FORMULATION AND CHARACTERIZATION OF PARAFFIN-BASED SOLID FUELS
CONTAINING NOVEL ADDITIVES FOR USE IN HYBRID ROCKET MOTORS

A Thesis in
Mechanical Engineering
by
Daniel B. Larson

© 2012 Daniel B. Larson

Submitted in Partial Fulfillment
of the Requirements
for the Degree of

Master of Science

August 2012
The thesis of Daniel B. Larson was reviewed and approved* by the following:

Kenneth K. Kuo
Distinguished Professor of Mechanical Engineering
Thesis Advisor

Richard Yetter
Professor of Mechanical Engineering

Karen A. Thole
Professor of Mechanical Engineering
Head of the Department of Mechanical and Nuclear Engineering

*Signatures are on file in the Graduate School
ABSTRACT

In an attempt to improve the mass burning rates of solid fuels for hybrid rocket motors, this investigation examines the impact of including various additives to paraffin wax. Several paraffin-based formulations, with additives including lithium aluminum hydride (LiAlH₄), diisobutylaluminum hydride (DAH), triethylaluminum (TEA), multi-walled carbon nanotubes (MWNT), and RDX, were cast and tested. Most solid-fuel formulations were cast into paper phenolic motor casings to be tested in the Long-Grain Center-Perforated (LGCP) hybrid rocket motor located at the Pennsylvania State University’s High Pressure Combustion Lab. Casting of fuel grains containing TEA, DAH, and LiAlH₄ was performed at the Aerospace Corporation. After testing a first batch of solid-fuel grains containing LiAlH₄ in the LGCP hybrid rocket motor, it was determined that the particles of LiAlH₄, coupled with a wax that had too low a melting point, were causing unstable, non-uniform burning. New fuel grains with ball-milled LiAlH₄ particles and a higher melting point wax demonstrated much more uniform burning. It was found that with 10% LiAlH₄ in paraffin, the solid-fuel grains showed improved linear regression rates (7-10%) over baseline paraffin. The grains also demonstrated increased mass burn rates. Fuel samples containing MWNT were sent to the University of Texas – Austin’s Center for Nano and Molecular Science and Technology for DSC, SEM, and TGA testing. SEM results showed good dispersion of MWNT in the paraffin; however, there was local clumping of MWNT. Testing of a paraffin/MWNT fuel grain in the LGCP hybrid rocket motor showed that the paraffin/MWNT fuel grain had slightly lower regression that baseline paraffin. However, fuel grains containing MWNT have the potential for improved mechanical properties over pure paraffin. Finally, several methods of dispersing RDX in paraffin wax were attempted, but in each case, the RDX particles came out too large. The particles sank in the melted paraffin, creating a heterogeneous mixture not suitable for casting into a casting to be used in a hybrid rocket motor.
TABLE OF CONTENTS

List of Figures........................................................................................................................................ vi
List of Tables ........................................................................................................................................... ix
Nomenclature .......................................................................................................................................... x
Acknowledgements ............................................................................................................................... xii

Chapter 1 Introduction ........................................................................................................................... 1
  1.1 Background..................................................................................................................................... 1
  1.2 Research Motivation ......................................................................................................................... 3
  1.3 Research Objectives ......................................................................................................................... 4

Chapter 2 Literature Review ................................................................................................................ 6
  2.1 Paraffin Wax as a Hybrid Rocket Fuel............................................................................................ 6
  2.1.1 LiAlH₄ as an Additive to Paraffin Wax ....................................................................................... 7
  2.1.2 RDX as an Additive to Paraffin Wax ......................................................................................... 9
  2.1.3 Multi-Walled Carbon Nanotubes (MWNT) as an Additive to Paraffin Wax.................. 10
  2.2 Long-Grain Center-Perforated Hybrid Rocket Motor ............................................................... 11

Chapter 3 Method of Approach ............................................................................................................ 13
  3.1 Chemical Equilibrium Analysis ..................................................................................................... 13
  3.2 Casting LiAlH₄, DAH, and TEA into Paraffin Wax ......................................................................... 13
      Batch 1 ........................................................................................................................................... 16
      Batch 2 ........................................................................................................................................... 17
      Batch 3 ........................................................................................................................................... 17
  3.3 HPCL Casting Equipment Setup and Procedure Summary ....................................................... 18
  3.4 Casting RDX into Paraffin Wax ...................................................................................................... 21
  3.5 Casting MWNT into Paraffin Wax .................................................................................................. 23
  3.6 Testing Solid-Fuel Grains in the LGCP Hybrid Rocket Motor .................................................. 25
  3.7 Data Reduction Methods ................................................................................................................. 28

Chapter 4 Results and Discussion ....................................................................................................... 30
  4.1 Chemical Equilibrium Analysis ..................................................................................................... 30
  4.2 Paraffin/LiAlH₄ Casting Results ....................................................................................................... 32
      Batch 1 ........................................................................................................................................... 32
      Batch 2 ........................................................................................................................................... 34
      Batch 3 ........................................................................................................................................... 35
  4.3 Paraffin/RDX Cast Results ............................................................................................................. 36
  4.4 Paraffin/MWNT Cast Results ......................................................................................................... 40
  4.5 Test Results from LGCP Hybrid Rocket Motor Test Firings ....................................................... 44
Batch 1 .................................................................................................................. 44
Batch 2 .................................................................................................................. 44
Batch 3 and Paraffin/MWNT Fuel Grains ......................................................... 52
Chapter 5 Conclusions ...................................................................................... 61
  5.1 Solid-Fuel Grain Formulation and Casting .............................................. 61
  Paraffin/LiAlH$_4$ Fuels ................................................................................. 61
  Paraffin/MWNT Fuels .................................................................................. 62
  Paraffin/RDX Fuels ..................................................................................... 63
  5.2 Long-Grain Center-Perforated Hybrid Rocket Motor Tests .................... 64
  5.3 Future Work ............................................................................................ 65
References ............................................................................................................. 67
Appendix A CEA2 Sample Input Files ............................................................... 72
Appendix B MWNT Standard Operating Procedure .......................................... 74
Appendix C Paraffin/RDX Casting Hazard Analysis ......................................... 78
Appendix D LGCP Hybrid Rocket Motor Part Updates ..................................... 87
Appendix E LCGP Hybrid Rocket Motor Test Checklist and Data Sheet .......... 89
Appendix F Error Analysis ............................................................................... 94
Appendix G Batch 2 Test Notes .................................................................... 97
Appendix H Batch 3 Test Notes and Data (Including HPCL-Manufactured Fuel Grains) .... 99
LIST OF FIGURES

Figure 1-1. Comparison of hybrid rocket system with a liquid rocket and solid rocket systems. ................................................................................................................................. 2

Figure 2-1. Simplified schematic of droplet entrainment of a liquefying fuel. .........................7

Figure 2-2. Schematic diagram of the LGCP hybrid rocket motor ........................................... 12

Figure 3-1. Photograph of aluminum casting mold (1.5” inner diameter) with end caps .........14

Figure 3-2. Photograph of aluminum casting mold for spinning on lathe at the Aerospace Corporation ............................................................................................................................... 16

Figure 3-3. Photograph of environmental chamber and casting equipment setup. ..................19

Figure 3-4. Photograph of solid fuel grain casting assembly ..................................................20

Figure 3-5. Plot of temperature versus DMF vapor pressure with paraffin wax melting and casting temperatures .................................................................................................................... 22

Figure 3-6. Modified schematic diagram of the LGCP hybrid rocket motor with graphite spacers and new post-combustion downstream transition section ......................................... 26

Figure 3-7. Modified schematic diagram of the LGCP hybrid rocket system with new check valve and relocated Venturi with pressure transducers ......................................................... 27

Figure 4-1. Plot of theoretical specific impulse versus oxidizer-to-fuel ratio for paraffin wax fuel with various percentages of LiAlH$_4$. [41] ..............................................................................................................................30

Figure 4-2. Plot of produce molecular weight, flame temperature divided by 100, a various mole fractions for paraffin fuel with various percentages of LiAlH$_4$ additive (plotted for O/F = 1.7). [41] ..................................................................................................................... 31

Figure 4-3. First batch casting fuel completely debonded and removed from paper phenolic motor casing .............................................................................................................. 33

Figure 4-4. Cross Sections and close up images shown bubbles, layers, and nonuniformity. .......................................................................................................................................... 33

Figure 4-5. X-ray images of solid-fuel grains with undesirable features ..................................34

Figure 4-6. X-ray images of solid-fuel grains with acceptable features .................................35

Figure 4-7. Typical solid-fuel grain (20% LiAlH$_4$ in paraffin before testing in the LGCP) .... 35

Figure 4-8. SEM images of LiAlH$_4$ particles after a) 3 hours, b) 35 hours, and c) 300 hours of ball milling ..........................................................................................................................36
Figure 4-9. Image of liquid paraffin fuel inside the heated mixing bowl with RDX particles after removal of mixing blades................................. 38

Figure 4-10. SEM images of broken fuel sample surfaces with progressive magnification from 1mm to 50 µm (4% MWNT in paraffin)........................... 42

Figure 4-11. SEM image of broken paraffin fuel with MWNT clumping (8% MWNT in paraffin) ................................................... 42

Figure 4-12. SEM image of broken paraffin fuel sample coated with gold and palladium for reduced surface charge buildup and better, up-close image quality (8% MWNT in paraffin) ................................................... 43

Figure 4-13. LGCP hybrid rocket motor test firing of paraffin with 28% LiAlH$_4$ additive ... 45

Figure 4-14. Typical set of pressure-time traces from paraffin fuel grain with 15% LiAlH$_4$ .......................................................................... 46

Figure 4-15. (a) Transition section and (b) nozzle showing unburned, unreacted fuel accumulation after an LGCP test firing ................................................... 47

Figure 4-16. Pressure time traces from various Batch 2 LGCP hybrid rocket motor firings with initial O$_2$ fluxes of 220 kg/m$^2$-s, with test synchronized to t = 0 seconds as ignition ................................................... 51

Figure 4-17. Load cell thrust data from various Batch 2 LGCP hybrid rocket motor firings.............................................................................. 51

Figure 4-18. LCGP hybrid rocket motor firing of an Aerospace Corporation Batch 3 pure paraffin fuel grain.............................................................................. 52

Figure 4-19. Pressure-time history from an LGCP hybrid rocket motor firing containing a solid-fuel grain manufactured by the Aerospace Corporation containing 10% LiAlH$_4$ at an O/F $\equiv 2.1$........................................................................................................... 54

Figure 4-20. Deduced regression rates for different paraffin-based fuel formulations tested in a gaseous oxygen system (Fuel grains run with O/F lower than the typical values between 2.0 and 2.4 are marked with a *) ................................................... 55

Figure 4-21. Mass burning rates of various solid-fuel formulations tested in a gaseous oxygen hybrid rocket motor (Fuel grains run with O/F lower than the typical values between 2.0 and 2.4 are marked with a *). ................................................... 58

Figure C-1. Hazard analysis procedural flow chart ..................................................... 83

Figure C-2. Hazard identification example #1 ......................................................... 86

Figure C-3. Hazard identification example #2 ......................................................... 86

Figure D-1. New post combustion transition section for downstream section of LGCP ...... 87
Figure D-2. Example of upstream graphite filler piece (4-inch length shown). ..................... 88
LIST OF TABLES

Table 3-1. Table of casting batches of paraffin wax-based fuel samples containing RDX..... 23
Table 4-1. Table of casting batches of paraffin wax-based fuel samples containing MWNT. .......................................................... 43
Table 4-2. Chamber pressure, oxidizer flux, fuel grain mass loss, and test duration data ..... 49
Table C-1. Hazard review mandatory mishap checklist ......................................................... 82
Table C-2. Risk assessment levels ............................................................................. 84
Table C-3. Risk Assessment Matrix ................................................................................. 84
Table C-4. Hazard severity ......................................................................................... 85
Table C-5. Hazard probability ...................................................................................... 85
Table F-1. Various Uncertainty Parameters ................................................................. 94
Table G-1. Post-test comments on fuel grains, motor components, and test data .......... 97
Table G-2. Post-test comments on fuel grains, motor components, and test data from high pressure tests (200 – 330 psig during stable burning) ......................................................... 98
Test data and parameters from Batch 3 fuel grains and HPCL-manufactured fuel grains (Pressure values are taken from the chamber head end pressure transducer) ................. 99
Table H-2. Test notes from Batch 3 fuel grains and HPCL-manufactured fuel grains (Pressure values are taken from the chamber head end pressure transducer) .......... 100
# NOMENCLATURE

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>AP</td>
<td>Ammonium Perchlorate</td>
</tr>
<tr>
<td>A_t</td>
<td>Nozzle throat area</td>
</tr>
<tr>
<td>c*</td>
<td>Characteristic velocity</td>
</tr>
<tr>
<td>c_{theo}</td>
<td>Theoretical characteristic velocity</td>
</tr>
<tr>
<td>δ_{av}</td>
<td>Average specific gravity</td>
</tr>
<tr>
<td>δ_f</td>
<td>Specific gravity of fuel</td>
</tr>
<tr>
<td>δ_o</td>
<td>Specific gravity of oxidizer</td>
</tr>
<tr>
<td>D_f</td>
<td>Final fuel grain port diameter (post-test)</td>
</tr>
<tr>
<td>D_i</td>
<td>Initial fuel grain port diameter (pre test)</td>
</tr>
<tr>
<td>D_{p}</td>
<td>Instantaneous fuel grain port diameter</td>
</tr>
<tr>
<td>DAH</td>
<td>Diisobutylaluminum hydride</td>
</tr>
<tr>
<td>DMF</td>
<td>Dimethylformamide</td>
</tr>
<tr>
<td>η_{c*}</td>
<td>c* efficiency</td>
</tr>
<tr>
<td>g*</td>
<td>Number of molecules in critical radius (size of critical nucleus)</td>
</tr>
<tr>
<td>G_{ox}</td>
<td>Instantaneous oxidizer mass flux</td>
</tr>
<tr>
<td>(\overline{G}_{ox})</td>
<td>Time averaged oxidizer mass flux</td>
</tr>
<tr>
<td>HPCL</td>
<td>High Pressure Combustion Laboratory</td>
</tr>
<tr>
<td>I_d</td>
<td>Density specific impulse</td>
</tr>
<tr>
<td>I_{sp}</td>
<td>Specific impulse</td>
</tr>
<tr>
<td>k</td>
<td>Boltzmann’s constant</td>
</tr>
<tr>
<td>L</td>
<td>Fuel grain length</td>
</tr>
<tr>
<td>LiAlH_4, LAH</td>
<td>Lithium Aluminum Hydride</td>
</tr>
</tbody>
</table>
\( \dot{m}_b \) Fuel mass burn rate  
\( \dot{m}_{\text{ox}} \) Oxidizer mass flow rate  
\( M_f \) Final fuel grain mass (post-test)  
\( M_i \) Initial fuel grain mass (pre-test)  
MWNT Multi-walled carbon nanotubes  
O/F Oxidizer-to-fuel ratio by mass  
P_c Instantaneous chamber pressure  
PSU The Pennsylvania State University  
\( \dot{r} \) Instantaneous regression rate  
\( \bar{r} \) Time averaged regression rate  
RDX Cyclotrimethylene trinitramine  
\( \rho_{\text{fuel}} \) Fuel density  
S Supersaturation Ratio  
\( \sigma \) Interfacial tension between solid and fluid  
T Temperature  
TEA Triethylaluminum  
\( u_s \) Solute solid-phase molecular volume  
\( y \) Instantaneous mole fraction of solute  
\( y_{\text{eq}} \) Equilibrium mole fraction of solute
ACKNOWLEDGEMENTS

The author would like to thank his advisor, Dr. Kenneth K. Kuo. Without Dr. Kuo, this thesis and the opportunities that came with it would not have been possible. Dr. Kuo’s work ethic and drive toward success have been inspirational.

This work was supported (in part) by a NASA Office of the Chief Technologist’s Space Technology Research Fellowship. The author would like to thank Mr. George Story (the Technical Officer on this NASA fellowship project) and Mr. George Fletcher of NASA-Marshall Space Flight Center for their thoughtful questions and suggestions. The author is also grateful to Mr. Gregory Zilliac of NASA-Ames Research Center for giving the author the chance to work as a member of the Peregrine Sounding Rocket Program in 2012. The knowledge and opportunities provided by the on-site experience at NASA-Ames are long-lasting and invaluable.

The author also owes sincere thank you to Dr. John DeSain of the Aerospace Corporation for his constant support and help throughout the duration of this Master’s project. The author would also like to acknowledge Dr. Brian Brady and Dr. Thomas Curtiss of the Aerospace Corporation. The author would like to thank Dr. Joseph Koo and his student, Mr. Russell Borduin, for their help analyzing solid-fuel samples at the Center for Nano and Molecular Science and Technology at University of Texas-Austin.

Thank you to the members, past and present, of the High Pressure Combustion Lab including: Mr. Heath Martin, Mr. Drew Cortopassi, Dr. Jonathan Essel, Mr. Matt Sirignano, Dr. Ryan Houim, Prof. Bao Qi Zhang, Mr. Garrett Lee, Mr. Mike Morgan, and Mr. Seth Lynch for all of your friendship, constructive criticism, and project help. The author would like to thank Dr. Eric Boyer for his guidance, assistance, and continued friendship. In addition, the author would especially like to thank Trevor Wachs for the long hours, suggestions, and hard work on this project.
DEDICATION

This thesis is dedicated to my parents, Bruce and Patricia, and my brothers, Thomas and Joseph, for their unconditional love and support. “To the stars and back.”
Chapter 1

Introduction

1.1 Background

A classical hybrid rocket consists of a solid fuel and a liquid oxidizer. A simplified representation of a hybrid rocket compared to a liquid and a solid system is provided in Figure 1-1. There are several advantages to a hybrid rocket. One major advantage is safety. Unlike a solid rocket system, where the fuel and oxidizer are homogeneously mixed, a hybrid rocket is composed of an inert fuel with a separated oxidizer. The solid fuel grain of a hybrid rocket is more robust, because cracks and imperfections do not lead to catastrophic failures seen in solid rockets. Another advantage of a hybrid system is simplicity. To throttle a hybrid, flow of the oxidizer is changed, unlike liquid systems which require flow rate modulation of both the fuel and the oxidizer via an often complex series of pumps and valves. Hybrid rocket shutdown is achieved by cutting oxidizer flow rate, which a considerable advantage if system termination is required. These benefits help reduce the overall cost of the hybrid system due to lower shipping and manufacturing costs [1].

