

Extruded Composite Propellant Technology Development

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This paper discusses the development of technology for the production of extruded composite propellant formulations. These formulations are based on thermoplastic elastomers and curable polymer gumstocks as the propellant binder, and utilize ammonium perchlorate as the primary oxidizer. Propellant mixing and other processing procedures are addressed. Evaluations of potential binders, processing details, and theoretical performance characteristics are included to define the parameters used as acceptability criteria. The primary candidates for continued development are identified and details of the processing and evaluation are given.

Introduction

HISTORICALLY, extruded propellant used in gun systems and small tactical rocket motors has been based on homogeneous single- and double-base propellants. Later development of triple-base and modified double-base propellants for guns and rocket motors has shown that nonhomogeneous propellants may be successfully extruded.

Most recently, the development of propellants suitable for Low Vulnerability Ammunition (LOVA) using cyclotrimethyl trinitramine (RDX) or cyclotetramethylene tetranitramine (HMX) as energetic materials and a thermoplastic binder has produced extrudable propellants, although these systems use solvent extrusion processes.¹⁻³ Solids loadings of up to 90% have been produced successfully.

Prior efforts, reported in the restricted literature, in the development of extruded composite propellant technology used systems similar to castable composite propellants based on liquid polymers such as hydroxy-terminated polybutadiene and carboxy-terminated polybutadiene acrylonitriles.⁴ These systems were partially cured to produce a semisolid propellant which was then extruded. The success of this approach was limited by difficulty in terminating the curing reaction at the proper time to produce an extrudable propellant without damage to the polymer network during the extrusion process. The extrusion was followed by a continuation of the cure to produce the final mechanical properties desired. The variability in precure times and concomitant curing within the network resulted in nonuniform hardness throughout the precured mass of propellant. The nonuniformity of the propellant resulted in generally unsatisfactory propellant grains during the extrusion process.

Initial development of the LOVA gun propellants was based on liquid polymer binders and the same basic problem was encountered. One thermoplastic elastomer binder, Kraton G 1652 (Shell Chemical Co.), was included in the study. Thermoplastic elastomers as a class are nonhomogeneous polymeric systems, in which an elastomeric polymer has been copolymerized with a thermoplastic material to produce a molecule having a central block of elastomer and terminated at each end with thermoplastic blocks. The end result of this molecular construction is a material which has the properties of a vulcanized rubber but which, unlike thermosetting or vulcanizing rubbers, can be processed in a manner similar to thermoplastics. The system may be remelted and reprocessed a number of times and provides the user with significant advantages

over the thermosetting or vulcanizing rubbers. The initial processing attempts were successful, and additional thermoplastic elastomeric binders have been evaluated with varying success.

Elastomeric gumstocks, which are basically prepolymerized liquid polymers, may also be used as propellant binders. The following approaches have been in general use for many years. The elastomeric gumstock may be softened by heating and plasticization and the solids incorporated either through solvent or high-shear mixing. Polyisoprene has been used in this manner for both high-energy and gas generator propellants.

Polyisoprene has been used to produce propellants which are thermoplastic as well as thermosetting. Similar gumstocks have been produced from virtually all of the liquid polymer systems (polyurethanes, polybutadienes, etc.). The incorporation of curing agents is required for propellants which must operate at higher temperatures.

The application of thermoplastic elastomers or gumstocks to the development of extrudable composite propellants for rocket motors, with performance and physical characteristics similar to those found earlier with the castable composite propellants, was a natural outgrowth of the progress in the LOVA program.

Experimental Program

Objectives

The objective of this program is to develop and evaluate extruded composite propellants based upon thermoplastic elastomers and/or gumstocks using ammonium perchlorate as the principal oxidizer. Formulations are to be without primary smoke. The potential applications are in mass-produced rocket motors, gas generators, starter cartridges, and other propellant-actuated devices.

