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Report AFRPL-TR-75-10

INTEGRAL RAMJET BOOSTER DEMONSTRATION PROGRAM (U)

Donald A. Wiederecht

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Lockbeed Propulsion Company Redlands, CA 92373

February 1975

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Prepared for Air Force Rocket Propulsion Laboratory Director of Science and Technology Air Force Systems Command Edwards, California 93523





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(2) study and development of methods of accommodating a unique bond interface system which required radial outgassing holes in the silicone internal booster insulation and (3) laboratory evaluation of key aspects of the stress free viscous system (SVS) as they relate to the booster requirements. Design and fabrication of five analog motors and heavywall full scale motor hardware was also initiated prior to redirection of the program scope by AFRPL.

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Section 1

INTRODU_ FION

The hydrocarbon-fueled integral rocket/ramjet engine is a candidate propulsion system for a number of potential missile applications requiring operation over a wide Mach number-altitude envelope. The most demanding of these potential applications, from a propulsion standpoint, is the Advanced Strategic Air-Launched Missile (ASALM), formerly called the multi-purpose missile (MPM). Other applications for which the integral rocket/ramjet appears attractive include air-launched tactical missiles, air-to-ground strategic missiles, surface-launched defensive missiles, and a variety of air-to-air missiles.

This program, undertaken by Lockheed Propulsion Company for the U.S. Air Force under Contract F04611-74-C-0040, was designed to provide integral rocket motor booster propulsion technology specifically applicable to the ASALM and generally applicable to other future hydrocarbon-fueled, integral rocket/ramjet engine applications.

The ramburner configuration for the ASALM is primarily dictated by the volume required for the rocket booster. Since reduced propellant volume translates directly into reduced system size and weight, a high densityimpulse propellant is required. In addition to volume limitations, the transition from booster to ramjet operation is critical. Reproducible, rapid motor burnout and minimum combustibles in the ramburner after booster motor burnout are required to eliminate ramjet transition difficulties such as inlet unstarts or complicated ramjet fuel control devices. These requirements can be met by applying conventional rocket motor design practice to a casebonded, high thrust, low volume integral rocket ramjet booster. As an alternative to case bonding, the LPC Stress-free Viscous System (SVS) method of grain retention also provides an avenue to meeting these requirements.

The overall objective of the program was to develop and demonstrate a high-performance, low volume integral rocket booster for ramjet missile applications. To achieve this objective, a program consisting of three phases was undertaken.

Phase I, Design and Analysis, consisted of performance and configuration analyses of both the case-bonded and SVS motor approaches, followed by completion of a full flightweight motor design.

Phase II, Laboratory and Analog Motor Testing, consisted of laboratory evaluation and optimization of propellant, bond system, and fabrication techniques, followed by analog motor and age life verification of the ability of selected grain design, propellant and case bonding techniques to meet rocket operating criteria. Phase III, Heavywall and Flightweight Full-Scale Motor Design and Test, consisted of fabrication and test of three heavywall, full-scale motors to prove feasibility of the selected design, followed by 6 full-scale motors in flightweight hardware to demonstrate the design under varying environmental conditions.

The overall expected accomplishment from this program was the demonstration of a high-volumetric loaded, minimum-sliver booster ready for free jet and transition ramjet testing.

After the completion of Phase I and part of Phase II through the preparation of analog motors for casting, LPC announced its intention of ceasing business operations beyond mid calendar year 1975. Since this program and possible follow-on activity associated with transition free-jet testing project beyond that date, the program was redirected by RPL to stop technical activity following completion of short-term laboratory specimen testing. Accordingly, the report contained herein will cover those portions of the original program, as defined within the scope of the redirected program.

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Section 2

SUMMARY

(U) Lockheed Propulsion Company undertook an 18-month program to complete the Air Force objectives to develop and demonstrate an integral rocket booster for ramjet missile applications. The program consisted of a 15month technical effort, with 3 months for final report preparation and publication. The critical technology to the integral booster development is the materials system, which must interface with the selected silicone elastomer ramjet combustor insulator. Key technical questions are the ability to bond booster propellant to this insulation and compatibility over extended periods of storage, with rocket motor internal materials that do not leave significant residuals to impair ramjet start-up operation. Program logic is shown in Figure 2-1.

(U) Phases I and II were initiated concurrently to provide an integrated analytical and laboratory evaluation of design and materials system approaches. Although case bonding is the preferred approach, both case-bonded and the LPC Stress-free Viscous System (SVS) methods of grain retention were evaluated during this portion of the program. Case bonding was the primary approach with SVS carried as an alternative, in the event that bonding propellant to the silicone ramjet combustor insulator proved infeasible.

(U) Analog motors of approximately one-quarter full scale in size and containing the optimized propellant and bond system from Phase II, plus the selected design configuration from Phase I, were used as the final tool for structural evaluation of the bonded system. The essentially stress-free condition of the grain in the SVS approach does not require similar evaluation. After completion of analog motor testing over environmental extremes, final selection of the grain retention system was to be made.

(U) Evaluation of the selected design and materials system approach was planned to be evaluated then in motor firings and a 12-month age-life study. Previous successful subscale motor firings conducted by LPC had demonstrated the feasibility of the materials and design approaches being studied under the program. Furthermore, availability of existing full-scale size heavy-weight hardware at LPC made it economically possible to scale-up directly to full-scale rather than an intermediate subscale size. A series of three full-scale, heavyweight motor firings, at ambient and each of the temperature extremes, was therefore planned. In addition to providing ballistic and structural data in full size prior to commitment to the final flightweight motor demonstrations, these motors provide early data on propellant and motor process scale-up that permits time for refinement. Plantscale propellant mixes for the heavywall motors also provide propellant for the age-life specimens.

(U) Upon completion of the heavywall series, a final update of the flightweight motor design was to be conducted. Following Air Force approval of this design, the flightweight motor demonstration firings were planned at various conditions of thermal cycling and vibration.

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Figure 2-1 Program Logic

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(U) The schedule for accomplishment of this program is shown in Figure 2-2. One of the principal program features influencing the schedule is the 12-month age-life program and the need to finalize the propellant and materials system prior to start of preparation of specimens for that study. Program work through the evaluation of analog motor results must be completed, therefore, in a 5-month time period. The conduct of Phase II concurrently with Phase I accommodates this. Another principal feature is the provision of time after each full-scale motor firing for the complete evaluation of results prior to committing the test of the next motor. In addition, the flightweight motors are cast two at a time, and provision has been made to test and evaluate the first two motors in that series prior to casting the second group of motors. This feature provides an opportunity for minor motor modification and/or change in test conditions following each test event. Although the resulting time span for Phase III is longer using this approach, the early completion of Phases I and II accommodate it.

(U) Lockheed Propulsion Company's case-bonded design approach is presented in Figure 2-3. The design is based upon the results of previous Air Force programs, earlier LPC studies, and the results of Phases I and II under this contract up to the point of work stoppage.

(C) Characteristics of the proposed motor design and the reasons for the selected approach are as follows:

CASE-BONDED DESIGN

1	Characteristics	Reasons For Selection		
(1)	Sliverless, neutral burning grain with "keyhole" port and 0.75 web fraction	(1)	 Optimum tailoff Minimum case weight	
			 Good processability and reliability 	
(2)	87-percent solids, 18-percent aluminum propellant (LPC-691D),	(2)	 Exceeds performance requirements 	
	with ultra-fine ammonium per- chlorate (UFAP) to provide high burning rate		 No liquid burning rate catalyst 	
			 Good physical properties, and processability 	
(3)	Booster phase insulation (if required) provided by slight	(3)	• Avoids introducing another material	
	thickening of ramjet Dow Corning DC 93-104 silicone insulation		 Minimum post-burnout effects on transition 	
			• Serves as efficient booster insulation	

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Figure 2-2 Program Schedule

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(C) **CASE-BONDED DESIGN** (Continued) Characteristics **Reasons For Selection** (4) Propellant-to-DC 93-104 insula-(4) • Excellent bonding to DC tion bonding with LPL-63 liner/ 93-104 etched fluorinated ethylene pro-Minimum residuals pylene (FEP) film (LPE-18) substrate (5) LPE-17 silicone elastomer grain (5) • Minimum post-burnout stress relief flap (forward end effects on transition only) Good bonding (6) Plaster-celogen pellets inserted (6) • **Provides** inert compatible in off-gas holes filler to support grain during rocket operation Low-temperature (300°F) decomposition under ramjet operation to vacate holes (7) Air Force-developed ejectable (7) • Retention/ejection mechanozzle (if final tests successnism is contract requireful on ejectable nozzle conment tract) Nozzle design to be proven in tests Proven in service on SRAM (8) Hot particle magnesium-Teflon (8) • igniter Low-cost

(C) The design provides a high grain volumetric efficiency with a high solids loading/performance HTPB/UFAP propellant (LPC-691D) that is capable of providing the required structural properties. This propellant combines good processability and a minimum plasticizer content to reduce the potential for migration, which could adversely affect either the bond to the silicone chamber insulator, or the insulation material itself. The keyhole-slotted grain design provides a theoretically sliverless tailoff with a peak-to-average pressure (neutrality) of 1.07.

(U) All essential features of the design and its performance capability have been demonstrated in subscale motor hardware and laboratory test specimens. Verification has included ballistic design and structural integrity margins, propellant characteristics, interface materials system bond capability and compatibility, motor fabrication processes, and rocket materials system residuals. Subscale motor tests were conducted previously under LPC funding. A summary of these efforts is presented in Appendix A. In addition, LPC successfully loaded and tested two full-scale, case-bonded HARM (High-velocity Anti-Radiation Missile) demonstration motors (10 by 83.5 inches) using HTPB propellant, DC 93-104 insulation, and the LPC-proposed bond and materials interface system.

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(U) A number of the subscale integral ramjet booster motor tests demonstrated features of the proposed ballistic design, including port/throat ratio, web fraction, chamber pressure range, temperature range, plus materials and processes. LPC-691 propellant formulation was tailored and characterized specifically to meet the integral booster ballistic requirements.

(U) Subscale motor tests, analog motors, laboratory tests, and analysis have now demonstrated the structural integrity of the proposed design. Motor firings at the temperature extremes of -65 and $\pm 165^{\circ}$ F, plus analog motor tests with cooldown to $\pm 100^{\circ}$ F following repeated thermal cycles between the temperature extremes, have been successfully conducted. Descriptions of these previously conducted motor firings and analog motor tests are presented in Appendix A, and in Subsection 3.2.2.

(U) In support of the design effort for the case-bonded motor, LPC had previously conducted detailed evaluations of three different pretreatment methods for overcoming the inherent problems associated with silicone elastomers, as follows:

- Chemical pretreatment with a silicone-type resin
- Use of films as an intermediate substrate between DC 93-104 and conventional liner. This work included use of an etched fluorinated ethylene propylene (FEP) film and metal foil.
- Pretreatment of DC 93-104 with plasma inert gas to enhance surface wettability (hence adhesion)

(U) The LPC tests showed all three systems to be promising, however, the FEP film using liner provided superior propellant bonds to the silicone insulator that meet all structural requirements. Peel strengths of 13 pli and greater at ambient and low temperatures are provided. The FEP film (LPE-18) approach has been used successfully by LPC in the subscale integral ramjet booster motors and found to be simple to process in motor hardware. It was therefore selected as the baseline approach. LPC's optimum, balanced liner system for the integral ramjet booster was developed during the Air Force's Interface Materials Investigation program with Marquardt (AF Contract F33615-72-C-1234), and resulted in the identification of the LPL-59 liner formulation. Final results of that program were obtained with this liner, which contains TDI curative. In arriving at this system, a series of parameters were studied including not only the type of curative, but also equivalence ratio, additives such as bonding agents, liner thickness, number of coats, cured versus uncured liner, surface preparation, and propellant cure time and temperature. When used with the FEP film bond system, the LPL-59 liner was found to yield good results over a wide variety of conditions. As finalized, LPL-59 was further modified to include a catalyst as a hydrogen scavenger and re-designated as LPL-63. These efforts are described in Subsection 4.4.1

(U) During the course of LPC's continuing efforts to develop optimum interface materials systems, subsequent to completion of the Materials Interface program, a potential problem in processing compatibility between DC 93-104

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(U) insulation and the propellant was discovered, and resolved after extensive study. It involved the formation and evolution of gaseous species from DC 93-104 insulation at propellant grain cure temperatures, with migration into the grain during the cure cycle. This leads to fissures or voids in the cured grain adjacent to the interfaces. Such a problem would lead to serious consequences in the development of the integral ramjet booster if not recognized and counteracted. The nature of the problem is such that it is not detectable in laboratory interface specimens.

(U) Three alternative approaches to solving the problem were successfully demonstrated in application to motor processing. The selected approach is the use of a higher DC 93-104 cure temperature than initially used by LPC, and the addition of a very small amount of catalyst to the liner to promote scavenging of any minute quantities of gas that might still evolve from the insulation.

(U) The requirement of minimum combustibles remaining after booster burnout is achieved by minimizing the liner thickness and by the use of silicone elastomers (which have low gas-producing characteristics) for both the stress release flaps and the insulation required for the booster phase. Particular emphasis has been placed on proper control of dimensional tolerances and burning reproducibility, so that the theoretically sliverless performance of the grain can be approached.

(U) To further support the design development eafort, LPC conducted a series of subscale motor test firings in 1973 to evaluate motor operation during the critical transition to ramjet operation. Included in these evaluations were the effects of residual combustibles remaining from booster operation. The earlier laboratory and analytical data showed that adverse effects could occur from either delays in transition or creation of an over-pressure due to residuals combustion during initial ramjet operation.

(U) However, excellent transition performance was demonstrated in three Martin-Marietta/LPC subscale, case-bonded motor, integral rocket-toramjet transition tests conducted during the last half of 1973. Smooth transition was achieved for all three tests. In two of the three tests, the transition times were well below the specified 0.75 second, including a motor with a purposely offset grain port to produce a sliver at burnout. In the third test, utilizing a motor having grain defects near the insulation interface, the transition time was 0.84 second. These test results therefore indicate that by proper design and materials selection, plus careful motor processing, the potentially detrimental effects of residuals on transition can be significantly minimized and possibly eliminated. Reference (a) describes these efforts.

 ⁽a) R. V. Williams and D. A. Wiederecht, Lockheed Propulsion Company, H. Readey and E. Cobb, Martin-Marietta Orlando, "Integral Rocket Ramjet Booster Transition Tests", JANNAF 1974 Propulsion Meeting, San Diego, California, 22-24 October, 1974.

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(U) Efforts accomplished under this contract were devoted to optimize the propellant system to extend pot life for plant scale operations, and augmenting the bond/interface materials system to accommodate insulation off-gas holes. Incorporation of a pot-life extender of the UOP-36/DBTH anti-oxidant system provided dramatic improvement in pot-life to 16 hours or greater, with comparable or better physical properties to the earlier LPC-691B formulation, and identical burn rate. An increase in cure time from 7 to 12 days accompanies this change, but poses no problem to utilization of the propellant system provided schedule time is adjusted. The excellent processing characteristics give a high degree of confidence for successful scale-up to plant scale mix and motor cast operations.

(U) A system for accommodating the insulation off-gas hole array in the substrate of the propellant grain was developed and tested successfully. Results obtained prior to work stoppage on the contract indicated no adverse effects on bond strength yet full support of the grain against induced loads, with no impairment of the off-gassing function. The system employs hole-filling pellets cast out of a mixture of plaster and celogen (blowing agent), and inserted into the holes after drilling through the FEP film bonding aid into the DC 93-104 insulation. Normal lining and casting of the motor follows. After rocket burnout, the ramjet operation will rapidly decompose the hole fillers to a powder at about 300° F. Use of this system provides support to the grain at each hole site against chamber pressure, and precludes the presence of liner or propellant material in the holes. For SVS retention, this system is not required (except beneath the seal bond), since the fluid will readily occupy the holes during booster operation and vacate them at transition.

(U) The design, analysis, laboratory, and motor test data thus show promising avenues of approach for meeting the stated performance objectives with the baseline case-bonded approach. All features of the proposed baseline design have been successfully demonstrated, thus contributing to a minimum-risk for the remaining phases of the program. At the time of work stoppage, analog motors were ready for lining and propellant casting. The complete insulation and case-bonded grain retention substrate systems were in place. Work was also progressing on fabrication of tooling for the heavyweight motors, and was scheduled to commence on preparation of specimens for the age-life program.

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Section 3

MOTOR DESIGN AND ANALYSIS

3.1 PERFORMANCE AND CONFIGURATION ANALYSIS

3.1.1 System Requirements and Interface Considerations

(U) For eventual Air Force applications, the integral ramjet booster is designed and sized to perform its basic function of accelerating a missile from launch conditions to ramjet takeover conditions. In general, a highthrust, short-burn-time rocket motor is desired to reduce impulse losses due to drag during boost. End-burning grain configurations are excluded because of the very high burn rate that would be required. The definition of explicit "ramjet takeover conditions" involves the ramjet vehicle design, trajectory or guidance specification, and launch aircraft flight conditions. Some of these may reasonably be expected to change from the present baseline. However, regardless of the numerical differences that may occur during the evolution of this missile system, the priorities in the booster design concept are expected to remain reasonably fixed. The areas of emphasis placed on an ASALM integral ramjet booster design, in addition to normal rocket motor considerations, are summarized in the following paragraphs.

- The booster must perform to allow transition to ramjet operation (1) under the worst-case Air Force requirement. This worst-case requirement may significantly exceed nominal requirements and the missile may not simply "under perform", it may fail outright to achieve transition to ramjet operation. For example, a lowaltitude launch on a hot day (with a specified maneuver) is expected to require the largest integral booster velocity increment (ΔV). This results because the ramjet requires approximately a fixed takeover Mach number, which occurs at a higher velocity as ambient temperature increases. The ΔV requirement is further increased if the booster propellant is cold and the booster burns at lower thrust level for a longer burn time, thus increasing drag losses. Consideration of all such worst-case requirements is thus critical for an integral ramjet booster propulsion system.
- (2) The time required to achieve the transition from booster to ramjet operation directly increases the total booster impulse required, because of the missile deceleration due to drag during the transition phase coast period. Thus, a minimum sliver design with minimum residual or combustible materials is critical to avoid either an excessive loss in system performance capability, or an undue risk of ramjet unstart or failure to effect takeover. Specific evaluation of transition time involves system and fuel control studies beyond the scope of integral booster development alone. However, the booster design criteria that tend to minimize transition time are readily defined. Close coordination during booster development between rocket contractor, ramjet contractor, and systems integrator is required.

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- (U) (3) A high density-impulse integral booster is desired, but with a higher priority than in conventional booster designs as a result of ramjet constraints. Typically, the volume of the ramburner is determined by booster requirements, while the case material (L-605 alloy) is selected for ramjet operation. This material is a high-temperature rather than a high-strength alloy, leading to a heavier case and thus to an increased sensitivity of system weight to booster volume. A high density-impulse is used in the broad sense, and includes the volume fraction, propellant, and insulation requirements of the design. An additional design criterion, due to the high sensitivity to system weight, is that the booster must have a nearly neutral chamber pressure trace.
 - (4) The long ramjet burn time and high chamber temperature required to meet the system performance goals place heavy demands on chamber insulator performance capabilities. Establishment of a silicone elastomer (Dow Corning DC 93-104) as the insulation material to provide these capabilities creates the need for new rocket motor technology to achieve reliable, high-strength bonds of the propellant grain to this material. Silicones inherently have poor bond qualities, so that special techniques must be established and reduced to process procedures to ensure adequate structural integrity under all environments imposel by an airlaunched missile system.
 - (5) The design of the frangible dome cover, provided as an integral part of the combustor, is also an important consideration since it will dictate propellant grain processing approaches and tool configuration. Additionally such items as the ramjet nozzle material and booster ejectable nozzle design and materials must be carefully evaluated as to sealing capability and survivability in the booster burn environment.
 - (6) Some adjustment of the booster design may be expected as additional details of the system become available from other Air Force programs. The current booster design requirements are based on the Martin Co. Configuration Control Performance Bulletin (-008). However, it is understood that a more current configuration, CCPB-009 now exists and dictates somewhat different performance requirements for the booster motor. These changes will be integrated into the booster design as directed by the Air Force.

These considerations of the unique and demanding factors that must be emphasized for an integral ramjet booster propulsion system have led LPC to establish the following major objectives, to meet the overall Air Force objectives:

- Minimum booster volume and weight (i.e., maximize volumetric efficiency)
- Maximum integrity of bonds to silicone insulator (for case-bonded design)

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(U)

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 Minimum combustible residuals and effect created thereby on ramjet takeover

(U) Table 3-1 lists the booster design requirements. Figure 3-1 shows a typical booster internal configuration as it existed at the onset of the Booster Development Program.

3.1.2 Ballistic Analysis

(U) Ballistic analyses of the case-bonded design and the alternate SVS design are discussed in the following sections.

3.1.2.1 Trade Studies and Baseline Case-Bonded Design

(C) <u>Selection Criteria</u>. Selection criteria for the case-bonded grain configuration and propellant formulation used in preliminary tradeoff studies were established from the performance requirements specified in Table I of Exhibit A of the contract as related to various solid rocket motor design factors. These criteria and design relationships are discussed below.

- (1) A neutral pressure-versus-time curve consistent with the specified 1820-psia maximum expected operating pressure (MEOP) is desired to minimize the ratio of maximum to average pressure (P_{max}/P_{avg}) , thereby minimizing chamber length and weight. Sizing of the motor chamber wall is entirely constrained by the booster operating conditions. This is because the nominal operating pressure of the booster motor is much higher than that of the ramjet sustainer. It is especially important, therefore, that the booster operate at the most efficient conditions with respect to chamber design. A neutral-burning booster motor will provide the highest performance for a given chamber weight because the wall thickness of the pressure vessel (chamber) depends on the highest expected pressure condition, and delivered specific impulse increases with pressure. Consequently, full utilization of the allowable working pressure over the entire burn duration is highly desirable.
- (2) Minimum total motor weight and motor case length are desired. Volumetric efficiency should be maximized and propellant density-impulse should be at a maximum consistent with requirements.
- (3) Minimum propellant sliver and residual combustibles must be achieved during rocket motor tailoff (ramburner startup). This can be accomplished by proper grain and grain retention design and by minimizing sliver-producing elements such as erosive burning.

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TABLE 3-1

INTEGRAL ROCKET BOOSTER DESIGN REQUIREMENTS

ITEM	VALUE	PERMISSIBLE VARIATION
Nominal Motor Action Time, Seconds (70°F)	4.1	
Boost Thrust, Pounds (70°F)	30,000	\$4.0%
Maximum Thrust Variation, Percent of Nominal	*10 ·	1
Maximum Chamber Pressure, PSIA (Any Temp.)	1,820	
Minimum Delivered Isp, Pound-Seconds/Pound	2451	and a state of the
Maximum Propellant Ik, %/degree	0.20	
Action Time Total Impulse, Pound-Seconds (70°F)	122,100 ¹	±1.0%
Maximum Total Motor Weight, Pounds	. 862.5 ²	-
Maximum Case Length, Inches	44.0 ²	-
Case Outside Diameter, Inches	18.0	-
Case Wall Thickness, Inches	0.128	-
Insulation Material	Dow-Corning 93-104	-
Insulation Thickness, Inches	0.25 to 0.50	
Maximum Throat Erosion Rate, Inches/sec	0.002	124 C

¹Motor performance is based on 70°F sea level operation. Isp is defined at 1000 psia chamber pressure and expansion to 14.7 psia with a nozzle half angle of 15 degrees.

2The major program objective is to minimize total motor weight (includes propellant, chamber, booster nozzle, ramjet nozzle, insulation liner, igniter, inlet attachment, and nozzle release mechanism), and motor case length.

Classified by ASD/XR	
Exempt from General D-classification	
Schedule of Executive Order 11652	1
Exemption Category 3	
Declassify onIndefinite	

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* The length dimensions are maximums; the finai case length will be defined during Phase I., and will be the minimum possible that is consistent with the Table I total impulse requirements.

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A grain sliver is defined as the propellant remaining in a motor at the time corresponding to major web burnout. All of the grain design candidates given serious consideration should possess minimal sliver and ideally should be sliverless. Consequently, tailoff will approach that of simple chamber blowdown, with very small slivers due only to inherent manufacturing variations. Combustible residual materials include rocket motor insulation and stress relief flaps. To achieve minimum combustible materials, the total quantities of these items must be minimized. Influencing factors include nonuniform propellant webs, which require use of local peripheral insulation, and web fraction in conjunction with propellant structural allowables, which defines the stress relief flap configuration. Internal ballistic phenomena, such as erosive burning, must be controlled since they typically produce nonuniform propellant burning at various stations in the motor, generating propellant slivers and causing premature exposure and consequent burning of insulation. It is desirable to achieve a booster motor design that will exhibit negligible erosive or nonuniform burning effects. To aid in this goal, physical and effective port-to-throat area ratios of 1.30 and 1.62, minimum, were established as groundrules for the minimumrisk baseline design. Grain structural integrity also influenced this limitation for the case-honded motor. Motor test data demonstrated negligible erosivity at 1.3 port/throat ratio. Secondary flow effects are expected to be minimal because no nozzle submergence is proposed. The technique of designing to lower port-to-throat area ratios, while at the same time tapering the grain port to minimize the flow effects, is disallowed because it is inconsistent with the sharp tailoff requirements.

(4) State-of-the-technical-art selection of grain configurations and propellant formulations will be adhered to, assuring credible design values for ballistic characteristics and propellant properties.

(U) Grain configuration and performance tradeoff analyses were conducted to determine an optimum design that best fits the selection criteria. The grain design studies involved consideration of ballistic requirements, grain structural capabilities, and minimum sliver and tailoff impulse. A discussion of the detailed grain design tradeoffs follows.

(U) Grain Design Studies. Several controlling assumptions were made at the outset of the grain design study effort, including the following:

• A nominal instulation thickness of 0.375 inch is required for ramjet operation. Consequently, all of the thermal protection required during booster operation was as an additional insulation requirement.

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(C)

 (U) A minimum structural safety factor of 1.5 for the grain was imposed. This factor includes an assumed 20-percent degradation of propellant physical properties due to aging.

(C) Four basic grain configurations were evaluated for the case-bonded approach. These were as follows:

(1) Keyhole: This configuration, shown in Figure 2-3, meets the basic design goals and is ideally sliverless. The neutrality of this grain, which burns internally and on its aft face, is exceptional, having a maximum-to-average burn area variation during web consumption of less than 3 percent. This relates to a maximum-to-average thrust variation of 7 percent, well within the required 10-percent limit. Estimates of pressure and thrust versus time are shown in Figures 3-2, 3-3 and 3-4 for +70, +165, and -65°F, respectively. This design was ultimately selected and demonstrated in a number of subscale motor firings.

As illustrated, the keyhole configuration is basically a circular port with a single longitudinal slot whose depth is slightly less than the thickness of the web. The keyhole is "straight-through", that is, it runs the full length of grain.

- (2) Radial slot: The configuration shown in Figure 3-5 meets the basic goals in that it is ideally sliverless and provides reasonably neutral performance. The pressure-versus-time performance for this preliminary design is presented in Figure 3-6. The maximum-to-average performance variation for this configuration is 1.13, which exceeds the specified 1.10 maximum value. Nevertheless, it is quite possible that the required neutrality could be achieved by additional tailoring of the grain geometry. As illustrated, the radial slot is located near the middle of the grain. Other slot locations, such as near the forward closure (conocyl), were evaluated. However, these locations were less desirable in terms of grain structural integrity and motor processability.
- (3) Forward circular-port/aft-star configuration (similar to Figure 1 of Exhibit A of the contract). This grain design has a progressive, cylindrical port in the forward portion of the grain, and a regressive three-or four-point star configuration in the aft grain portion. This combination provides a reasonably neutral trace and ideally sliverless geometry. However, this configuration did not excel in any of the desirable grain design and performance areas, and this class of grain design is the least desirable from a structural integrity standpoint.
- (4) Modified keyhole: This design is very similar to the previous keyhole design except that it incorporates an anchor at the bottom of a shortened keyhole slot (see Figure 3-7). This modification permits use of a conventional, one-piece mandrel for motor loading. With the exception of the mandrel-removal feature, this configuration was not outstanding from a performance or weight standpoint. -27-







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Figure 3-7 Modified Keyhole Grain Design



(U) All of the above grain configurations are characterized by high volumetric loading, relatively neutral burning, and ideally sliverless burnout (or nearly so).

(U) Grain configurations requiring a center support structure (e.g., rod and tube) were not considered, because of their inherent complexity. Furthermore, grains that require propellant surface restriction material were rejected because they result in more residual material at burnout, are more costly to fabricate, and tend to degrade performance reproducibility.

(U) <u>Baseline Design Selection</u>. Of the candidate grain configurations discussed above, the keyhole and center-located radial slot designs were evaluated in greatest detail as offering the most desirable features. The results of a detailed performance tradeoff between these two designs are presented in Table 3-2.

(C) In each case, the propellant physical properties were adequate to permit maximum web fractions consistent with a minimum physical port-to-throat area (1.30) constraint. Because of insulation requirements and the volume lost to a radial slot, the keyhole configuration shows a slight performance advantage in terms of lower motor weight and case length. In addition, the keyhole grain is more neutral than the radial slot design.

(U) Both designs presented are structurally adequate for an 87-percent solidsloaded propellant formulation.

(U) On the basis of a lower motor weight and case length as well as more favorable curve neutrality, i.e., lower ratio of maximum to average thrust, and less complex grain forming processes, the keyhole was selected as LPC's baseline case-bonded grain design approach.

(C) <u>Motor Characteristics</u>. The selected keyhole grain design, with a web fraction of 0.75, delivers a thrust of 30,000 lbf over an action time of 4.07 seconds, and a total impulse of 122,100 lfb-sec within the MEOP constraint as specified in the contract. These values correspond to $+70^{\circ}$ F and sea level conditions. Pertinent ballistic performance and design parameters are summarized in Table 3-3. A component weight summary is presented in Table 3-4. Ignition and tailoff transients for $+70^{\circ}$ F are presented in Figures 3-8 and 3-9.

(C) The data in Table 3-3 are based on properties for the baseline 87-percent solids-loaded propellant formulation. The ballistic values presented in the table are referenced to the nozzle centerline. It is recognized that a small nozzle cant angle may be desired, as indicated in Figure 1 of Exhibit A to the contract. Due to the small angle, motor-centerline and nozzle-centerline performance values are not expected to differ by more than one-tenth of one-percent.