The various advantages of a hybrid rocket system come with some drawbacks. First, hybrid rocket fuels have a history of low regression rate and low bulk density. The low regression rate and low bulk density means that the solid-fuel grains have reduced thrust and often require multi-port complex geometries for booster applications. Another disadvantage of hybrids is O/F shift. As the motor runs, the fuel port(s) open up, and the O/F varies during the burn. Motor-scaling effects and internal diffusion flame combustion behavior are also complex problems in hybrids which can result in lower combustion efficiency, reducing $I_{sp}$ [1,2].
Early hybrid rocket development began in the 1930’s. Later, in the 1960’s, hybrids were developed as target drones. As the technology developed, high-performance upper stages were designed and boost stages were also investigated. Between 1985 and 1993, American Rocket Company (AMROC) performed many static motor firings of hybrids with vacuum thrust levels ranging from 5000 lb to 272,000 lb [1,2]. In 2004, a privately funded and built spacecraft called SpaceShipOne made by Scaled Composites flew two suborbital flights into space (>100 km) within two weeks to win the Ansari X-Prize [4]. SpaceShipOne was powered by a hybrid rocket system developed at SpaceDev (which had acquired the technical rights and data generated by AMROC in 1998) [1,2]. Another current project, a joint collaboration between NASA Ames Research Center, NASA Wallops, Stanford University and the Space Propulsion Group, Inc.
called the Peregrine Sounding Rocket Program, has a goal to fly a paraffin fuel hybrid rocket to an altitude of 100 km [5]. Details of the design and testing of the Peregrine rocket have been documented and published [6].

1.2 Research Motivation

In an effort to increase thrust without resorting to complex port geometries, much of the recent work on hybrid rockets has been to develop fast-burning solid fuels. One approach has been to use a low melting point fuel such as paraffin wax. The inclusion of various nano-sized and/or energetic additives is another way to further increase the mass burning rates of the solid fuels [1,7,8]. Developing uniform, dense, fast-burning solid fuels is essential to improve the viability of hybrid rocket propulsion systems.

In order to address the low density and regression rate, several paraffin wax-based fuel formulations were developed in this investigation, with additives including: lithium aluminum hydride \( \text{LiAlH}_4 \), diisobutyaluminum hydride \( \text{DAH}, [(\text{CH}_3)_2\text{CHCH}_2]_2\text{AlH}] \), triethylaluminum \( \text{TEA}, (\text{C}_2\text{H}_5)\text{Al} \), cyclotrimethylenetrimetramine \( \text{RDX}, \text{C}_3\text{H}_6\text{N}_6\text{O}_6 \), and multi-walled carbon nanotubes (MWNT). Each of the mentioned additives provides potential benefits when added to paraffin wax. \( \text{LiAlH}_4 \) has been shown to be hypergolic with strong acids (i.e., nitric acid) providing the benefit of restart on demand for the hybrid rocket motor. \[9\] \( \text{DAH} \) and \( \text{TEA} \) are pyrophoric liquids and could help increase regression rate. \( \text{RDX} \), a common ingredient in gun and rocket propellants, is a highly energetic material with a higher density than paraffin, which would increase the bulk density of the paraffin wax and possibly improve the regression rate as well. Finally, inclusion of MWNT could increase the mechanical strength of the paraffin.
The development of new fuels which are safe and cost effective would allow for cheaper space access and more efficient space launch practices. After developing the fuels, each homogeneous fuel formulation was tested using a laboratory-scale hybrid rocket motor located at the High Pressure Combustion Laboratory (HPCL) at the Pennsylvania State University (PSU). This small-scale rocket motor, called the Long-Grain Center-Perforated (LGCP) hybrid rocket motor, allows for fuel formulation characterization while validating its potential use in larger hybrid rocket systems. Large-scale space launch systems can use the research in this study to produce fuel grains with higher density and increased regression rates over baseline paraffin or HTPB.

1.3 Research Objectives

The overall goal of this research is to develop and characterize fast-burning, high density solid fuels for hybrid rocket propulsion applications to provide more affordable and efficient space access. Individual objectives of this investigation were:

1) Formulate paraffin wax-based solid-fuel grains containing LiAlH$_4$ for testing in the LGCP hybrid rocket motor to determine the effect of the LiAlH$_4$ additive on the regression rate and combustion behavior of the solid fuel.

2) Develop a novel casting technique to incorporate RDX in paraffin wax by dissolving the RDX in a solvent compatible with the paraffin wax.

3) Develop a casting technique for inclusion of MWNT in paraffin wax and generate solid-fuel grains for testing in the LGCP hybrid rocket motor.

4) Modify the LGCP hybrid rocket motor so that it promotes complete burning and so that varying lengths of solid-fuel grains can be effectively tested with rapid turnaround time.
5) Test the formulated solid-fuel grains in the LGCP hybrid rocket motor at the HPCL to determine fuel combustion and regression characteristics.
Chapter 2

Literature Review

Before generating or testing paraffin wax-based, solid-fuel grains, it is important to understand the fundamentals behind paraffin wax as a fuel in hybrid rockets and the additives examined in this research. In addition to the detailed additive and fuel information, specific correlations are necessary for the characterization of the performance of various solid-fuel formulations.

2.1 Paraffin Wax as a Hybrid Rocket Fuel

In an effort to increase regression rates in hybrid rockets, a successful method is to use a fuel which forms a melt layer such as paraffin wax. The use of such liquefying fuels in hybrid rocket motors promotes surface regression rate; paraffin wax has demonstrated regression rates 3-4 times higher than conventional hydroxyl-terminated polybutadiene (HTPB) under similar oxidizer mass flux conditions [10,11]. HTPB is a common hybrid rocket fuel and solid rocket propellant binder which gasifies and is transported from the condensed phase surface through a pyrolysis process [12]. The physical mechanism for regression of paraffin wax and other liquefying propellants is generally believed to be from droplet entrainment due to liquid surface layer instabilities. A generalized schematic of the droplet entrainment with roll waves can be seen in Figure 2-1 [10]. The inclusion of energetic and/or nano-sized particles has been shown to improve the mass burning rates and overall fuel density of the solid fuels used in hybrid rockets, including already fast burning fuels like paraffin wax [7,8].
Various additives have the potential to improve the characteristics of the paraffin wax in diverse ways. Some additives can cause the fuel to be hypergolic with other substances like acids, which allows combustion to be restarted on demand without the need for replacement igniters. Other additives, such as metals (e.g., aluminum), can greatly improve the fuel density. Physical properties of the fuel can be modified as well. Inclusion of additives with an oxidizer such as ammonium perchlorate can make the paraffin fuel a fuel-rich propellant. Other additives have the potential to change the mechanical properties of the paraffin wax. With inclusion of multiple additives, a solid-fuel can be tailored to meet various mission and design criteria. Several of these additives are discussed in the subsequent sections.

2.1.1 LiAlH₄ as an Additive to Paraffin Wax

LiAlH₄ is a white powder with a density of 0.917 g/cm³ [13], which is very close to the density of pure paraffin making it possible to spin cast the LiAlH₄ without significant consideration of spinout effects. With additive particles of higher density than the paraffin wax,
centrifugal forces during spin casting cause the additive particles to migrate toward the motor case walls, generating a non-uniform solid-fuel grain structure. Previous work conducted at the Aerospace Corporation [9] examined hypergolic ignition of paraffin wax fuel containing various percentages of LiAlH$_4$ with strong acids such as nitric acid. The ignition event was likely due to the acid-base reaction in the oxygen environment. This caused rapid heat release, which melted and gasified the paraffin wax, elevating the temperature of the wax above its auto-ignition temperature. The result of this is the potential for restart on demand and solid-fuel grains which demonstrate increased regression rates over baseline paraffin. [9]

LiAlH$_4$ has the potential to increase regression rate through the formation of gaseous hydrogen generated by the decomposing aluminum hydride. This can result in increased entrainment of molten fuel droplets into the gaseous oxidizer stream. Hydrogen desorbs from LiAlH$_4$ in 3 reactions, shown below by Reaction 1 – 3 (R1 – R3) [14]. Before these reactions, the LiAlH$_4$ undergoes a phase change from solid to liquid [15], shown as step 1 (S1) below.

\[
\text{LiAlH}_4(s) \rightarrow \text{LiAlH}_4(l) \quad \text{(S1)}
\]

\[
\text{LiAlH}_4 \rightarrow \frac{1}{3}\text{Li}_3\text{AlH}_6 + \frac{2}{3}\text{Al} + \text{H}_2 \quad \text{(R1)}
\]

\[
\frac{1}{3}\text{Li}_3\text{AlH}_6 \rightarrow \text{LiH} + \frac{1}{3}\text{Al} + \frac{1}{2}\text{H}_2 \quad \text{(R2)}
\]

\[
\text{LiH} \rightarrow \text{Li} + \frac{1}{2}\text{H}_2 \quad \text{(R3)}
\]

The reaction time for dehydrogenation reaction R1 can be decreased by decreasing the size of the LiAlH$_4$ particles, which can be done through ball milling, however R2 was shown to be relatively insensitive to the ball-milling time [14]. In other words, smaller particles of LiAlH$_4$ results in faster dehydrogenation of the LiAlH$_4$.

The inclusion of LiAlH$_4$ in paraffin has been studied before. Fuel grains of baseline paraffin (containing 3% polymeric foam) were compared with fuel grains containing 3 and 6% LiAlH$_4$. The fuel samples of paraffin with 6% LiAlH$_4$ showed regression rate increases over the
baseline paraffin [16]; however, operating pressures in the motor (1.5 bar) were far below those typically found in hybrid rocket motors. Another study, performed at the Aerospace Corporation [17] examined open flame burning in air of paraffin with and without LiAlH$_4$ additive. Fuel samples containing LiAlH$_4$ were found to have enhanced mass loss rates compared to pure paraffin. The study also found that the paraffin/LiAlH$_4$ fuel was storable and stable when exposed to air, and was even stable when submerged in water. This is significant, because LiAlH$_4$ is reactive with water.

Osmon [18] demonstrated the use of LiAlH$_4$ in a hybrid rocket with a solid fuel consisting of 95% LiAlH$_4$ with 5% polyethylene. Using hydrogen peroxide as the liquid oxidizer, the LiAlH$_4$-based fuel was found to be slightly pressure sensitive. As the aluminum particles left the surface, they developed an oxide shell in the oxidizing environment, possibly inhibiting further and full reaction of the aluminum particles. It is possible that the aluminum particles were not reaching high enough temperatures to quickly ignite and fully combust, which would account for the downstream accumulation that Osmon observed in the nozzle after the test. It is known that aluminum particles tend to agglomerate at the burning surface [19]. Evans, et. al [20], using SEM techniques, observed aluminum particle accumulations at the surface of burned HTPB hybrid rocket fuel with Silberline® aluminum flakes.

### 2.1.2 RDX as an Additive to Paraffin Wax

Cyclotrimethylenetrinitramine (C$_3$H$_6$N$_6$O$_6$), commonly known as RDX, is an energetic, white powder with a density of 1.82 g/cm$^3$ [21]. Although the RDX is much denser than paraffin, its ability to increase mass burning rate and regression rate of the solid fuel comes from the fact that RDX is highly energetic. It is a common ingredient in rocket and high power gun propellants. Former investigations of including ammonium perchlorate (AP) in an HTPB/polypropylene
glycol (PPG) binder have been performed. One formulation included 60% AP in the HTPB/PPG binder. This fuel-rich propellant achieved a DOT Class 1.4C rating [22,23]. This is important because RDX poses safety concerns (the bulk material is a Hazard Class/Division 1.1D high explosive), but in a paraffin fuel matrix, the fuel grains can remain essentially inert. Stability and thermal behavior of RDX is documented and predictable [21].

By dissolving the RDX in a solvent, a safe mixture can be combined with melted paraffin. RDX is soluble in many solvents including acetone and dimethylformamide (C$_3$H$_7$NO); the solubility of RDX in the various solvents is well understood and has been widely documented [24]. Dimethylformamide (DMF) is particularly desirable not only because of RDX’s solubility in the DMF, but also because of its compatibility with paraffin wax. DMF can be used for the removal of aromatic hydrocarbons in liquid paraffin wax [25]. Another important quality of dimethylformamide is its high boiling point of ~153 °C [26], which is substantially higher than the melted paraffin casting temperature. The boiling point of the solvent can be changed by drawing a vacuum [27], allowing the solvent to be evaporated out of the melted paraffin without changing the paraffin wax casting temperature.

2.1.3 Multi-Walled Carbon Nanotubes (MWNT) as an Additive to Paraffin Wax

Mechanical properties of paraffin wax have been tested and published [28], but better mechanical properties are required. Multi-walled carbon nanotubes (MWNT) consist of multiple graphene layers wrapped around themselves [29]. The MWNT used in this study are branded Baytubes® C 150 P from Bayer MaterialScience. They have between 3 and 15 walls with a mean diameter between 13 and 16 nanometers. The overall length of the MWNT various from 1 to >10 µm, and the bulk density of the material is between 140 and 160 kg/m$^3$ [30].
MWNT have the potential to change the thermal properties of the paraffin wax as well as advance the mechanical characteristics of the fuel. In a study conducted by Allaoui et. al [29], Young’s modulus and yield strength were doubled and quadrupled, respectively, for MWNT/epoxy composites with 4 wt% MWNT. MWNT have also been added to paraffin wax samples in another study [31]. These paraffin/MWNT samples showed an increase in thermal conductivity as additive amount increased. These MWNT were ball milled before being sonicated in the paraffin wax [31]. MWNT have also been used in ablative applications in a phenolic resin and in polymers [32,33]. The mixing procedures for distributing the MWNT in the resin and polymers were also documented.

The low cost of MWNT make them a beneficial, practical additive. This presents a scale up advantage. Expensive single-walled nanotubes could be used for laboratory scale samples and experiments, but as hybrid rocket motor size and fuel grain size increase, fuel cost becomes substantial. Paraffin/MWNT could be a significant cost savings compared to paraffin based fuels with similar percentages of single-walled nanotubes.

### 2.2 Long-Grain Center-Perforated Hybrid Rocket Motor

The Long-Grain Center-Perforated (LGCP) hybrid rocket motor is a laboratory scale hybrid rocket motor located at PSU’s High Pressure Combustion Laboratory. The motor has been used with gaseous oxygen and liquid nitrous oxide (N₂O) oxidizers. Capable of running oxygen mass fluxes up to 0.8 lbm/s (0.36 kg/s), the LGCP can operate at pressures up to 1,750 psig (12 MPa). Fuel grains are cast into 1.5-inch outer diameter paper phenolic tubes which can be loaded into the test apparatus. This allows for rapid turnaround times so that many tests can be run. A schematic of the LGCP is provided in
Figure 2-2. A remotely operated data acquisition system is used for data collection from four pressure transducers and a load cell attached to the test deck. During motor firings, a video system is used to record the tests. The LGCP has been an accurate and reliable test system and has proven to be an effective way of testing HTPB and paraffin-based solid fuels with various nano-sized and/or energetic additive particles [34,35,36].

Figure 2-2. Schematic diagram of the LGCP hybrid rocket motor.
Chapter 3

Method of Approach

3.1 Chemical Equilibrium Analysis

Prior to any testing in the Long-Grain Center-Perforated (LGCP) hybrid rocket motor or casting new fuel grains for testing, chemical equilibrium analysis was performed to determine optimal O/F ratios, changes in specific impulse ($I_{sp}$), characteristic velocity (c*), etc. at a variety of O/F values. This was done using a computer program developed at NASA called CEA2 (Chemical Equilibrium with Applications) [37]. For consistent comparisons between fuel formulations, the program was run with a chamber pressure of 1000 psia (6.9 MPa) and a nozzle expansion ratio of 10. These values were chosen since they are representative of a booster operating in the upper atmosphere. As an example, SpaceDev, the same company that made the 20-inch hybrid motor that powered SpaceShipOne, developed a motor called the Streaker™ with a common core booster with an expansion ratio of 10:1 [38]. Chamber pressures can vary, such as 700 psi for the Delta II or 3700 psi for the Atlas V [39]. A sample CEA2 input file is provided in Appendix A. In all cases, the paraffin wax was assumed to be pure C$_{32}$H$_{66}$ with a heat of formation of -231.65 kcal/mol [40].

3.2 Casting LiAlH$_4$, DAH, and TEA into Paraffin Wax

Paraffin wax-based solid-fuel grains containing LiAlH$_4$, DAH, and TEA additives were cast at the Aerospace Corporation in El Segundo, CA using a process similar to one previously developed at Penn State University’s High Pressure Combustion Laboratory. These grains were
cast and tested in several batches as the formulations and casting consistencies were tailored to achieve more uniform, complete combustion. The cast procedures for the two batches are similar, but the differences are important. In all cases, the batches were cast into 16-inch (406.4-mm) long paper phenolic tubes with an inner diameter of 1.25 inches (31.75 mm) and an outer of 1.5 inches (38.1 mm). Prior to each cast, the inner surface of the phenolic tube, which acts as the fuel cartridge, was roughed using a wire brush to promote better bonding between the paraffin-based fuel and the cartridge wall. The phenolic tube was then placed inside an aluminum mold with sealable end caps to facilitate spinning on a lathe. An image showing the aluminum mold and end caps is provided in Figure 3-1.

![Figure 3-1. Photograph of aluminum casting mold (1.5" inner diameter) with end caps [41].](image)

To cast each solid-fuel grain, paraffin wax was melted and the additives were mixed into the paraffin. The purities of the lithium aluminum hydride, triethylaluminum, and diisobutylaluminum hydride were >95%, ~93%, and reagent grade Aldrich, respectively. Because the TEA and DAH were in liquid form, they were measured and added to the paraffin wax slightly differently than the LiAlH₄ additive. Equation 3.1 and Equation 3.2 show how mass and volume percentages were determined for the three different additives.
\[
\text{mass percentage of LiAlH}_4 = \frac{\text{(mass of LiAlH}_4)}{\text{(mass of LiAlH}_4 + \text{mass of paraffin wax})} \times 100\% \tag{3.1}
\]

\[
\text{volume percentage of liquid additive} = \frac{\text{(volume of additive)}}{\text{(volume of additive + volume of paraffin wax})} \times 100\% \tag{3.2}
\]

Due to the volatility and reactivity of each of the various additives, casting was done under a nitrogen purge (in a purged bag). For casting the LiAlH\(_4\), the additive was added to the melted paraffin in the nitrogen purged bag, while the wax was stirred for 15 minutes and maintained at a constant temperature of ~100 °C. Then, the mixture of paraffin wax with LiAlH\(_4\) was poured into the phenolic fuel cartridge inside of the aluminum mold. The volume of the pour was generally between 100 – 125 mL. After each pour, the end caps of the aluminum mold were sealed, and the assembly was placed on a lathe as shown in Figure 3-2. The mixture was spun at 400 RPM (1500 RPM for “Batch 1”) for approximately 25 minutes to allow for solidification of the paraffin wax. Prior to the next layer pour, the mold was reheated to ~50 °C to avoid fast cooling of the newly poured paraffin wax and to promote a strong, homogeneous bond between the old and new layers [41].
Figure 3-2. Photograph of aluminum casting mold for spinning on lathe at the Aerospace Corporation.