Approach

The general approach consists of the development of propellant formulations based on polymer and oxidizer studies, evaluation of processability and extrusion capability, demonstration of inhibitor feasibility, selection or development of propellant grain configurations, and manufacture of propellant grains for testing and evaluation.

Research and development has been conducted in seven primary areas. These were: the selection of viable polymeric materials (binders), the evaluation of plasticizer types and levels, the incorporation of curing agents, the evaluation of extrusion parameters and grain configuration, the evaluation of ammonium perchlorate and RDX as oxidizers, the evaluation of inhibiting materials, and the performance of ballistic and mechanical properties testing.

Propellant mixes based on initial formulations were extruded, and strand burning rate, mechanical properties, and

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Table 1 Plasticization of Kraton G 1652

Characteristic	Tufflo 6056 formulations		Tufflo 6016 formulations	
	1	2	1	2
Plasticizer, % of binder	20.0	20.0	45.0	55.0
Solids loading, %	90.0	89.0	90.0	90.0
Maximum tensile strength at -55°C, MPa	5.0	5.0	4.4	5.6
Strain at maximum tensile at -55°C, %	3.2	2.8	4.5	3.4
Maximum tensile strength at 74°C, MPa	0.2	1.7	0.7	0.6
Strain at maximum tensile at 74°C, %	5.1	4.5	3.9	3.0
Density, g/cm ³	1.67	1.68	1.71	1.72
Specific impulse, s	250.0	247.0	250.0	250.0

Table 2 Ballistic and physical properties of Kraton G 1652 and Estane 5712 formulations

Characteristic	Kraton formulations						Estane formulation
	1	2	3	4	5	6	
Binder, % of weight	11.84	7.98	7.84	5.48	5.39	5.48	10.24
Plasticizer, % of weight	2.96	1.99	1.96	4.49	4.41	4.49	2.56
Solids, % of weight	85.20	90.03	90.20	90.03	90.20	90.03	87.20
Burning rate at 6.9 MPa, mm/s	9.4	15.4	20.3	17.0	23.4	23.2	—
Burning rate slope	0.54	0.61	0.70	0.58	0.68	0.68	—
Density, g/cm ³	1.59	1.67	1.68	1.71	1.69	—	—
Tensile strength at -55°C, MPa	8.2	5.0	5.0	4.4	4.6	5.6	9.6
Elongation at maximum stress at -55°C, %	5.4	3.2	2.8	4.5	4.3	3.4	3.8
Tensile strength at 74°C, MPa	0.4	1.5	1.7	0.7	0.7	0.6	0.7
Elongation at maximum stress at 74°C, %	4.1	5.1	4.5	3.9	3.1	3.0	10.1

Table 3 Cure ratio effects on Hycar 4004

Characteristic	Cure ratio, NCO : OH							
	0.6 : 1	0.7 : 1	0.8 : 1	0.8 : 1	0.8 : 1	0.83 : 1	0.87 : 1	0.9 : 1
Functionality	2.0	2.0	2.0	3.0	2/3	2.0	2.0	2.0
Solids loading, %	90.0	87.0	87.0	87.0	87.0	87.0	87.0	87.0
Maximum tensile strength at -55°C, MPa	4.0	4.0	3.7	4.1	3.8	10.6	8.8	4.5
Strain at maximum tensile at -55°C, %	8.9	15.0	26.0	7.7	14.0	8.2	16.2	18.0
Maximum tensile strength at 74°C, MPa	0.4	0.5	0.7	0.6	0.6	1.4	1.2	0.7
Strain at maximum tensile at 74°C, %	6.5	44.0	25.0	6.5	25.0	24.0	33.0	47.0

density were determined. From these initial formulations, additional modifications were introduced to evaluate burning rate range capability and mechanical properties improvement. Working life evaluations were also performed as applicable.

