Environmental Security Technology Certification Program
(ESTCP WP-9804)

Final Report
2.75-Inch Rocket Motor Manufacturing Waste Minimization Project

Indian Head Division, Naval Surface Warfare Center
Alliant Techsystems
US Army Armament Research and Development Engineering Center

19 June 2006

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE (DD-MM-YYYY)
19 June 2006

2. REPORT TYPE
Final Report

3. DATES COVERED (From - To)

4. TITLE AND SUBTITLE
Environmental Security Technology Certification Program (ESTCP)
2.75-Inch Motor Manufacturing Waste Minimization Project

5a. CONTRACT NUMBER

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

5d. PROJECT NUMBER

5e. TASK NUMBER
WP-9804

5f. WORK UNIT NUMBER

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8. PERFORMING ORGANIZATION REPORT NUMBER

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
Environmental Security Technology Certification Program
901 North Stuart Street
Suite 303
Arlington, VA 22203

10. SPONSOR/MONITOR'S ACRONYM(S)
ESTCP

11. SPONSOR/MONITOR'S REPORT NUMBER(S)
WP-9804

12a. DISTRIBUTION/AVAILABILITY STATEMENT
Approved for public release; distribution is unlimited.

13. SUPPLEMENTARY NOTES

14. ABSTRACT

All Department of Defense (DOD) military services use a 2.75-inch rocket system, produced by a conventional batch method that produces large amounts of waste propellant, nitroglycerin, and process water, and are labor intensive. This project sought to demonstrate a lower cost manufacturing process that reduces the amount of waste and pollution generated in the manufacture of the Mk 90 double-base propellant grain used in the Mk 66 2.75-inch rocket system. The new process explored the use of a continuous shear roll mill and twin screw mixer/extruder to reduce the propellant scrap, nitroglycerin emissions, and touch labor while increasing safety by utilizing remote control technology.

15. SUBJECT TERMS
Mk 66
2.75-inch rocket
Double-Base Propellant

16. SECURITY CLASSIFICATION OF:			**17. LIMITATION OF ABSTRACT**	**18. NUMBER OF PAGES**	**19a. NAME OF RESPONSIBLE PERSON**
a. REPORT	**b. ABSTRACT**	**c. THIS PAGE**	SAR	98	Susan Simpson
U	U	U			**19b. TELEPHONE NUMBER (Include area code)** (301) 744-4284

Standard Form 298 (Rev. 8/98)
Prescribed by ANSI Std. Z39-18

TABLE OF CONTENTS

Heading *Page*

LIST OF TABLES

LIST OF FIGURES

LIST OF ACRONYMS

ARDEC	US Army Armament Research and Development Engineering Center
ARL	Army Research Laboratory
ATK	Alliant Techsystems
BAH	Booz-Allen & Hamilton
CAA	Clean Air Act
CLEVER	Closed Loop Energetics with VOC Emission Reduction
CWA	Clean Water Act
DEM/VAL	Demonstration and Validation
DOD	Department of Defense
EMO	Environmental Management Office
EPA	Environmental Protection Agency
EPCRA	Emergency Planning and Community Right-to-Know Act
ESH	Environmental, safety, and health
ESTCP	Environmental Security Technology Certification Program
FEM	Finite element model
HFMI	Highly Filled Materials Institute
HOE	Heat of explosion
ICT	Institute of Chemical Technology
IHDIV	Indian Head Division
IR	Infra-red
IRP	Installation Restoration Plan
LAP	Load, assemble, and pack program
L/D	Length to screw diameter ratio
LEPC	Local Emergency Planning Committee
LIW	Loss-in-weight
LOVA	Low vulnerability ammunition
MACS	Modular Artillery Charge System
NC	Nitrocellulose
NEPA	National Environmental Policy Act
NFPA	National Fire Protection Association
NG	Nitroglycerin
NSWC	Naval Surface Warfare Center
PRB	Process Review Board
RAAP	Radford Army Ammunition Plant
RAB	Restoration Advisory Board
RCRA	Resource Conservation and Recovery Act
RDX	Cyclotrimethylenetrinitramine or 1,3,5-trinitroperhydro-1,3,5-triazine
RMP	Risk Management Plan
RPM	Revolutions per minute
SHA	Systems Hazard Analysis
SIT	Stevens Institute of Technology

LIST OF ACRONYMS—Continued

SMAW	Shoulder-Launched Multipurpose Assault Weapon
SRM	Shear roll mill
TACOM	Tank-Automotive and Armaments Command
TAP	Toxic air pollutants
TIME	Totally Integrated Manufacturing Enterprise
TMD	Theoretical maximum density
TNT	Trinitrotoluene
TOW	Tube-launched, optically tracked, wire-guided missile
TPE	Thermoplastic elastomers
TSE	Twin screw mixer/extruder
VPDES	Virginia Pollution Discharge Elimination System
W&P	Werner & Pfleiderer

Acknowledgements

The Team wishes to acknowledge the support of Mr. Charles Pellerin and Dr. Jeff Marquesee of the ESTCP Office for their guidance during the many project scope and schedule changes during the progress of this project.

Abstract

All Department of Defense (DOD) military services use a 2.75-inch rocket system, produced by a conventional batch method that produces large amounts of waste propellant, nitroglycerin, and process water, and is labor intensive. This project sought to demonstrate a lower cost manufacturing process that reduces the amount of waste and pollution generated in the manufacture of the Mk 90 double-base propellant grain used in the Mk 66 2.75-inch rocket system. The new process explored the use of a continuous shear roll mill and twin screw mixer/extruder to reduce the propellant scrap, nitroglycerin emissions, and touch labor while increasing safety by utilizing remote control technology.

1. Introduction

1.1. Background

The Mk 66 2.75-Inch Rocket Motor is an integral part of US Army and Navy power projection strategies now and in the future. The propulsive energy for the Mk 66 rocket motor is derived from the Mk 90 solid propellant grain. The propellant, designated AA-2, is a double-base type, meaning that its main energetic ingredients are nitroglycerin (NG) and nitrocellulose (NC). The current Mk 90 propellant grain manufacturing process creates several sizeable waste streams to include NG emissions, contaminated waste water, solid propellant waste, and lead-contaminated ash. Several regulatory drivers were the impetus for developing the more environmentally-friendly process that was partially demonstrated during this project. Those regulatory measures include:

- The Pollution Prevention Act of 1990 identifies waste reduction at the source as the highest priority.
- Executive Order 12856 of 1993 requires that federally-owned facilities comply with the Pollution Prevention Act and take action to reduce the amount of toxic materials treated or disposed.
- The Clean Air Amendment of 1990 requires reductions in the discharge of toxic air pollutants (TAP). A major volatile component of the Mk 90 propellant, NG, is a TAP that is emitted from the current process in significant quantities.
- The waste propellant from the process is either burned in the open or in an approved incinerator. The open burning of energetics is coming under increasing scrutiny by regulators and could, eventually, become banned. The incineration process, while meeting Clean Air Act requirements, does produce a heavy metal contaminated solid waste that must be disposed of in a landfill.

The Department of Defense (DOD) has spent millions of dollars developing environmental strategies and installing equipment that safely processes these wastes in compliance with Federal and state regulations. However, the actual amount of waste was not significantly reduced. The DOD is in need of significant grain manufacturing process changes that reduce the amount of waste produced and, if possible, reduce the propellant grain's unit cost simultaneously.

[Content containing proprietary information has been removed and is presented in Appendix A.]

Unfortunately, the team was unable to completely demonstrate the technologies described above, mostly due to unresolved technical problems at the twin screw mixer/extruder (TSE) facility and a significant reduction in the anticipated funding from one of the program sponsors. This report will describe how the objectives listed in the Demonstration and Validation (DEM/VAL) plan were modified and the extent to which the remaining objectives were demonstrated.

1.2. Objectives of the Demonstration

The overall objectives of this project were to demonstrate a pilot-scale Mk 90 grain manufacturing process that, when scaled up, has:

- Lower environmental impact than the current process (waste minimization)
- Lower unit labor costs
- The ability to produce propellant grains that meet every aspect of the Mk 66 rocket motor specification.

As a follow-on, the project team was to develop a transition plan that identified the qualification requirements, costs, schedule, risks, and return-on-investment associated with the transition from the current batch production process to the demonstrated process at the production scale facility in the private sector.

The process demonstrations necessary to achieve these objectives were performed at four different sites. Each site and its role within the demonstration are described as follows.

- NitrochemieAschau GMBH, a German-based energetics processing firm, had an operating shear roll mill (SRM) capability, so they performed the initial processing of AA-2 paste supplied by Alliant Techsystems (ATK). The SRM converts the paste into well-mixed, homogenous pellets that can be fed into the TSE. The work completed here validated the feasibility of using the SRM and provided optimum paste processing parameters.
- The pellets were transported to Indian Head Division, Naval Surface Warfare Center (IHDIV/NSWC), and extruded at the pilot-scale TSE facility. Significant technical resources were applied to safety-related facility/equipment modifications, characterization of the rheological properties of AA-2 propellant, and testing the modified equipment using both inert and live propellant feedstock.
- The ATK manufacturing facility in Radford, VA (Radford Army Ammunition Plant (RAAP)) took the lead in overseeing the pellet production in Germany and installing a new pilot-scale SRM facility at RAAP. They successfully operated the new facility and produced acceptable pellets. It was hoped that once this new SRM was operational, the decrease in NG emissions from the SRM process would be validated during the

production of additional pellets.

- The US Army Armament Research and Development Engineering Center (ARDEC), Picatinny, NJ, conducted an independent review of the environmental and cost analysis, and managed the development of a SRM process model.

A significant portion of the original objectives were met, even in the face of considerable technical challenges and a sizeable reduction in leveraged funding. The following technical advantages were proven during the project:

- The use of the SRM to effectively and safely process AA-2 paste into pellets was proven and documented. This will, if adopted at the production scale, reduce hazardous waste emissions and reduce labor costs significantly.
- The TSE was successfully modified and safely extruded AA-2 propellant, and showed great promise as a grain production technology. The knowledge gained as a result of the rheological studies and die design efforts is extremely valuable to future efforts related to double-base propellant processing. The additional resource investment required to fully validate the TSE would be relatively small compared to the potential labor cost savings and decrease in hazardous waste processing costs.
- Very capable and user-friendly SRM process model software was delivered to the Government. It will be valuable to those who wish to process propellant on the SRM in the future and aid in the scale-up efforts at ATK.

Several other secondary objectives were met and are described in detail in Section 4.

1.3. Regulatory Drivers

The RAAP and IHDIV/NSWC are subject to the provisions of a number of regulatory requirements established by Federal, state, or local authorities. Environmental consequences of Mk 90 production include, but are not limited to:

- The generation, treatment, and disposal of hazardous and otherwise regulated waste streams, including production scrap and lead-contaminated ash from deactivation processes
- The discharge of liquid process wastes, including NG contaminated waters, into waste water treatment facilities
- The release of volatile or hazardous ambient air pollutants, including emissions of NG
- Workplace exposure of personnel to hazardous and potentially hazardous substances, including NG, lead, and adhesives.

The work at IHDIV/NSWC was conducted under existing permits. The proposed introduction of new processes or changes/modifications to existing permitted processes required ATK to apply for permit modifications. This is discussed in detail in Section 6.

The development of this technology addresses a high priority requirement of the Compliance Pillar of the DOD Environmental Security program:

ID#:	3.I.6.c
Title:	Energetics production pollution prevention
Tech Area:	Pollution Prevention
Priority:	High
Media:	Air, water, soil
Containment:	Metals, energetics
Drivers:	Clean Air Act (CAA), Clean Water Act (CWA), Resource Conservation and Recovery Act (RCRA)
Description:	Manufacturing and processing of energetic materials for ordnance items results in large amounts of waste (e.g., extrusion scrap, contaminated salts, redwater) which are not recycled or recovered because of specification, safety, technological, or market constraints. New manufacturing processes are required to reduce hazardous waste and effluent generation, avoiding the necessity for open burning/open detonation, costly commercial disposal of hazardous waste, and expensive end-of-pipe treatment.

1.4. Stakeholder/End-User Issues

The major stakeholder and end-user decision-making factors affected by this demonstration include:

- The successful pilot-scale use of SRM and the potential reduction in labor costs in conjunction with improved environmental performance should be perceived as very promising to the US Army 2.75-inch rocket system program manager. These positive results will influence the decision to move forward with further development of the technology and potentially qualify a production-scale process.

- The TSE also shows great promise and much of the difficult technical work needed to apply the technology effectively has been completed. Although not completely exhibited during this demonstration, the full potential of the technology could be realized if additional funding could be obtained. Again, the benefits would need to be presented to the weapon system program manager so that he/she could decide how to proceed with completing the demonstration.

2. Technology Description

2.1. Technology Development and Application

This demonstration utilized the current AA-2 propellant formulation, the present Mk 90 grain production process and 2.75-inch rocket motor performance as the baseline. The steps in the existing grain manufacture are shown in Figure 2-1. There were two major changes to the baseline process facilitated by the integration of the SRM and TSE technologies. The SRM mixes and creates a colloid of the AA-2 propellant paste, which is then discharged in pellet form. This process replaces the carpet rolling steps in Figure 2-1 (within green dashed line). The pellets are then fed into the TSE where they are extruded into the grains final physical shape. The TSE replaces the batch extrusions steps and the machining steps shown in Figure 2-1 (within the green dashed line). These two major process changes are augmented by smaller process modifications that further minimize the amount of waste. These modifications are as follows:

- Eliminating the sawing operation by cutting the billets to their final length at the TSE
- Eliminating the dowel rod operation by extruding the grains to their final inner and outer diameters
- Modifying end and outer diameter inhibiting as necessary.

The eliminated processes are shown in Figure 2-1 (within green dashed line). The steps in the advanced manufacturing process are shown in Figure 2-2. The key design criteria for SRM and TSE processing are listed in Table 2-1. The criteria are discussed in detail in Section 4.2.

Table 2-1. Key Design Criteria for SRM and TSE

Key Design Criteria	
SRM	***TSE***
Paste Composition	Feed Rate of Pellets
Feed Rate of Paste	Screw rpm
Temperature	Screw Design
Process Length	Process Temperature
Roller Speed	Die Design
Roller Gap	Material Rheology

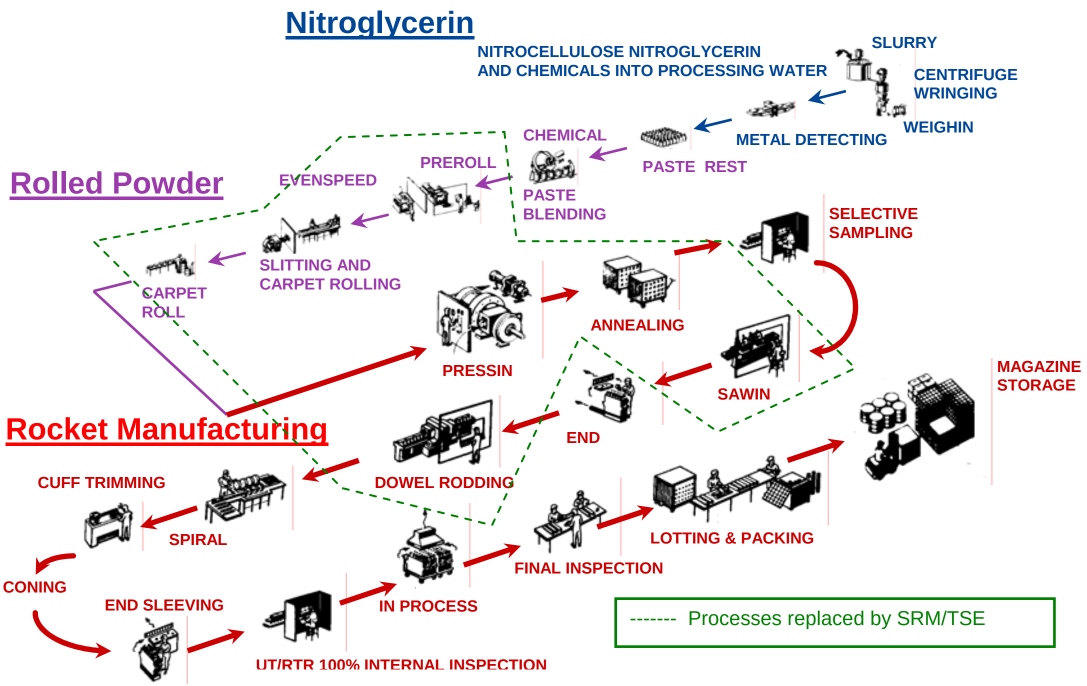


Figure 2-1. Mk 90 Current Batch Production Process

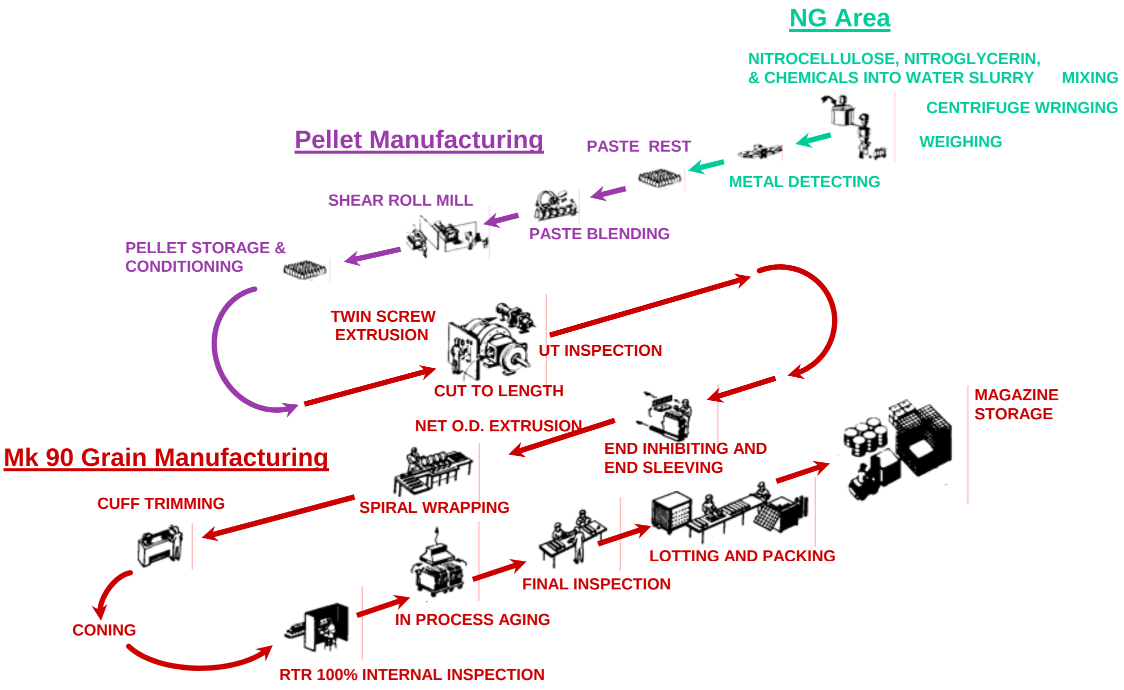


Figure 2-2. Mk 90 Advanced Manufacturing Process

2.1.1. Pellet Manufacturing. The use of continuous roll mills in polymer processing has existed in the rubber and plastics industries for years as a convenient means of creating a granulated, premixed feed for a TSE. In the 1980s, NWC-Nitrochemie of Aschau, Germany undertook a program to devise a method for making solventless gun propellants which would be less labor intensive than the conventional batch rolling and extruding process. Nitrochemie adapted a continuous roll mill, made by ColorMetal for compounding/pelletizing plastics, to the task of dewatering, mixing, colloiding, and pelletizing a water-wet paste of NC, energetic and non-energetic plasticizers, ballistic modifiers, and stabilizers. They found that the pellets produced by this process made an ideal feedstock for a TSE, which was used to form the material by heat and pressure. In 1990, a US Patent was awarded to NWC-Nitrochemie for the combined process of SRM and TSE processing of double-base propellants. ATK (Hercules Inc. at that time) licensed the technology through BOWAS Induplan Chemie in 1992. In general, Nitrochemie found that any nitrocellulose-based propellant composition with a total plasticizer level greater than about 30 percent (by weight) is a candidate for continuous processing on the SRM**.**

2.1.2. SRM Process Description. The SRM is a continuous, open, universal processing machine for the homogenizing, melting, dispersing, compressing, and granulation of materials of medium to high viscosity within a temperature range of 20 to 230 °C. The processing is carried out in the gap between two long, horizontally opposed rolls which rotate in opposite directions. The outside diameters of the rolls never come in contact with each other and the gap between them is adjustable between 0.5 and 5.0 mm. Helical shearing and conveying grooves are machined into the outside diameters of both rolls. Each roll can be heated or cooled throughout its whole length in two separate zones. Powdered material is fed into the processing gap between the rolls by a feeder of the appropriate type for the particular material. By adjusting the temperature and the turning speed of each of the rolls, a layer of product is formed around the front roll. This layer of product can weigh between 0.5 and 5.0 kg, depending on the size of the machine and the gap between the rolls. In a continuous process, the grooves of both rolls shear, disperse, and transport the product from the input end of the machine to the output end, where it is taken off as pellets. The critical elements for maximum dispersion and homogenization of a product are the shearing force, the width of the processing gap, the temperature, and the shearing rate. All of these factors can be adjusted individually. A photograph of the 200 mm SRM that was installed at RAAP in support of this project is shown in Figure 2-3.

Figure 2-3. Shear Roll Mill

2.1.3. TSE Process Description. Twin screw processing has been used in the plastics and food industries for years. The first TSE for use in polymer processing were developed in the late 1930s in Italy. In the 1960s, thrust bearings were developed to increase their reliability. Twin screw compounding and extrusion have been used by several European countries since the early 1970s to manufacture energetics—primarily single-base, double-base, and triple-base gun propellants. There was a surge of interest in the application of twin screw technology in the United States in the 1980s. During the same period, the Europeans began investigating the application of twin screw technology to different types of energetic materials such as plastic-bonded explosives and composite propellants. IHDIV/NSWC began researching this technology in the early 1980s. Within the last 15 years, there have been remarkable improvements in the technology areas of machine design, controls, feeders, and computational models. These improvements have matured the development of the continuous process and expanded the scientific understanding of the operation of the TSE.

2.1.3.1. Grain Manufacturing Process. The description below identifies how the propellant grain is manufactured. The TSE process includes material feeding, mixing/extrusion, deaerating, and cutting. These operations are done within one process, which results in a lower manufacturing cost when compared to the batch process. The entire TSE process at the IHDIV/NSWC facility is controlled remotely from a control room located approximately 300 feet from the processing building.

2.1.3.2. Material Feeding. The characteristics of the feed material are critical; the flow of material must be constant so the extruder does not experience interruptions. For some formulations, several loss-in-weight solid feeders are required to deliver the dry solid ingredients and several pump systems are required to meter the liquid ingredients to the TSE. For this demonstration, only one solid feed stream was needed at steady state. A new vibratory tray feeder was procured and utilized to deliver this feed stream. The existing four feeders were not appropriate for this application.

The IHDIV/NSWC TSE facility is a process development facility built around a continuous process. Unlike a production size facility, this facility has a limited solid refill capability. The total amount of material that can be fed to the extruder determines the length of each processing run. The IHDIV/NSWC facility has one refill hopper for each of the four loss-in-weight solid feeders. Each refill hopper consists of four refill cylinders. Three of the four refill hoppers are shown in Figure 2-4.

Figure 2-4. Solid Refill Hoppers

These refill hoppers allow for longer TSE runs; thus, a longer continuous manufacturing process can be demonstrated. When refill of the solid feeder is required, the contents of one refill cylinder are emptied into the feeder in a very short time period. Therefore, the operation of the solid feeder is not disturbed. This procedure is repeated for the other three refill cylinders as required.

2.1.3.3. Twin Screw Extrusion. The TSE facility is centered around a Werner & Pfleiderer (W&P) ZSK-40 co-rotating twin screw extruder with cantilevered screw shafts. The extruder is powered by a 20 hp variable speed explosion-proof motor. A safety slip clutch is provided, which will disengage the motor from the extruder screws in the event of an over torque situation. This machine utilizes two 40 mm diameter screws (Figure 2-5) centered in the extruder barrels.

Figure 2-5. W&P 40-mm Extruder Segmented Screws

The screws are co-rotating, fully intermeshing, and self-wiping. The screw profiles are designed so that the tip of one screw wipes the flank and root of the other screw, resulting in a self-cleaning action. This type of twin screw mechanism provides very good conveying, pressure build up, and self-cleaning capabilities. Segmented screw sections are used to maintain flexibility in screw configurations. The screws can be made up of various different screw elements that slide onto a splined shaft. The various types of screw elements are right-handed conveying elements, left-handed mixing elements, and kneading blocks. All of these elements can vary in length and pitch. Each type of screw element provides distinct conveying, shearing, and pressure-building action. Thus, the various types of screw elements can be arranged on the screw shafts as needed to provide the required conveying or mixing actions at desired locations. The screw configuration used depends on the specific energetic formulation. The screw configuration used in this demonstration was designed in accordance with established protocols, taking advantage of existing expertise in Germany and elsewhere.

