The Influence Of Reprocessing On The Characteristics Of EX-101 Propellant

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ABSTRACT

As part of the GEM Program new gun propellants that utilize thermoplastic elastomers (TPE) are under evaluation by NSWC/IH and Thiokol Propulsion. A significant advantage of this class of materials is their ability to be reprocessed and reused. Under the Green Energetic Materials (GEM) Program a study is being performed to carefully evaluate the influence of multiple reprocessing cycles on propellant properties. Data gathered in this study will include: laboratory safety characteristics, propellant mechanical properties, binder filler interaction, polymer molecular weights, closed bomb burning rates, processing characteristics, and ballistic performance in a 40 mm gun. This paper will present the results of this study.

INTRODUCTION

TPE propellants are candidates to replace conventional gun propellant to both improve performance and eliminate the adverse environmental aspects of propellant manufacturing for several different gun systems.[1] TPE propellant can potentially be easily processed without the use of solvent, reprocessed and separated into its ingredients[2]. Under the GEM program a gun propellant designated as EX-101 has been developed and is currently being characterized. This propellant contains ground RDX an energetic thermoplastic elastomer binder utilizing BAMO and AMMO and a small amount of graphite as a processing aid.

A key element in the GEM program is the demonstration of technologies that will reduce the environmental burden associated with the manufacture and disposal of energetic materials. Because of previous work with the TPE binder used in EX-101, it was known that the propellant could be reprocessed. However, the specific effects of reprocessing on physical and chemical properties of EX-101 propellant were not known at the start of this program. To address the potential effect of multiple reprocessing cycles on propellant properties a study was designed and executed. The results of this study are summarized in this paper.

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TECHNICAL DISCUSSION

A flow diagram showing the major elements in this study is shown in Figure 1. As shown in Figure 1, the study began by processing an initial lot of propellant Thiokol's 19 mm twin screw extruder (TSE). A photograph of the extruder is shown in Figure 2.[2] A 7-perf die was selected for use on this effort and was utilized during all propellant extrusion. Following initial extrusion, samples were removed for testing and evaluation. The remaining propellant was then reprocessed and re-extruded. Samples were taken and the process was repeated until the propellant had been processed through the TSE a total of four times.

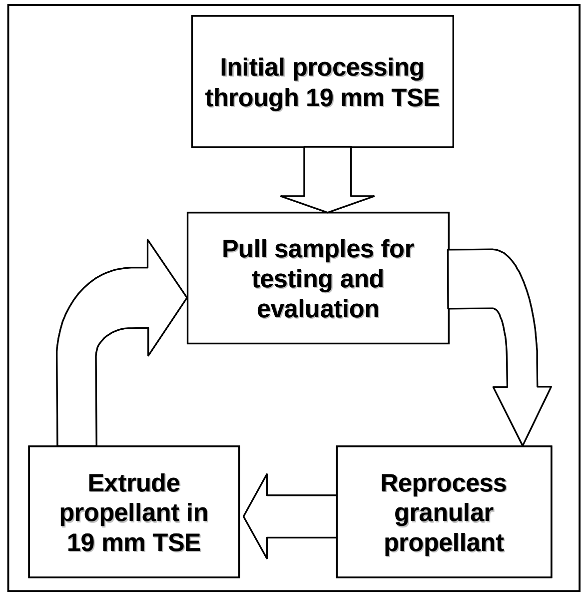


Figure 1. Reprocessing study flow diagram.

Testing performed on propellant from each processing run included: density, laboratory safety testing, compressive mechanical properties, microscopic analysis using scanning electron microscopy (SEM), closed bomb burning rates and TPE molecular weights. In addition to gathering data from these tests, processing information was obtained each time the propellant was mixed and extruded.

Figure 2. Thiokol's 19 mm TSE at building M-241.