Materials Selection and Discussion

Polymeric Materials

Selection of polymeric materials suitable for inclusion in this program was based on the evaluation of 27 potential binder materials using the manufacturers' data sheets and application recommendations. Using glass transition temperature and softening point as ranking factors, 17 binders were selected for laboratory evaluation. The laboratory evaluations were conducted using solvation, subscale pellet pressing, and thermal properties as criteria. Laboratory testing resulted in the selection of eight binders for evaluation in intermediate-scale propellant mixes.

Propellant mixes were successfully produced and extruded from three of the eight binders. The binders comprise one

nonreactive thermoplastic elastomer, Kraton G 1652, and two reactive elastomers, Hycar 4004 and Estane 5712 (B. F. Goodrich Co.). See Tables 1-4.

Plasticization

Hycar 4004 formulations were formulated using dioctyl adipate (DOA), as this plasticizer has long been utilized in castable propellant systems with no significant adverse effects. The plasticizer was incorporated into the binders at the 19 to 20% level.

Kraton G 1652 formulations initially incorporated Tufflo 6056, the plasticizer used in the LOVA formulations, though at lower concentration levels. The initial formulations using Tufflo 6056 were at the 20% plasticizer level and the propellant modulus was very high; later formulations incorporated Tufflo 6016 at 45 and 55% concentrations and the propellant modulus was reduced, although still quite high (Table 1). The higher plasticizer levels did not adversely affect the density or specific impulse of the propellant formulations, but did enhance processibility somewhat (Table 2). Extruded propellant

Table 4 Ballistic and physical properties of Hycar 4004 formulations

Characteristic	Formulations						
	1	2	3	4	5	6	7
Curative ratio, HCO:OH	1.10	0.80	0.86	0.86	0.86	0.70	0.90
Binder, % of weight	12.30	10.45	10.95	10.95	8.76	10.45	10.45
Plasticizer, % of weight	2.70	2.55	2.55	2.55	1.87	2.55	2.55
Solids, % of weight	85.00	85.00	86.50	85.00	90.30	85.00	85.00
Iron oxide, % of weight	—	2.00	—	1.50	—	2.00	2.00
Burning rate at 6.9 MPa, mm/s	6.6	15.5	11.2	14.7	13.2	14.2	—
Burning rate slope	0.38	0.46	0.45	0.49	0.41	0.45	—
Density, g/cm ³	1.73	—	—	1.75	1.79	1.76	—
Tensile strength at -55°C, MPa	3.5	4.0	3.9	3.8	3.3	4.0	4.5
Elongation at maximum stress at -55°C, %	14.0	25.5	12.2	14.0	3.3	15.0	18.0
Tensile strength at 74°C, MPa	0.5	0.8	0.6	0.6	0.6	0.5	0.7
Elongation at maximum stress at 74°C, %	12.4	26.4	15.5	24.8	4.6	44.0	47.0

incorporating Tufflo 6016 at higher plasticizer levels is somewhat more flexible than comparably loaded Tufflo 6056 propellant, and as such extrudes into longer strands with less risk of breakage.

Estane 5712 was processed in only one formulation with 20% plasticizer and no further reformulation was performed (Table 2).

Curing Agents

Hycar 4004 binder is hydroxylated polymeric material, and as such is curable using isocyanates. To reduce the moisture sensitivity of the curing reaction, isophorone diisocyanate (IPDI) was selected as the primary curing agent to be evaluated. Generally acceptable results were obtained using this material. Both a difunctional and a trifunctional IPDI were used in the studies, as well as a combination of the two agents. Curative ratios of the difunctional IPDI ranging from 0.6:1 to 0.9:1 (NCO:OH) were evaluated (Table 3).

Generally, propellant mechanical properties approached those of similar high-solids loading cast composite systems, although some anomalies were experienced which require further study. Repetitions of some formulations which had exhibited acceptable stress and strain properties no longer were acceptable (Table 4).

Propellant Mixing

Propellant mixing was performed both during early development and for the large-scale mix in horizontal Baker-Perkins sigma blade mixers which are jacketed for temperature control.