(C) <u>Nozzle Configuration Considerations</u>. A preliminary nozzle design optimization study was conducted for the purpose of arriving at a configuration upon which to base performance predictions and cost estimates. The two major design goals, minimum total motor weight and motor length, were found to be mutually exclusive when applied to nozzle design. Assuming a constant motor total impulse, the minimum motor-weight design optimizes at a relatively high expansion ratio (>9) and low half-angle. On the other hand, the minimum-length



Table 3-2

PERFORMANCE TRADEOFFS FOR CASE-BONDED DESIGN

Grain Configuration

•	Gian	Configuration
	Keyhole	Center-Located Radial Slot
Port-to-throat ratio	1.3	1.3
Base burn rate at 1,000 psia, in./sec	1.34	1.27
Action time, sec	4.07	4.07
Grain length, in. ^(a)	37.2	37.6
Ratio of maximum to average thrust	1.07	1.13
Web fraction	0.75	0.72
Propellant solids loading, %	87	87
Grain safety factor ^(b) (with 20-percent aging degradation)	1.90	2.5

(a) Provides 122, 100 lb-sec impulse

(b) Propellant allowables based on measured data

Table 3-3

BASELINE MOTOR BALLISTIC PERFORMANCE AND DESIGN SUMMARY (Sea-Level Conditions)

	Contract	Propos	ed Baseline	Design
Item	Requirements +70°F	+70 ⁰ F	+165°F	-65 ⁰ F
Nominal motor action time, sec	4.1	4.07	3.52	5.00
Boost thrust, 1b	30,000	30,000	34, 960	24, 100
Max thrust variation, % of nominal	10	7.0	7.0	6.9
MEOP (any temperature), psia	1,820		1,820	a within
Delivered I _{sp} , sec	245 ^(a)	248	250	245
Maximum propellant π_k , $\%/^0F$	0.20	0.15	0.15	0.15
Action time total impulse, lb-sec	122, 100	122, 100	123,070	120, 300
Action time average pressure, psia		1,372	1, 586	1,116
Nozzle throat diameter, in.		4.234	4.234	4.234
Nozzle exit diameter, in.		11.935	11.935	11.935
Nozzle half angle, deg	- uner	20	20	20
Nozzle expansion ratio		7.95	7.95	7.95
Minimum port-to-throat, physical		1.30	1.30	1,30
Minimum port-to-throat, effective		1.62	1.62	1.62
Max-to-average action time pressure		1.066	1.067	1.065
Maximum web fraction		0.75	0.75	0.75
Grain length, in.		37.2	37.4	37.0
Propellant weight, 1b		491.7	491.7	491.7
Propellant density, lb/in. ³		0.0650		
Burn rate at 1,000 psi, in./sec		1.34	1.46	1.26
Burn rate exponent (n)		0.59	0.59	0.59

(a) Minimum delivered specific impulse at 1,000-psia chamber pressure and expansion to 14.7 psia with a nozzle half-angle of 15 degrees. Motor delivered value corrected to the same conditions is 247 seconds.

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Table 3-4

RAMJET BOOSTER WEIGHT SUMMARY (Keyhole Grain Design, Case Bonded)

Component	Weight, 1b
GFE hardware ^(a)	
Chamber	160.9
Booster nozzle	43.5
Ramjet nozzle	85.3
Inlet attachment	8.5
Nozzle release mechanism	8.1
Subtotal	306.3
Propellant grain	491.7
Igniter	0.5
Liner	1.9
Ramjet	48.3 ^(b)
Rocket	4.5
Release flaps	3.8
Total inert	365.3
Total weight	857.0

 (a) Values from Martin-Marietta Corp Document,
 "Configuration Control and Performance Bulletin for Integral Rocket/Ramjet," dated January 1973.



⁽b) DC 93-104 material only: 0.375-inch nominal thickness.



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(C) design optimizes at a relatively low expansion ratio (≤ 5) and high half-angle. Since the thrust-to-weight ratio of the vehicle during booster operation is relatively high, the ideal velocity increment (ΔV) is believed to be a better optimization parameter for nozzle design purposes. Optimization to this parameter also results in a reasonable compromise between motor minimum weight and length. The results of the optimization are presented in Figure 3-10. Inspection reveals that a nozzle half-angle of 20 degrees is near optimum. In addition, maximum ΔV is seen to be a relatively weak function of nozzle expansion ratio. With a half-angle of 20 degrees, optimum performance is achievable with an expansion ratio anywhere within a range of from about 6 to 8.

(C) The design point value of 7.95, indicated in the figure, was selected after a review of UTC Drawing Cl0444, received as a part of Appendix A to Exhibit A of the RFQ. The selected design point requires the least change to the nozzle design as presented in the drawing. This change is minor, consisting only of a decrease in nozzle throat and exit diameters of 0.136 inch. The 20-degree nozzle half-angle is thereby retained. Such a minor change in internal nozzle dimensions is consistent with the requirements of Paragraph 4.3.2 of Exhibit A of the contract.

3.1.22 Tradeoffs and Alternate SVS Design

(C) For the alternate approach using the SVS for grain retention, the following criteria were used in conducting preliminary tradeoffs and in selecting the proposed propellant formulation and grain design:

- (1) A neutral pressure-versus-time curve consistent with the specified 1820-psia MEOP is desirable in order to minimize the P /P ratio.
 C avg
- (2) Because minimum case length is desired, volumetric loading and impulse-density of the propellant should be maximized.
- (3) The same goal (as for the case-bonded design) of achieving minimum propellant sliver and other residual combustibles at motor burnout was maintained, even though the SVS approach is more forgiving because the propellant cup and its residual contents will be ejected when ram-air enters the chamber.
- (4) The SVS design approach should represent demonstrated, stateof-the-art technology as developed at LPC since May 1970 on the AFRPL-funded Unique Grain Retention System Program.
- (5) A minimum port-to-throat ratio of 1.2 was established for the alternate SVS approach to take advantage of the higher web. fraction permitted by the stress-relieving grain retention system.

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Figure 3-10 Vehicle Ideal ΔV -versus-Nozzle Expansion Ratio and Divergence Angle



(U) Laboratory testing and test firings were conducted to assess and verify proposed details of materials and design approach.

(U) The principal tradeoff conducted to establish the proposed grain design for the SVS approach was related to grain configuration and propellant formulation. The same general design philosophy prevailed for selection of the grain configuration as in the case-bonded motor, but the propellant selection criteria were somewhat different.

(U) The principal grain configuration considerations were related to Items 1, 2, 3, and 5 above. Whereas the principal constraint on propellant selection in the case-bonded motor was achievement of adequate physical properties, processability is the most significant constraint in the SVS design.

(U) The same four basic grain designs were evaluated for the alternate approach as described in Subsection 3.1.2.1 for the case-bonded approach These were:

- (1) Keyhole
- (2) Radial slot
 - Forward end slot
 - Center slot
- (3) Forward circular port/aft star configuration
- (4) Modified keyhole

(U) All of these grain configurations are characterized by high volumetric loading, relatively neutral burning, and ideally sliverless burnout (or nearly so).

(U) As for the case-bonded design, the keyhole and radial slot (in longitudinal center of grain) were selected for detailed tradeoffs.

(U) A decision was made to propose a minimum port-to-throat area ratio of 1.2 for the alternate SVS approach since it is a more significant constraint on volumetric efficiency for the SVS design than for the case-bonded design. For the case-bonded design, the physical properties of the propellant are a major constraint on the web thickness (port size). However, the physical properties are not a constraint on the SVS approach, permitting selection of a port-to-throat area ratio entirely on the anticipated limits due to erosive burning. The threshold ratio, below which unacceptable erosive burning occurs, has not been established, if indeed such a ratio exists for the proposed propellant.

(U) Several subscale motor tests have demonstrated negligible erosive burning at a port-to-throat ratio of 1.3. The data from one motor with a 33-inch-long grain and a port-to-throat ratio of 1.0 demonstrated that this propellant has a very low susceptibility to erosive burning. On this basis, the selection of a port-to-throat area ratio value of 1.2 is not considered overly optimistic.



(U) Table 3-5 presents a summary of the data for the performance tradeoff for the keyhole and radial slot grain configurations in an SVS design. An 88-percent solids propellant was selected as having the highest solids loading with demonstrated processing characteristics acceptable for this program.

(C) On the basis of these tradeoff data, and for the reasons shown on the lower portion of Table 3-5 the keyhole grain was selected for the SVS approach. As a point of interest, snould propellant tailoring result in an 89-percent solids propellant that would be adequately processable for the SVS design, the potential decrease in grain length is af follows:

Solids, %	Grain Length, inches
88	37.20
89	36.97

(U) SVS Grain and Performance

The keyhole grain selected for the alternate motor design is shown in Figure 3-6. A summary of its characteristics is presented in Table 3-6. Also shown in the table for comparison are the contract requirements. Thrust and chamber pressure-versus-time curves for +70, +165 and -65° F (at sea-level) are given in Figures 3-11, 3-12 and 3-13, respectively. Table 3-7 presents a weight summary for the motor. It can be seen in Table 3-6 that all requirements are met.

(U) The selected propellant grain has a forward end web because it is structurally acceptable with the SVS retention approach. The selected grain is theoretically sliverless.

Table 3-5

SVS MOTOR GRAIN DESIGN TRADEOFFS (88-Percent Solids Propellant, +70°F, Sea Level)

Grain Co	nfiguration
Keyhole	Radial Slot
1.20	1.20
1.34	1. 27
4.07	4.07
37. 2	37.6
1.10	1,13
0.76	. 72
	Grain Co <u>Keyhole</u> 1. 20 1. 34 4. 07 37. 2 1. 10 0. 76

Selection

Reasons

- Keyhole Design
- (1) Lower motor weight and case length
- (2) Lower $P_c \max/P_c avg$
- (3) Better processability

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Table 3-6

SVS MOTOR BALLISTIC PERFORMANCE AND DESIGN SUMMARY (Sea-Level Conditions)

	Contract	Alter	nate (SVS) I	Design
	Requirements +70°F	+70 ⁰ F	+165°F	-65°F
Item				
Nominal motor action time, sec	4.1	4.07	3, 52	4.99
Boost thrust, 1b	30,000	30,000	34, 950	24, 120
Max thrust variation, % of nominal	10	10.0	10.0	9.9
MEOP (any temperature), psia	1,820		1,820	
Delivered I _{sp} , sec	245 ^(a)	248	250	245
Maximum propellant $\pi_k^{}$, $\%^{/0}F$	0.20	0.15	0.15	0.15
Action time total impulse, lb-sec	122, 100	122, 100	123,070	120,400
Action time average pressure, psia		1,340	1, 548	1,090
Nozzle throat diameter, in.		4.234	4.234	4.234
Nozzle exit diameter, in.		11.935	11.935	11.935
Nozzle half-angle, deg		20	20	20
Nozzle expansion ratio		7.95	7.95	7.95
Minimum port-to-throat, physical		1.20	1.20	1.20
Minimum port-to-throat, effective		1.50	1.50	1.50
Max-to-average action time pressur	e	1.099	1.0,99	1.099
Maximum web fraction		0.76	0.76	0.76
Grain length, in.	1	37.2	37.4	37.0
Propellant weight, lb		491.7	491.7	491.7
Propellant density, lb/in. ³		0.0653		·
Burn rate at 1,000 psi, in./sec		1.34	1.46	1.26
Burn rate exponent, n		0.59	0.59	0.59

 (a) Minimum delivered specific impulse at 1,000-psia chamber pressure and expansion to 14.7 psia with a nozzle half-angle of 15 degrees. Motor delivered value corrected to the same conditions is 247 seconds.

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Table 3-7

RAMJET BOOSTER WEIGHT SUMMARY (Keyhole Grain Design, SVS Alternate)

Component		Weight, 1b
GFE hardware ^(a)		ing and provide a
Chamber		160.9
Booster nozzle		43.5
Ramjet nozzle		85.3
Inlet attachment		8.5
Nozzle release mee	chanism	8.1
	Subtotal	306.3
Propellant grain		491.7
Igniter		0.5
Liner		1.9
Insulation Ramjet		48.3 ^(b)
Rocket		1.0
Cup/fluid		9.0
	Total inert	367.0
	Total weight	858.7

 (a) Values from Martin-Marietta Corp Document,
 "Configuration Control and Performance Bulletin for Integral Rocket/Ramjet," dated January 1973.

(b) DC 93-104 material only: 0.375-inch nominal thickness.



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3.1.3 Structural Analysis

(U) The basic propellant grain configuration for the fullscale case bonded Integral Ramjet Booster motor has remained essentially unchanged since originally conceived and proposed for utilization in the development program. The keyhole grain concept offers both ballistic and structural advantages over other concepts studied by LPC and extensive subscale testing has shown that it also affords significant total system advantages.

(U) At the onset of the program the case-bonded motor contained a combination full head end inhibitor and release flap and an aft end release flap. This arrangement represents what LPC considers to be a widely accepted standard conservative approach to grain design and retention.

(U) A thorough structural analysis of this configuration was performed on the basis of the physical properties measured for the 87-percent solids propellant (LPC-691B). These properties are discussed in Subsection 4.2. They were further reduced for use in the analysis, as shown in Figures 3-14 through 3-17. Figures 3-14 and 3-15 show the allowables as a function of time and temperature for the thermal loading and pressure loading, respectively. Figures 3-16 and 3-17 show the master relaxation modulus curve and the accompanying log aT shift factor curve, which was also used to shift the allowables to their locations in Figures 3-14 and 3-15. Based on these allowable and modulus curves, the induced loads are summarized and compared to the allowables in Table 3-8. The minimum safety factor for the unaged condition was +2.4 and for the aged condition was +1.9, both of which are for the innerbore hoop strain for the worst-case combination of thermal and pressurization loads at -65° F. This last safety factor includes a 20-percent reduction in allowables for aging. These safety factors show that the grain will successfully perform under all environmental and operational loading conditions.

(U) Therefore, the status of the analysis at the onset of the program indicated that for the physical properties displayed by LPC 691B propellant cured at +115°F, combined with the keyhole grain which incorporated release flaps at both ends, the structural margins of safety were well in excess of the minimum required values.

(U) As is recognized in the Integral Rocket Ramjet System inert residuals remaining at the end of booster burn are of concern as related to transition performance and should be held to an absolute minimum. The residuals consist largely of the unconsumed liner, release flaps and inhibitors. The keyhole grain design minimizes inert residuals through careful control of liner thickness and utilization of silicone release flaps and inhibitor components. However, in full consideration of the stress relieving aspects of the keyhole grain and the apparent design conservatism as indicated by the structural safety margins, it was deemed worthwhile by both AFRPL and LPC to analytically evaluate the possibilities of removal of one or both of the release flap components. If successful, this approach offers the obvious advantages of reduced residuals, lower cost and improved reliability. To facilitate this approach a comprehensive grain structural analysis was initiated in the first month of the program. In addition to evaluation of the elimination of release



Figure 3-14 Thermal Allowables versus Log Ra_T



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Figure 3-16 Stress Relaxation Modulus

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Figure 3-17 Time/Temperature Shift Factor Curve

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CRITICAL LOADS

2.63 7.3 1.9 Aged Factor of Safety 3.6 8.1 6.3 ۲ 1.1 5.0 1 ь ľ 2.4 3.3 9.1 Unaged Factor of Safety Ψ 7.9 4.4 10.1 + 6.2 1 1 ь 22.5 22.5 14.5 Ψ Allowable 138 1120 138 ۲ 145 1 1 ь <u>6.9</u> <u>1.6</u> 6.9 1.6 Ψ 13.6 142 <u>13.6</u> <u>142</u> F Induced Compressive Compressive 23.5 ь Pressure Pressure Thermal Thermal +

Table 3-8

RAMJET BOOSTER FACTORS OF SAFETY

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(U) flaps an additional objective was to determine minimum bond strength requirements including the influence of the filled combustion insulation outgassing holes at the bond interfaces. This work was conducted on a parallel basis with final analytical optimization of the grain dimensions. As previously mentioned the grain remained essentially unchanged with exception of a minor thinning from 0.85 to 0.05 inches in the web under the keyhole slot to produce a slightly higher onset thrust curve and further neutralize the performance curve shape. Additionally, the comprehensive stress analysis included the effects of a higher propellant cure temperature (145°F) as opposed to 115°F used in the initial analysis. The 145°F cure temperature was selected for use on the program to minimize the schedule impact which would have been imposed by the extended time required for curing at 115°F.

Comprehensive Stress Analysis

(U) Structural loads imposed on solid propellant grains range from relatively simple thermal, pressure, and acceleration conditions to complex phenomena involving transients and mechanically coupled nonlinear responses. In most applications transportation and handling loads of ± 10 g are required although these are not always permitted at the temperature extremes. Vibration spectrums must also be withstood particularly in external air carry applications where supersonic flight regimes can induce severe dynamic conditions. Transient thermal gradients can occur under aeroheat or aerocooling conditions in some systems and are capable of producing failures analagous to "thermal shock" phenomena. Coupled response structural phenomena include acoustic instabilities or resonant burning which produce pressure excursions that in turn result in case failures. Secondary conditions can cause concern for other structures. One such condition is low frequency vibration modes which could result in structural difficulties for the carrying vehicle. An example of this would be a fundamental vibration mode of less than 10 cps in a missile intended for air carry on certain supersonic aircraft.

(U) Because the propellant is a tailored polymeric material, it is possible to adjust its mechanical properties over a wide range and obtain an optimum for a specific set of design load requirements. Criteria for the propellant properties arise from the system loads and environments as well as from the type of geometry selected for internal ballistic considerations. An end burning design for example may require a higher modulus than a star perforation design for an identical application.

(U) Tailoring a propellant for optimum mechanical properties should relate to specific load conditions which in general are poorly defined in the early stages of development. In general it is not possible to achieve large structural margins through such tailoring without sacrificing specific capabilities. High strain capabilities, for example, can be obtained at the expense of stress capabilities or through modulus reductions which may result in unfavorable dynamic response capabilities.

(U) Clearly the design process needs an early guide to structural adequacy without a prohibitive amount of analytic computations and without leading to ill advised manipulations of the mechanical properties of the propellant to

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(U) meet vaguely defined loads. Fortunately, there is a method of achieving this structural integrity by concentrating the early attention on specific loads. Low temperature storage and operation have been the most critical loading condition encountered by most tactical solid rocket motors produced. If a propellant grain design is capable of meeting these historically critical conditions without recourse to unusual design variants or to extensive propellant tailoring, it will tend to have structural margins that are greater under the more complex yet less severe conditions. In particular, if these critical conditions are met while adhering to structurally sound engineering practice as regards the other loads anticipated, the design will have a high probability of meeting all specific load requirements when they are subsequently defined. Finally, by postponing the mechanical property optimization, this avenue is available for exploitation should some unusual condition be encountered later during development. Lockheed Propulsion Company has followed this practice in this program giving careful attention to the geometric characteristics that lead to high structural margins under typical tactical weapon applications.

(U) The Integral Ramjet Booster motor propellant grain is of the single slot keyhole configuration which represents a favorable structural geometry. Thermal loads are relieved by the slot, which penetrates close to the outer wall while acceleration is easily supported by the large bond area. Difficulties under sustained dynamic loads such as encountered in high speed air carry conditions are predominantly due to thermal effects caused by large deformations. These deformations are low in designs of this type due to the absence of free cantilevered portions such as those that are obtained in star perforation geometries. Acoustic instabilities are also less probable in asymmetric configurations such as this, and the structural advantages of these designs have been well understood for many years. The combination of structurally favorable geometry with good propellant mechanical properties results in a grain that is capable of withstanding all the currently envisioned structural loads for the system. The most critical loading condition is considered to be operation at low temperature. Development motors were therefore subjected to a detailed evaluation of the structural adequacy under this condition. Other loads should result in greater structural margins although specific conditions should be checked when the system is further defined, as some extreme load such as severe aerodynamic heating may result in unanticipated difficulties. Specific loads of this type are not clearly defined at this time due to the early stage of development.

(U) Structural adequacy under the low temperature operation condition was established by performing a linear viscoelastic analysis of the design and by supplementing the computations with structural analog motor experiments. Mechanical properties of the propellant were interpreted as linear viscoelastic responses by imposing time/temperature shift assumptions on test data obtained over the anticipated range. This characterization is depicted in Figure 3-16 where the regularity of the resultant curves indicates that the characterization is valid. Computer analyses were next made using these properties. Finite element computer codes were employed for these calculations which consisted of plane and axisymmetric representations subsequently combined to represent the three dimensional structure. The keyhole propellant grain has the configuration of a solid cylinder with an axial perforation consisting of a circular cylinder bore, concentric with the outside of the grain

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(U) and a single radial slot. This type of configuration lacks simple symmetries and therefore requires a three-dimensional stress analysis. In this analysis, the three-dimensional character of the stress-strain fields was obtained by combining two-dimensional solutions which represent orthogonal sections of the geometry.

(U) The most significant geometric characteristic from a structural consideration is the constraint imposed by case-bonding of the propellant grain. Geometrical constraints prevent thermal expansion and contraction deformations, thereby producing high stresses. Because these constraints are three-dimensional in character, it was necessary to account for this aspect in the analyses. Mathematical complexities associated with these structural problems are such that computer evaluations are necessary. LPC has developed a finite element numerical analysis computer code specifically for such problems and employed it in this analysis. The computer program is capable of resolving three-dimensional stresses in three types of geometries, solids of revolution, planar solids, and infinite length solids of constant cross section. Clearly the booster grain does not fit any of these categories, so a composite solution method was employed. First, the axisymmetric portion of the propellant grain was analyzed taking care to include all constraining aspects. Next, an analysis was made of a transverse section using the planar solid representation.

(U) In order to apply this latter result to the first solution, it is necessary to compute the stress variation that occurs in the circumferential direction, interpret this as a stress or strain factor, and multiply the first solution by these factors. Consider, for example, the circumferential or "hoop" strain. The integral of hoop strain around the circumference at a constant radius cannot be significantly changed by strain variations around the circumference, as the motor case maintains the outer boundary circular. What does happen is that increased hoop strains in one part of the circumference are compensated by decreases in other locations. The maximum strain increase is the ratio of maximum planar solution strain to average planar solution strain as computed around a constant radius circumference. This ratio is multiplied by the axisymmetric result to yield the maximum hoop strains for the grain. A similar process yields the maximum bond stresses.

(U) Factors obtained in this manner were combined with the axisymmetric computer solution to yield the maximum values and factors of safety presented in Table 3-9.

(U) The variance in bond stress was taken as a stress factor, which was subsequently applied to all bond stresses computed through the axisymmetric computer analysis. In addition to this nonaxisymmetric bond stress variation, the periodic holes drilled in the insulation cause local stress fluctuations. The influence of these holes was accounted for by factoring the results by 100/91 to represent the 91-percent of the total area that does not contain holes, and by a stress factor of 2. This latter factor is derived from the parabolic stress pattern that is obtained under thin bond pads.

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PROPELLANT GRAIN STRUCTURAL ANALYSIS SUMMARY

Head-End		:	Critical	Unag	ed Fa Safet	ctor	Age	d Fac f Safet	tor	
Condition	Loading	Combination	Location	0	-	w	0	-	۳	
Jnbond /2 Unbond Sonded	Thermal		Head End	3.1 2.9			2.3			
Jnbond /2 Unbord Sonded	Thermal		, Head End		6.5 5.7 3.7			5.2 4.5 2.9		
Jnbond /2 Unbond Sonded		Thermal plus Pressure	Head End		4.0 3.5 2.2			3.2 2.8 1.7		
Jnbonded	Thermal		Aft End Aft End Long. Center	4.2	7.6	3.2	3.4	6.1	2.6	
Jnbonded	Pressure		Aft End Aft End Long. Center		11.3	4.3	1	8.0	3.5	
Jnbonded		Thermal plus Pressure	Aft End Aft End Long. Center	1	5.4	1.8	1	4.3	1.5	

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(U) Specific factors obtained for use in the computer program were:

Nonsymmetric strain factor5.9Bond stress total factors3.47

(U) Included as an integral part of the comprehensive stress analysis was the analysis to establish the structural significance of removal of the release flaps. The analysis assumed that no release existed at the aft end in either case, inclusion or exclusion of the forward release. Thus a direct comparison of the effects on grain structural capability could be made. The bonded configuration was modeled as a grain with a base that equalled the inner circle of the keyhole at the forward end, and tapered to the web outward of the slot tip aft of 2.5 inches from the forward dome, as shown in Figure 3-18. A comparison of the fully bonded result with the previous forward release flap design indicated that maximum stresses and strains were essentially unchanged, but that a greater portion of the grain is subjected to slightly higher stress/strain levels in the bonded design. The structural margins are thus not changed significantly, and the analysis indicates that the forward release may be eliminated without significant structural risk to the design. Although, as previously stated, the total elimination of release flaps has both cost and transition performance advantages, there are other technical considerations which must be acknowledged. These points relate primarily to the forward release flap, which also constitutes an inhibitor for the forward portion of the grain. The forward inhibitor is necessary to maintain the proper ballistic profile. This must be done by either inhibiting, as in the current configuration, or by bonding to the forward dome of the motor.

(U) As recognized, LPC's primary approach for bonding utilizes the FEP film. In a bonded head-end approach, without the integral release flap/ inhibitor, compound contouring of this FEP film would be required to provide the necessary bond interface. Development of the tooling and techniques to achieve this is certainly possible, but in consideration of the nature of the program and schedule constraints, LPC's decision was to retain the forward inhibitor/release flap component while eliminating the aft end release.

(U) As recognized, a unique aspect of the integral ramjet booster propellant grain design, which is distinct from other solid rocket motors, is the configuration of the DC 93-104 insulation between the grain and the motor case. The characteristics and configuration of this insulation are dictated by the ramjet, not the solid booster, so its construction is unusual for solid rockets. In particular, the insulation is perforated with approximately $3000 \frac{9}{32}$ -inchdiameter radial outgassing holes, each hole having a non-elastomeric material filling the perforation. In a solid rocket motor it is important that the insulation be, for all practical purposes, incompressible, a condition that can be met if the perforated portions of the insulation must be capable of supporting all the bond stresses. Periodic bond patterns such as this have been extensively studied in conjunction with stress-relieving liner concepts, and found to be favorable under low temperature thermal conditions. Howeever, due to the reduced bond area, a periodic stress variation results in a

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(U) stress factor of two above the average for normal non-interrupted bonds. Both the reduced area and the increased stress factors were applied to the computer solution (which was related to a continuous bond) and effects of these factors are included in the computed structural safety margins.

(U) <u>Conclusions</u>. Table 3-9 summarizes the results of the propellant grain structural analysis. For purposes of clarification, the following is a summary of the conditions under which the analysis was performed.

- These analyses were performed using an axisymmetric computer program, assuming a 145°F propellant cure temperature
- An integration of the hoop strains from the generalized plane strain run was performed to determine an effective web to be used in the axisymmetric run
- Three configurations were run: full head-end bond, half headend bond (to edge of glass closure), and no head-end bond
- Each configuration was run with and without an aft release flap
- An accounting for the reduced area due to the insulation holes was made, increasing the induced stresses

(U) The following are the conclusions derived from the analysis:

- The critical region for the minimum bondline safety factor occurs at the head-end for shear stress for the combination of thermal plus pressure loading.
- Each bond configuration shows an adequate factor of safety to guarantee the structural integrity of the grain. Therefore, flaps are not required by the grain structural analysis.
- The minimum safety factor (1.5) remains strain critical, and occurs at the longitudinal center of the grain. This safety factor is adequate to guarantee the grain structural integrity.

3.1.4 Thermal Analysis

(U) Presented in this section is the thermal analysis of the integral rocket/ ramjet booster. Components analyzed included booster internal insulation, ramjet case forward dome cover, booster nozzle and ramjet nozzle.

3.1.4.1 Booster Internal Insulation (Case Bonded)

(U) Insulation thickness requirements for the side wall of the ASALM combustion chamber/booster case are basically set by the needs of ramjet mode operation. Work to date under the Ramburner Freejet program by The Marquardt Corporation has established this requirement to be 0.375 inch thickness uniformly over the side-wall, of the baseline DC 93-104 material. However, consideration of the total integrated propulsion system must necessarily factor into the design the impact of booster operation on this insulation material prior to the onset of ramjet operation. Although of comparatively short duration, booster operating temperatures are somewhat higher than ramjet operating temperatures, and local flow velocities associated with grain configuration can be high. Degradation of the insulation available for ramjet operation can, therefore, be expected, at least locally.

(U) The specified DC 93-104 insulation is a silica- and silicon-carbidefilled elastomer containing carbon reinforcement fibers. This material forms a firm, porous char layer, resulting in minimal postfire afterburning and ejectable debris as compared to more commonly used rocket motor insulations. Lockheed has assessed the impact of booster operation on ramburner insulation design. This assessment is well founded and based on analysis backed by empirical data in actual motor firings. The impact is minor, but does require careful consideration of what, if any, effect accrues during ramjet operation. The design basis assumed was that the thickness of virgin DC 93-104 insulation at the start of ramjet operation must be at least equal to the minimum, nominal thickness of 0.375 inch. This requires that a maximum additional thickness of 0,140 inch insulation be added during initial fabrication to the location beneath the grain slot. The additional insulation thickness is less at locations away from the slot, and at angular locations beyond about 60 degrees away on each side, no additional insulation is required beyond that needed for ramjet operation. This tapered configuration thus results in a non-circular mandrel for insulation casting.

(U) Joint review of these results in coordination meetings with ramjet and systems contractors has indicated the desirability of maintaining a circular insulation configuration. This simplifies insulation mandrel design and the off-gas hole and filler fabrication sequence. Since the 0.375-inch uniform side-wall insulation thickness specified for ramjet mode operation is more than adequate for case protection during rocket mode operation, the possibility exists for making no local increase in thickness, providing that local degradation of insulation at the start of ramjet mode operation is acceptable. LPC data indicate that this local degradation consists of thermal conversion and normal char formation by the DC 93-104, with the char remaining in place. This corresponds to normal behavior of the material under ramjet operating environment, under which the material is designed to char through completely before ramjet burnout. Thus, the net effect of

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(U) rocket operation appears to be a somewhat earlier completion of char formation in the local region beneath the grain slot. This question of design basis for rocket mode insulation requires Air Force resolution.

(U) Analysis. Rocket booster internal insulation requirements and thermal analysis locations are summarized in Figure 3-19. The design requirement to maintain a +400°F maximum temperature at the ramburner insulation interface prior to ramjet ignition was established. This isotherm represents the initiation of density change determined by thermogravimetric analysis. This requirement is met at all locations by the insulation design. The DC 93-104 booster insulation tapers uniformly both longitudinally and circumferentially. Beneath the keyhole slot centerline, the insulation is 140 mils thick at the aft end and tapers to 70 mils at the forward end. The insulation also tapers circumferentially from a maximum beneath the slot centerline to 55 degrees on either side, where it blends into the existing ramjet insulation. The remaining 250 degrees of the motor circumference has only 20 mils of LPL-59 liner. The ramjet nozzle entrance requires an additional 100 mils of silica-phenolic or 200 mils of DC 93-104 for booster operation. The motor forward closure requires a uniform 70 mils of DC 93-104.

(U) Thermal gradients for the booster insulation (DC 93-104), including char depth $(+800^{\circ}F$ isotherm) and $+400^{\circ}F$ isotherm locations, were obtained from the Charring Material Thermal Response and Ablation Program (CMA III). As a further check on the thermal model, LPC conducted an independent char depth correlation study based on all available industry firing data with DC 93-104. This study revealed good agreement with the char depths as predicted by the CMA III program.

(U) The complete analytical sequence of the thermal analysis is depicted in Figure 3-20. The molecular composition of the combustion products in the motor chamber for both equilibrium and frozen flow were determined with the LPC Chamber Gas Thermochemistry Program. In addition, this program calculated, as a function of static pressure, the static flame temperature, enthalpy, entropy, molecular weight, specific heat at constant pressure, one-dimensional velocity, and gas density for the nozzle flow expansion process.

(U) The data obtained from the Thermochemistry program were subsequently used in the LPC Chamber Gas Transport Properties Program to determine the viscosity, heat capacity, thermal conductivity, Prandtl number, and density of the gas mixture, required for the flow field analysis.