Processing Detail

As noted above, all propellant was processed through Thiokol's 19 mm TSE. This extruder has a length to diameter of 25/1, utilizes segmented screws, has vacuum capability, and four independently controlled temperature zones. The die block temperature is controlled independently from the extrusion barrels.

The initial feedstock was prepared by combining ground RDX, graphite and the TPE to form a molding powder. The molding powder was fed into the TSE using a loss-in-weight feeder at a rate of 5 lbs per hour (this rate was maintained during each TSE run). The 7-perf die had a nominal diameter of 0.39-inches and a pin diameter of 0.039-inches. After extrusion, approximately 7-pounds of propellant was cut into granular form with an l/d of 1.0. The remaining propellant was cryogenically ground and reprocessed through the extruder. This process of extrusion and reprocessing was repeated until propellant had been processed through the extruder four times.

Laboratory Data

Laboratory safety tests were performed on propellant after each pass through the TSE. The results of these tests are summarized in Table I. As shown by these data, laboratory safety properties were unaffected by reprocessing the propellant through the 19 mm TSE. The lower TC impact for the first iteration propellant is within experimental variation for this material. It should also be noted that the data presented in Table I indicate that this material has good safety and handling characteristics.

Table I. Results Of Safety Testing On EX-101 Propellant

	First TSE Iteration	Second TSE Iteration	Third TSE Iteration	Fourth TSE Iteration
ABL Impact (cm)	11	11	11	11
TC Impact (in)	34.5	44.5	44.1	40.9
ABL Friction (lb@ 8 ft/s)	800	800	800	800
TC Friction (lb)	>64	>64	>64	>64
TD ESD (J)	>8	>8	>8	>8
SBAT Onset (ºC)	149	153	154	154

The second important series of data generated in this study was propellant density. Density is perhaps the most important factor in determining the overall quality of the composition. Propellant with low density invariably has highly variable surface area due to entrapped air and normally performs poorly when tested in high performance gun systems. As shown in Figure 3 the propellant from each processing iteration had a measured density well above 99% of the calculated theoretical maximum value for this propellant. The values were also very consistent within a run and between runs which indicates the propellant was well mixed and uniform throughout.

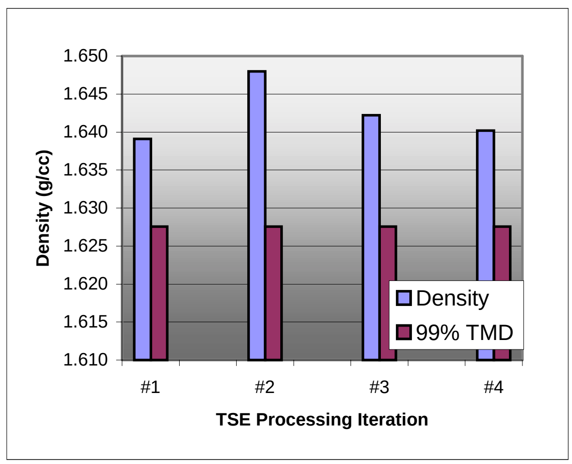


Figure 3. Density Of EX-101 Propellant From TSE Reprocessing Study.

Propellant grains were randomly selected after each iteration and submitted for examination using a scanning electron microscope (SEM). The purpose of the SEM evaluation was to determine if the continued reprocessing of this propellant caused any visually apparent changes in binder filler interaction or porosity. SEM photos were taken at 50X, 200X and 500X of propellant grains on a cut surface (end of the grain) and an extruded surface (side of the grain). Representative SEM photos showing propellant from two different processing cycles are shown in Figures 4 through 9. Examination of these photographs indicated that all propellant samples were well mixed and had relatively few voids or other defects.