Mixing in the 9.5×10^{-3} -m³ (2.5-gal) mixer or the 1.32-m³ (350-gal) mixer followed the same basic procedures. The order of addition was as follows:

- 1) Weigh out binder and soak overnight in ethyl acetate.
- 2) Transfer swollen binder and solvent to mixer with heating jacket at 60°C (140°F).
- 3) Disperse binder in solvent.
- 4) Add all materials except ammonium perchlorate.
- 5) Mix 15 min.
- 6) Add one-half of ammonium perchlorate.
- 7) Mix 15 min.
- 8) Add remainder of ammonium perchlorate.
- 9) Mix 15 min and inspect.
- 10) Open mixer and sparge with dry nitrogen while mixing to drive off ethyl acetate, approximately 30 min.
- 11) Inspect and check for ethyl acetate.
- 12) Continue sparge and mix for additional 30 min, or until all traces of ethyl acetate are removed.

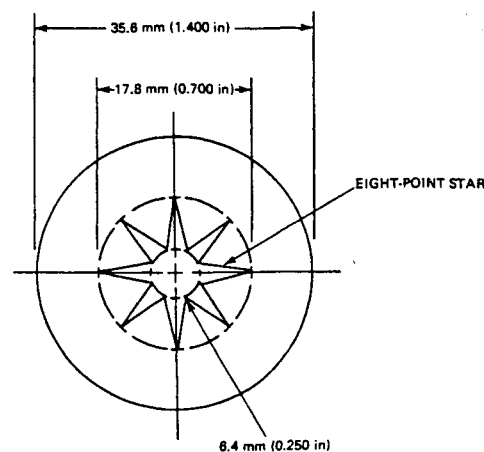


Fig. 1 Eight-pointed star extruded grain configuration.

13) Discharge mixer and protect propellant from atmosphere in conductive plastic bags and transport to blocking press.

Mixes produced a uniform material the consistency of putty, and this material was used without further processing for blocking or extrusion in the smaller presses.

Extrusion Evaluation Conditions

Three extrusion presses were used in these evaluations. The initial tests were performed using a 63.5-mm (2.5-in.) extrusion press fitted with a 9.65-mm (0.38-in.) strand die. Temperatures used were ambient conditions of 24 to 29°C (75 to 85°F) for the Hycar formulation and 104 to 108°C (220 to 227°F) for the Kraton formulations. Minimum pressures for extrusion were 12.4 and 11.6 MPa (1800 and 1690 psi), respectively.

The second extrusion press, 88.9 mm (3½ in.), was used with the 9.65-mm die as well as with two additional die configurations. These die configurations were a right circular cylinder with a cylindrical perforation, having a 40.6-mm (1.6-in.) outside diameter (o.d.) and a 15.2-mm (0.6-in.) inside diameter (i.d.); and a right circular cylinder with a 35.6-mm (1.4-in.) o.d. and an eight-pointed star central perforation of 17.8-mm (0.7-in.) nominal i.d. (Fig. 1). Temperatures used for extrusion were unchanged from those used in the original extrusion evaluations. Extrusion pressures were increased up to 43.4 and 116.7 MPa (6300 and 16,913 psi), respectively, for the two binder systems.

The strands produced were subjected to mechanical properties evaluation and the larger grains were examined for structural integrity. The larger propellant grains were smooth and uniform in appearance. The star-pointed grain was cut to evaluate the internal surface condition. Propellant flow around the extrusion stake was uniform and no tearing or irregular flow patterns were observed.