**Batch 1**

Batch 1, which was considered a test batch for evaluating the casting procedure, was cast in the same manner as Batch 2 below. Major differences among Batch 1 and the other batches were high lathe spinning (1500 RPM), the phenolic fuel cartridge was not roughed prior to casting, black candle dye was used in place of carbon black, and a single pour was used to fill the phenolic fuel cartridge for Batch 1. The results of this test batch are briefly discussed in Chapter 4 of this investigation. Pure paraffin, 25% LiAlH$_4$, and 50% LiAlH$_4$ motors were cast.
**Batch 2**

In the second batch, a paraffin wax with a melting temperature between 57.8 and 61.9 °C was used. Approximately 0.2% carbon black particles were added to the wax as well as ~1% low density polyethylene (LDPE). Typically, 3 to 4 pours were required to fill the grain. After the final cooling, a center port of 0.375 inches (9.5 mm) was drilled. In between pours, the fuel grains were X-rayed to observe the homogeneity of the fuel grain and adherence of the layers to one another. If any major defect (e.g., void) was detected, the fuel was remelted, and the process was started again. The grains containing the liquid additives (TEA and DAH) were cast similar to those containing LiAlH$_4$. The difference for these other grains was that they were cast and maintained in a nitrogen purged-environment through the entire process, until they were sealed in the aluminum mold; aluminum mold assembly was done inside a nitrogen purged bag. This was done as a safety precaution due to the pyrophoric behavior of the TEA and DAH [41]. After the grains were cast in 16-inch lengths, they were sent to PSU for testing. The fuel grains were then cut to lengths of 4.5 inches before testing in the LGCP hybrid rocket motor.

**Batch 3**

Due to issues in the second batch, which are discussed later in Chapter 4, a third batch of solid-fuel grains containing LiAlH$_4$ was cast. In this batch, the as-received LiAlH$_4$ particles were ball-milled for 300 hours to achieve smaller-sized particles. In addition, a higher melting point wax (between 70 and 80 °C) was used [42], and no LDPE was included. Also, the inner surface of the paper phenolic fuel cartridges was roughed more thoroughly with a wire wheel. Three pours of the melted wax into the phenolic cartridges were required for these solid-fuel grains. The
16 inch fuel grains were sent to PSU where they were cut to lengths of 4, 5, and 5.5 inches (101.6, 127, and 139.7 mm).

### 3.3 HPCL Casting Equipment Setup and Procedure Summary

The casting equipment at the HPCL consists of a heated mixing bowl and a stand mixer located inside an environmental chamber. An image of the casting setup inside the environmental chamber is shown below in Figure 3-3. Using a centrifugal pump, silicon fluid was pumped from a heated, external bath through chamber wall feedthroughs into a hollow cavity between the mixing bowl and an outer pot. Temperature of the silicon heating fluid was taken at the entrance of the hollow cavity. The hollow cavity around the pot is referred to as the “Heating fluid circulation jacket” in Figure 3-3. Once a steady temperature is achieved, the melted paraffin inside the pot is assumed to be at a temperature close to this value. Temperature is controlled via a manual external heater and a variable transformer.

The environmental chamber can be run as a vacuum chamber or it can be fitted with an exhaust line for purge gas. For vacuum casts with solvents or hazardous chemicals, a cryogenic trap was used inline before the vacuum pump to condense the vapors leaving the environmental chamber. This reduced the risk of hazardous vapors entering the work area and prevented the solvent vapors from entering the vacuum pump, where the vapors could damage seals. The cryotrap was constructed with a reservoir surrounded by a bath of ethanol cooled by dry ice. For casts involving flammable vapors (i.e., toluene) not under vacuum, an exhaust line was attached to the environmental chamber and nitrogen purge gas flow was established.
Once a fuel formulation was melted and thoroughly mixed, the paraffin-based mixture was poured into the paper phenolic fuel cartridge. An image of this assembly is shown in Figure 3-3, although the phenolic tube shown is shorter than the one that was typically used (16 – 18 inches). It should be noted that prior to pouring, the phenolic tube interior was roughed with sand paper, and the entire cast assembly was heat soaked in an oven at ~60 °C. This promoted good adhesion between the case wall and the fuel, and it prevented the paraffin from instantly solidifying and forming air pockets against the various casting pieces. After pouring the melted paraffin fuel mixture, the assembly was then placed on a lathe and spun at 400 RPM for up to 75 minutes to allow full solidification of the paraffin wax. This centrifugal style of casting promotes good contact between the phenolic case wall and the paraffin wax. Paraffin wax shrinks as it cools, so the centrifugal casting method prevents the wax from pulling away from the paper phenolic case wall.
Because paraffin wax shrinks as it cools (the fuel grain was poured nearly full on the first pour), it was necessary to make a second pour of the melted fuel to fill the rest of the fuel cartridge. Prior to this second pour, the casting assembly shown in Figure 3-3 was disassembled and allowed to heat soak again. This softens the first pour wax, and promotes good bonding between the poured layers. The original fuel mixture, which was kept under constant mixing and heat, was then poured into the fuel cartridge, and the lathe spinning process was repeated. After removing the fuel grain assembly from the lathe, the end retaining pieces, end caps, and center mandrel were removed. The fuel grain was cut and then faced on a lathe until the proper length was achieved. Because the center mandrel generally leaves a somewhat nonuniform inner surface, a center bore was drilled to a uniform diameter in each grain. Typically, center-bore diameters were around 0.375 inches.

Figure 3-3. Photograph of solid-fuel grain casting assembly.
3.4 Casting RDX into Paraffin Wax

It should be noted that a detailed hazard analysis was performed (Appendix C) in accordance with DoD standards so that controls were implemented to ensure safe handling of the RDX. RDX casting took place in several steps. First, small amounts (< 4 grams) of wetted, Class I, Type II military grade RDX powder was weighed and baked at low temperature to remove any moisture in the damp powder. The next step was to dissolve the RDX in the dimethylformamide (DMF) solvent. The typical ratio at room temperature, based on solubility data [24], was 1 gram of RDX/5 mL of DMF. After dissolving the RDX, the DMF/RDX solution could safely be moved to the environmental chamber where the mixing equipment was located. The paraffin wax had been melted and was held at around 100 °C. As an important safety note, mixer clearances were set to avoid any contact of the mixing blades with pot walls, and all equipment was grounded in compliance with DoD standards.

The mixer was turned on, and the DMF/RDX solution was added to the melted paraffin. After the solution was poured into the melted paraffin, the environmental chamber door was closed. A camera system, enabled with a live video feed, was placed near the window of the environmental chamber so that the casting process could be monitored remotely. The camera was set to focus on the paraffin/DMF/RDX mixture. The other unused window of the environmental chamber was shielded with a canvass covering to prevent any glass fragments from scattering in the event of a chamber overpressurization and subsequent window failure. Also, CEA2 was performed to determine the level of pressure in the environmental chamber through the closed volume combustion of 8 grams of RDX. The input file for this calculation was placed in Appendix A.

After the environmental door was closed, the chamber was pumped down to appropriate pressures based on the test procedure. The various cast batch pressure levels are shown below in
Figure 3-5. In each case, after the vacuum was started, the temperature was slowly increased to boil out the DMF. In the case where the DMF was boiled out slowly (“Cast Condition 2”), after approximately 1.5 hours of continuous mixing, a deep vacuum (~1psia) was drawn to check for any bubbling which would indicate remaining DMF. If there was none, the mixer was stopped, the vacuum pump was shut down, and the chamber was pressurized. Images of the RDX were taken before the paraffin cooled, to qualitatively check for the degree of RDX particle size and dispersion in the melted paraffin.

A fourth cast condition was also attempted. For this condition, the DMF was allowed to stay in solution. This was done due to DMF’s compatibility with paraffin. For this condition, the RDX was dissolved in the DMF at an elevated temperature, which increases the solubility of the RDX. This also means that less DMF is required to dissolve the appropriate amount of RDX. The
heated DMF/RDX solution was then transferred to the environmental chamber and poured into the paraffin mixture under constant mixing. Table 3-1 details the various cast conditions for the paraffin-based fuel containing RDX.

Table 3-1. Table of casting batches of paraffin wax-based fuel samples containing RDX.

<table>
<thead>
<tr>
<th>Cast Condition</th>
<th>wt% RDX (nominal)</th>
<th>Nominal Ratio Paraffin/RDX (grams)</th>
<th>DMF Boiling Rate</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4%</td>
<td>48/2</td>
<td>Rapid</td>
</tr>
<tr>
<td>2</td>
<td>4%</td>
<td>48/2</td>
<td>Slow</td>
</tr>
<tr>
<td>3</td>
<td>2%</td>
<td>98/2</td>
<td>Moderate</td>
</tr>
<tr>
<td>4</td>
<td>2%</td>
<td>89/2 (9 gms DMF)</td>
<td>None</td>
</tr>
</tbody>
</table>

3.5 Casting MWNT into Paraffin Wax

Casting of the multi-walled carbon nanotubes into paraffin wax took several steps. The first step was to “debulk” the MWNT. Typically, this is done via a sonication process, as communicated by laboratory members at University of Texas – Austin’s Center for Nano and Molecular Science and Technology. It is necessary to unwrap the nanotubes and then evenly distribute them in the paraffin matrix. In this investigation, the MWNT were debulked by sonication in toluene. This was done by floating an open beaker of toluene in a water sonication bath. The usual ratio of MWNT nanotubes to toluene was approximately 1 to 2 grams per 100 mL of toluene. Toluene has been observed to break down under intense sonication or long periods of sonication [43], so sonication times were typically limited to 20 minutes. For verification, this phenomenon was also observed in the HPCL bath sonicator with a small beaker of toluene floated in the sonication bath. The toluene breakdown observed at the HPCL was observed after
approximately 40 minutes; however, the beginnings of breakdown may have occurred after approximately 30 minutes.

After the MWNT had been dispersed in toluene, the mixture was added to pre-melted paraffin. It is important to note that the paraffin temperature was held at around 100 °C, which is below the boiling point of toluene (~110 °C). This was important so that the toluene did not boil as soon as it contacted the paraffin wax. Toluene was selected specifically for this purpose as its boiling point is in the range of standard operating procedure melting temperatures of the paraffin wax. The mixer was started and set at a low speed, and the environmental chamber door was closed.

Using the nitrogen purge line attached to the environmental chamber and an exhaust line to the exterior of the building, a nitrogen flow was established and run constantly as the temperature of the mixture was increased. This was done as a safety precaution due to toluene’s high flammability in air. A fan was also used to circulate the gases inside the chamber and stir up toluene’s dense vapors in an effort to exhaust as much of the toluene as possible. The length of time varied for boiling off toluene. Generally, the casting equipment had an appreciable odor from the toluene, but when the smell was gone, it was assumed that the majority of the toluene had been evaporated out of the mixture. The full standard operating procedure for preparing the MWNT was placed in Appendix B.

Small-scale fuel samples (~50 grams) were generated and sent to the University of Texas – Austin for TGA, DSC, and SEM testing at the Center for Nano and Molecular Science and Technology. These samples had MWNT additive ranges between 0 and 8 wt% in the paraffin wax. In addition to the small scale samples a fuel grain for testing in the LGCP was also generated using the prescribed methods from “3.3 HPCL Casting Equipment Setup.” Details of the cast batches of paraffin wax containing MWNT were placed in Table 4-1. It should be noted that several pure paraffin solid-fuel grains were cast into paper phenolic tubes so that they could
be compared with fuel grains manufactured by the Aerospace Corporation as well as the fuel grain with paraaffin/4% MWNT.

3.6 Testing Solid-Fuel Grains in the LGCP Hybrid Rocket Motor

Because the LGCP can accommodate HTPB-based fuel formulations, the fuel grain length for paraffin-based fuels is much shorter at optimal O/F values. Fuel grains were shortened to lengths where desirable O/F ratios could be achieved during the LGCP test firings. The short fuel grains (as short as 2.5 inches) from Batch 1 were extended to the full 16-inch LGCP length by adding a section of empty phenolic tube; however, after tests revealed accumulations of unburned fuel in the nozzle and original post-combustion chamber, further modifications were required. Since the empty phenolic tube filler was an expendable that needed to fabricated and used for each test, a more sustainable option was desired. To accommodate various lengths of solid-fuel grains, graphite spacers of different lengths were fabricated to fill upstream portions of the LGCP hybrid rocket motor, instead of the empty phenolic tube. Since the combustion products from early tests were not fully reacting, an extended downstream transition section was fabricated to replace the original post-combustion chamber. This extended post-combustion transition section provided additional time for the combustion products to fully react and combust. Drawings of these parts have been placed in Appendix D, and the modified LGCP hybrid rocket motor schematic is shown below in Figure 3-5.
The LGCP hybrid rocket motor was run with gaseous oxygen for all tests in this study, and a nitrogen purge was used to extinguish the flame and cool the motor at the end of each test. Typically, chamber pressures were between 160 and 350 psig (1.1 and 2.41 MPa). The solid-fuel grains inserted in the motor were varied in length to achieve various O/F values. Batch 2 oxidizer mass fluxes were focused around 220 kg/m²-s, while Batch 3 and other grains generated at the HPCL had varying oxidizer mass fluxes and test conditions. A more detailed description of the various tests and results is outlined in Chapter 4. The LGCP testing checklist was placed in Appendix E for reference.

Data was acquired from four pressure transducers and a load cell by a data acquisition system, and valve and igniter timing was controlled by an automated computer system with LabVIEW. A live-feed camera was also used to record the test firings so that the burn could be evaluated on a qualitative, visual basis (i.e., streaks in plume, steadiness of burn, etc.). Due to
some valve timing issues and delays in nitrogen purge, grains after Batch 2 were tested with a modified apparatus setup. The before and after schematic of the LGCP hybrid rocket system is detailed in Figure 3-7 and discussed further in Chapter 4.

To summarize the changes made to the LGCP hybrid rocket system, the Venturi grouping (Venturi and accompanying pressure transducers PT1 and PT2) was relocated, and a new check valve was added downstream of PV2 in the oxygen line. The Venturi (a calibrated orifice) was relocated so that both the oxygen and nitrogen must travel through the Venturi before reaching the motor. Although this change limited the flow of nitrogen purge gas on its way to the combustion chamber, it also allowed the high pressure nitrogen purge gas to snap a new check valve (inserted in the oxygen line) closed, eliminating the flow of oxygen after that point. When the Venturi was relocated, the upstream and downstream pressure transducers were also relocated.

Figure 3-6. Modified schematic diagram of the LGCP hybrid rocket system with new check valve and relocated Venturi with pressure transducers.
so that all existing calibrations could be used. The goal of this was to reduce start up and shut down transients.

3.7 Data Reduction Methods

Along with CEA2 results, it was necessary to deduce regression rate data, since this was a major topic of interest in this investigation. Equation (3.3) and Equation (3.5) [44] were used to generate plots of regression rate versus oxidizer mass flux.

\[
\hat{r} = \frac{D_f - D_i}{2l_0}
\]  

(3.3)

\[
D_f = \sqrt{D_i^2 + \frac{4(M_1 - M_f)}{\pi L_f' fuel}}
\]  

(3.4)

\[
\bar{G}_{ox} = \frac{16\dot{m}_{ox}}{\pi (D_f + D_i)^2}
\]  

(3.5)

\[
\dot{r} = aG_{ox}^n p_c^m D_p^l
\]  

(3.6)

It should be noted that \(D_f\) can be determined by measuring down the port of the solid fuel grain after each test and averaging at various locations, or \(D_f\) can be calculated using Equation (3.4). Unless otherwise noted, \(D_f\) was calculated using Equation (3.4) in this study. \(D_f\) can be calculated from the measured values of fuel length, initial diameter, fuel density, initial fuel grain mass, and final fuel grain mass. Then, regression rate (3.3) can be determined with burn time calculated from ignition to burnout using the pressure-time history plots recorded for each test. From the plots of regression rate versus oxidizer flux, regression rate coefficients for \(a\) and \(n\) for a time averaged version of Equation (3.6) [2] can be determined. It should be noted that \(a, l, m,\) and \(n\) are empirical constants, and \(l\) and \(m\) are usually taken as zero, unless there is a pressure or port...
diameter dependence. In the case of this investigation, \( l \) and \( m \) were taken to be zero due to the limited test matrix. Finally, mass burn rate of the fuel was taken as the equation shown in (3.7).

\[
\dot{m}_b \equiv \frac{M_i - M_f}{t_b}
\]  

(3.7)

Average chamber pressures, oxidizer mass fluxes, oxidizer mass flow rates, average thrust cell measurements, and all pertinent calculations based on collected data were deduced using MATLAB and Microsoft Excel. Details of the error analysis used in the investigation are located in Appendix F.
Chapter 4
Results and Discussion

4.1 Chemical Equilibrium Analysis

CEA2 calculations of LiAlH$_4$ additive to paraffin show favorable results, as shown below in Figure 4-1. As LiAlH$_4$ is added to paraffin wax, the dependency of I$_{sp}$ on O/F is reduced. The plateaus of the curves on the plot are flatter at peak I$_{sp}$ values as LiAlH$_4$ additive is increased. This is beneficial in a hybrid rocket system, because it allows for operation over a wider range of O/F as the port diameter inside the hybrid rocket motor changes. This benefit would be helpful in accommodating O/F shift which occurs in hybrid rockets. Reduced amounts of oxidizer are necessary (i.e. lower O/F) for peak performance as LiAlH$_4$ content in paraffin is increased. [41]

Figure 4-1. Plot of theoretical specific impulse versus oxidizer-to-fuel ratio for paraffin wax fuel with various percentages of LiAlH$_4$. [41]
Figure 4-2 shows CEA2 calculation results comparing mole fractions of H, OH, and H\(_2\) in the combustion products, as well as molecular weight and flame temperature with increasing LiAlH\(_4\) additive percentage. It was noted that the ratio of flame temperature to product molecular weight tended to increase as LiAlH\(_4\) additive increased, although this ratio peaked at the 40%-LiAlH\(_4\) additive data point. An O/F of 1.7 was selected for comparison as it is near the peak \(I_\text{sp}\) for most of the various additives as shown in Figure 4-1. [41]

![Figure 4-2. Plot of produce molecular weight, flame temperature divided by 100, a various mole fractions for paraffin fuel with various percentages of LiAlH\(_4\) additive (plotted for O/F = 1.7). [41]](image)

CEA2 calculations for density specific impulse showed that as RDX content increased (from 0 to 10% RDX), the density specific impulse increased slightly up to O/F values of around 2.2. Density specific impulse was calculated using Equation 4.1 and Equation 4.2 [2] using gaseous oxygen with paraffin wax containing various amounts of RDX up to 10%.
\[ I_d \approx \delta_{av} I_{sp} \quad (4.1) \]

\[ \delta_{av} = \frac{\delta_o \delta_t (1+\frac{\delta_t}{\delta_p})}{(\frac{\delta_t}{\delta_p})\delta_t + \delta_o} \quad (4.2) \]

### 4.2 Paraffin/LiAlH₄ Casting Results

**Batch 1**

When Batch 1 solid fuel grains arrived at PSU from the Aerospace Corporation for testing, the fuel grains were non-uniform and heterogeneous throughout and even debonded from the phenolic case walls. This can be seen in Figure 4-3 and Figure 4-4. Despite the improper bonding and the non-uniformity, the grains were repaired and cut to lengths ranging from 2.5 to 8 inches (63.5 to 203.2 mm). The grains were repaired by roughing the inner surface of the phenolic tube and gluing the grains in place using cyanoacrylate glue. The inconsistencies in the solid-fuel grains and debonding issues indicated that changes in the casting procedure were required for better uniformity and bonding to the case wall. Changes in the casting process included roughing the phenolic tube more thoroughly, reduced lathe rotational speeds, and heat soak of the fuel grain prior to new wax layer pours.
Figure 4-3. First batch casting fuel completely debonded and removed from paper phenolic motor casing.

