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HIGH TEMPERATURE, LONG SERVICE LIFE FUEL CELL BLADDER MATERIALS

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14. ABSTRACT

This report was developed under SBIR Topic AF98-196. Modern aircraft engines use recycled fuel loops as coolants to remove excessive heat from engine components. This heated fuel is returned to the bladder, exposing the fuel inlet area to fuel that has not had time to cool. The temperature of the returned fuel was initially expected to be in the range of 135°F to 160°F. In today's fighter aircraft, fuel used to support engine cooling operations can create temperature spikes in the range of 300°F to 400°F at the bladder inlet of the return loop, and prolonged exposure to temperatures between 200°F and 250°F are now quite common. Under this program, METSS clearly demonstrated the technical feasibility of using commercially available material technologies to support the development of high temperature resistant fuel bladders that can be readily integrated into the existing fuel bladder manufacturing processes. Under the Phase II program, METSS identified two specific rubber materials based on hydrogenated nitrile rubber and epichlorohydrin that meet the criteria needed for a fuel cell bladder operating at 225°F. Of these, the hydrogenated nitrile rubber compound was particularly adaptable to the manufacture of fuel bladders. This relationship will establish a solid foundation for Phase III product commercialization efforts, providing a viable path for rapid technology transfer and implementation.

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EXECUTIVE SUMMARY

Existing systems have performed exceptionally for over 50 years; however they were not designed to withstand the temperature demands imposed by today's high performance aircraft. The current bladder systems are qualified only to temperatures of 135°F. In today's fighter aircraft, fuel used to support engine cooling operations can create temperature spikes in the range of 300ºF to 400°F at the bladder inlet of the return loop, and prolonged exposure to temperatures between 200ºF and 250°F are now quite common. This has created a problem in some aircraft systems, leading to fuel bladder failure and reduced service life.

The existing high temperature performance deficiencies have created a need for a new generation of aircraft fuel bladders. This program addresses this need by systematically screening and testing candidate material technologies to identify classes of rubber materials that are capable of sustaining service in high temperature fuel bladder applications. Specifically, this report identifies specific rubber compounds that are capable of long-term service at 225°F in JP8+100 and further demonstrates these materials can be used to fabricate high performance inner liner materials for high temperature fuel bladder applications.

The results of this program demonstrate several classes of rubber that are suitable for high temperature fuel cell applications with an upper used temperature of at least 225°F, specifically in the presence of JP8+100. Of these materials, hydrogenated nitrile rubber (HNBR) and epichlorohydrin (ECO) represent the most cost effective classes of materials demonstrating high temperature resistance to JP8+100. The performance of specific formulations based on each of these systems was demonstrated against the program requirements and the applicable military performance specifications for fuel cell materials. The primary difference between the ECO and the HNBR inner liner materials developed under this program is low temperature performance capability; the HNBR systems demonstrated low temperature flexibility down to -65°F while the ECO materials demonstrated marginal performance, even at -40°F.

The inner liner materials developed under this program can be provided as ready to calendar gums or fully calendared (but uncured) inner liner materials to support full qualification trails and manufacturing efforts.

1.0 INTRODUCTION

1.1 Background

Fuel bladders have been used in aircraft since the 1940's. These bladders are designed to fit into various open spaces within the aircraft to contain volatile fuels and provide an added level of protection to the aircrew and ground personnel (especially in enclosed environments, e.g., hangars, ships). Essentially a collapsible rubber structure, the bladders are inserted into confined spaces within the airframe where they are expanded, suspended or otherwise supported, and connected to the fuel system. The bladders contain the liquid fuel and prevent fuel fumes from escaping.

There are three types of fuel bladders that are commonly used to contain fuel in fixed wing aircraft and helicopters:

- Bladder Tank - used extensively as supported tanks in military, general aviation, and commercial aircraft.
- Self-Sealing Tank - used in military and law enforcement aircraft; designed to temporarily seal off leaks when small munitions puncture tank.
- Crash-Resistant Self-Sealing Tank - used in military combat aircraft (and Indy race cars); constructed to provide safety by eliminating post-crash fires.

Applicable Military Specifications include:

- MIL-T-6396E - Tanks, Aircraft Propulsion Fluid System, Internal, Removable, Non-Self-Sealing
- MIL-T-5578C - Tank, Fuel, Aircraft, Self-Sealing
- MIL-T-27422B - Tank, Fuel, Crash & Resistant, Aircraft.

The primary function of each of these bladders is to contain fuel and fuel vapors. The ability of the bladder to perform in this regard is dictated by the inner ply of a composite structure, which consists of a fabric reinforced rubber layer for mechanical strength and structure, and a barrier material to contain the fuel vapors. Additional materials, laminated to the inner liner, impart secondary

characteristics such as self-sealing and crash resistance and provide additional structural stability for strength and support. Thus, successful integration of high temperature/fuel resistant materials into the design of inner liner will support the development of high temperature resistant fuel bladders for a number of existing aircraft applications.

1.2 Current Bladder Designs and Materials

Fuel bladders are complicated structures, which are difficult to install into the aircraft and generally require depot level maintenance if leakage occurs. While it was originally planned to have fuel bladders remain in service for the life of the airframe, nitrile rubber bladders typically have a service life of about 7-9 years and urethane systems have service lives of about 10 years. The major problem with current fuel bladders is unscheduled depot maintenance attributed to the reduced service life of the bladder, presumably brought about by high temperature degradation processes or accelerated aging. Many bladder systems are presently failing after less than three years of service. Pin holing, blistering, and de-bonding are the primary failure modes being observed. The internal pressures of the bladder vary between 2-5 psi so the bladder appears to be under little physical stress that could otherwise accelerate the degradation process. Therefore, the failures are primarily being caused by material incompatibility problems with the high temperature fuels.

Current fuel bladder technology is based on the use of polyurethane and nitrile rubber chemistry. Nitrile rubbers are copolymers of isobutylene and acrylonitrile. These materials have been available since the 1950's and were specifically developed for use in fuel bladders. The nitrile rubber systems represent complex mixtures of curatives, reinforcing fillers and tackifiers that are cured in an autoclave to form a fuel resistant inner liner material. Most of the older bladder systems in use are constructed of nitrile rubbers. Inner liners based on nitrile systems are typically fabricated using lay-up methods from fabric-reinforced sheets of calendared material. While typically more robust than their urethane counterparts, the lay up process makes them more difficult and more expensive to fabricate and necessitates the use of seams that could act as failure initiation sites. Furthermore, the sensitivity of the current nitrile rubber systems to weathering and ozone attack necessitates strict maintenance requirements for bladders removed in the field (e.g., oiling and storage if removed for extended periods of time).

A typical composite wall of a nitrile rubber fuel tank is composed of the following:

- Calendared, nylon fabric reinforced nitrile rubber inner liner next to fuel
- Tie cement to facilitate inner liner bonding to barrier material
- Liquid applied nylon barrier material applied to restrict fuel vapor transport
- Tie cement to facilitate bonding barrier material to outer plies
- Outer plies - may be a combination of laminated materials for crash-resistant and self-sealing bladders, or another fabric reinforced nitrile rubber layer for Type II, Class A tanks.

Urethane chemistries were adapted to fuel bladder use in the early 1980's, their high toughness and abrasion resistance making them an excellent counterpart to the nitrile rubber systems. The use of liquid urethane chemistry is highly desirable as it facilitates the use of spray-up processes for fuel bladder construction, significantly reducing the cost of fuel bladder manufacture (less labor intensive; air cured systems eliminated the need for autoclave curing) and eliminating the presence of seams in the bladder's construction. Urethane bladders are also lighter and tougher than their existing nitrile rubber counterparts and less susceptible to ozone attack and weathering (inherently more UV resistant).

A typical composite wall of a sprayed polyurethane fuel tank is composed of the following:

- Liquid urethane sprayed onto bladder mold, air cured (urethane next to fuel)
- Liquid applied nylon barrier material applied to restrict fuel vapor transport
- Liquid urethane overcoat sprayed onto nylon barrier
- Polyester fabric reinforcement layer (solvent bonded to cured urethane substrate)
- Liquid urethane overcoat sprayed onto polyester fabric (Type II, Class A bladder)
- Additional outer plies - a combination of laminated materials for crash-resistant and self-sealing bladders; a tie layer is typically required.

While the existing systems have performed exceptionally for over 50 years, they were not designed to withstand the temperature demands imposed by today's high performance aircraft. The current bladder systems are qualified only to temperatures of 135°F. In today's fighter aircraft, fuel used to support engine cooling operations can create temperature spikes in the range of 300ºF to 400°F at the

bladder inlet of the return loop, and prolonged exposure to temperatures between 200ºF and 250°F are now quite common. This has created a problem in some aircraft systems, leading to fuel bladder failure and reduced service life. The existing high temperature performance deficiencies have created a need for a new generation of aircraft fuel bladders.

1.3 The Need for New Materials

The basic problem is that the current fuel bladders were designed to operate at fuel temperatures of 160°F. Modern aircraft engines use recycled fuel loops as coolants to remove excessive heat from engine components. This heated fuel is returned to the bladder, exposing the fuel inlet area to fuel that has not had time to cool. The temperature of the returned fuel was initially expected to be in the range of 135ºF to 160°F. In today's fighter aircraft, fuel used to support engine-cooling operations can create temperature spikes in the range of 300ºF to 400°F at the bladder inlet of the return loop, and prolonged exposure to temperatures between 200ºF and 250°F are now quite common.

Recent testing with JP-8 fuel showed that, after 28 days at 200°F, bladder inner-liner materials were severely degraded. Both the nitrile rubber and urethane technologies that are currently used passed at 160°F, but failed at 200°F. As a consequence, the service life of current fuel cell bladders is too short to handle aging aircraft requirements. Bladder service life on some aircraft is only 4-5 years, with some failures after 2 years.

As a consequence, more durable material is needed to help extend the service life and avoid added maintenance costs. This program addresses this need by systematically screening and testing candidate material technologies to identify classes of rubber materials that are capable of sustaining service in high temperature fuel bladder applications. Specifically, this report identifies specific rubber compounds that are capable of long-term service at 225°F in JP8+100 and further demonstrates these materials can be used to fabricate high performance inner liner materials for high temperature fuel bladder applications.

2.0 CANDIDATE MATERIALS

2.1 Commercial Materials

An extensive effort was conducted to identify candidate material technologies and suppliers to support the program efforts. Methods of identification included literature and patent searches, discussions with rubber and raw material suppliers, rubber compounders, and a search for available information on the Internet. Based on the results of the Phase I program efforts, the work conducted under the Phase II program emphasized the identification and evaluation of suitable candidates based on the following materials:

- ***Nitrile Rubbers and Highly Saturated Nitrile Rubbers*** – Nitrile rubbers (NBR) demonstrate excellent resistance to hydrocarbons. However conventional nitrile rubbers offer limited high temperature performance and must be heavily plasticized to achieve good low temperature performance. Saturated (hydrogenated) nitrile rubbers (HNBR) offer exceptional performance characteristics and superior thermal-oxidative stability over a much broader temperature range. Originally intended to be an extension of standard nitrile rubbers with higher oxidation resistance, these materials are competing with fluorinated materials for high temperature and severe service environments. Several commercially available formulations provide excellent high temperature resistance and low temperature performance through specific modification of the precursor materials and specialty compounding. These materials offer other favorable characteristics, including good tensile properties, wear resistance, and durability. Commercially available materials claim service performance over a temperature range of -65 to 350°F.
- ***Epichlorohydrin Rubbers*** – Epichlorohydrin rubber materials have been commercially available since the mid 1960's and, due to the presence of oxygen in their backbone, exhibit excellent chemical resistance to hydrocarbons. Recent refinements of these materials have produced materials with increased low temperature flexibility.
- ***Fluoroelastomers*** – Fluoroelastomers are known for their chemical resistance and would be an ideal fuel bladder candidate for the present application if their cost structure were not so prohibitive. Under the Phase II program particular emphasis was placed on evaluating new advancements in fluoroelastomer materials, including a new class of perfluoroethers that

offer exceptional low temperature and high temperature performance, excellent chemical resistance, and good mechanical properties.

- ***Other materials*** – Other elastomers were also included in the testing program to provide a thorough evaluation of the major classes of rubbers. These other materials included fluorosilicones, high acrylonitrile content nitriles, linear and crosslinked polyurethanes, and thermoplastic elastomers (primarily polypropylene and EPDM blends).

At the beginning of the Phase II program, 60 rubber compounds from various suppliers were identified for possible consideration under the program. Samples of all of these materials were obtained and tested under the program. The Phase II candidate materials are summarized by general materials type in Table 1.[1] A quick evaluation of this list produces the following summary of test materials based on general material classification:

Material Classification	# of Materials
• Nitrile Rubbers (NBR)	12
• Hydrogenated Nitrile Rubbers (HNBR)	10
• Epichlorohydrin Rubbers (ECO)	5
• Fluoroelastomers (FKM)	14
• Perfluoroethers (PFE)	4
• Fluorosilicones (FMVQ)	2
• Polyurethanes (PUR)	4
• Thermoplastic Elastomers/Urethanes (TPE/TPU)	6

During the course of the program, each of the materials suppliers was provided information on the performance of their materials after testing and evaluation against the stated performance criteria. Willing suppliers were allowed to reformulate and resubmit samples for further consideration. Several of the program suppliers were very active participants in the Phase II program, submitting multiple formulation iterations or material advancements to support the program efforts.