(U) The Equilibrium Surface Thermochemistry Program (EST III) was then used to calculate the variation of enthalpy with temperature for the molecular composition of the boundary layer edge gas. The thermodynamic state at the surface of the ablating insulation as a function of pyrolysis gas and char rates, normalized with respect to mass transfer coefficient, was also determined from the EST III program. The data obtained from this program were subsequently used in the CMA program to perform surface energy balance calculations.

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Thermal Insulation Requirements for Case-Bonded Design Figure 3-19



Figure 3-20 Method of Thermal Analysis of Internal Insulation

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(U) The next step was to determine the necessary input for the Charring Material Thermal Response and Ablation Program, CMA III. This program is an explicit finite difference solution for transient transport of thermal energy in a three-dimensional isotropic material that can ablate from a surface and can decompose in depth. The program calculates the transient thermal response of a composite slab containing several in-depth charring and noncharring materials, as well as the surface recession rate resulting from diffusion or kinetically controlled surface chemical reactions and/or mechanical melt removal.

(U) The CMA code, developed by Aerotherm/Acurex Corporation, is one of the most sophisticated ablation models but does not possess the degree of generality required to accurately account for the effects of swelling, "coking", and in-depth changes in pyrolysis gas chemical composition. In practice no ablation code exists that incorporates all of these effects. To allow for these effects, which are prevalent in the DC 93-104 insulation material, the CMA thermal modeling was partially modified in accordance with the results reported by the Naval Weapons Center (NAVWPNSCEN) in References 3-1 and 3-2. Thermal modeling conducted by NAVWPNSCEN is briefly described below, as quoted from Reference 3-1:

"NAVWPNSCEN developed a set of thermochemical properties for use in the CMA code from available in-depth and external surface temperature measurements. These temperature data were accumulated from tests at NAVWPNSCEN, The Marquardt Company, and UARL. All known properties of DC 93-104 (e g, virgin thermal properties, decomposition temperature thresholds, resin fraction, virgin density) were input to CMA. The unknowns (e g, chemistry of pyrolysis gases, decomposition rates, high-temperature thermal properties, elemental composition of char) were simply estimated and adjusted in such a way as to force agreement between CMA predictions and the bulk of the temperature data."

(U) The CMA III thermochemical properties used by LPC are presented in Figure 3-21 and Table 3-10. Additional CMA III input involved the convective heat transfer coefficients and radiation heat fluxes. The derivation of these parameters is discussed in the subsequent text.

(U) At this point, a flow field analysis was conducted to determine the timedependent velocity profiles necessary for the calculation of the convective heat transfer coefficient at the analysis locations. The velocity profiles were obtained using a one-dimensional continuity mass flow balance as shown below:

$$V_{G} = \frac{r_{b}A_{b}\rho_{p}}{\rho_{G}A_{f}}$$
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Table 3-10

THREE-COMPONENT DECOMPOSITION MODEL

Component	ρ _{oi} lbm/ft ³	ρ _{ri} lbm/ft ³	Bi Sec ⁻¹	Ψ _i	Ei/R, ⁰ R	Minimum Temperature of Reaction, ⁰ R
A (Resin)	60.75	32.4	0.448×10^{10}	3.0	0.368×10^5	+1360
B (Resin)	20.24	0	0.140×10^5	3.0	0.154×10^5	+1360
C (Reinf.)	110.00	110.00	0	0	0	+9000

Resin Decomposition Gas Enthalpy

Temperature, ⁰ R	900	1800	2700	3600	4500	5400
Enthalpy, Btu/lbm	-2450	-1340	110	2200	3250	4500

 $\frac{\partial \rho_{i}}{\partial t} - B_{i} \rho_{oi} \left[\frac{\rho_{i} - \rho_{ri}}{\rho_{oi}} \right] \psi_{i} \exp \left[-E_{i} / RT \right]$

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(U) Where

 V_{C} = gas velocity at the station investigated

A_L = propellant exposed surface area at corresponding time

 A_{f} = corresponding flow area

 $\rho_{\rm p}$ = propellant density

 $P_{C} = gas density$

(U) After establishing the flow field definition, the convective heat transfer coefficients and the radiant heat flux to the surface were calculated. Convective heat transfer coefficients were calculated with either the conventional Bartz equation for rocket nozzles, or the Colburn analogy for a turbulent flat plate, along the chamber sidewall:

$$Nu_{\rm m} = 0.0296 \ \mathrm{Re_{+0.80}} \ \mathrm{Pr^{+0.33}}$$

with Reynolds numbers selected to reflect the time-dependent insulation exposure to gas flow.

(U) The radiative heat flux boundary condition was calculated on the basis of the assumption that the particle-laden gas stream was optically thick, and that the particles and the wall exchange radiant energy as if they were two parallel plates. In addition, it was assumed that both the stream and the insulation behave as gray bodies, and that they emit and reflect radiant energy diffusely. Based on the above assumptions, the radiative heat flux boundary condition is:

$$q_r = \epsilon_{eff} (\sigma T_s^4 - \sigma T_w^4)$$

where

$$\epsilon_{\text{eff}} \text{ (effective emissivity)} = \frac{1}{\left(\frac{1}{\epsilon_{\text{w}}} + \frac{1}{\epsilon_{\text{s}}} - 1\right)}$$

 σ = Stephen Boltzman constant

 $\epsilon_{\rm w}$ = wall emissivity

 ϵ_{c} = particle-laden stream emissivity

T₂ = local stream static temperature

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(U) The effective emissivity was evaluated using a value of 0.9 for the wall emissivity and using the following relationship for the emissivity of the particle-laden combustion products:

$$\epsilon_{\rm g} = 1 - \exp\left(-c \frac{n}{16} \rho D\right)$$

where

C = propellant formulation dependent experimental constant

n = percentage of aluminum loading

 ρ = local density of propellant combustion species

D = local beam length

(U) To check the char depth obtained from the CMA III analyses, a separate char depth correlation study for DC 93-104 was conducted. Previous rocket motor firing experience is listed in Table 3-11. The material has been under' evaluation and in use as a lower-cost, superior-performance ablative material by the Navy, Air Force, LPC, and industry since 1968. DC 93-104 has been used in variety of motors in various component locations (e g, chamber sidewall, blast tube) that encompass wide ranges of heat flux, gas velocity, and propellant composition (covering the entire operating regime of the rocket booster motor).

(U) Investigated variables that influence char rate were local pressure, combustion temperature, insulation internal diameter, local gas velocity, and exposure time. The three primary variables influencing char rate were found to be gas velocity, combustion temperature, and exposure time. Computer analyses of the available data resulted in an exponential curve-fit equation. All data points examined fitted this equation within a ± 2 sigma limit where sigma equals 10.25 percent. Based on the rocket booster operating conditions, a set of design curves was generated, as shown in Figure 3-22. As stated previously, this char rate correlation was in good agreement with the CMA III analyses. For purposes of rapidly determining preliminary DC 93-104 insulation sizing, this char rate correlation appears to be a good design tool.

3.1.4.2 Booster Internal Insulation (SVS)

(U) This Subsection describes the thermal design of the booster insulation for the alternate SVS approach. The primary function of the booster insulation, as discussed in Subsection 3.1.4.1 is to thermally protect the ramjet combustor insulator during booster burn. For design purposes, this requires that the ramjet insulator interface temperature be maintained below +400°F prior to ramburner ignition. This requirement is met by the insulation design presented in Figure 3-23.

10-Inch-Dia HARM 1 Chamber Boost 1657 3.5 HTPB/86/1(a) Demonstration Motor 3 Chamber 1200 3.0 HTPB/86/1(a) Subscale Motors 3 Chamber 1200 3.0 HTPB/86/1(a) Subscale Motors 1 Chamber 1200 3.0 HTPB/86/1(a) 6-Inch-Dia HARM 1 Chamber 1200 3.0 HTPB/86/1(a) 6-Inch-Dia HARM 1 Chamber 1200 3.0 HTPB/86/1(a) Subscale Motors 1 Chamber 1200 3.0 HTPB/86/1(a) Subscale Motors 1 Chamber 1200 3.0 HTPB/86/1(a) Subscale Motors 6 Chamber 1200 3.0 HTPB/86/1(a) Subscale Motors 6 Chamber 1200 1.5 HTPB/86/1(a) Subscale Motors 6 Chamber 1000 1.5 HTPB/86/1(a) Subscale Motors 6 Chamber 1000 1.5 HTPB/86/1(a) Subscale Motors 6 Chamber 1000 1.5 HTPB/86/1(a) Demonstration Motors 6 Blast Tube 800 7.4 CTPB/88/17.5 Agile Nozzle Material <th>3.5 HTPB/86/1^(a) 9.0 (LPC-801A) 9.0 HTPB/86/1^(a) 3.0 HTPB/86/1^(a) 3.0 (LPC-801A) 3.0 HTPB/86/1^(a) 3.0 (LPC-801A) 3.0 HTPB/86/1^(a) 3.0 HTPB/86/1 1.5 HTPB/86/1 1.5 HTPB/86/1 1.5 HTPB/86/18 1.5 (LPC-691A) 15.0 (LPC-691A) 15.0 (LPC-691A) 15.0 TPPB/86/18 7.4 CTPB/88/17 7.4 CTPB/88/17.5 7.4 CTPB/88/17.5 7.4 TPB/86/0</th> <th>LPC LPC LPC LPC LPC LPC LPC LPC</th>	3.5 HTPB/86/1 ^(a) 9.0 (LPC-801A) 9.0 HTPB/86/1 ^(a) 3.0 HTPB/86/1 ^(a) 3.0 (LPC-801A) 3.0 HTPB/86/1 ^(a) 3.0 (LPC-801A) 3.0 HTPB/86/1 ^(a) 3.0 HTPB/86/1 1.5 HTPB/86/1 1.5 HTPB/86/1 1.5 HTPB/86/18 1.5 (LPC-691A) 15.0 (LPC-691A) 15.0 (LPC-691A) 15.0 TPPB/86/18 7.4 CTPB/88/17 7.4 CTPB/88/17.5 7.4 CTPB/88/17.5 7.4 TPB/86/0	LPC LPC LPC LPC LPC LPC LPC LPC
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Table 3-11

DC 93-104/SOLID PROPELLANT FIRING EXPERIENCE

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Figure 3-22 DC 93-104 Char Rate Curves

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Figure 3-23

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(U) The SVS system utilizes a propellant cup that is externally supported by silicone fluid. In the alternate design, the combustor insulator is thermally protected by the insulative capabilities of the propellant cup, with only the aft closure needing additional insulation for booster operation. As for all previous SVS motors, the propellant cup is fabricated from a Lockheed-developed carbon black and asbestos-filled polybutadiene-polyisoprene elastomer, designated LPE-6.

(U) Thermal insulation requirements were determined with one of Lockheed's Charring and Material Ablation computer codes, CHIRP IV. The code employs an nth order rate equation to define the decomposition of a material, while calculating the in-depth, one-dimensional thermal response utilizing an explicit finite difference formation.

(U) The six analysis stations are basically the same as for the baseline design. The primary difference is Station 1 in the forward closure, where the exposure time has been reduced by a head-end web over the entire dome. The analytical procedures used are thus identical to those of the baseline case-bonded design except that the Chamber Gas Thermochemistry and CHIRP IV programs have been substituted for the EST III and the CMA III programs (Refer to Figure 3-20).

(U) For the entire forward dome and 250 degrees circumferentially of the chamber sidewall, 40 mils of LPE-6 cup insulation is required. This thickness is the minimum practical calendered sheet size for reliable, defect-free fabrication and handling. The LPE-6 cup thickness beneath the keyhole slot region tapers both longitudinally and circumferentially. The taper is uniform longitudinally from 250 mils at the aft end to 40 mils at the forward end beneath the keyhole centerline. Circumferentially, the LPE-6 cup insulation tapers from a maximum beneath the slot to a minimum of 40 mils, blending into the remaining cup insulation at the 55-degree web burnout location on either side of the keyhole slot. The aft closure/ramjet nozzle entrance section requires 100 mils of silica-phenolic or 200 mils of DC 93-104 for booster operation.

3.1.4.3 Ramjet Case Forward Dome Cover

(U) In the case-bonded booster design, the dome cover is thermally protected from direct exposure to the high temperature combustion gases by 70 mils of secondarily bonded LPE-17. The insulator and the adhesive system is identical to that utilized for the stress relief flaps. This forms a protective layer, that in the postfire, charred condition would not be expected to prevent the proper fracturing of the plug when it is blown out. General Electric RTV-102 silicone was used to protect the glass plug in the Martin-Marietta/LPC transition test motors. This material satisfactorily insulated the plug, yet allowed it to fracture upon command.

(U) With the SVS booster configuration dome cover, insulation is not required in that the dome is protected from the combustion gases by the SVS cup and silicone fluid.

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3.1.4.4 Booster Nozzle

(U) The baseline booster nozzle is Air Force-supplied and analysis was therefore limited to a cursory evaluation to ensure compatibility with the LPC booster motor designs. Evaluation items included basic dimensions, tolerances, materials and simple thermostructural analysis at critical locations. The GFE design is compatible with the LPC booster designs. Final analysis will require waiting for the conclusion of nozzle development testing under separate Air Force contract.

3.1.4.5 Ramjet Nozzle

(U) During the course of the program, and at the request of The Marquardt Corporation, DC 93-104 was evaluated for potential use as the ramjet nozzle liner material. Parameters evaluated were surface recession and the sealing of the booster/ramjet nozzle interface against gas flow during booster operation. Figure 3-24 shows the ramjet nozzle surface recession and char profiles which occur during booster operation. Circumferential location is in line with the propellant slot, which is the limit condition. The recession ranges from 0.060 to 0.175 inch and char depth (400°F isotherm) ranges from 0.140 to 0.275 inch. This amount of recession results in a significant step in the entrance to the throat region of the ramjet nozzle. This step will most probably degrade the ramjet nozzle performance, the magnitude of which can only be established by extensive flow analysis or testing. Industry experience would suggest a maximum nozzle performance degradation of 6 percent.

(U) In regard to gas sealing, the DC 93-104 can readily be processed to form the proper surface for low pressure sealing, but the compressibility under motor operating pressure would result in a weak or nonfunctional seal.

3.2 FLIGHTWEIGHT MOTOR DESIGN

3.2.1 Baseline Design Description

(U) The motor design with the baseline case-bonded grain retention system is shown in Figure 3-25 and a comparison of its characteristics with the specified requirements is given in Table 3-12. This table shows that the case-bonded approach is capable of meeting or exceeding all requirements as specified in the RFP. Characteristics of the baseline design and the reasons for the selected approach were presented in Section 2.

(U) The motor cases are government-furnished equipment items, complete with DC 93-104 insulation in place. The ejectable booster nozzle design is based upon results from the RPL Ejectable Nozzle Development contract, and procured per government-furnished design drawings.

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Figure 3-24 Ramjet Nozzle/Booster Insulation Surface Recession and Char Profiles

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Table 3-12

CASE-BONDED MOTOR PERFORMANCE SUMMARY (Sea Level and +70°F)

Parameter	RFP Requirement	Proposed Design
Nominal action time, sec	4.1	4.07
Boost thrust, 1bf	30,000	30,000
Maximum chamber pressure, any temperature, psia	1820	1820
Propellant π_k , $\%/^0 F$	0.20 max	0.15
Delivered specific impulse, sec	245 min ^(a)	250 ^(b)
Action time total impulse, lbf-sec	122,100	122,100
Case length, in.	44.0 max	41.65
Total motor weight, 1b	862.5 max	846.8 ^(c)

 (a) 1000 psi P to 14.7 psi expansion with a 15-degree half-angle; motor delivered value corrected to same conditions is 247 seconds.

(b) Delivered under motor conditions

⁽c) Based on hardware weights reported in Martin-Marietta Company's Configuration Control and Performance Bulletin, dated January 1973. See Table 2-3 for detailed breakdown.





Figure 3-25 Case-Bonded Motor Design

(The reverse is blank)

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(U) The motor case design includes the ramburner nozzle housing as an integral part of the ramburner/rocket booster chamber. Case material is an L-605 cobalt-base alloy. The chamber is 18 inches in outside diameter with the cylindrical length established at the minimum required to meet booster performance requirements.

(U) Based on the design studies in this phase of the contract, the cylindrical chamber length for the case-bonded design is 41.65 inches from the forward dome flange to end of the cylindrical section. For the alternate SVS design, the chamber cylindrical length is 42.07 inches. The forward dome of the chamber includes a port which interfaces with the ramjet air intake duct. The dome port incorporates mounting and sealing surfaces for the dome closure, which separates the air intake duct from the chamber during booster operation.

(U) The chamber/ramburner nozzle entrance is insulated with DC 93-104. The cylindrical section and forward dome insulation is 0.375-inch thick, except for the area under the propellant port keyhole slot, where it is increased 0.140-inch to 0.515-inch directly under the keyhole slot, and tapers uniformly for 55 degrees on each side of the slot to the basic 0.375inch thickness. The insulation thickness varies through the inlet, throat, and exit cone sections of the nozzle.

(U) The ejectable rocket booster nozzle seats within the ramburner nozzle and is retained at the exit cone outer diameter by a retention/ejection mechanism. An O-ring is used to seal the interface between the rocket booster nozzle and the ramburner nozzle throat.

(U) To provide insulation aeroheat off-gassing paths, the cylindrical section of the case insulation is drilled to provide a pattern of $\frac{9}{32}$ -inchdiameter holes on 0.75-inch centers for the escape of pyrolysis gases. The holes are refilled with plaster-celogen pellets to support the propellant grain in the case-bonded design. In the SVS design, the holes are filled with the silicone fluid except for the area under the SVS seal, where the holes are filled with pellets to support the seal.

(U) The case-bonded grain is provided with a release flap at the forward end of the cylindrical section of the grain. This forward release flap is bonded to and inhibits the forward face of the propellant grain, and extends aft along the cylindrical section of the grain for 1.50 inches beyond the case tangency point. The release flap material is 0.070-inch-thick LPE-17.

(U) The ramjet booster motor igniter consists of a tubular Teflon cannister filled with a granulated magnesium Teflon pyrotechnic output charge, and contains two initiators for ignition redundancy. The igniter cannister is fabricated from two vacuum-formed tetrafluoroethylene (TFE) end caps which house the initiators and a TFE tube which contains the output charge. A groove is machined lengthwise on the outside of the tube wall to provide a reduced wall thickness for directing the burning particles toward the propellant surface. The TFE end caps and tube are assembled by a heatsealing technique that makes the igniter airtight.

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(U) The initiators are potted into the end caps and the assembly is installed in a molded rubber holder mounted on the nozzle. The high-energy initiator, previously used on the SRAM missile, has a high no-fire capability (5 amp/ 1.5 watts for 5 minutes). Firing current for the initiator is 15 amps at 28 volts DC.

(U) A critical review of the proposed GFE case design identified two design areas which require additional analysis: the capability of the L-605 case material and of the frangible pre-stressed glass dome to perform adequately under booster rocket motor operating conditions. The L-605 alloy has limited solid rocket motor case usage, and most of the available test data are for elevated temperatures where the alloy has adequate ductility for low pressure ramjet applications. The solid rocket motor operates at temperatures as low as $-65^{\circ}F$ and at very high pressures. Resistance to flaw growth and brittle fracture, therefore, is a prime requirements for the chamber material and the chamber must be designed to meet fracture mechanics, as well as structural strength, criteria.

(U) The potential problem areas resulting from the design environments are:

- The development motor chambers will be subjected to thermal cycles and vibration loads before motor firing at -65, +70, and +165°F.
- (2) The chambers are fabricated from roll and welded (TIG) L-605 sheet that is cold reduced by shear spinning and then girth welded (EB). The chamber also contains numerous spot welds to anchor the convoluted retaining bonds for the internal insulation.
- (3) The L-605 (20 percent cold worked and EB welded) has low ductility (1 percent elongation).
- (4) The fracture strength and flaw growth resistance of L-605 (cold worked and welded) at room temperature and -65°F are unknown.
- (5) Catastrophic chamber fracture during proof-testing or motor firing appears to be a potential problem.
- (6) Fracture toughness and flaw growth data for L-605 are needed so that a fracture mechanics analysis can be conducted to arrive at the allowable chamber loads and the required proof test conditions.

(U) The forward dome closure to chamber boss joint, as designed in the government-furnished drawings, is not adequate for rocket motor operation:

 No positive seal is provided between the closure and chamber. An O-ring is normally used to seal this type of joint in rocket motor design. An O-ring seal allows for tolerance differences

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- (U) between the mating parts and will function to seal the joint increased gap due to chamber boss rotation under rocket motor operating pressure.
 - (2) No positive retention is provided against inward movement of the closure. The rocket motor propellant grain will shrink away from the closure during cure, and therefore provides no support.
 - (3) Any dome closure which must be installed from the aft end should be removable for motor processing. Access through the forward boss is required for installation of the pellets which are used to fill the holes in the insulation and to simplify motor processing.
 - (4) If the dome closure should become damaged after the rocket motor propellant grain is cast, with the design shown, the closure could not be replaced without the loss of the booster grain.
 - (5) Data for cyclic tests (pressure and temperature) and burst tests at room temperature and -65°F are required so that design criteria and inspection requirements for the prestressed glass dome closure can be defined.

(U) Design changes to correct the noted deficiencies and test programs required to provide data in the critical areas noted should be completed prior to the initiation of work on Phase III of the program. In addition, any changes and/or data generated should be subject to review by the ramjet booster contractor to ensure that the rocket motor environmental conditions can be met.

(U) These two items have been discussed at coordination meetings with other contractors. A program to develop fracture toughness data for the case material is underway at Air Force Materials Laboratory. However, it is considered essential that a fracture mechanics analysis be performed by the booster contractor to ensure acceptable performance in flightweight motor demonstration tests.

(U) Furthermore, a task was undertaken by the ramjet contractor to examine redesign details of the forward dome closure to chamber joint. The objective of this task is to answer the points raised above by appropriate design changes.

3.2.2 Summary of Demonstration of Baseline Design Approach

(U) Extensive design, analysis, and testing work has been conducted on all aspects and features of the baseline design. This Subsection illustrates the direct relationship of these tasks to the features of the proposed full-scale design.

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(U) Table 3-13 provides a comprehensive overview of the tests conducted. Figure 3-26 shows the configuration of the subscale case-bonded motor used in the LPC/Martin transition demonstration tests. This configuration, without ejectable nozzle and frangible glass port cover, and approximately 2 inches shorter in length, is identical to that used in LPC's other subscale motor firings and analog motor tests.

(U) These subscale motors incorporated all of the features and functions of a full-scale motor. They are in fact essentially identical except for size (6-inch versus 18-inch diameter) to the full-scale heavyweight motors which were planned for the program. These 6-inch subscale motors had test objectives keyed to proof of specific areas of the design such as ballistic performance, propellant grain structural integrity, interface bond integrity, process development and verification, and residuals effects. These are the same objectives that apply to the full-scale heavyweight motor test program specified.

(U) Following are those features, functions, and components of the fullscale motor design that are deemed critical to successful operation, together with a summary of how each has been proven through test at LPC:

	LPC Approach	How Proven
Propellant	LPC-691	Fully characterized ballistically:
		 10 subscale motors at full-scale motor condition
		Fully characterized structurally:
		• In subscale motors -65 to +165°F
		• In analog motors to -100°F
		Fully characterized processability:
		 In 65 mixes ranging from 1 to 10 gallon sizes
Grain Configuration	Keyhole	12 successful subscale tests have proven:
		• Desired ballistic curve shape
		Minimum sliver
		• Sharp tailoff
		• Minimum inert residuals
		 Successful transition and ramjet burn
	-84-	 No erosive burning at port-to- throat of 1.3
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Table 3-13

SUMMARY OF TESTS CONDUCTED IN SUPPORT OF CASE-BONDED INTEGRAL RAMJET BOOSTER TECHNOLOGY

Test	Date	Description	Results
1	March 1973	Four 6- by 11-inch ballistic motor firings at -65 to +165°F	Success: Propellant burn rate, slope, and w_k verified
2	March 1973	6- by 33-inch motor fired at +70 ⁰ F with port-to-throat ratio of 1.0	Success: No evidence of erosive burning pressure spike
3	April 1973	6- by 11-inch case-bonded prototype motor fired at +70°F	Success: Materials interface grain support and ballistics demonstrated
4	June 1973	6- by 14-inch case-bonded motor (Martin/ LPC) fired at +70°F	Success: Static verification of case- bonded transition motor configuration
5	August 1973	6- by 14-inch case-bonded motor (Martin/ LPC) fired at +70°F in Orlando with nozzle and port cover ejection, plus transition to ramjet combustor burn at sea level conditions	Success: Full transition in <836 milli- seconds with minor residuals effect
6	October 1973	6- by 14-inch case-bonded motor (Martin/ LPC) fired at $+70^{\circ}$ F in Orlando with ::ozzle and port cover ejection, plus transition to ramjet combustor burn at sea level condi- tions with offset grain mandrel	Success: Full transition in <442 milli- seconds with minor residuals effect
7	October 1973	6- by 14-inch case-bonded motor (Martin/ LPC) fired at +70°F in Orlahdo with nozzle and port cover ejection, plus transition to ramjet combustor burn at 24K-ft conditions	Success: Full transition in <646 milli- seconds with minor residuals effect
8	October 1973	6- by 11-inch case-bonded motor fired at -65°F	Success: Materials interface, grain support and ballistics demonstrated at low temperature
9	October 1973	6- by 11-inch case-bonded motor fired at +70°F with alternate bond system	Success: Alternate bond system materials interface, grain support, and ballistics demonstrated
10	December 1973	Two 6- by 14-inch structural analog motors cooled to -90°F, then thermal cycled between -65 and +165°F with freeze-to- failure	Success: No structural or bond failure to -90°F or during thermal cycling
11	December 1973	6- by 11-inch case-bonded motor fired at -65°F with alternate materials interface system	Success: Alternate materials interface system, grain support, and ballistics demonstrated at low temperature
12	December 1973	6- by 11-inch case-bonded motor fired at +165°F with alternate materials interface system	Success: Alternate materials interface system, grain support, and ballistics demonstrated at high temperature
13	December 1974	6- by ll-inch case-bonded motor fired at +70°F with off-gas holes and hole fillers	Success: Materials interface system including holes and fillers, grain support, and ballistics demonstrated
14	December 1974	6- by 11-inch case-bonded motor fired at -65°F with off-gas holes and hole fillers	Success: Materials interface system including holes and fillers, grain support, and ballistics demonstrated at low temperature

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Figure 3-26 Case-Bonded Transition Test Demonstration Motor

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(U)	LPC Approach	How Proven
DC 93-104/Propella bond interface	ant LPL-63/ LPE-18/ DC 93-104	Over 400 bond specimens fabri- cated and tested
		42 motors successfully fabricated, containing over 35 pounds of DC $93-104(a)$
		Successful temperature cycle and firing from -65 to +165°F
		Three successful transition tests with no effect of residuals
Release flap configuration and material	LPE-17/ LPE-18	Over 80 components successfully fabricated and installed
		Proven in 18 tests from -65 to +165°F
		Proven structurally to -100°F
Minimum inert residuals	LPC keyhole grain LPE-18/DC 93-104 bond interface	Three successful case-bonded transition tests with no over- pressure during ramjet burn.
		Successful transition demon- stration in less than allowable 750 milliseconds
Off-gas hole filler	Plaster/celogen pellets in drilled holes	Successful demonstration in motor tests at $+70$ and $-65^{\circ}F$ with no effect on ballistics, grain retention, thermal
		protection or residuals

3.3 SVS APPROACH EVALUATION

3.3.1 Basic SVS Concept

(U) The basic SVS concept is shown schematically in Figure 3-27. The grain rests in the case with viscous liquid surrounding it on three sides. (In the schematic sketch, the annulus is magnified for clarity.) The annulus gap is of sufficient size to provide a small, positive clearance between the

⁽a) Includes motors fabricated on other LPC programs.

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(U) cup and insulation at the upper temperature extreme. A flexible seal retains the liquid, and a nozzle closure maintains atmospheric pressure during storage and captive flight.

(U) The supporting liquid and the plenum pressure retain the grain. The flexible seal serves only to retain the liquid and is under very little load except momentarily at ignition.

(U) The key concept in this retention system is that of a controlled response to loading conditions. The viscosity of the supporting liquid and the width of the annulus dictate how the SVS responds to the load. By varying these two parameters, an SVS may be designed so that the grain can move easily under slowly applied loads and yet be retained during rapidly applied, short-duration loads with only small movements. These characteristics are ideally suited to the needs of a grain retention system that must resist movement during events of short duration (seconds), such as vibration, shock, acceleration, and pressurization, while allowing stress relief during events of long duration (hours), such as thermal cycling. The response of the SVS to each of the above loading conditions is described in Appendix C.

(U) The motor design with the alternate SVS grain retention system is shown in Figure 3-28 and a comparison of its characteristics with the specified requirements is given in Table 3-14. The SVS approach is capable of meeting or exceeding all requirements as specified in the RFP.

(U) In the SVS concept the grain is supported in the case by a viscous liquid. The grain is contained in a rubber cup, and the support liquid fills the space between the cup and the motor case insulation. A flexible seal between the aft end of the cup and the insulation retains the liquid. This seal is shown in Figure 3-29. The reinforcement material is LPE-15, a nylon-fabricreinforced neoprene rubber that is vulcanized to the propellant cup. Cup thickness is 0.060 inch, locally thickened to a maximum of 0.250 inch in the region of the propellant grain slot. The fluid cavity between the propellant cup and the case insulation is a 0.030-inch annulus along the cylindrical section and a 0.42-inch longitudinal gap between the cup and forward dome.

(U) While the case-bonded approach meets all requirements and appears to be the lowest-risk approach, there are potential advantages of the SVS as an alternative configuration for the integral ramjet booster:

- (1) If yet unforeseen bonding problems are encountered with the case-bonded approach, the SVS concept provides a means of eliminating the need for bonding (except in the low-stressed SVS seal area).
- (2) If the presence of residuals creates transition problems (not now anticipated), the SVS approach permits expulsion of the propellant cup and the residuals left in it during the transition event.

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Table 3-14

ALTERNATE (SVS) MOTOR PERFORMANCE SUMMARY (Sea Level and +70°F)

RFP Requirement	Proposed Design
4.1	4.07
30,000	30,000
1820	1820
0.20 max	0.15
245 min ^(a)	250 ^(b)
122,100	122,100
44.0 max	42.07
862.5 max	848.5 ^(c)
	RFP Requirement 4.1 30,000 1820 0.20 max 245 min ^(a) 122,100 44.0 max 862.5 max

 (a) 1000 psi P_c to 14.7 psi expansion with 15-degree half-angle; motor delivered value corrected to same conditions is 247 seconds

(b) Delivered under motor conditions

(c) Based on hardware weights reported in Martin-Marietta Company's <u>Configuration Control and Performance Bulletin</u>, dated January 1973. See Table 2-17 for detailed breakdown.