The TSE facility was designed to have the capability of easily changing from one energetic formulation to another. Therefore, an extruder with a modular barrel design is used (Figure 2-6). This extruder has six segmented barrel sections and three liquid injection plates, which can be arranged to serve different ingredient addition locations and operating requirements. Each individual barrel section is 165 mm in length; the total processing section of the extruder has a barrel length to screw diameter ratio (L/D) of approximately 28. The extruder is equipped with five separate temperature control zones for maintaining process temperature. The extrusion pressure and temperature is measured by a pressure transducer/thermocouple located at the entrance region to the die. Pressures and temperatures along the length of the extruder are also measured at pertinent locations using transducers/thermocouple assemblies.

The ingredients are mixed, consolidated, and pressurized for extrusion in the processing section of the extruder. The product is extruded through a die mounted in a die holder at the end of the extruder. The (interchangeable) single die is designed for a particular product and grain configuration. Extrusion of a 2.75-inch extruded composite propellant grain is shown in Figure 2-7. These grains were cut using a flying cutter as were some of the Mk 90 grains made during this demonstration. A hydraulic clamping mechanism retains the die holder during operation. The die holder is equipped with a shearing mechanism that will release the die at 3000 psi during an over pressure event.

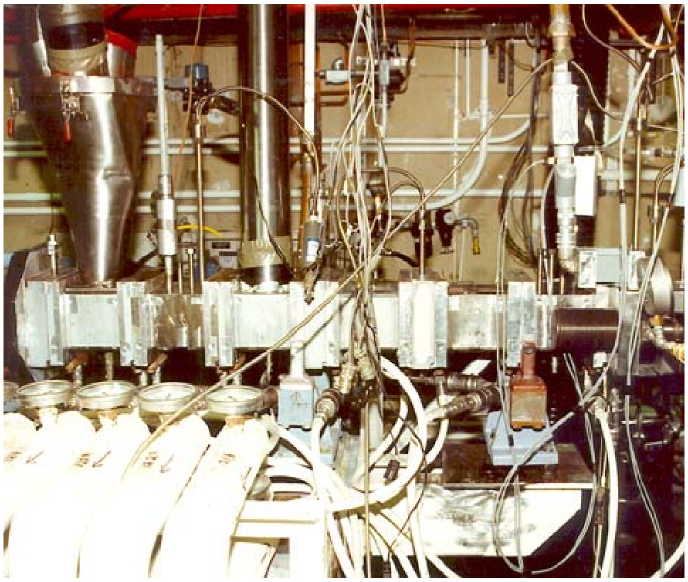


Figure 2-6. W&P 40-mm Extruder Barrel Sections (Side View)

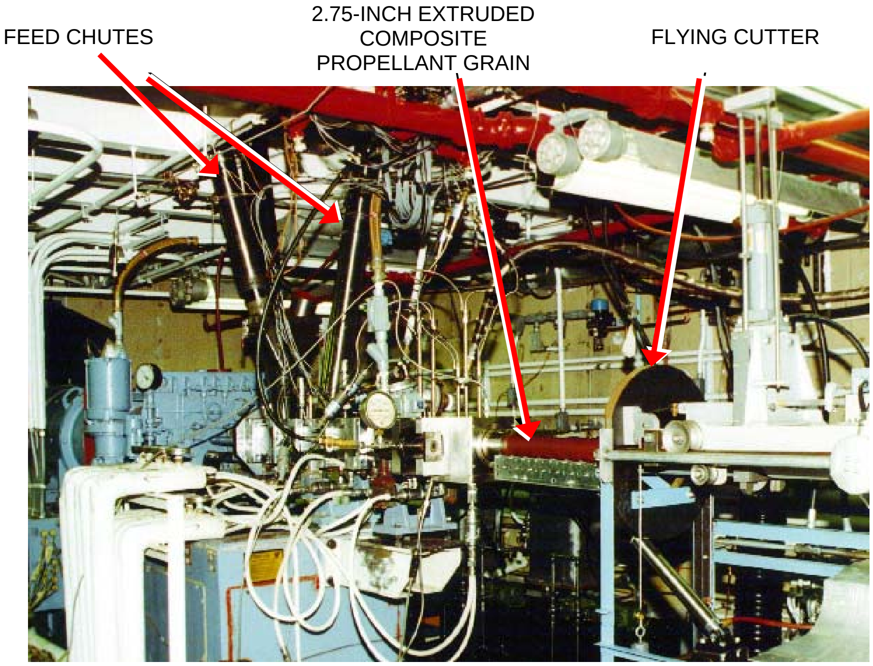


Figure 2-7. W&P 40-mm Extruder and Flying Cutter

2.1.3.4. Propellant Cutting. The flying guillotine cutter mentioned above was used to perform all of the cutting operations during the demonstration. There were plans to use a computer-controlled, water-jet cutter but this technology was not utilized. There were insufficient grains produced to justify the startup of this rather complex equipment.

2.1.3.5. TSE Process Control. All of the above TSE operations are controlled and monitored remotely from a control room. An Allen Bradley programmable logic controller is used to continuously control and monitor the process conditions, as well as to control the various alarms and interlocks of the system. The alarms and interlocks include features such as shutdown of the extruder in event of an over torque or over pressure event. A PC-based data acquisition system is used to log the process data at a rate of once per second. The system features trend screens, status screens, history recall, instrumentation displays, and subsystem overview screens. This system is ideal for monitoring feeder stability, process instrumentation, and extruder performance.

2.1.4. Motor Firing Operations. No static firing operations were necessary at the static firing facility at IHDIV/NSWC because no acceptable grains were produced. Propellant grains produced using carpet rolled sheetstock made from SRM-produced pellet were fired at RAAP.

2.1.5. Totally Integrated Manufacturing Enterprise (TIME). This portion of the project was removed from the task list due to a reduction in funding from the 2.75-inch rocket motor program manager. No data were collected concerning the use of this capability.

2.2. Previous Testing of the Technology

Continuous processing of single base and double base materials has been conducted on a production scale in a number of European facilities for many years. The French have produced a solventless double-base propellant grain for their version of the 2.75-inch rocket continuously and report a labor savings of 25 percent over their batch process. Additionally, the Germans have many years of experience processing double-base materials on the continuous processor. In both instances, continuous processing produced a high quality product very affordably.

In 1991, ATK (then Hercules, Inc.) at their Kenvil, NJ facility purchased manufacturing technology and equipment for solventless propellant production from BOWAS Induplan Chemie, of Salzberg, Austria. The equipment purchases included a 200 mm SRM and a 96 mm high-pressure TSE, manufactured by Berstorff. The 200 mm SRM is considered to be development scale. It had a demonstrated capacity of up to 125 lb per hour (dry weight) when processing two other double-base formulations, JA-2 and DIGL-RP. The 300-mm production-scale unit has nearly twice this capacity.

The 200-mm SRM was installed and operated successfully with both simulated double-base and live propellant. Approximately 200,000 lb of DIGL-RP (a double-base tank propellant formula) were successfully processed on the SRM. Small amounts (several thousand pounds each) of other formulations were also processed during developmental testing. As for the TSE, prior to the installation of the production equipment at Kenvil, extrusion trials were conducted on a 58-mm W&P extruder. These trials showed that JA-2, for the Army 120-mm M829A1 round, and DIGL-RP, for the Army 120-mm M830/831 rounds, could be extruded from a TSE.

The propellants were indistinguishable in terms of physical properties and burning rate from those produced from the current batch process. One observation made by investigators during examination of JA-2 propellant at the Army Research Laboratory (ARL) was that the product from the TSE was "totally void free," a highly desirable gun propellant characteristic.

The installation of the 96-mm TSE at Kenvil was completed in October 1993. Approximately 40,000 lb of DIGL-RP propellant was produced at throughput rates of up to 300 lb per hour. An explosive incident occurred on 1 November 1993 that ejected the die block, causing minor damage to the extruder and significant damage to the building. The root cause was traced to metal-to-metal contact between the screw and the barrel of the extruder during a feed upset. The extruder and its building were not rebuilt because of a prior re-direction of tank propellant production back to RAAP. Both the SRM and TSE were moved to RAAP when the Kenvil plant was closed in 1996. It was this SRM that was installed and used during this demonstration.

Extensive gun and rocket propellant experience, as well as an excellent scientific knowledge base, exists at IHDIV/NSWC. The history of the twin screw processing efforts at IHDIV/NSWC follows:

1982–1984 *Technology Evaluation:* Research began on the technology. The initial application was the processing of low vulnerability ammunition (LOVA) nitramine gun propellant. Facilities were visited to determine what equipment current users were utilizing to process energetic materials. Universities and private companies were funded to perform inert studies with continuous processing equipment.

1985–1987 *Pilot Plant Definition:* Although LOVA nitramine gun propellant was the initial product, the facility was designed to be flexible so that a wide variety of energetic materials could be processed in that facility. With this flexibility in mind, a 40-mm W&P was procured and a facility chosen for the installation of the extruder and associated equipment.

1987–1988 *Process Development Facility Construction:* The initial facility construction and equipment installation was completed in 1988 at a cost of approximately $1 million.

1988–1989 *Inert Studies (LOVA):* The objective of the inert work with LOVA was twofold. First, facility start-up and initial familiarization had to occur with inert material. The second objective was to develop operating parameters for the live LOVA propellant processing. A total of 32 processing trials were performed, resulting in the manufacture of over 900 lb of inert propellant. An extensive documentation package was prepared to obtain approval for live operations.

1990–1993 *Live Operations (LOVA):* A similar continuous processor was built at NSWC-White Oak during the same period. The continuous processor at White Oak was the first facility in the US to process live material. Live processing at the IHDIV/NSWC facility followed approximately six months later in May 1990. Both facilities were started up with LOVA nitramine gun propellant. Since then, 40 processing trials at IHDIV/NSWC have yielded over 500 lb of live LOVA. These trials used a LOVA preblend manufactured using a standard vertical mixer. Additionally, a rheological study of a high energy LOVA propellant as processed on the TSE was conducted with the use of an on-line, adjustable gap rheometer. This work was performed in collaboration with the Highly Filled Materials Institute (HFMI) at Stevens Institute of Technology (SIT) and NSWC-White Oak prior to the merger of the White Oak and IHDIV divisions.

1993–1994 *Facility Upgrade:* The initial facility design had a limited feed capacity. There were only two loss-in-weight solid feeders and there were no solid refill hoppers. Additionally, the facility lacked humidity control and other controls were minimal. The facility upgrade removed these limitations. The facility was upgraded specifically to provide the capabilities required to execute the Continuous Processing of Composite Propellants project. These capabilities included additional loss-in-weight solid feeders, automatic solid refill, humidity control, improved extruder barrel temperature control, and data acquisition/process control. This facility upgrade incorporated many of the lessons learned from the LOVA processing work and from the TSE-user community to create a very flexible, modular, and capable research and development facility.

1994–1997 *Continuous Processing of Composite Propellants:* IHDIV/NSWC entered into a cooperative agreement with the French to develop continuous processing for the manufacture of composite propellants. This project combined the research and development resources of both countries to develop this technology faster than either country could achieve independently. A secondary IHDIV/NSWC objective was the development of TSE processing science. During this time, a 2.75-inch extruded composite rocket motor grain was successfully extruded through a die designed with the aid of computational fluid dynamic modeling.

1995–1996	*LOVA Demonstration Lot:* The objective was to test fire, in a gun, a demonstration lot of M43 nitramine gun propellant manufactured in the TSE and compare the test results to that obtained from the batch-processed propellant. The TSE feed material was prepared using a precipitation process that required ground RDX as the starting material. The propellant grains were successfully extruded and test fired.
1996–1997	*Inert TPE Processing:* Thermoplastic elastomers (TPE) are ideal binders for “green energetics” because they do not require cross-linking and, therefore, can be recycled more easily. TPEs are very difficult, if not impossible, to process in standard large batch mixers. However, TPEs can be processed readily in twin-screw extruders. The objective of this project was to develop the process for manufacturing a TPE-based gun propellant.
1997 - 2000	*CLEVER:* The Closed Loop Energetics with VOC Emission Reduction (CLEVER) program incorporated the Bofors Precipitation Process with TSE. The first objective of this program was to verify solvent emission reductions when replacing the conventional solvent processing of nitramine gun propellants with the CLEVER process. The second objective was to demonstrate that a new, cost-efficient, and environmentally-friendly process can produce acceptable gun propellant.

2.3. Factors Affecting Cost and Performance

2.3.1. SRM Process. The pellets will be made from batch-produced, water-wet AA-2 paste. The pellets produced by the SRM process will have a composition nearly identical to that of carpet-rolled AA-2 paste. These composition requirements are fairly broad, in recognition of the need to make small changes during production so that the performance requirements of the 2.75-inch Mk 90 propellant grain can be adjusted as needed. As an example, slight variations are made in ballistic modifier concentration to accommodate lot-to-lot variation in modifier effectiveness. Because the ballistic modifier is essentially inert, any variation, intentional or otherwise, in its concentration has a significant effect on energy as measured by the heat of explosion (HOE).

A partial listing of factors known or expected to affect SRM pellet processing and ultimate suitability of pellets for use in the TSE is shown below.

- *Composition of paste*—Affects energy content, burning rate, and effectiveness of selected SRM operating parameters.

- *Moisture content of paste*—Affects effectiveness of selected SRM operating parameters and moisture content of pellets.

- *Homogeneity of paste*—Affects feed rate control of paste and uniformity of pellet composition.

- *Distribution of LC-12-15 ballistic modifier*—Distribution of modifier has a likely effect on propellant burning rate.

- *Nitrocellulose properties*—Affects energy content, rate of colloiding, and physical properties.
- *Feed rate of paste to SRM*—Affects effectiveness of selected SRM operating parameters and moisture content of pellets. Must be experimentally optimized.

- *Temperature of processing zones on SRM rollers*—Affects effectiveness of selected SRM operating parameters, specific work input, and moisture content of pellets. Must be experimentally optimized.

- *Processing length of SRM*—Affects effectiveness of selected SRM operating parameters, specific work input, and moisture content of pellets. Must be experimentally optimized.

- *Roller RPM*—Affects effectiveness of selected SRM operating parameters, specific work input, and moisture content of pellets. Must be experimentally optimized.

- *RPM difference between SRM rollers*—Affects effectiveness of selected SRM operating parameters, specific work input, and moisture content of pellets. Must be experimentally optimized.

- *Gap between rollers*—Affects effectiveness of selected SRM operating parameters, specific work input, moisture content of pellets, and pellet dimensions. Must be experimentally optimized.

- *Final moisture content of discharged pellet*—Indicative of specific work input on SRM, used for process control of feed rates (i.e., too low a moisture content—danger of fire, too high a moisture content—insufficient work input). Has a significant effect on processing in TSE. Must be experimentally optimized.

- *Final absolute density of pellet*—Indicative of specific work input and degree of colloiding obtained on SRM; near 100 percent theoretical maximum density (TMD) desired. Low densities may lead to low densities of TSE-extruded grain and improper ballistic performance.

- *Bulk density of pellet*—Function of absolute density and geometry; affects loss-in-weight (LIW) feeder selection and maximum throughput of TSE at a particular RPM/screw fill.

The ARDEC SRM modeling effort has resulted in an improved understanding of how the

SRM operates. The SRM model will reduce the start-up time/risk required to process new propellant formulations on the 200-mm pilot plant size SRM at RAAP and reduce scale-up risk/time for transitioning propellant formulations from the 200-mm to the 300-mm production scale SRM. The model is based on empirical data; it provides the operator the knowledge to make real-time adjustments to operating parameters. These adjustments ensure that the optimum propellant properties are realized.

2.3.2. TSE Process. Manufacture of the final propellant by the TSE process has many variables as well. These include: pellet delivery rate (lb/hr), extruder screw configuration, barrel configuration, screw speed (rpm), process temperature (can have multiple zones), extrusion die temperature, extrusion pressure (process measurement), die design, and torque (process measurement).

The AA-2 feedstock delivery rate is a key area. The delivery rate challenge is simplified through the pellet manufacture process. The pellets are free flowing and easily fed in a new vibratory tray feeder. It is anticipated that there will only be one feed stream at steady state, which helps to simplify the process design. However, if the moisture content of the pellets is low, additional water may have to be added. This could be easily achieved by including an injection port in the modular barrel configuration.

The screw configuration, product throughput, screw speed, and process temperature are operating parameters that have to be evaluated. If the TSE is operated in a “starved” configuration (screws not filled), the pellet feed rate controls the rate of product output. The rate of product output can affect product quality, for example surface finish and die swell. When the TSE is operated in a “flooded” condition, the screw speed controls the rate of product output. Under both conditions, the shear rate imparted to the material is dependent on screw speed, which affects product quality. The screw design depends on the amount of mixing, conveying, and pressurization needed to manufacture a quality product. For this application, the design is simplified by the fact that the pellets are fully colloided prior to being introduced to the extruder. Thus, the screw consisted primarily of conveying elements with some kneading elements. The kneading elements were used to create a fluid bearing to help center the screws and prevent screw-to-screw and screw-to-barrel contact. This screw design and the relatively slow screw speed meant that there was no need to apply vacuum to the barrels. The kneading block formed a barrier which allowed air to escape through the feed port. Their pitch and length were experimentally optimized. The die is the most probable place for material stagnation and cook-off, so particular attention was paid to the die design, as well as the flow characteristics of the material through the die. Characterization of the material rheology, as a function of temperature, was critical. The work imparted by the extrusion process and the heated TSE segments can alter the material’s temperature greatly and the resulting rheological effects must be well understood. A detailed description of the testing and analysis that determined the initial TSE operating parameters is provided in Section 3.6.

2.3.3. Costs. The key to controlling and minimizing the operating costs associated with both the

SRM and the TSE is a complete understanding of the numerous process variables listed above. If each variable's effect is clearly understood then the equipment can be started up quickly, brought to steady-state with minimal waste, and produce very few reject pellet batches or propellant grains. This keeps costs low by conserving the expensive raw materials, reducing labor costs per grain, and ensuring that waste disposal costs are minimized. For the SRM, this is an area where the process model can be extremely helpful if it can allow technicians and engineers the opportunity to investigate changes in process variables without having to process propellant. Any reduction in the time it takes to reach optimal operating conditions is valuable and the effort to achieve the reduction deserves consideration and investment. Similar efforts to model the TSE die flow are equally valuable.

2.4. Advantages and Limitations of the Technology

[Content containing proprietary information has been removed and is presented in Appendix A.]

3. Demonstration Design

3.1. Performance Objectives

The performance objectives for the project are provided in Table 3-1. Those objectives that were fully validated are green. Those objectives that were partially validated are yellow and those that were not validated at all are red.

Table 3-1. Performance Objectives

Type of Performance Objective	Primary Performance Criteria	Expected Performance (Metric)	Actual Performance (Objective Met?)
Quantitative	1. Reduce the per unit discharge of NG vapor emissions	NG emissions reduced by 14.3 percent	Partially met, independent material balance completed, no actual process validations completed
	2. Reduce the per unit discharge of contaminated waste water	Contaminated waste water discharge reduced by 63 percent	Partially met, independent material balance completed, no actual process validations completed
	3. Reduce the per unit discharge of propellant waste	Propellant waste discharge reduced by 52.8 percent	Partially met, independent material balance completed, no actual process validations completed
	4. Reduce the per unit weight of created lead-contaminated ash	Lead-contaminated ash reduced by 23.1 percent	Partially met, independent material balance completed, no actual process validations completed
	5. Reduce the unit direct labor cost	Direct labor costs reduced by 28 percent	Partially met, independent cost analysis completed, no actual validations completed
Qualitative	1. Produce Mk 90 propellant grains that meet DOD physical, chemical, and performance specifications	All specification requirements met	Partially met, grains manufactured from SRM-produced pellets but extruded through legacy ram presses met specification requirements, no acceptable grains produced using TSE
	2. Establish pilot-scale SRM facility and produce acceptable AA-2 propellant pellets	Facility operating and pellets characterized as acceptable	Met, facility operating at ATK site, produced acceptable pellets
	3. Reconfigure and operate existing TSE facility, produce acceptable Mk 90	Facility operating and acceptable Mk 90 grains	Partially met, facility reconfigured and

Type of Performance Objective	Primary Performance Criteria	Expected Performance (Metric)	Actual Performance (Objective Met?)
	grains from AA-2 pellets	produced	operated, significant progress made in propellant flow characterization and die design, no acceptable grains produced
	4. Develop a mathematical model of the SRM process	Model developed that can be used to predict process behavior when key variables are adjusted	Model completed, delivered to and accepted by the Government, meets objective fully
	5. Develop transition plan for the advanced MK 90 grain manufacturing process	Transition plan completed and approved by all team members	No formal transition plan submitted, ATK has internal plans in place for production-scale facility

3.2. Selecting Test Platforms/Facilities

The project team members are IHDIV/NSWC, ATK, RAAP, and ARDEC, Picatinny Arsenal. These sites were selected based on the extensive technical expertise and robust equipment/facilities existing at each site. ATK investigated the utility of using the SRM to produce pellets from their standard production AA-2 paste and installed a pilot-scale SRM facility in one of their existing buildings. Using a development scale TSE, IHDIV/NSWC had produced extruded composite propellant grains for the 2.75-inch rocket and successfully static fired them. The IHDIV/NSWC utilized this TSE expertise to make needed modifications to the TSE facility, optimize the operating parameters, and extrude AA-2 pellets. TACOM-ARDEC has also invested in continuous processing technology development. ARDEC funded and managed the development of the SRM process model. Their resident engineering and modeling expertise, when teamed with ATK processing expertise, was uniquely qualified for this effort.

3.3. Test Platform/Facility Characteristics/History

3.3.1. ATK. RAAP has been located near Radford, VA since 1940, when Hercules signed a contract with the US Government to build and operate Radford Ordnance Works and the New River Plant. After the Korean Conflict, the Nitroglycerin and Rocket Area were constructed. In 1968, a continuous TNT nitration plant was constructed. In 1995, ATK became the operating contractor of RAAP. Today, RAAP consists of two primary operating groups: Radford Army Ammunition Plant (RAAP) and New River Energetics. The RAAP facility produces a variety of propellant types, including those used in medium caliber ammunition, tank rounds (tactical and training), rocket motors (TOW Launch, Hydra 70, and SMAW), and specialty applications. New River Energetics, which occupies a portion of the plant, specializes in small caliber shot gun, rifle, rimfire, and centerfire gun powders for the commercial and military markets. Recently, ATK had chosen RAAP as the preferred location of its load, assemble, and pack (LAP) program for medium-caliber ammunition rounds and Modular Artillery Charge System (MACS). The two locations consist of 800 buildings on 6,901 acres and approximately 1,400 employees.

3.3.2. IHDIV/NSWC Facility. IHDIV/NSWC is the oldest, continuously operating Naval ordnance facility in the US. IHDIV/NSWC was established in 1890 as the Naval Proving Ground. It became the Naval Powder Factory in 1932, the Naval Propellant Plant in 1958, the Naval Ordnance Station in 1966, and the Indian Head Division, Naval Surface Warfare Center in 1992. IHDIV/NSWC is located on a peninsula bordered by the Mattawoman Creek and the Potomac River in Charles County, Maryland (Figure 3-1). The activity consists of 1,600 buildings on 3,500 acres and approximately 2,000 employees. The total plant asset value is $1.5 billion with over $50 million invested in the last five years in environmental efforts.

IHDIV/NSWC carries out a full spectrum of functions for energetics research, development, manufacturing, and in-service engineering. This activity possesses the unique capability to transition all energetics from laboratory to production, and on to service. Energetics is a term which applies to explosives, propellants, pyrotechnics, and specialty chemicals. This includes their immediately related component applications; for example rocket and missile propulsion units, warheads, mines, gun projectiles and propelling charges, and cartridge-actuated and propellant-actuated devices. The scope of capabilities at IHDIV/NSWC allows for efficient use of the specialized expertise and facilities required for research and development, scale-up, manufacture, and testing of energetics.

Figure 3-1. IHDIV/NSWC

3.4. Present Operations

The operations that this advanced Mk 90 grain manufacturing process is intended to replace are depicted in Figure 2-1 (within green dashed line). The waste streams created by this legacy process are shown in the mass balance diagrams provided as Figures 3-2 and 3-3. The key differences are the following:

- SRM replaces the pre-rolling, even-speed rolling and carpet rolling step with a single rolling operation that produces pelletized AA-2 propellant vice the rolled sheet propellant form (sheetstock).

- TSE replaces the ram extrusion process and further improves upon it by extruding to a more exact outer diameter, which eliminates the need for the legacy dowel rod operation.

- Water-jet cutting the grains immediately after extrusion and doing so accurately eliminates the need for the legacy saw-to-length operation.

- TSE extrusion of pellets does not leave the same internal stresses in the extruded grain as compared to conventional ram extrusion, this means that the annealing step can be eliminated.

In summary, the legacy grain manufacturing process will be radically modified due to the elimination of five keys steps. The advanced process is depicted in Figure 2-2 and its associated mass balance diagrams are presented in Figures 3-4 and 3-5. The waste streams associated with each of the eliminated steps is significant and their elimination contributes to the project's overall waste minimization objective. Their elimination also makes possible the reductions in direct labor costs outlined in Section 1.2.

[Figure 3-2 containing proprietary information has been removed and is presented in Appendix A.]