Generally, the samples materials evaluated under the program were provided to METSS as cured plaques of compression molded rubber to support the initial testing and evaluation portion of the

[1] Specific materials information has been provided to the Air Force, including suppliers and product codes. Generic material descriptions are used in this report for reasons of supplier confidentiality.

program. However, more advanced or modified formulations were typically compounded from raw materials and molded in-house to support the program efforts.

2.2 Liquid Systems

In addition to laying up calendared sheets on bladder pre-forms, fuel cell bladders can also made by spraying rubber solutions over discardable pre-forms, with fabric layers and barrier coatings interspersed among the sprayed layers of rubber to impart the requisite physical and functional properties to the bladder. Currently, the rubber used in the production of bladders made by this method is a polyurethane material that lacks the high temperature fuel resistance desired by the Air Force. As sprayed bladders offer advantages over calendared systems including reduced weight and a continuous layer of cured rubber, a portion of the program efforts were directed at evaluating alternative liquid based rubber systems and developing new liquid formulations for testing and evaluation under the program for high temperature fuel cell application.

The primary objective of this effort was to identify or produce a low VOC with resistance to JP8+100 at temperatures up to 225°F. In pursuit of this, METSS investigated a number of commercially available liquid sprayable systems as well as a number of formulations and blends developed in-house. Both solvent-based and water-based formulations were considered, however strong emphasis was placed on the use of environmentally friendly solvents and low VOC formulations.

Eight manufacturers supplied METSS with 17 different commercially available liquid elastomer based compositions or elastomeric materials that could be dissolved in a liquid carrier. Many of the commercial materials were modified or blended by METSS with other materials during the course of the formulation development efforts in an effort to improve their physical/chemical properties and film forming capability. In other efforts, formulations based on HNBRs or carboxylated NBRs blended with an epoxy-terminated polysulfide were developed by METSS to improve the various properties of the nitrile rubbers.

A list of the 17 liquid systems evaluated under the Phase II program or used to support in-house formulation development efforts is provided in Table 2. A quick evaluation of this list produces the following summary of test materials based on general material classification:

Material Classification	# of Materials
• Polyurethane (PU)	2
• Hydrogenated Nitrile Rubbers (HNBR)	3
• Carboxylated Nitrile Rubbers (CNBR)	2
• Nitrile Rubbers (NBR)	1
• Fluoroelastomers (FKM)	9

Test samples for sprayable systems were cut from cast or sprayed, dried and cured films of the candidate liquid spray systems. Multiple castings were typically required to produce samples of suitable thickness for characterization. A professional-quality, automotive spray gun (Devilbiss) and air compressor was used to support spraying trials. The specific formulation development efforts related to the liquid systems are presented and discussed in Section 4.2, along with the presentation of the test results related to these development efforts.

2.3 Inner Liner Materials

Inner liner materials were prepared for testing and evaluation under the program using rubber compounds demonstrating compliance with existing military performance specifications and high temperature stability to JP8+100. The inner liner is the portion of the fuel cell that must be resistant to JP8+100 jet fuel at temperatures up to 225°F. During fuel cell manufacturing (for calendared systems), the inner liner laminate is placed over a plaster form and coated with a barrier coating of nylon 11 in an ethanol-toluene solution produced by Nycote Laboratories under the trade name Nycote® 7-11. The subsequent structural and functional (crash resistance, self-sealing) of the fuel cell are then built up upon the inner layer to create the final bladder structure per the manufacturer's design specifications.

The inner liners constructed under the program were made by a calendaring process in which uncured molten rubber was laminated to and adhered to both sides of a nylon reinforcing fabric through contact in the nip of a pair of calendaring rolls. The nylon fabric used in the construction of the inner liner had a density of 3.8 oz/yd^2, made from 210 denier fibers in a 78 by 61 plain weave construction. The nylon fabric was treated with resorcinol formaldehyde latex to promote adhesion to the rubber prior to the calendaring process. During processing, the upper roll of the calendaring press was held at 120°F, while the bottom one was held at 105°F. The laminates were cured into 0.080 inch thick sections in a press at 350°F to support the inner liner testing and evaluation portions of the program.

All of the calendaring under the Phase II program was performed for METSS at Akron Rubber Development Labs (ARDL) using material formulations provided by METSS.

2.4 Fuel Cell Construction

Fuel cell designs are proprietary to the commercial fuel cell manufacturers. As such, commercial fuel cell manufacturers were contacted to construct additional samples needed to support composite wall testing and fuel cell testing.

Table 1. Candidate Materials

Material ID	Material Type	Material ID	Material Type
1	Fluoroelastomer (FKM)	31	Fluorosilicone (FMVQ)
2	Fluoroelastomer (FKM)	32	Hydrogenated Nitrile (HNBR)
3	Fluoroelastomer (FKM)	33	Polyurethane (PUR)
4	Polyurethane (PUR)	34	Hydrogenated Nitrile (HNBR)
5	Fluoroelastomer (FKM)	35	Hydrogenated Nitrile (HNBR)
6	Fluoroelastomer (FKM)	36	Hydrogenated Nitrile (HNBR)
7	Epichlorohydrin (ECO)	37	Hydrogenated Nitrile (HNBR)
8	Hydrogenated Nitrile (HNBR)	38	Nitrile (NBR)
9	Hydrogenated Nitrile (HNBR)	39	Fluoroelastomer (FKM)
10	Fluorosilicone (FMVQ)	40	Fluoroelastomer (FKM)
11	Fluoroelastomer (FKM)	41	Fluoroelastomer (FKM)
12	Fluoroelastomer (FKM)	42	Thermoplastic Elastomer (TPE)
13	Perfluoroether (PFE)	43	Thermoplastic Elastomer (TPE)
14	Hydrogenated Nitrile (HNBR)	44	Thermoplastic Elastomer (TPE)
15	Fluoroelastomer (FKM)	45	Polyurethane (PUR)
16	Fluoroelastomer (FKM)	46	Polyurethane (PUR)
17	Epichlorohydrin (ECO)	47	Epichlorohydrin (ECO)
18	Perfluoroether (PFE)	48	Nitrile (NBR)
19	Perfluoroether (PFE)	49	Nitrile (NBR)
20	Nitrile (NBR)	50	Thermoplastic Elastomer (TPE)
21	Perfluoroether (PFE)	51	Thermoplastic Elastomer (TPE)
22	Nitrile (NBR)	52	Thermoplastic Elastomer (TPE)
23	Fluoroelastomer (FKM)	53	Thermoplastic Polyurethane (TPU)
24	Nitrile (NBR)	54	Thermoplastic Polyurethane (TPU)
25	Nitrile (NBR)	55	Thermoplastic Polyurethane (TPU)
26	Hydrogenated Nitrile (HNBR)	56	Thermoplastic Elastomer (TPE)
27	Epichlorohydrin (ECO)	57	Epichlorohydrin (ECO)
28	Hydrogenated Nitrile (HNBR)	58	Nitrile (NBR)
29	Nitrile (NBR)	59	Nitrile (NBR)
30	Nitrile (NBR)	60	Fluoroelastomer (FKM)

Table 2. Sprayable Formulations

Material ID	Material Type
S-1	Fluoroelastomer (FKM)
S-2	Fluoroelastomer (FKM)
S-3	Fluoroelastomer (FKM)
S-4	Fluoroelastomer (FKM)
S-5	Fluoroelastomer (FKM)
S-6	Hydrogenated Nitrile (HNBR)
S-7	Polyurethane (PUR)
S-8	Polyurethane (PUR)
S-9	Fluoroelastomer (FKM)
S-10	Fluoroelastomer (FKM)
S-11	Fluoroelastomer (FKM)
S-12	Fluoroelastomer (FKM)
S-13	Hydrogenated Nitrile (HNBR)
S-14	Hydrogenated Nitrile (HNBR)
S-15	Nitrile (NBR)
S-16	Carboxylated Nitrile (CNBR)
S-17	Carboxylated Nitrile (CNBR)

3.0 EXPERIMENTAL

The experimental portion of the program was extensive, covering multiple exposures to JP8+100 for 3 and 28 days at temperatures of 200°F and 225°F. Experimental methods are described in this section. Experimental results and discussion are presented in Section 4.0. The experimental efforts were conducted using a tiered approach so poor performing materials could be identified early during the course of the experimental work using simple test methods, saving more extensive testing and evaluation for the best performing materials. Multiple sets of tests were conducted on the best performing materials to verify performance and validate program results.

3.1 Testing Sequence

The test methods that were employed to support the Phase II program efforts were selected after carefully considering the following:

- The objectives of the Phase II program
- The design requirements for high temperature resistant fuel bladder materials
- The current manufacturing processes for constructing aircraft fuel bladders
- Testing needed to demonstrate compliance with military performance specifications.

METSS selected a number of standardized tests to support the Phase II program testing and evaluation efforts and defined the applicable military performance testing needed to qualify new fuel bladder inner liner materials developed for integration into MIL-T-6396E, MIL-T-5578C, and MIL-T-27422B bladders. A summary of the standardized test methods used to support the program efforts is provided in the following bulleted list along with a brief notation on their use and relevance. Details of the actual testing performed under the Phase II program are presented in the remainder of this section.

- ASTM D471: *Test Method for Rubber Property - Effects of Liquids*
    - general procedure for sample immersion
- ASTM D2240: *Rubber Property - Durometer Hardness*
    - Shore A rubber hardness measurement
- ASTM D412: *Rubber Properties in Tension*

    - general procedure for tensile testing rubber materials
- ASTM D751: *Standard Test Methods for Coated Fabrics*
    - general procedure for tensile testing coated fabrics (inner liner materials)
- ASTM D624*: Standard Test Method for Tear Strength of Conventional Vulcanized Rubber and Thermoplastic Elastomers*
    - general test method for evaluating tear resistance of rubbers
- ASTM D2231: *Rubber Properties in Forced Vibration (DMA)*
    - low temperature performance/overall elasticity
- ASTM D413: *Rubber Property - Adhesion to Flexible Substrates*
    - general test method for evaluating bonding between rubber and fabric or barrier
- ASTM D2137: *Rubber Property - Brittleness Point of Flexible Polymers and Coated Fabrics*
    - low temperature performance of fabric reinforced rubber

Fuel bladder test methods outlined in military performance specifications MIL-T-6396E and MIL-T-27422B selected to evaluate the candidate inner liner materials are presented in Table 3. As noted, there is a high degree of overlap in the target performance specifications, especially with regard to the inner liner materials.

The testing and evaluation efforts were divided by level of difficulty and applicability to each phase of the materials development effort, establishing the basis for the testing sequence that was followed during the course of the program to screen, evaluate and qualify new inner liner materials for high temperature fuel bladder application. The final test sequence selected to support the Phase II program development efforts is presented in Table 4.

3.2 Tier I - Screening Tests

Screening studies were performed using test samples that were die-cut from cured rubber samples to characterize the resistance of candidate materials to high temperature JP8+100. The use of die-cut samples allowed more materials to be evaluated under the program in a time efficient manner. In the beginning of the program all of the plaques used to support the program efforts were prepared by the material providers for optimum performance. The data generated on samples cut from the molded plaques provided a solid basis for selecting candidate materials for further program consideration and progression to more advanced testing. As the program progressed METSS assumed responsibility for

sample preparation and modification. Actual compounding was performed at ARDL under the direction METSS.

High temperature resistance to jet fuel was determined by aging test samples in accordance with ASTM D 471: *Test Method for Rubber Property - Effects of Liquids*. Fluid aging experiments were performed in friction air ovens for 3 day and 28 day periods with samples fully immersed in JP8+100 at 200ºF and 225ºF. Test temperatures were maintained within ±3°F for the duration of the high temperature fluid aging experiments. Individual or replicate samples of the same material were aged in individual vessels to eliminate the possibility of cross-contamination between materials. Care was taken to make sure replicate samples did not touch one another during immersion, thereby ensuring full contact of all of the fuel with surfaces of the sample. Sample vials were sealed with Teflon® lined lids to prevent fuel loss and possible contamination from lid/liner materials. All test measurements performed on fluid aged samples were performed after excess fluid was removed from the samples and the samples were allowed to cool to room temperature.

Volume swell, weight gain and hardness change measurements were performed on all of the candidate test materials after high temperature fluid aging. Initial screening experiments were conducted using ¾-inch diameter samples that were die-cut from cured sheets of the candidate test materials. After initial characterization (weight, hardness and dimensional volume), replicate samples (3 for each test) were immersed in separate 2 ounce vials of JP8+100 and placed in preheated friction air ovens for 3 and 28 days. A glass marble was placed in the bottom of the vial so the test sample would rest in an upright position to maximize fluid exposure. Hardness measurements were performed in accordance with ASTM D 2240: *Test Method for Rubber Property - Durometer Hardness*. Due to the thickness of the test specimens, replicate samples had to be stacked (as allowable under the test method) to support accurate hardness determination. All hardness measurements were performed using a Gardner Shore A hardness tester and test stand. Hardness readings were taken immediately after full contact between the tester and sample.