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Figure 3-28 Alternate -93-Motor Design

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Figure 3-29 Cup and Seal Configuration

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- (U) (3) A potential problem is the use of a large number of small radial holes in the case insulation to permit the escape of pyrolysis gases given off by the insulation as a result of aerodynamically-induced backside heating during ramjet operation. For a case-bonded booster grain, these holes must contain a filler until after booster operation is completed. The SVS design overcomes such a potential complication. The fluid of the SVS system would fill the holes until booster/ramjet transition.
 - (4) The SVS approach provides an essentially stress-free propellant grain, thus permitting the use of higher energy propellants--not suitable for a case-bonded design because of their poorer physical properties. Use of SVS would also permit use of concepts such as LPC's Grain Burn Pattern Regulation (GBPR) which provides essentially 100-percent volumetric loading. Use of GBPR in the Internal Ramjet Booster motor results in a 2.3-inch reduction in motor length. Thus the SVS approach provides a greater performance growth potential than available with the case-bonded design.

(C) Characteristics of the proposed alternate motor design and the reasons for the selected approach are summarized as follows:

SVS DESIGN

	Characteristics			Reasons For Selection
(1)	Sliverless, neutral-burning grain with "keyhole" port and a 0.76 web fraction	(1)	•	Optimum tailoff Minimum case weight Good processability and reliability
(2)	87-percent solids, 18-percent aluminum propellant, LPC-691D with ultra-fine ammonium per- chlorate (UFAP) oxidizer to provide high burning rate	(2)	•	Exceeds performance requirements No liquid burning rate catalyst Good physical properties and processability
(3)	LPE-6 propellant cup with SVS grain retention	(3)	•	Permits ejection of cup and residuals within it LPE-6 proven in SVS applications Cup also serves as booster insulation

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(U) Characteristics

(4) DC-510 fluid/flexible seal SVS system

Reasons for Selection

- (4) No grain/case loads, avoids potential case-bonding problems
 - Fluid and seal design proven in SVS application
 - Fluid shown to be compatible with DC 93-104
 - Seal bond to DC 93-104 demonstrated

(C) The SVS design provides high volumetric efficiency, with a high solidsloading and performance HTPB propellant. In the SVS design, the volume required to accommodate cup and fluid is compensated by the addition of a head-end web with the keyhole-slotted grain design, and the increased propellant solids loading. Solids loadings of up to 89 percent appear to be possible, with the only limitation being that of processability. Propellant structural properties are not a limiting consideration in this essentially stress-free system. The keyhole-slotted grain design provides tailoff and neutrality characteristics similar to those of the case-bonded design.

(U) In support of the design effort for the SVS motor, LPC conducted laboratory evaluation of two of the three pretreatment methods mentioned for the case-bonded design, for making the bond of the SVS seal to the silicone insulator. While both the FEP film process and plasma gas treatment process are capable of meeting the structural requirements for this bond, the FEP film is proposed. LPC also conducted tests of compatibility between the SVS support fluid and the DC 93-104 silicone insulator. These tests gave no evidence of adverse effects even under accelerated aging conditions.

(U) A series of successful tests were conducted by LPC to evaluate ejection of the SVS cup (which removes all propellant and elastomeric residual materials). These tests included cold gas ejection tests and three subscale motor firings. Transition times well below the 0.750-second specification were demonstrated. One of these motor tests was part of the Martin-Marietta/LPC transition test series.

(U) Design, analysis, and laboratory test data thus show that the SVS provides a means for meeting all of the stated performance objectives. Successful motor firings with nozzle and cup ejection offers proof of the capability that can be provided. When combined with concepts such as grain burn pattern regulation (GBPR) for achieving a 100-percent volumetric loading with radial-burning ballistics, and higher solids-loading higher performance propellants, the SVS provides true growth potential for the ASALM application.

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- 3.4 REFERENCES
- 3-1 Theilbar, W.H., Cohen, L.S., and Couch, H.T., <u>Summary of Results</u> with a Reinforced Silicone Elastomer, CPIA Publication 228, Vol II October 1972

3-2 Patton, J.B., Robinson, A.T., <u>Ablative Silicone Elastomer Blast</u> Tube Liner, CPIA Publication 228, Vol III, November 1972

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Section 4

LABORATORY AND ANALOG MOTOR TESTS

4.1 REVIEW OF AIR FORCE PROGRAM DATA

(U) Several Air Force programs dealing with related technology or system interfaces have been completed or are in progress. As an initial task, and a continuing effort throughout this phase of the program, these programs were reviewed and pertinent information evaluated therefrom. Key considerations were booster interfaces with other system components and booster motor performance as related to overall system requirements.

(U) Table 4-1 summarizes these programs and the booster components or subsystems affected by them. The specific impact derived from the programs for each booster item affected is shown in Table 4-2. The most significant program in terms of impact was the "Integral Ramjet Rocket Materials Interface Investigation" conducted by the Marquardt Corporation under Air Force Contract AFAPL F33615-72-C-1234. Lockheed Propulsion Company was a subcontractor to Marquardt under this program, and developed the basics of case-bonding HTPB propellants to silicone insulators as part of that work. The film-bonding technique was found to offer highly promising results then, and ultimately led to the optimized system achieved in this program.

(U) Significant impact was also received from the MPM Freejet Program, conducted by Marquardt under Air Force Contract AFAPL F33615-73-C-2002, since this program is directed toward development of the ramburner combustor which constitutes the direct internal interface for the booster. Important coordination was required regarding the insulation off-gas holes, ramjet nozzle to rocket nozzle seals and retention, and dome closure shape, sealing, and installation.

(U) Several details relating to other interfacing programs were under active coordination at the time of work stoppage. These are summarized as follows:

- Insulation Design Work under this contract raised the question of whether additional internal insulation thickness is needed to provide a full ³/₈-inch thickness of virgin material at the start of ramjet burn. Some localized, shallow charring of the insulation will occur during booster burn. However, the insulation is designed to char through completely during ramjet operation. This question was referred to the Air Force by LPC and is being studied.
- Case Material Work undor this contract identified a need for L-605 case material flaw growth data at low temperature and an accompanying fracture mechanics analysis as normally performed for rocket chambers prior to test firing. Existing data are limited and confined to ambient temperatures and above corresponding to

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Table 4-1

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Table 4-2

REVIEW AIR FORCE PROGRAM DATA

BOOSTER ITEM	IMPACT OF OTHER AIR FORCE PROGRAMS
BOND SYSTEM	 COMPARED BOND SYSTEMS IDENTIFIED DESIRABILITY OF INTERMEDIATE BOND AID INDICATED NECESSITY OF OPTIMIZING LINER FOR THIS SYSTEM
INSULATION MATERIALS SYSTEM	 ESTABLISHED NEED FOR OFFGAS HOLES IN INSULATION PROVIDED HEATING PROFILES IMPOSED ON HOLE FILLER MATERIAL PROVIDED TIME SPAN FOR HOLE FILLER TO VACATE IDENTIFIED QUESTION REBUILDUP UNDER AND AFT OF GRAIN SLOT
CASE MATERIAL	• IDENTIFIED NEED FOR LOW TEMPERATURE FLAW GROWTH DATA
DOME CLOSURE	 INDICATED NEED FOR CHANGE TO FORWARD VERSUS REAR INSERTION IDENTIFIED NEED FOR IMPROVED HIGH PRESSURE SEAL DESIGN
EJECTABLE NOZZLE	• PROVIDES GRAIN/INSULATION/NOZZLE INTERFACE DETAILS
PERFORMANCE REQUIREMENTS	 PROVIDES UPDATE OF SYSTEM PERFORMANCE NEED

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- (U) ramjet mode operation. A rocket mode analysis needs to be performed, since flightweight motor firings at temperature extremes and following vibration constitute part of the Phase III motor demonstration. The Air Force Materials Laboratory has undertaken the performance of flaw growth data testing. This work needs to be completed prior to Phase III flightweight testing.
 - Dome Closure A revision was identified to the dome closure retention method to permit forward-end insertion after rocket procenting, and improve sealing against the high pressure and temperature rocket gases. This work has been undertaken and is being studied.
 - Ejectable Nozzle The Air Force contract covering development of technology for this component is still under way. Final completion of testing is necessary to determine the adequacy of both the ejection/retention mechanism design and function, and the nozzle design/materials performance. Once this work is complete, there is additional coordination needed with ramjet contractors to complete the design of rocket-to-ramjet nozzle sealing, and flightweight chamber retention of rocket nozzle against ejection loads.

4.2 LABORATORY PROPELLANT STUDIES

(U) The Integral Ramjet Booster Development Program utilizes LPC-691D, an 87% total solids HTPB propellant. The LPC-691D propellant formulation is, in essence, identical to LPC's basic HTPB propellant for the integral ramjet booster, LPC-691B, with UOP-36/DBTH anti-oxidant added to improve pot life and processability. The basic family of LPC-691 HTPB propellants has been under development at LPC for an extended period of time. It is therefore appropriate to review some of the history of the evolution of this propellant prior to a detailed discussion of the laboratory propellant study portion of the contract effort.

4.2.1 Propellant Requirements

(C) Based upon the goals of the program RFQ and preliminary analysis of motor design parameters, the following requirements have been established:

- Burn rate: 1.34 in./sec at 1000 psi and $+70^{\circ}$ F
- Specific impulse: minimum 245 lbf-sec/lbm delivered at 1000 psi exhausting to 14.7 psi, 15-degree half-angle
- Pressure exponent: minimum consistent with other requirements
- Temperature sensitivity $(\pi_{\rm L})$: 0.20%/°F max

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- Physical properties: consistent with 75- to 76-percent web fraction design, capable of thermal cycling and operation over a temperature range of -65 to +165°F with a minimum structural safety factor of 1.5
 - Pot life: minimum 4 hours after curative addition to ensure confidence in motor processing

(U) Although propellant density is not a specified design requirement, the available envelope together with the major program objective of minimizing total motor weight and case length has led LPC to the conclusion that a theoretical density of 0.065 lb/in.³ minimum should be established as a specific program goal, in addition to the propellant requirements listed above.

(U) In summary, the requirements of high burn rate, high impulse-density, excellent mechanical properties, and minimum mobile constituents impose severe constraints on propellant selection.

4.2.2 Evolution of LPC-69! Propellant

(C) When development of a propellant for the integral ramjet booster application was initiated at LPC in the first half of 1972, the design requirements for the motor established a burn rate of 1.9 in./sec at 1500 psi. In accordance with performance criteria noted earlier, the formulation target was established at 87-percent solids minimum with 18-percent aluminum and an Fe_2O_3 catalyst level of 1 percent. To provide the desired burn rate with good mechanical properties and processability, nominal 0.7-micron UFAP was chosen as the fine-ground component of the oxidizer system. In LPC's experience, the best balance of processability, mechanical, and ballistic properties in UFAP HTPB propellants is achieved by combinations of UFAP and nominal 15-micron AP in a bimodal oxidizer system, the ratio of UFAP to 15-micron AP being varied as necessary to achieve the required burn rate. Isophorone diisocyanate (IPDI) was selected as the curative in order to maximize pot life, and MT-4 was utilized as the bonding agent.

(C) Using 0.7-micron UFAP procured from Naval Weapons Center, LPC performed formulation studies on 1-gallon mixes with the objectives of examining plasticizer tradeoffs evaluating solids loading versus physical properties and processing characteristics, and defining the particle size ratio required to achieve the desired burn rate. As a result of these studies, the formulation designated LPC-691A (Table 4-3) was selected for further scale-up to the 10-gallon (140-pound) mix scale. The propellant properties in Table 4-3 were measured on a 10-gallon characterization batch.

(U) Beginning in January 1973, additional testing and characterization of LPC-691 type propellant was initiated. This effort had several objectives:

 In propellant work during 1972, UFAP procured from the Naval Weapons Center was used. In January 1973, LPC activated a 30-gallon Union Process Attritor facility for the pilot plant

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Table 4-3

FORMULATION AND PROPERTIES OF LPC-691 TYPE PROPELLANTS

	LPC-619A, %(a)	LPC-619B, % ^(b)	LPC-691C, %(C)
Formulation			I THE STATE OF THE PARTY
R-45M HTPB	11.55	10.55	10.55
Isodecvl pelargonate	1.00	2.00	2.00
Ethyl 736/HAA	0.10/0.20	0.10/0.20	0.10/0.20
MT-4	0.15		
HX-752		0.15	0.15
Aluminum	18.00	18.00	18.00
FeO	1.00	1.00	1.00
NH.CIO. (15a)	37.50	40.80	37.50
(0.70 NWC)	30.50		
(1.0µ LPC)		27.20	30.50
Theoretical Properties	in a state of the	and the second	- unit south A - a
Ions, lbf-sec/lbm	262.7	262.5	262.5
Density, 1b/in. ³	0.0648	0.0647	0.0647
c*, in /sec	5.174	5. 168	5 168
T _c , ⁰ F	5,781	5, 772	5, 772
Ballistic Properties, Cured Strands	1.1.1.1.1.1.1.1.1		or the process of
Burn rate 1000 psia/+70° F.	A CONTRACTOR OF THE PARTY OF	A STATISTICS AND A STATISTICS	Deres of Tree of Sub
in./sec	1.48	1.30	1.46
Pressure exponent	0.63	0.59	0.63
σ , (-65 to +160°F), %/°F	0.09	0.10	
π_{k}^{p} , (-65 to +160°F), %/°F	0.19	0.19	asta Denildulinan
Ballistic Properties, 6-inch Motors	and the second second		of the state of the
Burn rate (1000 psia/+70°F),			
in./sec		1.31	1.43
Pressure exponent		0.59	0.61
p_{1}^{σ} (-05 to +105 F), $\frac{76}{10}$ F		0.063	
"k, (-05 to +165°F'), %/°F		0.153	
	σm, εm, E.05	, σ _m , ε _m , E.05,	σ E. 05,
Physical Properties	psi % psi	psi % psi	psi % psi
		- <u> </u>	<u></u>
JANNAF uniaxial at +165°F	122 48 269	154 47 400	76 40 392
+70	187 39 697	209 39 740	198 24 710
0	341 34 2,293	359 37 1,753	
-65	1,137 11 20,500	958 15 10,520	1024 14 12,730
Processing Characteristics	and subsystems	Could be seen to be	a chart would also also
Brookfield viscosity, kos			- Bertsting and Adding
1 hour after curative addition	20	25	27
5 hour after curative addition	31	37	48
	provide the second second		
Pot life, hr	7	7	5

(a) 10-gallon mix 0156-2C

(b) 10-gallon mix 0156-49C (c) 1-gallon mix 0156-49C

(c)_{1-gallon} mix 0156-41C

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- scale manufacture of UFAP, as described in Appendix B.
 Since there were some process changes relative to the material processed by Naval Weapons Center in the Sweco Vibra-Energy Mill, it was necessary to establish that LPC-691 propellant could be successfully prepared with LPC attritor-ground UFAP.
 - (2) Studies at LPC and Thiokol under two Air Force contracts, F04611-72-C-0069 "Development of HTPB Propellants for Air Launched Missiles", and F04611-72-C-0048, "Development of HTPB Propellant for Ballistic Missiles", had indicated that HX-752 was a superior bonding agent as compared to the MT-4 used in LPC-691A. MT-4 is not a commercially produced ingredient, and must either be prepared by the user, or purchased from a specialty chemical company. It is, moreover, a reaction product of variable and indefinite composition, and does not have good storage stability. Some studies indicate that the aging behavior of propellants containing MT-4 is inferior to that of similar propellants containing HX-752. For these reasons, it was desirable to replace MT-4 with HX-752.
- (C) (3) Continuing motor design studies by the Air Force had indicated that a somewhat lower burn rate (approximately 1.3 in./sec at 1000 psi) would be optimum for the ramjet booster design. This required minor burn rate tailoring of the formulation.

(U) In addition to the propellant tailoring, it was desired to obtain reproducibility data in various mixer types, conduct further ballistic and structural characterization, and provide subscale motors to Martin-Marietta for ramjet transition tests.

(C) In early mixes, it was observed that the combination of 0.6-micron UFAP ground in the LPC attritor and incorporation of HX-752 bonding agent led to higher mix viscosities than had prevailed earlier with LPC-691A processed with Naval Weapons Center UFAP. To correct this condition, two additional modifications were made, as discussed below.

(C) First, the content of IDP plasticizer in the propellant was increased from 1.0 to 2.0 percent. In initially formulating LPC-691A propellant, it had been LPC's intent to minimize the content of plasticizer because of uncertainties regarding the effect on DC 93-104 properties of plasticizer that could migrate from the propellant. However, this is no longer considered a constraint because LPC's primary bonding approach is based on the use of an FEP film between the propellant and the silicone insulation. LPC's studies have shown that fluorinated ethylene propylene (FEP) film is a highly effective barrier in preventing migration of IDP plasticizer.

(C) Secondly, it was found that essentially identical burn rates were obtained regardless of whether 1.0- or 0.6-micron UFAP was utilized in the formulation, but lower mix viscosities were obtained with 1.0-micron UFAP. Accordingly, the UFAP particle size has been respecified at 1.1 ± 0.15 microns. This change is also desirable for cost reasons, since it is less costly to grind to 1 micron than to 0.6 micron.

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(U) Two versions of this modified propellant have been processed during the year, as noted in Table 4-3. LPC-691B was tailored to the design requirements of the integral ramjet booster program. LPC-691C, having the same burn rate as its predecessor, LPC-691A, was processed for use in the motors supplied to Martin for ramjet transition tests, since this design optimized at the higher burning rate.

(U) Over 65 mixes of LPC-691 type propellants ranging in size from 1 pint to 10 gallons have been processed at LPC in order to investigate formulation and process variables, conduct propellant characterization tests, and load motors for subscale transition tests at Martin, Orlando. Table 4-4 summarizes results from some of the more significant mixes evaluated. Several points of interest may be noted in these data:

- (C) The effect of UFAP particle size on end-of-mix viscosity may be observed by comparing Mix 0156-14A of LPC-691B propellant (0.61 micron), and Mix 0156-14D of LPC-691C propellant (0.58 micron) with other mixes containing nominal 1-micron UFAP. Both mixes with the finer grind of UFAP showed an end-of-mix viscosity of 47 kps at +110°F, whereas all other mixes are in the acceptable range of 14 to 24 kilopoise at +110°F.
 - However, it is also evident that neither of these mixes containing 0.6-micron UFAP is higher in burn rate than counterpart mixes containing 1.0- to 1.2-micron UFAP.
 - Mixes processed in the Ross 10-gallon mixer show burn rates approximately 0.10 in./sec lower than equivalent mixes prepared in the 1-gallon Baker Perkins mixer. This is not unexpected, since the Ross mixer produces much less shear and will be less efficient in dispersing UFAP.
 - The FEPT hydrogenation catalyst can be added to LPC-691 propellant, if needed, at the 0.05-percent level without affecting propellant properties. However, the effectiveness of baking the DC 93-104 insulation and adding a catalyst to the liner make it unnecessary to use this additive in the propellant.
 - Except for the two mixes containing 0.6-micron UFAP, processing characteristics of all these mixes were good, with pot life exceeding 5 hours after curative addition.

4.2.3 Characterization of LPC-691B Propellant

(U) Characterization of LPC-691B propellant has encompassed the firing of 6- by 11-inch test motors for measurement of motor burn rate and temperature sensitivity, extensive physical property testing and analysis of grain design safety factors, fabrication and thermal cycling of structural analog motors, and propellant/liner/DC 93-104 insulation bond tests.

Optimum NCO/OH ratio of 0.78/1.0 Characterization mix LPC/Martin transition test motors Lot check using LPC UFAP Structur d analog motors New R-45 polymer lot Remarks Characterization min 6 x 11 motor tests 6 x 11 motor tests E. 05, psi 6, 335 2697 1.080 1.079 656 454 581 381 670 1.094 1.049 1.047 12, 300 11. 520 740 400 110 .. 653 JANNAF, Uniaxial Properties PROPERTIES OF LPC-691 PROPELLANT BATCHES *m. 5 5 6 2 195 175 16.8 12 12 54 177 188 198E 125 3254 19 129 sured Not me "m. psi 1, 137 187 122 1. 372 1.259 1, 224 186 141 1. 213 205 162 162 1. 049 243 179 1.040 1, 176 1, 164 162 119 961 1,068 1.024 900 958 209 154 Temp. °F +70++70++ +70 +165 -65 29-+165 +165 145 +165 +165 +165 +165 +165 +165 +70 +70 +70 +70 +70 +70 +70 -65 +70 Pr Asure Exponent 0.60 0.60 0.63 0.59 0.63 0.69 0.58 0.59 0.46 0.58 0.59 0. 55 0.61 . FUM Burn Rate Viscusity, kps 1000 psi/+70°F Not measured 1.60 1.50 1.33 1.31 1.23 1.30 1.35 1.60 1.48 1.41 1.41 1.43 1.28 20 N 20 23 19 53 2 23 25 ç 27 Ļ, 18 8 12 WMD, H 0.72 0.72 0.72 1.02 0.93 0.80 1.12 0.98 0.98 0.58 0.61 0, 98 0. 98 0.93 1.08 UFAP Data LPC-1003/4 Grind No. NWC-153 LPC-1011 LPC-1011 LPC-1012 LPC-1004 LPC-1011 LPC-1012 LPC-1011 NWC-135 LPC-1003 LPC-1005 NWC-135 NWC-135 LPC-1001 Mixer, gallon 2-1/2 2-1/2 -10 ~ 2 2 10 2 2

Table 4-4

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Mix contained 0.05% FEPT catalyst

53

= 5

1.064

-65

0.57

1.45

17

1.2

LPC-1009

LPC-691C 0156-43D

LPC, Martin transition test motors

909

222

1.137

+70

0.55

1.50

2

1.2

LPC-1009

LPC-691C 0156-43C

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LPC-691B 0156-46G

LPC-691B 0156-47A

LPC-691B 0156-47E

LPC-691B 0156-48A

LPC-691B 0156-49A

LPC-691B 0156-49C

LPC-691C 0156-14D LPC-691C 0156-41C

LPC-691B 0156-14A

LPC-691A 0156-10B

Propellant Mix No.

LPC-691A 0156-2A

LPC-691A 0156-2B

LPC-691A 0156-2C

LPC-691A 0156-11B

LPC-691B 0156-12D

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(C) <u>Ballistic Characterization</u>. Seven laboratory batches of LPC-691B propellant were cast into 6-inch-diameter Ballistic Test Motors (BTMs) for ballistic characterization. The motors were statically fired to obtain subscale ballistic performance data, to evaluate the keyhole grain design, to obtain burn rate and temperature sensitivity data, and to evaluate erosive burning characteristics.

(C) Ten BTMs were test fired. A matrix of batch data and test objectives is presented in Table 4-5. Ballistic data are summarized in Table 4-6. Significant test results are discussed below:

- An average standard burn rate of 1.31 in./sec at +70°F was derived for LPC-691B propellant from 6- by 11-inch motors. Burn rate scale factors from cured strand data were not consistent. Data from the first 10-gallon batch indicated a scaleup factor from strand data of 1.05. Data from subsequent batches, however, indicated scale-down factors ranging from 0.91 to 0.99.
- (2) No effects of erosive burning could be identified in a special test configuration where the port-to-throat area ratio equaled 1.0. An initial pressure ratio of 1.20 was determined from measured head-end pressure and predicted "end-burner" pressure. A factor of this magnitude is generally attributable to mass addition and it appears that this propellant has a low susceptibility to erosive burning.
- (3) In the last four motors tested, the keyhole grain design yielded a curve shape in good agreement with the prediction with minor variations due primarily to deviations in as-built and grain design dimensions. In the first motor tested, variations in predicted and measured curve profiles were attributed to propellant defects at the grain periphery.
- (4) The 6- by 11.4-inch motor ballistic data were satisfactory and demonstrated both performance and reproducibility characteristics commensurate with data gathered on numerous propellants tested in 6- by 11.4-inch motors at LPC.

The average c* was 5,039 ft/sec for the five keyhole grain motors with a c* efficiency of 0.975. The average standard specific impulse was 238.8 seconds with an I_{sp} efficiency of 0.91. The nozzle discharge coefficient (C_D) averaged 0.94 for these motors. As is characteristic with all subscale ballistic test motors, because of the grain weight restriction, the measured energy-related parameters are lower than can be expected for comparable full-scale units. LPC experience indicates that a minimum 5-percent gain in efficiency can be expected in the proposed full-scale motor as compared to the 6- by 11.4-inch motor. This increase is more than sufficient to meet the minimum required specific impulse of 245.0 seconds.

ch No. Size, 12D 10 46 G&H 1 47AB 1	. Gal	UFAP Particle	Tb 1000, in./sec 1.28 1.33 1.41 1.41 (-65 ⁰ F) 1.30	ta, +70'F ^{Tb} 2000, in./sec 2.05 2.15 2.15 (-65 ⁹ F) 1.88	0.59 0.59 0.59	Use Runs 2297 - 2301 (Table 2-11) (1) Four 6 by 11.45 w/3-in diameter circular ports (2) One 6 by 33.8 w/3-in diameter circular port and $A_p/A_t = 1.0$, initial Run 2310 One 6 by 11.4 w/subscale keyhole grain design and 1/4-inch-thick (S/N PT001) Run 2403 One 6 by 11.4 w/subscale keyhole grain design and 1/4-inch-thick (S/N 3834-01) Run 2406 One 6 by 11.4 w/subscale keyhole grain design and 1/4-inch-thick DC 93-104 case insulation - plasma arc treated propellant/ liner interface (S/N 3834-01) Run 2406 One 6 by 11.4 w/subscale keyhole grain design and 1/4-inch-thick DC 93-104 case insulation - plasma arc treated propellant/ liner interface (S/N 3834-01) Run 2406 One 6 by 11.4 w/subscale keyhole grain design and 1/4-inch-thick DC 93-104 case insulation - FEP film at propellant/liner interface (S/N 3834-02)	Objective (1) Burning rate slope (2) Temperature sensitivity (3) Burning rate scale factor (4) Erosive burning susceptibility (2) Curve profile evaluation (2) Curve profile evaluation (3) Performance evaluation (4) Propellant/liner bond quality (5) Performance evaluation (1) Propellant/liner bond quality (3) Performance evaluation (1) Propellant/liner bond quality (3) Performance evaluation (4) Burning rate scale factor (1) Propellant/liner bond quality (3) Performance evaluation (4) Burning rate scale factor (1) Propellant/liner bond quality
8A 10		0.98	[-65 ⁰ F) 1.31 (+165 ⁰ F) 1.50	i.82 (-65 ⁰ F) 1.60 (+165 ⁰ F) 2.08	0.46	Run 2504 and 2505 Two 6 by 11.4s w/subscale keyhole grain design and 1/4-inh-thick DC 93-104 case insulation (S/Ns 3834-03 and 3834-05)	 Subscale keyhole grain design performance evaluation Temperature sensitivity Burning rate scale factor

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Table 4-5

6-INCH MOTOR TEST MATRIX

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Table 4-6

6 BY 11.4 BTM TEST RESULTS

					I.	un				
	22.97	2298	2299	2300	2301	2310	2403	2406	2504	2505
Test temperature, ^o F	Amb	+165	Amb	-65	Amb	Amb	Amb	-65	+165	-65
Throat diameter, in,	1.772	1.770	3.00	1.360	1.501	1.531	1.555	1.553	1.199	1.200
Exit diameter, in.						3.2	3.2	3.2	3.6	3.65
Expansion ratio						4.40	4.29	4.30	9.20	9.23
Propellant weight, 1b						14.8	14.3	14.3	13.65	13.63
Web, in.	1.5	1.5	1.5	1.5	1.5	2.01	2.01	2.01	2.01	2.01
Time										
Web burnout, sec	1.389	1.225	1.196	0.843	0.880	2.209	2.245	3.052	1.142	1.612
Total burn, sec	1.58	1.40	1.50	1.20	0.96	2.49	2.53	3.20	1.25	1.76
Burning rate, in./sec	1.080	1.224	1.254	1.779	1.705	0.910	0.895	0.659	1.762	1.247
Pressure										
Average over burn time, psia	659	745	819	1,820	1,486	572	493	378	1,634	1, 148
c*, ft/sec						5,111	4,948	4,973	5,056	5,046
c*, std, ft/sec						5,123	4,962	5,019	5,027	5,062
Thrust										
Average over burn time, lb						1,502	1, 326	977	2,803	1, 962
Delivered specific impulse, sec						226.5	218.0	210.6	238.8	237.1
Delivered, std, specific impulse, sec						245.2	237.1	237.0	234.4	239.7
Coefficients										
Nozzle efficiency						0.953	0.952	0.942	0.927	0.942
Burnrate Scale Factor										
^{rb} BTM ^{rb} Strand	-		- 1.05 -	_		0.95	0.96	0.91	0.93	0.99

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(5) The results of the ballistic test motor program show the temperature sensitivity (π_k) of LPC-691B propellant to be 0.153 %/°F over the temperature range of -65 to +165°F. These data were derived from 6- by 11.4-inch motors cast from a single batch of propellant and tested at constant area ratio (κ_n) conditions. As shown in Appendix A, this total range of π_k values obtained throughout the ballistic test program is 0.119 to 0.194 %/°F. However, some earlier test motors displayed minor performance deviations due to propellant grain defects, which introduced difficulty in assessing motor burn rates. This resulted in less representative temperature sensitivity values.

(U) <u>Structural Characterization</u>. LPC-691B propellant has been structurally characterized. Table 4-7 shows the physical property tests that were performed and the purpose of each test. Figures 4-1 through 4-3 show the results of these tests, and Table 4-8 gives the cure shrinkage and thermal coefficient of expansion values.

(U) Figure 4-3 shows the high stress and strain capability of the LPC-691B propellant system. These data show a strain capability that exceeds that of most propellant systems at all temperatures. The data for pressurized conditions show an expected increase in strain capability of approximately 50-percent over the unpressurized strains. This increase is conservative since a pressure of 500 psi was applied instead of the 1500-psi operating pressure, which would give an additional increase in capability.

(U) Biaxial stress and strain values are excellent. A high strain capability is exhibited at the critical low temperatures. Coefficient of expansion and cure shrinkage are less than those of most propellants, which are normally around 5×10^{-5} in./in./⁰F with a linear shrinkage equivalent to a temperature differential of 16⁰F. These lower values for LPC-691B, as shown in Table 4-8, give lower induced loads, resulting in higher grain structural safety factors.

(U) Stress relaxation modulus shows a normal profile with low to moderate values over the temperature range, which results in low induced stresses in the grain. All of the structural tests show the LPC-691B propellant to be an excellent propellant compared to other propellant systems.

4.2.4 Propellant Selection-Summary and Conclusions

(C) Based on the foregoing discussion, the following conclusions are presented relative to the propellant for the integral rocket/ramjet motor:

• An 87-percent solids/18-percent aluminum R-45M HTPB propellant containing nominal 1-micron UFAP meets the performance requirements of this motor and exhibits satisfactory ballistic properties, structural characteristics, and processability. This propellant, designated LPC-691B, was selected as the basic formulation for the program.

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Table 4-7

STRUCTURAL TEST SUMMARY FOR LPC-691B TYPE PROPELLANTS

Test	Propellant Batch	Purpose of Test
Uniaxial	0156-49C	Batch-to-batch variation baseline
Uniaxial under pressure		Verify pressure loaded allowable
Strip biaxial		Determine thermally loaded tensile allowable
Stress relaxation		Determine time/temperature relaxation modulus capability
Coefficient of expansion		Determine linear expansion of propellant for thermal loading condition
Cure shrinkage		Determine equivalent volumetric contraction for thermal loading

Analog motor

0156-48A

Verify safety factors for in-situ condition



Figure 4-1 Uniaxial JANNAF Test Results



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Figure 4-3 Stress Relaxation Modulus

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Table 4-8

THERMAL EXPANSION AND CURE SHRINKAGE FOR LPC-691B, BATCH 0156-49C

Coefficient of Thermal Expansion, in. /in. / ⁰F

 4.09×10^{-5} 4.12×10^{-5} 4.1×10^{-5}

Cure Shrinkage

0.157% Volumetric

= 0.0523% Linear

 $= +12.8^{\circ}F$



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(C) The only tailoring that was required for this propellant during the program was the normal adjustment of curative equivalence ratios to accommodate the raw material lots procured for the program and the evaluation of the UOP-36/DBTH anti-oxidant material on processability and physical properties.