Based on the success of the foregoing evaluations, a large-scale mix of a Hycar formulation was prepared. The propellant, as discharged from the mixer with no further processing, was blocked into 381-mm (15-in.) diam by 152-mm (6-in.) high cylinders for loading in the extrusion press. The blocking press was evacuated prior to and during the propellant blocking operation. The 381-mm (15-in.) extrusion press was also evacuated prior to advancing the ram for extrusion through the die. The die configuration was a 116.6-mm (4.59-in.) o.d. with an eight-pointed stake with a nominal diameter of 50.8 mm (2.0 in.). The extrusion rate was 902 mm (35.5 in.) per minute.

Extrusion Results and Discussion

The results of extrusion studies of the propellant formulations and the extrusion parameters and die configurations used are described below and are classified by polymeric systems.

Hycar 4004

Propellant formulations ranging from 85 to 90% solids were processed through the extrusion presses in the first three die configurations with propellant pressures ranging from 12.4 to 43.4 MPa (1800 to 6300 psi). All propellant grains appeared structurally sound, and the 9.65-mm (0.38-in.) strands were used for ballistic and mechanical properties testing; in addition the 40.6-mm (1.6-in.) grains were test-fired in a rocket motor in a neutral burning configuration.

Propellant formulations were also held at ambient temperature for up to 96 h before extrusion to determine the working life of the propellants after mixing. These materials extruded well and without significant increase in extrusion pressure. Extrusion press residual material was retained up to 140 hr and Shore hardnesses were taken on the material over that period. No significant increase in hardness was noted, indicating that the formulations were not curing significantly at ambient temperature.

The large-scale mix resulted in the extrusion of approximately 7620 mm (300 in.) of propellant grain from a 136-kg (300-lb) mix of propellant. The grain was very good in appearance and exhibited no slumping during the curing cycle. The extrusion die and stake configurations were reasonably

reproduced by the propellant grain, and the extruded grain was reasonably close to the dimensions of the die and stake values. For this and other formulations of similar characteristics, the extrusion of propellant grains to the desired dimensions appears possible, thus eliminating a requirement for postcure machining of the propellant grain to final dimensions (Table 5).

Kraton G 1652

Propellant formulations using this binder were processed with two plasticizers at several levels. This binder is a nonreactive thermoplastic and thus has infinite working life after mixing. These formulations included 85 to 90% solids loadings. Extrusion pressures ranged from 11.6 to 116.7 MPa (1690 to 16,913 psi). Extrusion die configurations were the same as for the Hycar 4004 except that the 40.6-mm o.d. (1.6-in. o.d.) cylinder was not used.

The 9.65-mm (0.38-in.) strands were used for ballistic and mechanical properties, and the eight-point perforated grain was made for structural evaluation. No motor test firings have been attempted.

Estane 5712

Only one formulation of this propellant has been processed, and that with 16- μ m ammonium perchlorate. Extrusion was accomplished through the 9.65-mm (0.38-in.) die at 79°C (175°F) and 20.7 MPa (3000 psi) pressure, and mechanical properties of the strands were obtained.

Oxidizer Loadings/Burning Rate Evaluations and Discussion

Propellant formulations were devised to obtain the maximum specific impulse attainable using the NASA Lewis Thermochemical Computer Program 5 as a predictive tool (Table 6). The propellants calculated as having maximum specific impulse were generally used for the mixing and extrusion evaluations.

Propellants were processed using up to 15% RDX substituted for ammonium perchlorate. Ammonium perchlorate particle size distributions were varied over a fairly wide range and iron oxide was used as the burning rate catalyst (Tables 7 and 8).

It was found that the usual ammonium perchlorate propellant responses to those variables were produced in the extruded system.

Initial trial mixes of Kraton G 1652 and Estane 5712 as the binder were processed, including 200 μ m ammonium perchlorate in the total oxidizer used. Inspection of these mixes after sparging of the solvent revealed that the 200 μ m ammonium perchlorate was separated from the main mass of propellant in the mixer. This failure to incorporate into the main mass of propellant results in a nonuniform formulation, not to the designed solids loading, and therefore a nonrepresentative product. The necessity of the 200 μ m ammonium perchlorate in these formulations had not been demonstrated, and 100 μ m and finer ammonium perchlorate used in subsequent trials did incorporate into the mixes. The larger particles do not appear

Table 5 Propellant grain dimensions, die diameter 115.6 mm (4.550 in.)

