appeared to "flow" laterally on the surface under the influence of a mild lateral cross flow due to side-venting of the flushing gas. Since the material is presumably vaporizing at its surface, it is difficult to be sure that the apparent motion is really "flow."

With aluminized formulations, the surface accumulation did not have this appearance of flow. It was clear that aluminum burning occurred in the char layer. In fact, the luminosity was so bright that other features of the surface layer were difficult to resolve (because of low luminosity). In the motion pictures the luminous material appears to exhibit local, more or less random, motions, but careful examination suggests that the sponge-like structures are not moving, but the luminous processes are. It seems likely that portions of the structure are sintered aluminum accumulations, and that combustion spreads through these structures. Unlike conventional propellants, this does not lead to formation of detaching aluminum agglomerates. The aluminum seems to usually burn in place, with the intense reaction sites probably being governed by extent of aluminum concentration, extent of the local oxidizer flow, and heat-up by the laser beam.

The detailed description in the test characterizes these surface features for each formulation. These descriptions do not allow much insight into formulation effects because a) the extraordinarily complex behavior was not

conspicuously different for the different propellants, and b) it is difficult to rank the propellants according to specific formulation variables because formulation variables were generally changed more than one at a time. Aside from the features mentioned above, the most distinctive behavior was with formulations C-652-82 and C-494-29C. C-652-82 exhibited very large and persistent accumulate structures on the surface. It continued to burn after laser cut-off. the top of the surface structures became less bright, while the under side continued to glow brightly, apparently due to continued aluminum combustion. Formulation C494-27C burned somewhat more like conventional propellants, with aluminum agglomerating and moving away from the surface when first ignited. Some surface accumulation developed as burning continued, but to a lesser degree than most of the aluminized samples.

Unfortunately, the laser experiment became operational so late in the program that it was not fully exploited (most of the tests were run after the end of the contract). It would be desirable (at the very least) to do a chemical and microscopic study of the residue on sample surfaces remaining after laser cut-off.

Summary

Combustion of A3L propellants appears to proceed by an exothermic oxidizer decomposition at the surface with possible emission of an oxidizer aerosol as well. It is not clear how the polymeric fuel decomposes, but it appears to accumulate on the surface as a porous structure, through which the oxidizer or its products must pass. In our tests it could not be determined whether the accumulation oxidized in place or broke away from the surface.

In metalized formulations the aluminum was impeded from leaving the surface by the surface concentration, and burned instead in this layer (and probably added aluminum oxide structures to the layer). Because of the high temperatures associated with the aluminum combustion, the polymeric fuel structures would be decomposed and then oxidized. The remaining surface structures would then presumably be aluminum oxides, carbides, and nitrides. These energetic reactions must contribute to the heat flow that sustains burning, but probably in a very pressure dependent way, presumably modified on the motor environment also by the combustor gas flow.

To the extent that surface concentration occurs in a motor environment, it has several practical implications:

a) Ignition is not fully achieved until the layer is built up.

b) Because the layer is the site of energetic reactions, processes that modify accumulation and reaction there should affect burning rate.

c) If, as seems likely, accumulated material "tears away" from the surface, it may not react fully in small motors.

d) Formation of aluminum concentrations may lead to relatively slow combustion of the aluminum, and to a larger than usual size of particles or droplets in the two-phase flow, with correspondingly modified two-phase flow problems.

Of course, these singular effects would be absent if no surface concentration occurred in the flow environment of a motor. Then burning would be more fully dominated by the primary reactions.

Table 1

Composition of C478-74-X Series of Propellants

Component	1	2	3	4	5	6	7	8
AL MDX-65	15.0	7.5	18.0	9.0	22.0	11.0	26.0	13.0
H-60	--	7.5	---	9.0	---	11.0	---	13.0
PVA	13.0	16.0	13.0	16.0	16.0	13.0	16.0	13.0
Burning Rate Add.	2.0	3.5	3.5	2.0	2.0	3.5	3.5	2.0
Oxidizer	70.0	65.6	65.5	64.0	60.0	61.5	54.5	54.0

Table 2

Composition of C494-29 and 652-81 Series of Propellants

	C494-29A	29B	29C	29D	29E	29F	29G	652-81	652-82
S-HAN-5	65.3	62.0	65.0	65.0	81.0	70.0	75.0	62.0	65.3
GNX	3.7	4.0	--	--	--	--	--	4.0	3.7
Al MDX-65	18.0	15.0	15.0	10.0	--	5.0	2.5	15.0	18.0
Al H-60	--	5.0	5.0	10.0	--	5.0	2.5	5.0	--
PVA HT	12.0	13.0	15.0	7.5	9.5	18.0	18.0	13.0*	12.0*
PVA	1.0	1.0	--	7.5	9.5	2.0	2.0	1.0	1.0

* PVA HT from Cone Dryer

Table 3

Results for Dry Pressed AP/AN/Al/Wax Samples
at 500 and 1000 psi in Nitrogen

AN	AP	Al	Wax	500 psi *	1000 psi *
85	0	10	5	NB	NB
75	10	10	5	NB	NB
65	20	10	5	NB	NB
55	30	10	5	NB	NB
45	40	10	5	NB	NB
35	50	10	5	P	P
25	60	10	5	B (Uneven)	B (Uneven)
15	70	10	5	B	B
5	80	10	5	B	B

* NB: No burn

P: Partial (3 mm or less) burn

B: Burned to completion

Appendix A

EXPLANATION OF VIDEO RECORDS

In the experimental set-up, the test sample is arranged relative to the camera as in Fig. A-1, with external illumination from the upper right and mild N_2 flushing flow from the bottom, vented to the left. The sample is tilted relative to the focal plane so that the image is in focus across the middle of the surface ignited by the laser beam. The camera is started before the laser exposure starts, runs until well after the laser beam shut-off. Multiple tests were made on most formulations for a variety of reasons.

a. Poor laser beam positioning

b. Camera problems

c. Singular combustion behavior

d. Run tests with and without neutral density filters to cope with extreme difference in brightness due to metal flame.

The descriptions of results in the text are based on review of all tests. The video tape provided gives short scenes from typical pictures of the propellants in Table 2.

In viewing the tape it should be understood that the scene is a diagonal downward view of a VERY complex surface, consisting of what is often a forest of "accumulate" structures. Sometimes there are aluminum agglomerates or bursts of fine droplets leaving the surface; the low video framing rate and N_2 flow prevent tracking of the progress of this detached aluminum. However, most of the aluminum combustion (most formulations) occurs in the forest of accumulated surface material. The resulting bright sites seem to have high mobility. However, most of this "mobility" is actually spreading of inflamation through the structure of a relatively stationary accumulate forest.

Comments in the text pertain to such features as brightness of combustion, presence or absence of detaching aluminum agglomerates or spewing of fine droplets, extent or structure of surface accumulation, and behavior after laser cut-off (the top of the accumulate forest darkens quickly, but active aluminum combustion sites sometimes continue to burn, so that the under side of the surface accumulation remains luminous after the top has turned dark.