4.2.5 Laboratory Studies of LPC-691D

(U) As discussed earlier in this section, LPC-691B was the basic formulation selected for the contract effort to be modified to LPC-691D through addition of the UOP-36/DBTH pot life extenders. Table 4-9 shows the details of the formulation change from LPC-691B to the modified formulation, LPC-691D. The reason for making the change was to take advantage of the much longer casting life of LPC-691D which had been demonstrated by LPC on an independent research and development program. Table 4-10 compares the pertinent properties of LPC-691D with similar properties of LPC-691B. From these data it is clear that the strand burning properties at 70°F and 100 psi and the JANNAF uniaxial properties of the two formulations are essentially equivalent, and that the casting life of LPC-691D is more than double the casting life of LPC-691B.

(U) It was also agreed at this technical coordination meeting that two laboratory scale propellant studies would be made to minimize any risks associated with propellant scale-up to the 300-gallon mixer. The program schedules and budgets were revised to incorporate these two laboratory studies.

(C) Objective of the first study was to define the NCO/OH equivalence ratio for best cured LPC-691D mechanical properties with available set of ingredient lots. Four 1-gallon batches of LPC-691D were made varying the curative equivalence ratio (NCO/OH) over the range 0.80/1.0 to 0.90/1.0. Table 4-11 summarizes test results. The first two batches were made varying the curative equivalence ratio from 0.80/1.0 to 0.90/1.0. Propellant properties measured were viscosity, uncured and cured strand burning rates, and JANNAF uniaxial properties. Raw material lots and key processing variables (particularly mix times and mix temperatures) were held constant for this study. Results from the first two batches indicated the desired propellant properties were achievable with curative equivalence ratio in the range of 0.81/1.0 to 0.84/1.0. Consequently, the third and fourth batches were made with 0.84/1.0 and 0.81/1.0 curative equivalence ratios, respectively. Strip biaxial mechanical properties were measured on these two batches, in addition to all the properties measured on the first two batches. General conclusions from this study are as follows:

- JANNAF modulus at 70°F and 5-percent strain increases approximately linearly with increasing NCO/OH equivalence ratio. Minimum NCO/OH equivalence ratio with reasonable safety margin required to meet minimum modulus requirement of 500 psi is 0.84/1.0 (Figure 4-4).
- JANNAF maximum true stress at 70°F decreases approximately linearly with decreasing NCO/OH from 0.90/1.0 to 0.84/1.0, but then decreases sharply nonlinearly from NCO/OH 0.84/1.0 to 0.80/1.0 (Figure 4-5).



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LPC-691D (wt%) 40.80 27.20 18.00 0.04 10.77 0.04 1.00 0.15 2.00 100.00 LPC-691B (wt%) 18.00 10.55 40.80 27.20 2.00 0.10 1.00 0.15 0.20 100.00 HTPB Binder (R-45 + IPDI) 7 - 9μ (Fisher Subsieve) Ammonium Perchlorate Formulation

1μ (MSA)

Iron Oxide

HX-752

IDP

HAA

Ethy1 736

UOP-36

DTBH

Aluminum (S-792)

Table 4-9

COMPARISON OF LPC-691B AND LPC-691D FORMULATIONS

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Formulation	LPC-691B	LPC-691D
NCO/OH Ratio Typical Properties	0.8	0.84
Apparent Viscosities at 110 ⁰ F, kps Before curative addition 1 hour after curative addition 16 hours after curative addition	 25 40 kps at 7 hours	14 14 40
Burning Rate at 70 ⁰ F and 1000 psi, in./sec	1.31	1.34
IANNAF Uniaxial Properties $a = -65^{0}F$, psi $e = at -65^{0}F$, % $a = +70^{0}F$, psi $e = at +70^{0}F$, psi $E = 05 = at +70^{0}F$, psi	958 15 209 39 740	968 16 185 37 620
Strip Biaxial Properties on at -65°F, psi on at -65°F, % on at +70°F, psi on at +70°F, psi on at +70°F, %		780 12 163 27

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Table 4-10

COMPARISON OF LPC-691B AND LPC-691D PROPELLANT PROPERTIES

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Table 4-11

MPO 763 - SUMMARY OF PROPELLANT DATA LPC - 691D Variation of Curative Equivalents Ratio

Ingredient	Lot No.	-1	-2	-3	
R 45	3108811A	10.07	9 98	10.03	10.06
PDI	3109011A	0.70	0.79	0.74	0 71
IDP	30022114	2.00			
DTBH	31089114	0.04			
(IP th	10619114	0.04			
	31003114	0.15			
77. 72	5109511A	0.15			
ron Oxide	3063811A	1.00			
Aluminum, 5-792	3108711A	18.00			
UFAP LOH	AG - 1022	27,20			
AP 5µ	Ray 055	40.80			
NCO OH Ratio		0.80/1 0	0.90/1.0	0.84/1.0	0.81 1 0
Brookfield apparen	t viscosities				
TF bar IRPM. II	(0°F), kps				
Before curative a	ddition	10	12	15.7	9.5
I hour after curat	tive addition	15	13	13	12
Haake Rotovisco pr	operties		· ·		
ist sity at 110 F	and a shear				
rate of 0 19 sec"),	kps				
hour after curat	tive addition	14.5	15		
5 hours after cu	rative addition	38	39		
Incured strand bur	ning rate				
betwee curative add	lition at 70°E				
and 1000 per up /e		1.1.8	1.22	1.15	1 19
and roov par. m.rs	et	1 10	1.66	1.19	1 17
NCO OH Ratio		0.80/1.0	0,90/1.0	0.84 1 0	0.81/1.0
- acuum mix tempe	rature. "F	145 155	145 150	150 155	145 155
Vacuum mix 'ime a	fter AP added	45	45	45	45
ured strand burni	ng rates in sec				
at 1500 psi and 6	552	1.51	1.54	1,45	1.50
1000 psi +7	0°F	1.34	1.41	1.32	1 34
-500 psi +7	'0'F	1 72	1.76	1 66	1.68
- J00 psi +7	0°F	2 00	2.02	1.91	1.92
1500 psi +1	65°F	1 86	1.97	1 84	188
Presente exponent	1000 to 2000				
riessure exponent	1000102000	0.69	0.52	0.64	0.52
pairange		0.30	0.52	0.90	0,55
ANNAF Uniaxial F	Properties				
J. at 65°F		813	1168	968	846
n 65°F		14	13	16	15
0°F		203	419		
т. 0°F		46	27		
m .70°F		108	252	185	126
m .70°F		41	20	27	34
F. 05 - 1 70 ⁰ T		100	1005	31	24
2. US at + /UF		500	1095	620	430
at - Los'F		78	104	133	05
'm +165°F		1	32	43	41
shore A hardness	at +70°F .3 sec:	48	75	60	59
NLAD 'DH Ratio		0.80/1.0	0.90/1.0	0.84/1 u	0.81 - 1 0
Strip biaxial proper	ties				
at 65°F				780	658
6505				12	12
17 . ZOVE				163	110
TT				27	24
m			••	122	22
0m +165°F				132	87
6 165'F			••	35	38







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5 JANNAF Maximum True Stress at 70°F versus NCO/OH LPC-691D Propellant

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- (C) Both strip biaxial and JANNAF uniaxial 70°F strain values increase with decreasing NCO/OH.
 - Desired mechanical properties are maximum 70°F strain value acceptable modulus and stress. Therefore optimum NCO/OH equivalence ratio is 0.84/1.0.
 - No significant changes in strand burning rate, pressure exponent, or temperature sensitivity were observed when NCO/OH ratio was varied over the range 0.80/1.0 to 0.84/1.0. Slightly faster burning rates were observed at NCO/OH ratio of 0.90/1.0.
 - Brookfield viscosities on all batches one hour after curative addition at 110°F were within range of 12 to 15 kilopoise. Castability on all batches was excellent and viscosities 16 hours after curative addition were typically less than 40 kilopoise.

(C) The curative equivalence ratio of 0.84/1.0 was selected for the first scale-up batch to the 300-gallon mixer. Work on the program was terminated before the scale-up batch was made.

(U) Purpose of the second laboratory scale study was to provide additional information on effects of processing conditions on LPC-691D propellant properties. Four 1-gallon batches were made varying two process variables at two levels. The selected process variables were vacuum mix temperature and vacuum mix time (after oxidizer addition and before curative addition). Propellant properties measured were viscosity, uncured and cured strand burning rates, and JANNAF uniaxial mechanical properties. Raw material lots, curative equivalence ratio (NCO/OH), and other processing variables were held constant for the study. Results of tests performed on these four 1-gallon batches are shown in Table 4-12. General conclusions from these results of this study are as follows:

- Longer vacuum mix times appeared to reduce propellant viscosity for the lower temperature mixes (135 to 145°F).
- Longer mix times did not increase propellant strand burning rates
- The elevated mix temperatures appeared to show a trend to faster strand burning rates.
- Propellant modulus and stress at 70°F tended to be reduced for elevated mix temperatures and short vacuum mix cycles.
- Propellant modulus and stress tended to increase for elevated mix temperatures and long vacuum mix cycles.



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Table 4-12

MPO 763 - SUMMARY OF PROPELLANT DATA LPC-691D Variation of Mix Time and Temperature

Ingredient	Lot No.	-5	-6	-7	-8
R-45	3108811A	10.07			
IPDI	3109011A	0,70			
IDP	3002211A	2.00			
DTBH	3108911A	0.04			
UOP-36	3061811A	0.04			
HX-752	3109311A	0.15			
Iron Oxide	3063811A	1.00			
Aluminum	3108711A	18.00			
UFAP. 1.04	AG-1022	27.20			
AP, 8µ	Ray 055	40.80			
NCO/OH Ratio		0.80 - 1.0			
Vacuum mix t	emperature. ⁰ F	160 - 170	155 - 165	135 - 145	135 - 145
Vacuum mix ti	ime after AP added	90	180	90	180 -
Brookfield app (TF bar, 1 RF	PM. 100°F), kps				
Before curat	ive addition	12	14	16	9.5
l hour after	curative addition	12	13	16	11,5
Uncured stran curative additi	d burning rate before ion at 70°F and 1000				
psi, in./sec		1.19	1.19	1.20	1.18
NCO/OH Ratio		0.80 - 1.0			
Vacuum mix to	emperature, ⁰ F	160 - 170	155 - 165	135 - 145	135 - 145
Vacuum mix ti	ime after AP added	90	180	90	180
Cured strand	burning rates, in./sec				
at 1500 psi a	nd -65°F	1.61	1.51	1.58	1.43
1000 psi	+70°F	1.40	1.32	1.35	1.29
1500 psi	+70°F	1.76	1.66	1.73	1.63
2000 psi	+70°F	2.21	1.90	1.93	1.87
1500 psi	+165°F	1.97	1.85	1.94	1.86
Pressure expo	onent (1000 to 2000	1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 - 1999 -			
psi range)		0.56	0.55	0.55	0.55
JANNAF Unia	kial Properties				
o at -65°F		741	820	857	738
€65°F		17	16	15	18
$\sigma_{\rm m}^{\rm m} = 0^0 {\rm F}$		160	165	192	153
$\epsilon_{\rm m}^{\rm m} = 0^0 {\rm F}$		56	48	26	67
$\sigma_{}^{m}$ +70°F		73	76	110	65
$\epsilon_{\rm m}^{\rm m}$ +70°F		93	58	35	86
E.05 at +70°E	r.	210	300	440	161
g at +165°F	Contraction and Section	40	41	29	31
€ +165°F		144	85	28	94
Shore "A" har	dness at +70°F (3 sec)	38	45	58	37

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(C) Since propellant viscosities were all excellent and no increase in burning ratio was achieved by the longer mix cycle, the preferred mix conditions are a short vacuum mix cycle at elevated mix temperature from economic considerations. Quantitative conditions for optimum mixing conditions in the 300-gallon mixer would have to be defined at that mixer scale. Close control of process variables appear necessary to achieve reproducible propellant properties.

4.3 HOLE FILLER LABORATORY STUDIES

(U) A unique requirement of the Integral Ramjet Booster Propulsion System is that the prescribed DC 93-104 ramjet internal insulation contain a fixed number of radial outgassing holes. The function of the holes is to permit outgassing of the external insulation, which occurs during ramjet operation as a result of the combined external/internal thermal environment. It has been shown through extensive testing conducted by the ramjet subcontractor that without the holes, retention of the internal insulation throughout the ramjet burn can not be reliably achieved. The quantity, size, spacing and method of hole fabricating has been determined by the ramjet subcontractor tests, and as such has become an interface requirement which must be accommodated by the solid booster grain.

(U) Lockheed Propulsion Company's primary approach for achieving a propellant grain bond to the DC 93-104 combustor insulator is through the in situ application of an etched FEP film barrier to the internal surface of the combustor insulator. This process, which involves application of the film to the male insulation casting mandrel, has been successfully implemented for numerous motors and subjected to successful tests from -65 to +165°F.

(U) Since the time that program evolution pointed to the need for the outgassing holes, LPC investigated methods of accommodating this configuration. The criteria used for evaluation were:

- Holes will be formed by conventional drilling operation after in situ application of FEP film.
- Holes should be filled to preclude the potential of "blow through" and associated pressure spikes attendant with propellant web fracture.
- Filler material will be structurally capable of supporting environmental loads and act as an insulator during booster grain burn, but not impede purpose of holes, i.e., permit DC 93-104 outgassing.
- Filler material will be amenable to processing without compromising the bondability of the FEP film system.

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(U) To implement this evaluation, a combustor hole filler laboratory study effort was initiated consisting of process, thermal, and short-term aging bond studies of two candidate hole filler materials. The initial effort consisted of fabrication and thermal testing of eight 4- by 4-inch by $\frac{3}{8}$ -inchthick DC 93-104 test panels. The panels contained the convoluted stainless steel band for mechanical retention of the DC 93-104 to the combustor wall and the hole configuration as currently specified in the Marquardt Corporation combustor. The tests were conducted in the LPC combustion laboratory. The basic test arrangement provides for both backside and internal ramjet heat inputs. Backside heating is provided for by a calrod resistance heater and internal heat flux is applied by an oxyacetelene torch. Each test panel is subjected to preheat of the backside to approximately 750^{0} F prior to torch ignition. The torch test duration is 30 seconds at central heat flux levels currently being used by the Marquardt Corporation in similar panel tests. The torch test arrangement is shown in Figure 4-6.

(U) Two candidate hole filler materials were selected for process evaluation: (1) a (75/25%) mixture of celogen and plaster, and (2) a 100% pellet of ammonium sulfamate (NH₄NH₂SO₃). The pretest appearance of the thermal panels with the celogen/plaster filler materials installed is shown in Figure 4-7. The basis of selection of these two materials has its origin in earlier work conducted at LPC to screen candidate filler materials. The celogen/ plaster compound offers the advantage of easy processability in that a wet mixture of these ingredients is easily cast into a Teflon mold, as shown in Figure 4-8, thus permitting processing of a quantity of pellets in a single operation. Following cure of the mixture, the pellets are immersed in a solution of sodium silicate and dried. This treatment hardens the outer surface of the pellet and minimizes "dusting". The pellets are sized to permit an approximate 0.003 inch interference fit in the $\frac{1}{32}$ -inch-diameter DC 93-104 combustor insulation holes. An additional advantage of this compound is that its constituents are basically inert. The celogen is a gassing or "blowing" agent which decomposes at approximately 300°F forming nitrogen gas. Observations of thermal tests conducted to date show that these pellets are essentially self-expelling due to the decomposition of the celogen and the build-up of gas pressure within the DC 93-104. Further, the products of decomposition will minimize potential energy contributions to the system which could adversely affect transition performance. The post-test condition of a typical celogen/plaster thermal test panel is shown in Figure 4-9. As can be seen in Figure 4-9, the holes in the rectangular test area are either clean or contain a soft white deposit which would not interfere with the original intended function of the holes.

(U) The second hole filler material evaluated, 100% ammonium sulfamate, is a subliming salt which transforms into a gas composition at approximately $325^{\circ}F$. This material is not castable and must be formed in a pelletizing machine. This process does provide a harder, more dense product should structural considerations indicate this desirable. The pretest condition of a typical ammonium sulfamate-filled panel is shown in Figure 4-10. The posttest appearance is shown in Figure 4-11. As can be seen, a majority of the holes in the test area are clear or contain a small amount of re-solidified residue which appears on cooldown. The re-solidification would not occur during actual ramjet operation, therefore all holes exposed to internal heat flux would remain clear.

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Figure 4-6 Torch Test Arrangement

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Figure 4-7 75% Celogen/25% Plaster Thermal Test Panel - Pretest





Figure 4-9 75% Celogen/25% Plaster Thermal Test Panel - Post-test

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Figure 4-10 100% Ammonium Sulfamate Thermal Test Panel - Pretest

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Figure 4-11 100% Ammonium Sulfamate Thermal Test Panel - Post-test

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(U) At the completion of this test series, which contained four thermal tests for each of the two filler caudidates, fabrication of the bond-in-tension and peel samples for the second phase of the study was initiated. To fully evaluate the effects of the filler pellets on the system bond and peel strengths, the laboratory test matrix, shown in Table 4-13, was established. The matrix consists of a total of 54 specimens. The specimen configurations are shown in Figures 4-12 and 4-13. Standard specimen dimensions were retained to allow comparison with past data. The specimens were cast from two 1-gallon propellant mixes. The data are summarized in Table 4-14.

(U) Study of the test data and types of failures experienced leads to the general conclusion that the propellant curative equivalence ratio of 0.80/1.0 selected for the test series is somewhat lower than optimum. This is supported by the fact that the majority of the failures at +165 and +70°F occurred in the propellant at lower values than experienced with numerous past propellant batches. Program schedule requirements dictated the necessity to choose an equivalence ratio based on data available at the time prior to completion of the laboratory propellant program. As discussed in the laboratory propellant studies portion of this report, later data revealed that the propellant physical properties are much closer to optimum at an equivalence ratio of 0.84/1.0 with the program propellant materials. However, the data do provide certain information which is key to the program.

- Although bond-in-tension failures occurred at lower than desired values at +165 and +70°F in the propellant, this indicates that there was no gross effect of either of the pellet materials on the bond interface.
- The -65°F specimens failed at values close to those experienced in past tests and in the same manner which further supports the conclusion that the filler materials had no adverse effect on bond.
- The peel data generally experienced the same type failures as were developed in the bond samples. The same general conclusions can therefore be applied to these results.

(U) Based on results to date, current program emphasis is being placed on use of the celogen/plaster molded pellet. This is primarily related to the comparative ease with which the cast celogen pellets can be processed as compared to the individual pelletizing operation required for the ammonium sulfamate.

4.4 BOND AND ACCELERATED AGE TESTS

(U) Lockheed Propulsion Company's participation in the AFAPL Contract F33615-72-C-1234 with the Marquardt Corporation, on the Integral Rocket/ Ramjet Interface Materials Investigation, led to the establishment of the feasibility of a materials interface system for HTPB propellants. Extensive

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Table 4-13

COMBUSTOR HOLE FILLER MATERIAL TEST MATRIX (Candidates A and B)

	Bond Accel	-In-Te erated	nsion Age T	and 'ests	Accele	Peel	and Age T	ests
Thermal Evaluation	Time	Tem	Test peratu	re, ⁰ F	Time	Tem	Test peratu	re, ⁰ F
4 x 4 Panels	(weeks)	-65	+70	+165	(weeks)	-65	<u>+70</u>	+165
	0	3	3	3	0	3	3	3
6	2	3	3	3	2	3	3	3
	4	3	3	3	4	3	3	3

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Bond-in-Tension Specimen Configuration Figure 4-12

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Figure 4-13 Peel Specimen Configuration



BOND-IN-TENSION AND PEEL TEST RESULTS

Туре	Temperature (°F)	Stress (pei)	Strength (lb/in.)	Failure Mode
Zero Age Time				
Control (BIT) (No Holes)	+165 +70	40 87 390		CP CP CI
P/C (BIT) (1 Hole)	+ 165 +70 -65	40 55 193		CP CP CI
A/S (BIT) (1 Hole)	+165 +70 -65	36 77 323		CP CP CI
P/C (BIT) (4 Holes)	+165 +70 -65	22 37 294		CP CP ALF
A/S (BIT) (4 Holes)	+ 165 +70 -65	14 57 243		CP CP CI
Control (Peel) (No Holes)	+165 +70 -65		9.0 17.0 53.0	CP CP ALF
P/C (Peel) (3 Holes)	+165 +70 -65		8.0 15.0 55.0	CP CP CI
A/S (Peel) (3 Holes)	+165 +70 -65		11.0 17.0 69.0	CP CP CI
2. Weeks Age at 4	165°F			1.1
Control (BIT) (No Holes)	+165 +70 -65	40 105 225		CP CP CI
P/C (BIT) (1 Hole)	+ 165 + 70 - 65	32 92 212		CP CP CPI
A/S (BIT) (1 Hole)	+ 165 +70 -65	44 85 284		CP CP CPI
P/C (BIT) (4 Holes)	+ 165 + 70 - 65	19 37 293		CP CP CI
A/S (BIT) (4 Holes)	+165 +70 -65	20.6 42 215		CP CP CI
Control (Peel) (No Holes)	+165 +70 -65		7.0 16.0 70.0	CP CP CI
P/C (Peel) (3 Holes)	+165 +70 -65		8.5 21.0 50.0	CP CP AIF
A/S (Peel) (3 Holes)	+165 +70 -65		9.0 21.0 63.0	CP CP AIF
4 Weeks Age at +	165 T			
Control (BIT) (No Holes)	+165 +70 -65	47 120 240		CP CP CI
P/C (BIT) (1 Hole)	+ 165 + 70 - 65	49 97 265		CP CP CP
A/S (BIT) (I Hole)	+165 +70 -65	50 106 322		CP CP ALP
P/C (BIT) (4 Holes)	+165 +70 -65	25 48 205		CP CP CI
A/S (BIT) (4 Holes)	+165 +70 -65	16 50 214		CP CP CI
Control (Peel) (No Holes)	+165 +70 -65		12.0 22.0 60.0	CP CP AIF
P/C (Peel) (3 Holes)	+165 +70 -65		11.0 19.0 55.0	CP CP CI
A/S (Peel) (3 Holes)	+165 +70 -65		13.0 21.0 62.0	CP CP AIF

KEY	

P/C	-	Plaster/Celogen Pellet
A/S	-	Ammonium Sulfamate Pellet
CP	-	Cohesive in Propellant
CI	-	Cohesive DC 93-104 Insulation
ALF	-	Adhesive LPL-63 Liner/FEP Film
AIF	-	Adhesive DC 93-104/FEP Film
CPI	-	Cohesive Propellant/Interface
ALP	-	Adhesive Liner/Propellant

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and the standard standard standard standards.

(U) work since then under LPC funds has led to the optimization of this system, confirmation of its superior performance compared with other systems, and finally to the verification of its performance in a wide variety of motor tests.

(U) <u>Background</u>. The operational requirements of the integral rocket/ ramjet are such that the thermal insulation for the combustion chamber must possess a unique combination of mechanical, physical, and thermal properties. To meet the combination of long-burn-time, long-range missions, and short-burn-time, high-velocity missions, in a single vehicle, the material must be low in density, thermally stable for the duration of burn time, and resistant to high shear forces. It must also be processable into high-quality components, and low in cost. The unusually long burn time for the ramjet imposes the most severe of the above requirements.

(U) Review of the combustion chamber insulator work conducted by Marquardt under Air Force sponsorship¹ indicates that four different insulator systems were selected for evaluation in 15-inch-diameter combustion chamber tests. The systems were (1) plasma-sprayed ceramic, (2) trowellable ceramic, (3) reinforced plastic, and (4) elastomeric ablative.

(U) The plasma-sprayed ceramic materials were reported to be inadequate in a number of areas. Thermal protection was limited by the thickness of the coating that could be applied. Spalling occurred in areas of high thermal stress. Adequate thermal protection could be obtained using the honeycombreinforced trowellable material; however, the density was high, resulting in weight penalties. Also, the honeycomb attachment method exhibited problems under the vibration and acoustic conditions within the test chamber.

(U) The reinforced plastic insulator proved to be difficult and expensive to fabricate and install. Delamination of the reinforcement plies led to rapid insulator deterioration.

(U) The elastomeric ablative material (DC 93-104) demonstrated adequate thermal protection under the most severe thermal environments. It is quite durable in that the carbon-fiber-reinforced silicone rubber forms a hard, tenacious char during early stages of ombustion chamber operation.

(U) The char forms the primary insulator during extended flight times. The density of the insulation was lower than that of the other systems tested: 0.053 lb/in.^3 , compared to 0.063 lb/in.^3 for the reinforced plastics and approximately 0.1 lb/in.^3 for the ceramics.

(U) Low cost and simple processing are significant attributes of the silicone ablative. It is a castable material that can be molded into the combustion chamber with inexpensive tooling. Curing takes place at room temperature, or can be accelerated by heating the assembly. Marquardt, in its report, indicates that the material is readily adaptable to mass production techniques.

¹ Contracts F33615-70-C-1778 and F33615-71-C-1100, Report No. AFRPL-TR-72-2.

(U) Clearly, then, DC 93-104 is a good selection for the baseline thermal protector in the combustion chamber. However, the operational characteristics of the integral rocket/ramjet, and the silicone ablative insulation specified for it, present a unique challenge in solid rocket motor adhesive bonding technology.

(U) <u>Objectives</u>. The major objectives in the design and fabrication of the case-bonded grain with respect to the interface system are listed below:

- Structural adequacy
- Reproducible bonding to silicone
- Minimum residual combustibles at burnout
- Long-term aging capability

(U) The objectives are not listed in order of their importance, but they serve to show the complex problems involved in conducting this program. To successfully meet them requires that they be integrated into a plan that demonstrates theoretically and practically that a solution has been found.

(U) <u>Structural Adequacy</u>. The structural adequacy of the case-bonded grain includes, of course, an adequate bond between the selected propellant and the silicone insulation. It is expected that structural analysis will indicate the desirability of obtaining tensile, shear, and peel strengths greater than the cohesive strength of the selected propellant.

(U) <u>Reproducible Bonding to Silicone</u>. The achievement of a reproducible bond to the DC 93-104 silicone ablative material is one of the key objectives in meeting the requirements of the ramjet booster. Achieving this goal requires the application of sound adhesive bonding theory along with some unique approaches to treating the silicone to render it bondable.

(U) <u>Minimum Residuals at Burnout</u>. The successful transition from booster burnout to ramjet "take-over" requires that little or no combustible material remain in the combustion chamber at the time of startup. Therefore, selection of interface materials for adhesive bonding, which could be present at burnout, must depend heavily on their thermodynamic characteristics. These materials must burn out early during transition and produce a minimum of gas.

(U) <u>Long-Term Aging Capability</u>. In keeping with standard practice, the interface materials selected for the bond of the grain to the silicone interface must be capable of performing under all conditions and periods of long-term aging. Of particular concern must be the ability of the bond to withstand exposure to any plasticizer or process aid ingredients that could be present in the propellant and the insulation materials.

(U) Data obtained under AFAPL Contract F33615-72-C-1234 with Marquardt, on Integral Rocket/Ramjet Interface Materials Investigation, have shown that it is feasible to meet these objectives. LPC participated in this

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(U) program as a subcontractor to Marquardt. The task remaining was to optimize the interface system and to complete verification.

(U) <u>Approaches to the Solution</u>. Cured silicone rubber (e.g., DC 93-104) is normally considered unbondable with the use of adhesives and processing techniques suitable for solid propellant rocket motors. The silicones (sometimes called RTVs) are not bondable because of their low surface tension or surface energy. Also, because of their inert nature, silicones do not respond to conventional surface treatment methods for raising surface energy to allow adhesion to take place.

(U) Lockheed Propulsion Company has studied the theoretical barriers to achieving a bond between propellant and cured silicone. There are three general techniques that could be used to treat the problem, as discussed below.

(U) One method would be to establish a chemical bridge between the insulation and the propellant. This could be accomplished by coating the insulation with an adhesive that is known to adhere well to silicone and to propellant. Work in this area has been performed by LPC and the Naval Weapons Center at China Lake, California.

(U) The second method is to use a high-energy medium to increase the surface energy of the silicone insulation. The CASING (Crosslinking by Activated Species of Inert Gas) process has been shown to improve the bondability of various materials, including silicones. This is a process whereby an inert gas is excited to the glow discharge state in contact with the insulation. The resulting bombardment crosslinks the chains in the silicone, making it bondable.

(U) The third method, which is LPC's primary approach, is to bond a film (or a metal foil) to the inside face of the silicone insulation. This is best accomplished in situ during the cure of the silicone. Adhesion between the film and the insulation is accomplished by a silicone primer. The film used would be bondable on both sides, thereby allowing adhesion to the propellant using conventional methods.

(U) <u>Technical Discussion</u>. The integrated solution of the stated interface objectives then, is a process that provides a high-quality bond between the HTPB propellant and the silicone ablative insulation, as well as between the release flap and the insulation, and that provides a minimum of combustible residual material.

(U) Detailed Technical Requirements. Clear definition of the technical requirements to be achieved in this program is the key to their solution. The interface system that is finally selected to go into Phase III must have been demonstrated through extensive testing to be capable of achieving the stated requirements in a practical design. These requirements for the interface adhesive system between the propellant and the liner are listed below. The

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(U) peel strength value is based on standard, center-perforated grain design practice at LPC.

Bond Strength

90-degree peel	1.5 pli	
Friaxial bond-in- tension (poker chip)	Cohesive in the propellant	-65 to +165°F
Double lap shear	Cohesive in the propellant	
Aging Capability	No change in peel strength or tensile/shear failure mode after 4 weeks of aging at 165°F	-65 to +165°F
Residual Combustibles	Minimum remaining at	

Processability

start of ramjet mode

Controllable to degree required to obtain no residual combustibles

(U) A similar set of requirements exists for the bond between the grain release flap at the forward and aft ends of the grain. This bond must be made between the insulation and the release flap material. The detailed requirements are basically as previously stated, except for elimination of the "cohesive in the propellant" failure modes.

(U) Silicone Bonding Methods. Lockheed Propulsion Company has conducted laboratory studies on each of the three approaches outlined earlier for bonding to silicone insulation. These studies have developed data and preliminary processes for meeting the objectives of this program. approaches and selection of the primary "bondable film" approach for the program. The following is a brief description of the approaches considered, with a detailed discussion of the bondable film method.

Method 1: Chemical Modification of Surface

(U) Chemical modification of the DC 93-104 surface to promote adhesion to hydrocarbons was one of the first techniques explored by LPC and other organizations (ref NWC Report TP 4100-2i¹). This is a direct approach that has actually been tested in solid propellant rocket motors.

(U) Silicone resin can be used to provide a tie coat between the insulation and the hydrocarbon surfaces of liner or propellant. Silicone resins bond well to silicone rubber surfaces and present a more compatible surface for adhesion to hydrocarbons.