Figure 3-2. Mass Balance of Carpet Roll Production

[Figure 3-3 containing proprietary information has been removed and is presented in Appendix A.]

Figure 3-3. Mass Balance of Batch Grain Production

[Figure 3-4 containing proprietary information has been removed and is presented in Appendix A.]

Figure 3-4. Mass Balance of SRM Pellet Production

[Figure 3-5 containing proprietary information has been removed and is presented in Appendix A.]

Figure 3-5. Mass Balance of TSE Grain Production

3.5. Pre-Demonstration Testing and Analysis

The engineering consulting firm Booz-Allen & Hamilton (BAH) was contracted to conduct waste, emissions, and cost analysis for the conventional and advanced processes. These estimates were to be refined during the course of this program. The BAH report supported the preliminary work completed before the demonstration began. These preliminary efforts are described below.

The waste and emissions estimates for the current batch process rely on data, sampling, and testing conducted by the Environmental Management Office at ATK. Figures 3-2 and 3-3 are mass balances of the current carpet roll production process and the batch grain manufacturing process, respectively. Table 3-2 shows the total waste and emissions based on 200,000 delivered Mk 90 batch produced grains (with inspection lots of 44 motors per lot of 20,000 motors). Emissions of NG into the air and water during the carpet roll process are not included. These emissions are very difficult to measure directly; the amount of NG that leaches into the process water also depends on how often the water has been recycled. The SRM process will also generate these waste streams. It is anticipated, however, that less waste will be generated during the advanced process; less material is processed to produce the same number of propellant grains. The waste water from the SRM will contain contaminants from the ingredients in NOSIH-AA2. ATK does not monitor each building for the Virginia Pollution Discharge Elimination System (VPDES) permit. The concentrations of constituents from the SRM are not anticipated to be higher than the current batch process. The SRM should only affect the waste water volume, not the total amount of constituents at the Waste Water Treatment Plant. Waste emissions and cost data on the batch process will continue to be analyzed.

Estimates have been made to determine the waste and emissions from the proposed advanced production process. Figures 3-4 and 3-5 are mass balances of the pellet production process and the continuous TSE grain manufacturing process, respectively. The 144 lb of waste generated during TSE is due to losses during start-up and shutdown. Table 3-2 show the waste and emission estimates based on 200,000 Mk 90 grains, delivered.

A large amount of data exist on AA-2 propellant carpet roll and Mk 90 propellant grains. The Mk 90 propellant grain testing is performed in accordance with product specification AS 2544L. Testing of AA-2 propellant carpet roll is conducted in accordance with AS 2543D. These data will be used as the comparison baseline for the advanced process.

Table 3-2. Waste Emission Data for Advanced Process

					Pounds In	Pounds Out	Sample			Solid Waste (lbs)	Water Waste (gallons)	Vapor Waste (lbs)	
Slurry Mix					1,677,766	1,644,211				33,555	1,025,000		0.61 gal water/lb prop ~2% mix - solid waste
Blender					1,712,775	1,701,080				11,695	7,790,000		Add 4.17% LC-12-15 0.68% Solid Waste
PreRoll Mill					1,701,080	1,664,640				36,440			2.14% Solid Waste 4.58 gal water/lb prop
Even Speed Roll Mill					1,664,640	1,664,094				546			0.03% Solid Waste
Carpet Roll					1,664,094	1,661,930	2,164						0.13% Sample
Total										**82,236**	**8,815,000**		
	Grain Weight In (lbs)	**Grain Weight Out (lbs)**	**Grains In**	**Grains Out**	**Pounds In**	**Pounds Out**	**GLAT**	**Reject Grains**	**Grains Waste (lbs)**	**Solid Waste (lbs)**	**Water Waste (gallons)**	**Vapor Waste (lb)**	
Press	7.87	7.65	---	211,173	1,661,930	1,615,472				46,458			0.22 lb per grain
Anneal	7.65	7.64	211,173	211,173	1,615,472	1,613,360						2,112	0.01 lb vapor per grain
Saw	7.64	7.45	211,173	211,081	1,613,360	1,572,552		92	703	40,808	3,703,000		0.04% reject 0.19 lb per grain 2.30 gal water/lb prop
End Inhibit	7.45	7.45	211,081	210,989	1,572,552	1,571,867		92	685	685			0.04% reject
Dowel Rod	7.45	7.20	210,989	210,989	1,571,867	1,519,120				52,747	4,232,002		0.25 lb per grain 2.69 gal water/lb prop
Coning	7.20	7.14	210,989	210,989	1,519,120	1,506,461				12,659	2,645,001		0.06 lb per grain 1.74 gal water/lb prop
Aging	7.14	7.08	210,989	210,989	1,506,461	1,493,802						12,659	0.06 lb vapor per grain
Final Inspection	7.08	7.08	210,989	200,440	1,493,802	1,419,115		10,549	74,687	74,687			~5% reject
GLAT	7.08	7.08	200,440	200,000	1,419,115	1,416,000	440						44 Grains/Lot, 10 Lots
Total										**228,044**	**10,580,003**	**14,771**	
Total Waste										***310,280***	***19,395,003***	***14,771***	

3.6. Testing and Evaluation Plan

3.6.1 Demonstration Set-up and Start-up. The project was initiated in late 1999. In each of three areas, initial steps were taken to prepare for the demonstration. The ATK engineers established a relationship with Nitrochemie to begin the development of a SRM feasibility study. The IHDIV/NSWC TSE engineering staff began their review of the potential facility modifications necessary to extrude the pellets. As part of this analysis, they established a contract with the SIT HFMI led by Dr. Dilhan Kalyon. Dr. Kalyon is an internationally recognized expert in the analysis and modeling of propellant material flow within processing equipment like the TSE. He and his team performed all of the rheological characterization studies on AA-2 propellant and used the study results to develop a new die design. His group's work will be discussed in detail later in this section. In addition to Dr. Kalyon's expertise, IHDIV/NSWC engineers obtained the services of Dr. Dietmer Muellar, also an internationally recognized expert in propellant processing, from the Institute of Chemical Technology (ICT) located in Karlsrhue, Germany. Dr. Muellar advised the IHDIV/NSWC engineers during the initial processing runs, both inert and live, at the TSE. His work is also presented in more detail later in this section. ARDEC initiated a contract with BAH to perform the environmental and cost analysis of the advanced and legacy processes. They also initiated a contract with SIT to perform the SRM process model development. A more detailed description of each of these efforts is provided below.

3.6.1.1. AA-2 Pellet Manufacture. The feasibility studies were conducted at Nitrochemie-Aschau (Germany) on the 200- and 300-mm SRMs with AA-2 paste produced at RAAP. Optimum operating parameters and throughput rates for producing the AA-2 pellets were determined. It was now possible for ATK to begin planning for the installation of the 200-mm SRM they already had in storage at RAAP. The facility design was initiated in January 2001 and completed in September 2001. An existing building was to have obsolete equipment removed, its utilities reconfigured, and the new SRM installed within the same building footprint. Construction was initiated immediately following design approval. The SRM facility construction was completed in October 2002 and the SRM was ready to support the demonstration in December 2002.

In addition to the facility readiness efforts, RAAP conducted a preliminary quality evaluation of the Nitrochemie pellets. In June 2001, the pellets were used to manufacture conventional, rolled sheetstock propellant. They were rolled flat and consolidated using the legacy rolling equipment. This meant the propellant could be loaded in the conventional ram press and be extruded, forming Mk 90 propellant grains. These grains were then analyzed in accordance with the Mk 90 propellant grain specification to include ballistic firings in October 2001. The very promising results are provided, in detail, in Section 3.6.6.2. These results provided additional evidence as to the feasibility of the project objectives.

3.6.1.2. Propellant Grain Manufacture. Four major tasks needed to be completed before the TSE portion of the demonstration could begin. First, a Process Review Board (PRB) was convened to evaluate the hazards posed by the extrusion of AA-2 propellant at the TSE facility. Secondly, any facility or equipment modifications required by the PRB hazards analysis would need to be planned and executed. Thirdly, the rheological behavior of AA-2 propellant would need to be well-characterized so that an appropriate screw configuration could be selected and a new die could be designed. The propellant's rheological properties also factored into safety concerns related to potential flow stagnation points within the extruder as well as a determination of the need for extruding under a vacuum. Lastly, optimal processing parameters needed to be found so that the production of grains could be begin. Each of these efforts will be discussed in greater detail in the following sections.

3.6.1.2.1. Process Review Board. The PRB was a group of senior technical and management personnel who had considerable experience with the TSE technology and the AA-2 propellant. Once the members were selected, a systems hazard analysis (SHA) was conducted to identify the hazards within the facility, equipment, and procedures. Based on this analysis and the lessons learned during several inert processing campaigns, some key modifications to the TSE facility were implemented.

3.6.1.2.2. Facility and Equipment Modifications. The facility and equipment modifications were outcomes either directly from the SHA or based on the results of the inert processing trials. In particular, the screw/barrel configuration and the TSE operating parameters selected for the initial live runs were only decided upon after extensive review of inert processing trials. The key facility and equipment modifications are described in detail below, including an extensive review of the inert work.

- *Die release system*—This addition to the TSE provided a means to vent pressure from the extruder as the pressure rose above 3,000 psi. It ensured that any overpressure event did not transition to a detonation.
- *Screw removal system*—This new equipment allowed for the safe removal of the screws if the equipment was shutdown abruptly and could not be restarted. It allowed for the remote removal of the screws and therefore reduced operator exposure to the associated hazards. Two photographs of the equipment are provided in Figure 3-6.
- *Engelhardt vibratory tray feeder*—Based on feeder studies conducted at IHDIV/NSWC we added a vibratory feeder to our 40-mm facility. This allowed us to feed the AA-2 pellets without distorting them. The feeder required extensive modifications to meet the National Electric Code for a Class 1 Division 1 and Class 2 Division 1 environment. We applied NFPA requirement 496 to meet code. Engelhardt, Inc is located in Germany. A further modification to the feeding system included a vented hopper. This hopper was tested with 30 lb of AA-2 propellant in a bomb proof at IHDIV/NSWC. This proved to us that this amount of AA-2 would not transition to detonation. We documented this with a formal drawing and set the ratio of open area as a standard for AA-2. Two photographs of this new equipment are provided in Figure 3-7.

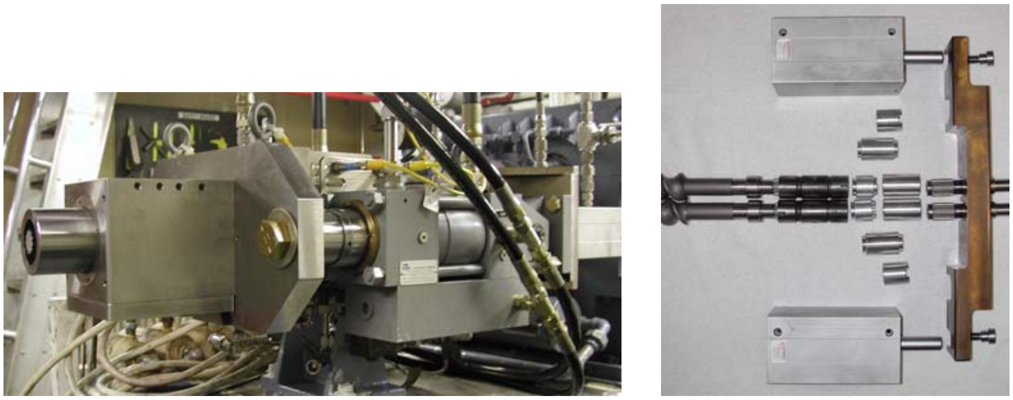


Figure 3-6. Die Lift-Off System (Left) and Barrel Screw Removal System Disassembled (Right)

Figure 3-7. Engelhardt Vibratory Tray Feeder Installed (Left) and Vented Hopper (Right)

- *Propagation break funnel*—There was a concern that an incident in the extruder could propagate to the feed hopper which could contain as much as 30 lb of pellets and create a significantly greater explosive event. To help mitigate this problem, a propagation break funnel was installed between the feeder and extruder. The funnel was designed and patented by a private sector explosives company. A photograph of the funnel is provided in Figure 3-8.

Figure 3-8. Propagation Break Funnel

- *Barrel/screw configuration*—The barrel and screw configuration were determined during initial inert extruder campaigns, which took place from August 2000 to May 2001, and through the AA-2 propellant characterization research. There were two efforts initiated to determine the appropriate configuration. One effort utilized the expertise of Dr. Kalyon and his staff at HFMI which, as mentioned earlier, had as an objective the complete rheological characterization of AA-2 propellant. The other effort took advantage of Dr. Muellar's propellant processing expertise with him witnessing both inert and live propellant extrusion runs. Both of these efforts occurred simultaneously and important knowledge from one was transferred to the other. At this point in this report, Dr. Muellar's work will be discussed in detail. The HFMI work will be reviewed in the following section.

  Dr. Muellar was present for three of these inert campaigns and provided detailed reports containing his observations and recommendations. These reports had significant influence on the project's direction and are summarized in the paragraphs below.

  The objectives of the three initial runs were to test the proposed operating conditions for the live runs and ensure there were no flow stagnation points within the extruder barrels, the barrel-to-die transition zone, or the die itself. The two different inert simulants that were extruded were designed to have fluid flow properties that were similar to AA-2 propellant. (Note: It would be determined that there were appreciable rheological differences between the live propellant and the simulants. These differences would hinder the optimization process for the remainder of the project. Due to cost and schedule constraints, no new simulant was developed.) For Runs 1 and 2 an inert simulant designated CAB-D, developed at ICT, was processed using the barrel/screw configuration shown in Figure 3-9. The simulant used for Run 3 was designated Sim-5 and had been produced at ATK. The barrel/screw configuration used for Run 3 is shown in Figure 3-10. The simulants had coloring die added to them during their respective extrusions so that the simulants flow could be traced. After an appropriate amount of time, the TSE would be shutdown, disassembled, and inspected to ensure the colored stimulant had moved through the extruder components leaving no uncolored simulant behind. The concern was that if propellant flow stagnates within the heated TSE components, the propellant may be in contact with the hot surfaces for too long and decompose resulting in a fire or explosion.

The operating set points and processing results for Runs 1 through 3 are provided in Table 3-3.

A list of Dr. Muellar's conclusions and recommendations based on the above results are provided below:

- *Conclusion 1*: There were no stagnation points in the barrel, the barrel-to-die transition area (also know as eight-to-round since the cross-sectional view of the barrel is a figure-8 and the die is round), or the die. Figure 3-11 shows the extruded inert billet. Note that the colored propellant has been completely processed through the extruder by the uncolored simulant, a clear indicator that there are no stagnation points.

- *Conclusion 2*: The L/D of 12 is optimum. Since the machine was only temporarily configured to mimic this L/D (actual L/D was 24 but only second half of screw/barrel utilized), it should be permanently reconfigured to this L/D for any additional runs of this type, inert or live. This means that the entire extruder should be shortened to half the length used during these tests.

- *Conclusion 3*: The screw configuration needs to be adjusted so that higher die pressures can be maintained without the screws touching as was the case in Run 3. High pressures created in the eight-to-round area caused screws to touch and created the observed mechanical noises. When shorter screws in the permanent setup are installed and the combination of kneading and conveying elements are optimized, the screw contact should be eliminated.

- *Conclusion 4*: The barrel temperatures used in Run 3 are too low for AA-2 propellant and the die pressures in Run 2 are too low so the new screw/barrel configuration must create higher pressures at the higher temperature.

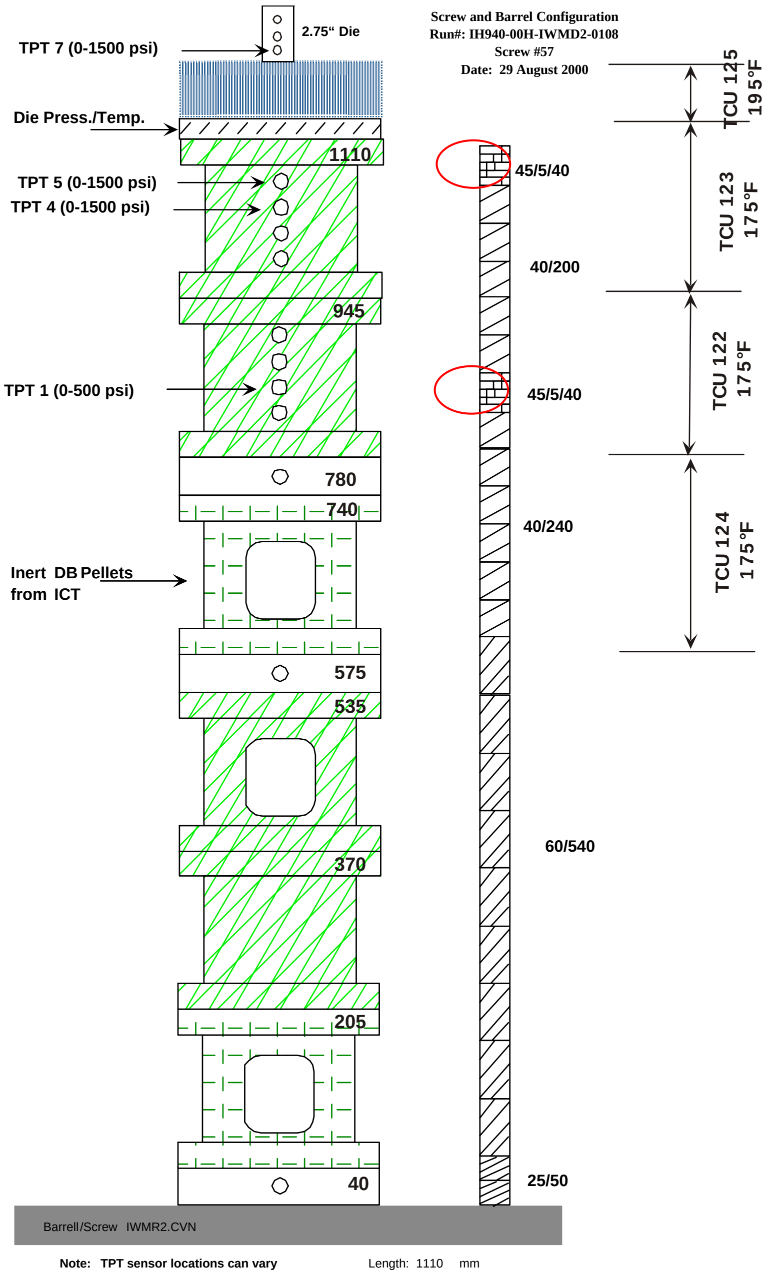


Figure 3-9. Barrel/Screw Configuration for Inert Runs 1 and 2 Showing Two Kneading Elements (Red Ovals) Positioned Within Conveying Elements

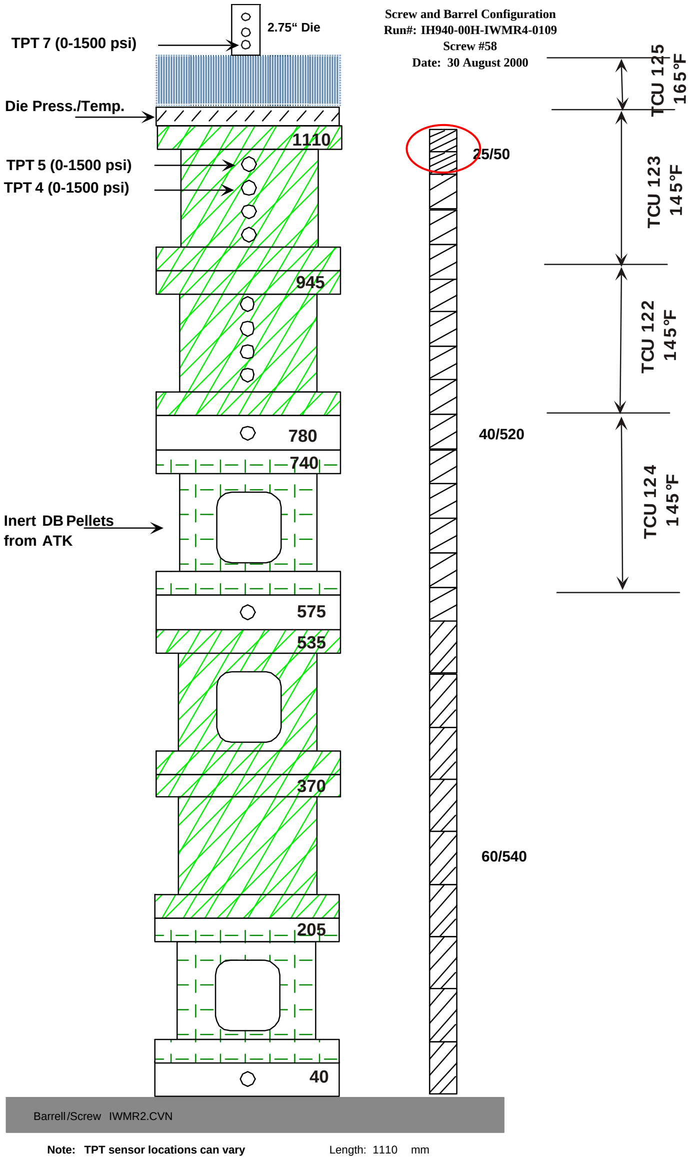


Figure 3-10. Barrel/Screw Configuration for Inert Run 3 Showing Conveying Element Tips on Screws (Red Oval) and Lower Initial Barrel Temperatures

Table 3-3. Campaign 1 Inert Run Processing Parameters

Parameter	Run 1	Run 2	Run 3
Propellant simulant	CAB-D	CAB-D	Sim-5
Barrel L/D	12	12	12
Die configuration	2.75-in w/11-point stake	2.75-in w/11-point stake	2.75-inch w/11-point stake
Screw RPM	8	12	8 to 15
Pellet feed rate (lb/hr)	8	8	8
Barrel temperature set point (°F)	175	175	146 decreased to 126
Die temperature set point	194	204	164 decreased to 146
Screw/barrel configuration	See Figure 3-9	See Figure 3-9	See Figure 3-10
Resulting die pressure (psi)	89	26	18 to 77
Propellant stagnation	None	None	None
Resulting torque (in-lb)	Not measured	Not measured	71 to 91 Noises were heard at the higher torque values
Inert grain quality	Fair	Fair	Good at low temp.

Figure 3-11. Inert Simulant Extruded Billet Showing No Flow Stagnation Points

- *Recommendation 1*: Use a screw configuration with two separate kneading elements to help center the screws. Also, if vacuum is necessary, one of the elements can be used near the vacuum port.

- *Recommendation 2*: Install die release system similar to the one used at ICT 58-mm TSE (this modification was described earlier in this section).
- *Recommendation 3*: Use an infra-red (IR) camera to monitor temperature profile in propellant layer directly in contact with eight-point stake.
- *Recommendation 4*: Monitor in-process viscosity in real-time to ensure shear stress is not increasing to dangerous levels (overheat propellant).
- *Recommendation 5*: Ensure any pellets being produced on the SRM are well characterized and compared to the legacy sheetstock propellant.

In campaign 2, the barrel and screws were reconfigured. The L/D was permanently set at 12 which shortened the barrel/screw assembly. The screws were configured with the two kneading elements spaced such that when they were filled with propellant they would support the screws. Conveying elements were positioned at the screw tips. Additional pressure-temperature transducers were installed so that an accurate pressure-temperature profile could be obtained particularly in the eight-to-round and die sections of the assembly. A schematic of the configuration is provided in Figure 3-12.

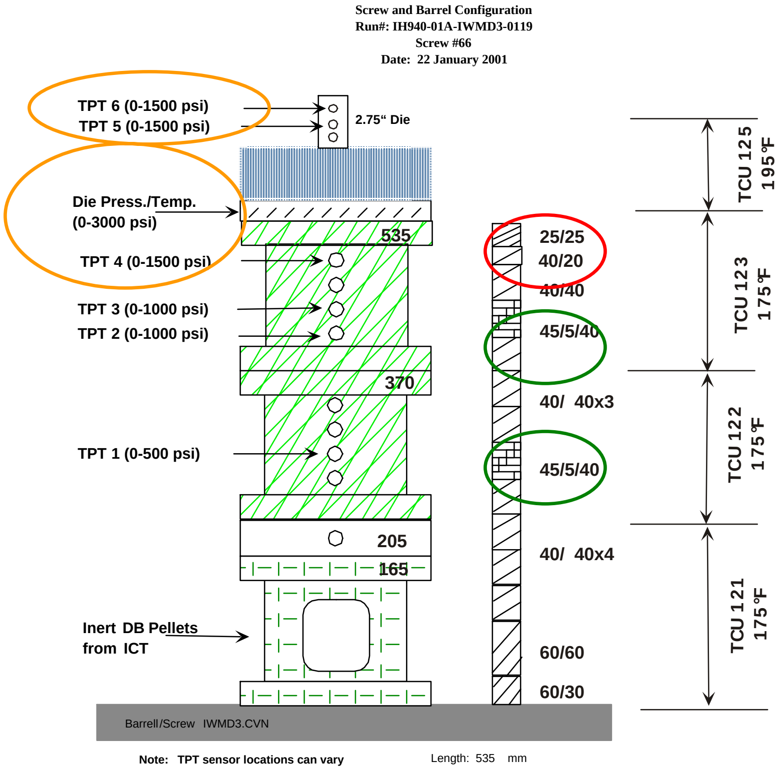


Figure 3-12. Barrel/Screw Configuration for Campaign 2 Runs With Two Kneading Elements (Green Ovals), Conveying Element at Screw Tips (Red Oval) and

Additional Pressure-Temperature Ports (Orange Oval)

This campaign's objectives were to validate the configuration, measure the pressure drop over the length of the die, and determine the level to which the screws were filled during the extrusion. The screw fill level was an important variable that was critical to determining if the live extrusion could be performed without vacuum. If the screws were not filled, air could escape from the extruder and would not be adiabatically compressed. Adiabatic compression of entrapped air had to be avoided as it could cause an explosive incident. In addition, the new die release system had been installed and was evaluated. Table 3-4 provides the operating conditions for each run.