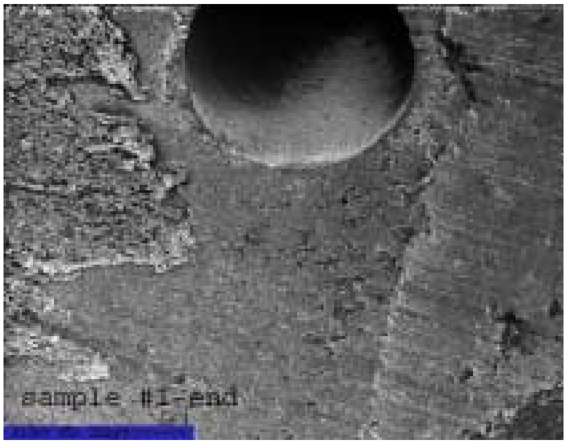


Figure 4. Iteration #1 propellant, end of grain at 50X.

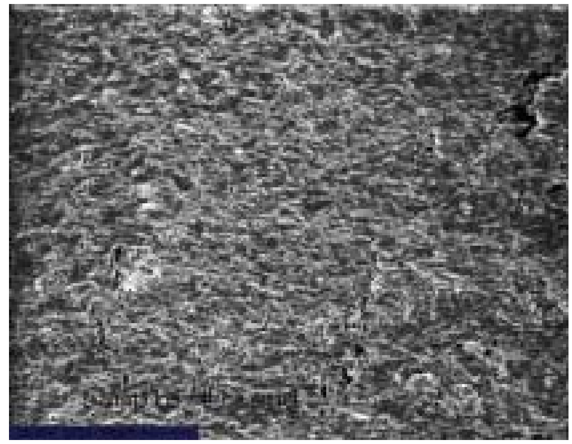


Figure 5. Iteration #1 propellant, end of grain at 200X.

Figure 6. Iteration #1 propellant, end of grain at 50X.

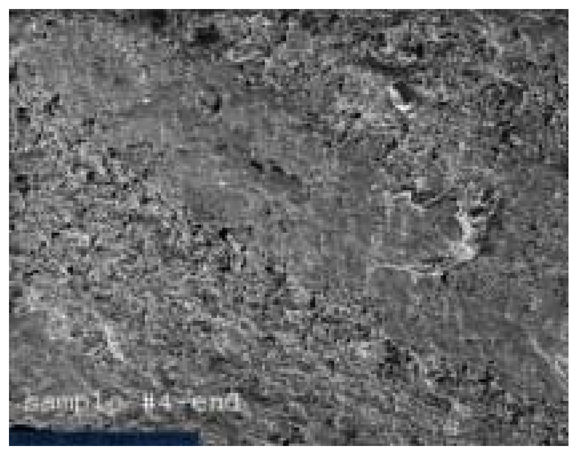
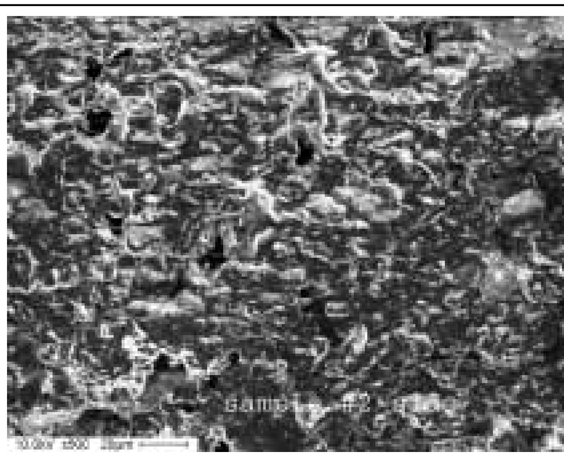


Figure 7. Iteration #4 propellant, end of grain at 200X.