3.3 Tier II – Property Screening

Tier II testing consisted of characterizing the physical and mechanical properties of the best performing rubber materials before and after aging in JP8+100 at 225°F. Unless otherwise noted, the high temperature fluid aging experiments were performed using the same methods outlined in Section 3.2. At the end of this test sequence, nonvolatile and stoved gum residue testing were performed on

the best performing rubber inner liner materials to ensure compliance with performance specifications prior to construction and more advanced testing of inner liner barrier materials.

Existing performance specifications for full bladder materials require that the inner liner gum not lose more than half of their initial tensile strength (50% retention) and elongation at break after three days aging in the hot jet fuel. Tensile property measurements were performed in accordance with ASTM D 412: *Standard Test Methods for Rubber Properties in Tension*, using Type C dumbbell specimens (5 replicates per test condition) that were die-cut from the cured rubber plaques. Tensile property testing was performed on as-received materials as well as tensile specimens that were fluid aged for 3 and 28 days in the JP8+100. During fluid aging, tensile test specimens were fixed vertically on a rack and placed in 1-quart jars containing the appropriate test fluid; care was taken to make sure test specimens were separated during aging. Reported results include tensile strength (psi) and elongation at break (%) for un-aged samples, and change (%) in tensile strength and elongation at break for aged samples. All tensile property measurements were performed at a constant cross-head displacement of 2 inches per minute using a Tinius Olsen 5000 universal testing machine. Elongation measurements reported in this document reflect crosshead displacement and not actual specimen strain data.

The tear strength of the candidate rubber materials was determined in accordance with methods outlined in ASTM D624: *Standard Test Method for Tear Strength of Conventional Vulcanized Rubber and Thermoplastic Elastomers*, before and after aging in JP8+100 at 225°F for 3 and 28 days. All testing was performed on a Tinius Olsen 5000 universal testing machine using type C tear specimens (5 replicates per test condition) die-cut from the cured rubber plaques and an extension rate of 2 inches per minute. The maximum allowable tear strength reduction is 50% after 3 days of high temperature fuel aging.

Dynamic Mechanical Analysis (DMA) experiments were performed on candidate materials, before and after aging in JP8+100, to characterize low temperature mobility and define low temperature transitions.[2] Materials used in fuel cell applications must maintain low temperature performance down to -40°F (for JP8+100). These goals are not tied to a parameter, such as T_g or T_o, determined by DMA, but to a brittleness test discussed in Section 3.4. However, the use of DMA provides a useful means of screening materials for low temperature flexibility. DMA test samples measured ½-inch wide by 3-inches long; sample thickness was dependent on the thickness of the test plaques provided

[2] ASTM D 2231: *Standard Practice for Rubber Properties in Forced Vibration*

by the material suppliers, which were nominally 0.08 inches.[3] Fluid aged samples were aged in 4 oz jars. Two test samples (one each for 3 and 28 day aging) were aged in each jar, using a stainless steel wire spacer to separate samples and hold them in a vertical position during aging. All experiments were performed using a TA Instruments DMA 983 equipped with a liquid nitrogen cooling accessory (LNCA). DMA experiments were conducted in dual cantilever mode, with a grip spacing of 45 mm and 7 in-lb clamping force holding the sample. All DMA experiments were conducted at 1 Hz, scanning a 5°C/min from -100ºC to 50ºC.

DMA measures the dynamic modulus of materials over a range of temperatures, providing a quick and easy method to generate information that can be used to evaluate low temperature performance. The existence of low temperature transitions can be related directly to low temperature flexibility, mechanical hysteresis, and resilience. Departure from the high modulus behavior exhibited by materials below their glass transition temperature to the rubbery plateau modulus characteristic of elastomers occurs over a range of temperature, with the glass transition temperature (T_g) being the mid-point in this transition. The onset of the glass transition region is associated with the transformation from brittle-to-ductile behavior when examining a material that is heated from a low temperature to a high temperature, and, therefore, provides a measure of the material's ability to function adequately at a given operational temperature. The use of this temperature as an indicator of low temperature performance in rubbers has been substantiated by Thomas.[4] Thomas showed that the low temperature sealing ability of a variety of fluoroelastomers was maintained down to approximately 25°F below the glass transition temperature. METSS subsequently established that this difference represented the differential between T_g and the onset of T_g (designated as T_o).

Additional testing included at the end of the Tier II testing and evaluation sequence included stoved and nonvolatile gum residue testing to ensure compliance with the existing military performance specifications for fuel cell bladders. These tests were included to ensure materials that have excessive amounts of extractables or dissolved matter (which may not show up in the D471 experiments – Section 3.2 – due to fuel retention) are eliminated from consideration. Stoved and Nonvolatile Gum Residue tests were conducted in accordance with methods outlined in ASTM D-381: *Standard Test Method for Gum Content in Fuels by Jet Evaporation*. According to MIL-T-6396 and MIL-T-27422, Stoved Gum Residue of bladder materials should be less than 20 mg/100 ml, and the Nonvolatile Gum Residue should be less than 60 mg/100 ml. These tests were performed by Texas Oiltech Labs.

[3] The actual dimensions (average of three per dimension) were determined for each sample tested and the data were input in the DMA analysis software for proper data analysis.
[4] E. W. Thomas, SAE Technical Paper 2001-01-2974, *"Fluoroelastomer Compatibility with Advanced Jet Engine Oils"*

3.4 Tier III – Fabric Inner Liner Testing

Fabric reinforced inner samples were prepared by ARDL from candidate rubber materials demonstrating the best overall resistance to high temperature aging in JP8+100 and subjected to more advanced testing. All inner liner materials were prepared by ARDL using methods outlined in Section 2.3.

Fabric inner liner strength was determined in accordance with methods outlined in Federal Test Method Standard No. 191, Method 5100. All samples were tested before and after 3 and 28-day high temperature fuel aging in JP8+100 at 225°F. The military performance specifications provide for a maximum change in fabric inner liner strength of 20% change after 3 days of fuel aging.

The tensile properties and hardness of the fabric reinforced inner liner materials were determined after high temperature humidity exposure for a period of 30 days at 95% RH and 160°F. The humidity exposure conditions were taken from the military performance specification for fuel cell bladders. The tensile properties were determined using methods provided in ASTM D751: *Standard Test Methods for Coated Fabrics*. Allowable property changes after high temperature humidity exposure are ±45% tensile, ±30% elongation, and ±15 points hardness.

Seam strength testing was performed according to Federal Test Method Standard No. 601, Method 8011 to determine the ability of the inner liner materials to bond to themselves, a basic requirement for bladders manufactured by hand lay-up. Seam strength was determined before and after 3 day and 28 day immersion in JP8+100 at 225ºF. The performance specifications require no less than 6 lbs/inch seam strength or that tension failures of samples containing seams perpendicular to the direction of load occur in the rubber and not at the seam. Samples made for this test were produced by compression molding an uncured layer of calendared rubber against a cured layer in a mold with a 7 mil thick sheet of Mylar® film inserted between the two layers to a distance of 1-inch. This provided a means for separating and gripping two layers of the rubber in order to measure the strength developed at the interface between the two layers.

The adhesion strength of the calendared rubbers to the nylon fabric was also tested before and after 3 day and 28 day immersion in JP8+100 at 225ºF, using methods outlined in ASTM D 413: *Adhesion to Flexible Substrates (Rubber to Fabric).* The performance specifications require no less than 6 lbs/inch adhesion strength between the rubber gum and inner liner fabric. Samples made for this test were

produced by separating the nylon fabric from one of the uncured layers of calendared rubber produced during the calendaring process and placing a 7 mil thick sheet of Mylar® film between the fabric and the gum to create a 1-inch wide space where the fabric was prevented from bonding to the separated rubber during the subsequent compression molding step. This provided a means for separating and gripping the rubber and pulling it away from the fabric inner line so the adhesion strength could be determined after molding.

The low temperature flexibility of the fabric reinforced inner liner was determined using methods outlined in ASTM D2137: *Standard Test Methods for Rubber Property-Brittleness Point of Flexible Polymers and Coated Fabrics*. This test essentially entails striking cantilever samples cooled to -65°F and -40°F and examining the samples for evidence of delaminating or cracking. The results of this test are reported on a pass/fail basis.

3.5 Tier IV – Testing of Inner Liner with Barrier Material

As described in Section 2.3, during fuel cell manufacturing the fabric reinforced inner liner is typically coated with a coating of nylon 11 (to create a barrier to fuel vapor) before the structural and functional layers of the fuel cell are added to the structure. The adhesion strength of the inner liner adhesion to the nylon barrier film is a critical aspect of the bladder design. Thus, additional adhesion testing was performed in accordance with ASTM D413 to determine the degree of bonding between the inner liner materials and the nylon barrier film. The minimum permissible bonding strength is 6 lbs/inch. The procedure used to produce the test samples was to place a 7 mil film of Mylar® along the edge of a sheet of rubber. The nylon barrier material was coated over the rubber and Mylar® film. When dried, the Mylar® film could be lifted, thereby providing a means for separating the barrier coating from the rubber substrate so the adhesion strength can be determined.

The vapor barrier properties of the inner liner materials were determined using permeation cup measurements. According the performance specifications, the room temperature permeation rate of JP8+100 should be less than 0.025-fl.oz./ft²/day for each of the replicates tested (this is a pass/fail test). METSS performed permeation measurements on each of the candidate materials with and without the nylon barrier film to see if any of the candidate materials demonstrated inherent resistance to fuel vapor permeation. If so, the use of the nylon barrier film may be eliminated during manufacturing, thereby reducing processing time and cost. The permeation tests were performed for a period of eight days at 70°F and 50-60% relative humidity.

Stress aging testing of the fabric reinforced inner liner materials (with barrier coating) was conducted to support MIL-T-24722B qualification. This test involves exposing folded samples to JP8+100 for 7 days at 160°F and examining samples for evidence of blistering, cracking, separation, or other material failure. This is a Pass/Fail test, with no evidence of degradation of the samples permitted after 7 days of aging.

3.6 Tier V – Composite Wall Testing

Once the inner liner materials are qualified, the remaining structure of the fuel cell remains unchanged, so one may determine that substitution of the new high temperature resistant inner liner materials into existing fuel cell designs could be qualified by inference. The same goes for the fully constructed fuel cell. However, to verify this, METSS arranged additional testing to be supported by the fuel cell manufacturing community.

In support of this effort, METSS provided a number of samples of both inner liner materials and compounded rubber gums to fuel cell manufacturers to support sample preparation efforts and testing and evaluation efforts. Testing and evaluation efforts were to include testing by the fuel cell manufacturers as well as METSS. The test jigs required to support internal testing efforts were constructed by METSS in accordance with the guidelines provided in the military performance specifications.

After preparing and supplying numerous samples to support the construction of composite wall samples for internal and external testing and evaluation, METSS was finally forced to suspend all efforts related to this task due to lack of focused support from the fuel cell manufacturing community and sufficient program funding to continue to support the materials development efforts. A description of the materials METSS supplied to support this effort is provided in Section 4.6.

3.7 Tier VI – Fuel Cell Testing

Fuel cell testing was suspended for the same reasons provided in Section 3.6. However, the effect of changing the inner liner material on fuel cell performance is anticipated to be negligible, provided the inner line material demonstrates high temperature resistance to JP8+100.