Table 3-4. Campaign 2 Inert Run Processing Parameters

Parameter	Run 1	Run 2	Run 3
Propellant simulant	Sim-5	Sim-5	CAB-D
Barrel L/D	12	12	12
Die configuration	2.75-inch w/11-point stake	2.75-inch w/11-point stake	2.75-inch w/11-point stake
Screw RPM	15	15	15
Pellet feed rate (lb/hr)	10	10	10
Barrel temperature set point (°F)	175	175	175
Die temperature set point (°F)	195	195	195
Screw/barrel configuration	See Figure 3-12	See Figure 3-12	See Figure 3-12
Resulting die pressure (psi)	115	115	~60
Resulting die temperature (°F)	205	205	204
Screw fill level	Low	Low	High
Inert grain quality	Good	Good	Good

These processing runs were considered to be very successful. The barrel and die pressures were excellent and the pressure fluctuations during the extrusion were appropriate for this type of material. These results indicate that the conveying elements located at the screw tips were increasing the pressure adequately. The temperature rise along the barrel and in the die was acceptable. There were no mechanical noises coming from the extruder which indicated that the shorter screws and the kneading element locations were maintaining the proper screw alignment.

The important question related to screw fill level was also answered with a positive result. The measure of screw fill is represented by a dimensionless value called Q*. The Q* value is a function of feed rate, screw speed (rpm), feedstock density, and the screw diameters. The following equation defines the relationships:

$$Q^* = Q/(\text{Screw Speed} \times \rho \times (\text{Screw Radius})^3)$$

Where Q is mass throughput in kg/s, screw speed is in radians/sec, ρ is density in kg/m^3, and screw radius is in meters. The higher the value of Q*, the higher the screw fill. At Q* values near 0.2, the screws are very full or operating at a "flooded" condition. At Q* values less than 0.1, the screws are not full and operating in a "starved" condition. The Q* value for these runs was 0.0761 and were being operated in what was hoped to be a starved condition. The extruder was disassembled during the runs and inspected to verify this condition. Figure 3-13 shows the screws with the simulant still in place and they were indeed operating in the starved condition:

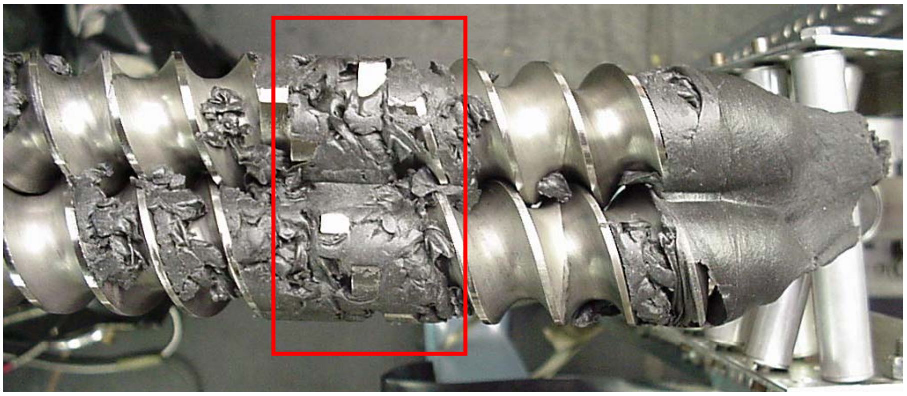


Figure 3-13. 40-mm TSE Screws Removed From Barrel During Inert Run Showing Starved Condition and Kneading Elements Filled Providing Screw Support (Red Box)

Thus an important decision could now be made. The TSE could be run without vacuum because air would be able to flow back through the extruder barrels and the risk of adiabatic compression would be negligible.

Dr. Muellar's conclusions and recommendations supported a decision to move to live operations but he had one technical concern. The die body, the outer portion of the die that forms the outside diameter of the extruded grain, was temperature controlled, but the die stake temperature was not because it did not have any heated fluid flowing through its completely solid construction. Since the multi-pointed stake forms the internal configuration of the grain, this inability to control its temperature may cause problems with successfully forming the internal annular perforation. Dr. Muellar provides a possible remedy to this problem in the following list of conclusions and recommendations:

- *Conclusion*: The L/D and screw configuration are correct and should be used for AA-2 processing.

- *Conclusion*: The process temperature, feed rate, pressures, and screw speed are in the correct range.

- *Conclusion*: The live processing runs can be started with inert material being fed and then transitioning to live AA-2 pellets but it may take as long as 45 minutes for the inert material to clear the extruder.

- *Recommendation*: IHDIV/NSWC engineers should maintain a record of the pressure drop across the die and use these data together with feed rate and temperature to monitor the "process viscosity."

- *Recommendation*: IHDIV/NSWC engineers should consider fabricating a metal filling ring that can be placed over the die stake so as to transfer heat from the die body to the stake. This ring should be used before each run to bring the two to nearly the same temperature.

- *Recommendation*: IHDIV/NSWC engineers should complete all components of the die release/screw removal system and have it properly tested.

With the inert work now completed, the findings were provided to the PRB members in the "Justification for Scale-up Memorandum." This memorandum described the findings from the inert processing campaigns and provided the rationale for the initial live propellant extrusion operating conditions. It also outlined the reasoning for operating without the use of vacuum.

This completes the description of both the facility/equipment modifications and the determination of the screw/barrel configuration. The parallel effort to characterize the AA-2 propellant's rheological properties so that a new die could be designed will be described in the following section. The new die design was not necessary for live propellant extrusions to proceed per the PRB. However, in order to produce propellant grains that met the grain specification, the new design was critical.

3.6.1.2.3. Rheological Characterization of AA-2 Propellant. This was an extensive effort that actually required more than two years to complete. Dr. Kalyon and the HFMI staff began there research in mid-2000 and submitted their die design to the IHDIV/NSWC engineers in December 2002. To put this in chronological perspective, the initial live extrusions, using the legacy die/stake assembly from the extruded composite propellant grain project, were completed in December 2001 and January 2002. They were both reasonably successful considering they were the first double-base propellant extrusions ever performed at IHDIV/NSWC. The new die/stake assembly would be the next step towards extruding acceptable grains and operating for the substantial run times necessary to collect data that was statistically significant enough to verify the waste reduction estimates outlined earlier in this report. However, before any die could be fabricated, the rheological behavior of AA-2 propellant had to be characterized. The following is a selection of report summaries that describe in sufficient detail the work that led to the new design. The amount of technical data collected by IHDIV/NSWC engineers and the analytical information generated by HFMI is considerable, and in many cases, theoretical. These summaries capture key discoveries and critical recommendations that led to the new design.

– Report 1, Flow and Deformation Behavior of AA-2 Formulation, 26 December 2000

This report provides results of extensive testing performed on live AA-2 propellant in capillary rheometers using dies of different diameters and L/D ratios. Capillary dies are circular dies of known diameter through which propellant is extruded under different conditions. In this case, the propellant was extruded through instrumented and jacketed dies of 5- and 6-mm diameter using a conventional hydraulic press with a 2-in diameter ram/cylinder. A tapering transition section connects the larger diameter cylinder to the capillary diameter. The data collected provided key pressure versus shear rate/stress information at various temperatures, and rheometer die length to die diameter ratio. These data were most useful in determining the appropriate die configuration for the AA-2 extrusion. The dependencies between temperature, pressure, and L/D were graphically presented. The report clearly indicates that additional work was necessary to adequately complete the rheological characterization of AA-2.

– Report 2, Mathematical Modeling of the Flow of AA-2 Through the 2.75-Inch Die at a Screw Rotational Speed of 15 RPM, at 11.5 lb/hr, and at a Die Temperature of 160 °F, 16 May 2001

The report provides the results of a finite element model (FEM) run on the flow of AA-2 through a 2.75-in diameter die with an 11-point stake (extruded composite configuration used in inert and initial live extrusions). It reported on the expected shear stresses that would prevail in different areas of the eight-to-round transition and in the die itself. It assumed a temperature of 165 °F and a flow rate of 11.5 lb/hr. It provided relational equations showing the shear rate dependency on temperature as well as three-dimensional results of the FEM. The results indicated a pressure drop of nearly 2,200 psi was required to move the AA-2 through the die. This is a very high value for the TSE. The anticipated temperature rise was 13 to 178 °F, which was seen as conservative and reasonable. However, there was concern that the viscous energy generated when the screws attempt to create the required pressure mentioned above would drive the temperature up to some unknown endpoint. Dr. Kalyon states that more analysis would be required to quantify this temperature rise. Some of the key recommendations were:

- Set the die temperature to 190 °F so that the required pressure drop is reduced to approximately 600–700 psi.

- The actual pressure drop and propellant temperature should be measured during the next live extrusion and compared with the report data.

- Additional analysis should be performed on the extruder section to determine the potential temperature rise as it generates the required pressures.

– <u>Report 3, Extrudate Swell Behavior of AA-2, 17 July 2001</u>

This report provides results on die swell experiments performed with AA-2 propellant by IHDIV/NSWC personnel. Both the outside diameter swell and the wall thickness swell were investigated. The experiments used a 2-in diameter ram press and an annular die specifically designed to match the expected residence time of the eight-point star die. The ram rate of the press was operated at various rates to simulate various mass flow rates at the TSE. The tests were done at two temperatures, 160 and 190 °F. The results are given in Table 3-5.

Table 3-5. AA-2 Propellant Die Swell Test Results

Test Parameter	Temperature (°F)	Extrusion Speed Range (in/min)	Die Swell Range (%)
Diameter swell	160	0.4–2.0	1.9–3.7
Diameter swell	190	0.2–2.0	1.9–3.2
Wall thickness swell	160	0.4–2.0	6.0–13.0
Wall thickness swell	190	0.2–2.0	7.0–13.0

Dr. Kalyon's report indicates that these values are typical for highly filled materials. The die swell increases with shear rate (extrusion speed) and has only a minor dependence on temperature. These were not considered to be unusual findings. This information was used to design the new AA-2 propellant extrusion die.

– <u>Report 4, Analysis of the Shear Viscosity and Wall Slip Data of AA-2 Collected at IHDIV/NSWC, 14 December 2002</u>

This report describes the additional work called out in Report 1 where slightly larger capillary rheometers were built (8 and 9 mm vice 5 and 6 mm) to better characterize the flow behavior of AA-2 propellant. These tests exposed some problems with the ram press used to extrude the material through the capillaries. The actual values obtained from the 200 °F testing did not agree well with the predicted values based on temperature dependency equations. This suggested that the AA-2 was not behaving as expected at higher temperatures. The 170 °F testing did agree well so that information was used to continue with the design of the new die.

Another area of concern was the calculation of entrance and exit effects on the pressure drop. At 190 °F these effects are negligible, while at 170 °F these effects constitute a majority of the pressure drop. These results were not expected and seemed to disagree with the normal behavior of materials like AA-2 propellant. The greatest difference between the two temperatures occurred at low shear rates, which is the area where they were expecting to operate when actually extruding AA-2 propellant with the TSE.

Recommendations included an altering of the ram rate determination procedure and performing additional testing using the IR thermal imaging camera so that the differences in entrance and exit effects could be better understood. The use of the camera would verify the actual temperatures of the material as it exited the capillary.

– Report 5, Mathematical Modeling of the Flow and Heat Transfer of AA-2 in the 40-mm Twin Screw Extruder and 8-Point Star Die, 19 December 2002

This a very important report that described the results of a FEM evaluation of the proposed new die design. The initial AA-2 propellant extrusion was already completed at this point but was accomplished using the 11-point star die from the extruded composite grain program. This new die would allow the TSE to produce grains that met the applicable rocket motor specifications. Dr. Kalyon's group was in the final stages of the design and utilized an FEM analysis to ensure that the pressure drop across the length of the die and any temperature increases due to viscous heating would be safe and obtainable at the 40-mm TSE. The die was designed to initially have a perfectly straight stake resulting in a constant annular space along its post-transition area length. However, the design was modular, allowing for the interchanging of stakes, which allowed for later adjustments in final grain dimensions. With that in mind, the FEM analysis included stakes with a 10 percent decreasing diameter and a 10 percent increasing diameter over the stake length.

Some interesting comments concerning some of the die design constraints are included in this report. A first constraint was to keep as much of the original extruded composite design unchanged and this resulted in a relatively long transition zone from the extruder barrels to the die. An additional constraint was to maintain the overall length of the original die body so that auxiliary TSE components would not need to be modified. These two constraints would reduce the length of the straight section of the die to just over 7 in. This is less than half the length of the same die section used in the conventional AA-2 propellant grain extrusion press which is slightly more than 15 in. The impact of these constraints will be discussed later.

The FEM analysis was run with the following assumptions:

- Feed rate at 11 lb/hr
- Propellant entering the die at 190 °F
- The barrel walls and die are maintained at 170 or 190 °F and do not change

- The stake is assumed to be mechanically unheated and absorbs heat energy without transferring it away.

The conclusion of the analysis was that the maximum average pressure drop associated with these conditions and configurations was 750 psi. This pressure can easily be created by the TSE so the design from this perspective looked acceptable. There was also a negligible temperature rise within the die due to the very low levels of shear stress being imparted to the propellant. Again, this was a desirable result. It also concluded that at a screw speed of 15 rpm and a feed rate of 10-11 lb/hr, the screws were not running full, which was also a desirable condition. The final die design would now be submitted.

The rheological characterization had progressed adequately to initiate the fabrication of the new die. While this research effort was progressing, the IHDIV/NSWC engineers had presented all of their inert processing run information and their startup recommendations to the PRB panel in August 2001. The panel reviewed the information and responded with 13 Class 1 corrective actions. The Class 1 corrective actions needed to be completed before live operations could begin. The actions were completed in October 2001 and final approval was granted in November 2001. A significant milestone had been reached. The first live AA-2 propellant extrusions from the 40-mm TSE would begin in December 2001. There would be five attempts at extruding propellant from the extruder over the following four years. These efforts never moved from a operating condition test phase to the demonstration phase and for that reason, all of the live processing runs will be described in the following section.

3.6.1.2.4. Live AA-2 Propellant Extrusions at the 40-mm TSE. In much the same procedure that was followed with the inert processing runs, the live runs can be broken into several separate campaigns. Each campaign will be described in detail below. The initial campaigns utilized the legacy extruded composite grain die. The new die/stake assembly was fabricated and introduced into the live propellant extrusion testing regime. The assembly did not perform as expected so the efforts of Dr. Kalyon's HFMI team to analyze the processing problems will be included where appropriate.

- <u>Live AA-2 Extrusion Campaign #1, Two Runs, 11–13 December 2001</u>

  The initial live extrusion, Run 1, was executed using the operating parameters determined during the inert processing runs. Dr. Muellar was present for this campaign. The extrusion began with the feeding of inert pellets into the TSE using the legacy screw-type feeders. The inert pellet feed rate began at 8 lb/hr and then was stepped down to 5 lb/hr and finally reduced to zero. The live pellet flow rate was correspondingly increased up to a 10-lb/hr feed rate. This procedure was followed for all of the live extrusions but with slightly different step increase rates and times. Table 3-6 provides the operating parameters and resultant maximum temperatures/pressures for the two runs.

Table 3-6. Live Campaign 1 Processing Parameters

Parameter	Run 1	Run 2
IHDIV/NSWC Lot. No. for extruded propellant	IH230-01M-AA21-0062	IH230-01M-AA22-0063
AA-2 Pellet Lot No.	12300	12300
Inert pellets used	Sim-5 (start), CAB-D (end)	CAB-D
Die configuration	2.75-inch w/11-point stake	2.75-inch w/11-point stake
Screw RPM	15	15
Pellet feed rate (lb/hr)	10	10
Barrel temperature set point (°F)	175	170
Die temperature set point (°F)	175	170
Screw/barrel configuration	See Figure 3-12	See Figure 3-12
Maximum die pressure (psi)	1635	460
Steady state die pressure (psi)	700	300
Resulting die temperature at sensor T6 (°F)	180–188	172–177
Torque range (in-lb)	200–250	150–180
Propellant grain results	Good	Poor

The extrusion runs were considered successes because they represented the first double-base propellant extrusions from the 40-mm TSE and there were no safety-related problems. The 40-mm TSE generates significant amounts of processing data during each run. This data is in spreadsheet format and can be used to create graphical representations of the pressure, temperature, screw speed, and torque during the extrusion period. The pressure, torque, and screw speed curves for both runs are provided in Figures 3-14 and 3-15. The temperature curves for both runs are provided in Figures 3-16 and 3-17. When reviewing these curves, the RHEO_P values are the pressure readings at locations along the barrel and die. The numbers after the P refer to the sensor location shown on Figure 3-12. For example, if the sensor location is designated TPT4 then the pressure data would be designated RHEO_P4. The same convention holds for temperature. The temperature data would be designated RHEO_T4. Some exceptions to this are the sensors located in the die which will be designated DIE_T or P with the location number immediately following. The numbering always increases in the direction of flow so DIE_T7 is closer to the propellant exit than DIE_T6. The feedback from the temperature controllers are designated TEMP_Z followed by a number.

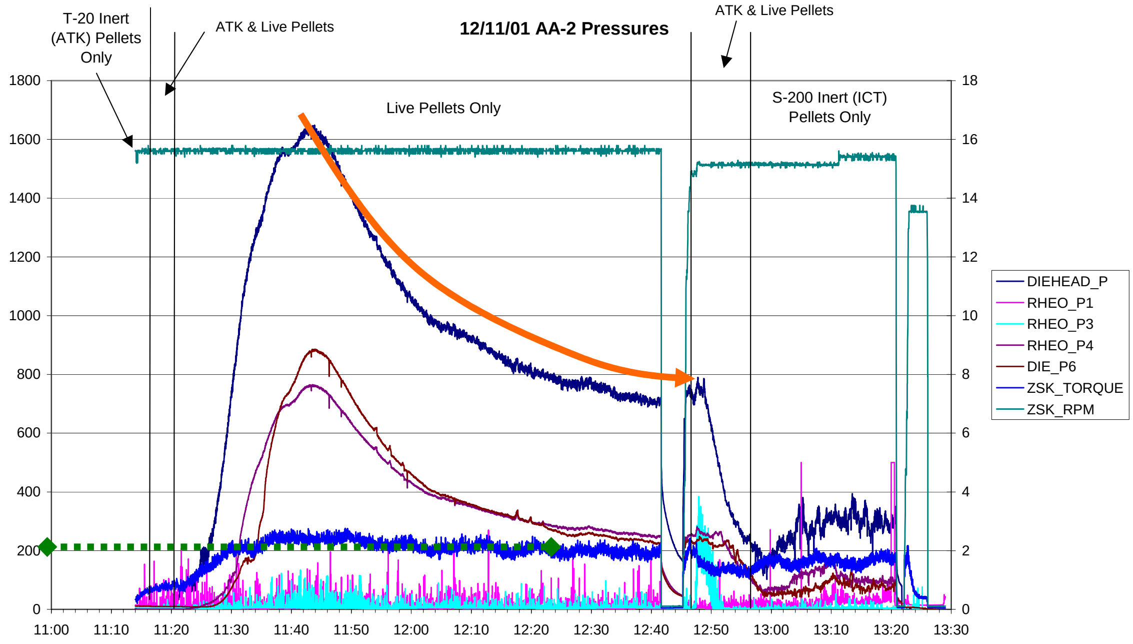


Figure 3-14. Pressure, Torque, and Screw Speed Trace IHDIV/NSWC Lot. No. IH230-01M-AA21-0062 (Run 1)

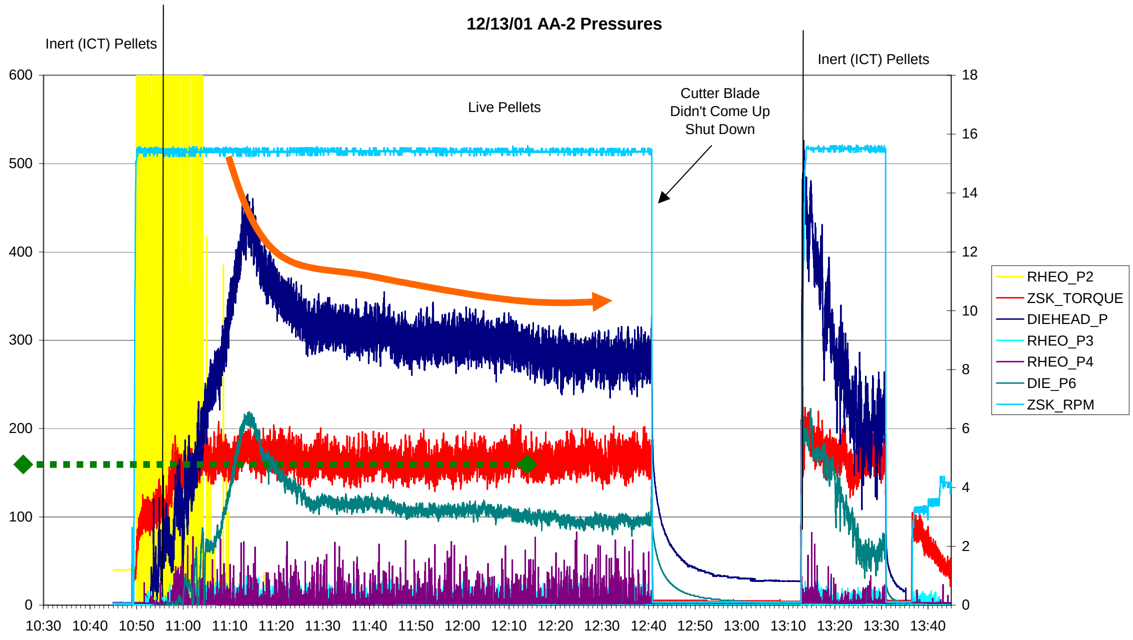


Figure 3-15. Pressure, Torque, and Screw Speed Trace IHDIV/NSWC Lot. No. IH230-01M-AA22-0063 (Run 2)

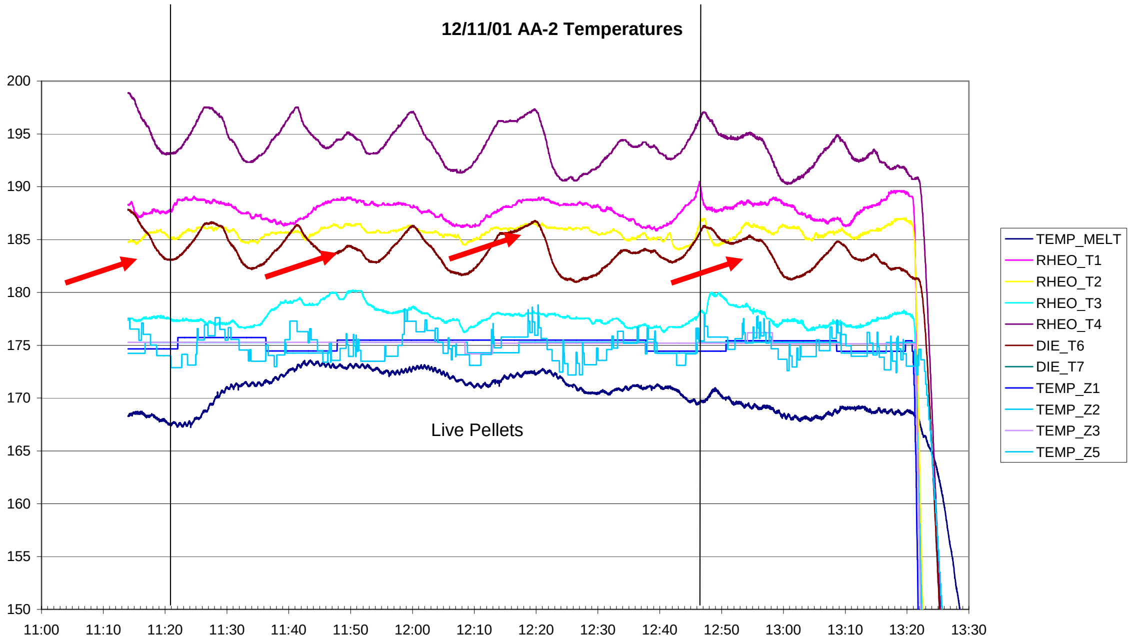


Figure 3-16. Temperature Trace
IHDIV/NSWC Lot No. IH230-01M-AA21-0062 (Run 1)

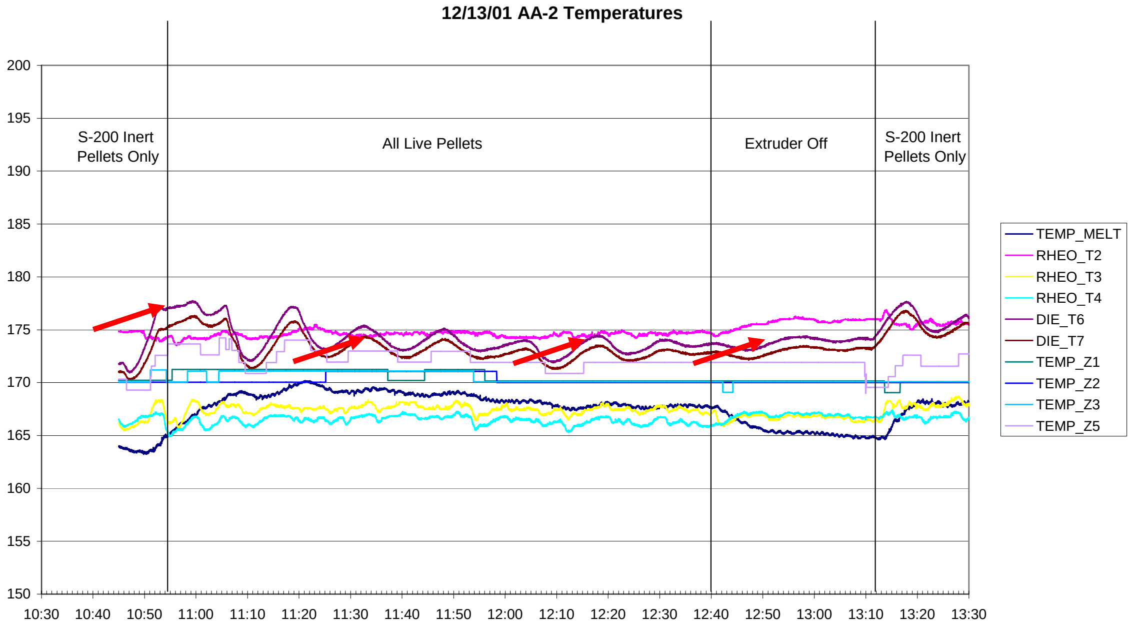


Figure 3-17. Temperature Trace
IHDIV/NSWC Lot No. IH230-01M-AA22-0063 (Run 2)

The key observation in Figures 3-14 and 3-15 is the difference in die head pressure. This is the pressure reading at the entrance to the die, where the supports that hold the stake centered in the die are located. In Figure 3-14, note how the pressure increases rapidly once AA-2 pellets are introduced into the TSE. The pressure peaks are 1,635 psi and then decreases steadily to approximately 700 psi (see orange arrow). Interestingly, when Dr. Kalyon calculated the anticipated pressure drop across the die, he had predicted it to be 600–700 psi at die temperatures of 190 °F (Report 2). These empirical results indicate that the pressure drop can be in the predicted range at temperatures lower than 190 °F. In Figure 3-15, the die pressure increases but not nearly as much, reaching only 430 psi. The pressure then decreases to approximately 290 psi (see orange arrow). The torque values were also lower for Run 2 (see the green dashed line in both figures). Remembering that Run 2 was at a slightly lower temperature, these data suggest that pressure at the die head is inversely related to temperature. This relationship will be examined further when additional extrusions are reviewed.