Figure 4-4. Cross Sections and close up images shown bubbles, layers, and nonuniformity.
Batch 2

Batch 2 grains manufactured at the Aerospace Corporation incorporated the casting procedure modifications identified from Batch 1 analysis and testing, and contained paraffin wax fuel grains with various amounts of LiAlH$_4$ additive (up to 28%). In addition, fuel grains with 10% liquid additives (TEA and DAH) were cast and used for comparison tests in the LGCP. The percentage additives of LiAlH$_4$ were 10%, 15%, 20%, and 28%, as well as a pure paraffin fuel grain (as a control). During the casting, the fuel grains were X-rayed between pours to check for inconsistencies in the fuel grain. If one existed, the grain was remelted and redone. Examples of undesirable solid-fuel grain features are shown in Figure 4-5. Fuel grains with better consistency, capable of undergoing testing in the LGCP hybrid rocket motor, are shown in Figure 4-6. The grains were cut to lengths of 4.5 inches (114.3 mm), and a small transition section was carved into the front section of the fuel grain. A typical solid-fuel grain, cut to the proper length and ready for testing, is shown in Figure 4-7. [41]
Figure 4-6. X-ray images of solid-fuel grains with acceptable features.

Figure 4-7. Typical solid-fuel grain (20% LiAlH$_4$ in paraffin) before testing in the LGCP.

Batch 3

Batch 3 fuel grains from the Aerospace Corporation consisted of a pure paraffin grain, a 5% LiAlH$_4$ in paraffin grain, and a 10% LiAlH$_4$ in paraffin grain. Differences for Batch 3 compared to prior batches were that there was no addition of LDPE, the melting point wax was a
higher melting point wax (70 to 80 °C), and the LiAlH$_4$ additive particles were ball-milled for 300 hours. The effect of various ball-milling times on the LiAlH$_4$ additive particle sizes can be observed in SEM images in Figure 4-8. SEM images of LiAlH$_4$ particles after a) 3 hours, b) 35 hours, and c) 300 hours of ball milling. The majority of the particles were sub-micron in size with many of the particles less than 100 nm; however, many of the particles have an alumina layer which could delay the combustion of the particles during decomposition [45], which could be even more damaging if the particles were completely covered as Al$_2$O$_3$ is inert. When the solid fuel grains were cut to proper lengths, the cross sections showed uniform fuel with few defects or voids in the grain structure. Several grains had varying center-port diameters, but the grains were of high enough quality that they could be tested in the LGCP hybrid rocket motor after average initial port diameters were measured.

![SEM images](image)

Figure 4-8. SEM images of LiAlH$_4$ particles after a) 3 hours, b) 35 hours, and c) 300 hours of ball milling. [45]

### 4.3 Paraffin/RDX Cast Results

As discussed in Chapter 3, four different sample casts were generated with RDX added to paraffin. In “Cast Condition 1” (refer to Figure 3-4), after the dimethylformamide (DMF) and
RDX solution was added to the melted paraffin, a vacuum was drawn. However, as can be seen in Figure 3-4, the boiling point of the DMF was 20 to 30 °C below the temperature of the melted paraffin wax. This led to possible superheating, and a violent boiling event splattering bubbles, melted paraffin, and nucleated particles of RDX all around the mixing bowl, mixer, and environmental chamber windows and walls.

In an effort to eliminate the rapid boiling event, a second cast (“Cast Condition 2”) was used. In this case, the mixing and evaporating off of the DMF took place over several hours. Because the solution only became slightly supersaturated over time, it was speculated that the RDX particles that nucleated out of the DMF during the slow boil were much larger than those that came out during the violent boiling. At the end of the process, before the paraffin was allowed to cool and solidify, images were taken to show the state of the RDX dispersion in the paraffin wax. An image inside the heated mixing bowl of the “Cast Condition 2” result is shown in Figure 4-9.
Figure 4-9. Image of liquid paraffin fuel inside the heated mixing bowl with RDX particles after removal of mixing blades.

Not long after the photograph shown in Figure 4-9 was taken, most of the RDX particles had settled to the bottom of the melted paraffin in the mixing pot, forming a very heterogeneous fuel sample. “Cast Condition 2” was deemed far from satisfactory, so a third cast condition, “Cast Condition 3,” was performed in an attempt to find an intermediate result between violent, rapid boiling of the DMF solvent and the slow evaporation of DMF which resulted in large RDX particles which sank to the bottom of the mixing bowl. For “Cast Condition 3” it was attempted to hold pressure at a steady level where the boiling point of the DMF was only slightly below the temperature of the melted paraffin. However, this was more difficult that originally predicted as the point at which the DMF rapidly boils was very fine. In other words, although attempts were made to avoid rapid boiling, some spattering from the violent release of DMF was still observed.
Even after all the DMF was removed through a faster boiling process, the result of “Cast Condition 3” was similar to that of “Cast Condition 2”. Because the pot walls are not perfectly smooth, it could be that nucleation sites along the pot walls allowed the RDX particles to be too large. It should be noted that “Cast Condition 3” used around twice as much wax with the same amount of RDX, creating a mixture of 2% RDX in paraffin as opposed to 4% RDX in paraffin. This was done to promote more uniform mixing.

It was noted that although the rapid boiling was not desirable, it may have promoted smaller RDX particles. The size of the critical nucleus, $g^*$, is reduced as the supersaturation ratio, $S$, increases. This is shown through Equations 4.3 [46] and 4.4 [47], where $y$ is the instantaneous mole fraction of the solute, and $y_{eq}$ is the equilibrium mole fraction of the solute in the solution.

$$g^* = \left(\frac{32\pi}{3}\right)\left(\frac{\sigma(v_g)^2}{kT}\right)^3 \left(\frac{1}{\ln S}\right)^3$$  \hspace{1cm} (4.3)  

$$S = \frac{y}{y_{eq}}$$  \hspace{1cm} (4.4)

Therefore, as the instantaneous mole fraction is increased quickly, as during a rapid boil, this would serve to increase the supersaturation ratio. When the evaporation of DMF was slow, it allowed the instantaneous mole fraction of RDX to remain very close to the equilibrium mole fraction, which was not conducive to generating small particles of RDX. Note that $g^*$ is not in units of length. It represents the number of molecules inside the critical nucleus. So, in essence, as $g^*$ is reduced, the RDX particle sizes are also reduced by having to contain less molecules in the critical nucleus.

Because DMF is a flammable liquid, it was speculated that the inclusion of the liquid in the final fuel grain may not be undesirable. So, for a fourth cast condition, the DMF was kept in the paraffin solution. This condition was slightly different, as the DMF temperature was elevated in order to dissolve a larger amount of RDX. Three grams of RDX was mixed on a hot plat into six grams of DMF at elevated temperature (~60 °C) for about 5 minutes. This was done so that
less DMF was necessary, and given the DMF’s compatibility with paraffin, it was speculated that once poured into the paraffin, the mixture would be homogeneous. However, when pouring the DMF/RDX solution into the mixing pot, as soon as the solution touched the pot walls, the RDX nucleated out. The sides of the pot were scraped down, and the mixer was run. After mixing, clear separation could still be seen, and when pouring the pot into a sample tray, the RDX could be seen settling in the bottom of the mixing pot, showing that this method requires further investigation to determine the proper solubility limits of the paraffin/DMF/RDX mixture so that a homogeneous mixture might be achieved. Solid-fuel grains for testing in the LGCP hybrid rocket motor were not manufactured due to concerns with the fuel structure and nonuniformity.

4.4 Paraffin/MWNT Cast Results

It was noted during the casting process that higher concentrations of MWNT in the melted paraffin significantly affected the viscosity of the melted fuel, and higher percentages and larger amounts of MWNT became increasingly difficult to sonicate and process. Properly debulking enough MWNT needed to generate a fuel grain for testing with the LGCP hybrid rocket motor required more than 1.5 L of toluene during sonication. Despite this large volume of toluene, there was still a great deal of settling during the pouring of the toluene/MWNT into the mixing pot. The nanotubes also appeared to agglomerate and settle in the mixing pot. This was also noticed at a small scale from the SEM images of the small scale (<50 grams) samples sent to University of Texas–Austin and imaged at UT-Austin’s Center for Nano and Molecular Science. A detail of the various fuel casts of paraffin with MWNT additives is provided in Table 4-1. It should be noted that additive percentages are nominal, and it is likely that the weight percentage of MWNT in the paraffin is lower than the value stated due to losses in the casting process (i.e., MWNT sticking to beakers, mixing pot walls, mixing blades, etc.).
The SEM images of broken paraffin/MWNT fuel samples in Figure 4-10 and Figure 4-11 show surface charge buildup, which appear as bright spots. To reduce the bright spots and remove this charge buildup on the surfaces, sputter coating with gold and palladium particles was attempted. A close-up SEM image of a coated fuel sample is shown in Figure 4-12. The MWNT were seen as light colored strings, and bundling in the paraffin wax was observed. The MWNT are either not being debundled enough during sonication in toluene, or they are agglomerating during the long mixing process. The sonication time could not be increased due to the possibility of toluene breakdown [43], and the mixing time could not be reduced because the time is purely depended on the time necessary to evaporate the toluene out of the paraffin wax. A solution to this problem would be to sonicate the MWNT in melted paraffin with a wand sonicator, eliminating the need for toluene. The use of a surfactant may also be beneficial to better wet the MWNT with the paraffin.
Figure 4-10. SEM images of broken fuel sample surfaces with progressive magnification from 1mm to 50 µm (4% MWNT in paraffin).

Figure 4-11. SEM image of broken paraffin fuel with MWNT clumping (8% MWNT in paraffin).
Figure 4-12. SEM image of broken paraffin fuel sample coated with gold and palladium for reduced surface charge buildup and better, up-close image quality (8% MWNT in paraffin).

<table>
<thead>
<tr>
<th>wt% MWNT</th>
<th>Ratio Paraffin/MWNT (gms)</th>
<th>Amount of Toluene Used (mL)</th>
<th>Casting Notes/Observations</th>
</tr>
</thead>
<tbody>
<tr>
<td>5%</td>
<td>Test batch</td>
<td>Test batch</td>
<td>Test batch, not enough toluene used, MWNT sank to bottom</td>
</tr>
<tr>
<td>4%</td>
<td>48/2</td>
<td>~130+</td>
<td>Very brittle, fuel has faint toluene odor, homogeneous</td>
</tr>
<tr>
<td>0% with Toluene</td>
<td>48/2</td>
<td>~100</td>
<td>Mixer broke during cast, less brittle than last cast, ripples on surface</td>
</tr>
<tr>
<td>2%</td>
<td>49/1</td>
<td>~150</td>
<td>Paraffin appeared to sweat during removal</td>
</tr>
<tr>
<td>0%</td>
<td>50/0</td>
<td>0</td>
<td>Mixed with heat for ~30 minutes, paraffin still appears to sweat</td>
</tr>
<tr>
<td>2%</td>
<td>49/1</td>
<td>~150</td>
<td>Good cast, sonicated with beaker level to bottom of bath, higher temp cast</td>
</tr>
<tr>
<td>4%</td>
<td>48/2</td>
<td>~175</td>
<td>Increase in viscosity of melted fuel, settling of MWNT after sonication</td>
</tr>
<tr>
<td>8%</td>
<td>46/4</td>
<td>~400</td>
<td>Needed to scrape sides of mixing bowl during process, even higher viscosity</td>
</tr>
<tr>
<td>4%</td>
<td>432/18</td>
<td>~1500+</td>
<td>Lots of toluene settling in environmental chamber, took 3+ hours to remove toluene, great deal of settling at bottom of pot, stratification, grain delaminated from case wall</td>
</tr>
</tbody>
</table>
4.5 Test Results from LGCP Hybrid Rocket Motor Test Firings

**Batch 1**

General results from the LGCP hybrid rocket motor test firings were inconclusive due to significant accumulations of unburned/partially reacted fuel in the downstream combustion chamber and nozzle. Some grains also demonstrated uneven burning, which was observed by examining the grain geometry after each test firing. The uneven burning and low combustion efficiency (i.e., unburned fuel in downstream sections of the motor) indicated that a change to the casting procedure and some motor changes were necessary to produce better burning results. Changes made to the LGCP hybrid rocket motor were discussed in Chapter 3, such as a new, longer transition section and upstream graphite filler pieces.

**Batch 2**

Many of the results discussed in this section were described in the paper by Larson et. al [41] and presented at the 2011 Joint Propulsion Conference in San Diego, CA. It was noted during the testing of the Batch 2 fuel grains cast by the Aerospace Corporation that several grains experienced burnouts (consumption of all fuel as well as phenolic wall charring). In fact, tests run on the baseline paraffin fuel grains as well as the fuel grains containing 10% LiAlH$_4$ showed this effect as the test sequence was tailored. All other fuel grains after these initial ones were run at consistent test conditions. Figure 4-13 shows an LGCP hybrid rocket motor test firing of a paraffin fuel grain containing 28% LiAlH$_4$. 
Figure 4-13. LGCP hybrid rocket motor test firing of paraffin with 28% LiAlH$_4$ additive. [41]

A typical set of pressure-time traces is provided in Figure 4-14. Although target test durations were only 1.3 seconds to ensure only a portion of the fuel was consumed, due to startup and shutdown transients the full burn time of the test firings was usually around 2.7 seconds. Ignition and flame spreading occurs at approximately 0.1 seconds as chamber pressure continuously rises. Combustion product energy was transferred to the fuel surface to constantly melt the paraffin wax fuel, which maintained the diffusion flame. The pressure-time trace shows approximately 1 second of steady state burning. Around the middle of the steady state burning, the oxygen valve was shut to begin shutdown.

It could be observed that high pressure nitrogen drove up the chamber pressure as remaining oxygen was mixed with the nitrogen until the nitrogen quenched the flame and the combustion process was terminated, which was seen as an exponential decay from the chamber pressure. It was also speculated that the increase in chamber pressure may have been caused by a partially blocked nozzle from unburned fuel collection. The sequence delay timing in the valves likely could have been reduced, but for consistency, the LGCP hybrid rocket motor setup was run in the same configuration for all tests in order to easily compare the results from the various solid-fuel grain formulations. One other possibility is that there is a continued reaction of LiAlH$_4$ particles with remaining oxygen or water vapor in the combustion products; however this was less likely since the similar pressure increases were seen from fuel grains containing TEA and DAH.
Figure 4-14. Typical set or pressure-time traces from paraffin fuel grain with 15% LiAlH$_4$.

A key result from these tests was that in nearly all cases, a layer of unburned and unreacted fuel had accumulated on the downstream sections of the hybrid rocket motor (i.e., transition section and the converging section of the nozzle). After each test, the graphite nozzle and transition section were placed in warm water. The two sections bubbled and fizzed showing that the fuel which was deposited on these sections was at least partially unreacted since LiAlH$_4$ is highly reactive with water. The two sections were allowed to remain in the water to passivate any of the rest of the unreacted fuel. Prior to submerging the parts in water, masses were recorded. Typically, there was between 3 and 6 grams of fuel accumulated on the transition section and 0.2 and 1.7 grams of fuel on the nozzle. Since the mass loss from the fuel grains was between 20 and 48 grams total, the amount of fuel trapped in the downstream sections of the LGCP hybrid rocket motor was significant (10% - 22%). Sample photographs of the nozzle and transition piece are shown in Figure 4-15, with large accumulations of paraffin wax/LiAlH$_4$ which are gray colored on the black graphite.
Figure 4-15. (a) Transition section and (b) nozzle showing unburned, unreacted fuel accumulation after an LGCP test firing.

One possible reason for the downstream fuel buildup was that the melt layer was too thick, which captured the LiAlH$_4$ particles and carried them downstream into the transition section and nozzle. This thick layer also may have been pushed downstream during the purge period. A higher melting point wax with a smaller melt layer would have allowed the LiAlH$_4$ particles to react closer to the paraffin fuel surface, transferring more energy to the fuel. The higher melting point wax could also have reduced the amount of fuel pushed downstream through a thinner melt layer. It was also possible that the LiAlH$_4$ particles cast into the paraffin wax were too large. As the large particles of LiAlH$_4$ were entrained in the flow, they may not have had time to fully react and burn before traveling into the downstream sections of the motor. A previous study showed that LiAlH$_4$’s phase change from solid to liquid at 170 °C occurs during an endothermic peak period, followed almost immediately by an exothermic peak at slightly higher temperature where the total energy released/absorbed is nearly neutral [48]. This would imply that the reaction of the LiAlH$_4$ particles is governed by the energy input by the system. By mass, smaller particles would allow the heat transfer to occur more rapidly and achieve more rapid
reaction before they are carried downstream. In essence, smaller LiAlH₄ particles may aid in attaining more complete combustion.

The solid-fuel grains containing triethylaluminum and diisobutylaluminum hydride showed similar characteristics when burning compared to the grains with LiAlH₄. The DAH and TEA grains still left deposits of paraffin fuel in the downstream transition section; however, the fuel was fully-reacted. The downstream sections were placed in water, and unlike the sections filled with paraffin/LiAlH₄, these sections were unresponsive to the water. This could simply be because of the high reactivity of these two liquid additives. In any case, it appeared that the total amount of fuel deposited from the TEA and DAH additive fuel grains was reduced compared to the LiAlH₄. The first TEA grain burned evenly and uniformly. The second test caused pitting in the transition piece which had broken into two pieces. Upon close inspection, it was found that there were deep pits in the transition section after the test firing. One of these pits had burned all the way through the transition section to the LGCP’s inner steel wall. It was speculated that this could be attributed to the volatile behavior of the TEA, as it may have still been reacting in the post combustion chamber.