Grain/end	Dimensions at extrusion			
	o.d., mm		i.d., mm	
	0 deg	90 deg	0 deg	60 deg
1 A	117.0	116.5	66.5	67.8
1 B	117.3	116.7	67.5	65.8
1 Center	117.0	116.8	—	—
2 A	117.0	116.2	—	—
2 B	117.1	116.8	66.0	66.7
2 Center	117.0	116.6	—	—
Grain/end	Dimensions after cure			
	o.d., mm		i.d., mm	
	0 deg	90 deg	0 deg	60 deg
1 A	116.7	116.0	64.1	65.8
1 B	116.5	116.5	66.3	65.2
1 Center	116.4	116.6	—	—
2 A	117.0	116.2	—	—
2 B	117.0	116.5	67.0	67.0

Table 6 Solids loading vs theoretical specific impulse

Binder formulations	Characteristic	
	Oxidizer loading, %	Specific impulse, s
Estane	84.0	242.0
Texin	84.0	244.0
Kraton 1	84.0	236.0
Kraton 2	88.5	250.0
Hycar 1	84.0	243.0
Hycar 2	85.5	246.0
Hycar 3	89.3	233.0

Table 7 Effects of oxidizer particle size on burning rate

Characteristic	Hycar 4004 formulations		Kraton G 1652 formulations	
	1	2	1	2
Oxidizer loading, %	84.0	84.0	84.0	84.0
Oxidizer average particle size, μm	200.0	80.0	200.0	90.0
Burning rate at 6.0 MPa, mm/s	7.6	11.2	10.2	15.2
Burning rate slope	0.4	0.5	0.5	0.6

Table 8 Effects of burning rate catalyst on burning rate

Characteristic	Hycar 4004 formulations			Kraton G 1652 formulations	
	1	2	3	1	2
Oxidizer loading, %	84.0	84.0	84.0	88.0	88.0
Oxidizer average particle size, μm	80.0	80.0	80.0	90.0	90.0
Iron oxide, %	0	1.5	2.0	0	1.5
Burning rate at 6.0 MPa, mm/s	11.2	14.7	15.2	15.2	20.3
Burning rate slope	0.5	0.5	0.5	0.6	0.7

to be required for burning rate modification at this time, and no further investigation of the phenomenon was conducted.

The Hycar 4004 and Kraton G 1652 binder systems were evaluated at 85% solids loading, including 15% RDX (4 μm). The RDX was substituted for ammonium perchlorate and the primary effect in these formulations was to reduce the flame temperature and reduction in specific impulse based on NASA Lewis thermodynamic calculation results. The formulations incorporated zirconium carbide and graphite as potential stability additives, although no resonance evaluations were made during this period. The inclusion of such materials at this time allowed the evaluation of processing, burning rate, etc., which could be projected into later formulation evaluations without additions of new materials. The anticipated necessity of stability additives was based on the lack of particulates in the gas stream of reduced smoke propellant compositions.

Iron oxide was included in the formulation matrix to evaluate the response of the formulations to a burning rate catalyst. The response of the formulations was typical of ammonium perchlorate-oxidized propellants and the increases in burning rate were typical of this additive at the indicated levels (Table 8).

Inhibiting

During this study period, only the Hycar 4004 propellant was qualitatively analyzed for inhibition. Two basic approaches were used: overwrapping with ethyl cellulose using ethyl lactate-butyl acetate (ELBA) solvent as a bond promoter, and a painted-on hydroxy-terminated polybutadiene (HTPB)/isocyanate-cured liner system.

The ethyl cellulose/ELBA solvent system produced a marginal bond with a failure mode characterized as propellant failure at the interface; i.e., a blush of propellant was left on the surface of the peeled ethyl cellulose.