Throughout this section the DIE_T6 temperature will be used for comparative purposes. In Figure 3-16, the DIE_T6 temperature fluctuates between 182 and 187 °F, which indicates a temperature rise of 7 to 12 °F above the 175 °F set point (red arrows).

In Figure 3-17, the DIE_T6 temperature in Run 2 ranges from 172 to 177 °F, a full 10 degrees lower than Run 1 (see red arrows). As stated above, these lower temperatures resulted in lower pressures and lower torque.

However, the Run 1 conditions produced a much better extruded grain. The perforation was smooth as was the outer diameter. The grain was slightly twisted and did not swell at all having an outside diameter of 2.31–2.34 in. Run 2 produced a grain of poor quality with rough inner and outer surfaces. The propellant also did not reconsolidate after flowing around the stake support legs so there were longitudinal cracks along the grain. The consensus was that the propellant was not warm enough, nor was it subjected to adequately high pressures to reconsolidate and form a smooth, monolithic grain. We considered that the inability to control the temperature of the stake was contributing to the poor surface condition of the perforation.

We considered the following for future live extrusions:

- Increasing feed rate to 13 to 15 lb/hr may increase pressure and improve grain quality.

- Keep other operating conditions the same as Run 1, the higher die temperatures gave a superior grain.

- Consider raising barrel and die temperature to 180 °F.

- Live AA-2 Extrusion Campaign #2, Two Runs, 6–8 February 2002

The objective of Campaign #2 was to validate that the operating conditions from Campaign #1, Run 1 were indeed optimum and to investigate the suggestions to increase the feed rate and screw speed. The processing parameters associated with the two runs are provided in Table 3-7.

Table 3-7. Live Campaign 2 Processing Parameters

Parameter	Run 1	Run 2
IHDIV/NSWC Lot. No. for extruded propellant	IH230-03B-AA23-009	IH230-02B-AA24-010
AA-2 Pellet Lot No.	12300	12300
Inert pellets used	Sim-5	Sim-5
Die configuration	2.75-inch w/11-point stake	2.75-inch w/11-point stake
Screw RPM	15 and 19	15 and 19
Pellet feed rate (lb/hr)	10 and 13	10 and 13
Barrel temperature set point (°F)	178	Data lost
Die temperature set point (°F)	Oscillating from 165–180	Data lost
Screw/barrel configuration	See Figure 3-12	See Figure 3-12
Maximum die pressure (psi)	1100	681
Steady state die pressure (psi)	No steady-state attained	Data lost
Resulting die temperature at sensor T6 (°F)	Oscillating from 180–210	Data lost
Torque range (in-lb)	190–200	205
Propellant grain results	Poor	Poor

Both of these runs were plagued with equipment problems. In Run 1, the temperature controller that controls the temperature of the fluid being delivered to the die was fluctuating wildly. In Run 2, there was a power outage, during the run which resulted in a loss of electronic data. This is the reason for the limited available data and the absence of temperature, pressure, torque and speed traces for Run 2. The data from Run 1 will be examined in detail. The pressure trace for Run 1 is provided in Figure 3-18.

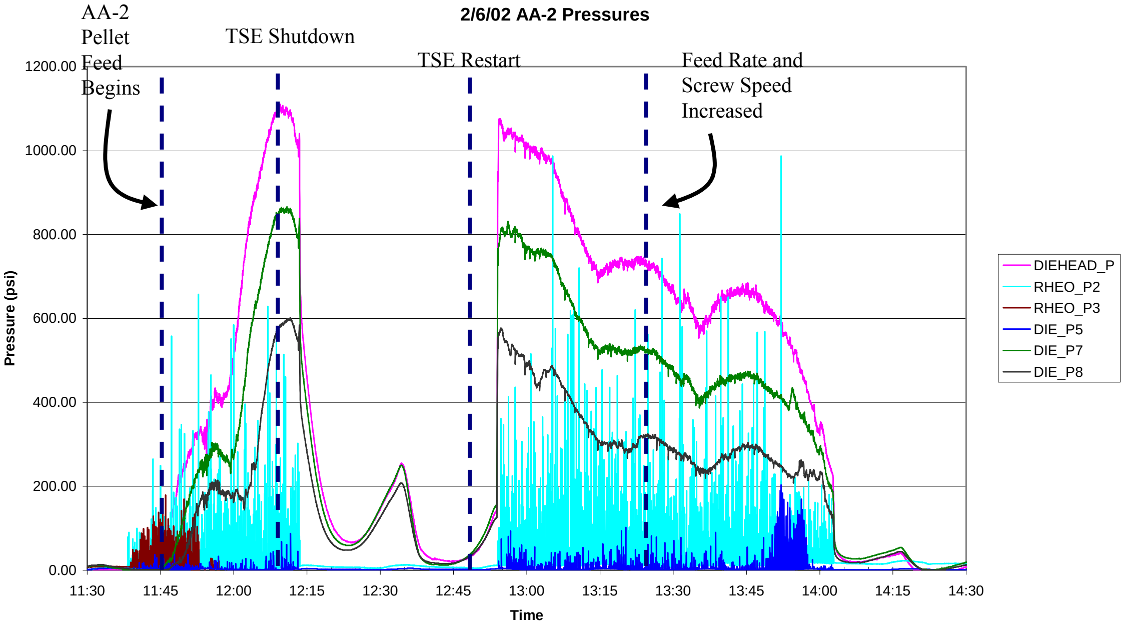


Figure 3-18. Pressure Trace, IHDIV/NSWC Lot No. IH230-03B-AA23-009 (Run 1)

The pressure trace begins similarly to Campaign #1, Run 1 (C1R1) with a steep climb in die head pressure after the AA-2 pellet addition begins. The pressure peaks at approximately 1,100 psi and then begins to decline. At 12:13, the TSE is shutdown for a cutter malfunction. When it is restarted after a 40-minute shutdown the pressure rises to 1,030 psi immediately. It then declines steadily to approximately 700 psi, levels out for 10 minutes and then begins to decrease once again. At 13:30 the feed rate was increased to 13 lb/hr and the screw speed was increased to 19 rpm. The die head pressure rises for 10 minutes reaching 670 psi before falling to nearly 300 psi before final shutdown. This pressure trace departs from C1R1 immediately after restart because it never approaches a steady-state pressure. The pressure drops in three distinct steps and continues to decline after the feed rate and speed increases. However, a review of the temperature data will reveal why these results cannot be compared to C1R1 in any meaningful manner. The actual temperature traces for the eight-to-round transition and the die are provided in Figure 3-19, while the die temperature controller output trace is provided in Figure 3-20.

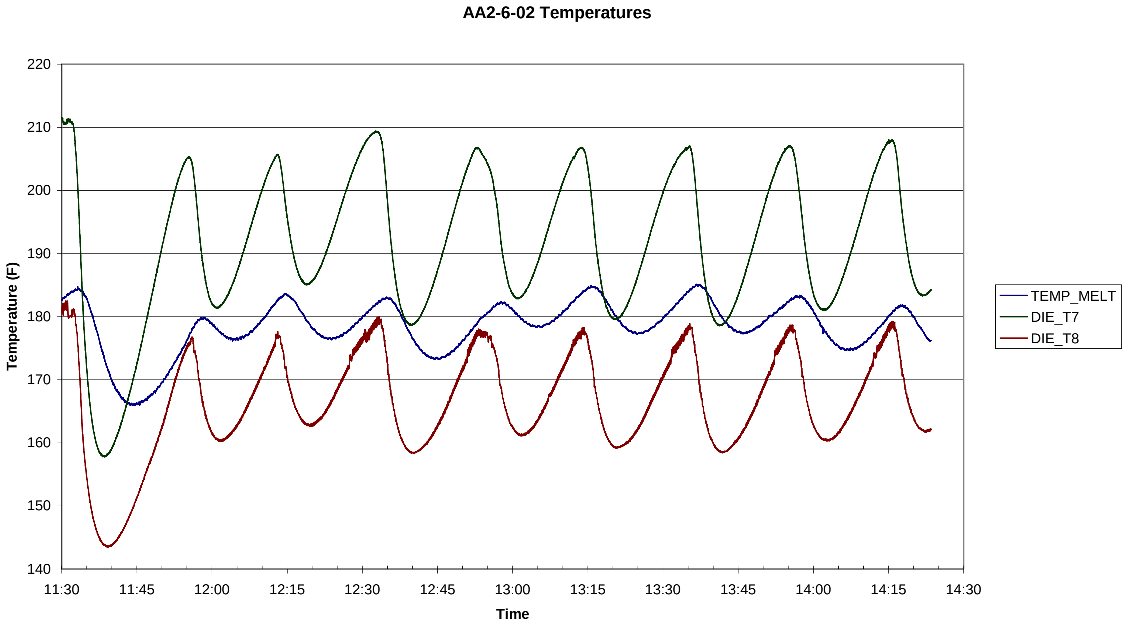


Figure 3-19. Temperature Trace
IHDIV/NSWC Lot No. IH230-03B-AA23-009 (Run 1)

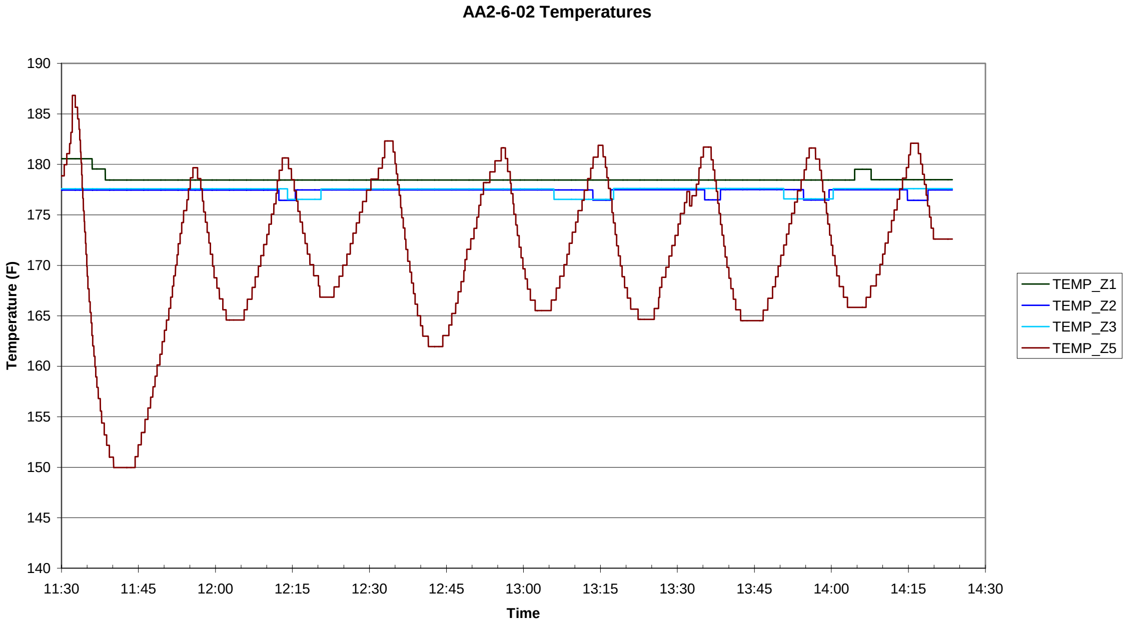


Figure 3-20. Temperature Controller Output Trace
IHDIV/NSWC Lot No. IH230-03B-AA23-009 (Run 1)

In Figure 3-19, note how the two die temperatures, DIE_T7 and Die_T8, are oscillating across significant temperature ranges. The DIE_T7 sensor, slightly further from the die exit, fluctuates between 180 and 210 °F, while the DIE_T8 sensor oscillates between 160 and 178 °F. The cause of these oscillations is clearly evident in Figure 3-20, which shows the TEMP_Z5 controller (this controller is connected to the die heating system) is out of control and oscillating. The effect of these temperature fluctuations is significant. The propellant is moving very slowly through the die so it is subjected to die temperatures for several minutes. As mentioned earlier, the propellant's rheological behavior is significantly temperature–dependent, so as the temperature changes so does the pressure required to move the material through the die. This means that the pressure readings for Run 1 are not comparable because of the wide temperature fluctuations.

There are few electronic data available related to Run 2 because of the power outage that occurred during the run. However, a significant amount of propellant was extruded. The written notes indicate that nearly 90 inches of propellant grain was extruded. The grains, four of varying lengths, were all longitudinally twisted due to the stresses imparted to the propellant by the rotating screws. It was clear that this problem would need to be resolved if acceptable grains were to be produced. The maximum pressure indicated in the written notes indicates a maximum die pressure of 681 psi, which is lower than previous extrusions at these operating conditions. There is no indication in the notes explaining this lower pressure nor are there any additional descriptions of the grain quality.

Campaign #2 ended with its objectives only partially met. The feed rate and screw speed had been increased with no appreciable increase in die head pressure. That determination was valuable for planning future extrusions. The validation of the C1R1 operating conditions was partially accomplished in that there were two more extrusions that safely extruded double-base propellant from the TSE. No progress related to process optimization was made due to the equipment problems encountered.

As mentioned in Section 3.6.1.2.3, Dr. Kalyon was performing the rheological characterization of AA-2 propellant during this same time period. That work would result in the design of new die in April 2003. The new die's fabrication would take nearly nine months of additional time. Also during this period, the Engelhardt vibratory feeder began to have electro-mechanical problems. It would be January 2004 before the feeder and the new die were ready to use as part of additional extrusions. There were some inert extrusion runs performed in March 2004 that were used to validate some operating condition modifications that were investigated by Dr. Kalyon's HFMI staff. Two major modifications were under consideration:

- Creating two temperature zones along the die length, 190 °F at the die entrance and 170 °F to cool the propellant before it exited the die because at the higher temperatures investigated in the earlier extrusions, the grain tended to slump under its own weight**.**

- The design and fabrication of a flow breaker plate that would be installed just before the die to break up the propellant flow and reduce the torsional forces imparted to the propellant and eliminate the grain twisting problem**.**

Dr. Kalyon and his staff analyzed both of these modifications in detail and concluded that both were feasible with regards to the increase in the required pressure for extrusion. The next live extrusion was scheduled for August 2004. The first modification was implemented, while the implementation of the second was delayed.

– Live AA-2 Extrusion Campaign #3, One Run, 4 August 2004

The goal of this campaign was to successfully consolidate AA-2 pellets into a 2.75-inch propellant grain that had an 8-point star perforation. The new die and stake were installed and there was the potential to extrude a grain that would meet the propellant grain specification. The operating conditions are provided in Table 3-8.

Table 3-8. Live Campaign #3 Processing Parameters

Parameter	Run 1
IHDIV/NSWC Lot. No. for extruded propellant	?
AA-2 Pellet Lot No.	12300
Inert pellets used	Sim-5
Die configuration	2.75-inch w/8-point stake
Screw RPM	15
Pellet feed rate (lb/hr)	10
Barrel temperature set point (°F)	185
Die temperature set point (°F)	185
Screw/barrel configuration	See Figure 3-12
Maximum die pressure (psi)	1500
Steady state die pressure (psi)	No steady-state attained
Resulting die temperature at sensor T6 (°F)	192
Torque range (in-lb)	110–270
Propellant grain results	No grains extruded

Some key differences in the operating conditions include the higher barrel and die temperatures, 185 °F vice 175 °F, and the use of the new die/stake assembly. Unfortunately, this run did not perform well. The die pressure and temperature increased to levels that exceeded the limits called out in the processing documentation. The TSE was shutdown before any grains could be extruded. It would be determined upon TSE disassembly that the screws had been damaged during the run. The damage to the screws was significant enough to warrant an engineering investigation. This investigation would further delay the project. A review of the pressure trace for the run, Figure 3-21, shows the rapid increase in die head pressure (orange arrow) after the feed had become entirely live propellant.

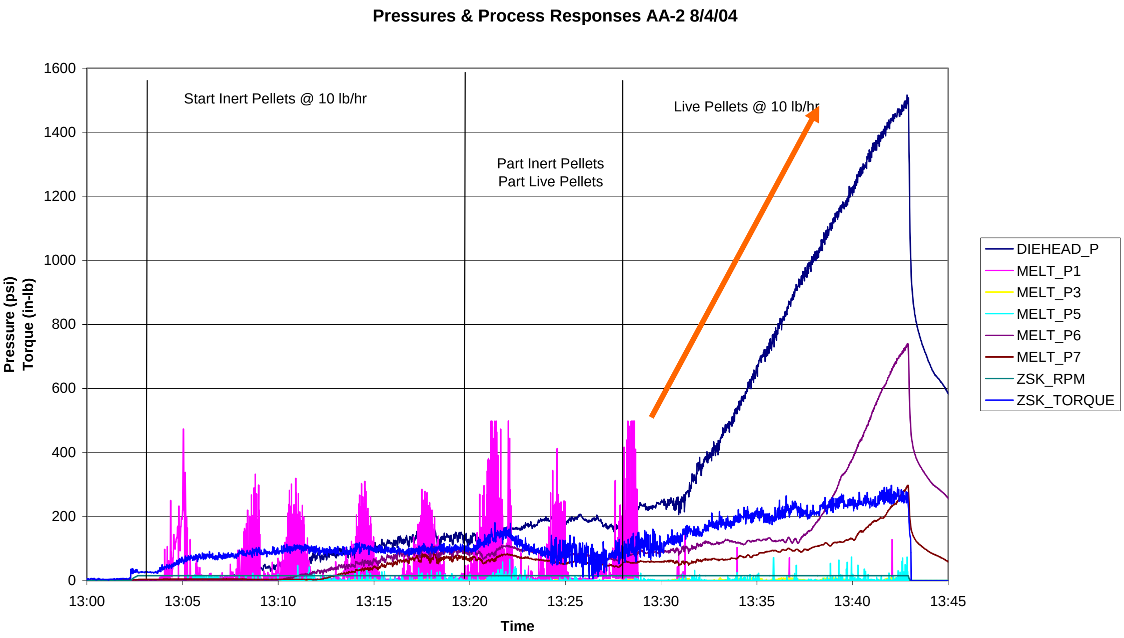


Figure 3-21. Pressure, Torque, and Screw Speed Trace IHDIV/NSWC Lot. No. ? (Run 1)

Note also that the torque increases steadily to a level not seen during earlier extrusions. The ensuing investigation would conclude that the screws had contacted one another and this contact created chips in the screws' finished surface. A photograph of one of the damaged areas is shown in Figure 3-22.

Figure 3-22. Example of Screw Damage on 40-mm TSE

The cause of the contact was not positively identified, but the most likely cause was the application of unbalanced forces to the screws. The source of the unbalanced forces was not determined. However, one of the investigation report's recommendations did address the feed rates of the inert material during start-up:

> *In order to lessen the potential for unbalanced forces caused by differing rheologies in the extruder, the feeding of inert stimulant should be limited to the minimum required to establish the material bearing supports before ramp-up of the double-base feed.*

The IHDIV/NSWC engineers implemented this and the other recommendations outlined in the report which was published in March 2005. The next live extrusion would not occur until June 2005.

– Live AA-2 Extrusion Campaign #4, Three Runs, 23–30 June 2005

At this point in the project, it was clear that the revised objectives outlined in the project's restructured DEM/VAL plan (2002) were not going to be accomplished. The ESTCP funding had been exhausted in 2003. The lengthy equipment-related delays had pushed the project well past its scheduled completion date. With the limited time and funding available the project's objective list was reduced to one. The objective for these three extrusion runs was to find operating conditions that produced propellant grains that could be processed through the remaining grain processing steps. If a grain was fully processed and was found to be free of physical defects, it could be ballistically tested. With that as the goal, the three extrusion runs were executed.

The processing parameters for Campaign #4 are provided in Table 3-9 and the Run 1 pressure, speed and torque trace is provided in Figure 3-23.

Table 3-9. Live Campaign #4 Processing Parameters

Parameter	Run 1	Run 2	Run 3
IHDIV/NSWC Lot. No. for extruded propellant	IH230-05F-AA2-0288	IH230-05F-AA2-0295	IH230-05F-AA2-0297
AA-2 Pellet Lot No.	12300	12300	12300
Inert pellets used	Sim-5	Sim-5	Sim-5
Die configuration	2.75-inch w/8-point stake	2.75-inch w/8-point stake	2.75-inch w/8-point stake
Screw RPM	15, reduced to 12	15	18, increased to 24
Pellet feed rate (lb/hr)	10, reduced to 8	8, increased to 12	12, increased to 16
Barrel temperature set point (°F)	175	175	170
Die temperature set point (°F)	175	175	170
Screw/barrel configuration	See Figure 3-12	See Figure 3-12	See Figure 3-12
Maximum die pressure (psi)	1650	1200	610
Steady state die pressure (psi)	No steady state attained	420	360
Resulting die temperature at sensor T6 (°F)	171–175	173–175	167–168
Torque range (in-lb)	200–650	400–575	360–470
Propellant grain results	Short sections extruded, fairly well formed	36-in grain extruded, passed radiographic inspection, diameter too small for further processing	Grain "hooked" as it left the die, small diameter, numerous longitudinal splits

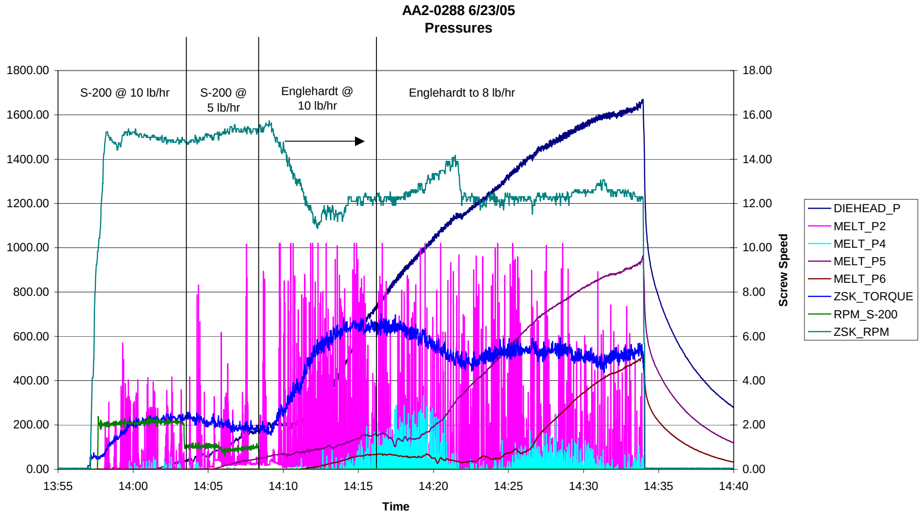


Figure 3-23. Pressure, Speed, and Torque Trace, IHDIV/NSWC Lot No. IH230-05F-AA2-0288 (Run 1)

In Run 1, there are two key data outputs to note, the torque and maximum die pressure. Both of these values are higher than normal. The torque is three times higher than the values seen in earlier extrusions. From Figure 3-23, it can be seen that the torque began increasing rapidly after the live pellet feed rate was increased to 10 lb/hr. The screw speed decreases from 15 to 12 rpm as the torque increases. In response to the decreasing speed, the TSE operator lowers the AA-2 pellet feed rate to 8 lb/hr in to maintain a constant screw fill level. The die head pressure continues to increase during these adjustments, climbing to over 1,600 psi. Several small propellant sections were extruded and cut. They were well consolidated and held their shape better than previous grains. Unfortunately, the extrusion run was terminated because the guillotine cutter blade became stuck in the extruded propellant and would not free itself. The run was not restarted.