Table 4-2 shows the test matrix for Batch 2 fuel grains. The typical time for each test was around 2.7 seconds, measured from ignition to burnout. An additional table (Table G-1) is provided in Appendix G and contains detailed test notes. Both tables are ordered the same.
Table 4-2. Chamber pressure, oxidizer flux, fuel grain mass loss, and test duration data.

<table>
<thead>
<tr>
<th>Fuel Grain</th>
<th>Maximum Pressure (psig)</th>
<th>Maximum Pressure (MPa)</th>
<th>Steady Region Pressure (psig)</th>
<th>Steady Region Pressure (MPa)</th>
<th>Initial Oxidizer Flux, $G_{Ox, i}$ (kg/m$^2$-s)</th>
<th>Fuel Grain Mass Loss (g)</th>
<th>Test Duration (s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure Paraffin</td>
<td>311</td>
<td>2.14</td>
<td>231</td>
<td>1.59</td>
<td>307</td>
<td>78</td>
<td>2</td>
</tr>
<tr>
<td>Pure Paraffin</td>
<td>156</td>
<td>1.08</td>
<td>124</td>
<td>0.85</td>
<td>198</td>
<td>70.9</td>
<td>2.85</td>
</tr>
<tr>
<td>Pure Paraffin</td>
<td>189</td>
<td>1.30</td>
<td>126</td>
<td>0.87</td>
<td>154</td>
<td>28.8</td>
<td>2.66</td>
</tr>
<tr>
<td>10% LiAlH$_4$</td>
<td>159</td>
<td>1.10</td>
<td>139</td>
<td>0.96</td>
<td>178</td>
<td>33.4</td>
<td>2.69</td>
</tr>
<tr>
<td>20% LiAlH$_4$</td>
<td>221</td>
<td>1.52</td>
<td>190</td>
<td>1.31</td>
<td>226</td>
<td>49.1</td>
<td>3.09</td>
</tr>
<tr>
<td>30% LiAlH$_4$</td>
<td>305</td>
<td>2.10</td>
<td>179</td>
<td>1.23</td>
<td>220</td>
<td>54.2</td>
<td>4.25</td>
</tr>
<tr>
<td>15% LiAlH$_4$ †</td>
<td>290</td>
<td>2.00</td>
<td>189</td>
<td>1.30</td>
<td>223</td>
<td>47.9</td>
<td>2.77</td>
</tr>
<tr>
<td>20% LiAlH$_4$ †</td>
<td>272</td>
<td>1.88</td>
<td>188</td>
<td>1.30</td>
<td>222</td>
<td>37.8</td>
<td>2.7</td>
</tr>
<tr>
<td>25% LiAlH$_4$ †</td>
<td>294</td>
<td>2.03</td>
<td>201</td>
<td>1.39</td>
<td>218</td>
<td>31.4</td>
<td>2.68</td>
</tr>
<tr>
<td>30% LiAlH$_4$ †</td>
<td>262</td>
<td>1.81</td>
<td>207</td>
<td>1.43</td>
<td>223</td>
<td>28</td>
<td>2.75</td>
</tr>
<tr>
<td>25% LiAlH$_4$ †</td>
<td>285</td>
<td>1.97</td>
<td>221</td>
<td>1.52</td>
<td>220</td>
<td>31.8</td>
<td>2.69</td>
</tr>
<tr>
<td>10% TEA</td>
<td>320</td>
<td>1.59</td>
<td>167</td>
<td>1.15</td>
<td>218</td>
<td>30.9</td>
<td>2.71</td>
</tr>
<tr>
<td>10% DAH</td>
<td>241</td>
<td>1.66</td>
<td>173</td>
<td>1.19</td>
<td>218</td>
<td>33.9</td>
<td>2.77</td>
</tr>
<tr>
<td>10% DAH</td>
<td>262</td>
<td>1.81</td>
<td>197</td>
<td>1.36</td>
<td>228</td>
<td>27.4</td>
<td>2.83</td>
</tr>
<tr>
<td>10% DAH</td>
<td>254</td>
<td>1.75</td>
<td>194</td>
<td>1.34</td>
<td>222</td>
<td>20.9</td>
<td>2.8</td>
</tr>
</tbody>
</table>

* Shown in Figure 10 and Figure 11
† Shown in Figure 11
‡ Shown in Figure 12

It was difficult to determine whether increases in LiAlH$_4$ contributed to increases in regression rate based on tabulated data and summary plots. It was difficult to determine regression rate correlations, because there was so much fuel accumulated in downstream sections or due to uneven burning. However, a pressure relationship can be seen from a comparison plot (Figure 4-16) of pressure time traces from assorted tests. This was confirmed by CEA2 calculations. The CEA2 calculations showed that as additive percentage of LiAlH$_4$ increased at a given O/F, so too did the theoretical c*. Since c* is a function of chamber pressure, it makes sense that the LGCP test firing results showed an increase in chamber pressure as LiAlH$_4$ content increased. It can be seen in Figure 4-16 that as LiAlH$_4$ content increases, the average steady state burning chamber pressure also increases. This increase in pressure was attributed to the increase in the ratio of flame temperature to the molecular weight of the products, which corresponds to an increase in c*, as discussed in the section 4.1 results for CEA2 calculations.
The pressure relationship was confirmed by thrust measurements made by a load cell on the thrust stand, which are shown in Figure 4-17. It should be noted that the fuel grains containing pure paraffin and 10% LiAlH$_4$ were not plotted in Figure 4-16 or Figure 4-17, because they were run at varying test conditions. It was also observed that the two tests run with 20% LiAlH$_4$ additive had nearly the same thrust measurement, with data points which lie on top of one another in Figure 4-17. The post test grain geometries were also very similar, which indicated similar, repeatable burning characteristics.

To generate a more diverse test matrix, hybrid rocket motor tests were run at higher intended chamber pressures. These tests were run under the same oxidizer conditions, but with a smaller nozzle throat to obtain stable region burning pressures ranging from 200 – 330 psig (1.4 – 2.3 MPa). There was only one test of each grain performed at higher pressures, and during those tests, several grain debondings occurred. Due to these facts, pressure correlations were not presented. The unburned, unreacted fuel was left the downstream sections of the motor, just the same as the other tests. Although high pressure test data correlations were inconclusive, notes from these tests were placed in Table G-2 in Appendix G.
Figure 4-16. Pressure time traces from various Batch 2 LGCP hybrid rocket motor firings with initial O$_2$ fluxes of 220 kg/m$^2$-s, with test synchronized to $t = 0$ seconds as ignition.

Figure 4-17. Load cell thrust data from various Batch 2 LGCP hybrid rocket motor firings.
Batch 3 and Paraffin/MWNT Fuel Grains

The results and discussion in this section were submitted for journal review and, at the time of this writing, are to be presented at the 9th International Symposium on Special Topics in Chemical Propulsion (9-ISICP) in Quebec City, Canada [49]. The third batch of solid fuel grains for the LGCP hybrid rocket motor included fuel grains with ball milled LiAlH_4 particles in the paraffin wax as well as a baseline paraffin wax motor. These three grains (5\% LiAlH_4, 10\% LiAlH_4, pure paraffin) were cast at the Aerospace Corporation. The fuel grains were cut to various lengths so that they could be run at nearly the same O/F ratios while varying oxidizer mass flux values. Paraffin-based fuel grains cast with MWNT additives were also tested, as well as baseline paraffin grains cast at PSU’s HPCL. It should be noted that, for simplicity, the entrance region carved into Batch 2 grains was not carved into the Batch 3 grains. An image of an Aerospace Corporation pure paraffin fuel grain during testing in the LGCP is shown in Figure 4-18.

Figure 4-18. LCGP hybrid rocket motor firing of an Aerospace Corporation Batch 3 pure paraffin fuel grain.

The Batch 3 tests as well as the pure paraffin and paraffin/MWNT fuel grains manufactured at the HPCL were tested with the new LGCP system setup (detailed in the
schematic shown in Figure 3-6). A typical pressure-time history from an LGCP test firing is shown in Figure 4-19. Generally, the average chamber pressure during the “steady state burning” portion of the test was ~245 psig (1.69 MPa). The test shown in Figure 4-19 displayed steady burning. In the figure, ignition is shown just after t = 0 seconds, and combustion ends at approximately 1.9 seconds. Note that after the oxygen run valve closed, the pressure dropped slightly before the high pressure nitrogen purge gas snapped the oxygen check valve closed and drove up the pressure. The N\textsubscript{2} purge gas was intentionally set at a higher pressure than O\textsubscript{2} feed pressure in order to shut that check valve and extinguish combustion. The HPCL hybrid rocket facility was originally designed for high flow rates (up to 360 g/s) with multi-hybrid system capabilities, which made rapid startup and shutdown timing difficult at much lower flow rates due to large valve sizes and residence times associated with large tubing volumes. The relocation of check valve and overdriving with N\textsubscript{2} purge gas reduced the delays in startup and shutdown, addressing the original system design issues.

The timing delays and issues were speculated to be insignificant, although time for oxygen to clear the plumbing calculations were still performed. A key reason why the timing delays were more significant for paraffin tests was that the LGCP was originally designed for HTPB, a much slower burning fuel. With longer tests with the HTPB, the startup and shutdown transients were less significant. Because paraffin has a substantially higher burn rate, the startup and shutdown transients were a more substantial portion of the burn. The LGCP’s small diameter cartridge provides a very limited burn time for the paraffin-based solid-fuel grains, and the change in oxidizer flux over the length of the burn is also quite significant. However, overall, the LGCP hybrid rocket motor is used for fuel formulation comparisons more than it is used for high-accuracy regression rate measurements, so the initial and final motor transients can be somewhat neglected, since they are nearly the same for the various fuel formulations. For accurate, real-time regression rate data, a larger, X-ray transparent hybrid rocket motor is used. This larger motor
allows for longer burn times, and startup and shutdown transients are negligible since the regression rate behavior is taken instantaneously via X-ray images.

Figure 4-19. Pressure-time history from an LGCP hybrid rocket motor firing containing a solid-fuel grain manufactured by the Aerospace Corporation containing 10% LiAlH$_4$ at an O/F $\equiv 2.1$

A comparison plot of regression rate versus oxidizer flux of usable tests is shown in Figure 4-20. The density of the fuel grains containing LiAlH$_4$ was taken as 0.86 g/cm$^3$, the density of the Aerospace Corporation’s pure paraffin was taken to be 0.898 g/cm$^3$, PSU’s paraffin was listed as 0.86 g/cm$^3$, and finally, the PSU’s MWNT additive grains was taken as 0.865 g/cm$^3$. These densities are important, because, as discussed in Chapter 3, they are important for calculating the final port diameter ($D_f$). Many of the fuel grains had some end burning, and in other cases, some burning to the phenolic case wall was observed, which would cause $D_f$ values to be inflated. When comparing calculated $D_f$ to measured $D_f$, data values were typically within 10% of one another, somewhat validating the use of calculated $D_f$. 
Figure 4-20. Deduced regression rates for different paraffin-based fuel formulations tested in a gaseous oxygen system (Fuel grains run with O/F lower than the typical values between 2.0 and 2.4 are marked with a *).

From the plot in Figure 4-20, it can be seen that the fuel grains manufactured by the Aerospace Corporation containing 10% LiAlH$_4$ had slightly elevated regression rates over the baseline Aerospace Corporation paraffin. The paraffin/10% LiAlH$_4$ fuel grains had 7 – 10% higher regression than the paraffin wax in average oxidizer mass flux ranges between 60 and 100 kg/m$^2$-s. It was speculated that some of this increase could be attributed to the lower density of the paraffin/LiAlH$_4$ fuel grains; however, mass regression rates showed a similar trend. This is
shown in Figure 4-21. Post-firing observations of transition and nozzle pieces from the fuel grains with LiAlH$_4$ showed only a small film of particles which thinly coated the sections. The mass of this film was not appreciable, and the overall deposition of fuel/particles was negligible. Submerging the transition piece and nozzle in water did result in some slow bubbling, but it was far reduced from the Batch 2 bubbling and fizzing from the same pieces. So, although there is still some unreacted fuel in these downstream sections, it was significantly reduced compared to the previous tests.

The pure paraffin grains manufactured at PSU’s HPCL have an uncharacteristically high regression rate, which is even higher than the paraffin/LiAlH$_4$ fuel grains. This is because of the low density of the as cast fuel, which is can be seen in Figure 4-21 as the HPCL paraffin has lower mass regression than the paraffin/10% LiAlH$_4$ fuel grain. HPCL’s cast paraffin was only slightly higher than the Aerospace Corporation’s paraffin in terms of mass burning rates. The low density fuel would tend to burn more rapidly, but low density is not desirable. The low density of the HPCL as cast paraffin indicates issues in the casting procedure which would allow air to remain trapped in the solid-fuel grain. This could have occurred after the first pour with the paraffin wax, during reheating. It was observed that a white, foamy layer forms on the surface of the paraffin after spinning on the lathe. This foamy layer may not have been getting softened enough during the heat soak. It is also possible that the second pour of the paraffin wax was not at a sufficiently high temperature to eliminate the foamy layer. This would have kept the surface bubbles from completely melting away. It could also be that the paraffin was not spun fast enough on the lathe to push the wax to the fuel grain wall, leaving bubbles near the inside surface, although particle spin out effects become a concern as lathe speeds increase.

O/F values were typically between 2 and 2.4, although grains run at other conditions or fuel grains which burned to the paper phenolic cartridge wall (5% LiAlH$_4$) were different. Grains with these issues are denoted in Figure 4-20 and Figure 4-21. Fuel grains marked with a “*” were
noted because they were run with O/F ratios (1.5 – 1.75) which were lower than the other fuel grains. For the fuel grains manufactured at the Aerospace Corporation, the 10% LiAlH₄ fuel grains were run with an O/F of between 2.0 and 2.2, and the pure paraffin fuel grains were run with O/F values between 2.3 and 2.4. As shown in Figure 4-1, peak Iₛₚ for paraffin containing LiAlH₄ shifts left to lower O/F values. So, although the O/F values for tests with 10% LiAlH₄ were lower than for tests with pure paraffin, it is satisfactory because the grains with LiAlH₄ have a peak Iₛₚ at lower O/F values. This point is clear when looking at data points from Figure 4-20, where in most cases, the 10% LiAlH₄ achieved the same regression as the corresponding pure paraffin grains, but the grains had lower average oxidizer mass fluxes. It could be concluded then, that increasing the content of LiAlH₄ in paraffin wax could improve the regression rate of the solid fuel. However, it should be noted that this is a very limited series of tests, and additional runs are necessary to validate these results. Fuel grains containing 15 – 25% ball milled LiAlH₄ could be tested to generate a more complete test matrix.
It was expected that the fuel formulation with 4% MWNT would have a lower regression rate due to the higher amount of carbon in the fuel grain. It was speculated that MWNT increases conductivity to the subsurface layers of the fuel, since thermal conductivity was shown to increase in paraffin containing MWNT [31]. This increase in conductivity to the subsurface fuel layers may reduce the overall burning rate of the fuel by possibly decreasing the surface temperature of the fuel. It is also possible that this heat transfer to the fuel subsurface could cause increases in the melt layer of the fuel, which may affect the fuel burn rate. MWNT could be used
with energetic additives to overcome these shortcomings. It is also possible that the MWNT could replace the use of chemical dyes or carbon black in futures solid fuel grains, and despite lower regression rates, the MWNT could still provide a mechanical strength advantage over pure paraffin fuel grains. More testing would be required in order to prove this result. Inclusion of MWNT in a paraffin wax fuel grain would reduce the density of the solid-fuel grain due to the lower density of MWNT compared to paraffin wax.

Note that the 5% LiAlH$_4$ fuel grain at the highest oxygen mass flux had a very high mass burn rate and linear regression rate. After this test, it was 1.9 grams of unburned fuel was found in the downstream component of the LGCP hybrid rocket motor, which likely contributed to inflated regression numbers. Also, the burnout to the phenolic cartridge wall may have also contributed to the high regression figures. This was because the fuel had non-uniform regression characteristics. One part of the solid-fuel grain had web remaining, while the other had burned out. It is also possible that pieces of the fuel broke off of the main web as burning approached the phenolic tube wall and the fuel web was nearly completely consumed, which would inflate the regression rate.

A brief analysis comparing the c* and c*-efficiencies ($\eta_{c^*}$) of the Aerospace Corporation’s pure paraffin and paraffin/10% LiAlH$_4$ fuel revealed that the 10% LiAlH$_4$-additive fuel grains had slightly higher c* and c*-efficiencies (< 5%) over the baseline paraffin. Average c*-efficiencies between the six tests (three of each formulation) ranged between 0.76 and 0.81. Although these are somewhat low efficiency numbers, the efficiency numbers among the tests are very consistent. The equations used to calculate these values are shown below in Equation 4.5 and Equation 4.6 [2]. Note that $P_c$ was taken at the steady-state pressure from each test. Theoretical c* ($c_{\text{theo}}^*$) was taken from equilibrium (CEA2) calculations.

$$c^* = \frac{P_c A_t}{\dot{m}} \tag{4.5}$$

$$\eta_{c^*} = \frac{c^*}{c_{\text{theo}}^*} \tag{4.6}$$
A final note on Batch 3 was that there was load cell data collected. These data, although useful, was not conclusive when analyzing for differences in thrust between different fuel grains. This is because nozzle sizes were changed to hold chamber pressure close to constant. Test chamber pressures for pure paraffin fuel grains and grains containing LiAlH₄ were very similar. In fact, chamber pressures between the pure paraffin and 10% LiAlH₄ at each test condition were within 5% of one another. Notes and basic test information were placed in Appendix H.
Chapter 5

Conclusions

5.1 Solid-Fuel Grain Formulation and Casting

Paraffin/LiAlH$_4$ Fuels

Equilibrium calculations showed that as LiAlH$_4$ content is increased in the paraffin wax, peak $I_\text{sp}$ values become flatter, and peak $I_\text{sp}$ curves shift to lower O/F ranges. This shows that grains with LiAlH$_4$ are more accommodating of O/F shift encountered during normal hybrid rocket motor operation and throttling. CEA2 calculations also showed an increase in the ratio flame temperature to the molecular weight of the products (up to ~40% LiAlH$_4$) as LiAlH$_4$ content was increased. This ratio increase corresponded to an increase in theoretical $c^*$.