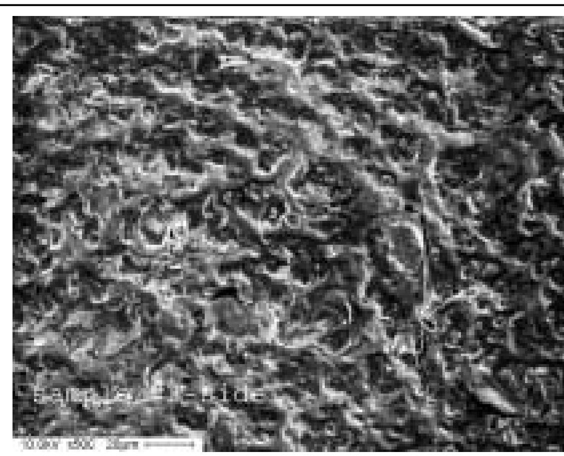


Figure 8. Iteration #2 propellant, side of grain at 500X.

Figure 9. Iteration #3 propellant, side of grain at 500X.

of repeated high shear mixing and extrusion at 85°C followed by cryogenic grinding at temperatures approaching –200ºC would change the TPE used in EX-101. To address this question, the TPE molecular weight was determined using GPC after each pass through the 19 mm TSE. The results of these analyses are shown in Table II along with the initial evaluation of the polymer used in this study before it was combined with RDX and graphite to form EX-101 propellant. It should be noted that the initial GPC testing on the polymer from all propellant samples was performed at the same time while the virgin polymer was analyzed several months earlier. All data shown in Table II are with reference to a polystyrene standard. The actual GPC data for the propellant from the polymer recycling study is shown in Figure 10.

Table II. GPC Analysis Of Polymer In Reprocessing Study.

Lot	Mn	Mw	Mw/Mn
Initial polymer	17,720	83,840	4.73
Iteration one propellant	13,182	89,174	6.43
Iteration two propellant	16,199	109,914	6.79
Iteration three propellant	16,309	113,211	6.94
Iteration four propellant	15,331	109,033	7.11