The HTPB system adhered very well and peel attempts resulted in failure in the liner coating. This system was used to inhibit certain surfaces of test motor scale propellant grains to ensure neutral burning of the propellant grains. The inhibiting coating held up well during the test firings at -55 and 74°C (-65 and 165°F) as well as at ambient temperature.

Tradeoff Study

Preliminary propellant grain tradeoff studies were performed for both Hycar 4004 and Kraton G 1652 propellant

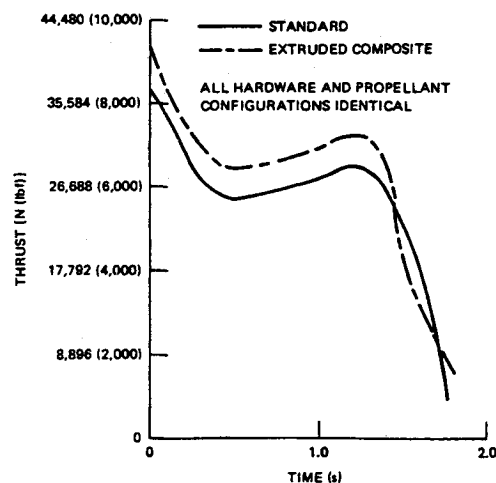


Fig. 2 Tradeoff study for Rocket Motor 1.

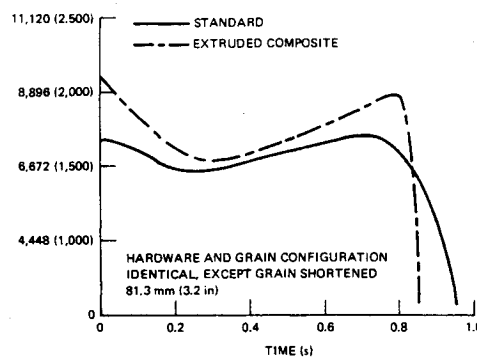


Fig. 3 Tradeoff study for Rocket Motor 2.

formulations for application to two extruded, double-base loaded rocket motors. The rocket motor hardware configuration was unchanged; the propellant grain was assumed to be manufactured by extrusion with the current extrusion dies with no modification. Propellant ballistic and physical properties were limited to the burning rate, etc., which have been demonstrated to date.

Kraton G 1652 propellant could not be made to fit the current performance characteristics of either rocket motor. This failure to duplicate the performance is due to the significantly higher burning rate exponent which has been characteristic of the Kraton formulations thus far prepared.

Hycar 4004 propellant formulations provided a better fit of performance characteristics, although the thrust values are consistently higher for the composite propellant than for the double-base baseline values (Figs. 2 and 3).

For Rocket Motor 1, no changes were evaluated except for reductions in burning rate and use of the lowest values of ballistic parameters supplied. It must be noted that the density of the extruded composite propellant is approximately 7% higher than that of the double-base propellant replaced.

Rocket Motor 2 used the lower ballistic values again, and in addition the propellant grain was shortened 81.3 mm (3.2 in.) in an attempt to reduce the overperformance.

The tradeoff study indicates that if exact duplication of performance of rocket motors were required, the extruded composite would have to be modified to negate some of the advantages gained; e.g., higher density and specific impulse. However, a significant gain in performance could be obtained by the use of extruded composite as a direct substitute for the extruded, double-base grains currently loaded. One additional factor which will affect the actual performance, as opposed to the theoretical, is that the efficiencies of composite propellants

are somewhat less than for double-base propellant. Therefore, only test firings will determine the true comparability of the two propellant types.

Conclusions

The technology development described in this paper has demonstrated that this approach to extruded composite propellant manufacture is realistic and propellants suitable for cartridge loaded rocket motors have been produced. Currently available mixing and extruding equipment may be utilized in the processing of these propellants.

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