Table 3. Test References for MIL-T-6396E and MIL-T-27422B

Test Description	Section Reference	
	MIL-T-6396E	MIL-T-27422B
Nonvolatile Gum Residue	4.6.13.1	4.6.4.1
Stoved Gum Residue	4.6.13.2	4.6.4.2
Gum Inner Liner Strength (a)	4.6.15.1	4.6.4.3
Fabric Inner Liner Strength (b)	4.6.15.2	4.6.4.4
Seam Adhesion (b)	4.6.16	4.6.4.6
Humidity	4.6.10	NR (c)
Permeability	4.6.12	4.6.4.5
Puncture Resistance	4.6.17	NR
Fluid Resistance of Exterior Surface	4.6.11	4.6.6.1
Leakage	4.6.1.5	NR
Slosh Resistance	4.6.8	4.6.6.3
Stand Test	4.6.9	4.6.6.6
Additional Tests Required to Support MI-T-27422 B Phase I Pre-production Testing		
Slit Resistance	NR	4.6.4.7
Inner Liner Adhesion to Sealant	NR	4.6.4.8
Stress Aging	NR	4.6.4.9
Constant Rate Tear	NR	4.6.5.1
Impact Penetration	NR	4.6.5.2
Impact Tear	NR	4.6.5.3
Panel Strength Calibration	NR	4.6.5.4
Crash Impact	NR	4.6.6.2
Fitting Strength	NR	4.6.5.5
Gunfire Resistance	NR	4.6.6.4
Post Gunfire Fuel Aging	NR	4.6.6.5

(a) - liquid spray rubber systems only; (b) - fabric reinforced rubber (lay-up) only; NR - not required

Table 4. Phase II Test Sequence

Tier I: Screening Tests - Candidate Materials
• ASTM D471: Test Method for Rubber Property - Effects of Liquids
Tier II: Property Screening - Candidate Materials
• Gum Inner Liner Strength (FED-STD-601, method 4111) / ASTM D412: Rubber Properties in Tension* • ASTM D624: Tear Resistance* • ASTM D2231: Rubber Properties in Forced Vibration (DMA)* • Nonvolatile Gum Residue (FED-STD-791, method 3302; ASTM D381) • Stoved Gum Residue (FED-STD-791, method 3302; ASTM D381)
Tier III: Inner Liner Material Characterization
• Fabric Inner Liner Strength (FED-STD-191, method 5100)* • Humidity Testing • ASTM D751: Standard Test Methods for Coated Fabrics* • Seam Adhesion (FED-STD-601, method 8011)* • ASTM D413: Adhesion to Flexible Substrates (rubber to fabric)* • ASTM D2137: Brittleness Point of Flexible Polymers and Coated Fabrics*
Tier IV: Characterization of Inner Liner with Barrier Material
• ASTM D413: Adhesion to Flexible Substrates (inner liner to barrier) • Permeability • Inner Liner Adhesion to Sealant (FED-STD-601, method 8011) • Stress Aging
Tier V: Composite Structure Testing
• Puncture Resistance (FED-STD-101C, 2065.1) • Slit Resistance • Constant Rate Tear • Impact Penetration • Impact Tear • Panel Strength Calibration
Tier VI: Fuel Cell Testing
• Leakage • Slosh Resistance • Stand Test • Crash Impact • Gunfire Resistance • Post Gunfire Aging

* Tests were performed before and after high temperature exposure to JP8+100.

4.0 RESULTS AND DISCUSSION

The testing and evaluation efforts performed under the Phase II program progressed through a series of steps starting with the screening tests conducted on compression molded rubber test plaques to eliminate obviously poor performers and rank the other materials being evaluated under the program according to performance. Fluid aging resistance was emphasized during the screening experiments. Physical property evaluations were performed using samples that were die-cut from the better performing test materials. Multiple sets of screening experiments (D471 fluid aging) and physical property testing were performed to ensure replication of test results prior to down-selection to the best performing candidate materials. To avoid repetition, the data presented in this report represent the final series of tests conducted on each sample. Testing and evaluation efforts of the best performing materials proceeded to materials which were calendared into the nylon fabric to support testing for the fabric reinforce inner liner materials. These samples were prepared using methods typical of those used in fuel cell manufacture.

The results presented in this section are provided in the same general sequence as the testing and evaluation efforts conducted under the program. Additional materials development and sample preparation efforts are presented as needed to support the discussion provided in this section of the report. Emphasis is placed on creating and presenting the basis for selecting the best performing program materials. As such, once a basis for eliminating a given material from further program consideration is presented, additional data may not be presented or discussed so emphasis can be placed on supporting the decisions made to move forward with testing, evaluation and qualification of the best performing materials identified under the program. For ease of presentation, data tables are presented at the end of each of sub-section.

4.1 Evaluation of Molded Samples

Samples die-cut from compression molded test plaques were used to support the majority of the screening experiments performed under the Phase II program. These samples were typically provided by the materials suppliers supporting the Phase II program efforts in the fully cured form and ready for testing. The objective of this portion of the program was to thoroughly evaluate which available material technologies, or classes of rubber materials, were capable of meeting the stringent requirements for high temperature fuel cell application, so the materials development efforts

performed under the Phase II program could be directed toward the best and most appropriate material technologies.

4.1.1 Volume Swell, Weight Gain and Hardness Change

The results of the fluid aging experiments performed on samples die-cut from compression molded test plaques are presented in this section. The actual performance of individual test samples or material classes is discussed in the context of the present application. Reasons for eliminating samples from further program consideration are presented along with a discussion of the relative ranking of materials used to identify which materials were emphasized in subsequent testing and evaluation efforts. The data presented in this section is limited to the data collected during the ASTM D417 fluid aging experiments in JP8+100 at 200°F and 225°F.

As previously discussed, fluid aging experiments were conducted in accordance with methods outlined in ASTM D 471: *Test Method for Rubber Property - Effects of Liquids*. Experiments were performed in for 3 day and 28 day periods with samples fully immersed in JP8+100 at 200ºF and 225ºF. Volume swell, weight gain and hardness change measurements were performed on all of the candidate test materials after high temperature fluid aging. All tests were performed in triplicate using methods previously described. The results reported are the average of measurements taken on the three replicate samples for fluid weight gain and dimensional volume swell. The hardness measurements reported (initial hardness and hardness change) for each sample are the average of nine (9) measurements for each sample condition – three hardness measurements for each of the three replicate samples. All test measurements performed on fluid aged samples were performed after excess fluid was removed from the samples and the samples were allowed to cool to room temperature.

For ease of presentation, the tabulated fluid aging data are presented as follows at this sub-section:

- Table 5. D471 Results – 3 Day Aging in JP8+100 @ 200°F
- Table 6. D471 Results – 28 Day Aging in JP8+100 @ 200°F
- Table 7. D471 Results – 3 Day Aging in JP8+100 @ 225°F
- Table 8. D471 Results – 28 Day Aging in JP8+100 @ 225°F.

Most of the candidate materials demonstrated good high temperature stability to JP8+100 at 200°F and 225°F. This was not surprising as most of the material classes were selected based on the results of the Phase I program efforts. Notable exceptions were one of the HNBR materials (14), two NBR materials (20 and 59) and two FKM materials (41 and 60), all of which exhibited excessive volume swell in all of the fluid aging experiments. As a group, the NBR materials all exhibited more volume swell than the other materials. One of the FKM materials that exhibited excessive volume swell (41) was an experimental material and not a pure fluorocarbon material; no additional data is available on the second FKM (60). Most of the TPE and TPU materials demonstrated excessive volume swell. The polyurethane samples demonstrated mixed response, either losing volume (33 and 45) or demonstrated excessive volume swell (46). Sample 27 (ECO) demonstrated weight loss under all test conditions. One of the FKM materials (5) demonstrated consistent weight gain at 200°F and consistent weight loss at 225°F.

While hardness and hardness change were not generally an issue, some of the TPE (42, 44, 44 and 56), TPU (54 and 55) and one of the FKM (6) materials were excessively hard and not well suited for fuel cell application. Other materials, including ECOs (17 and 57), HNBRs (34, 35, 36 and 37), NBR (22, 25, 58 and 59) also demonstrated higher hardness than may be desired for the proposed application. Excessive hardness change (greater than 10 points) was exhibited by the following materials: PUR (4, 33, 46), ECO (17, 57), NBR (20, 29, 38, 48), FKM (41, 60) and most of the TPEs and TPUs.

Table 5. D471 Results – 3 Day Aging in JP8+100 @ 200°F

Material ID	Class		ΔV (%)	ΔM (%)	Initial Hardness	Hardness (3 days)
1	FKM	Mean:	12.48	3.66	70.67	64.67
		Std. Dev:	8.80	0.20	1.73	1.22
2	FKM	Mean:	4.85	2.03	78.22	73.11
		Std. Dev:	3.59	0.08	0.83	0.93
3	FKM	Mean:	13.02	4.04	66.33	60.00
		Std. Dev:	2.89	0.32	1.12	1.58
4	PUR	Mean:	16.85	14.14	78.33	67.78
		Std. Dev:	1.72	0.58	1.00	0.83
5	FKM	Mean:	14.76	8.70	86.22	81.44
		Std. Dev:	1.39	0.66	0.44	0.53
6	FKM	Mean:	12.29	2.05	82.67	78.22
		Std. Dev:	4.11	0.12	1.32	1.56
7	ECO	Mean:	7.07	2.96	80.00	74.56
		Std. Dev:	1.06	1.34	0.50	0.53
8	HNBR	Mean:	12.89	7.33	78.44	69.44
		Std. Dev:	9.43	4.87	1.01	2.70
9	HNBR	Mean:	13.34	7.86	74.67	67.33
		Std. Dev:	7.70	4.41	0.50	2.78
10	FKMQ	Mean:	16.03	7.45	61.11	52.78
		Std. Dev:	2.28	0.17	1.45	1.09
11	FKM	Mean:	6.87	2.30	74.78	71.22
		Std. Dev:	1.95	0.10	0.44	0.44
12	FKM	Mean:	4.79	2.20	78.78	76.89
		Std. Dev:	2.08	0.09	2.17	0.78
13	PFE	Mean:	2.54	1.92	60.22	58.44
		Std. Dev:	2.10	0.01	2.44	1.01
14	HNBR	Mean:	33.23	19.58	69.00	59.89
		Std. Dev:	2.68	0.11	1.00	0.60
15	FKM	Mean:	6.64	2.58	79.11	75.67
		Std. Dev:	1.44	0.04	0.78	0.50
16	FKM	Mean:	7.66	2.12	73.67	68.89
		Std. Dev:	2.57	0.06	0.87	1.27
17	ECO	Mean:	15.62	10.21	83.56	70.11
		Std. Dev:	2.79	0.06	0.73	0.60
18	PFE	Mean:	5.62	2.83	56.44	57.11
		Std. Dev:	2.09	0.01	0.73	1.36
19	PFE	Mean:	15.86	10.19	84.00	70.89
		Std. Dev:	2.78	1.15	0.87	0.60
20	NBR	Mean:	56.60	32.36	82.00	53.33
		Std. Dev:	3.52	0.08	0.71	0.87

Table 5. D471 Results – 3 Day Aging in JP8+100 @ 200°F (Cont'd.)

Material ID	Class		ΔV (%)	ΔM (%)	Initial Hardness	Hardness (3 days)
21	PFE	Mean:	1.88	1.59	69.78	70.67
		Std. Dev:	1.01	0.01	0.67	0.71
22	NBR	Mean:	10.64	8.76	85.67	81.00
		Std. Dev:	1.43	0.14	0.50	0.50
23	FKM	Mean:	3.70	2.21	72.00	72.11
		Std. Dev:	1.96	0.70	3.43	1.27
24	NBR	Mean:	17.05	10.74	80.44	72.67
		Std. Dev:	1.36	0.14	0.53	1.00
25	NBR	Mean:	13.35	9.86	79.00	72.78
		Std. Dev:	0.59	0.12	0.50	1.09
26	HNBR	Mean:	3.34	3.41	63.78	59.33
		Std. Dev:	1.63	0.12	0.44	1.41
27	ECO	Mean:	-4.34	-2.86	61.67	64.00
		Std. Dev:	1.27	0.16	1.00	0.87
28	HNBR	Mean:	16.82	13.30	71.44	65.33
		Std. Dev:	1.36	0.62	1.42	1.22
29	NBR	Mean:	19.17	14.47	74.89	65.56
		Std. Dev:	1.71	0.18	0.33	0.53
30	NBR	Mean:	3.56	4.92	73.33	71.67
		Std. Dev:	0.76	0.02	0.50	0.50
31	FMVQ	Mean:	5.29	4.33	83.00	78.78
		Std. Dev:	0.59	0.06	0.00	0.97
32	HNBR	Mean:	14.76	11.60	78.33	69.89
		Std. Dev:	0.18	0.14	0.50	9.75
33	PUR	Mean:	-6.63	-3.46	74.22	81.22
		Std. Dev:	3.27	0.63	1.09	3.35
34	HNBR	Mean:	8.59	8.95	83.22	77.44
		Std. Dev:	1.39	0.07	0.44	0.73
35	HNBR	Mean:	10.03	9.01	83.56	78.11
		Std. Dev:	0.96	0.04	0.53	0.60
36	HNBR	Mean:	8.95	8.64	83.67	77.78
		Std. Dev:	1.39	0.05	0.50	0.44
37	HNBR	Mean:	8.70	8.69	83.89	76.89
		Std. Dev:	4.12	3.84	0.33	0.33
38	NBR	Mean:	15.04	15.01	71.89	61.22
		Std. Dev:	2.50	0.07	0.33	1.09
39	FMK	Mean:	9.18	7.08	72.89	69.78
		Std. Dev:	1.36	0.12	0.60	0.83
40	FKM	Mean:	4.27	1.40	73.11	72.33
		Std. Dev:	4.31	0.03	1.05	0.71
41	FKM	Mean:	38.00	21.40	72.78	54.78
		Std. Dev:	3.74	0.14	0.83	0.67