Initial batches (Batch 1 and Batch 2) did not have desirable burning or, in many cases, homogeneous grain properties. It was speculated that an increase in the wax melting point and/or reduced particle size was necessary. Batch 3 fuel grains yielded much better results. Fuel grains were more uniform, and LiAlH$_4$ particles were much smaller in size. After 300 hours of ball milling, particle sizes were generally sub-micron with many particles less than 100 nm in size, although many of the ball milled particles showed an alumina layer which was speculated to possibly reduce the reactivity of the LiAlH$_4$ additive. When cutting the grains open, there were few voids or defects, and the fuel grains appeared homogeneous. Center port diameter varied among the grains, often differing within a single grain, but the overall composition was good enough for testing.
Paraffin-based solid-fuel samples containing multi-walled carbon nanotubes were generated by debulking the MWNT through sonication in toluene and then mixing the toluene/MWNT mixture into melted paraffin. The paraffin temperature was then elevated to evaporate out the toluene, leaving paraffin with MWNT evenly distributed in the wax matrix. The paraffin/MWNT fuel samples were sent to University of Texas–Austin’s Center for Nano and Molecular Science and Technology where they were imaged using SEM techniques. The fuel samples, with percentages of MWNT as high as 8% showed that the MWNT were uniformly distributed, but were often bundled and clumped together. This indicated that the casting procedure of sonicating the MWNT in toluene and then boiling the toluene out of the paraffin wax was not sufficient to fully debulk the MWNT. It was also speculated that the MWNT may have been getting clumped during the extended mixing time required to boil out toluene from the paraffin/MWNT fuel mixture.

It was observed that the MWNT significantly changed the viscosity of the melted wax during the mixing and casting process. When pouring nanotubes from beakers and from the final mixing pot, a great deal of settling was observed. This is likely another indication of insufficient sonication time or intensity, which is reduced due to toluene’s temperamental behavior during sonication. A solid fuel grain was cast for testing in the LGCP hybrid rocket motor. The fuel grain was highly uniform and the fuel grain appeared homogeneous. No carbon black was required since MWNT were used.
Paraffin/RDX Fuels

Cast procedures and hazard analyses were developed, and solid-fuel casting equipment was modified (inclusion of cryogenic trap, remote monitoring, etc.) and validated to generate fuel samples. Solid fuel samples containing RDX were generated using various casting conditions. It was observed in each case that the RDX nucleated out of the DMF solvent as larger particles and the particles did not remain suspended in the paraffin wax. Rather, the particles sank to the bottom of the mixing pot, creating a two layer, heterogeneous fuel. This would have caused significant issues during centrifugal cooling on a lathe, so fuel grains for testing in the LGCP hybrid rocket motor were not tested. Four total sample batches were generated using four slightly differing methods.

The first cast condition caused rapid boiling due to superheating of the DMF solvent. This splattered the liquid fuel mixture all over the environmental chamber. The second batch used a slow boiling method, but this slow process caused large particles of RDX to nucleate out, rather than the planned small particles. The third cast condition, intended to be a middle point between the first two cast conditions resulted in some rapid boiling, but even when the DMF was evaporated out of the mixture, large particles of RDX were the result, and they sank to the bottom of the mixing pot. A final cast condition was to dissolve RDX at elevated DMF temperature and insert the mixture into the paraffin wax. Stratification of the DMF/RDX solution was observed, and any of the solution that contacted mixing bowl walls released RDX particles on contact. It was speculated that the mixing bowl may not have been smooth enough, and therefore provided nucleation sites for the RDX.
5.2 Long-Grain Center-Perforated Hybrid Rocket Motor Tests

Batch 1 fuel grains containing LiAlH₄ (manufactured at the Aerospace Corporation) had uneven burning and very poor fuel grain characteristics. It was determined that changes to the casting procedure were necessary to get better burning and more uniform solid-fuel grains. Batch 2 solid-fuel grains containing LiAlH₄ showed better burning characteristics, but there was non-negligible accumulation of unreacted and unburned fuel (10 – 22% of the total fuel mass loss) in the downstream sections of the LGCP. This caused difficulty in determining regression rates. Batch 2 results did show that increased percentages of LiAlH₄ in the paraffin wax resulted in higher chamber pressures. This validated CEA2 calculations which showed an increase in the ratio flame temperature to the molecular weight of the products (up to ~40% LiAlH₄ additive). The increase in chamber pressure as LiAlH₄ additive increased was confirmed by load cell results. It was speculated that in order to reduce the amount of unburned and unreacted fuel in the downstream components of the hybrid rocket motor, reduced LiAlH₄ particle size and/or higher melting point wax were required. Increasing the oxidizer-to-fuel ratio was also speculated as a solution to reduce downstream fuel accumulation.

The third batch of solid-fuel grains manufactured by the Aerospace Corporation had very even burning in the motor, with few fuel grain delamination issues and minimal end burning. There was a small film of unreacted fuel in the downstream motor sections, but it was not an appreciable mass of fuel and was neglected. Fuel grains containing 10% LiAlH₄ showed an increase in linear regression rate between 7 – 10%, although some of this increase may be due to the lower density of the paraffin/LiAlH₄ fuel grains. Mass burn rates were also higher for fuel grains containing LiAlH₄. Paraffin fuel grains containing MWNT demonstrated uniform, even burning, although the burn rates with the MWNT were lower than the HPCL paraffin fuel grains. It was speculated that this reduced burning rate was due to the increase in the fuel’s thermal
conductivity with the inclusion of MWNT. This increase in conductivity may reduce the fuel surface temperature by transferring heat to the fuel subsurface layers. This could also cause a larger melt layer.

Thrust data were collected but was not conclusive for making determinations about the differences between the fuel grain formulations. The LGCP hybrid rocket motor was modified and tested numerous times, validating its use as a functional apparatus for testing and comparing new fuel formulations while consuming minimal resources. Modifications to the motor and overall setup included: addition of upstream graphite filler to accommodate shortened solid-fuel grains, relocation of check valves, relocation of pressure transducers, relocation of the calibrated orifice, and the inclusion of a new downstream transition section which acted as the post-combustion mixing chamber. The system also includes an updated control system based in LabVIEW which includes an igniter trigger signal to sync ignition data between multiple runs.

5.3 Future Work

Many parts of this study can be continued to further develop and advance the solid-fuel formulations. Outlined below are several important elements of future work to improve the fuel formulations described in this investigation.

1) Test additional solid-fuel grains containing LiAlH₄. Fuel grains with ball-milled particles should be cast with 20% additive loading as well as various other percentages. In addition, grains should be cast without ball milling the particles to see if running the LGCP at higher O/F values is what contributed to the reduction in accumulated fuel in downstream sections of the hybrid rocket motor.

2) Cast new grains for use in the larger X-ray Transparent Center-perforated (XTC) hybrid rocket motor located at PSU’s High Pressure Combustion Laboratory. The
real time regression rate analysis will allow for accurate determination of regression rate correlations.

3) Continue to manipulate casting procedures to try to incorporate RDX in paraffin wax, or explore the possibility of using other energetic/nano-sized additives in the paraffin wax in order to increase the fuel density and regression rate.

4) Generate small fuel samples with MWNT for mechanical testing.

5) Cast new grains with the same additive percentage of carbon black as MWNT to determine any differences in the solid-fuel regression rate.

6) Improve sonication processes and/or mixing processes to reduce clumping of MWNT in the paraffin wax. One possible way of doing this would be to use a wand sonication device for in situ sonication of the MWNT in paraffin. This would generate uniform dispersion of the MWNT. Otherwise, methods for sonicating the melted paraffin/MWNT in a bath sonicator may need to be developed.
References


Appendix A

CEA2 Sample Input Files

CEA2 Input File for 10% LiAlH₄ in Paraffin

!LAH/Paraffin 90 Hybrid
problem
  rocket equilibrium
  p, psia=1000
  o/f=0.1, 0.3, 0.5, 0.7, 0.9, 1.1, 1.3, 1.5, 1.7, 1.9, 2.1, 2.3, 2.5, 2.7, 2.9, 3.1, 3.3, 3.5, 3.7, 3.9, 4.1, 4.3, 4.4, 4.7, 4.9, 5.1, 5.3, 5.5
  sup, ae/at=10
react
  fuel=LAH Li 1 Al 1 H 4  h, cal/mole=-27980.8  wt%=10
  oxid=02  wt%=100
  fuel = Paraffin  C 32 H 66  h, cal/mole= -231650.0
  wt%=90  t(k)=298.15
output  plot isp
end
CEA2 Input File 8 grams of RDX burning to complete in environmental chamber

```
problem  
   uv   rho,kg/m**3=.022, t,k=3800  
react    
   name=RDX  wt=100  t,k=298  C 3 H 6 N 6 O 6  
end
```

**Summary of RDX Combustion Results:**

The output file from CEA2 showed that a total pressure increase from the burning inside the calculated volume of the environmental chamber was less than 4 psi. Although the environmental chamber is only rated to 2 psi, it was speculated that the burning would not cause a catastrophic chamber failure, but more likely, the chamber windows would be the failure mode. Therefore, special precautions were taken (i.e., remote operation and covering unused windows) to ensure the safety of all test personnel.
Appendix B

MWNT Standard Operating Procedure

PROCEDURE FOR PREPARING PARAFFIN IMPREGNATED WITH MULTI-WALL CARBON NANOTUBES (MWNT) FOR LGCP CASTING

High Pressure Combustion Laboratory (HPCL)
Department of Mechanical and Nuclear Engineering
The Pennsylvania State University

Written by: Trevor Wachs
Research Assistant
Date: __________________________

Edited by: Daniel Larson
M.S. Candidate
Date: __________________________

Reviewed by: J. Eric Boyer
Senior Researcher
Date: __________________________

June 12, 2012
THE PENNSYLVANIA STATE UNIVERSITY
HIGH PRESSURE COMBUSTION LABORATORY
UNIVERSITY PARK, PA 16801
STANDARD OPERATING PROCEDURE  

12 JUNE, 2012

VERSION: NSRTF 2

SAFETY

Purpose
To establish safe operating procedures associated with casting solid fuels and assign responsibilities for the operation of the Long-Grained Center-Perforated hybrid rocket motor casting equipment.

Scope
This SOP applies to operations associated with preparation of fuel mixtures containing MWNT. Operations that are not adequately covered by this SOP will be addressed in a separate SOP or a supplement to this SOP.

Applicability
This SOP applies to all Penn State, HPCL, and visiting personnel and all other personnel assigned to support the operation of the LGCP casting experiment.

Responsibility
A. The Director of the High Pressure Combustion Laboratory and Distinguished Professor of Mechanical Engineering, Dr. Kenneth K. Kuo, is responsible for the overall enforcement of this SOP. Dr. Kuo is further responsible that only trained personnel are assigned to these operations and that all personnel are informed of all of the hazards associated with this experimental procedure.

B. The Experimentalist/Researcher/Graduate Student (E/R/GS) is responsible for application and enforcement of this SOP and overall on-site supervision of the experiments, to include taking necessary actions to protect all personnel, equipment, and facilities from any blast, fragmentation, or fire resulting from an experiment under his (or her) control. The E/R/GS is responsible that all personnel assigned to the testing programs have been adequately trained. In addition, prior to any operation, they have been thoroughly briefed on duties, responsibilities, and hazards that may be involved.

Personnel Limits
A. Personnel will be limited to the minimum required for safe and efficient operation of each cast.
B. A minimum of two trained persons must be present for any hazardous operation.
C. Observing personnel will be limited to those having an official interest in the experiment.

Hazardous Material Limits

Hazardous material at the fuel processing site will be limited to the amount necessary for safe and efficient operation of the program. All material not involved in the current test will be adequately protected and located so that it is not exposed to any personnel.

General Safety Requirements

A. Solid-fuel preparations will be conducted in the Chemical Room, Annex, and the machine shop (Room 123).
B. Heat and other sources of ignition will be kept away from the solid-fuel grains.

Personal Protective Equipment Requirements

A. Safety glasses and/or face shield will be worn for all eye hazard operations.
B. Face masks (rated for nano particles) will be worn whenever fine particulates are being mixed or measured.
C. Nitrile gloves and lab jacket will be used whenever handling MWNT.
D. High-temperature gloves will be used whenever working with molten paraffin or paraffin fuel mixtures.

Sequence of Operations

**SAFETY:** Utilize the necessary safety gear for each respective section of the procedure. To conduct any part of this procedure, protective eyewear, a lab coat, long pants, and shoes that cover the entire foot are REQUIRED. Consult the lab safety manual as frequently as needed. However, each section may have specific safety precautions, which will be noted at the beginning of that section.

1. **Multi-Wall Nanotubes (MWNT) / Paraffin Fuel Preparation:**
   1.1. Verify heat-transfer-fluid (silicone oil) level is above the bend in the heating element, approximately 1” from the top of the pot.
   1.2. Turn on heater, bath stirrer, and pump for mixing system
   1.2.1. Heater temperature and variable transformer settings should be recorded and used to determine settings for future tests. Currently 80% on the variable transformer and 120 °C on the heating element does not result in local boiling.
   1.2.2. During operation, ensure that heating fluid temperature, as measured by the thermocouple installed in the base of the mixing bowl does not exceed 130 °C.
   1.3. As pot temperature approaches 80 °C, add pre-massed paraffin.
   1.4. Ensure paraffin does not exceed 100 °C but is above 80 – 90 °C as measured by a thermometer inserted directly into the paraffin.
   1.5. Refer to the attached MSDS for Toluene and MWNT.
      1.5.1. When working with open particulate MWNT, dust mask, lab jacket, nitrile gloves, and safety glasses must be worn.
1.6. Tare a small plastic sample bowl on the scale in the Chemical Room.
1.7. Using a sample stirrer, add compacted MWNT powder to sample tray until desired mass is obtained, and put aside. DO NOT RETURN EXCESS POWDER INTO THE ORIGINAL CONTAINER, dispose of properly.
1.8. Fill sonication bath with water.
1.9. Tare glass beaker of adequate size for the required toluene (typical ratio is 100 mL for 1 – 2 grams of MWNT)
1.10. Add toluene into beaker until the desired volume is reached. DO NOT RETURN EXCESS LIQUID INTO THE ORIGINAL CONTAINER, dispose of properly.
1.11. Slowly add MWNT into toluene while mixing with flat sample stirrer. Ensure all of the nanotubes are added.
1.12. Place beaker container in water sonication bath and use electrical tape to balance/support the beaker in the bath. Bottom of beaker should be level with the bottom of the sonication bath.
1.13. Turn on sonicator and run for 15 minutes. DO NOT RUN LONGER: TOLUENE BREAKDOWN MAY RESULT.
1.14. Recheck paraffin temperature,
   1.14.1. If temperature is 100 ± 5 °C
       1.14.1.1. Add toluene/MWNT mixture while slowly stirring the paraffin. Safety glasses, face shield and lab coat must be worn.
   1.14.2. If temperature is outside of this range (100 ± 5 °C)
       1.14.2.1. Adjust heater settings to ensure the paraffin temperature is returned to this range.
1.15. Using the chamber gloves covered with Silver Shield, or other toluene compatible over-gloves, turn on the mixer to low speed (setting 2), pour in toluene/MWNT mixture, and confirm the integration of the paraffin and toluene/MWNT mixture.
1.16. Close chamber door, ensure all environmental exhaust valves are open, and open nitrogen purge bottle.
   1.16.1. Set regulator until flow is confirmed in exhaust line. NOTE: The environmental chamber is not rated as a high pressure vessel, so simply confirm flow of N₂ purge gas.
1.17. Increase heater temperature until inlet thermocouple temperatures read in the range of 120 to 130 °C.
1.18. Open Annex doors and run fans to promote air circulation.
1.19. Mix for 30 minutes, or until odor of toluene has left the Annex.
1.20. Turn off mixer, heater, and pump.
1.21. Secure the N₂ purge, keep the outlet valve open
1.22. Ensure the pressure gauge on the outlet valve has returned to zero
1.23. While standing away from the door and seal, open a latch close to door hinge.
1.25. Continue to open latches slowly, leaving the furthest from the hinge for last
1.26. When opening the last latch, stand outside of the door opening arc.
1.27. Continue with LGCP casting procedure (available at the HPCL) as normal; refer to this procedure for all subsequent pours.
Appendix C

Paraffin/RDX Casting Hazard Analysis

Hazard Analysis

of

RDX Casting Procedures in Paraffin Wax

at the

High Pressure Combustion Laboratory

The Pennsylvania State University

Project Name: NASA (NSTRF) RDX CASTING
Prepared by: Dan Larson, Trevor Wachs
Version: DBL.01

Reviewed and Approved by:

Director of the HPCL: ______________________

HPCL Safety Officer: ______________________
<table>
<thead>
<tr>
<th>Hazard No.</th>
<th>Energy Source and Hazard Description</th>
<th>Potential Effects</th>
<th>Potential Causal Factors</th>
<th>Initial Risk</th>
<th>Recommended Control Mechanisms/Barriers</th>
<th>Final Risk</th>
<th>Status</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>RDX Overheating During Casting</td>
<td>Decomposition of RDX</td>
<td>Allowing Casting Equipment to Overheat</td>
<td>III-D</td>
<td>Use oil which boils above 135 degrees C and Variac Control</td>
<td>III-E</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>RDX</td>
<td>Serious Injury, Death, Loss of Equipment</td>
<td>Sparking, shocking, excess friction, causing accidental ignition of RDX crystals</td>
<td>I-D</td>
<td>Wear appropriate PPE and handle RDX according to state and federal safety regulations</td>
<td>I-E</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>Dimethylformamide (DMF) Exposure</td>
<td>Injury</td>
<td>Spilled DMF on user, inhalation of excess fumes</td>
<td>III-D</td>
<td>Wear proper PPE and adhere to MSDS guidelines for handling and storage, limit exposure, properly vent environmental chamber and cryo trap</td>
<td>IV-D</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>Ignition of DMF</td>
<td>Loss of equipment, injury</td>
<td>Exposing DMF to ignition source or static electricity</td>
<td>III-D</td>
<td>Wear proper PPE and adhere to MSDS guidelines for handling and storage, keep away from ignition sources, ground equipment and containers</td>
<td>IV-D</td>
<td></td>
</tr>
<tr>
<td>Hazard No.</td>
<td>Energy Source and Hazard Description</td>
<td>Potential Effects</td>
<td>Potential Causal Factors</td>
<td>Initial Risk</td>
<td>Recommended Control Mechanisms/Barriers</td>
<td>Final Risk</td>
<td>Status</td>
</tr>
<tr>
<td>-----------</td>
<td>-------------------------------------</td>
<td>-------------------</td>
<td>-------------------------</td>
<td>--------------</td>
<td>-----------------------------------------</td>
<td>------------</td>
<td>--------</td>
</tr>
<tr>
<td>5</td>
<td>Overheating in vacuum chamber pump</td>
<td>Loss of equipment</td>
<td>Insufficient oil in motor</td>
<td>IV-D</td>
<td>Check and maintain proper oil level in motor</td>
<td>IV-E</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>Hot paraffin casting equipment</td>
<td>Minor burns</td>
<td>Touching equipment while casting fuel</td>
<td>IV-C</td>
<td>Wear heat resistant gloves, and safety apparel when working around equipment</td>
<td>IV-D</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>Hot paraffin</td>
<td>Minor burns</td>
<td>Spilling of paraffin onto skin</td>
<td>IV-C</td>
<td>Wear heat resistant gloves and safety apparel when working with hot paraffin</td>
<td>IV-D</td>
<td></td>
</tr>
<tr>
<td>8</td>
<td>Lathe spinning</td>
<td>Injury, death, loss of equipment</td>
<td>Parts not secured to lathe while spinning, individual injured by moving parts</td>
<td>I-C</td>
<td>Ensure all parts are secured to lathe, read manual, stay away from moving parts</td>
<td>I-E</td>
<td></td>
</tr>
<tr>
<td>Hazard No.</td>
<td>Energy Source and Hazard Description</td>
<td>Potential Effects</td>
<td>Potential Causal Factors</td>
<td>Initial Risk</td>
<td>Recommended Control Mechanisms/Barriers</td>
<td>Final Risk</td>
<td>Status</td>
</tr>
<tr>
<td>-----------</td>
<td>--------------------------------------------------------------------------</td>
<td>-------------------------------------------</td>
<td>----------------------------------------------------------------------------------------</td>
<td>--------------</td>
<td>----------------------------------------------------------------------------------------------------------------</td>
<td>------------</td>
<td>--------</td>
</tr>
<tr>
<td>9</td>
<td>Electric shock from instrumentation / casting equipment</td>
<td>Personal injury, death</td>
<td>Exposed and energized power lines</td>
<td>I-D</td>
<td>Keep power lines covered, keep power off until ready for use, keep environment dry</td>
<td>I-E</td>
<td></td>
</tr>
<tr>
<td>10</td>
<td>Static build up</td>
<td>Equipment damage, ignition of RDX/Paraffin Grain</td>
<td>Discharge through instrumentation, clothing static buildup</td>
<td>III-C</td>
<td>Discharge static to ground prior to handling fuel or casting materials</td>
<td>IV-C</td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>Friction from removal of center mandrill and end caps</td>
<td>Ignition of RDX/Paraffin fuel grain</td>
<td>Removing End caps with twisting motion, spinning out center mandrill</td>
<td>III-D</td>
<td>Avoid overfilling phenolic tube so that mandrill removes easily, coat end caps with mold release and avoid twisting</td>
<td>III-E</td>
<td></td>
</tr>
<tr>
<td>12</td>
<td>Friction and/or sparking when cutting fuel casing and fuel</td>
<td>Ignition of RDX/Paraffin fuel grain</td>
<td>Cutting grain using hacksaw or band saw, facing fuel grain while spinning on lathe</td>
<td>III-D</td>
<td>Avoid blade overheating, spin grain on lathe at lower RPM (&lt; 500), always check RDX particle distribution before excessive machining</td>
<td>III-E</td>
<td></td>
</tr>
</tbody>
</table>
Table C-1. Hazard review mandatory mishap checklist

<table>
<thead>
<tr>
<th>Fuels and propellants</th>
<th>Actuating devices</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical initiators</td>
<td>Noise or vibrations</td>
</tr>
<tr>
<td>Explosive charges</td>
<td>High velocities</td>
</tr>
<tr>
<td>Chemical reactions</td>
<td>Contamination</td>
</tr>
<tr>
<td>Electrical current</td>
<td>Unbalanced objects</td>
</tr>
<tr>
<td>Shock (mechanical)</td>
<td>Excessive friction</td>
</tr>
<tr>
<td>Stored electrical sources</td>
<td>Penetrating or lacerating surfaces</td>
</tr>
<tr>
<td>Energy in any form</td>
<td>Moving mechanisms or machinery</td>
</tr>
<tr>
<td>High concentrations of toxicity</td>
<td>Condition which can lead to fire</td>
</tr>
<tr>
<td>Stress reversals or concentrations</td>
<td>Structural weakness, damage, or failure</td>
</tr>
<tr>
<td>Radiation sources</td>
<td>Incompatible chemicals</td>
</tr>
<tr>
<td>Heat or toxicity sources</td>
<td>High levels of noise</td>
</tr>
<tr>
<td>Oxidation/corrosion</td>
<td>High levels of vibrations</td>
</tr>
<tr>
<td>Moisture conditions</td>
<td>Objects that can move or fall</td>
</tr>
<tr>
<td>OSHA violations</td>
<td>Static-generating sources</td>
</tr>
<tr>
<td>Spring-loaded devices</td>
<td>Mechanical power sources</td>
</tr>
<tr>
<td>Objects that can be catapulted</td>
<td>Leakage of fluids or electrical current</td>
</tr>
<tr>
<td>Objects that can disintegrate</td>
<td>Condition which can lead to explosion</td>
</tr>
<tr>
<td>Falling, moving, or catapulted objects</td>
<td>Hazards where controls may have been removed</td>
</tr>
<tr>
<td>Elements which have become defective</td>
<td>Hazards which personnel might be exposed</td>
</tr>
<tr>
<td>Initiation sensitivity</td>
<td></td>
</tr>
<tr>
<td>Rate of burning or deflagration</td>
<td></td>
</tr>
<tr>
<td>Protection capabilities of shields</td>
<td></td>
</tr>
<tr>
<td>Prevent propagation due to blast overpressure</td>
<td></td>
</tr>
<tr>
<td>Possible spillage of reactive materials</td>
<td></td>
</tr>
<tr>
<td>Pressure Sources high pressure</td>
<td>Hazardous Pressures unusually high pressure</td>
</tr>
<tr>
<td>low pressure</td>
<td>unusually low pressure</td>
</tr>
<tr>
<td>dynamic pressure</td>
<td>unusually dynamic pressure</td>
</tr>
<tr>
<td>Electrical Conditions energized surfaces</td>
<td>Hazardous Temperatures unusually high temperatures</td>
</tr>
<tr>
<td>inadvertent activation</td>
<td>unusually low temperatures</td>
</tr>
<tr>
<td>power source failure</td>
<td>unusually dynamic temperatures</td>
</tr>
</tbody>
</table>
Figure C-1. Hazard analysis procedural flow chart
Table C-2. Risk assessment levels

<table>
<thead>
<tr>
<th>Mishap Risk Assessment Value</th>
<th>Mishap Risk Category</th>
<th>Suggested Criteria</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 – 5</td>
<td>High Risk</td>
<td>Unacceptable (Director of the HPCL Review and Decision Required)</td>
</tr>
<tr>
<td>6 – 9</td>
<td>Serious Risk</td>
<td>Undesirable (Director of the HPCL Decision Required)</td>
</tr>
<tr>
<td>10 – 17</td>
<td>Medium Risk</td>
<td>Acceptable (with Safety Officer’s Review)</td>
</tr>
<tr>
<td>18 – 20</td>
<td>Low Risk</td>
<td>Acceptable</td>
</tr>
</tbody>
</table>

Table C-3. Risk Assessment Matrix

<table>
<thead>
<tr>
<th>HAZARD SEVERITY</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>1</td>
<td>2</td>
<td>4</td>
<td>8</td>
<td>12</td>
</tr>
<tr>
<td>II</td>
<td>3</td>
<td>5</td>
<td>6</td>
<td>10</td>
<td>13</td>
</tr>
<tr>
<td>III</td>
<td>7</td>
<td>9</td>
<td>10</td>
<td>14</td>
<td>17</td>
</tr>
<tr>
<td>IV</td>
<td>13</td>
<td>16</td>
<td>18</td>
<td>19</td>
<td>20</td>
</tr>
</tbody>
</table>

HAZARD PROBABILITY

The table shows a risk assessment matrix where the hazard probability and severity are categorized and rated accordingly. The matrix helps in determining the risk level and the appropriate action to be taken based on the assessment.
Table C-4. Hazard severity

<table>
<thead>
<tr>
<th>Category</th>
<th>Description</th>
<th>Environmental, Safety, and Health Result Criteria</th>
</tr>
</thead>
<tbody>
<tr>
<td>I</td>
<td>Catastrophic</td>
<td>Could result in death, permanent total disability, loss exceeding $1M, or irreversible severe environmental damage that violates law or regulation</td>
</tr>
<tr>
<td>II</td>
<td>Critical</td>
<td>Could result in permanent partial disability, injuries or occupational illness that may result in loss exceeding $200K but less than $1M, hospitalization of at least 3 personnel, or reversible environmental damage causing a violation of law or regulation</td>
</tr>
<tr>
<td>III</td>
<td>Marginal</td>
<td>Could result in injury or occupational illness resulting in 1 or more lost work day(s), loss exceeding $10K but less than $200K, or mitigable environmental damage without violation of law or regulation where restoration activities can be accomplished</td>
</tr>
<tr>
<td>IV</td>
<td>Negligible</td>
<td>Could result in injury or illness not resulting in a lost day of work, loss exceeding $2K but less than $10K, or minimal environmental damage not violating law or regulation</td>
</tr>
</tbody>
</table>

Table C-5. Hazard probability

<table>
<thead>
<tr>
<th>Category</th>
<th>Description</th>
<th>Specific Individual Item</th>
<th>Probability</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Frequent</td>
<td>Likely to occur often in the life of an item</td>
<td>( P \geq 10^{-1} )</td>
</tr>
<tr>
<td>B</td>
<td>Probable</td>
<td>Will occur several times in the life of an item</td>
<td>( 10^{-2} \leq P \leq 10^{-1} )</td>
</tr>
<tr>
<td>C</td>
<td>Occasional</td>
<td>Likely to occur some time in the life of an item</td>
<td>( 10^{-3} \leq P \leq 10^{-2} )</td>
</tr>
<tr>
<td>D</td>
<td>Remote</td>
<td>Unlikely but possible to occur in the life of an item</td>
<td>( 10^{-6} \leq P \leq 10^{-3} )</td>
</tr>
<tr>
<td>E</td>
<td>Improbable</td>
<td>So unlikely, it can be assumed occurrence may not be experienced</td>
<td>( P \leq 10^{-6} )</td>
</tr>
</tbody>
</table>
Figure C-2. Hazard identification example #1

Identify Potential Energy Sources and Hazards

- Identify Potential Causal Factors of Hazard
- Potential Effects Caused by Hazard to Operators or Equipment
- Identify Severity Level of Hazard
- Identify Initial Risk of Hazard
- Identify Probability of Hazard Occurrence
- Acceptable

Evaluate Initial Risk of Hazard

Recommend Control Mechanisms or Barriers

Re-assess Severity Level of Hazard

Re-assess Probability of Hazard Occurrence

Assess Final Risk of Hazard

Evaluate Acceptable Risk of Hazard

overpressurization in pot

- pressure vent was closed
- lid blows off and injures chef or damages fan above stove

II: critical

B: probable

RAL = 5 unacceptable without restaurant owner's review

verify vent on lid cannot close and make sure lid does not seal on pot

II-D

D: remote

RAL = 10 unacceptable without kitchen manager's review

hot outer surface of pot

- pot is being heated
- serious burns to chef

III: marginal

C: occasional

RAL = 11 unacceptable without kitchen manager's review

chef wears protective gloves or mitts when handling pot

IV: negligible

C: occasional

RAL = 18 acceptable

Figure C-3. Hazard identification example #2
Figure D-1. New post combustion transition section for downstream section of LGCP.
Figure D-2. Example of upstream graphite filler piece (4-inch length shown).
Appendix E

LCGP Hybrid Rocket Motor Test Checklist and Data Sheet

CHECKLIST FOR LGCP MOTOR FIRING

Prior to testing:

☐ Verify sufficient reserves of nitrogen and oxygen in bottles.

☐ Ensure all shop air is connected to all remotely operated valves and greater than 90 psig in room 127.

☐ Title the VHS video display and set VHS tape to a new recording section.

☐ Mount outdoor test camera, install A/V lines

☐ Set up exhaust fan to blow exhaust products out of test cell.

☐ Ensure all pressure transducers are connected to patch panel.

☐ Plug in power supply to patch panel

☐ Make sure igniter circuit is disconnected from electric match and match leads are twisted together. Length of igniter wire should be 1.75” Plus length of paper phenolic before paraffin

☐ Ensure the proper size burst disk is installed.

☐ Remove Load Block/ ensure Test deck is restrained to load cell.

☐ Ensure oxygen pintle on venturi is set to 2” (or full open).

☐ Ensure Nitrogen motor purge line ball valve is aligned to LGCP.

☐ Ensure all pressure lines and network cable are connected and turn on Nicolet Vision.

☐ Verify sampling rate is 1ks/s.

☐ Verify Pressure transducer signals on Nicolet Vision are around 0 psig +/-5.

☐ With the board off, open the control program, Hybrid Slab Motor Control V.4 and name the file.

☐ Verify that program is in “Automatic” mode.
☐ Turn on control board and ensure all valve indication and fuse lights are lit.

☐ Ensure Oxygen run valve and nitrogen purge are shut.

**Testing:**

☐ Ensure all non-testing personnel are clear from the test cell and fenced-in area.

☐ Open nitrogen bottle valves for purge.

☐ Set nitrogen regulator for purge to amount specified on test data sheet.

☐ Open nitrogen bottle attached to oxygen regulator.

☐ Verify oxygen line hand valve and all cluster hand valves are open (and hand valves leading to other systems are shut)

☐ **Slowly** open oxygen bottle valves.

☐ Set Oxygen regulator to value written on test data sheet.

☐ Make an announcement for starting the test firing: “We will be conducting a motor firing in room 127. Please stand clear of the test cell and the fenced-in area.”

☐ Connect igniter lines to electric match leads.

☐ Check ALL timing parameters

☐ Press “Record” on the VCR and start timer on title screen.

☐ Press “Record” on the Nicolet Vision.

☐ Ensure all lights necessary for LGCP test are illuminated.

☐ Use the control program to initiate the test.

**************************TEST INITIATED**************************

☐ If misfire occurs, stop using this portion of checklist and move onto the Misfire Section.

**************************TEST CONCLUDED**************************

☐ Press “Stop” on VCR and data acquisition systems.

☐ Check that the Oxygen run and manifold valves have shut.
Close Oxygen bottles at the cluster.

Announce “We will now be venting gases in room 127, all personnel stay clear”.

Using the manual mode of operation of the LabVIEW Control Program, open the oxygen run and manifold valves and allow to vent.

Close N\textsubscript{2} bottle in chamber purge line and vent using LabVIEW control program.

Announce “Test completed in room 127”.

Test cell may now be entered.

**Misfire Procedure:**

Immediately turn off Hybrid control Panel by turning key; This will start nitrogen flow and interrupt oxygen flow.

Announce “Misfire has occurred in room 127, stay clear of the test cell and fenced in area until directed otherwise”.

Continue nitrogen flow for 5-10 seconds.

On the Hybrid Control Rev 2 control program; Restart and turn on manual mode and ensure all relays are in the shut position.

Turn oxygen valve switches on the HCP to the off position.

Turn on control panel. Verify all flows have stopped and no smoldering is observed. If smoldering is observed, continue to purge the system.

Isolate Oxygen by closing oxygen bottles at the cluster.

Continue recording event on handy cam.

Press “Stop” on Nicolet vision.

Record time of misfire.

Verify combustion has subsided via handy-cam.
☐ After 10 minutes personnel may enter test chamber.

☐ Announce “Test completed in room 127”.

☐ Gather evidence and determine cause of problem.
LGCP TEST DATA SHEET

<table>
<thead>
<tr>
<th>Aero Test #</th>
<th>Date</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### Test Parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oxidizer flow est.</td>
<td>g/s</td>
</tr>
<tr>
<td>Act.</td>
<td></td>
</tr>
<tr>
<td>O2 Reg. Press.</td>
<td>psi</td>
</tr>
<tr>
<td>O2 Pintle Pos.</td>
<td>in</td>
</tr>
<tr>
<td>Fuel assembly mass</td>
<td></td>
</tr>
<tr>
<td>O2 Mass flow rate</td>
<td>g/s</td>
</tr>
<tr>
<td>Orifice size/CD</td>
<td>in</td>
</tr>
<tr>
<td>O2 Flux</td>
<td>kg/(m²*s)</td>
</tr>
<tr>
<td>Test duration est</td>
<td>s</td>
</tr>
<tr>
<td>Fuel Length</td>
<td>in</td>
</tr>
<tr>
<td>Bore Preburn</td>
<td>in (dia)</td>
</tr>
<tr>
<td>Nozzle size</td>
<td>in (dia)</td>
</tr>
<tr>
<td>Bore Postburn</td>
<td>in (dia)</td>
</tr>
<tr>
<td>N2 Purge Press.</td>
<td>psi</td>
</tr>
<tr>
<td>O2 Manifold</td>
<td>psi</td>
</tr>
<tr>
<td>N2 Purge Bottle</td>
<td>psi</td>
</tr>
</tbody>
</table>

### Data Acquisition

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sampling rate</td>
<td>sec-1</td>
</tr>
<tr>
<td>DAQ file name</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Pressure #1</th>
<th>Channel</th>
<th>Color</th>
<th>Pressure #3</th>
<th>Channel</th>
<th>Color</th>
<th>Pressure #4</th>
<th>Channel</th>
<th>Color</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Channel</td>
<td></td>
<td></td>
<td>Channel</td>
<td></td>
<td></td>
<td>Channel</td>
<td></td>
</tr>
<tr>
<td>Pressure #2</td>
<td>Channel</td>
<td>Color</td>
<td>Pressure #3</td>
<td>Channel</td>
<td>Color</td>
<td>Pressure #4</td>
<td>Channel</td>
<td>Color</td>
</tr>
<tr>
<td></td>
<td>Channel</td>
<td></td>
<td></td>
<td>Channel</td>
<td></td>
<td></td>
<td>Channel</td>
<td></td>
</tr>
</tbody>
</table>

### Comments

<p>| |</p>
<table>
<thead>
<tr>
<th></th>
</tr>
</thead>
</table>

### Test Personnel

<p>| |</p>
<table>
<thead>
<tr>
<th></th>
</tr>
</thead>
</table>
Appendix F

Error Analysis

The error analysis in this investigation primarily follows the error analysis described in the Ph.D. dissertation of Risha [50], which follows the method prescribed by Kline and McClintok [51], outlined below in Equation F.1. Uncertainties of the various independent parameters were estimated and are summarized in Table F-1.