(U) The chemical method of treatment satisfies all of the objectives except the very important one of structural capability at -65°F. This deficiency can possibly be alleviated by using materials with better lowtemperature properties. The aging capability has been demonstrated by

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(U) other workers in the field. The silicone resins used as a chemical bond do not present a reaiduals problem.

Method 2: Inert Gas Plasma

(U) Numerous reports^{1, 2, 3} have discussed the development of a process that improves the bondability of various materials. This process is designated CASING (Crosslinking by Activated Species of Inert Gas). Recently, there has been a significant amount of work^{4, 5} to characterize this process for improving the bondability of RTV silicones. Also, it was reported⁶ in March 1972 that a commercial process, herein called the "plasma process", had been developed and is currently in broad use by many industries such as those manufacturing integrated circuits and electrical connectors.

(U) The plasma process is a method whereby an inert gas is excited to the glow discharge state in contact with a substrate material, e g, the silicone insulation. The resulting bombardment cross links the latent hydrocarbon chains in the silicone. The surface energy is raised significantly and wetting and adhesion can take place. The process, though new to the solid rocket motor industry, was discovered in about 1956 and has undergone steady development since.

(U) Continuing review of the literature has indicated that this technique has potential for achieving bondability to the silicone insulation in the integral rocket/ramjet. To investigate this potential, LPC undertook an in-house laboratory investigation. A detailed description of the process and the preliminary laboratory investigations conducted to determine its suitability, including a series of Scanning Electron Microscope (SEM) photographs taken of DC 93-104 with and without treatment, are presented in Appendix D. The results of these investigations indicate that peel strength values superior to those obtained by chemically modifying the silicone surface (Method 1) are achievable, with cohesive type failures in the DC 93-104 substrate.

(U) The structural capability of the plasma method is also shown to be adequate when liner is made part of the propellant/insulation bonding system. The aging capability is expected to be adequate. This method therefore meets all of the objectives except the one of no residuals. If a thin film of hydrocarbon liner can be tolerated, this system will meet all the objectives,

- ⁴ DeLollis, N.J. and Montoya, O., <u>Journal of Adhesion</u>, pp 57-67, Sept 1971
- ⁵ DeLollis, N.J., <u>The Use of RF Activated Gas Treatment to Improve</u> Bondability, Sandia Laboratories Report SC-RR-71-0920, 1972
- ⁶ Bersin, R.L., <u>Adhesives Age</u>, pp 37-40, March 1972

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¹ Schonhorn, F.W. and Hansen, R.H., <u>Journal of Adhesion</u>, pp 93-99, April 1970

² Hall, R. J., et al, <u>Surface Treatment of Polymers with Activated Gas</u> <u>Plasma for Adhesive Bonding</u>, Picatinny Arsenal Technical Report 3788, 1969

³ Cagle, Charles V., Adhesive Bonding, McGraw-Hill, New York, 1968

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(U) with the possible exception of existing plasma treatment equipment size limitations and potential scale-up difficulties.

Method 3: Bondable Film

(U) In situ attachment of a bondable film to the DC 93-104 is LPC's primary bonding concept. This concept simply involves the bonding of FEP (fluorinated ethylene-propylene) film, etched on both sides, to the insulation during the silicone curing process. Application of silicone primer to one side of the film allows the bonding to take place.

(U) Lockheed Propulsion Company introduced this process on the SRAM program by bonding etched-one-side FEP film to the cup wall during vulcanization of the rubber. This forms a positive release from the case insulation, thereby guaranteeing a stress-free condition in the release areas of the motor. Use of etched-both-sides FEP is an extension of this approach. Both of these types of film are off-the-shelf, commercially available materials. Alternately, a thin (1 to 2 mil) metal foil could be used instead of the FEP film. Some of the advantages and disadvantages of this approach are listed below.

Advantages

- (U) This concept has many advantages, as discussed below.
 - (1) Simple Processing
- (U) The film allows bonding to the cured silicone without additional treatment. Other methods discussed (chemical and plasma) require further treatment of the insulation after receipt by the contractor. The first step at the start of processing would be a simple wipedown of the FEP with a suitable solvent. Communications with Marquardt personnel on Contract F33615-72-C-1234, The IRR Interface Materials Investigation Program, indicate that addition of FEP film to their processing plan would be a simple matter and is entirely feasible. LPC experience in fabricating a number of rocket motors on Company funds since completion of that contract has verified the simplicity of this process.
 - (2) Mobile Ingredients Barrier
- (U) The film acts as a good barrier to mobile ingredients that might be contained in the insulation and/or the selected propellant. Migration of such ingredients is known to affect the burning rate of propellant, thereby causing a possible change in the sliver at booster burnout.
- Lockheed Propulsion Company conducted a test to determine the permeability of a common propellant processing aid, isodecyl pelargonate (IDP), through the FEP film. A 1-mil-thick film was found to resist passage of IDP through it, or absorption in it, after exposure to a 100-percent concentration for 144 hours at +165°F.



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An IR spectra was taken to detect the presence of carbon/oxygen species on or in the FEP film, and no evidence of their presence was found.

(U) A test to assess the quantity of mobile ingredients in the DC 93-104 was conducted by exposing the cured insulation to normal processing temperatures. Figure 4-14 indicates a potentially significant loss of material that could affect the propellant burning rate when the material migrates to the interface. The use of FEP should preclude this migration. The species lost has not been identified, but checks have indicated it is not water vapor.

(3) Damage Control

- (U) DC 93-104 is known to be very tear-sensitive. Damage received during processing could propagate into the insulation, causing later failure. The addition of FEP film to the system also serves to protect the silicone from damage and to preserve its integrity if damage were to occur.
 - (4) Versatility
- (U) FEP film is known to be inert in the presence of any of the common materials used in solid rocket motor processing. Additionally, any of these materials in the adhesives category will be bondable to the FEP. Clearly then, the film allows the designer, the M&P Engineer, and the propellant chemist great versatility in their individual disciplines.
 - (5) Good Bonding and Structural Adequacy
- Lockheed Propulsion Company has conducted bond studies using various combinations of DC 93-104, FEP film, liner, and propellant. The systems tested are listed in Table 4-15. Bond-in-tension and peel data are presented for tests at -65, +70, and +165°F.
- (U) The data in Table 4-15 indicate that successful bonding systems can be achieved with FEP film. The best overall system from a bonding standpoint is FEP in combination with liner. This is to be expected because the liner is designed to impart better bonding between propellant and the insulation substrate. The two systems omitting liner do not produce cohesive-in-the-propellant failure modes at all temperatures. They are also substantially lower in peel strength.
- (U) When applied with liner, the FEP film system provides good structural integrity and is capable of bonding to silicone. The aging capability should be excellent. The low-temperature strain capability of the materials in the above systems is excellent (e.g., FEP film at -65°F has more than 33-percent strain). The DC 93-104, LPL-49, DC-1200 primer, and HTPB materials are well known for their good low-temperature properties.

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Figure 4-14 Weight Loss of DC 93-104 after 89 Hours at High Temperature

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Table 4-15

BOND STRENGTH TEST DATA

	Test Temperature, ⁰ F	Bond-in- Tension, psi	Failure Mode	Peel, pli	Failure Mode
Propellant Systems					
DC 93-104/DC-1200/FEP 1 mil/ HTPB (UFAP)	-65 +70 +165	444 100 65	AP/FEP CPI CP	3.8 1.7 1.1	AP/FEP CPI CPI
DC 93-104/DC-1200/FEP 5 mil/ HTPB (UFAP)	-65 +70 +165	374 50	AP/FEP AP/FEP	3.7 2.0 1.0	AP/FEP CPI CPI
DC 93-104/DC-1200/FEP 1 mil/ LPL-49 liner/HTPB (UFAP)	-65 +70 +165	408 175 80	CPI CP CP	19.8 10.0 5.5	CPI CPI CPI
DC 93-104/DC-1200/DC 93-104/ DC-1200/A1 foil 2 mil/chemlock 205/LPL-59 liner/HTPB (UFAP)	-65 +70 +165	212 137 57	CI CP A205/59	7.8	A205/A1
DC 93-104/DC-1200/DC 93-104/ DC-1200/Cu foil 2 mil/ chemlock 205/LPL-59 liner/HTPB (UFAP)	+70	125	CP	9.5	A205/Cu
DC 93-104/DC-1200/DC 93-104/ DC-1200/Cu foil 2 mil/chemlock 205/HTPB (UFAP)	+70	130	A205/Cu	8.1	СРІ
Release Flap Systems					
DC 93-104/DC-1200/FEP/ chemlock 234/FEP/LPE-17	-65	223	A234/ FEP	3.6	A234; FEP
	+70	108	A234/	4.6	A1200/
	+165	100	CI	3.5	A234/

Definitions:

CPI - Cohesive in the propellant at the interface CP - Cohesive in the propellant A x/y - Adhesive material "x" to material "y" CI - Cohesive in DC 93-104

FEP - Fluorinated ethylene-proylene film 1 to 5 mil thick, etched on both sides for bonding

DC-1200 - Silicone primer

LPL-49 - CTPB based liner LPE-17 - Silicone release flap elastomer LPL-59 - HTPB based liner

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(U) Good bonds are also indicated for the metal foil systems. The data shown are for foils post-bonded to cured DC 93-104 insulation using DC 93-104 itself as an adhesive. These systems offer the option of installing a bondable surface into the chamber after the DC 93-104 insulation has been cured.

Disadvantages

- (U) The bondable film concept has only one minor disadvantage, as discussed below:
 - (1) Low Residuals
- (U) FEP film in the bonding system means that at burnout of the grain, there will be a 1-mil layer that must be burned away prior to ramjet operation. If liner is used, it will also have to be consumed. Based on mass loss rate data obtained by LPC, these types of materials burn at the rate of about 0.01 in./sec in a high heat flux environment. The FEP film would, therefore, be consumed in 100 milliseconds or less with negligible effect. The question of whether to use liner or not is expected to be common to all substrate preparation for bonding techniques considered.
 - (2) Processing (Metal Foils)
- (U) Data acquired at LPC indicate adequate bonds for the foils tested. However either in situ or post-bonding of the foil systems to the DC 93-104 may introduce additional processing difficulties as compared to the film material. LPC experience indicates potential problems with handling of the foils, particularly in secondary bonding applications. Also the foils lack the extensive production use history of the film which has seen extensive application in the short range attack missile (SRAM) motor produced by LPC.

4.4.1 Insulation/Propellant Processing Compatibility

(U) During the course of LPC's continuing efforts to develop optimum interface materials systems for integral ramjet boosters, two potential problem areas were discovered and resolved after extensive study. Both concern the processing compatibility of these advanced materials systems and could lead to serious consequences if not recognized and counteracted. The first of these is the interfacial migration of curative out of isocyanate cured R-45 HTPB propellant into the adjacent substrate. This leads to local depletion of curative in the propellant with the resultant formation of a soft propellant layer near the interface which results in low strength level failures in tension or peel. The second of these is the formation and evolution of gaseous species from DC 93-104 insulation at propellant grain cure temperatures, with migration into the grain during the cure cycle. This leads to fissures or tear-like voids in the cured grain adjacent to the interface, which can result in confined burning of additional propellant surface during rocket firing, with possible catastrophic failure.

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(U) The first of these phenomena was discovered during the course of the subcontract work in support of the Marquardt Interface Materials Investigation Program (AFAPL Contract F33615-72-C-1234). This led to the development of a new, improved liner system for HTPB propellant bonding. The second of these phenomena was discovered during company-sponsored development and demonstration programs in 1973. This led to the development of an improved processing/cure cycle and chemical additives to eliminate the gas evolved. Both phenomena are discussed further as follows.

(U) <u>Propellant Curative Migration</u>. In the development of advanced HTPB propellant formulations for this (ASALM) and other applications, part of the effort has been directed toward lowering end-of-mix viscosity and extending pot life. Success in the achievement of these goals has led to a significant increase in the time from grain casting to propellant gellation. Coincident with this result, the occurrence of soft layers of propellant adjacent to bond interfaces was observed. Study of the problem led to the establishment of a model, which generally explains the phenomena.

(U) Figure 4-15 presents the model in a series of schematic diagrams which depict curative concentration across the interface after propellant gellation. LPC's initial work in bonding HTPB propellants was with a liner system containing an imine curative. With no isocyanate curative in the liner, a concentration imbalance exists at the interface, and the isocyanate curative will tend to migrate out of the propellant into the liner. However, as shown in Part A of Figure 4-15, good bonds can still be obtained provided propellant viscosity is high and/or pot-life is short, thereby keeping the time for curative migration before propellant cure to a small value. This was the circumstance during LPC's early work on bond systems for silicone insulators.

(U) The bondable film bond system aids this situation, since it provides a barrier and limits the volume of substrate into which curative can migrate to the liner, which is less than one-tenth the thickness of the insulator.

(U) The effect of improvements in HTPB propellant formulation and processing, which reduced viscosity and increased pot-life, is shown in Part B. With increased time before the onset of cure, significant curative is lost from the interfacial propellant to prevent adequate cure. The resulting soft layer leads to low bond strengths for the system since failure occurs to this layer. This was the circumstance during LPC's initial work on the Interface Materials Investigation program, using LPL-49 liner.

(U) With this model confirmed in numerous tests at LPC, the effort to overcome this difficulty was directed toward formulating an HTPB liner (isocyanate cured) containing a curative level well in excess of that required to cure the liner itself. This excess curative is provided to diffuse into the propellant from the liner. TDI, DDI and IPDI cured liner systems were considered. More favorable results were obtained with the faster-reacting curatives TDI and DDI. Best results were obtained when the FEP bondable film was used, since the excess curative could only migrate into the propellant as desired. Part C of Figure 4-15 illustrates the resulting condition. By proper optimization, a balanced system was

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(U) obtained in which excellent bonds were achieved even when combined with low viscosity, good pot-life propellant.

(U) LPC's optimum, balanced liner system was developed during the Marquardt Interface Materials Investigation Program, and resulted in the identification of the LPL-59 liner formulation. Final results of that program were obtained with this liner, which contains TDI curative. In arriving at this system, a series of parameters were studied including not only the type of curative, but also equivalence ratio, additives such as bonding agents, liner thickness, number of coats, cured versus uncured liner, surface preparation, and propellant cure time and temperature. When used with the FEP film bond system, the LPL-59 liner was found to yield good results over a wide variety of conditions. As finalized, LPC's process employs a single 20-mil coat of LPL-59, uncured, and with propellant normally cast within 8 hours after liner application, although excellent results have been obtained with casting up to 16 hours after lining.

(U) The propellant curative migration phenomenon discovered with HTPB propellant bond systems during the Interface Materials Investigation Program was thus resolved. Table 4-16 shows the relative importance on bond strengths achievable, of having a balanced liner system.

Table 4-16

(U)

EFFECT OF LINER BALANCE ON BOND STRENGTH

	Relativ	ve Bond S	trength (unaged)
	Ten	sion	P	eel
	$+70^{\circ}\mathrm{F}$	+165°F	+70°F	+165°F
Balanced liner system	1.00	1.00	1,00	1.00
Unbalanced liner system	0.83	0.67	0.91	0.69

(U) For bond systems which do not provide a barrier to curative migration into the substrate insulation, achievement of a balanced propellant interfacial to bulk cure is somewhat more difficult. This situation is shown in Part D of Figure 4-15. Fair bond values can be achieved, but diminishing improvements are realized as the excess curative level is increased further. Somewhat better bonds are obtained by increasing the thickness of liner used (values up to 50 mils thick were considered). However, such thick layers of liner add considerably to the inert residuals present during transition. Further, processing becomes difficult due to liner runs and wipes, with resultant non-uniformity in propellant burn-out.

(U) <u>Insulation Gas Evolution</u>. Early in 1973, 6-inch motors containing DC 93-104 were found to have cracks and tear-like voids in the propellant grain near the insulation surface. Each of these defects individually was small (less than 1 inch in length), but were numerous and randomly dispersed around the periphery and along the length adjacent to the insulation interface.

(U) The occurrence of this condition was unexpected in that it had not been previously noted in any laboratory specimens. The problem occurred to various degrees in several motors and with different propellant systems. Since the phenomenon was evident only in larger-scale hardware, a series of test motors was made to isolate the cause of the problem. Following an extensive study of these and other tests performed in conjunction with Dow Corning, it was established that the propellant defects resulted from gaseous products released from the DC 93-104 insulation during the elevated temperature period of propellant cure. A synopsis of the test results is as follows:

- A subscale motor was processed in which a CTPB propellant was substituted for the HTPB but which contained the same DC 93-104 insulation. The same type of defects were encountered.
- A subscale motor was processed with the HTPB propellant, but without the DC 93-104 insulation. This motor was free of the grain defects.

 Subscale motors were processed with several variations of propellant formulation, cure time, cure temperature, and cooldown cycles. All motors had defects.

A subscale motor was processed and x-rayed before start of cure and found to be defect-free. The same motor was then cured and x-rayed again while hot, prior to cooldown. The motor had defects.

• Stress-free block specimens (9- by 9-inch) with DC 93-104 insulation over a steel base plate were prepared. An aluminum foil dam was placed around the periphery and propellant was cast onto the insulation and bond system. These specimens, similar in configuration to a bond specimen but much larger, had defects similar to those in motors.

• A motor was manufactured in which aluminum foil was used, rather than FEP film, on the DC 93-104. No defects occurred in the motor grain, but the foil separated from the insulation in several areas in the form of blisters. This was apparent in x-rays of the motor while hot with only a partial cure in the propellant.

• A motor was made in which the insulation was dried 16 hours at $+160^{\circ}$ F prior to casting of the propellant. The motor had defects.

• A motor was made in which the insulation was dried 5 days at +160°F under a 27-inch Hg vacuum. This motor was defectfree.

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(U) To verify the presence of gases and identify the species present, an off-gas analysis was performed on several material combinations using the mass spectrometer. The results of this analysis were:

 Specimens containing DC 93-104 plus propellant and bond system materials were found to evolve a significant quantity of hydrogen gas, and a lesser quantity of methane.

Specimens containing only propellant and bond system materials evidenced negligible quantities of these or other gases.

(U) The determination that the principal gas being evolved was hydrogen helps to explain the absence of defects in laboratory bond specimens. Such a low-molecular-size gas can be expected to diffuse comparatively rapidly through the heavily loaded structure of the propellant polymer. The thin FEP film (a fluorocarbon) is also permeable to hydrogen. Diffusion path lengths to the free surface of the specimens are less than 1 inch, which would allow the hydrogen to escape. This conclusion is supported by the appearance of motors containing defects due to gas evolution, in which the number of such defects was notably reduced near the ends of the grains.

(U) The results of a laboratory investigation indicated that the basic chemistry of the DC 93-104 insulation system leads to formation of hydrogen gas at temperatures in the range of propellant cure temperatures. Although the quantities of gas are small in absolute terms, they are significant in terms of the defects they can form in a propellant grain. The process leading to cracks in the grain is both complex and subtle. It is dependent upon insulation cure temperature, propellant cure temperature, solubility of the gas in the propellant grain, and diffusion path length to free surfaces. For diffusion path lengths as large as those found in motor hardware, solubility and temperature become the controlling factors.

(U) <u>Solutions to Gas Evolution</u>. Lockheed Propulsion Company studied and identified three solutions to the problem of gas evolution from silicone insulation in integral rocket ramjet materials interface systems. These potential solutions were:

- (1) Modification of motor processing procedures
- (2) Chemical modification of the liner and/or propellant
- (3) Chemical modification of the insulation

(U) <u>Modification of Motor Processing</u>. The strong temperature-dependence of the gas evolution phenomena provides one means for averting damage to the rocket grain, by accelerating gas formation and diffusion out of the insulation during its cure cycle prior to propellant casting. The test motor discussed earlier (in which the insulation was vacuum-dried for 5 days and resulted in a defect-free grain) is evidence that the problem condition can be eliminated by physical treatment of the insulation.

(U) <u>Chemical Modification to Liner and/or Propellant</u>. Under Air Force Contract F04611-69-C-0038, LPC developed and demonstrated that the addition of a catalyst and acceptor to the aluminum hydride propellant promoted scavenging of hydrogen that was being released within the propellant. Thus the problem was controlled at its source. The reaction is shown below wherein the H_2 acceptor is the R-45 polybutadiene polymer.

> palladium on carbon H₂ + R-CH=CH-R $\frac{(catalyst)}{R}$ R-CH₂ - CH₂ - R

(U) The composition of the catalyst is as follows:

Ingredient	%
Palladium	4.5
Platinum	0.5
Iron	5.0
Carbon	90.0

Very small quantities of this catalyst were found to be effective in controlling hydrogen, under the Air Force Aluminum Hydride Contract.

(U) <u>Chemical Modification of DC 93-104</u>. Although the first approach to chemical modification was to add the catalyst to the liner and propellant, another possible solution would be the scavenging of the gas being given off by the insulation at its source (in the insulation). This approach was evaluated in laboratory tests and found to be effective, but was not considered as a primary fix due to the possibility of compromise of the thermal performance of the DC 93-104.

(U) <u>Conclusions on Solutions to Insulation/Gas Evolution</u>. As a result of the studies conducted at LPC, hydrogen gas evolution from DC 93-104 at the temperatures associated with rocket motor cure cycles has been identified as a potentially serious hazard to successful processing of integral rocket/ramjet interface systems. Three solutions to this problem have been developed and proven successful in actual motor hardware. For the conduct of the integral booster development program, LPC elected to use a modified motor process procedure plus the addition of 1.0-percent palladium-on-carbon catalyst to LPL-59 liner. The modified process procedure employs a vacuum bake-out/cure cycle for the DC 93-104 insulation. This combined approach, identical to that used in the Martin/LPC demonstration motor program, with the exception of no catalyst addition to the propellant, provides the least departure from established technology on the ramjet insulator and rocket propellant.

4.4.2 Optimized System

(U) Lockheed Propulsion Company's continued study of materials interface systems for integral rocket/ramjets has led to the development of an

(U) optimized system as described in the previous subsections. This system consists of the following materials:

Propellant	LPC-691B
Liner	LPL-59 with 1-percent palladium-on-carbon catalyst (re-identified as LPL-63), applied as a single 0.020-inch-thick uncured coat
Bond System	LPE-18, etched, FEP-bondable film, 0.002- inch-thick, bonded in situ to the insulation at casting
Primer	DC-1200
Insulation	DC 93-104 with a moderately elevated tem- perature bake-out/cure cycle

(U) Bond results for this optimized system are shown in Table 4-17. Values at all conditions are excellent and quite equivalent to bonds obtainable with non-silicone insulators, since the failures are occurring in the propellant. Specimens do occasionally show cohesive failure in the DC 93-104 insulation itself; the material has a relatively low tear strength compared to its tensile strength. The effects of accelerated aging are minimal in the optimized system with an indication of a small increase in propellant strength due to additional curing.

(U) For the bonding of inert release-flap materials, LPC's studies have led to the development of a corresponding optimized system that provides minimum residuals effects during initial ramjet operation. This system consists of the following materials:

Release Flap	LPE-17 silicone elastomer
Bond System	LPE-18, etched, FEB-bondable film, 0.002- inch-thick, bonded in situ to both sides of the LPE-17, and to the insulation at casting
Adhesive (FEP to FEP)	Chemlok 234
Primer	DC-1200
Insulation	DC 93-104 with a moderately evaluated tem- perature bake-out/cure cycle

(U) Bond results for this optimized system are shown in Table 4-18. Values are also excellent, and reflect process improvements achieved. Therefore, with the bond test results obtained on both other contracts and LPC in-house work, the task remaining for the integral booster development program was to round out the data matrix, filling in those data points which were not obtained in previous contract and company-sponsored efforts.

ST. (Propellant	RENGTH TEST System: DC 9.	DATA FOR 3-104/DC-1	OPTIMIZ 200/LPE-	ED PRO 18/LPL	PELLAN1 -59 (with	SYSTEM BOND 1% catalyst)/LPC-6	(91-B)
	Test Temperature, ⁰ F	Bond-in- Tension, psi	Failure Mode	Peel, pli	Failure Mode	Double-Lap Shear Stress/Strain, psi/%	Failure Mode
Unaged	-65 +70 +165	385 138 72	C C C C	30.2 12.0 6.8	CPI CPI CPI	 123/56 71/58	C C :
Aged & weeks at +145 ⁰ F	-65 +70 +165	359 168 74	ចក្ខភួត	31.6 13.0 7.2	CPI CPI CPI	 124/56 	: <mark>6</mark> :

Table 4-17

Definitions:

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CPI - Cohesive in the propellant at the interfaceCP - Cohesive in the propellantCI - Cohesive in the insulation

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BOND STRENGTH DATA FOR OPTIMIZED RELEASE FLAP SYSTEM (Release Flap System: DC 93-104/DC-1200/LPE-18/Chemlok 234/LPE-18/LPE-17)

Failure Mode	;	A1200/FEP	A1200/FEP	A1200/FEP	
Double-Lap ear-Stress/ rain,psi/%	1	53/	14/	/85	
Failure Sh Mode St	A234/FEP	A234/FEP	A234/FEP	A234/FEP	
Peel, psi	8.5	6.2	5.0	6.1	
Failure Mode	A234/FEP	A234/FEP	A234/FEP	A234/FEP	
Bond-in- Tension, psi	235	125	112	125	
Test Temperature, ⁰ F	-65	+70	+165	02+	
	Unaged)		Aged 4 weeks at +165°F	

Definitions:

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A234/FEP - Adhesive between Chemlok 234 and FEP film A1200/FEP - Adhesive between DC-1200 and FEP film

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(U) The laboratory bonding tests were performed with a $2\frac{1}{2}$ -gallon propellant mix to evaluate the adhesive systems in the motor. There are two major bond interfaces: the silicone insulation/liner/propellant interface, and the silicone/adhesive/release flap interface. Prime considerations are structural adequacy, aging capability, and suitability for rocket/ramjet takeover conditions.

(U) The baseline materials for case insulation, release flap, liner, and propellant are as follows:

Component	Material	Туре
Insulation Release flap Liner Propellant	DC 93-104 ^(a) LPE-17 LPL-63 LPC-691B	Silicone Silicone HTPB based HTPB
Fropenant		

(U) It was planned to test (where applicable) each of the systems in the two modes that are listed below with the data to be reported for each:

Double-lap shear test	Bond strength, failure mode
90-degree peel test	Peel strength, failure mode

(U) The configuration, quantity, and test conditions for each system are shown in Table 4-19. As previously noted, data are already available for part of the series, and the matrix has been optimized so as to evaluate only those interface systems considered critical to structural integrity. Further, where data were to become available from another task such as analog motors, the matrix was also optimized to preclude repetition of data acquisition. The test matrix consists of 30 peel and 6 double-lap shear specimens, which fills out the matrix of existing data on the primary bond system approach. Aging was conducted over a 4-week period at $+165^{\circ}F$, with withdrawals at 2 and 4 weeks. Table 4-20 shows the test results of the bond and accelerated age program. As can be seen, where a direct comparison is possible, the test data show favorable comparison with the earlier results, shown in Table 4-17. This leads to the conclusion that the elevated temperature aging exposure had no adverse effect on the adhesive properties of the system.

4.5 ANALOG MOTORS

(U) The analog motor program planned for the integral booster development program was only partially completed at the time of termination of contract work. At that point, all 5 motors were prepared to the point of liner application and propellant cast.

⁽a) Prepared with bondable fluorinated ethylene propylene (FEP) film on exposed interface surface.

rpe of Test	Sample Configuration	Motor Area	Samples	Aging Weeks at +165°F	<u>Tem</u> - 65	Test peratu +70	re, ⁰ F + 165
Jouble - Lap Shear	Steel/DC 1200/DC 93-104/ DC 1200/FEP/Liner/ Propellant/Repeat	Insulation/ Liner/ Propellant	9	0	3	*(a)	m
)0-Degree Peel	DC 93-104/DC 1200/FEP/ Liner/Propellant	Insulation/ Liner/ Propellant	m m vo	0 2(b) 4	* ~ ~	* *	m m
	Steel/DC 1200/DC 93-104/ DC 1200/FEP/Chemlok 234/FEP/LPE-17	Insulation/ Adhesive/ Release Boot	\$ M \$	004	n n n	* m	m m

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Table 4-19

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		Teet	Test R	esults
Type Sample	Weeks Aging	Temperature (⁰ F)	Strain (Maximum)	Stress (Maximum)
ouble Lap Shear	0	- 65	76%	257 psi
:eel/DC 1200/DC 93-104/ C 1200/FEP/Liner/ ropellant/Repeat		+165	69%	48 psi
			Peel S	trength
)-Degree Peel	•	+165	8.() pli
C 93-104/DC 1200/FEP/	2	- 65	30.0) pli
iner/Propellant	4	+165 -65	9.4 50.0	t pli) pli
eel/DC 1200/DC 93-104/ C 1200/FEP/Chemlok	0	+165 -65	0.0	ild (ild b
4/FEP/LPE-17	2	-65	1.1	, pli
	4	+165	1.8	3 pli
		+70 -65	3.1	i pli

Table 4-20

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(U) The analog motor program was designed to evaluate the propellant and bond system in a motor configuration. The motors were to be instrumented to determine innerbore hoop strain and bondline stress capability. The analog motor configuration is shown in Figure 4-16. Table 4-21 shows the 7-motor test plan for the 6- by 12-inch analogs. Because two of the analog tests were already successfully completed on prior company-sponsored work, the total test matrix consists of only 5 motors. The analogs were to contain LPC-691B propellant, DC 93-104 insulation with the specified hole pattern and filler material, and LPE-17 release flaps as defined in the Phase I analysis. The grain configuration to be used in five of these analog motors was a circular port with end releases, if required. Induced loads in this configuration are easily predictable, therefore, test results can be readily correlated with predictions. Of the two additional motors having the actual keyhole configuration, one has been tested and the other would have been tested for comparison with the circular-port test results, had the program been completed.

(U) The circular-port configuration for two motors was designed to give an innerbore hoop strain equivalent to that in the full-scale keyhole design at the required low temperature of -65° F. After thermal cycling or vibration, the motors were to be taken to failure at their low-temperature limit. Two of the circular-port grains are under-designed to force failure at the inner-bore. One has been tested. The remaining motor was to be used to force failure at the bondline. These tests would have verified the stress and strain safety factors for the full-scale grain. Two keyhole designs would have established confidence in the full-scale safety factors.

(U) The instrumentation planned for use in these motors was selected on the basis of LPC experience (with respect to adequacy and response) in measuring the displacements at the locations and/or interfaces considered to be structurally germane to the motor design. The gage configuration is shown in Figure 4-17. The full-scale motor is designed so that the critical induced loads occur in the motor port rather than at the bonded interfaces on the grain periphery. Therefore, the use of more sophisticated (and costly) gages to measure normal and shear stresses at the grain periphery is not warranted.

4.6 SVS LABORATORY STUDIES

(U) The objective of the SVS laboratory study effort was to evaluate specific SVS design features related to the SVS configuration of the integral ramjet booster.

(U) The basic approach for identification of the tasks to be performed was to isolate specific technical areas for investigation which are unique to the SVS integral ramjet boost r and have not been developed and proven on other LPC contractual or company-sponsored programs.

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Figure 4-16 Analog Motor Configuration

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(a) Numbers shown are motor numbers.

(b) All motors will be x-rayed during thermal excursions.