Table 6. D471 Results – 28 Day Aging in JP8+100 @ 200°F

Material ID	Class		ΔV (%)	ΔM (%)	Initial Hardness	Hardness (28 days)
1	FKM	Mean:	16.32	4.27	70.67	62.67
		Std. Dev:	8.23	0.07	1.73	1.12
2	FKM	Mean:	6.27	1.87	78.22	72.44
		Std. Dev:	4.65	2.74	0.83	0.88
3	FKM	Mean:	18.00	5.22	66.33	58.78
		Std. Dev:	0.73	0.68	1.12	1.09
4	PUR	Mean:	15.34	13.94	78.33	61.44
		Std. Dev:	0.18	0.20	1.00	1.13
5	FKM	Mean:	13.82	7.55	86.22	83.11
		Std. Dev:	1.67	0.62	0.44	0.60
6	FKM	Mean:	9.63	1.95	82.67	78.44
		Std. Dev:	5.44	0.04	1.32	1.13
7	ECO	Mean:	7.03	2.98	80.00	71.33
		Std. Dev:	3.34	1.33	0.50	0.71
8	HNBR	Mean:	13.45	6.99	78.44	67.44
		Std. Dev:	8.48	4.88	1.01	3.88
9	HNBR	Mean:	11.93	7.43	74.67	68.11
		Std. Dev:	7.20	4.56	0.50	2.57
10	FKMQ	Mean:	12.84	7.18	61.11	48.67
		Std. Dev:	3.42	0.89	1.45	1.32
11	FKM	Mean:	5.54	2.32	74.78	69.33
		Std. Dev:	2.50	2.52	0.44	0.50
12	FKM	Mean:	4.59	2.25	78.78	76.00
		Std. Dev:	1.50	0.07	2.17	0.50
13	PFE	Mean:	1.15	1.77	60.22	58.11
		Std. Dev:	2.70	0.65	2.44	0.33
14	HNBR	Mean:	30.03	18.33	69.00	58.33
		Std. Dev:	0.44	0.45	1.00	0.71
15	FKM	Mean:	7.45	2.62	79.11	75.33
		Std. Dev:	2.01	0.11	0.78	0.50
16	FKM	Mean:	6.84	2.19	73.67	75.11
		Std. Dev:	1.37	0.12	0.87	5.23
17	ECO	Mean:	22.83	13.82	83.56	60.50
		Std. Dev:	1.26	0.29	0.73	0.84
18	PFE	Mean:	7.59	2.69	56.44	56.11
		Std. Dev:	3.91	1.30	0.73	0.93
19	PFE	Mean:	29.18	18.13	84.00	49.22
		Std. Dev:	2.36	1.41	0.87	2.11
20	NBR	Mean:	83.40	46.38	82.00	44.56
		Std. Dev:	0.73	0.25	0.71	1.13

Table 6. D471 Results – 28 Day Aging in JP8+100 @ 200°F (Cont'd.)

Material ID	Class		ΔV (%)	ΔM (%)	Initial Hardness	Hardness (28 days)
21	PFE	Mean:	0.90	-32.32	69.78	71.78
		Std. Dev:	0.61	58.61	0.67	0.44
22	NBR	Mean:	8.14	-11.17	85.67	83.89
		Std. Dev:	3.07	33.02	0.50	0.60
23	FKM	Mean:	1.90	1.92	72.00	72.89
		Std. Dev:	0.68	0.15	3.43	3.48
24	NBR	Mean:	14.70	10.34	80.44	76.33
		Std. Dev:	1.62	0.13	0.53	1.00
25	NBR	Mean:	11.18	9.23	79.00	77.11
		Std. Dev:	1.83	0.14	0.50	1.05
26	HNBR	Mean:	-0.34	2.60	63.78	61.78
		Std. Dev:	1.45	0.08	0.44	0.83
27	ECO	Mean:	-6.28	-3.70	61.67	66.22
		Std. Dev:	0.97	0.82	1.00	0.67
28	HNBR	Mean:	14.16	12.20	71.44	67.00
		Std. Dev:	0.69	0.74	1.42	1.00
29	NBR	Mean:	16.95	13.81	74.89	64.22
		Std. Dev:	0.99	0.05	0.33	0.67
30	NBR	Mean:	2.86	4.03	73.33	76.22
		Std. Dev:	3.28	1.39	0.50	1.20
31	FMVQ	Mean:	3.61	4.09	83.00	79.44
		Std. Dev:	0.64	0.79	0.00	0.88
32	HNBR	Mean:	10.27	11.49	78.33	73.11
		Std. Dev:	3.75	0.02	0.50	0.60
33	PUR	Mean:	-13.88	-12.27	74.22	nd
		Std. Dev:	1.35	0.27	1.09	
34	HNBR	Mean:	10.13	8.48	83.22	77.56
		Std. Dev:	4.14	0.04	0.44	0.53
35	HNBR	Mean:	9.02	8.43	83.56	76.00
		Std. Dev:	2.15	0.05	0.53	0.71
36	HNBR	Mean:	8.72	8.17	83.67	78.11
		Std. Dev:	1.48	0.13	0.50	0.60
37	HNBR	Mean:	9.84	8.14	83.89	78.33
		Std. Dev:	0.82	0.12	0.33	0.87
38	NBR	Mean:	121.58	12.33	71.89	71.44
		Std. Dev:	194.61	0.17	0.33	0.53
39	FMK	Mean:	9.52	7.92	72.89	69.33
		Std. Dev:	2.70	0.24	0.60	0.71
40	FKM	Mean:	1.84	1.36	73.11	72.56
		Std. Dev:	1.95	1.02	1.05	0.73
41	FKM	Mean:	36.30	21.34	72.78	53.11
		Std. Dev:	1.61	0.27	0.83	2.20

nd = not determined

Table 7. D471 Results – 3 Day Aging in JP8+100 @ 225°F

Material ID	Class		ΔV (%)	ΔM (%)	Initial Hardness	Hardness (3 days)
1	FKM	Mean:	12.74	4.17	69.11	62.22
		Std. Dev:	3.42	0.16	0.78	0.83
2	FKM	Mean:	5.03	1.60	77.22	69.11
		Std. Dev:	2.88	0.98	1.20	2.09
3	FKM	Mean:	9.34	4.16	69.67	62.22
		Std. Dev:	1.51	0.93	1.22	2.28
4	PUR	Mean:	12.69	15.19	75.67	60.00
		Std. Dev:	4.98	0.10	0.87	2.78
5	FKM	Mean:	-4.29	-3.22	76.67	77.56
		Std. Dev:	2.92	0.29	2.87	2.79
6	FKM	Mean:	36.99	33.57	>90	>90
		Std. Dev:	18.62	1.84	0.00	0.00
7	ECO	Mean:	7.34	5.34	81.00	74.44
		Std. Dev:	0.84	0.04	1.00	0.73
8	HNBR	Mean:	8.19	4.84	79.89	74.11
		Std. Dev:	3.54	0.12	0.60	0.60
9	HNBR	Mean:	13.92	26.63	78.56	70.22
		Std. Dev:	1.67	27.55	1.01	0.67
10	FKMQ	Mean:	8.98	6.96	60.89	53.89
		Std. Dev:	1.36	0.17	1.27	1.62
11	FKM	Mean:	6.65	2.73	74.11	71.00
		Std. Dev:	2.71	0.04	0.60	1.00
12	FKM	Mean:	5.79	1.09	79.00	74.44
		Std. Dev:	1.02	1.40	1.00	0.53
13	PFE	Mean:	3.89	1.99	62.67	62.33
		Std. Dev:	0.91	0.03	0.87	1.22
14	HNBR	Mean:	25.09	19.62	72.22	65.33
		Std. Dev:	4.15	0.08	1.09	1.41
15	FKM	Mean:	7.17	2.71	80.22	77.11
		Std. Dev:	0.15	0.42	0.83	0.78
16	FKM	Mean:	5.47	2.54	75.00	70.78
		Std. Dev:	2.66	0.26	0.50	0.97
17	ECO	Mean:	14.30	10.51	83.67	71.11
		Std. Dev:	0.49	0.05	0.50	1.05
18	PFE	Mean:	2.13	2.82	63.78	63.89
		Std. Dev:	1.63	0.02	1.09	1.17
19	PFE	Mean:	2.69	1.59	74.67	74.67
		Std. Dev:	3.22	0.02	0.50	1.00
20	NBR	Mean:	75.51	40.45	79.78	50.56
		Std. Dev:	2.99	1.23	1.09	1.33
21	PFE	Mean:	0.60	1.64	74.44	73.11
		Std. Dev:	1.43	0.08	0.73	1.05

Table 7. D471 Results – 3 Day Aging in JP8+100 @ 225°F (Cont'd.)

Material ID	Class		ΔV (%)	ΔM (%)	Initial Hardness	Hardness (3 days)
22	NBR	Mean:	14.75	9.16	86.56	83.22
		Std. Dev:	3.06	0.06	0.73	0.83
23	FKM	Mean:	4.95	2.00	73.33	73.22
		Std. Dev:	0.94	0.05	0.87	1.30
24	NBR	Mean:	21.43	11.69	82.89	75.22
		Std. Dev:	2.47	0.04	0.60	0.44
25	NBR	Mean:	18.42	10.97	81.00	75.00
		Std. Dev:	2.04	0.31	1.73	1.66
26	HNBR	Mean:	5.92	3.60	69.00	62.44
		Std. Dev:	2.12	0.12	1.00	0.88
27	ECO	Mean:	0.12	-2.55	66.11	65.56
		Std. Dev:	0.92	0.07	0.78	1.33
28	HNBR	Mean:	27.05	15.75	71.44	64.11
		Std. Dev:	5.18	1.90	3.50	4.91
29	NBR	Mean:	26.59	15.34	78.22	67.89
		Std. Dev:	1.18	0.22	0.67	1.27
30	NBR	Mean:	10.03	4.73	76.00	74.56
		Std. Dev:	1.87	0.37	1.12	0.88
31	FMVQ	Mean:	12.93	5.14	84.78	77.78
		Std. Dev:	1.56	0.15	0.83	1.09
32	HNBR	Mean:	19.41	12.26	80.22	74.78
		Std. Dev:	1.70	0.08	0.44	0.83
33	PUR	Mean:	-2.74	-3.95	80.56	>90
		Std. Dev:	9.89	0.59	3.21	0.00
34	HNBR	Mean:	14.84	9.54	86.11	79.33
		Std. Dev:	2.78	0.15	0.78	0.71
35	HNBR	Mean:	14.31	9.57	85.89	79.89
		Std. Dev:	1.43	0.06	0.60	0.60
36	HNBR	Mean:	12.97	9.28	85.89	79.22
		Std. Dev:	0.41	0.29	0.78	1.39
37	HNBR	Mean:	13.24	9.24	86.56	79.33
		Std. Dev:	0.49	0.09	0.53	0.87
38	NBR	Mean:	21.44	14.38	73.44	61.00
		Std. Dev:	5.33	0.05	0.88	1.58
39	FMK	Mean:	5.68	3.02	81.22	76.56
		Std. Dev:	0.36	0.13	0.83	0.73
40	FKM	Mean:	2.57	1.59	73.56	72.00
		Std. Dev:	1.52	0.01	1.01	1.00
41	FKM	Mean:	35.50	25.00	71.78	53.56
		Std. Dev:	1.93	2.78	0.83	1.51
42	TPE	Mean:	10.47	76.00	>90	81.33
		Std. Dev:	0.44	1.21	0.00	0.87

Table 7. D471 Results – 3 Day Aging in JP8+100 @ 225°F (Cont'd.)

Material ID	Class		ΔV (%)	ΔM (%)	Initial Hardness	Hardness (3 days)
43	TPE	Mean:	102.30	6.26	84.22	56.56
		Std. Dev:	1.77	1.08	0.97	0.73
44	TPE	Mean:	125.05	107.10	85.44	38.56
		Std. Dev:	8.63	0.35	1.01	7.30
45	PUR	Mean:	-1.35	-3.06	59.89	66.22
		Std. Dev:	1.31	1.85	1.17	0.67
46	PUR	Mean:	37.16	29.51	77.56	52.89
		Std. Dev:	2.06	0.15	1.01	0.78
47	ECO	Mean:	10.59	8.14	71.22	62.33
		Std. Dev:	0.31	0.56	0.97	0.87
48	NBR	Mean:	17.76	14.77	75.22	61.33
		Std. Dev:	4.35	0.13	0.83	1.32
49	NBR	Mean:	16.83	12.43	75.00	65.78
		Std. Dev:	1.80	0.70	0.71	1.72
50	TPE	Mean:	51.15	31.20	72.22	53.78
		Std. Dev:	4.35	2.27	0.44	1.79
51	TPE	Mean:	1.71	-3.80	81.78	73.33
		Std. Dev:	3.32	0.41	1.20	0.71
52	TPE	Mean:	126.22	106.31	77.11	40.56
		Std. Dev:	5.96	1.96	1.05	3.32
53	TPU	Mean:	138.87	111.91	78.78	23.78
		Std. Dev:	8.33	0.90	1.09	2.95
54	TPU	Mean:	7.53	5.32	>90	>90
		Std. Dev:	1.70	0.11	0.00	0.00
55	TPU	Mean:	7.44	1.77	>90	>90
		Std. Dev:	0.65	6.74	0.00	0.00
56	TPE	Mean:	7.16	4.52	>90	>90
		Std. Dev:	1.64	0.05	0.00	0.00
57	ECO	Mean:	14.58	11.50	87.56	73.22
		Std. Dev:	0.79	0.08	0.73	0.83
58	NBR	Mean:	16.11	8.49	82.33	75.22
		Std. Dev:	3.75	0.06	1.00	0.83
59	NBR	Mean:	18.58	13.07	82.00	73.11
		Std. Dev:	1.05	0.08	1.41	1.36
60	FKM	Mean:	33.88	21.26	78.33	67.33
		Std. Dev:	4.28	0.21	1.32	1.12