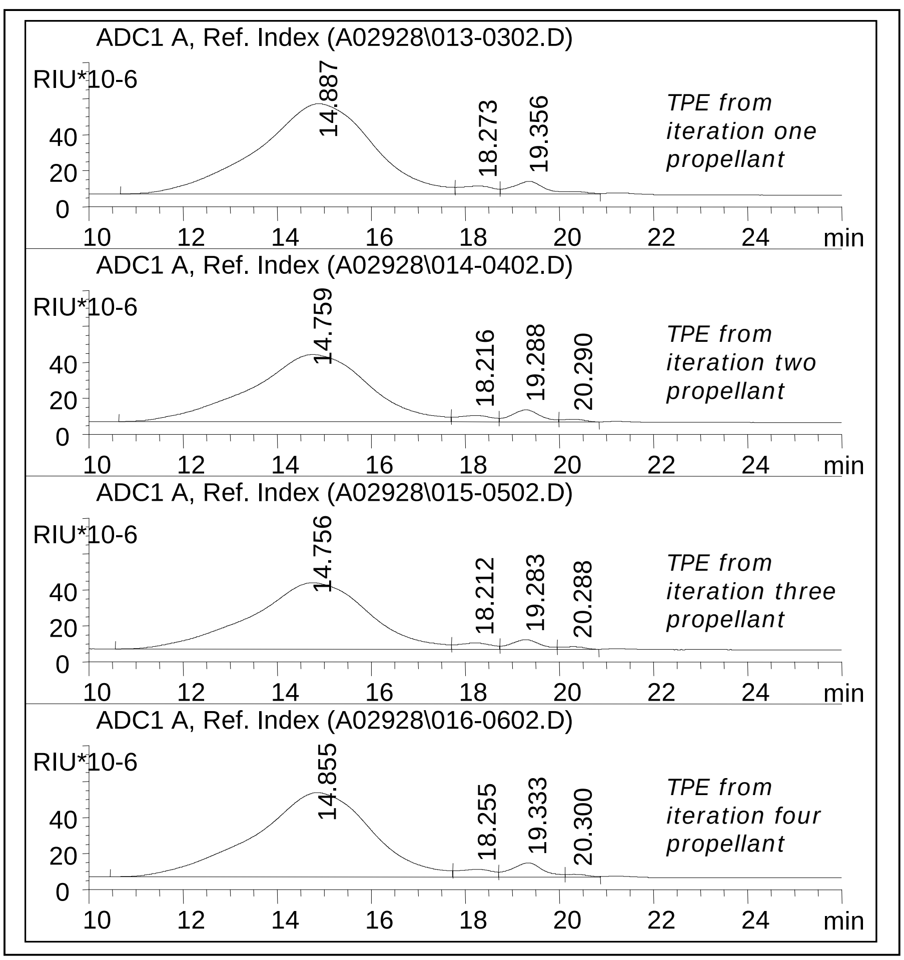


Figure 10. GPC Traces From TPE Used In Recycling Study.

As shown in Figure 10 and Table II, the changes in molecular weight after each processing cycle were not significant. There was no apparent reduction in chain length due to cryogenic fracturing.

In addition to GPC testing propellant grains were taken from each iteration and tested to determine their compressive mechanical properties. The maximum stress and yield stress for each sample are shown in Table III. All samples were compressed to 50% of their initial height at ambient temperature at a rate of 20 inches/minute. Again these data are remarkably consistent and support the overall theme that EX-101 propellant may be reprocessed several times without degradation.

Table III. Selected Mechanical Properties From Reprocessing Study		
Sample	Maximum Stress (ksi)	Yield Stress (ksi)
First iteration	2.24	0.95
Second iteration	2.45	0.97
Third iteration	2.54	1.03
Fourth iteration	2.33	1.02

One of the final sets of data obtained in this study were closed bomb burning rates. Three different tests were conducted on propellant from each processing iteration. All tests were conducted at ambient temperature and used a loading density of approximately 0.3 g/cc. Test results are summarized in Table IV.

Table IV. Closed Bomb Burning Rate From Reprocessing Study			
	Burning Rate (in/sec) @		
Sample	20 ksi	30 ksi	40 ksi
First iteration	3.318	5.153	6.904
Second iteration	3.357	5.173	6.781
Third iteration	3.332	5.127	6.661
Fourth iteration	3.550	5.427	7.003

As shown in Table IV the burning rates for the first three iterations were stable at 20,000 psi and 30,000 psi and showed a very slight downward trend at 40,000 psi. It is possible that additional mixing resulted in slightly better wetting of the RDX crystals which may reduce burning rate. However, propellant from the last iteration has a burning rate which was about 3% higher that the average of the other three runs at 40,000 psi. The reason for this increase is not known. Examination of the pressure time, burning rate versus pressure and vivacity for propellant from the fourth iteration did not shed any further light on this subject. Additional tests using this propellant in a 40 mm gun are planned but have not yet been conducted. It is expected that these tests will give conclusive evidence regarding the burning rate difference noted above.

CONCLUSIONS

Based on the data presented in this document it is concluded that EX-101 propellant may be reprocessed multiple times without significant changes in mechanical properties, TPE molecular weight, laboratory safety data, and binder-filler interaction. A potential change in burning rate observed in propellant from the last iteration should be investigated further. When these data are taken as a whole they strongly support the hypothesis that these TPE propellants are stable and may be reprocessed multiple times without degrading key propellant properties.

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[1] : L. E. Harris, T. Manning, K. Klingaman, P.C. Braithwaite, A. C. Haaland, R. B. Wardle, "Thermoplastic Elastomer Gun Propellant," Proceedings of 1999 NDIA IM/EM Technology Symposium, Tampa, Florida, Nov. – Dec 1999.

[2] : A.C. Haaland, M.K. Vernieuw, V.D. Lott, "Design and Operation of a Small Scale Extrusion Facility For Energetic Material Processing," Proceedings of 1997 NDIA IM/EM Technology Symposium, Tampa, Florida, Oct. 1997.