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ANALOG MOTOR TEST PROGRAM MATRIX^(a)

Table 4-21





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Figure 4-17 Analog Motor In Situ Strain Gage Configuration

(U) To accomplish this, it was necessary to identify the key technical considerations related to the SVS concept and individually assess the technical status of the important design features. Table 4-22 lists those design features which LPC consider to be critical to the SVS concept. Also on Table 4-22 are comments as to the technical status of each item. A review of the content of Table 4-22 shows that data exist on virtually all of the key design facets of the SVS concept to a level which fulfills the requirements of this program with the exception of one area. The only area in which basic data are lacking is that related to bond of the SVS seal to the DC 93-104 combustor insulator over the required filled outgassing holes. Therefore, the SVS laboratory study effort was structured to fill out the seal bond and material compatibility data matrix with particular emphasis on study of seal bond characteristics when bonded over filled combustor outgassing holes. The seal-to-combustor insulation bondline length to assure adequate bond strength was also verified to support Phase I analytical predictions.

(U) The test specimen configuration for the SVS laboratory studies is shown in Figure 4-18. The test matrix is shown in Table 4-23. The type of specimen selected was a 90-degree peel for as close as possible simulation of the loading conditions at the SVS seal-to-chamber insulation bond. The matrix included both candidate hole filler materials, i.e., the celogen/plaster and the pelletized ammonium sulfamate. At the onset of the experiment, available literature on these materials indicated decomposition or sublimation temperatures of approximately 350°F. With this knowledge, it was decided to construct the specimens using a 0.040-inch-thick uncured LPE-6 tie-ply, as shown in Figure 4-18. LPC experience with the SVS system indicates that either LPE-6 or EPDM rubber are acceptable materials for use as tie-plys. The only advantage EPDM offers is its somewhat superior resistance to ozone deterioration. This, however, is not an important consideration for the tieply material, since in application it is essentially unexposed to the surrounding atmospheric conditions. This led to the selection of LPE-6 because of the existence of more extensive use history and bond characteristics data from past LPC programs. Use of the uncured tie-ply is consistent with past practices developed at LPC to provide a strong, low permeability bond of the seal to the chamber insulation. The cure conditions of this system are dictated by the 300° F autoclave cure required for the tie-ply. The 50° F margin was thought to be sufficient to preclude any decomposition of the materials. However, upon cure of the initial group of specimens, visual examination showed that partial decomposition of both filler materials had occurred and had adversely effected the bond between the tie-ply and FEP film. Evidently minor variations in lots and purity of the constituent materials in the filler pellets were sufficient to introduce considerable variability into the actual temperature of decomposition.

(U) With this information in hand, it was decided to evaluate the use of a different hole filler material, selected from the candidate compounds tested early in the laboratory combustor hole-filler task. An examination of these data indicated that ammonium chloride, a subliming salt with a melting temperature of 635° F would provide a much greater margin between its decomposition temperature and the required 300° F cure temperature for the LPE-6 tie-ply. Prior to refabrication of the test specimens, the ammonium chloride results were subjected to a 300° F oven bake cycle for 4 hours

SVS LABORATORY DESIGN CONSIDERATIONS

KEY TECHNICAL CONSIDERATIONS

DESIGN FEATURE

- SEAL BOND LINE LENGTH
- SEAL MATERIAL AND CONSTRUCTION
- CUP MATERIAL AND FABRICATION
- SEAL BOND TO DC 93-104
- PROPELLANT TO CUP BOND

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- SVS FLUID COMPATIBILITY
- SEAL
- cup
- DC 93-104
- BOND OF SEAL TO DC 93-104 (OVER-FILLED OUTGASSING HOLES)

COMMENTS

- PROVEN ON OTHER LPC PROGRAMS
- LIMITED DATA EXISTS
- NO DATA EXISTS



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Table 4-23

SVS LABORATORY SPECIMEN EVALUATION TEST MATRIX (90-Degree Peel)

	+165°F	+70°F	-65°F
0 Time	3	3	3
*2 Weeks	3	3	3
*4 Weeks	3	3	3

* Aging to be conducted at +165°F in silicone support fluid.

NOTE: Each group of three specimens contains

(1) Control

(2) Ammonium chloride hole filler

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(U) to evaluate potential decomposition through weight loss. These tests showed negligible weight loss, which indicated the filler pellets would survive the tie-ply cure cycle without adverse effect on the seal bond to the DC 93-104. It should be noted that the ammonium chloride is not one of the primary candidate materials being tested for the case-bonded approach. The basic difference being the higher sublimate temperature of the ammonium chloride. It was judged that the lower temperatures of decomposition typical of the celogen/plaster and ammonium sulfamate compounds would be better for the case-bonded motors, since the entire internal cylindrical surface of the combustor requires hole fillers, and the lower decomposition temperature would appear to enhance rapid cleaning of the holes at ramjet take-over. However, in the SVS motor, only the small number of holes directly underneath the seal bond area require filling, therefore the maintenance of a low subliming temperature for the filler material is considered to be of less importance for this application.

(U) The zero time, 2-week, and 4-week aging data are shown in Table 4-24. The specimens were aged at $+165^{\circ}F$ immersed in the SVS silicone support fluid. For comparison purposes, Table 4-24 also includes peel data from tests conducted earlier at LPC on the same bond interface which did not include the holes and filler materials. Comparison of the SVS laboratory study test data with the earlier test results indicates that inclusion of the holes and ammonium chloride filler pellets had no pronounced adverse effect on the SVS seal peel strength values.

4.7 FULL-SCALE HEAVYWALL MOTORS

(U) The Phase III task related to full-scale heavywall motor and tooling design was completed prior to program redirection by the Air Force. The completed full-scale heavywall motor design drawing package consists of the following six drawings, shown as Figures 4-19 through 4-24, respectively.

Drawing 299871, Motor Assembly (Figure 4-19)

Drawing 299891, Can Assembly (Figure 4-20)

Drawing 299787, Nozzle Assembly (Figure 4-21)

Drawing 299875, Can and Insulation Assembly (Figure 4-22)

Drawing 299872, Cartridge Loaded (Figure 4-23)

Drawing 299876, Release Boot (Figure 4-24)

(U) As can be seen by review of the drawings, the overall full-scale heavywall motor design approach is essentially the same as planned at the onset of the program. The unit will be cast in a cylindrical steel cartridge which will later be potted into LPC's 20-inch-diameter Char motor for test firing. The cartridge will contain all the basic features of the full-scale flightweight motor in that the prescribed insulation outgassing hole pattern and filler

Table 4-24

SVS LABORATORY STUDIES 90-DEGREE PEEL TEST RESULTS (Pounds/Linear Inch)

Aging	Test Temperature, ⁰ F			
(weeks)	+165	+70	-65	
0	5.5/5.3	5.3/6.4	26/17.5	
2	6.0/4.2	9.6/8.4	16.5/15.8	
4	2.9/3.7	8.4/3.7	13.2/15.1	
	Previous LPC Data (No Pellets)			
0	3.5	4.5	4.7	

KEY: (Control, No Pellets)/with Pellets (Avg. 2 Specimens)

(The reverse is blank)





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Figure 4-21 Nozzle Assembly





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Figure 4-23 Cartridge Loaded

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(U) material will be incorporated. The design also eliminated the aft end release flap as was shown satisfactory by the stress analysis, but the head and release is retained as recommended for processing considerations. The propellant grain has been configured to yield ballistic performance as close as possible to that predicted for the full-scale flightweight motor.

(U) The tooling concept for the full-scale heavywall motors is shown on Figures 4-25a through 4-25e, which is a composite five-sheet drawing, 299875-501-CTS. The tool details depicted are all those necessary to cast and process the DC 93-104 combustor insulation, drill the combustor outgassing holes, and cast and process the propellant grain. The tooling concept provides for in situ placement of the forward release flap and inhibitor. Other key features of the tooling concept are a two-piece "dove tail" propellant casting mandrel design: This feature allows removal of the mandrel in two sections, which will be necessary when processing full-scale flightweight motors which incorporate the ramjet nozzle as an integral part of the motor case. The basic tooling approach is conventional in all respects and employs well proven concepts. Every effort has been made to incorporate features in the tool design concept which will allow use of a major portion of the tools for both the heavywall and flightweight motors with minor modifications.





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Set and Accessories Sheet 4 of 5

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Figure 4-25e Casting Tooling Set and Accessories Sheet 5 of 5

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Section 5

CONCLUSIONS AND RECOMMENDATIONS

(U) An assessment has been made of the results obtained as of the time the work effort was stopped, and the status of technology as developed for high performance integral ramjet boosters. The following conclusions have been reached.

CONCLUSIONS

- 1. Case bonding of an integral booster grain to the baseline DC 93-104 silicone combustor insulation is feasible using an HTPB propellant polymer. Feasibility has been established in subscale size and for an accelerated age time of one month in regards to performance requirements, bond reliability, structural integrity, materials compatibility, motor processability and minimum residuals. Scale-up to full size and exposure to long-term aging remain to be demonstrated, however, every indication from data now available is that both of these steps can be accomplished successfully without difficulty.
- 2. As an alternative to case bonding, the Lockheed developed Stress-free Viscous System (SVS) is also feasible with the baseline silicone insulator, HTPB propellant materials system. Feasibility has been established to a similar degree as for case bonding in this system. SVS has been proven under separate Air Force contract in full size flight-type hardware for air launched missile environments.
- 3. Performance achievable in the baseline booster designs for both case-bonded and SVS systems, meets or exceeds all system requirements established for the contract. Capacity exists therefore for design adjustment as requirements evolve from the studies of the TIS contractors.
- 4. The case-bonded booster requires the minimum departure from system proven concepts for solid rocket boosters including the integral-ramjet type, and has been the basis for studies to date under the CIS and TIS programs. Conversely, the SVS booster provides greater latitude for ultimate growth. The concept can accommodate higher solids loaded performance optimized propellants, and 100 percent web fraction concepts like Grain Burn Pattern Regulation (GBPR) and Wired End Burners (WEB). Furthermore, it provides extreme flexibility regarding grain length to diameter ratio or insulation configuration and chemistry since grain strain and bond considerations are eliminated.

- 5. The LPC 691 HTPB propellant system is well proven and characterized and is an excellent system optimized to the needs of ASALM integral booster. It is ready for scale-up to plant-scale mixes and full-size motors. The system has flexibility in burn rate adjustment and capacity for increased solids loading. Pot life extends to 16 hours.
- 6. The LPC in-situ FEP film/LPL 63 liner bond system is well proven and characterized and is an excellent system optimized to the needs of modern integral ramjet boosters. It is compatible with drilled insulation off-gas for backside heating accomodation, when combined with low-temperature reacting plastercelogen hole filler pellets. The combined system has been demonstrated in subscale motor tests including firing at -65°F. Data to date indicates that analog motor tests, which remain to be completed, can be accomplished successfully without difficulty.
- 7. Several component integration and interface coordination details remain to be finalized between booster and ramjet contractors.

<u>Case.</u> The flight-weight case material L605 requires verification of capability to accommodate rocket mode operating conditions under low temperature environment (-65° F). Fracture toughness data is being obtained by the Air Force Materials Lab (AFML). A fracture mechanics analysis for the rocket mode should be conducted by the continuation booster contractor.

Ejectable nozzle. Final verification of ejection mechanism and rocket nozzle materials and design needs to be completed. Rocket nozzle to ramjet nozzle sealing is a critical interface and is not yet settled. The ramjet nozzle material remains to be selected, and one of the candidates, DC93-104, is of concern. O-ring sealing against an elastomeric material in a nozzle entrance region during rocket mode operation, may prove to be difficult.

<u>Dome closure</u>. The dome closure design requires finalization. Technical comments provided by LPC are under study by the ramjet contractor and the resolution of these should be reviewed regarding technical acceptability by the continuation booster contractor. Since this component forms a part of the rocket mode pressure vessel, questions must be finalized regarding insertion and removal from the forward end, to permit processing of the booster grain and bond system, plus adequacy of dome cover to port sealing against the high pressures and temperatures of rocket mode operation.

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8. The program as being conducted at the time of work stoppage was achieving very good results and was expected to fully achieve the contract objectives as planned.

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RECOMMENDATIONS

- 1. It is recommended that the design and materials system as developed under Phase I and II of this contract be established as baselines from which to continue and complete full-scale motor development and demonstration. The design and systems are well founded, with excellent performance.
- 2. It is recommended that the case-bonded approach be retained as primary, since it appears to provide the least potential for overall system impact at this time, and is fully capable of meeting all requirements.
- 3. It is recommended that the SVS approach be retained in consideration for growth potential with higher performance, longer-lived capability under fatigue loads.

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Appendix A

SUBSCALE INTEGRAL RAMJET BOOSTER BALLISTIC CHARACTERIZATION TESTS

A. I SUMMARY

(U) Candidate propellant formulations for the integral ramjet booster were ballistically characterized with 6-inch-diameter subscale motors. The objectives of the tests were to obtain ballistic performance data, to evaluate erosive burning characteristics, to obtain burn rate and temperature sensitivity data, and to evaluate the booster keyhole grain design in subscale hardware.

(U) A total of seven batches of LPC-691B, the proposed booster propellant, were cast into 6-inch motors. Batch size ranged from 1 to 10 gallons. In addition, three 6-inch motors were cast from a 10-gallon batch of an alternate formulation in which fluid energy mill (FEM) ground oxidizer was substituted for UFAP.

(C) Thirteen 6-inch motors were statically fired. A program summary is presented in Table A-1 and performance data are presented in Table A-2. Significant test results for the characterization of the proposed LPC-691B formulation are as follows:

- (1) An average standard burn rate of 1.31 in./sec at +70° F was derived from 6 x 11.4s. Burn rate scale factors from cured strand data indicated minor scatter, most of which is within data accuracy limits. Data from the first 10-gallon batch indicated a scale-up factor from strand data of 1.05. However, data from subsequent batches indicated scale-down factors ranging from 0.91 to 0.99, which indicates that the scaling factor is not of great magnitude for a motor of this size.
- (2) The effects of erosive burning for the keyhole grain configuration were found to be negligible in a special test configuration with a port-to-throat area ratio of 1. An initial pressure ratio of 1. 20 was determined from measured head-end pressure and predicted equivalent "end-burner" pressure. A factor of this magnitude is attributed to mass addition and it verifies that the LPC-691B propellant and keyhole grain have a low susceptibility to erosive burning.

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		Stran	id Data (70°	F)		
Propellant and Batch No.	Size, gal	rb1000, in./sec	rb2000. in./sec	١¤	Configuration	Objectives 0
LPC-691B 0156-12D	10	1. 28	1. 90	0.59	Four 6C3 x 11. 4s One 6C3 x 33. 8, Ap/At, initial, = 1.0	 Burning rate and slope Temperature Temperature Burning rate scale factor Erosive burning
LPC-691B 0156-14A	2-1/2	1. 33	2.05	0.63	One 6 x 11.4 with subscale keyhole grain design and 1/4-inch DC 93-104 case insulation	 (1) Curve profile (2) Performance evaluation
LPC-691B 0156-46G & H	-	I, 41	. 2. 15	0.59	One 6 x 11. 4 with subscale keyhole grain design; DC 93-104 case insula- tion plasma-arc treated prior to propellant casting	 Propellant/liner bond quality evaluation Temperature sensitivity Performance, burning rate, and burning rate
LPC-691B 0156-47A & B	-	1.41	2. 15	0.59	One 6 x 11. 4 with subscale keyhole grain design; FEP film used between the DC 93104 case insu- lation and propellant	Same as above
LPC-691B 0156-48A	01	1.31	1.82	0.46	Two 6 x 11. 4s with sub- scale keyhole grain designs; DC 93-104 case insulation with FEP film interface	 Performance evaluation Temperature sensitivity mage and and and and and and and and and and
LPC-808A 0138-81A	10	1.17	1.72	0. 55	Three 6C3 x 11.4s	Evaluate alternate propel- lant for the booster

Table A. 1

6-INCII MOTOR TEST PROGRAM

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6-INCH MOTOR PERFORMANCE SUMMARY

Motor	2297	2298	2299	2300	2301	PT001 2310	3834-01 2403	3834-02 2406	3834-05 2504	3834-03 2505	3801-03 2529	3801-01 2530	3801-02 2534
t date	26 Apr 73	27 Apr 73	1 May 73	2 May 73	2 May 73	23 May 73	17 Oct 73	18 Oct 73	20 Dec 73	20 Dec 73	17 Jan 74	17 Jan 74	23 Jan 7-
t temperature.	Amb	+165	Amb	-65	Amb	Amb	+70	-65	+165	-65	+56	+162	-65
tial, in. bat area, in. ² diameter, in. ansion ratio	1. 772 2. 466	1. 770 2. 461	3.00 7.069	1. 360	1.501	1. 531 1. 841 5. 21 4. 40	1. 555 1. 899 3. 22 4. 29	1.553 1.894 3.22 4.30	1. 199 1. 137 3. 65 9. 20	1. 200 1. 134 3. 65 9. 23	1.473 1.704 3.18 4.66	1.473 1.705 3.16 4.66	1.480 1.720 3.18 4.62
, in.	1.5	1.5	1.5	1.5	1.5	14.8 2.01	14. 3 2. 01	14.3 2 01	13.65 2.01	13.63 2.01	14.66 1.5	14.69 1.5	14. 77
eb burnout, sec tal burn, sec	1. 389 1. 58	1. 225 1. 40	1. 196 1. 50	C. 843 1. 20	0.880 0.96	2. 209 2. 49	2. 245 2. 53	3. 052 3. 20	1. 142 1. 25	1.612 1.76	0.943	0.835 0.97	1. 167 1. 35
rning raie, o, /sec soure	1,080	1. 224	1. 254	1.779	1.705	0.910	0.895	0.659	1. 762	1. 247	1,591	1. 796	1. 285
sb time integral, sia-sec	126	517	971	1,540	1, 306	1, 264	1, 106	1, 155	1, 864	1, 850	1, 306	1, 3 43	1, 307
ral time inte- ral, psia-sec	957	940	1,024	1,629	1, 352	1, 288	1, 158	1, 167	1, 900	1, 890	1, 372	i, 378	1, 364
crage pressure, sis	659	745	819	1,820	1,486	572	493	378	1,634	1, 148	1, 385	1,609	1, 120
elivered						5, 112	4, 948	4, 973	5, 056	5, 046	5, 132	5, 148	5, 112
(1000 psia, +70° F) heoretical star efficiency						5, 123 5, 168 0. 991	4, 962 5, 168 0, 960	5,019 5,168 0.971	5,027 5,168 0,973	5, 062 5, 168 0, 979	5, 125 5, 168 0, 992	5, 117 5, 168 0, 990	5, 140 5, 168 0, 995
ib time integral,						3, 318	2, 977	2, 981	3, 197	3, 163	3, 153	3, 335	3, 360
ral time inte- ral, lb-sec erage thrust, lb scific impulse,						3, 367 1, 502	3, 117 1, 326	3,003 977	3, 254 2, 803	3, 223 1, 962	3, 307 3, 343	3, 412 3, 994	3, 493 2, 879
Delivered						226.5	218.0	210.6	238.8	237.1	225. 9	233.0	237.3
(0° 1/2 L)						262.5	262.5	262.5	262.5	262.5	262.5	262.5	262.5
(0° 1/2 L) efficiency						245. 2 0. 934	237.7 0.905	237.0 0.903	234.4 0.893	239.7 0.913	228. Z 0. 869	233.0 0.888	243.1 0.926
timum eoretical indard						1.455 1.527 1.614	1.449 1.509 1.614	1. 449 1. 472 1. 614	1.611 1.672 1.614	1.611 1.639 1.614	1.468 1.59£ 1.614	1.468 1.604 1.614	1. 466 1. 584 1. 614
Cd)						0. 953	0.952	0.942	0.927	0. 942	0.893	0.921	0.961

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- (3) The keyhole grain design yielded a curve shape in good agreement with the predictions, with minor variations due primarily to deviations in as-built and grain design dimensions in the last four motors tested. In the first motor tested, variations in predicted and measured curve profiles were attributed to propellant defects at the grain periphery.
- (4) The 6 x 11.4 ballistic data were quite satisfactory and demonstrated both performance and reproducibility characteristics commensurate with data gathered on numerous propellants tested in 6 x 11.4 motors at LPC. The average c* was 5,039 ft/sec for the five keyhole grain motors with a c* efficiency of 0,975. The average standard specific impulse was 238.8 seconds with an I_{SD} efficiency of 0.91. The nozzle discharge coefficient (CD) averaged 0.94 for these motors. As is characteristic with all subscale ballistic test motors, because of the grain weight restriction, the measured energyrelated parameters are lower than can be expected for comparable full-scale units. LPC experience indicates that a minimum 5-percent gain in efficiency can be expected in the proposed full-scale motor as compared to the 6 x 11.4. This increase is more than sufficient to meet the minimum required specific impulse of 245.0 seconds.
- (5) The results of the ballistic test motor program show the temperature sensitivity (π_k) of LPC-641B propellant to be 0.153%/°F over the temperature range of -65°F to +165°F. These data were derived from 6- by 11.4-inch motors cast from a single batch of propellant and tested at constant area ratio (K_n) conditions. As shown in this appendix, the total range of π_k values obtained throughout the ballistic test program is 0.119 to 0.194%/°F. However, some earlier test motors displayed minor defects that introduced difficulties in assessing motor burn rates, resulting in less representative temperatures sensitivity values.

(U) The following data were obtained for LPC-808A, the modification containing FEM-ground oxidizer:

- The burn rate determined from 6C3 x 11.4 motor data was

 32 in./sec at 1,000 psia, which meets the booster requirements. The 6 x 11.4 motor data indicated a cured strand-to-motor burning rate scale factor of 1.13.
- (2) Temperature sensitivity compared favorably with that of LPC-691B. A value of 0. 159%/°F was calculated for the temperature range of -65 to +165°F.
- (3) Pressure- and thrust-versus-time performance was typical of 6C3 x 11.4 motor performance. No performance anomalies were observed.

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Each series of tests is evaluated in the following subsection.

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A, 2 DATA EVALUATION

A. 2.1 Batch 0156-12D

(U) Four 6C3 6 x 11.4 ballistic test motors were cast from this batch and fired to provide the burn rate scale-up factor from cured strands to motors. The motors were standard 6 x 11.4s with 3-inch port diameters and 1.5inch webs. The circular-port grains were designed to burn in the bore and on the ends to provide a reasonably neutral pressure-time trace. The igniter design was a pellet bag containing B-KNO3 pellets. Different nozzle throat sizes were used to evaluate the burn rate over the pressure range of interest.

(U) All motors performed successfully. Pressure-versus-time curves are shown in Figure A-1. Cured strand data for Batch 0156-12D are presented in Figure A-2. A scale factor of 1.054 is indicated for calculation of motor burn rate from the strand rate at 1,000 psia.

(U) Temperature sensitivity values were calculated from burning rates at 1,000 psia as follows:

 $\pi_{K}(+70 \text{ to } +165^{\circ} \text{ F}) = 0.151\%/^{\circ} \text{ F}$ $\pi_{K}(+70 \text{ to } -65^{\circ} \text{ F}) = 0.119\%/^{\circ} \text{ F}$ $\pi_{K}(-65 \text{ to } +165^{\circ} \text{ F}) = 0.132\%/^{\circ} \text{ F}$

(U) Three 6 x 11.4 motors, also cast from Batch 0156-12D, were joined together to form a 6 x 34 motor. A 3-inch throat diameter was used to give a port-to-throat ratio of 1.0 for erosive burning evaluation. A low-output igniter was used to minimize igniter contribution to initial pressure. It consisted of an 8-inch-long core of magnesium/Teflon/Viton (MTV) installed in the radial slot between the forward and center grains. The motor fired satisfactorily, yielding the pressure-time history shown in Figure A-3.

(U) The evaluation of erosive burning consisted of comparing the measured head-end pressure with an equivalent non-erosive "end burner" pressure-time profile computed with the LS-241 computer code. These two curves are compared in Figure A-4. The initial pressure ratio of 1.2 can be accounted for by the mass addition effects for a port-to-throat ratio of 1. A plot of pressure ratio versus port-to-throat for mass addition is shown as an inset in Figure A-4. It was concluded that LPC-691B in the keyhole grain configuration shows little tendency toward erosive burning.



Measured Pressure versus Time for 6C3 x 11.4 Ballistic Test Motors, Batch 0156-12D Figure A-1







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Figure A-4 Measured and Predicted Pressure versus Time for 6C3 x 33.8 Ballistic Test Motor, Run 2299



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A. 2. 2 Batch 0156-14A

(U) One 6×11.4 motor was cast from this batch with a subscale keyhole grain design. This design (shown in Figure A-5) is one-third booster scale. The objective of this test was to evaluate ballistic performance of the prototype grain design.

(U) The motor performed successfully. Measured pressure and thrust versus time are plotted in Figure A-6. Ballistic data are summarized in Table A-2.

(U) Predicted and measured chamber pressure are compared in Figure A-7. The actual pressure shows only minor deviation from the theoretical prediction.

(U) Motor and cured strand burn rates are plotted versus pressure in Figure A-8. A scale-down factor of 0.95 is indicated for the strand-to-motor relationship.

A. 2.3 Batch 0156-46G and H (Motor 3834-01) and Batch 0156-47A and B (Motor 3834-02)

(U) Two 6 x 11.4 motors were cast from four laboratory batches of LPC-691B propellant with subscale keyhole slot grain designs. The objectives of the tests were to investigate propellant-to-insulation bond quality, to determine temperature sensitivity over the range of +70 and -65° F, and to obtain other ballistic data.

(U) Motor 3834-01 was fired at ambient temperature (about $+70^{0}$ F) Motor 3834-02 was fired at -65° F. Both motors were instrumented for chamber pressure and thrust. Measured pressure and thrust versus time curves are plotted in Figures A-9 and A-10 for Motors 3834-01 and 3834-02, respectively. Cured strand and motor burn rate data are presented in Figure A-11. Characteristics and ballistic data are summarized in Table A-2.

(U) Performance of both motors was satisfactory and met the primary objective of investigating bond quality. Comparisons of actual and predicted pressure versus time (Figures A-12 and A-13) show minor dispersions that are most probably due to differences between the as-built and design dimensions.

(U) Temperature sensitivity was determined from the following relationship:

 $\pi_{\kappa} = \frac{\ln \overline{P}_{+70} / \overline{P}_{-65}}{\Lambda T}$

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Figure A-5 Subscale Keyhole Grain Design

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Measured Pressure and Thrust versus Time for 6 x 11.4 Ballistic Test Motor 3834-01, Run 2403 Figure A-9









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Measured and Predicted Pressure versus Time for 6 x 11.4 Ballistic Test Motor 3834-01, Run 2403 Figure A-12





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where

 $\overline{P}_{+70} = 493 \text{ psia}$ $\overline{P}_{-65} = 378 \text{ psia}$ $\Delta T = +70 - (-65) = 135^{\circ} \text{ F}$

and

$$\pi_{\rm K} = \frac{\ln 483/378}{135} \times 100 = 0.194\%/{\rm °F}$$

(U) The value is somewhat higher than determined previously (Batch 0156-12D) and is likely due to burning rate variations between the propellant batches used to cast the motors.

A. 2.4 Batch 0156-48A

(U) Two 6 x 11.4 motors were cast from a 10-gallon batch for further temperature sensitivity and ballistic performance evaluation. Both motors had subscale keyhole grain designs. The motors were fired on
20 December 1973; Motor 3834-05 (Test 2504) at +165°F and Motor 3834-03 (Test 2505) at -65°F. The motors were instrumented to measure chamber pressure and thrust.

(U) Both tests were successful. Pressure and thrust versus time for both motors is plotted in Figures A-14 and A-15, respectively. Ballistic data are summarized in Table A-2.

(U) Temperature sensitivity was determined as follows:

$$\frac{\overline{P}_{+165}}{\overline{P}_{-65}} = \frac{1.634}{1.148} = 1.42333 = e^{\pi}$$

(U) Predicted and measured pressure-versus-time curves are shown in Figures A-16 and A-17, for Motors -05 and -03, respectively. Comparison shows reasonable agreement.

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Measured Pressure and Thrust versus Time for 6 x 11.4 Ballistic Test Motor 3834-05, Run 2504 Figure A-14

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Figure A-15 Measured Pressure and Thrust versus Time for 6 x 11.4 Ballistic Test Motor 3834-03, Run 2505






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Figure A-17 Measured and Predicted Pressure versus Time for 6 x 11.4 Ballistic Test Motor 3834-03, Run 2505



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(U) Pre- and postfire photographs of Motors -05 and -03 are presented in Figures A-18 through A-21.

A. 2. 5 Batch No. 0138-81A

(U) Three 6C3 x 11.4s were cast from this batch to provide burn rate, burn rate scale, and temperature sensitivity data for LPC-691B propellant modified with FEM ground oxidizer. The motors were fired at ambient $(+56^{\circ} F)$, high $(+162^{\circ} F)$, and low $(-65^{\circ} F)$ temperatures. The motors were instrumented for chamber pressure and thrust.

(U) All tests were successful. Pressure-time curves are shown in Figure A-22. Ballistic data are summarized in Table A-2.

(U) Burn rate data, presented in Figure A-23, show that this formulation has the required burn rate for the booster (i $e_1 \approx 1.3$ in./sec at 1,000 psia). Temperature sensitivity data are also acceptable, as indicated below:

	$\frac{\pi K}{K} (\%/^{\circ} F)$
+56 to +162°F	0.42
+56 to -65° F	0.174
-65 to +162°F	0.159

(U) The scale factor from cured strand data to the 6×11.4 motor burning rate was 1.13. This is substantially higher than factors determined for LPC-691B.

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Figure A-18 Prefire View of Motor 3834-05

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Figure A-20 Prefire View of Motor 3834-03

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Figure A-22 Measured Pressure versus Time for 6 x 11.4 Ballistic Test Motors 3801-01, -02, and -03, Runs 2529, 2530, and 2534

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Appendix B

ULTRA-FINE AMMONIUM PERCHLORATE (UFAP) PREPARATION FACILITY

Lockheed Propulsion Company, using Company funds, has engineered a pilot plant facility for the preparation of ultra-fine ammonium perchlorate (UFAP). This UFAP facility has consistently produced materials with two average particle sizes (0.5 to 0.6 and 0.9 to 1.1 microns).

In developing the UFAP fabrication process, LPC first conducted a laboratory investigation with a Union Process, Inc, laboratory-size attritor. This $1^{1}/_{2}$ -gallon attritor was obtained and evaluated, and was found to be a satisfactory mill for the rapid preparation of UFAP¹. The attritor process has the primary advantage of being able to grind UFAP in one-fourth the time required for vibro-energy mills.

On the basis of this evaluation and the need for larger quantities of UFAP, LPC subsequently purchased a 30-gallon attritor (Figure B-1). The attritor is essentially a ball mill, but of a unique character: it has a stationary grinding tank that is filled with small $({}^{3}\!/_{32}$ -inch) zirconia beads. During the grinding process these beads are kept in constant motion by a centrally mounted agitator. The LPC attritor utilizes recycled, conditioned water to maintain temperature control during the grinding process. Flow meters determine the quantity of water necessary for temperature control. The material of construction is stainless steel.

Associated with the attritor and made by LPC is a remote-controlled operating system. The attritor is located in a revetted building with the start/ stop controls located in a control station at a distance from the attritor. Ammeters and intercom systems are used to monitor the attritor. The LPC attritor facility has the capability not only for remote operation but also for local start-stop when the grinding tank is empty.