Table 8. D471 Results – 28 Day Aging in JP8+100 @ 225°F

Material ID	Class		ΔV (%)	ΔM (%)	Initial Hardness	Hardness (28 days)
1	FKM	Mean:	17.28	5.38	69.11	63.00
		Std. Dev:	1.74	0.33	0.78	1.87
2	FKM	Mean:	6.75	2.33	77.22	71.11
		Std. Dev:	2.92	0.07	1.20	1.36
3	FKM	Mean:	14.76	4.81	69.67	62.33
		Std. Dev:	1.59	0.62	1.22	1.87
4	PUR	Mean:	13.94	13.77	75.67	54.78
		Std. Dev:	7.08	0.06	0.87	1.39
5	FKM	Mean:	-4.12	-3.77	76.67	74.44
		Std. Dev:	2.20	0.28	2.87	11.19
6	FKM	Mean:	57.04	55.21	>90	>90
		Std. Dev:	4.66	12.03	0.00	0.00
7	ECO	Mean:	8.44	4.81	81.00	78.11
		Std. Dev:	1.58	0.24	1.00	1.17
8	HNBR	Mean:	7.84	4.53	79.89	77.56
		Std. Dev:	0.49	0.17	0.60	1.67
9	HNBR	Mean:	11.74	9.39	78.56	72.78
		Std. Dev:	0.98	0.16	1.01	1.48
10	FKMQ	Mean:	12.11	6.98	60.89	54.67
		Std. Dev:	2.10	1.75	1.27	1.41
11	FKM	Mean:	9.55	2.71	74.11	69.44
		Std. Dev:	1.05	0.91	0.60	0.88
12	FKM	Mean:	5.37	2.10	79.00	74.33
		Std. Dev:	2.57	1.69	1.00	1.73
13	PFE	Mean:	2.05	1.92	62.67	62.33
		Std. Dev:	1.40	0.04	0.87	1.12
14	HNBR	Mean:	25.10	-60.32	72.22	64.11
		Std. Dev:	1.70	68.73	1.09	1.45
15	FKM	Mean:	5.72	2.99	80.22	77.56
		Std. Dev:	1.81	0.37	0.83	1.13
16	FKM	Mean:	5.68	2.61	75.00	71.56
		Std. Dev:	1.70	0.20	0.50	1.51
17	ECO	Mean:	21.69	12.32	83.67	60.33
		Std. Dev:	4.61	2.74	0.50	6.52
18	PFE	Mean:	1.88	2.45	63.78	63.56
		Std. Dev:	3.00	0.20	1.09	1.74
19	PFE	Mean:	3.53	1.54	74.67	74.78
		Std. Dev:	2.17	0.02	0.50	0.83
20	NBR	Mean:	92.97	50.25	79.78	42.44
		Std. Dev:	6.82	1.47	1.09	4.59
21	PFE	Mean:	2.65	1.58	74.44	73.33
		Std. Dev:	1.86	0.07	0.73	1.66

Table 8. D471 Results – 28 Day Aging in JP8+100 @ 225°F (Cont'd.)

Material ID	Class		ΔV (%)	ΔM (%)	Initial Hardness	Hardness (28 days)
22	NBR	Mean:	11.94	7.36	86.56	86.89
		Std. Dev:	1.79	1.57	0.73	0.78
23	FKM	Mean:	3.12	2.50	73.33	73.22
		Std. Dev:	3.70	0.45	0.87	1.39
24	NBR	Mean:	19.39	10.85	82.89	79.78
		Std. Dev:	2.92	0.34	0.60	1.48
25	NBR	Mean:	15.58	9.56	81.00	79.00
		Std. Dev:	2.08	0.15	1.73	1.32
26	HNBR	Mean:	3.88	3.19	69.00	65.78
		Std. Dev:	2.56	0.39	1.00	1.20
27	ECO	Mean:	-0.96	-3.36	66.11	67.78
		Std. Dev:	2.59	0.28	0.78	1.56
28	HNBR	Mean:	9.39	15.03	71.44	66.33
		Std. Dev:	24.98	2.13	3.50	4.64
29	NBR	Mean:	23.89	15.17	78.22	68.11
		Std. Dev:	3.16	0.19	0.67	1.36
30	NBR	Mean:	9.38	4.41	76.00	79.56
		Std. Dev:	2.98	0.40	1.12	1.74
31	FMVQ	Mean:	10.94	5.41	84.78	76.78
		Std. Dev:	0.42	0.22	0.83	2.05
32	HNBR	Mean:	20.20	12.19	80.22	73.44
		Std. Dev:	0.14	0.05	0.44	1.81
33	PUR	Mean:	-17.76	-23.82	80.56	>90
		Std. Dev:	5.56	0.89	3.21	0.00
34	HNBR	Mean:	11.37	9.67	86.11	78.56
		Std. Dev:	1.44	0.25	0.78	0.88
35	HNBR	Mean:	12.71	9.34	85.89	80.00
		Std. Dev:	1.28	0.07	0.60	1.50
36	HNBR	Mean:	11.12	9.13	85.89	79.89
		Std. Dev:	0.85	0.15	0.78	1.17
37	HNBR	Mean:	12.07	9.13	86.56	79.11
		Std. Dev:	1.99	0.03	0.53	0.33
38	NBR	Mean:	18.32	13.01	73.44	68.78
		Std. Dev:	5.01	6.43	0.88	1.20
39	FMK	Mean:	10.38	3.78	81.22	75.67
		Std. Dev:	4.20	1.61	0.83	0.71
40	FKM	Mean:	6.96	1.58	73.56	72.67
		Std. Dev:	1.72	0.02	1.01	0.87
41	FKM	Mean:	38.11	24.93	71.78	51.00
		Std. Dev:	3.50	2.48	0.83	1.22
42	TPE	Mean:	9.24	0.84	>90	83.11
		Std. Dev:	1.84	0.22	0.00	0.78

Table 8. D471 Results – 28 Day Aging in JP8+100 @ 225°F (Cont'd.)

Material ID	Class		ΔV (%)	ΔM (%)	Initial Hardness	Hardness (28 days)
43	TPE	Mean:	107.70	89.88	84.22	53.89
		Std. Dev:	7.59	1.45	0.97	1.05
44	TPE	Mean:	142.86	111.10	85.44	>90
		Std. Dev:	13.33	0.70	1.01	0.00
45	PUR	Mean:	-3.20	-6.48	59.89	>90
		Std. Dev:	0.81	0.27	1.17	0.00
46	PUR	Mean:	29.87	24.15	77.56	43.33
		Std. Dev:	1.30	0.69	1.01	1.00
47	ECO	Mean:	14.40	7.64	71.22	58.67
		Std. Dev:	17.93	5.55	0.97	1.80
48	NBR	Mean:	13.27	11.91	75.22	84.67
		Std. Dev:	1.15	1.37	0.83	0.87
49	NBR	Mean:	15.52	10.23	75.00	87.44
		Std. Dev:	2.09	0.78	0.71	1.51
50	TPE	Mean:	62.21	34.35	72.22	46.33
		Std. Dev:	4.39	0.17	0.44	1.66
51	TPE	Mean:	-3.50	-7.18	81.78	75.44
		Std. Dev:	2.96	0.43	1.20	0.73
52	TPE	Mean:	137.90	111.38	77.11	40.33
		Std. Dev:	12.53	3.46	1.05	2.29
53	TPU	Mean:	146.90	115.26	78.78	>90
		Std. Dev:	7.01	1.20	1.09	0.00
54	TPU	Mean:	9.21	5.40	>90	>90
		Std. Dev:	1.71	0.45	0.00	0.00
55	TPU	Mean:	8.84	1.65	>90	>90
		Std. Dev:	0.68	7.00	0.00	0.00
56	TPE	Mean:	8.43	4.40	>90	>90
		Std. Dev:	0.79	0.09	0.00	0.00
57	ECO	Mean:	14.20	10.81	87.56	60.56
		Std. Dev:	2.36	0.39	0.73	1.81
58	NBR	Mean:	13.65	7.76	82.33	77.56
		Std. Dev:	1.78	0.18	1.00	1.51
59	NBR	Mean:	23.77	11.80	82.00	>90
		Std. Dev:	4.79	0.08	1.41	0.00
60	FKM	Mean:	27.28	19.57	78.33	69.78
		Std. Dev:	2.00	0.57	1.32	1.99

4.1.2 Identification of Best Performers

The results of the D471 fluid aging experiments were used as the primary screening criteria for evaluating the potential of the candidate materials for high temperature fuel bladder application. After the 225°F fluid aging experiments, candidate materials were placed into one of three groups based on their 3-day volume swell performance:

- Best Candidates – The best candidate materials demonstrated 0 to 6% volume swell, less than 10 points change in Shore A hardness, and less than 10% weight gain. All of these materials were subjected to more extensive testing and evaluation under the Phase II program. These best performing materials included the following:
- Good Candidates – Good candidate materials demonstrated 6 to 13% volume swell, less than 10 points change in Shore A hardness, and less than 10% weight gain. Modified formulations of these materials were often developed and tested to improve performance.
- Other Materials – Materials with greater than 13% volume swell.

The best and good candidate materials are listed separately in Tables 9 and 10 respectively. Some materials meeting the primary volume swell criterion are not included in these tables due to excessive hardness. This included the thermoplastic elastomers and thermoplastic polyurethane materials which otherwise exhibited good stability in JP8+100. Volume swell, weight gain and hardness data (for 3 day JP8+100 fluid aging at 225°F) are repeated in these tables to support further discussion.

Table 9. Best Performers (0 to 6% Volume Swell after 3 Days in JP8+100 @ 225°F)

Material ID	Class		ΔV (%)	ΔM (%)	Initial Hardness	Hardness (3 days)
2	FKM	Mean:	5.03	1.60	77.22	69.11
		Std. Dev:	2.88	0.98	1.20	2.09
12	FKM	Mean:	5.79	1.09	79.00	74.44
		Std. Dev:	1.02	1.40	1.00	0.53
13	PFE	Mean:	3.89	1.99	62.67	62.33
		Std. Dev:	0.91	0.03	0.87	1.22
16	FKM	Mean:	5.47	2.54	75.00	70.78
		Std. Dev:	2.66	0.26	0.50	0.97
18	PFE	Mean:	2.13	2.82	63.78	63.89
		Std. Dev:	1.63	0.02	1.09	1.17
19	PFE	Mean:	2.69	1.59	74.67	74.67
		Std. Dev:	3.22	0.02	0.50	1.00
21	PFE	Mean:	0.60	1.64	74.44	73.11
		Std. Dev:	1.43	0.08	0.73	1.05
23	FKM	Mean:	4.95	2.00	73.33	73.22
		Std. Dev:	0.94	0.05	0.87	1.30
26	HNBR	Mean:	5.92	3.60	69.00	62.44
		Std. Dev:	2.12	0.12	1.00	0.88
27	ECO	Mean:	0.12	-2.55	66.11	65.56
		Std. Dev:	0.92	0.07	0.78	1.33
39	FKM	Mean:	5.68	3.02	81.22	76.56
		Std. Dev:	0.36	0.13	0.83	0.73
40	FKM	Mean:	2.57	1.59	73.56	72.00
		Std. Dev:	1.52	0.01	1.01	1.00

Table 10. Good Performers (6 to 13% Volume Swell after 3 Days in JP8+100 @ 225°F)

Material ID	Class		ΔV (%)	ΔM (%)	Initial Hardness	Hardness (3 days)
1	FKM	Mean:	12.74	4.17	69.11	62.22
		Std. Dev:	3.42	0.16	0.78	0.83
3	FKM	Mean:	9.34	4.16	69.67	62.22
		Std. Dev:	1.51	0.93	1.22	2.28
7	ECO	Mean:	7.34	5.34	81.00	74.44
		Std. Dev:	0.84	0.04	1.00	0.73
8	HNBR	Mean:	8.19	4.84	79.89	74.11
		Std. Dev:	3.54	0.12	0.60	0.60
10	FKMQ	Mean:	8.98	6.96	60.89	53.89
		Std. Dev:	1.36	0.17	1.27	1.62
11	FKM	Mean:	6.65	2.73	74.11	71.00
		Std. Dev:	2.71	0.04	0.60	1.00
15	FKM	Mean:	7.17	2.71	80.22	77.11
		Std. Dev:	0.15	0.42	0.83	0.78
30	NBR	Mean:	10.03	4.73	76.00	74.56
		Std. Dev:	1.87	0.37	1.12	0.88
47	ECO	Mean:	10.59	8.14	71.22	62.33
		Std. Dev:	0.31	0.56	0.97	0.87

4.1.3 Physical Properties

The physical properties of the best performing candidate rubber materials were determined before and after fluid aging in JP8+100 at 225°F for 3 and 28 days. Tensile property measurements were performed in accordance with ASTM D 412: *Standard Test Methods for Rubber Properties in Tension*, using Type C dumbbell specimens (5 replicates per test condition) that were die-cut from the cured rubber plaques. The tear strength of the candidate rubber materials was determined in accordance with methods outlined in ASTM D624: *Standard Test Method for Tear Strength of Conventional Vulcanized Rubber and Thermoplastic Elastomers*, also using Type C dumbbell specimens (5 replicates per test condition) die-cut from the cured rubber plaques. Both tests were performed at an extension rate of 2 inches per minute. The results reported are the average and standard deviation of measurements taken on five replicate samples for unaged samples, and the relative change (%) in tensile or tear properties of the materials after fluid aging based on the average properties of the aged samples relative to the average properties of the unaged samples. The tabulated tensile and tear property data are presented in the following tables:

- Table 11. Tensile Data – 3 Day Aging in JP8+100 @ 2225°F
- Table 12. Tensile Data – 28 Day Aging in JP8+100 @ 225°F
- Table 13. Tear Data – 3 and 28 Day Aging in JP8+100 @ 225°F.