The die and barrel temperatures were well controlled at approximately 175 °F and will not be reviewed in detail since the run was aborted.

The IHDIV/NSWC engineers drew some conclusions from this run:

- The high pressures produced well consolidated grains
- The torque can be adjusted by adjusting feed rate and screw speed
- The guillotine cutter requires additional lubrication and maintenance.

It should be noted that the water-jet cutter described in the initial project description and in the DEM/VAL plan was never used as part of these extrusion campaigns. The costs associated with its implementation proved to be quite high and the IHDIV/NSWC engineering staff felt it was more important to apply resources to the extrusion issues rather than introduce the new cutting technology.

Run 2 was initiated with the same processing conditions as Run 1 with the exception of a slightly lower feed rate. The pressure, speed, and torque trace is shown is Figure 3-24.

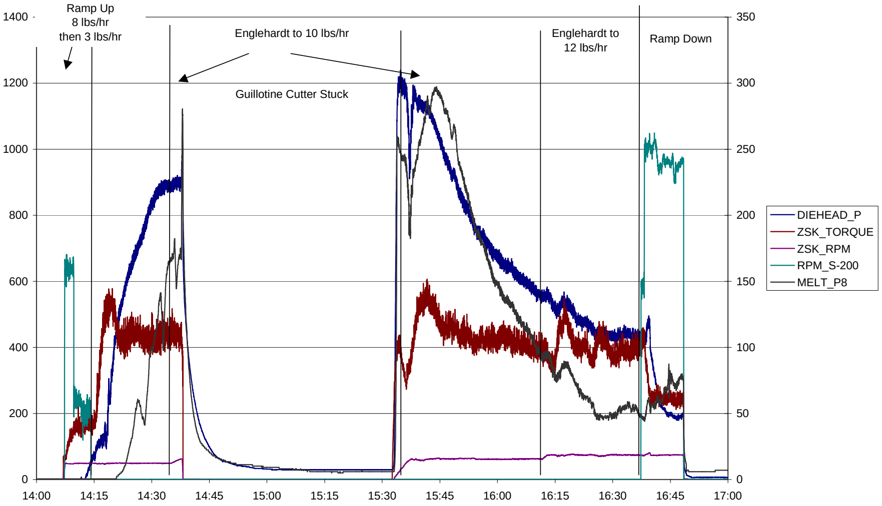


Figure 3-24. Pressure, Speed, and Torque Trace,
IHDIV/NSWC Lot No. IH230-05F-AA2-0295 (Run 2)

The engineers had decided to shorten the time taken to switch from feeding the inert pellets to feeding live AA-2 pellets during ramp-up. They were hoping this would increase the die head pressure and their predictions were well founded. After the short ramp-up, the initial setting of the AA-2 pellet feed rate was 8 lb/hr and produced a torque value of 450 in-lb and a die head pressure of 900 psi, which while not being as high as Run 1, were still higher than earlier campaigns. The feed rate was increased to 10 lb/hr. The torque and pressure values were holding steady for a few minutes when the guillotine cutter failed during its first grain cut of the run. It failed to release from the propellant and the run was halted. However, after the cutter blade was released manually, the extruder was restarted. Both the pressure and torque increased after restart to 1,200 psi and 550 in-lb, respectively. They both began to decline steadily after about 15 minutes of run time. When the die head pressure had decreased to 600 psi, the pellet feed rate was increased to 12 lb/hr in an attempt to increase the pressure. The pressure continued to decline until stabilizing at 420 psi. The torque stabilized at approximately 400 in-lb. At this point, a 54-in long grain had been produced and was reaching the end of the grain support table. The guillotine cutter was inoperative so the TSE operator stopped the run and began to reduce the live pellet feed rate and introducing the inert pellets. A 36-in long section of propellant grain was manually cut from the 54-in grain. The grain was radiographically inspected per the Mk 90 propellant grain specification. It passed this inspection. However, the grain's outside diameter was approximately 1/4 in smaller than a nominal grain. This meant it could not be processed safely though the grain finishing steps. The predicted propellant grain swell had not occurred and it meant that the processing conditions employed during this run could not produce an acceptable grain.

The temperature control during this run was excellent. The temperature trace is shown in Figure 3-25. Note that TEMP_T6 is very well controlled at approximately 174 °F. Also note the two-zone temperature profile along the die length was well executed. At temperature sensor DIE_T8, further from the die's exit, the temperature ranged from 171–174 °F, while the sensor at DIE_T9, at the die exit, shows the temperature ranging from 158–162 °F. This lower temperature over the second half of the die's length cools the propellant and minimizes the slumping that was mentioned earlier.

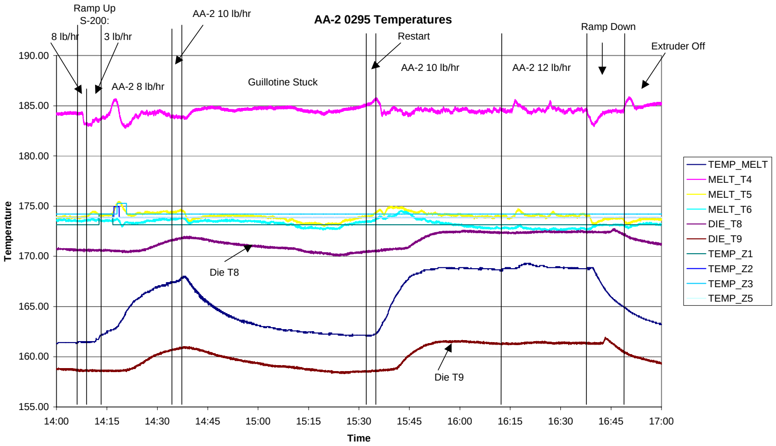
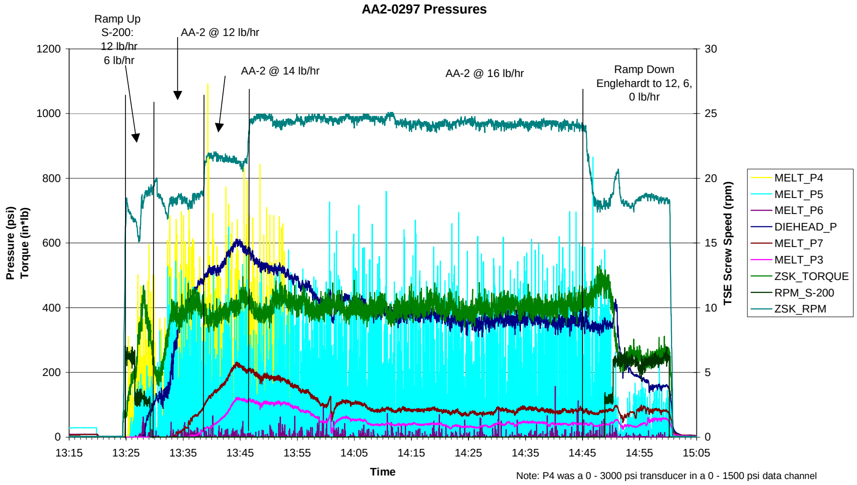


Figure 3-25. Temperature Trace,
IHDIV/NSWC Lot No. IH230-05F-AA2-0295 (Run 2)

The engineers had learned that reducing the ramp-up time would produce reasonably high pressure readings that produced better quality extrusions. They also now knew that the two-zone temperature control of the die was successfully cooling the grain and preventing the grain deformation. They also knew that the guillotine cutter was in need of additional work if it was going to be reliably used. The campaign's third run was planned for two days later.

The objective for Run 3 was to increase the steady state die head pressure by increasing the feed rate and screw speed. The temperature set points were lowered slightly to 170 °F. The shortened ramp-up time was utilized and the initial AA-2 pellet feed rate was set at 12 lb/hr. Since the torque and pressure levels remained low, the feed rate and screw speed were increased in two steps to 16 lb/hr and 24 rpm, respectively (see Figure 3-26).



Note: P4 was a 0 - 3000 psi transducer in a 0 - 1500 psi data channel

Figure 3-26. Pressure, Speed, and Torque Trace, IHDIV/NSWC Lot No. IH230-05F-AA2-0297 (Run 3)

The process had reached steady state and would remain there for over an hour. From a pressure and torque perspective, this was the most well controlled extrusion thus far in the project. Unfortunately, the grain quality was far from acceptable. The propellant grain hooked sharply to the right as it exited the die and did not consolidate well. There were longitudinal cracks visible along the grain's entire length. The propellant was not coming back together after flowing around the stake support legs. The temperatures of each barrel/die zone were well controlled. The die temperatures were increased about 30 minutes into the steady state extrusion in an attempt to remedy the consolidation problem but it did not resolve the problem.

Earlier in the project, the IHDIV/NSWC engineers had requested that Dr. Kalyon investigate the propellant consolidation problem. His analysis revealed that of the three variables known to affect consolidation—temperature, residence time in the die, and pressure—the most important was temperature. His report stated that the AA-2 propellant should reconsolidate well at temperatures near 220 °F. This temperature was much higher than the highest temperature (190 °F) deemed safe by the engineering staff. It was becoming evident that using the new die at the temperatures and pressures attempted thus far was unlikely to produce an acceptable grain. The campaign was halted and the engineering staff considered what changes to make before executing another extrusion run. It would be two more months before they would make their final attempt.

– Live AA-2 Extrusion Campaign #5, One Run, 24 August 2005

The final extrusion campaign was an attempt to replicate the reasonably successful extrusions accomplished during Campaign #4, Run 2 (C4R2) when a well consolidated grain was produced. The only readily apparent anomaly with that grain was its slightly small diameter. The engineers had requested and received approval for an increase in the allowable run time from 3 hours to 10 hours. The guillotine cutter had been repaired and was working well. The processing parameters would be the same as C4R2 (IHDIV/NSWC Lot No. IH230-05F-AA2-0295) as shown above in Table 3-6. Due to the proposed run length, the TSE refill hoppers were loaded with AA-2 pellets so that as the feeder emptied it could be automatically refilled with pellets. This run was given IHDIV/NSWC Lot No. IH230-05H-AA2-0388.

After the initial ramp-up, the AA-2 pellet feed rate was 10 lb/hr and the screw speed was 15 rpm, identical to C4R2. The process responded in a nearly identical fashion with the die head pressure increasing rapidly to nearly 1,700 psi and the torque increasing to 650 in-lb. The die head pressure then declined steadily to 400 psi, while the torque decreased erratically to approximately 350 in-lb. See Figure 3-27 for the pressure, screw speed, and torque trace.

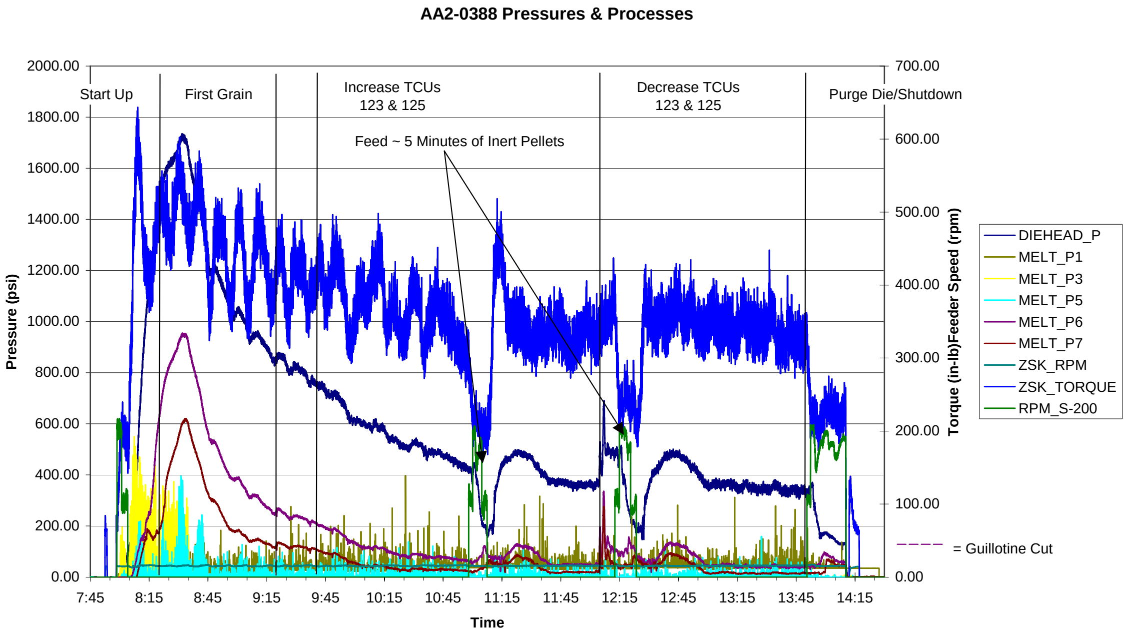


Figure 3-27. Pressure, Speed, and Torque Trace, IHDIV/NSWC Lot No. IH230-05H-AA2-0388 (Run 1)

The first grain was extruded within the 80 minutes and the guillotine cutter separated it with no problem. It had longitudinal cracks similar to those seen during the previous run. The temperature controller maintaining the die temperature (TEMP_Z5) and one of the barrel zones (TEMP_Z3) was increased in order to remedy the consolidation problem (see Figure 3-28).

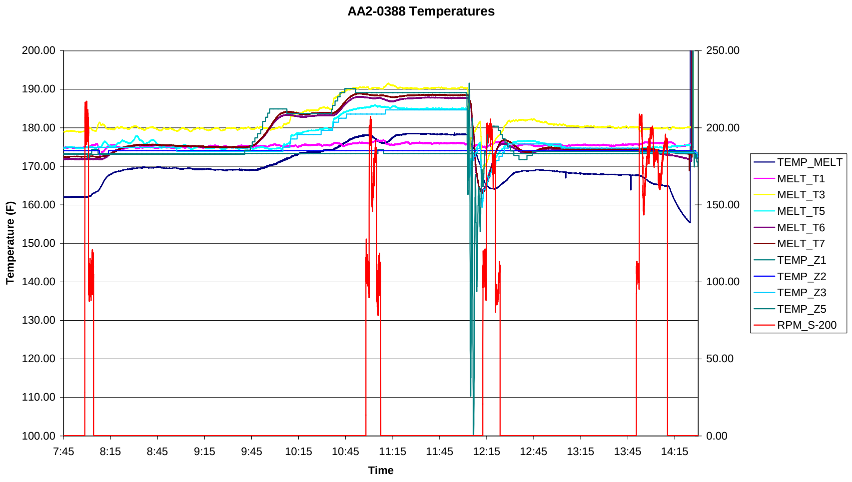


Figure 3-28. Temperature Trace
IHDIV/NSWC Lot No. IH230-05H-AA2-0388 (Run 1)

The temperature increases did not resolve the consolidation problems and the extruded propellant began to hook as it exited the die. After about 3 hours, it was noted that the propellant was not filling the annular space between the die and stake. In order to remedy this problem, inert pellets were fed into the extruder. After restarting the live pellets, it appeared that the crack had healed. However, the extrudate again began to hook as it exited the die and a crack appeared. A variety of conditions were attempted: the temperature controllers were set to their original position (see Figure 3-28) and inert pellets were again fed into the extruder in an attempt to increase the die head pressure. While this temporarily improved matters, hairline fractures and weld lines became apparent. After 6 hours of operation attempting to produce acceptable grains, the extruder was purged with inert pellets and shut down.

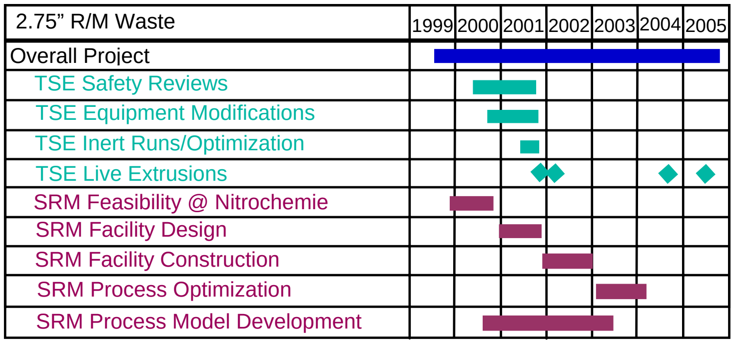
Conclusions: The work completed lead Indian Head personnel to conclude that the die was not capable of producing an acceptable grain. The pressure was not sufficient to consolidate the grain after it passed by the legs of the spider. Our recommendations for continuing are:

- Return to the inert work and find a safe method to increase the screw fill, i.e., increase Q*.
- The die must be redesigned to increase pressure and done so in a way to eliminate stagnation points.
- Improve the heating/cooling design of the water jacket.
- Implement the breaker plate.

3.6.1.3. Grain Finishing. The anticipated elimination of the annealing, saw-to-length and dowel rod operations was not investigated during this demonstration. There were no acceptable grains extruded from the TSE so there was no need to process a grain through the modified grain finishing process.

3.6.2. Period of Operation. The demonstration’s actual sequence of events is presented in Table 3-10. The long period between TSE live extrusions was caused by TSE equipment problems. The two main problems were the repair and reinstallation of the vibratory feeder and the new die design.

Table 3-10. 2.75-Inch Rocket Motor Waste Minimization Project Actual Schedule



3.6.3 Amount/Treatment Rate of Material to be Treated. The amount of AA-2 propellant processed at each location is provided in Table 3-11. The propellant form (paste, pellets, sheetstock, or grains) is also indicated.

Table 3-11. Quantities of Material Processes Using SRM Process

Location	Pellets or Sheetstock Produced[1] (lb)	Grains Produced (ea)	Grains Tested (ea)
Nitrochemie	1710	N/A	N/A
RAAP	200	50[2]	22[2]
IHDIV/NSWC	N/A	2	0

[1] Sheetstock manufactured from pellets. [2] Manufactured from Nitrochemie pellets.

3.6.4 Operating Parameters for the Technology. The 200-mm SRM at RAAP was put through an extensive optimization study under an ARDEC-sponsored contract. The results of the study are provided in Table 3-12. These results, when used in conjunction with the SRM process model developed by SIT, form the technical basis for determining the start-up settings for the 300-mm production-scale unit once it is installed.

Table 3-12. 200-mm SRM Adjusted Parameters

Operating Parameter	ATK Processing Range	Optimal Value
Paste moisture content (wt. %)	16.25–17.18	16.7
Paste feed rate (lbs/kg. per hr)	60–100/27–45	80/36
Temp. front roll feed (°F/°C)	165–205/74–96	185/85
Temp. front roll discharge (°F/°C)	145–185/63–85	165/74
Temp. back roll feed (°F/°C)	145–185/63–85	165/74
Temp. back roll discharge (°F/°C)	130–165/54–74	150/66
Roller RPM (front/back)	26–40/22–36	33/29
Roller RPM difference	4	4
Feed end roller gap (in/mm)	0.021–0.039/0.5–1.0	0.025/0.6

The TSE optimization work did not progress as well. This was due to several equipment problems and a nonexplosive incident involving screw-to-screw contact. However, the IHDIV/NSWC engineers did lay the groundwork for future optimization studies. Their results are shown in Table 3-13.

Table 3-13. 40-mm TSE Adjusted Parameters

Operating Parameter	IHDIV/NSWC Initial Settings or Recorded Range	Optimal Setting or Range
Pellet feed rate (lb/kg per hour)	10–13/4–6	Not found
Screw length-to-diameter ratio	12	12
Screw RPM	15–19	Not found
Temp. in zone 1–4 (°F/°C)	175–180/79–83	Not found
Die temperature (°F/°C)	168–180/76–83	Not found
Extrusion pressure range (psi/kp)	450–1635/3100–11270	Not found
Torque (ft-lb/n-m)	175–270/237–366	Not found

3.6.5 Experimental Design. The intended experimental design that was to be executed in support of this demonstration was not completed, in that the data that would have been used to validate the estimated reductions in waste streams and labor savings was not collected. The technical teams at both the SRM and TSE facilities worked diligently to bring their respective processes on-line so that the data could be collected. The SRM team at RAAP was able to optimize their process but the project ended before they could operate their equipment continuously for a period of time sufficient for the collection of validation data. The TSE team at IHDIV/NSWC was not able to optimize their process or produce any acceptable Mk 90 propellant grains so any data collected would not have accurately reflected the waste emission or labor reduction capabilities of the process.

It should be noted that although validation data were not collected at the 200 mm SRM facility, an extensive amount of processing data were collected. All of the parameters shown in Table 3-12 were adjusted carefully in an extensive process variable study. The optimal values shown are the result of this study. Additional process variable work was completed by SIT experts, under the direction of ARDEC personnel, who worked closely with Nitrochemie and ATK engineers during the development of the SRM process model. In addition to these successes, the ATK personnel at RAAP successfully converted Nitrochemie pellets into Mk 90 propellant grains using the legacy process and tested them to the applicable specification. The promising test results are discussed in detail below, but in general they provide substantial support to the argument that the SRM can produce pellets that can subsequently be extruded into acceptable propellant grains.

3.6.6 Product Testing. The product testing performed in support of this demonstration primarily evaluated the quality of both Nitrochemie and ATK AA-2 pellets and the Mk 90 grains produced from the pellets using the legacy rolling and extrusion equipment.

3.6.6.1 Pellet Testing. The first pellet testing that made a direct comparison between rolled sheetstock and SRM-produced pellets was performed by Nitrochemie. They found that the pellets they produced from ATK-supplied paste were nearly chemically identical to sheetstock. When Nitrochemie delivered pellets to RAAP, ATK performed their own testing. They tested the pellets and sheetstock made from the same paste lot in accordance with AS 2543, the controlling specification for sheetstock. The summarized results are provided in Table 3-14.

Table 3-14. Test Results—Sheetstock and Nitrochemie Pellet Analysis

Test Description	RAAP Sheetstock	Nitrochemie Pellets (Average of 8 batches)
% Nitrocellulose (NC)	52.52	53.24
% Nitroglycerin (NG)	37.35	36.98
% Triacetin (TA)	2.44	2.27
% Dinitrophenylamine (DNPA)	2.15	1.97
% 2-nitrodiphenylamine (2-NDPA)	2.02	1.91
% LC12-15	3.44	3.55
% Wax	0.08	0.07
% Water	0.36	0.73
Absolute density (g/ml)	N/A	1.60
Heat of explosion (HOE, cal/g)	994.9	992.1

These test results show that the SRM can produce AA-2 pellets with chemical properties that are very similar to sheetstock. The only results that indicate a significant difference is the percent water test. The pellets have twice the water content of the sheetstock which is not surprising since the propellant has a much shorter residence time on the heated SRM rolls than the propellant has on the various rolls that make up the legacy sheetstock rolling process. This difference can be remedied by adjusting key SRM processing parameters. Increasing roll temperature or reducing feed rate can lower the final water percentage. Overall, these initial results were very promising.

In early 2004, the SRM process optimization study funded by ARDEC provided more insight into the SRM's ability to produce acceptable propellant. The study consisted of nine processing runs setup in the matrix shown in Table 3-15. The shaded entries represent those variables that were adjusted during that specific process run. Using this methodology, each process parameter's affect on pellet quality, SRM throughput, and safety were evaluated. The pellets resulting from these runs were chemically tested. The weight percent of water, the only variable showing a marked difference from sheetstock made during the Nitrochemie process runs, varied considerably and did so in predictable fashion. The run information and moisture test results are provided in Table 3-16.