In addition, LPC has designed and built a remotely operated loading hopper for addition of the as-received ammonium perchlorate (AP) to the attritor bowl while the agi*ator is in motion. This allows immediate dispersion of the dry AP into the grinding tank, which contains the zirconia beads, Freon grinding fluid, phenethyaziridine coating agent (1 percent based on AP) and Twitchell Base 8240 (0.4 percent based on AP). The latter material is a grinding process aid.

¹ R. H. Epstein and P. G. Butts, "Preparation of UFAP Using the Union Process Attritor", 1971 JANNAF Combined Propulsion Meeting, Specialist Sessions Expanded Abstracts, Nov 1971



Figure B-1 30-Gallon Attritor

During the grinding operation, which requires 12 to 20 hours depending on the particle size desired, in-process samples are easily removed (by use of syringes) and subsequently analyzed. At the end of the grinding process, the contents of the grinding chamber are discharged through a bottom-out valve. The bead bed is washed with excess Freon to remove additional product.

The UFAP is tray-dried in air-circulating ovens to remove excess Freon. This results in a free-flowing product that can be handled in the conventional manner.

Each lot of UFAP produced at LPC by the 30-gallon attritor is analyzed by an MSA analyzer for particle size. Typical MSA curves are shown in Figure B-2 (two runs for each particle size). The organically coated UFAP is stored in sealed polyethylene bags with desiccant. The bags in turn are stored in dry, sealed 30-gallon drums. UFAP stored in this manner has been found to show no change in particle size after 8 months.



Figure B-2 MSA Distribution for UFAP

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Appendix C

RESPONSE OF SVS TO LOADING CONDITIONS

The Stress-free Viscous System (SVS) securely holds the propellant grain in the motor case during environmental shock, vibration, and acceleration, and yet allows thermal expansion and contraction of the grain with little restraint. Also, this system puts the grain in near-perfect equiaxial compression during firing. Thus, structural integrity during all modes of operation is ensured.

C.1 THERMAL CYCLING

For an 18-inch-diameter motor, thermal cycles typically occur over a time span of from 10 to 40 hours. As the motor heats and cools, the propellant expands and contracts approximately ten times as much as the case. The case, therefore, has a relatively constant volume, while the volume of propellant changes. To compensate for this differential in expansion, the SVS allows the grain to translate in the case as its volume contracts or expands.

In a typical thermal cycle, the system functions as follows: As the motor heats, starting from an initial uniform temperature of 70° F, the grain expands and closes the sidewall annular clearance, forcing the supporting liquid slowly forward into the head end, which in turn forces the grain aft. This is shown in Figure C-1, where the dashed lines represent the "hot" position.

	70° F POSITION	L
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Figure C-1 Grain Position at High Temperature

Conversely, as the motor cools the annulus enlarges, drawing the supporting liquid slowly into the annulus, and the grain is slowly pushed forward by atmospheric pressure in the nozzle plenum. The "cold" position is shown by the dashed lines in Figure C-2.

Stresses and strains induced in the grain are essentially zero during thermal cycling, because the grain does not change shape or deform as its volume contracts or expands with temperature change.

Karner and		anna
B	- TOUE DOSITION	
	- 10°F POSITION	

Figure C-2 Grain Position at Low Temperature

C.2 GRAVITATIONAL AND CAPTIVE FLIGHT LOADS

Under long-term storage in a horizontal position, or during transverse acceleration in captive flight, the grain will tend to sink to the bottom of the motor chamber or float to the top, depending on the relative densities of the liquid and propellant. Because of the small annulus width, the center of gravity shift will be well within tolerances dictated by present-day guidance systems; therefore, spacers to center the grains are not required. Of course, the grain will move very slowly under transverse loads because the support liquid must flow around the grain through the thin annulus to allow transverse motion. Therefore, transverse movement will be negligible under flight maneuvering loads even though transverse accelerations are high.

In contrast to transverse flight acceleration loads, longitudinal captive flight loads are relatively low. They will not exceed 1 g because the airplanes that carry the missile cannot exceed this acceleration level due to their thrust-to-weight ratio. Therefore, retention requirements in the longitudinal direction are much lower than in the transverse direction. The SVS relies on plenum pressure acting on the aft grain face to retain the grain under these longitudinal, captive flight loads (Figure C-3) because their duration is too long for shear restraint from the viscous support liquid to be effective. A plenum pressure of about one-third atmosphere is necessary to support a 75-inch-long grain under the above described loads. Thus, a nozzle closure is necessary to provide retention at altitude. Also, the plenum size must be large enough to ensure the one-third atmosphere after the temperature in the closed plenum drops to $-70^{\circ}F$.

During ground handling operations, the motor may be placed vertically with the aft end down and the nozzle closure removed. Under these conditions atmospheric pressure will retain the grain. The pressure differential across the seal under this longitudinal load will be zero if the retention liquid and the grain have the same density. If the liquid density is lower, which is desirable for optimum mass fraction, plenum pressure will exceed

annulus pressure at the seal. For a 75-inch grain and typical grain and liquid densities, this pressure differential across the grain will be only 2.0 psi.





C.3 VIBRATION AND SHOCK LOADS

Vibration loads imposed on the motor case must pass through the supporting liquid to reach the grain. This load path tends to isolate the grain from the environment. In addition, the grain cannot vibrate in any of its natural modes without moving with respect to the case. As the grain attempts to move, the supporting liquid must be displaced through the narrow annulus between the cup and insulation. This provides a very strong damping effect.

All acceleration loads during captive flight, storage, transportation, and handling greater than 1 g may be classified as shock loads because they are of relatively short duration. This is especially true of longitudinal accelerations. The retention system depends upon the viscosity of the supporting liquid to restrict motion resulting from these loads.

C.4 PRESSURIZATION

Pressurization loads are transmitted hydraulically from the combustion chamber to the forward parts of the motor by the support liquid and the grain. With comparatively rigid motor cases, the grain is put into equiaxial compression almost instantly. However, if the case is flexible, a significant pressure differential is developed across the seal. In order to understand why this occurs, the following significant events which occur during and immediately after the pressure transient will be examined:

- (1) The propellant is ignited and pressure rises.
- (2) The pressure is quickly transmitted to the support liquid in the forward end.

- (3) Pressure on both ends of the grain compress it axially and cause it to grow radially, pushing the case outward.
- (4) Distortion of the grain is resisted by its rigidity, which results in a lower pressure in the annulus than in the combustion chamber.
- (5) The grain slowly recovers from this distorted position as liquid flows from the head end into the annulus.

If the grain is rigid and the case is flexible, the initial pressure in the annulus will be zero. At the other extreme, a highly flexible grain will behave almost as a liquid and pressure in the annulus will equal the combustion pressure. Thus, the pressure differential across the seal can vary from zero with a highly flexible grain up to chamber pressure with a highly rigid grain. It turns out that propellant grains are relatively flexible and the pressure differentials resulting from grain rigidity can be expected to be less than one-tenth chamber pressure.

The rapid forward movement of the aft end of the grain also develops an inertial reaction force and a shear force along the sides of the grain. Both of these forces result from rapid axial movement of the aft portion of the grain as the grain is compressed. Therefore, they are of relatively short duration. However, they increase the pressure differential across the seal while they exist.

Figure C-4 summarizes forces acting on the grain and grain motion. Initially, the chamber pressure, P_c , rises. P_f follows it closely. The grain is then compressed into the shape shown by the dashed lines. At this point, the inertial and shear forces go to zero and the remaining pressure differential is due solely to grain rigidity. Liquid flows from the head end into the annulus and the rigidity of the grain restores it to its original shape, which is shown by the stippling. It is now forward of its initial position. The forward movement multiplied by the cross sectional area must equal the volume change of the flexible case forward of the seal.





The seal must be able to withstand the pressure differential from ignition until the propellant burns beyond the seal. After the grain burns beyond the seal, a secondary seal is formed by the cup as shown in Figure C-5. The large pressure differential developed before P_c and P_f become equalized is dissipated well before the burn front reaches the seal. However, the smaller differential associated with grain deformation remains and is withstood by the secondary seal until equilibrium is achieved by flow from the head end.





C.5 ACCELERATION DURING MOTOR OPERATION

During operation, the motor accelerates forward with an acceleration \ddot{X} , which tends to make the grain move toward the nozzle. This is shown in Figure C-6.



Figure C-6 Motor Acceleration

As the grain tends to move aft with respect to the case, a pressure differential is formed between the combustion chamber and the forward end, equilibrating the inertial force of the accelerating grain. This pressure differential is

$$\Delta P = \rho_n L \ddot{X}$$

where $\rho_p = \text{grain density}$

L = grain length

 $\ddot{\mathbf{X}}$ = absolute grain acceleration

However, the pressure differential across the seal is less than the pressure differential between the combustion chamber and the forward end. The expression for the differential across the seal is:

$$\Delta P_{s} = (\rho_{p} - \rho_{f}) L\ddot{X}$$

where ΔP_{a} = pressure differential across the seal

 ρ_{i} = liquid-density

Note that when the grain and liquid densities are equal, the pressure differential across the seal is zero.

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Appendix D

MATERIALS RESEARCH AND DEVELOPMENT REPORT

Descriptions and processing information for constituent items used by LPC in the development and demonstration of an integral low volume booster for ramjet-powered missile application:

(Item #1)

LPC-691D, 87% total solids HTPB propellant (see Exhibit #1 for ingredients and processing information).

(Item #2)

Ultra-fine ammonium perchlorate (see Exhibit #2 for processing information).

(Item #3)

LPL-63, HTPB base liner system (see Exhibit #3 for ingredients and processing information).

(Item #4)

Dehydrogenation catalyst (see Exhibit #4 for ingredients and processing procedure).

(Item #5)

Celogen plaster combustor insulation hole filler material (see Exhibit #5 for formulation and process procedure).

(Item #6)

DC 93-104, Dow Corning silicone combustor insulation material (commercially available from Dow Corning, mixed and cured in accordance with manufacturers specification).

(Item #7)

LPE-6, Polybutadiene isoproene rubber compound, formulation and manufacturing procedures proprietary to Lockheed Propulsion Company.

(Item #8)

LPE-15 (nylon-reinforced neoprene rubber, commercially available).

(Item #9)

LPE-18 (fluorinated ethylene propylene film, commercially available).

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(Item #10)

LPE-19 (EPDM, ethylene propylene rubber, commercially available).

(Item #11)

LPE-17 (millable, dimethyl silicone rubber compound, commercially available).

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Exhibit #1

PROPELLANT MIX RECORD

REQUEST NO.	RWO	CHEMIST		
MIX NO. 6002A	MPO 763-2-50	DATE9/18/74		
OPERATORS	MIX SIZE 5000 gram	_ PROPELLANT TYPE HTPB		
DATE MIXED 9/20/74	TYPE MIXER 1-gal VBP	FORMULATION $\frac{763-691D-12}{NCO/OH} = 0.84/1.0$		

_					
	MATERIAL WEIGHOUT	LOT NO.	WEIGHT PERCENT	WEIGH T GRAMS	MATERIAL CONDITIONING
1	AP, 7-9µ (Fisher Subsieve)	Ray 055	40.80	2,040.0	
2	UFAP, 1µ(MSA)	AG-1022	27.20	1,360.0	
3	Aluminum, S-792	31087-11A	18.00	900.0	
4	Iron Oxide	30638-'IA	1.00	50.0	
5	HX-752	31093-11A	0.15	7.5	
6	UOP-36	30618-11A	0.04	2.0	
7	DTBH	31089-11A	0.04	2.0	
8	IDP	30022-11A	2.00	100.0	
9	R-45	31088-11A	10.03	501.5	
10	IPDI	31090-11A	0.74	35.5	* Adjusted for sample
11			100,00	5,000.0	removal Step 11
12					
13					

	ORDER OF ADDITION	MIX TIME REQ	TIME	TIME STOP	TOTAL	TEMP REQ	ACT. TEMP	PRESS. MM ABS	REMARKS
1	8, 9, 6, 7, 5 & 3	20	0625	0645	20	150 -160			
2	1/8 of 1 & 2	10	0652	0702	10				Add AP by weight and
3		10	0706	0716	10		142		scrape down
4		10	0720	0730	10		150		
5		15	0734	0749	15		150		
6		30	0752	0823	30		158		Mix at 30 RPM
7		30	0820	0856			156		
8		60	0900	1000			154		
9	+ 4	45	1030	1115			155		
10	Cool to 110°F	30	1125	1155		110 5	112		
11	Withdraw 200 gr	ams (weigh	out)	n sea	lant t	ube fo	r wet	strands and Brookfield visc

(12) VISCOSITY INSTRUCTIONS 30 1300 1330 110 5 BROOKFIELD

K) EOM READING _____ TEMP _____ VIS COSITY _____ (CP)

() POT LIFE ______ HRS_____ PF RORVISKO

() POT LIFE _____ HRS ____ F ____ SPEEDS

	AMPLE CASTING AND CURE	c	AST		PRESS	CURE	c	URE	REMARK						
		START	STOP	TOTAL	ABS	TIME	Т	EMP			REM	REMARKS			
1						288	1	45	Vac	uum	cas	t at	40	mm	Hgab
2															
3															
4							Ι	V			1		1		
SP	ECIAL INSTRUCTIONS														
	•			-					_						
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Exhibit #2

LPC Document No. 2540-PP-030 Revision No. 1 September 1974

PROCESS PLAN

UFAP PREPARATION FOR PRODUCTION BATCHES OF LPC-691D

Prepared by:

W.R. West

APPROVED BY:

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R.V. Williams Project Engineer

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J.H. Bonin, Director J.W. White, Chief Technical Director Safety Engineer LOCKHEED PROPULSION COMPANY P.O.BOX111 REDLANDS, CALIFORNIA 92373

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PROGRAM PLAN

1.0 PURPOSE

A FIRE A

To define the detailed procedures for the grinding, drying, screening, packaging and storage of UFAP to be used in 300-gallon production batches of LPC-691D.

2.0 GENERAL REQUIREMENTS

- 2.1 A 1800 pound quantity of UFAP made from a composite of 50 lb Attritor grinds using a single set of raw material lots.
- 2.2 Sample from composite shall have a size of nominal 1.0# (MSA WMD 50 percent point)

3.0 SAFETY considerations

- 3.1 The Attritor will be operated remotely whenever the grinding tank agitator is in motion and the tank contains an $NH_4CIO_4/Freon TF$ slurry. Local operation of the Attritor is approved for discharge (no agitation).
- 3.2 If screening operation is done by hand, then during the operation the operator will bond and ground (use wrist stats) himself to the trays, the flour sifter, and the drum.

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4.0	REQUIRED CHEMICALS <u>Material</u>	Quantity Required	Lot No.
4.1	Freon TF (B. P. 117 ⁰ F	1980 gal (36 drums)	
4.2	NH ₄ CIO ₄ Type II Size 1 per EMS2015	2000 lbs	3108611A
4.3.1	Phenethyl aziridine	32 lbs	3109211A
4.4	Twitchell Base 8240	12 lbs	3109711A
4.5	Dispersing Agent Dessicant Bags, activated	1152 units (200 bags)	N/A

5.0 REQUIRED EQUIPMENT AND MATERIALS

- 5.1 Union Process Attritor, Model 30-S
- 5.2 Remotely Operated Loading Assembly, Dwg ERS-3205
- 5.3 Work Platform, Dwg ERS-3203
- 5.4 Zircoa Ceramic Beads, Size Z-10, approximately 800 lbs
- 5.5 Stainless Steel Beakers
- 5.6 Stainless Steel Pitchers
- 5.7 Non-sparking Spatulas
- 5.8 Stainless Steel Trays
- 5.9 Flour Sifter or Sweco Screener (Building 102)
- 5.10 Kimwipes
- 5.11 Waste Disposal Bags
- 5.12 Large Powder Scoop

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- 5.13 Aluminum Foil
- 5.14 Weston Thermometer
- 5.15 Attritor Data Sheet
- 5.16 Disposable Syringes
- 5.17 Drum Pump with Counter
- 5.18 5-Gallon P.E. Tanks

6.0 PROCESS PLAN

- 6.1 General Approach
- 6.1.1 The 1800 pound quantity of 1.0 UFAP will be obtained by making a composite of UFAP produced from 36 fifty-pound attritor grinds.

6.1.2 Planned utilization of the UFAP is as follows:

Material Use

Quantity Required (lbs)

300 gallon batches.

1800

- 6.1.3 The single set of faw material lots to be used for all grinds is shown in 4.0. Weighout for 50 lb grinds will be made per attritor data sheet.
- 6.1.4 Freon TF/AP dilution ration will be held constant for all grinds at 3/1.
- 6.1.5 Based on wet MSA analyses grind time will be controlled to control grind particle size.
- 6.1.6 MSA analyses on the dried and screened material will be used to select grinds to be used in the final composite.
- 6.1.7 Since the 1-gallon lot check batch of LPC-691D must be made before all the grinds have been completed, the lot check will be made using a singular typical grind which has been completely processed.
- 6.2 Attritor Set-Up
- 6.2.1 The Attritor is operated in Building 107. Both local and remote ON/OFF controls are available. City water is circulated through the Attritor jacket to maintain temperature in the grinding tank.

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- 6.2.2 Move work stand in position in front of Attritor. Lock wheels.
- 6.2.3 Remove lids from grinding tank.
- *6.2.4 Attach agitator to Attritor motor shaft. Ensure bolts are wrench tight.
- 6.2.5 Operate ON/OFF switches in both local and remote modes; ascertain that agitator revolves. Place lock-out on STOP switch in position.
- 6.2.6 Wipe grinding tank and agitator clean with Freon-wet kimwipe. Wipe dry.
- *6.2.7 From work stand, add ceramic beads which have been previously washed with hot water and dried for 48 - 72 hours at 250°F to grinding tank. Fill tank so that bead level is just above top agitator bar.
- *6.2.8 Turn on agitator in slow speed setting for 10 15 seconds to settle bead bed. Turn off agitator; replace lock-out on STOP switch. If bead bed settles below top agitator bar, add sufficient beads to reach level specified in 6.2.7.
- 6.2.9 Locate oxidizer remote addition equipment cart adjacent to work stand. Lock wheels. Attach air lines to operate remote addition equipment as follows:
- 6.2.9.1 Attach air line from airlift valve on cart to lower quick-disconnect on wall.
- 6.2.9.2 Attach air line from vibrator on hopper to middle quick-disconnect on wall.
- 6.2.9.3 Attach air line from air cylinder on hopper door to top quickdisconnect on wall.
- 6.2.10 Adjust regulator on lower air line to 20 30 psi.
- 6.2.11 Adjust pressure regulator on middle air line to 20 25 psi.
- 6.2.12 Check out remote addition equipment by operating values in both local and remote modes.
- 6.2.13 Lower oxidizer hopper to lowest level using air operated valve located on cart.
- 6.2.14 Remove oxidizer hopper lid.
- 6.2.15 From work stand or floor, fill hopper with required quantity of oxidizer.
- 6.2.16 Replace oxidizer hopper lid and tighten lid clamps.

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- 6.2.17 Unlock wheels on remote addition equipment cart and position cart adjacent to Attritor base. Relock wheels.
- 6.2.18 Using air-operated valve, raise oxidizer hopper to correct position for feeding oxidizer into Attritor. Lock sliding tube in place with pin.
- 6.2.19 Verify that bottom discharge valve on Attritor is in closed position.
- 6.2.20 Using drum pump, hand pump required gallons of Freon TF into Attritor bowl.
- 6.2.21 Add coating agent and dispersing agent diluted with one-half (1/2) gallon of Freon TF into Attritor bowl.
- 6.3 Grinding of UFAP
- 6.3.1 Start cooling water through cooling jacket by opening inlet water valve on wall; adjust flow to 2 to 4 on flowmeter.
- 6.3.1.1 The water flow through the Attritor cooling jacket is determined by the actuation of the green light on the panel of the Control Room. Check to see that green light is on; if light is not on, contact Chemist. Record in and out temperatures of cooling water as determined by thermocouples on data sheet.
- 6.3.2 Remove lock-out on STOP switch. Turn on Attritor locally in SLOW speed.
- 6.3.3 All operators go to Remote Control Room. Close doors to Building 107. Place black and yellow rope across road to Building 107.
- 6.3.4 Place key in lock-out and turn to RUN mode. Turn on speaker system.
- 6.3.5 Push FEED switch to start remote addition equipment. Run Attritor for 10 - 12 minutes.
- 6.3.6 Stop Attritor and remote addition equipment; wait 5 minutes in Control Room and then enter Building 107.
- 6.3.7 Disconnect and remove remote addition equipment from vicinity of Attritor. Lower hopper to bottom position and check to see that hopper is empty.
- 6.3.8 Check to see that NH4CIO4/Freon slurry reaches top of bead bed. If this condition does not exist, contact Chemist for instructions.
- 6.3.9 Check temperature of bead bed and record on data sheet.
- 6.3.10 Wipe off top lip of grinding chamber with Freen-wet Kimwipes. Place lids onto Attritor.

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- 6.3.11 Close doors at Building 107. All operators go to Remote Contol Room.
- 6.3.12 Turn on Attritor agitator remotely in FAST speed. Record time on data sheet.
- 6.3.13 Check Attritor at least once during each 2 to 3-hour period. Stop Attritor; wait 5 minutes in Control Room and then enter Building 107. Place lock-out in position on STOP switch. Remove lids, check temperature of bead bed and record on data sheet.
- 6.3.13.1 Record inlet and outlet cooling water temperatures at each check period on data sheet.
- 6.3.14 Wipe off top lip and lids with Freon-wet Kimwipes. Scrape down sides of grinding tank with large spatula. If NH₄/CIO₄/Freon slurry appears dry on surface, add Freon to grinding chamber. Replace lids. Remove lock-out from STOP switch. Record quantities of Freon added on data sheet.
- 6.3.15 After 3 hours of grinding, take wet sample of Freon/NH4CIO4 slurry using a disposable syringe. Take sample from area directly in front of agitator at a depth of 1" to 2". Remove needle from syringe and take sample using barrel and plunger. Clean off barrel tip, replace needle and label with grind number, sample number and time.
- 6.4 Discharge of Slurry
- 6.4.1 After grinding operation is completed as determined by Chemist using particle size analysis data, stop Attritor.

Wait 5 minutes and enter Building 107. Remove black and yellow rope from road to Building 107. Shut off cooling water to Attritor and record time on data sheet.

- 6.4.2 Remove lids from Attritor.
- 6.4.3 Remove work stand from in front of Attritor.
- 6.4.4 Place clean, dry 5-gallon size polyethylene tank under bottom-out valve. Ensure spigot on P.E. tank is closed.
- 6.4.5 Open bottom-out valve on Attritor and drain contents into P.E. tank. Fill tank 4/5 full. Close Attritor bottom-out valve.
- 6.4.6 Repeat section 6.4.4 and 6.4.5 until major contents of Attritor are discharged.
- 6.4.7 Add 5 to 10 gallons of Freon TF to bead bed as a rinse. Allow rinse Freon to discharge into a P.E. tank.

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- 6.4.8 Repeat section 6.4.7.
- 6.4.9 Remove P.E. tanks to oven area using truck.
- 6.4.10 Replace work stand in front of Attritor. Clean lids, top of agitator shaft and grinding tank top with Freon-wet Kimwipes.
- 6.4.11 Using drum pump, hand pump required gallons of Freon TF into Attritor bowl for subsequent grind. Replace lids.
- 6.5 Drying of Slurry
- 6.5.1 Prior to drying of $NH_4/CIO_4/F$ ron slurry, set two large air-circulating ovens to $100^+_{-}5^{\circ}F$ and adjust the dampers so that make-up air is kept at a minimum. This allows for a Freon-moist atmosphere during the drying operation and prevents the NH_4CIO_4 from caking.
- 6.5.2 Clean 6 each stainless steel trays with Freon wet Kimwipes.
- 6.5.3 Carefully drain contents of P.E. tanks into trays using discharge valve on tanks. Fill each tray about 5" high with slurry.
- 6.5.4 Place trays into oven. Cover each tray loosely with aluminum foil to reduce evaporation rate.
- 6.5.5 Dry contents of trays for 48 to 112 hours. Record start and stop times on data sheet.
- 6.6 Screening of UFAP
- 6.6.1 At the end of the drying cycle, remove trays from oven. Place in a covered building protected from water and allow to cool to ambient temperature.
- 6.6.2 Screening of the UFAP may be accomplished by either of two methods:
 - a) Screening by hand using flour sifter
 - b) Screening using Sweco screener in Building 102
- 6.6.3 If screening is done by hand using flour sifter, proceed as follows:
- 6.6.3.1 Make sure flour sifter is clean and dry.
- 6.6.3.2 Obtain a clean AP drum and prepare with a new polyethylene bag liner. Determine tare weight of drum with liner.
- 6.6.3.3 Accomplish the hand screening operation in either Building 120 or Building 106.
- 6.6.3.4 During the screening operation, the operator will ground himself, the scoops, the trays, flour sifter and drum.

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- 6.6.3.5 Scoop the dried UFAP from the trays and screen through flour sifter into clean, tared AP drun with polyethylene liner.
- 6.6.3.6 After the screening, sampling, and packaging of the UFAP has been completed, clean the trays, scoops, and flour sifter. Clean by using hot water and air dry.
- 6.6.4 If screening is done using Sweco screener in Building 107, proceed as follows:
- 6.6.4.1 Make sure Sweco screener is clean and dry. Assemble screener with fine screen on bottom and coarse screen on top.
- 6.6.4.2 Obtain a clean AP drum and prepare with a new polyethylene bag liner. Determine tare weight of drum with liner.
- 6.6.4.3 During the UFAP transfer operation to the screener, the operator will ground himself, the scoops, the trays, the drum, and the screener. The drum with the polyethylene bag liner must be located under the bottom discharge part of the screener.
- 6.6.4.4 Transfer one tray of the dried UFAP from trays onto top screen of Sweco screener using scoop.
- 6.6.4.5 Operate Sweco screener for the time period required for UFAP to clear the fine screen.
- 6.6.4.6 Repeat steps 6.6.4.4 and 6.6.4.5 until all the UFAP has been screened.
- 6.6.4.7 After the screening, sampling and packaging of the UFAP has been completed, clean the trays, scoops and Sweco screener. Clean using hot water and air dry.
- 6.7 Sampling and Testing of Dried UFAP
- 6.7.1 After the UFAP has been dried and screened, the UFAP shall be sampled by taking a center thief sample.
- 6.7.2 MSA particle size distribution shall be determined in duplicate on each grind. The WMD (Weight Mean Diameter) 50 percent point shall be determined from these distribution curves.
- 6.8 Packaging and Storage of UFAP
- 6.8.1 Only one grind will be packaged in a singular polyethylene bag.
- 6.8.2 The UFAP shall be packaged for storage immediately upon completion of screening and sampling operations.
- 6.8.3 Determine net weight of screened and sampled UFAP grind.

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6.8.4 Identify outside of bag with grind number, net weight, MPO, and date.

- 6.8.5 Obtain four bags (16 units) of activated, Type I, MIL-D-3464 desiccant bags. Place inside the polyethylene bag containing dried and screened AP.
- 6.8.6 Carefully squeeze air out of inside of bag. Twist top of bag and double over. Seal top of bag with tape.
- 6.8.7 Store a maximum of two packaged hags of UFAP inside a clean AP drum.
- 6.8.8 Place four bags (16 units) of activated, Type I, MIL-D-3464 desiccant bags in top of drum.
- 6.8.9 Place lid on drum and seal drum. Make sure drum is identified by material type, grind number, and MPO.
- 6.8.10 Store packaged UFAP drums in Building 112 magazines until needed for production batches.



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Exhibit #3

PROPELLANT MIX RECURD

REQUEST NO.		CHEMIST
MIX NO	MP0 763-2-60	DATE 11/7/74
OPERATORS	MIX SIZE 1000 gram	PROPELLANT TYPE HTPB
DATE MIXED	TYPE MIXER 1-gal VBP	FORMULATION 763-LPL-63-4

	MATERIAL WEIGHOUT	LOT NO.	WEIGHT PERCENT	WEIGH T GRAMS	MATERIAL CONDITIONING
1	LPL-63 Paste	74-3	74.29	742,9	
2	R-45	31088-11A	14.65	146.5	
3	HX-868	31157-11A	1,50	15,0	
4	FEPT Catalyst	65-26-2	1.00	10.0	
5	TDI	31158-11A	8,56	.85.6	
6	and a standard date and the standard date a standard standard standard standard standard standard a standard st		100,00	1,000.0	
7	40	4 0.000 · · · · · · · · · · · · · · · · ·			
8					
9					
10				1444-0 400-04-04-0-0-0-0-0-0-0-0-0-0-0-0-	
11					
12					
13				1222	The second second

	DRDER OF ADDITION	MIX TIME REQ	TIME	TIME	TOTAL	TEMP	ACT. TEMP	PRESS. MM ABS	REMARKS
1	1, 2, 3	10				90°F		20	
2	Scrape down					max			
3	4	5							
4	Scrape down								
5	5	10							
6									
7									
θ									
9									
10									
11									

VISCOSITY INSTRUCTIONS

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Exhibit #4

Pd on C -Fe(OH)₃ promoted Catalyst - Master Batch Processing

Forms catalyst with the following composition: 4.5% Pd/0.5% Pt/5.0% Fe⁺³/ 90% C. The catalyst is stored wet and is blended with the hydrogen acceptor/ C upon use.

Processing Procedure

- 1. For approximately 200 grams of the master batch material, use 5 L reaction flask.
- 2. Agitate and dissolve 144 g of NaCl in 720 g of water, at room temperature.
- 3. Add 12.18 g PdCl₂ and 2.016 g $H_2PtCl_6.6H_2O$ and stir at 30^oC until dissolved.
- 4. Add 2010 g water, 23.4 g FeCl₃, and 161.1 g Shawnigan Carbon Black. Stir for 15 minutes.
- 5. Add 432 g NaHCO₃ and 144 g water. Stir for 30 minutes at ambient temperature. Bring temperature up to 90-95°C gradually in a one-hour period, stirring vigorously and constantly. Stir vigorously for $1\frac{1}{2}$ hours at this temperature.
- 6. Rapidly cool to 60°C and filter. Wash by displacement with 570 g water and suck fairly dry.
- (NOTE): Specific catalyst used in Scavenger: 3.68% Pd/0.38% Pt/3.42% Fe/92.52% Carbon

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Exhibit #5

CELOGEN/PLASTER HOLE FILLER MATERIAL

- 1. Mix 75% Celogen and 25% Plaster of Paris in water to light paste mixture.
- 2. Spatula mixture into Teflon pellet mold.
- 3. Cure: 1 hour at ambient ($\approx 70^{\circ}$ F) (in mold) 1 hour at +160°F 2 hours at +180°F
- 4. Mix 10% solution of sodium silicate and water
- 5. Immerse mold with pellets in place in sodium silicate solution for 5±1.0 minutes
- 6. Cure: 1 hour at $+160^{\circ}$ F (in mold) 2 hours at $+180^{\circ}$ F
- 7. Tap mold to remove pellets
- 8. Immerse pellets in sodium silicate solution for 5 ± 1.0 minutes
- 9. Cure: 1 hour at $+160^{\circ}$ F (pellets 2 hours at $+180^{\circ}$ F only)

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