Retention of tensile properties after fluid aging was used as an important criterion for program consideration as property retention after aging provides an excellent indication of long service performance. Existing performance specifications for full bladder materials require that the inner liner gum not lose more than half of their initial tensile strength (50% retention) and elongation at break after three days aging in the hot jet fuel. As noted in Table 11 (3 day aging in JP8+100 @ 225°), all of the best performing materials met this requirement. After 28 days of high temperature fluid aging, ECO (7), FKMQ (10) and PFE (13) materials demonstrated in excess of 50% degradation in properties. All of the samples tested demonstrated less than 50% reduction in tear properties after 3 and 28 days of aging in JP8+100 @ 225°F, with the exception of PUR (4), FKMQ (10) and FKM (39) after 28 days of high temperature fluid aging.

DMA data, before and after 3 and 28 day fluid aging, are provided in Table 14. The DMA data provide an indication of low temperature flexibility as well as retention of flexibility during service.

Table 11. Tensile Data, Best Performers, 3 Days in JP-8+100 @ 225° F

Material ID	Material Type		Unaged		Fluid Aged	
			Tensile Strength (psi)	Elongation @ Break (%)	ΔTensile Strength (%)	ΔElongation @ Break (%)
1	FKM	Mean:	1610.43	681.42	36	34.8
		Std. Dev:	548.45	141.98		
2	FKM	Mean:	1828.66	372.77	-26.4	-5.5
		Std. Dev:	326.97	21.78		
3	FKM	Mean:	2265.26	877.86	nd	nd
		Std. Dev:	263.78	53.98		
7	ECO	Mean:	1533.09	452.67	-6.13	-19.96
		Std. Dev:	183.53	42.28		
8	HNBR	Mean:	4139.96	673.28	-31.5	-27.2
		Std. Dev:	270.37	22.49		
10	FKMQ	Mean:	759.26	590.84	-13.3	-20
		Std. Dev:	96.58	49.77		
11	FKM	Mean:	817.83	555.73	nd	nd
		Std. Dev:				
12	FKM	Mean:	1130.28	298.47	-13.1	-24.1
		Std. Dev:	343.91	71.35		
13	PFE	Mean:	1217.81	572.04	-17.47	-7.3
		Std. Dev:	463.95	125.93		
15	FKM	Mean:	1539.64	306.11	-17.1	-14.5
		Std. Dev:	98.84	45		
16	FKM	Mean:	1215.37	373.28	-21.42	-17.24
		Std. Dev:	375.2	85.66		
18	PFE	Mean:	1235.76	674.55	-38.85	-40.7
		Std. Dev:	157.43	101.09		
19	PFE	Mean:	1205.1	370.48	-23.48	-24.93
		Std. Dev:	257.14	59.6		
21	PFE	Mean:	826.85	381.17	1.58	-29.37
		Std. Dev:	25.45	135.72		
23	FKM	Mean:	1299.7	264.12	-14.27	-12.04
		Std. Dev:	510.3	58.87		
26	HNBR	Mean:	2192.7	1102.29	-19.3	-10.3
		Std. Dev:	188.51	72.72		
27	ECO	Mean:	874.9	505.1	0.1	0
		Std. Dev:	52.54	29.61		
30	NBR	Mean:	2358.39	721.12	-23.8	-41.6
		Std. Dev:	60.96	40.91		
39	FKM	Mean:	1195.97	409.16	-40.1	-28.9
		Std. Dev:	105.66	46.43		
40	FKM	Mean:	669.04	241.22	10.08	-1.74
		Std. Dev:	24.89	16.88		
47	ECO	Mean:	1729.53	740.08	nd	nd
		Std. Dev:	1187.56	55.6		

nd = not determined

Table 12. Tensile Data, Best Performers, 28 Days in JP-8+100 @ 225° F

Material ID	Material Type		Unaged		Fluid Aged	
			Tensile Strength (psi)	Elongation @ Break (%)	ΔTensile Strength (%)	ΔElongation @ Break (%)
1	FKM	Mean:	1610.43	681.42	-29.92	-8.42
		Std. Dev:	548.45	141.98		
2	FKM	Mean:	1828.66	372.77	-42.5	18.36
		Std. Dev:	326.66	21.78		
3	FKM	Mean:	2265.26	877.86	nd	nd
		Std. Dev:	263.78	53.98		
7	ECO	Mean:	1496.23	599.24	-70.61	-54.91
		Std. Dev:	144.3	259.46		
8	HNBR	Mean:	4139.96	673.28	-40.12	-41.84
		Std. Dev:	270.37	22.49		
10	FKMQ	Mean:	759.26	590.84	-84.01	-76.66
		Std. Dev:	96.58	49.77		
11	FKM	Mean:	817.83	555.73	nd	nd
		Std. Dev:				
12	FKM	Mean:	1130.28	298.47	7.48	-19.52
		Std. Dev:	343.91	71.35		
13	PFE	Mean:	1217.81	572.04	-66.82	-59.08
		Std. Dev:	463.95	125.93		
15	FKM	Mean:	1539.64	306.11	-22.46	-20.35
		Std. Dev:	98.84	45		
16	FKM	Mean:	1215.37	373.28	-45.86	-34.35
		Std. Dev:	375.2	85.66		
18	PFE	Mean:	1235.76	674.55	-46.88	-53.26
		Std. Dev:	157.43	101.09		
19	PFE	Mean:	1205.1	370.48	-34.47	-21.63
		Std. Dev:	257.14	59.6		
21	PFE	Mean:	826.85	381.17	-3.62	-33.84
		Std. Dev:	25.45	135.72		
23	FKM	Mean:	1299.7	264.12	-7.63	0.19
		Std. Dev:	510.3	58.87		
26	HNBR	Mean:	2192.7	1102.29	-8.92	-22.44
		Std. Dev:	188.51	72.72		
27	ECO	Mean:	874.9	505.1	-41.01	-49.92
		Std. Dev:	52.54	29.61		
30	NBR	Mean:	2358.39	721.12	-54.33	-4.59
		Std. Dev:	60.96	40.91		
39	FKM	Mean:	1195.97	409.16	-8.92	-1.93
		Std. Dev:	105.66	46.43		
40	FKM	Mean:	669.04	241.22	-27.83	-12.87
		Std. Dev:	24.89	16.88		
47	ECO	Mean:	1729.53	740.08	nd	nd
		Std. Dev:	1187.56	55.6		

nd = not determined

Table 13. TEAR DATA, BEST PERFORMERS, 3 AND 28 DAYS IN JP-8+100 @ 225° F

Material ID	Material Type		Unaged (lb/in)	%Change After 3 Days	%Change After 28 Days
1	FKM	Mean:	108.27	6.8%	-23.2%
		Std. Dev:	2.30		
2	FKM	Mean:	114.89	-27.4%	3.7%
		Std. Dev:	2.84		
3	FKM	Mean:	107.26	-3.6%	7.8%
		Std. Dev:	10.97		
4	PUR	Mean:	236.44	-30.9%	-81.3%
		Std. Dev:	31.26		
7	ECO	Mean:	206.39	-36.3%	-52.1%
		Std. Dev:	5.75		
8	HNBR	Mean:	156.14	-29.0%	-29.0%
		Std. Dev:	7.86		
10	FKMQ	Mean:	57.79	-34.2%	-72.9%
		Std. Dev:	1.70		
13	PFE	Mean:	33.19	41.7%	nd
		Std. Dev:	13.70		
19	PFE	Mean:	56.50	-27.5%	-26.2%
		Std. Dev:	4.83		
21	PFE	Mean:	51.86	-24.1%	-13.8%
		Std. Dev:	0.63		
26	HNBR	Mean:	219.30	-20.5%	-48.2%
		Std. Dev:	17.50		
27	ECO	Mean:	127.27	-17.9%	-39.7%
		Std. Dev:	4.36		
30	NBR	Mean:	245.73	-42.1%	-48.9%
		Std. Dev:	6.82		
39	FKM	Mean:	80.44	-25.8%	-58.7%
		Std. Dev:	4.78		

nd = not determined

Table 14. DMA Data, Best Performers, Before and After Aging in JP-8+100 @ 225° F

Material ID	Material Type	Control		3 Day		28 Day	
		Onset (°F)	T_g (°F)	Onset (°F)	T_g (°F)	Onset (°F)	T_g (°F)
1	FKM	3.0	21.9	4.4	25.3	13.1	27.4
2	FKM	18.4	36.2	12.7	32.8	16.2	35.5
3	FKM	5.2	25.0	19.8	39.1	8.8	27.1
4	PUR	nd	nd	nd	nd	nd	nd
7	ECO	nd	nd	-23.1	-2.9	-40.5	-20.8
8	HNBR	9.5	29.1	30.6	52.1	21.6	39.4
10	FVMQ	-103.4	-84.4	-79.3	-56.1	-77.1	-53.3
11	FKM	4.9	23.2	1.6	27.0	7.0	27.5
12	FKM	8.9	26.8	9.2	29.3	12.8	30.0
13	PFE	nd	nd	-60.3	-41.1	-57.6	-34.6
15	FKM	nd	nd	nd	nd	-18.0	2.2
16	FKM	9.0	29.0	6.7	28.8	-6.9	14.5
18	PFE	nd	nd	-40.8	-18.5	-45.6	-28.9
19	PFE	-28.9	-11.5	-41.8	-26.7	-49.1	-36.3
20	NBR	-34.1	-12.2	nd	nd	-23.2	20.2
21	PFE	-37.9	-19.0	-48.1	-32.2	-42.8	-24.3
23	FKM	-15.7	6.2	-26.7	-10.9	-23.2	-3.3
27	ECO	nd	nd	nd	nd	-23.7	-7.6
30	NBR	8.1	26.8	18.4	49.3	22.5	55.7
40	FKM	-44.7	-25.4	-72.4	-28.0	-49.7	-31.7

nd = not determined

4.2 Liquid Systems

The development efforts related to the liquid sprayable systems were directed by the results of the formulation development efforts. This was done by necessity due to the inherent difficulty in working with liquefied rubber materials and the impact the issues associated with processing these materials may have on fuel bladder manufacture. Primary challenges with all of the liquid systems included:

- the ability to form good stable solutions or dispersion
- difficulty spraying or casting void free films
- drying and curing of defect free films.

Nine of the liquid systems (see Table 2) evaluated under the Phase II program were based on fluoropolymer chemistry. This was primarily due to the good performance of FKM materials during the screening studies as well as the relative availability of these materials in the liquid form. All of the fluoropolymer systems were evaluated under the Phase II program were latexes (water-based). While METSS evaluated some solvent based nitrile (including NBR, HNBRs and CNBRs) systems during the Phase II program, all of the commercial systems evaluated were latex systems. Both of the polyurethane systems were solvent based. The polysulfide was a latex dispersion and the polyurea was a two-component 100% solids systems.

Samples of each of the commercial liquid systems were prepared by spraying or casting multiple layers of thin coatings to obtain a sample thick enough to support the testing and evaluation efforts (nominally 0.03 to 0.05 inches thick). In order to minimize defects in the films, the coatings were allowed to flash and dry in between each application. Various methods were used to accelerate drying and prevent defect formation, including the use of heated molds, air drying, vacuum, and heat lamps. Final sample compositions were cured in accordance with the manufacturer's recommendations.

High temperature fluid aging experiments were conducted on the liquid systems as previously described using methods outlined in ASTM D 471: *Test Method for Rubber Property - Effects of Liquids*. The fluid aging experiments were performed in for 3 day and 28 day periods with samples fully immersed in JP8+100 at 225ºF. Volume swell, weight gain and hardness change measurements were performed. All tests were performed in triplicate using test samples that were die-cut from the

cured sample plaques. Care was taken to cut test samples from defect free regions of the film. The results of the fluid aging experiments are presented in the following tables:

- Table 15. D471 Results – 3 Day Aging in JP8+100 @ 225°F
- Table 16. D471 Results – 28 Day Aging in JP8+100 @ 225°F.