\[
\delta Q = \sqrt{\left(\frac{\delta Q}{\delta v_1}\right)^2 + \cdots + \left(\frac{\delta Q}{\delta v_n}\right)^2} \tag{F.1}
\]

Where:

- \( Q \) is the dependant quantity to which the uncertainty is applied
- \( v_i \) represents the independent parameter
- \( \delta v \) is the uncertainty related to \( v_i \)

<table>
<thead>
<tr>
<th>Uncertainty Parameter</th>
<th>Definition</th>
<th>Value</th>
<th>Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \delta L )</td>
<td>Fuel Length</td>
<td>0.004</td>
<td>Inches</td>
</tr>
<tr>
<td>( \delta m_i )</td>
<td>Initial Fuel Mass</td>
<td>0.5</td>
<td>Grams</td>
</tr>
<tr>
<td>( \delta m_f )</td>
<td>Final Fuel Mass</td>
<td>1.01</td>
<td>Grams</td>
</tr>
<tr>
<td>( \delta t_b )</td>
<td>Burn Time</td>
<td>0.05</td>
<td>Seconds</td>
</tr>
<tr>
<td>( \delta \rho )</td>
<td>Fuel Density</td>
<td>50</td>
<td>kg/m(^3)</td>
</tr>
<tr>
<td>( \delta D_i )</td>
<td>Initial Center Port Diameter</td>
<td>0.015</td>
<td>Inches</td>
</tr>
</tbody>
</table>

The linear regression rate, shown in Equation 3.3, requires uncertainty calculation from various terms including \( D_i \), as shown in Equation 3.4. These equations are duplicated as (F.3) and (F.4) [44].
The error for linear regression rate, $\delta r$, is shown below:

$$\partial r_b = \sqrt{\left(\frac{\partial r_b}{\partial D_i} \delta D_i\right)^2 + \left(\frac{\partial r_b}{\partial D_f} \delta D_f\right)^2 + \left(\frac{\partial r_b}{\partial t_b} \partial t_b\right)^2}$$  \hspace{1cm} (F.4)

Where:

$$\frac{\partial r_b}{\partial D_i} = \frac{-1}{2t_b}$$  \hspace{1cm} (F.5)

$$\frac{\partial r_b}{\partial D_f} = \frac{1}{2t_b}$$  \hspace{1cm} (F.6)

$$\frac{\partial r_b}{\partial t_b} = \frac{(D_f-D_i)}{2t_b^2}$$  \hspace{1cm} (F.7)

And, the error in final port diameter, $\delta D_f$, must be calculated, since final port diameter is not a measured value, but rather, a calculated value.

$$\partial D_f = \sqrt{\left(\frac{\partial D_f}{\partial m_i} \partial m_i\right)^2 + \left(\frac{\partial D_f}{\partial m_f} \partial m_f\right)^2 + \left(\frac{\partial D_f}{\partial t_b} \partial t_b\right)^2 + \left(\frac{\partial D_f}{\partial L} \partial L\right)^2 + \left(\frac{\partial D_f}{\partial \rho_{fuel}} \partial \rho_{fuel}\right)^2}$$  \hspace{1cm} (F.8)

Where:

$$\frac{\partial D_f}{\partial m_i} = \frac{4}{\pi L \rho_f} \frac{\sqrt{\frac{4}{\pi L \rho_f} + D_i^2}}{2}$$  \hspace{1cm} (F.9)

$$\frac{\partial D_f}{\partial m_f} = \frac{-4}{\pi L \rho_f} \frac{\sqrt{\frac{4}{\pi L \rho_f} + D_i^2}}{2}$$  \hspace{1cm} (F.10)
\[
\frac{\partial D_f}{\partial D_i} = \frac{2D_i}{\sqrt{\frac{4(m_i-m_f)}{\pi L \rho_f} + D_i^2}} \tag{F.11}
\]

\[
\frac{\partial D_f}{\partial L} = \frac{-4(m_i-m_f)}{\pi \rho f L^2} \tag{F.12}
\]

\[
\frac{\partial D_f}{\partial \rho_{\text{fuel}}} = \frac{4}{\pi L \rho f} \frac{m_i-m_f}{\pi L \rho f + D_i^2} \tag{F.13}
\]

Similarly, the error in mass burn rate can be determined from Equation 3.7, duplicated as Equation F.14 here.

\[
\dot{m}_b \approx \frac{m_i-m_f}{t_b} \tag{F.14}
\]

Error in fuel mass burning rate is shown below.

\[
\partial \dot{m}_b = \sqrt{\left(\frac{\partial \dot{m}_b}{\partial m_f}\right)^2 + \left(\frac{\partial \dot{m}_b}{\partial m_i}\right)^2 + \left(\frac{\partial \dot{m}_b}{\partial t_b}\right)^2} \tag{F.15}
\]

Where:

\[
\frac{\partial \dot{m}_b}{\partial m_i} = \frac{1}{t_b} \tag{F.16}
\]

\[
\frac{\partial \dot{m}_b}{\partial m_f} = \frac{-1}{t_b} \tag{F.17}
\]

\[
\frac{\partial \dot{m}_b}{\partial t_b} = \frac{(m_i-m_f)}{t_b^2} \tag{F.18}
\]
**Appendix G**

**Batch 2 Test Notes**

Table G-1. Post-test comments on fuel grains, motor components, and test data [40].

<table>
<thead>
<tr>
<th>Fuel Grain</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure Paraffin</td>
<td>Burnout to phenolic tube observed, resulted in useless regression data</td>
</tr>
<tr>
<td>Pure Paraffin</td>
<td>Burnout to phenolic tube observed, resulted in useless regression data</td>
</tr>
<tr>
<td>Pure Paraffin</td>
<td>Minimal wax delamination, some burning on outside of wax wall between phenolic and fuel, phenolic tube was charred</td>
</tr>
<tr>
<td>10% LiAlH₄</td>
<td>Buildup of unburned/unreacted fuel in nozzle assembly and graphite transition section, uniform post-test grain geometry</td>
</tr>
<tr>
<td>10% LiAlH₄</td>
<td>White powder (could be aluminum oxide particles) deposited on surface of remaining fuel grain, unburned/unreacted fuel deposited in nozzle and transition section</td>
</tr>
<tr>
<td>10% LiAlH₄</td>
<td>Burnout on one portion of motor, wax appears to have run down sides of grain, test was much longer than expected, unburned/unreacted fuel deposited in nozzle and transition section</td>
</tr>
<tr>
<td>15% LiAlH₄</td>
<td>Gap between unburned fuel and phenolic tube was observed on top portion of fuel grain, phenolic tube not charred in the gap region where there was delamination (it is believed that the molten fuel ran down the sides of the grain near end of test), unreacted fuel deposited in nozzle and transition section</td>
</tr>
<tr>
<td>15% LiAlH₄</td>
<td>Less delamination issues than previous testing, burning on outside of grain indicates possible gas path on outside of grain, unreacted fuel deposited in nozzle and transition section</td>
</tr>
<tr>
<td>20% LiAlH₄</td>
<td>Unreacted fuel deposited in nozzle and transition section</td>
</tr>
<tr>
<td>20% LiAlH₄</td>
<td>Pressure spike observed in data, grain results very consistent with previous test of 20% LAH grain, unreacted fuel deposited in nozzle and transition section</td>
</tr>
<tr>
<td>28% LiAlH₄</td>
<td>Particle streaks observed in plume (likely caused by the ejection of partially burned fuel additive particles), unreacted fuel deposited in nozzle and transition section</td>
</tr>
<tr>
<td>28% LiAlH₄</td>
<td>White spots (could be aluminum oxide particles) on grain, charring out outside of phenolic tube, unreacted fuel deposited in nozzle and transition section</td>
</tr>
<tr>
<td>10% TEA</td>
<td>Much less fuel deposited in nozzle and transition section, fuel deposited on transition section and nozzle not reactive with water, showing favorable and uniform post-burn grain geometry</td>
</tr>
<tr>
<td>10% TEA</td>
<td>Graphite transition section was broken when removed from LGCP motor and showed pitting on the inside of the graphite (possibly due to volatility of the TEA additive), grain burned more unevenly than previous TEA grain, transition section unusable for future tests</td>
</tr>
<tr>
<td>10% DAH</td>
<td>Delamination observed on part of unconsumed fuel grain, less fuel deposits on nozzle and transition section than previous tests with TEA or LAH additives</td>
</tr>
<tr>
<td>10% DAH</td>
<td>Similar behavior to previous DAH grain test</td>
</tr>
</tbody>
</table>
Table G-2. Post-test comments on fuel grains, motor components, and test data from high pressure tests (200 – 330 psig during stable burning) [41].

<table>
<thead>
<tr>
<th>Fuel Grain</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>15% LiAlH₄</td>
<td>Divot in front of fuel grain (minor issue) observed prior to test, post-test: upon removal from motor, a large piece of fuel fell to the ground indicating delamination from phenolic tube, pressure spikes in data, uneven burning seen from video recordings</td>
</tr>
<tr>
<td>20% LiAlH₄</td>
<td>Grain weighed more than it did the week before (possibly due to oxidation), grain delamination observed, white powder (could be aluminum oxide particles) deposited on surface of remaining fuel grain on downstream portion of grain, gas path observed between solid fuel and paper phenolic tube (indicative of certain degree of delamination)</td>
</tr>
<tr>
<td>28% LiAlH₄</td>
<td>Grain weighed more than it did the week before by 0.3 grams (possibly due to oxidation), particle streaks observed in plume (likely caused by the ejection of partially burned fuel additive particles), more uniform burning with little to no grain delamination, fuel grain web thickness is thicker on one side (not the bottom side) – this could be caused by a non-centered grain perforation during grain fabrication</td>
</tr>
<tr>
<td>10% TEA</td>
<td>Smaller nozzle used than other high pressure tests resulting in data which is not comparable, grain delaminated and curled on itself in a plastic like behavior (only on upstream side), burn appeared smooth on video recording</td>
</tr>
<tr>
<td>10% DAH</td>
<td>Even burning observed with no debinding, minimal fuel deposited on nozzle, grain appeared bubbly on surface (possibly due to reaction of remaining DAH during burnout), greenish particles on surface of recovered fuel grain</td>
</tr>
</tbody>
</table>
Appendix H

Batch 3 Test Notes and Data (Including HPCL-Manufactured Fuel Grains)

Table H-1. Test data and parameters from Batch 3 fuel grains and HPCL-manufactured fuel grains (Pressure values are taken from the chamber head end pressure transducer).

<table>
<thead>
<tr>
<th>Test #</th>
<th>Fuel Formulation</th>
<th>Fuel Grain Length (in)</th>
<th>Maximum Chamber Pressure (psig)</th>
<th>Steady Region Pressure (psig)</th>
<th>Average Oxidizer Flux, $G_{ox}$ (kg/m²-s)</th>
<th>Fuel Mass Loss (gms)</th>
<th>Test Duration (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NASA 1</td>
<td>Pure Paraffin</td>
<td>4.506</td>
<td>246</td>
<td>204</td>
<td>53.8</td>
<td>36.2</td>
<td>2.87</td>
</tr>
<tr>
<td>NASA 2</td>
<td>Pure Paraffin</td>
<td>4.5</td>
<td>249</td>
<td>192</td>
<td>60.4</td>
<td>37</td>
<td>2.74</td>
</tr>
<tr>
<td>NASA 3</td>
<td>Pure Paraffin</td>
<td>4.51</td>
<td>240</td>
<td>204</td>
<td>60.6</td>
<td>31.8</td>
<td>2.47</td>
</tr>
<tr>
<td>NASA 4</td>
<td>Pure Paraffin</td>
<td>4.5</td>
<td>249</td>
<td>204</td>
<td>65.6</td>
<td>33.6</td>
<td>2.33</td>
</tr>
<tr>
<td>NASA 5</td>
<td>4% MWNT</td>
<td>4.515</td>
<td>247</td>
<td>202</td>
<td>63.8</td>
<td>29.7</td>
<td>2.4</td>
</tr>
<tr>
<td>NASA 6</td>
<td>4% MWNT</td>
<td>4.518</td>
<td>240</td>
<td>203</td>
<td>66.9</td>
<td>29.5</td>
<td>2.31</td>
</tr>
<tr>
<td>NASA 7</td>
<td>4% MWNT</td>
<td>4.52</td>
<td>260</td>
<td>202</td>
<td>67.1</td>
<td>29.7</td>
<td>2.31</td>
</tr>
<tr>
<td>NASA 8</td>
<td>Pure Paraffin</td>
<td>4.015</td>
<td>464</td>
<td>337</td>
<td>49.4</td>
<td>27.6</td>
<td>2.67</td>
</tr>
<tr>
<td>NASA 9</td>
<td>Pure Paraffin</td>
<td>4.027</td>
<td>300</td>
<td>256</td>
<td>52.8</td>
<td>30.6</td>
<td>2.59</td>
</tr>
<tr>
<td>NASA 10</td>
<td>Pure Paraffin</td>
<td>5.01</td>
<td>346</td>
<td>238</td>
<td>78.7</td>
<td>46.0</td>
<td>2.31</td>
</tr>
<tr>
<td>NASA 11</td>
<td>Pure Paraffin</td>
<td>5.5</td>
<td>374</td>
<td>314</td>
<td>95.6</td>
<td>50.3</td>
<td>2.03</td>
</tr>
<tr>
<td>AERO 31</td>
<td>5% LiAlH₄</td>
<td>4.006</td>
<td>407</td>
<td>349</td>
<td>55.7</td>
<td>28.0</td>
<td>2.48</td>
</tr>
<tr>
<td>AERO 32</td>
<td>5% LiAlH₄</td>
<td>4.005</td>
<td>350</td>
<td>296</td>
<td>59.8</td>
<td>29.8</td>
<td>2.36</td>
</tr>
<tr>
<td>AERO 33</td>
<td>Pure Paraffin</td>
<td>4.002</td>
<td>303</td>
<td>260</td>
<td>59.6</td>
<td>26.5</td>
<td>2.24</td>
</tr>
<tr>
<td>AERO 34</td>
<td>10% LiAlH₄</td>
<td>4.011</td>
<td>298</td>
<td>266</td>
<td>51.9</td>
<td>28.3</td>
<td>2.3</td>
</tr>
<tr>
<td>AERO 35</td>
<td>Pure Paraffin</td>
<td>5.004</td>
<td>347</td>
<td>235</td>
<td>80.4</td>
<td>44.4</td>
<td>2.41</td>
</tr>
<tr>
<td>AERO 36</td>
<td>5% LiAlH₄</td>
<td>5.011</td>
<td>371</td>
<td>302</td>
<td>96.7</td>
<td>57.6</td>
<td>2.17</td>
</tr>
<tr>
<td>AERO 37</td>
<td>10% LiAlH₄</td>
<td>5.022</td>
<td>306</td>
<td>245</td>
<td>73.0</td>
<td>38.8</td>
<td>1.92</td>
</tr>
<tr>
<td>AERO 38</td>
<td>Pure Paraffin</td>
<td>5.506</td>
<td>315</td>
<td>240</td>
<td>114.5</td>
<td>40.1</td>
<td>1.73</td>
</tr>
<tr>
<td>AERO 39</td>
<td>10% LiAlH₄</td>
<td>5.499</td>
<td>330</td>
<td>252</td>
<td>99.5</td>
<td>42.7</td>
<td>1.69</td>
</tr>
</tbody>
</table>

Note: Highlighted grains in Table H-1 and H-2 represent tests with burnouts or a broken transition section, and tests NASA 1 through NASA 7 were run with O/F values between 1.5 and 1.75.
Table H-2. Test notes from Batch 3 fuel grains and HPCL-manufactured fuel grains (Pressure values are taken from the chamber head end pressure transducer).

<table>
<thead>
<tr>
<th>Test #</th>
<th>Test Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>NASA 1</td>
<td>Burning appeared uniform, upstream oxygen tapered off, LGCP system change necessary</td>
</tr>
<tr>
<td>NASA 2</td>
<td>Moved N₂ purge below orifice, but run may have been out of orifice calibration, still verifying system</td>
</tr>
<tr>
<td>NASA 3</td>
<td>Leak in oxygen line discovered, and again may have been out of orifice calibration</td>
</tr>
<tr>
<td>NASA 4</td>
<td>Chamber aft pressure transducer may have been clogged</td>
</tr>
<tr>
<td>NASA 5</td>
<td>Even burning, but again, issues with downstream (aft end) pressure transducer</td>
</tr>
<tr>
<td>NASA 6</td>
<td>Uniform burning</td>
</tr>
<tr>
<td>NASA 7</td>
<td>Uniform burning, small “blip” in pressure during exponential pressure decay at burnout</td>
</tr>
<tr>
<td>NASA 8</td>
<td>Entrance region created to eliminate some spongy, non-uniform wax at the end of the grain. Not uniform grain, broken transition section, and nozzle blocked</td>
</tr>
<tr>
<td>NASA 9</td>
<td>Test timing improving, purge pressure may have been too low, some charring noted on the outside of the phenolic tube</td>
</tr>
<tr>
<td>NASA 10</td>
<td>Some burn around (phenolic charring/burning), lower pressure than expected, burned almost entire web</td>
</tr>
<tr>
<td>NASA 11</td>
<td>Reduced timing by 0.1 seconds, broken transition section, some burning to phenolic wall</td>
</tr>
<tr>
<td>AERO 31</td>
<td>Transition section failure</td>
</tr>
<tr>
<td>AERO 32</td>
<td>Pressure history shows increase in pressure during burn (non linear), but stable combustion. Thin layer of fuel on transition section</td>
</tr>
<tr>
<td>AERO 33</td>
<td>Some small white specks noted in the fuel grain, stable burning observed</td>
</tr>
<tr>
<td>AERO 34</td>
<td>Grain center port was non-uniform, still tested, but at lower oxidizer flux</td>
</tr>
<tr>
<td>AERO 35</td>
<td>Very uniform post-test center port geometry</td>
</tr>
<tr>
<td>AERO 36</td>
<td>Some burning to phenolic wall, 1.9 grams of accumulation in downstream section, some places with measurable web, but assumptions of web thickness were made</td>
</tr>
<tr>
<td>AERO 37</td>
<td>Timing reduced by 0.3 seconds to account for we burnout, timing may have been off</td>
</tr>
<tr>
<td>AERO 38</td>
<td>Very uniform post-test center port geometry, some charring of the phenolic fuel cartridge, timing reduced by a total of 0.5 seconds (0.1 sec less than AERO 37)</td>
</tr>
<tr>
<td>AERO 39</td>
<td>Very uniform post-test center port geometry, even more charring of the phenolic fuel cartridge the AERO 38 test, timing reduced by a total of 0.6 seconds</td>
</tr>
</tbody>
</table>