Table 3-15. SRM Process Optimization Study

	Paste Feed	Gap		RPM		Temperature			
Run	(Lb/Hr)	Feed End (In)	Discharge End (In)	Roll 1	Roll 2	Roll 1 °F Feed	Roll 1 °F Discharge	Roll 2 °F Feed	Roll 2 °F Discharge
1 - Baseline	80	0.025	0.030	33	29	185	165	165	150
2	60	0.025	0.030	33	29	185	165	165	150
3	100	0.025	0.030	33	29	185	165	165	150
4	80	0.021	0.023	33	29	185	165	165	150
5	80	0.039	0.042	33	29	185	165	165	150
6	80	0.025	0.030	26	22	185	165	165	150
7	80	0.025	0.030	40	36	185	165	165	150
8	80	0.025	0.030	33	29	205	185	185	165
9	80	0.025	0.030	33	29	165	145	145	130

Table 3-16. Pellet Moisture Content Summary

Run No.	Parameter Adjusted	Pellet Moisture Content (%)
1	Baseline	1.00
2	Reduced feed rate	0.56
3	Increased feed rate	1.55
4	Reduced roller gap	1.04
5	Increased roller gap	0.87
6	Reduced roller RPM	1.30
7	Increased roller RPM	0.91
8	Increased roll temperature	0.56
9	Decreased roll temperature	2.30

Note that runs 2 and 8 had reduced moisture content. The reduced feed rate in run 2 allowed for a longer propellant residence time on the rolls and more time for water to evaporate. The increased temperature in run 8 imparted additional energy to the propellant and drove the moisture down. The RAAP engineers felt that all of the runs except for run 9 were satisfactory, with the baseline run and run 8 producing the most pellets per unit time. They also felt that until the TSE was operational and fed with pellets made with different processing conditions, no firm conclusions could be drawn. The TSE would not become operational during this demonstration so the process study ended with an expanded knowledge of propellant moisture control and an enhanced understanding of those conditions that maximized output.

3.6.6.2. Propellant Grain Testing. The DEM/VAL plan stated that a demonstration objective was to produce Mk 90 propellant grains using Nitrochemie pellets that were rolled into sheetstock using a portion of the legacy rolling capability at RAAP. This objective was completed in October 2001 with the firing of Mk 90 propellant grains made from sheetstock produced using the entire legacy process and the SRM-produced pellets as an intermediate. The results, Figures 3-29 and 3-30, and Tables 3-17 through 3-19, show that carpet-rolled sheetstock made from pellets produces propellant grains that perform nearly identically to those produced in the conventional manner.

The first two graphs show the propellant strand burning rate for both sheetstock types as a function of pressure. Figure 3-29 provides results for high temperature tests, while Figure 3-30 provides low temperature results. The plots for each propellant type are very closely aligned and both meet the propellant specification requirements.

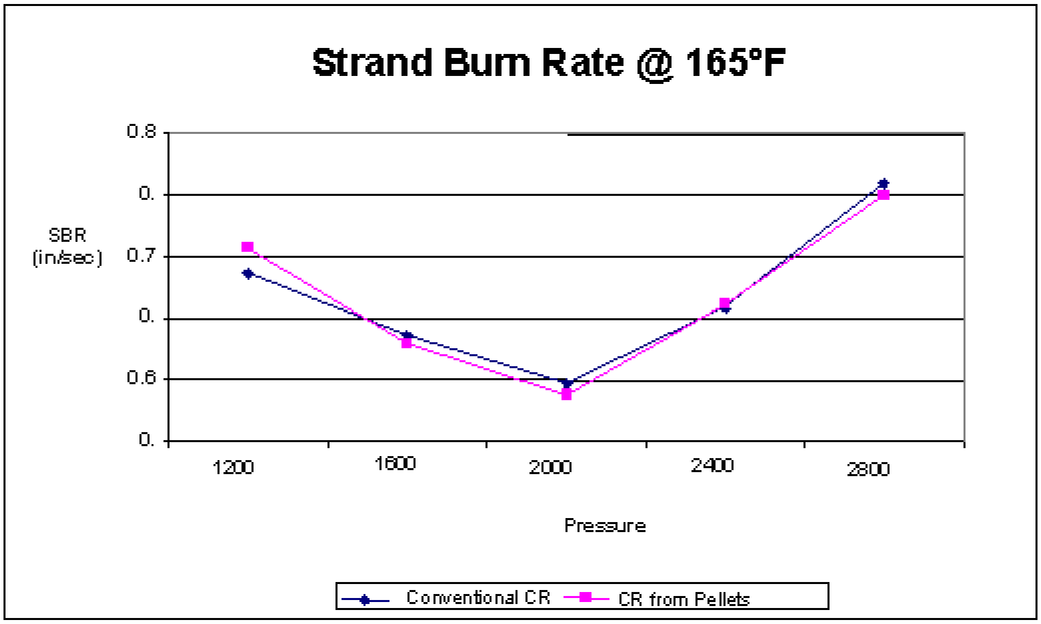


Figure 3-29. Strand Burning Rate at 165°F

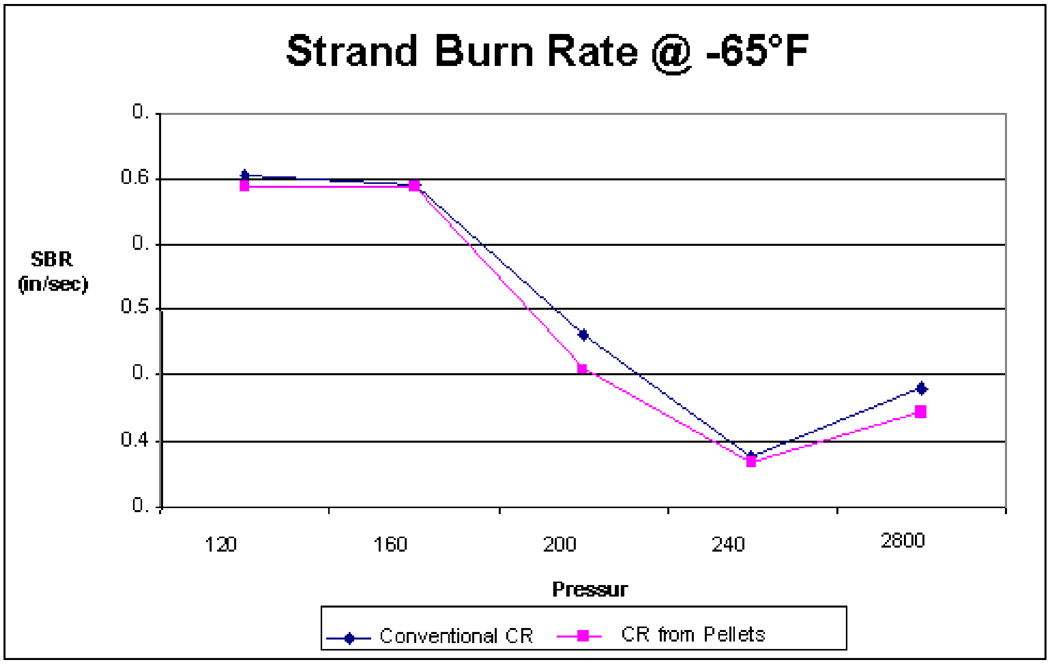


Figure 3-30. Strand Burning Rate at -65 °F

Table 3-17. Chemical Analysis Results for Conventional and SRM CR

Ingredient	Conventional CR	SRM CR
% NC	51.82	51.17
% NG	38.33	38.48
% TA	2.35	2.37
% DNPA	2.12	2.10
% 2-NDPA	2.01	1.99
% LC-12-15	3.33	3.59

Table 3-18. Average Grain Dimensions for Conventional and SRM CR

Measurements	Conventional CR	SRM CR
Length (in)	31.379	31.380
OD (in)	2.568	2.568
ID (in)	1.270	1.272
Aft web (in)	0.658	0.656
Forward web (in)	0.655	0.652
Weight (lb)	7.600	7.600

Table 3-19. Mk 90 Propellant Grains Produced Using Conventional CR and SRM CR–Ballistic Results

	Cold Motors @-50°F			Hot Motors @ +150°F		
	Normal Production	SRM		Normal Production	SRM	
Parameter		Normal	Pellets		Normal	Pellets
Action Time (sec)	1.164	1.139	1.148	1.026	1.032	1.025
Impulse (lbf-sec)	1490	1480.3	1480.2	1538	1527.6	1528.5
Max Thrust (lbf)	1650	1651.5	1650.8	1951	1879.5	1901.7
Spec. Impulse (lbf-sec/lb)	213.3	213.0	213.0	220.2	219.7	219.9

A chemical analysis of the propellant yielded similarly identical results as shown in Table 3-17. The average values for both types are provided. After both types of sheetstock were extruded from a conventional 15-in hydraulic ram press, the key grain dimensions were measured and recorded. The averages for both are provided in Table 3-18.

Table 3-18 shows that if the proper extrusion parameters are found, sheetstock manufactured using the SRM can produce grains that are dimensionally identical to the conventional process. The data also suggest that if the appropriate operating conditions can be found at the TSE facility, acceptable grains can be extruded. Both extrusion processes are simply physical transformations with the colloiding having occurred at the SRM. A redesigned die and further adjustments to the TSE feed rate and temperature setting should result in successfully extruded grains.

The last test performed was a set of actual grain firings using Mk 90 grains manufactured using both types of sheetstock and loaded into Mk 66 rocket motor hardware. The average ballistic performance of each at the performance specification's extreme temperature requirements is provided in Table 3-19. The normal production values are also provided for comparison.

The performance of the SRM sheetstock is similar to both the conventional sheetstock used in this study and the normal production values. The SRM values for impulse, the total energy delivered to the rocket motor, are within 0.6 percent of normal production value, while the specific impulse values, the energy provided per unit propellant weight, are within 0.14 percent of the same production values. This relationship holds for both hot and cold motor firings. This provides still further evidence that the SRM sheetstock can produce Mk 90 propellant grains, and subsequently Mk 66 rocket motors, that meet the specification requirement.

Although all of testing on TSE-manufactured grains envisioned by the DEM/VAL plan was not completed, the test results shown above demonstrate that the SRM pellet production process does produce propellant that, when conventionally extruded into propellant grains, can meet the chemical, physical, and ballistic specification requirements. The SRM has demonstrated great promise as an advanced technological replacement for the legacy process described in Section 2.1.

3.6.7. Demobilization

3.6.7.1. TSE. The IHDIV/NSWC TSE facility is very versatile, which meant that it was reconfigured after each AA-2 pellet extrusion to perform another process development task in other technology areas. The AA-2 propellant die was removed from the die holder, preserved and stored. The screws were removed, cleaned, and reconfigured for the next project. The pellets will remain in storage until it is determined if there will be additional TSE AA-2 extrusion development work. If not, the pellets will either be returned to RAAP or disposed of by burning. The tray feeder will remain available for use on this and other projects.

3.6.7.2. SRM. The SRM facility at RAAP remains in ready condition to perform further process development work. The 200-mm unit has always been considered a pilot-scale and will continue in this role. It will be used, in conjunction with the process study results and the SRM process modeling software developed under ARDEC direction, to improve ATK understanding of SRM processing and further refine operating parameters. This information will be used to design and start-up the 300-mm production-scale facility that is planned for installation at RAAP.

3.7. Selection of Analytical/Testing Methods

3.7.1. Selection of Analytical Method. Tests were carried out on the paste, pellets, and conventionally extruded grains. All tests are standard tests that are performed in conformance with established specifications or methods. The tests were chosen to either determine the acceptability of the grain for firing or to begin to develop a baseline to determine the necessary process controls for the advanced process.

3.7.1.1. Chemical Composition. This test was performed on both the pellets and extruded grains. The chemical composition of the pellets was compared to the requirements for AA-2 carpet roll, Product Specification AS 2543. The chemical composition of the propellant grains is for informational purposes. The procedures for the chemical analysis are listed in Table 3-20.

Table 3-20. Test Methods for the Chemical Composition of AA-2

Material	Method number of MIL-STD-286
Nitrocellulose	209.2.1
Nitroglycerin	208.1.3
2-Nitrodiphenylamine	218.4.3
Candellila Wax	T228.1
LC-12-15	T316.1
Triacetin	226.2
Di-n-propyl adipate	226.2

3.7.1.2. Heat of Explosion. The HOE provides an indication of the composition of the propellant. The ballistic modifier, LC-12-15, has a significant effect on the HOE. A weighed sample is burned in a bomb to measure the calorific value of the exothermic reaction. The procedure used is Method 802.1 of MIL-STD-286, except that pre-purified gas is used. This test was performed on both the pellets and extruded grains. The HOE of the pellets will be compared to the requirements for AA-2 carpet roll, Product Specification AS 2543.

3.7.1.3. Total Volatiles. The total volatiles content (percent moisture) was determined using Method 103.5.2 of MIL-STD-286. This test was performed on the pellets. The total volatiles of the pellets were compared to the requirements for AA-2 carpet roll, Product Specification AS 2543.

3.7.1.4. Strand Burning Rate. The strand burning rate was conducted in accordance with MIL-STD-286, Method T803.1 or OD 9376. The results were compared to the requirements for

AA-2 carpet roll, Product Specification AS 2543. Pellets were extruded into 1/4-in (nominal) strands prior to testing. Strands were cut from the propellant grain to determine the burning rate of the grain. However, differences in sample preparation may influence the results; thus, the test results are for information only.

3.7.1.5. Taliani Stability. This test is used to determine the stability of double-base propellants. The dried propellant is subjected to a temperature of 110 °C under a nitrogen atmosphere and the rate of gas evolution is measured. Different propellants have different normal gassing rates; this prevents using the raw data to determine various degrees of stability or chemical degradation. However, the relative stability of different samples of a given propellant can be determined. When nitrogen is used in the Taliani test, the presence of impurities in the standard composition may be detected. The data are reported as time in minutes to reach 100-mm pressure, slope of the pressure-time curve at 100-mm pressure, or slope of the pressure-time curve at 100 minutes. The tests were conducted in accordance with MIL-STD-286, Method 406.1.3. The stability of the pellets was compared to the requirements for AA-2 carpet roll, Product Specification AS 2543.

3.7.1.6. Propellant Density. The propellant density provides an indication of the quality of the propellant sample. An air pycnometer was used to determine the propellant density as described in Method 510.3.1 of MIL-STD-286. This test was performed on the pellets. This test was performed on both the pellets and extruded grains. The results were compared to the requirements for AA-2 carpet roll, Product Specification AS 2543.

3.7.1.7. Dimensional Analysis of Grains. The following dimensions of the extruded grains were measured in accordance with Drawing 233AS142 using a micrometer at 70 ± 5 °F: grain length, grain outer diameter, web thickness, and major inner diameter. After finishing, these dimensions were measured in accordance with Drawing 223AS106 at 70 ± 5 °F.

3.7.1.8. Static Firing. The static firings of the 2.75-inch propellant grain were conducted in a Mk 66 Mod 4 Static Fire Assembly. The ballistics were determined by firing a number of grains at two temperatures (–50 and 150 °F). From this testing the action time, impulse, and maximum thrust were determined. The data were compared to the performance requirements outlined in the Mk 90 Mod 0 Propellant Grain Product Specification, AS 2544L.

3.7.1.9. Internal Inspection. All billets and finished grains will be radioscopically inspected to ensure that they do not contain unacceptable internal defects. The results will be compared to Mk 90 Product Specification AS 2544.

3.8 Selection of Analytical/Testing Methods

All testing will be performed by ATK and/or IHDIV/NSWC, at their respective sites. The analytic, radiographic, and ballistic testing capabilities at both facilities are excellent. The laboratory personnel at each location are especially skilled at performing the tests listed in Section 3.7.1.

4. Performance Assessment

4.1. Performance Criteria

The criteria that were used to evaluate the performance of the advanced Mk 90 propellant grain manufacturing process are provided in Table 4-1. Each of the criteria are designated a primary or secondary criteria. Primary criteria are directly related to the project's original key objectives.

Table 4-1. SRM and TSE Performance Criteria

Performance Criteria	Description	Primary or Secondary
Process waste	1. Reduce NG emissions 14.3% 2. Reduce waste water amount by 63% 3. Reduce propellant waste by 52.8% 4. Reduce lead-contaminated ash by 23.1%	Primary
Labor costs	1. Reduce unit direct labor costs by 28.1%	Primary
Product testing	1. Completed Mk 90 grains must meet the requirements in AS 2544L. 2. SRM carpet roll propellant (AA-2) must meet the requirements in AS 2543. 3. Pellets (AA-2) were tested to many of the requirements listed in AS 2543 for comparison.	Primary
Factors affecting SRM technology performance	1. Solid composition of paste–Affects energy content, burning rate, and effectiveness of selected SRM operating parameters. 2. Moisture content of paste–Affects effectiveness of selected SRM operating parameters and moisture content of pellets. 3. Homogeneity of paste–Affects feed rate control of paste and uniformity of pellet composition. 4. Distribution of LC-12-15 ballistic modifier–Distribution of modifier has a likely effect on propellant burning rate. 5. Feed rate of paste to SRM–Affects effectiveness of selected SRM operating parameters and moisture content of pellets. Must be experimentally optimized.	Secondary

Performance Criteria	Description	Primary or Secondary
Factors affecting SRM technology performance (continued)	6. Temperature of processing zones on SRM rollers–Affects effectiveness of selected SRM operating parameters, specific work input, and moisture content of pellets. Must be experimentally optimized. 7. Processing length of SRM–Affects effectiveness of selected SRM operating parameters, specific work input, and moisture content of pellets. Must be experimentally optimized. 8. RPM of SRM rollers–Affects effectiveness of selected SRM operating parameters, specific work input, and moisture content of pellets. Must be experimentally optimized. 9. RPM difference between SRM rollers–Affects effectiveness of selected SRM operating parameters, specific work input, and moisture content of pellets. Must be experimentally optimized. 10. Gap between rollers–Affects effectiveness of selected SRM operating parameters, specific work input, moisture content of pellets, and pellet dimensions. Must be experimentally optimized. 11. Final moisture content of discharged pellet–Indicative of specific work input on SRM, used for process control of feed rates, i.e., too low a moisture content: danger of fire; too high a moisture content: insufficient work input. Has a significant effect on processing in TSE. Must be experimentally optimized. 12. Final absolute density of pellet–Indicative of specific work input and degree of colloiding obtained on SRM; near 100% theoretical maximum density (TMD) desired. Low densities may lead to low densities of TSE-extruded grain, and improper ballistic performance. 13. Bulk density of pellets–Function of absolute density and geometry: affects loss-in-weight (LIW) feeder selection and maximum throughput of TSE at a particular RPM/screw fill.	Secondary
Factors affecting TSE technology performance	1. Pellet delivery rate (lb/hr) 2. Extruder screw configuration 3. Barrel configuration 4. Screw speed (rpm) 5. Process temperature (can have multiple zones) 6. Extrusion die temperature	Secondary

Performance Criteria	Description	Primary or Secondary
	7. Extrusion pressure (process measurement) 8. Vacuum level (if required) 9. Die design 10. Torque (process measurement).	
Reliability	1. SRM–Exhibits high reliability, little mechanical maintenance required, some in process maintenance related to roller cleanliness and sharpening of granulating knife. 2. TSE–Many individual components, reliability increased through use of preventative maintenance plans, redundant components on hand if costs are reasonable.	Secondary
Ease of use	1. SRM–Continuously monitored, pilot process operated by 2–3 technicians or engineers, production process will require one lead and one trained operator, safety training is similar to legacy process. 2. TSE–Continuously monitored, pilot process operated by 2–3 engineers and technician, production process will require 2–3 technicians and one engineer, safety training is similar to legacy process.	Secondary
Versatility	1. SRM–Process can be used to manufacture pellets of different formulations after some optimization, quite versatile, could be set-up at alternate locations. 2. TSE–Process can extrude other double-base formulations after optimization, quite versatile, process could be used at other TSE sites after scale-up issues are resolved.	Secondary
Maintenance	1. SRM–Minor in-process maintenance required (roller cleaning) and regular bearing lubrication, preventative maintenance plan in place, no unusual training requirements. 2. TSE–Complex multi-component machine, maintenance plans for each component, maintenance based on hours of use, technicians and skilled trades personnel perform maintenance.	Secondary

Performance Criteria	Description	Primary or Secondary
SRM scale-up constraints	Scaling from 200-mm diameter rolls to 300-mm production rolls will require thorough understanding of total work utilized to create acceptable pellets and matching that work input on larger rolls. The SRM mathematical model developed by ARDEC/SIT will aid in this understanding.	Secondary
TSE scale-up constraints	In order to scale a TSE process, it is necessary to create the same mass transfer conditions in both the sub-scale and target extruders. Mathematical models of the die and extruder will aid in the scale up process. Practically, the key issues are matching shear rate and mechanical power input to the product. The following items should be followed when scaling between a lab machine and the larger scale unit: o *Process Length over Diameter (L/D)*–The L/D ratio of the barrel arrangement and the screw design of each process operation as well as the overall L/D should be matched as closely as possible between the different extruders. o S_{mech}–This is a measure of the power input per unit mass of material. For a fixed screw design, this value varies with the screw speed and throughput. o *Available Power*–The motor, gearbox, and shafts of the large-scale machine must be able to deliver the power and torque to the process section needed at the rates predicted by the scale-up calculations. o *Degree of Fill*–The large-scale machine should generate the same average degree of fill as that of the small-scale o *Shear Rate*–This parameter is critical to mixing intensity. The average shear rate is proportional to the extruder screw speed. Each machine has its own shear rate constant. The total shear input is, of course, dependent on the screw design.	Secondary

4.2. Performance Confirmation Methods

A summary of the demonstration results and the effectiveness of the advanced Mk 90 propellant grain manufacturing process are provided in Table 4-2. The data analysis procedures, baseline comparison protocols, and the data collection procedures are described in the *Discussion* section for each criterion.

Table 4-2. Performance Results

Performance Criteria	**Expected Performance Metric**	**Performance Confirmation Method**	**Actual Performance**
PRIMARY CRITERIA - Quantitative			
Process Waste 1. Reduce NG emissions 14.3% 2. Reduce waste water amount by 63% 3. Reduce propellant waste by 52.8% 4. Reduce lead-contaminated ash by 23.1%	1. Reduction goal obtained 2. Reduction goal obtained 3. Reduction goal obtained 4. Reduction goal obtained	Constituent weights recorded on SRM and TSE data collection sheets, waste amounts calculated from this data	SRM–No validation data supporting the waste amount calculation was collected TSE–No validation data supporting the waste amount calculation was collected, a representative amount of material was never processed
Discussion: SRM–The RAAP technical staff was focused on optimizing the SRM process and did not have ample opportunity to collect the data necessary to validate the waste reduction objective. TSE–There were several technical issues that were not resolved at the TSE facility. These issues surrounded both the die design and the operating parameters at which to process the AA-2 pellets. No acceptable grain was extruded and only a relatively small amount of propellant was actually processed which meant no statistically significant data could be collected. Hence, the waste reduction goals were not validated empirically.			
Labor Costs Reduce unit direct labor costs by 28.1%	Reduction goal obtained	Processing amounts recorded on data collection sheets, man-hour records maintained using standard time collection means	SRM-See Table 5-4 TSE–No validation data supporting the labor reduction calculation was collected, a representative amount of material was never processed
Discussion: SRM–The RAAP technical staff was focused on optimizing the SRM process and did not have ample opportunity to collect the data necessary to validate the waste reduction objectives. TSE–There were several technical issues that were not resolved at the TSE facility. These issues surrounded both the die design and the operating parameters at which to process the AA-2 pellets. No acceptable grain was extruded and only a relatively small amount of propellant was actually processed which meant no statistically significant data could be collected. Hence, the labor saving goals were not validated empirically.			

Performance Criteria	Expected Performance Metric	Performance Confirmation Method	Actual Performance
SECONDARY CRITERIA - Quantitative			
Product Testing 1. Completed Mk 90 grains must meet the requirements in AS 2544L 2. SRM carpet roll propellant (AA-2) must meet the requirements in AS 2543 3. Pellets (AA-2) were tested to many of the requirements listed in AS 2543 for comparison	1. Specification requirements met 2. Specification requirements met 3. Pellets compare favorably to carpet roll in selected tests	1. Perform chemical, physical and ballistic tests in accordance with AS 2544L 2. Perform chemical and physical tests in accordance with AS 2543 3. Perform selected tests in accordance with AS 2543	1. No testing performed on TSE extruded grains 2. SRM carpet roll propellant **passed** the specified chemical and physical tests 3. SRM pellets **compared favorably** to conventional carpet roll in selected tests
Discussion: SRM (Carpet Roll and Pellets)–materials tested and passed selected tests, details provided in Section 3.7. TSE–No acceptable grains were extruded for the reasons discussed above so no TSE-extruded grains were tested.			
SECONDARY CRITERIA - Quantitative			
Factors Affecting SRM Technology Performance 1. Solid composition of paste–Affects energy content, burning rate, and effectiveness of selected SRM operating parameters. 2. Moisture content of paste–Affects effectiveness of selected SRM operating parameters and moisture content of pellets. 3. Homogeneity of paste–Affects feed rate control of paste and uniformity of pellet composition. 4. Distribution of LC-12-15 ballistic modifier–Distribution of modifier has a likely effect on propellant burning rate. 5. Feed rate of paste to SRM–Affects effectiveness of selected SRM operating parameters and moisture content of pellets. Must be experimentally optimized. 6. Temperature of processing zones on SRM rollers–Affects effectiveness of selected SRM operating parameters, specific work input, and moisture content of pellets. Must be experimentally optimized.	**1 through 4.** It is expected that some minor adjustments to the AA-2 formulation may be necessary to optimize the SRM performance. Any adjustments would be done after a thorough engineering assessment that is approved by all team members. All raw and intermediate materials should pass all applicable receipt inspection and in-process quality assurance provisions. **5 through 11.** These processing variables will be optimized during the project. It is expected that through engineering studies at Nitrochemie and ATK, in conjunction with the SRM process model developed by SIT, the optimum operating conditions for AA-2 will be determined. **12 through 15.** These finished pellet characteristics will be optimized along with the processing parameters. They will also be optimized in conjunction with TSE process variability study. They should be comparable to AA-2 propellant with respect to composition (dry).	**1 through 4.** These are controlled by ATK quality assurance activities, both receipt inspection and in-process. **5 through 11.** All processing variables will be recorded as the test runs are completed. Final results will be recorded for each run. A final report will be submitted with recommended AA-2 optimum operating conditions. The SRM process model will be completed by SIT and submitted to ARDEC for review. **12 through 15.** The pellets will tested to many of the same specification requirements as the carpet rolled AA-2 propellant (AS-2544). They will be tested in the same lab and results will be recorded. This data will be reported to all team members.	**1 through 4.** These paste characteristics were adequately controlled throughout the demonstration's processing runs. **5 through 11.** The optimization study was **completed**. The process model was **completed and accepted** by ARDEC. **12 through 15.** Pellets were tested to many of the AA-2 sheetstock requirements and **passed** those tests.

Performance Criteria	Expected Performance Metric	Performance Confirmation Method	Actual Performance
Factors affecting SRM technology performance (continued) 7. Processing length of SRM–Affects effectiveness of selected SRM operating parameters, specific work input, and moisture content of pellets. Must be experimentally optimized. 8. RPM difference between SRM rollers–Affects effectiveness of selected SRM operating parameters, specific work input, and moisture content of pellets. Must be experimentally optimized. 9. RPM of SRM rollers–Affects effectiveness of selected SRM operating parameters, specific work input, and moisture content of pellets. Must be experimentally optimized. 10. Gap between rollers–Affects effectiveness of selected SRM operating parameters, specific work input, moisture content of pellets, and pellet dimensions. Must be experimentally optimized. 11. Final moisture content of discharged pellet–Indicative of specific work input on SRM, used for process control of feed rates, i.e., too low a moisture content: danger of fire; too high a moisture content: insufficient work input. Has a significant effect on processing in TSE. Must be experimentally optimized. 12. Final absolute density of pellet–Indicative of specific work input and degree of colloiding obtained on SRM; near 100% theoretical maximum density (TMD) desired. Low densities may lead to low densities of TSE-extruded grain, and improper ballistic performance.			

<table>
<tr><th>Performance Criteria</th><th>Expected Performance Metric</th><th>Performance Confirmation Method</th><th>Actual Performance</th></tr>
<tr><td>Factors affecting SRM technology performance (cont.)
13. Bulk density of pellets–Function of absolute density and geometry: Affects (LIW) feeder selection and maximum throughput of TSE at a particular RPM/screw fill.</td><td></td><td></td><td></td></tr>
<tr><td colspan="4"><i>Discussion: The SRM process optimization study, funded by ARDEC, was completed by RAAP engineers after the SRM was installed. The process study details are provided in Section 3.7. This was an important step along the path to scaling up this process. In addition, the SRM Process Model and Software developed by SIT under ARDEC direction, will be useful during the scale-up process. This portion of the demonstration project was quite successful.</i></td></tr>
<tr><td>Factors affecting TSE technology performance
1. Pellet delivery rate (lb/hr)
2. Extruder screw configuration
3. Barrel configuration
4. Screw speed (rpm)
5. Process temperature (can have multiple zones)
6. Extrusion die temperature
7. Extrusion pressure (process measurement)
8. Vacuum level (if required)
9. Die design
10. Torque (process measurement)</td><td>These processing parameters will be optimized during a process variable study. An optimum set of processing conditions will be established for AA-2 propellant. These conditions will not only produce acceptable Mk 90 grains but will comfortably fit within the safe TSE operating limits.</td><td>The processing variables will be recorded for each run. Some are measured automatically by the TSE control system while others are documented by engineers and technicians.</td><td>A total of eight runs were performed without an optimized process being developed. No acceptable grain was extruded. However, progress was made towards optimization. The operating ranges utilized during the process runs are provided in Section 3.6.1.2.4.</td></tr>
<tr><td colspan="4"><i>Discussion: The inability to produce an acceptable grain was disappointing. Progress was being made towards an optimum process but problems related to the die design, along with delays caused by equipment malfunctions, prevented the project from progressing satisfactorily. There were several lessons learned that could be used to design an extrusion die that would produce an acceptable grain. Further manipulation of operating conditions would further augment a new die design. Once optimized, the data collected during consistent operation would be utilized to validate the waste reduction and cost saving estimates.</i></td></tr>
<tr><td>Reliability
1. SRM–Exhibits high reliability, little mechanical maintenance required, some in-process maintenance related to roller cleanliness and sharpening of granulating knife.
2. TSE–Many individual components, reliability increased through use of preventative maintenance plans, redundant components on hand if costs are reasonable.</td><td>1. SRM–Expecting this equipment to be very reliable due to its simplicity and heavy construction.
2. TSE–The screw/barrel assembly is expected to be very reliable. The supporting equipment such as feeders, cutters, and temperature control systems can be less reliable during start-ups.</td><td>1. SRM–Records of mechanical breakdown will be kept during the process variable study, any delays caused by mechanical breakdown will be clearly evident.
2. TSE–Records of mechanical breakdown will be kept during the process variable study, any delays caused by mechanical breakdown will be clearly evident</td><td>1. SRM–The equipment exhibited excellent reliability with no maintenance-related downtime reported.
2. TSE–The vibratory feeder purchased for this project did have reliability problems and actually caused a considerable delay (9 months) during its repair period. The water-jet cutter was never made operational. There was</td></tr>
</table>

Performance Criteria	Expected Performance Metric	Performance Confirmation Method	Actual Performance
			also an incident involving screw-to-screw contact which resulted in a long delay. Some reliability problems with guillotine grain cutter.
Discussion: SRM–This equipment exhibited excellent mechanical reliability as expected. TSE–The feeder problem was certainly the project's major reliability issue. It was determined after an investigation that the problem was a wiring error and the feeder proved to be quite reliable after the problem was resolved. The TSE facility was not run for the expected number hours so a true picture of its reliability can not be ascertained.			
Ease of use 1. SRM–Continuously monitored, pilot process operated by 2-3 technicians and engineer, production process will require one lead and one trained operator, safety training is similar to legacy process. 2. TSE–Continuously monitored, pilot process operated by 2–3 engineers and technician, production process will require two operators and one engineer, safety training is similar to legacy process.	1. SRM–Expecting this equipment to be run with this manpower level, reduction in manpower for production feasible. 2. TSE–Expecting equipment to be run with this manpower level, reduction in manpower feasible for production.	1. SRM–Pilot-scale run records, production-scale estimates. 2. TSE–Pilot-scale run records, production-scale estimates.	1. SRM–The 200-mm SRM was run effectively with an engineer and one operator. It is expected that the production-scale unit will be run with 1.5 operators and an engineer in an oversight role. 2. TSE–The TSE was operated by 2–3 engineers but was not run for a sufficient length of time to draw any conclusions about manning requirements.
Discussion: SRM–The manpower expectations were met. ATK expects that the move to the SRM will allow them to realize the majority of the labor and waste reduction advantages foreseen by the entire advanced process. TSE–No further comment.			
Versatility 1. SRM–Process can be used to manufacture pellets of different formulations after some optimization, quite versatile, could be set-up at alternate locations. 2. TSE–Process can extrude other double-base formulations after optimization, quite versatile, process could be used at other TSE sites after scale-up issues are resolved.	1. SRM–Demonstration only to evaluate AA-2 propellant, but Nitrochemie has processed several different double-base formulations on their equipment. 2. TSE–Demonstration only to evaluate AA-2 propellant, but Nitrochemie has processed several different double-base formulations on their equipment.	1. SRM–Process optimization study records will indicate the facility's ability to handle other propellant types. 2. TSE–Process optimization study records will indicate the facility's ability to handle other propellant types.	1. SRM–Process optimization study did verify that there was a wide range of acceptable operating conditions suggesting that similar double-base propellants could be processed. 2. TSE–Insufficient run times did not allow for a process assessment.
Discussion: SRM–In addition to the information given above, it should be noted that the SIT process model development effort collected data related to the processing of JA-2 and DIGL propellants at Nitrochemie. Both of these were being successfully produced on the 300-mm SRM. TSE–This facility has shown considerable versatility in the past having produced both LOVA and extrude composite propellant formulations. If further optimization studies are completed and succeed at developing a satisfactory die design and operating conditions, the versatility of this equipment will allow other formulations to be processed.			

<table>
<tr><th>Performance Criteria</th><th>Expected Performance Metric</th><th>Performance Confirmation Method</th><th>Actual Performance</th></tr>
<tr><td>Maintenance
1. SRM–Minor in-process maintenance required (roller cleaning) and regular bearing lubrication, preventative maintenance plan in place, no unusual training requirements.
2. TSE–Complex multi-component machine, maintenance plans for each component, maintenance based on hours of use, technicians and skilled trades personnel perform maintenance.</td><td>1. SRM–There should be no unusual maintenance concerns and no significant delays due to maintenance issues.
2. TSE–Same as above.</td><td>1. SRM–Process run sheets will indicate if there are any significant problems. Quarterly reports will highlight any long-term delays that result from maintenance problems.
2. TSE–Same as above.</td><td>1. SRM–The facility operated without any significant maintenance-related delays.
2. TSE–The facility had several significant maintenance-related delays to include problems with the vibratory feeder and the guillotine cutter.</td></tr>
<tr><td colspan="4"><i>Discussion: SRM–No additional information. TSE–The maintenance problems which arose had a serious effect on the demonstration project. The vibratory feeder problems delayed the project nine months. The guillotine cutter issues created smaller delays. In order to move this process to the production-scale, the maintenance issues with the TSE support equipment will need to be resolved.</i></td></tr>
<tr><td>SRM scale-up constraints
Scaling from 200-mm diameter rolls to 300-mm production rolls will require thorough understanding of total work utilized to create acceptable pellets and matching that work input on larger rolls. The SRM mathematical model developed by ARDEC/SIT will aid in this understanding.</td><td>The SRM process model and the SRM optimization study provide key information that enables a thorough understanding of the total work imparted to the paste.</td><td>Review the results of the model development and optimization study which will be documented in technical reports.</td><td>Both reports were submitted and accepted by the Government. They did provide much of the needed information related to scale-up.</td></tr>
<tr><td colspan="4"><i>Discussion: The SRM Process Model developed by SIT will be particularly useful when ATK moves ahead with their scale-up plans. One of the contract deliverables was process design software that allows the user to provide key process inputs, to include roll diameter, and paste characteristics which are then modeled by the software. The output provides temperature and throughput estimates. It will also warn that dangerous conditions may result from certain operating conditions. This user-friendly software will help in introducing new formulations to both the 200- and 300-mm SRM facilities.</i></td></tr>
<tr><td>TSE scale-up constraints
In order to scale a TSE process, it is necessary to create the same mass transfer conditions in both the sub-scale and target extruders. Mathematical models of the die and extruder will aid in the scale up process. Practically, the key issues are matching shear rate and mechanical power input to the product.</td><td>The process optimization work performed at IHDIV/NSWC and the die modeling performed at SIT will lay the groundwork for any future scale-up work.</td><td>The IHDIV/NSWC technical staff provides a technical paper related to their optimization work and SIT experts provide a report on the die modeling.</td><td>The TSE process was not optimized so no report was submitted. There was a report submitted by SIT which provided technical information that would be helpful in designing a die for a production-scale facility.</td></tr>
<tr><td colspan="4"><i>Discussion: Some inert process optimization was completed at the TSE facility but no formal report related to this work was submitted. IHDIV/NSWC has installed an 88-mm TSE facility but has yet to use it to extrude materials similar to AA-2. This research facility could prove quite useful to any future scale-up efforts at RAAP.</i></td></tr>
</table>

5. Cost Assessment

5.1 Cost Reporting

In accordance with Environmental Security Technology Certification Program (ESTCP) guidelines, a cost estimate for the production-scale implementation of the advance Mk 90 propellant grain process was developed. This work was performed by the engineering consulting firm BAH and was presented to the government on 27 March 2000. Their work was reviewed and validated by ARDEC personnel. However, the demonstration participants did not collect the necessary data required to validate or revise these estimates. The technical personnel at both IHDIV/NSWC and RAAP concentrated their efforts on facility modifications, equipment installations, and process development work. They did not collect the data required to support the detailed cost analysis outlined in the ESTCP Final Report Guidance. Some facility installation costs related to the 200-mm SRM and the overall budgeted expenditures for each demonstration participant were recorded. These will be provided in the following section.

5.2 Cost Analysis

5.2.1 Actual Demonstration Costs. The actual funds provided in support of the demonstration, on a per fiscal year basis, are provided in Table 5-1.

Table 5-1. Actual Funding Provided Per FY ($K)

Participant/FY	1999	2000	2001	2002	2003	2004	2005	Total
ESTCP	600	405	500	245	0	0	0	1750
ARDEC	0	500	750	0	0	0	0	1250
ATK	250	750	1600	650	0	0	0	3250
IHDIV/NSWC	0	0	0	0	40	75	170	285
2.75-In Rocket Motor Program Office	0	750	0	0	0	0	0	750
Total	850	2405	2850	895	40	75	170	7285

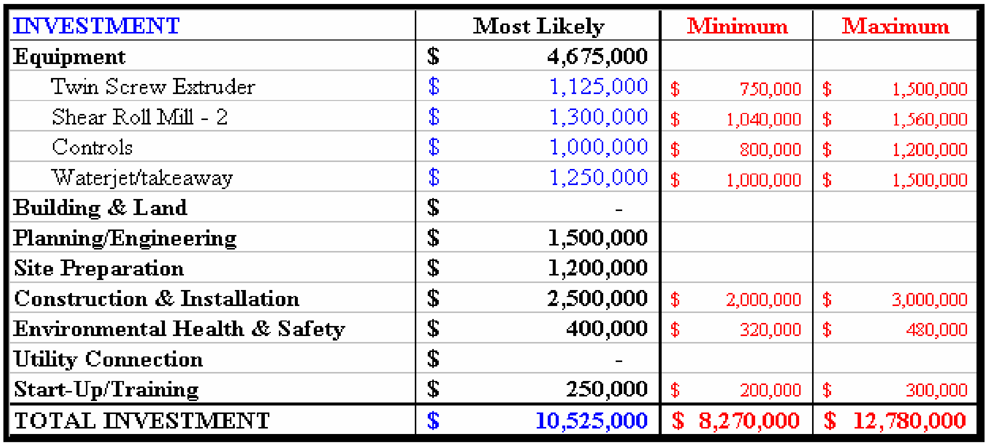
This demonstration was scheduled to be completed in early 2003 so the absence of funding in FY 2003–2005 is to be expected. This demonstration was an excellent example of resource sharing between the public and private sectors with the overall costs being shared almost equally.

The costs, in year 2000 dollars, to install the 200-mm SRM at RAAP are provided in Table 5-2 for comparison with the BAH summary further below. A summary of the original BAH work is provided in Tables 5-3 through 5-6. The cost basis for this analysis was 200,000 Mk 90 propellant grains per year. As stated earlier, ARDEC personnel validated these estimates in 2001. A summary of estimated start-up costs is provided in Table 5-3.

Table 5-2. Pilot-Scale SRM Facility Costs at RAAP

Project Phase	Cost ($K)
I. Design	180
II. Environmental Permitting	32
III. Equipment Removal	508
IV. SRM Refurbishment	326
V. SRM Installation	477
VI. Prove-out	74
Total	1597

Table 5-3. Costs to Start-Up Production-Scale Advanced Process



INVESTMENT	Most Likely	Minimum	Maximum
Equipment	**$ 4,675,000**		
Twin Screw Extruder	$ 1,125,000	$ 750,000	$ 1,500,000
Shear Roll Mill - 2	$ 1,300,000	$ 1,040,000	$ 1,560,000
Controls	$ 1,000,000	$ 800,000	$ 1,200,000
Waterjet/takeaway	$ 1,250,000	$ 1,000,000	$ 1,500,000
Building & Land	**$ -**		
Planning/Engineering	**$ 1,500,000**		
Site Preparation	**$ 1,200,000**		
Construction & Installation	**$ 2,500,000**	$ 2,000,000	$ 3,000,000
Environmental Health & Safety	**$ 400,000**	$ 320,000	$ 480,000
Utility Connection	**$ -**		
Start-Up/Training	**$ 250,000**	$ 200,000	$ 300,000
TOTAL INVESTMENT	**$ 10,525,000**	**$ 8,270,000**	**$ 12,780,000**

[Content containing proprietary information has been removed and is presented in Appendix A.]

Table 5-4. Direct Labor Cost Savings–Advanced Process

[Table 5-4 containing proprietary information has been removed and is presented in Appendix A.]

[Content containing proprietary information has been removed and is presented in Appendix A.]

Table 5-5. Annual Waste Treatment Costs for Conventional and Advanced Process

[Table 5-5 containing proprietary information has been removed and is presented in Appendix A.]

Table 5-6. Five-Year Cost Savings, Conventional Versus Advanced Process ($M)

[Table 5-6 containing proprietary information has been removed and is presented in Appendix A.]

[Content containing proprietary information has been removed and is presented in Appendix A.]

6. Implementation Issues

6.1 Environmental Permits

The regulatory environment governing the production of Mk 90 propellant and the subsequent treatment and disposal of waste materials was assessed in terms of organizational responsibilities, environmental regulations and requirements, and safety and health issues. Environmental, safety, and health (ESH) issues will be addressed based upon regulations and requirements established by:

- The Resource Conservation and Recovery Act
- The Clean Water Act
- The Clean Air Act
- The Emergency Planning and Community Right-to-Know Act
- The Safe Drinking Water Act
- The Pollution Prevention Act
- The Federal Facilities Compliance Act
- Presidential Executive Orders
- The Occupational Safety and Health Act
- DOD-specific requirements.

In order to move forward with this demonstration project, new environmental permits or modifications to existing permits were necessary. The SRM facility required a new permit to operate because it was a new process at RAAP. This permit was obtained with minor delays because the new Mk 90 Shear Roll Mill/Twin Screw Extruder production process has benefited from a history of close cooperation with federal, state, and local regulators. Beginning at the initial concept stage, RAAP's military and contractor personnel have partnered with authorities to ensure that they not only understand the engineering aspects of the process changes, but also recognize the positive ESH impacts that are expected to result from the use of the new equipment. As a result, the necessary environmental permit modifications were secured with minimal effort. Consultation and cooperation with authorities will continue as the Mk 90 Waste Minimization Program matures, and will continue throughout the effective life cycle of the new production equipment. This will serve to minimize the down time attributable to regulatory issues and ensure the ability of RAAP to produce Mk 90 propellant is not unduly impacted.

The introduction of double-base propellant and the accompanying NG emissions into the TSE facility did require a permit modification that was obtained by the IHDIV/NSWC Environmental Management Office (EMO). The EMO is staffed with personnel that understand propellant manufacturing processes, and have expertise in specific pollutant media. EMO personnel confer with production engineers and interact with regulators on a regular basis in order to ensure that the regulators have a clear understanding of energetic material production processes, associated ESH considerations, and the methods of environmental protection employed at IHDIV/NSWC. As a result, regulators are keenly aware of the fact that IHDIV/NSWC has initiated numerous projects that utilize pollution prevention technologies and is an industry leader in using pollution prevention as a means of complying with regulatory requirements.

6.2 Other Regulatory Issues

The installation of the production-scale SRM facility will require a new environmental permit to operate and the efforts to secure the permit have already begun. As stated above, the RAAP EMO has and continues to cultivate an excellent relationship with local, state and federal regulatory agencies. There should be no significant issues that delay the granting of the permit to operate. The regulators have been made aware of the environmental benefits of the new process and are looking to make the transition as smooth as possible.

If the permit acquisition process requires public participation, RAAP has numerous mechanisms at its disposal to encourage public participation and stakeholder involvement in various aspects of plant operations. Several are briefly discussed below.

The RAAP has been operational for approximately 60 years. The Environmental Protection Agency (EPA) has issued a RCRA Corrective Action Permit to address issues associated with past environmental contamination. An Installation Restoration Plan (IRP) has been developed to guide cleanup activities. As part of the IRP, a Restoration Advisory Board (RAB) has been formed. RABs are committees made up of community members, environmental regulators, local government representatives, installation representatives, and other interested parties. The RAB encourages community participation in the cleanup process and provides community members and other stakeholders the opportunity to have meaningful dialogue with facility environmental officials.

The RAAP is a member of the Local Emergency Planning Committee (LEPC). Under the Emergency Planning and Community Right-to-Know Act (EPCRA), facilities that store or manage certain hazardous or extremely hazardous substances in quantities greater than established threshold planning quantities are required to:

- Participate in the local emergency planning process
- Notify state and local emergency planning officials of the presence of hazardous substances above established thresholds
- Provide appropriate ESH data to state and local emergency planning officials
- Present the Risk Management Program to the public
- Report releases of such substances.

Additionally, facilities are required to notify the LEPC of changes that occur in facility operations such as the introduction of new processing techniques and equipment.

Under Section 112r of the Clean Air Act, RAAP is required to have a detailed Risk Management Plan (RMP) to minimize the potential consequences that may result from an accidental release of hazardous materials.

The RAAP aggressively implements the requirements of the National Environmental Policy Act (NEPA). NEPA requires federal agencies to evaluate the environmental consequences of proposed federal actions, to identify and assess reasonable alternatives to proposed actions in order to minimize or avoid adverse environmental effects, and to have procedures in place to ensure that relevant environmental information is made readily available to decision makers and the public before decisions are made and federal actions taken. ATK will conduct an NEPA review. If an environmental assessment is required, it will start within 30 days of start up of the SRM/TSE installation process. All NEPA documentation should be completed, through public comments as required, within 120 days of start up.

Public meetings are announced for both wastewater and hazardous waste permit modifications. These meetings are generally held off plant at locations convenient to the public. The meetings are announced in the “Legal Notices” sections of local newspapers and announced on local radio stations depending on the regulators’ guidelines. As discussed above, the permitting process results in close cooperation and interaction with federal, state, and local government officials. Both military and contractor personnel are always ready to meet with the public and local authorities on an as-needed basis to address community concerns as they may arise. The RAAP has public affairs offices with well-established procedures for distributing information related to plant operations and handling other public inquiries.

The IHDIV/NSWC will not be installing production-scale equipment so no new permits related to the transition of the advanced process are foreseen.

6.3 End-User Issues

In general, the end-users for this technology are procuring officers of any double-base propellant grain. Specifically, the end-user for this demonstration is the Mk 66 Program Office. The 2.75-Inch Rocket System acquisition strategy is to develop the lowest cost solution to meet the quality/reliability requirements set forth in the 2.75-Inch Rocket Motor Specification WS 33464B.

The Mk 66 rocket motor has had a history of intermittent combustion instability. Therefore, this continues to be a major concern. It has not been possible to directly correlate the instability problems with small-scale tests on the paste, carpet roll, or propellant grain. However, distribution of the ballistic modifiers in the propellant, and the amount of work introduced into the propellant grain during carpet roll manufacture and extrusion appear to be contributing factors. If the advanced process shows any inclination to exacerbate these problems it will not be readily supported. There were no test results that indicated this problem has worsened with the introduction of these new technologies.

Another issue is the development of acceptance criteria for the AA-2 pellets. The current AA-2 product specification requires that the material be provided in sheet or carpet roll form. While many of the tests can be conducted on the pellets (e.g., chemical composition, heat of explosion), others cannot without extruding the pellets to a different form. The sheetstock propellant specification AS 2543 requires *strand* burning rate testing and physical property testing on "dog" bones. Pellets can be extruded to the required shapes; however, the extrusion rate could affect the mechanical properties of the propellant. This, in turn, may affect both the burning rate and physical properties. Therefore, two alternate test methods are proposed: closed bomb testing for obtaining burning rate and a crush test to measure compressive strength. Sufficient testing will be conducted to begin to establish a database with which to modify the acceptance criteria for AA-2.

While it is important to establish acceptance criteria for the pellets, that alone is not sufficient to guarantee acceptable rocket motors. Processing parameters for producing carpet roll and finished grains greatly affect the quality of the rocket motors. Studies will be conducted to determine how the processing variables of the TSE and SRM affect the final propellant.

A final area of concern is stabilizer depletion in the pellets and finished grains. This will need to be addressed by accelerated aging tests to be performed in the future.

The ATK management at RAAP is committed to the installation of a production-scale SRM facility. This demonstration has laid important groundwork for the design and startup of such a facility. If even one-half of the projected savings are realized, the combined investment in this demonstration will be returned during a five-year grain procurement period.

7. References

There are no reference materials to be documented.

8. Points of Contact

Point of Contact	Organization	Phone	Email
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Appendix A.

PROPRIETARY INFORMATION

Appendix A is a separate document containing proprietary information, not appropriate for public release.