Of the nine FKM materials tested, only two (S1 and S4) demonstrated satisfactory performance in the 3 and 28 day fluid aging experiments. Both of these systems are aqueous dispersions that use a chemical accelerator to promote cure. From a processing standpoint, the primary problem with the FKM materials is that they were difficult to spray in a consistent manner without routine clogging the nozzle of the spray gun. This would have a significant impact on production efficiency during fuel cell manufacturing. Furthermore, being water based systems, these materials were slow to dry and external heating was needed to drive water off in a reasonable amount of time between coatings. However, void formation was still a common issue when working with these materials. Vacuum assisted drying produced the best samples. Other drawbacks to the use of fluorolatexes include cost ($500 + per gallon) and density.

Both of the polyurethane samples demonstrated excessive hardness after aging in JP8+100. The HNBR materials showed mixed results, with S13 exhibiting the best overall performance. The CNBR samples performed reasonably well in the 3 day experiments, with S16 being the better performer of the two CNBR systems tested. The NBR (S-15) sample demonstrated excessive volume swell.

Two of the HNBR materials (S-13 and S14) are latexes, the only difference between the two being solids content; S-13 had a viscosity of 6.6 cPs and a solids content of 32%, while S-14 had a viscosity of 13.6 cPs and a solids content of 41%. The hardness of S-14 was significantly less than S-13, possibly due to factors affecting cure and crosslink density at the higher solids content. The HNBR latexes were difficult to process as the suspensions were not stable, so constant agitation was required to prevent settling. Like the fluorolatexes, spraying was found to be difficult due to clogging of the spray gun nozzle, and the formation of void free samples continued to be a problem. Numerous modifications were made to the HNBR latexes in an effort to improve their spraying characteristics and performance, including adding additional carbon black, surfactants, peroxides, and resorcinol, but little improvement was made over the commercial formulations.

The solvent based HNBR formulations (S-6 and derivatives) were easy to spray and formed nice, functional films. However, the solutions were fairly dilute, so VOC content would be a concern. Interestingly, the solvent based system did not demonstrate improved performance in the high temperature fluid aging experiments relative to the latex dispersions. Various blends of acetone, MEK and MIBK were evaluated with HNBR. Acetone is a strong solvent for HNBR but it flashes too readily. MEK and MIBK are medium and slower drying solvents, which can be used to control flow, leveling and film formation. While these are not environmentally friendly solvents, they are good solvents for HNBR and served as a good starting point for supporting the liquid formulation development efforts. While film formation characteristics could be improved, little effect of the physical performance or chemical resistance of these blends was noted.

The use of environmentally solvent systems to support the use of liquid HNBR systems was also evaluated, but the results were limited. Candidate solvents were selected based on solubility models, and included diacetone alcohol, tetrahydrofuran, tetrahydrofuran diacetate, ethylene glycol diacetate and ethylene glycol monomethyl ether. Diacetone alcohol was found to be the best solvent for HNBR, followed by tetrahydrofuran diacetate and ethylene glycol diacetate. Glycol ethers, when present in small quantities, provided enhanced coalescence and film forming tendencies. However, the ability to achieve proper film formation tended to be an ongoing issue with these systems, especially when carbon black was incorporated into the formulations, something that is a necessity to form proper film strength and chemical resistance.

Additional liquid system development efforts focused on the development of reactive blends of modified nitrile rubber (CNBR and HNBR) and epoxy terminated polysulfide. Polysulfide was selected as a modifying rubber because its exhibit excellent low temperature flexibility, high temperature stability and good chemical resistance. Epoxy terminated polysulfides are prepared by reacting a molar excess of epoxy resin with liquid polysulfide.

While HNBR and CNBR latexes were readily available to support the formulation development efforts, polysulfides are not dispersible in water. To address this, METSS added a hydrophilic oligomer tail (a monoamine terminated polyether) to a fraction (about 15% of the available end groups) of the epoxy termination sites on the polysulfide chains to produce an emulsifiable epoxy terminated polysulfide.

Various percentages of CNBR and HNBR latexes were blended with the epoxy terminated polysulfide emulsions. Films were cast from the blended solutions, dried and cured at 250°F. Test samples that form tack-free, uniform films and exhibited good apparent strength were subjected to 3 and 28 day fluid aging in JP8+100 at 225°F. However, out of approximately 50 formulations prepared, only a few demonstrated volume swell of less than 20% on a consistent basis during the high temperature fluid aging experiments.

Table 15. D471 Results (Liquid Systems) – 3 Day Aging in JP8+100 @ 225°F

Material ID	Class		ΔV (%)	ΔM (%)	Initial Hardness	Hardness (3 days)
S1	FKM	Mean:	4.60	2.87	66.44	62.67
		Std. Dev:	19.27	1.22	2.13	1.73
S2	FKM	Mean:	-2.13	-1.47	72.89	79.00
		Std. Dev:	0.50	0.05	2.15	1.12
S3	FKM	Mean:	46.33	4.26	68.78	64.22
		Std. Dev:	9.96	0.28	1.72	2.91
S4	FKM	Mean:	8.30	1.23	76.89	76.89
		Std. Dev:	2.73	0.57	2.15	2.85
S5	FKM	Mean:	24.36	2.69	70.44	66.56
		Std. Dev:	26.89	20.75	1.67	1.33
S6	HNBR	Mean:	12.31	23.21	70.67	63.44
		Std. Dev:	3.69	1.19	1.66	1.88
S7	PUR	Mean:	-2.74	-3.95	80.56	>90
		Std. Dev:	9.89	0.59	3.21	0.00
S8	PUR	Mean:	-3.20	-6.48	59.89	>90
		Std. Dev:	0.81	0.27	1.17	0.00
S9	FKM	Mean:	-0.96	2.28	76.67	80.11
		Std. Dev:	12.43	0.17	1.80	3.72
S10	FKM	Mean:	-2.60	-0.71	63.67	70.44
		Std. Dev:	8.05	0.28	2.40	5.41
S11	FKM	Mean:	24.39	4.45	69.22	67.78
		Std. Dev:	37.41	3.19	2.99	4.41
S12	FKM	Mean:	28.65	8.78	79.67	77.33
		Std. Dev:	4.08	1.46	0.50	4.12
S13	HNBR	Mean:	12.24	15.39	65.56	54.78
		Std. Dev:	9.09	0.84	2.01	2.39
S14	HNBR	Mean:	18.69	22.42	40.67	29.33
		Std. Dev:	1.79	0.70	0.87	2.12
S15	NBR	Mean:	48.37	29.13	57.56	55.22
		Std. Dev:	12.97	1.55	1.24	1.30
S16	CNBR	Mean:	6.73	19.22	61.00	63.67
		Std. Dev:	nd	nd	8.08	1.53
S17	CNBR	Mean:	19.91	15.01	60.78	53.33
		Std. Dev:	4.87	0.83	3.77	4.42

Table 16. D471 Results (Liquid Systems) – 28 Day Aging in JP8+100 @ 225°F

Material ID	Class		ΔV (%)	ΔM (%)	Initial Hardness	Hardness (28 days)
S1	FKM	Mean:	12.21	5.23	66.44	59.22
		Std. Dev:	10.34	1.33	2.13	2.49
S2	FKM	Mean:	-5.52	0.10	72.89	76.56
		Std. Dev:	2.49	0.32	2.15	1.59
S3	FKM	Mean:	30.59	7.35	68.78	61.00
		Std. Dev:	7.10	0.88	1.72	2.06
S4	FKM	Mean:	4.94	0.63	76.89	75.00
		Std. Dev:	5.24	0.65	2.15	3.16
S5	FKM	Mean:	15.76	2.48	70.44	65.56
		Std. Dev:	3.95	1.41	1.67	1.13
S6	HNBR	Mean:	16.00	17.26	46.22	30.78
		Std. Dev:	25.78	21.39	3.87	2.91
S7	PUR	Mean:	-17.76	-23.82	80.56	>90
		Std. Dev:	5.56	0.89	3.21	0.00
S8	PUR	Mean:	-3.20	-6.48	59.89	>90
		Std. Dev:	0.81	0.27	1.17	0.00
S9	FKM	Mean:	3.81	4.26	76.67	75.00
		Std. Dev:	18.69	9.13	1.80	4.12
S10	FKM	Mean:	nd	nd	63.67	73.56
		Std. Dev:			2.40	8.41
S11	FKM	Mean:	21.33	13.73	69.22	61.67
		Std. Dev:	32.10	17.52	2.99	0.71
S12	FKM	Mean:	26.20	9.33	79.67	73.22
		Std. Dev:	9.82	19.31	0.50	1.72
S13	HNBR	Mean:	8.23	12.21	65.56	57.78
		Std. Dev:	7.18	2.08	2.01	1.20
S14	HNBR	Mean:	24.17	24.93	40.67	30.89
		Std. Dev:	3.68	1.44	0.87	3.22
S15	NBR	Mean:	34.41	24.87	57.56	75.44
		Std. Dev:	30.46	20.39	1.24	1.51

nd = not determined

4.3 Final Selection of Rubber Inner Liner Materials

As the liquid development efforts were not successful in providing new candidate materials suitable for the proposed applications, final selection of the inner liner materials focused on the calendared systems. Of the 22 good and best performing materials listed in Tables 9 and 10, sixteen are based on fluoropolymer chemistry (including one fluorosilicone). While resistant to high temperature fuels, these materials are too expensive and too heavy to be used effectively as fuel cell bladder materials. The fluorosilicone is not strong enough for the proposed application. This essentially leaves two HNBR materials (8 and 26), one NBR (30) and three epichlorohydrin materials (7, 27, and 47). The two HNBR materials were provided from the same supplier and are derivatives of the same basic rubber chemistry. The same comment applies to ECO samples 7 and 27. ECO 47 from was from an aftermarket supplier that could not provide the raw materials needed to support subsequent program efforts, so ECO 47 was dropped from testing. The NBR material, while demonstrating good performance in the high temperature screening studies, was not pursued as nitrile rubbers are known to fail in the present application. The main physical difference between HNBR samples 8 and 26 and ECO samples 7 and 27 were hardness, with samples 26 and 27 being the softer of the formulations.

Based on their overall performance, METSS selected these two materials to support the additional program development, testing and evaluation efforts. As the sample material evaluated under the program efforts were provided by the rubber supplier as cured plaques of sample material, before these materials could be used to support the fuel cell development efforts, METSS had to develop internal formulations based on these two raw materials and retest and re-qualify these formulations against the program requirements.

The product formulations used to support the inner liner production, testing and evaluation efforts are provided in Tables 17 and 18 for the final HNBR and ECO formulations, respectively. The HNBR material has an acrylonitrile content of 44%; 91% unsaturated; iodine number of 24; and Mooney viscosity between 71 and 85. The ECO in material has a Mooney viscosity of 72 and a chlorine content of 25.1% by weight.

Table 17. Formulation of HNBR Gum Inner Liner Material

Material	Parts by Weight
HNBR rubber	100.00
N762 – carbon black	55.00
PlastHall P7068 - plasticizer	5.00
Naugard 445 – antioxidant	1.50
Vanox MTI – stabilizer	1.00
Aflux 42 – processing aid	2.00
Maglite D – magnesium oxide	3.00
Saret SR-517 – acrylic co-agent	6.00
Vulcup 40KE – peroxide	8.00
Total	181.50

Table 18. Formulation of ECO Gum Inner Liner Material

Material	Parts by Weight
ECO rubber	100.00
N550 – carbon black	60.00
TP95 – alkoxyethyl adipate	5.00
Naugard 445 – antioxidant	1.00
Vanox MTI – stabilizer	0.50
Aflux 42 – processing aid	2.00
Calcium Carbonate – filler	5.00
Maglite D – magnesium oxide	3.00
Zisnet F-PT – triazine curing agent	0.90
DPG – diphenylguanidine -accelerator	0.30
Vulkalent E/C – retarder	0.90
Total	178.60

The procedure used to make these compounds was as follows:

- Preheat a Banbury mixer to 150°F (65.5°C), set the rotor speed to 390 rpm
- Add all ingredients to 75% fill, apply a ram pressure of 20 psi
- When internal temperature reaches 200°F, raise and lower ram to vacate gases
- When temperature reaches 250°F sweep
- Dump mixture onto roll mill when temperature reaches 300°F
- The compound is then roll milled at 122 F with three passes through the nip
- The compound is cooled and then reintroduced to the Banbury mixer at 100°F, 280 rpm, 20 psi ram pressure, at the same fill factor of 75%
- The Banbury is swept at 200°F, and the mixture dumped at 220°F
- The dumped mixture is roll milled at 122°F with five passes through the nip.

The final compounds were molded and cured into test slabs approximately 6 x 6 x 0.080 inches thick for testing and evaluation. Both materials were cured by heating for 20 minutes at 350°F in a compression press. The properties of the internally compounded gums (before and after fluid aging for 3 and 28 days in JP8+100 at 225°F) are provided in Tables 19, 20 and 21 for D471 fluid aging, tensile properties and tear properties, respectively.

As a final measure, stoved and nonvolatile gum residue testing was performed to ensure compliance with the existing military performance specifications for fuel cell bladders (Table 22). Stoved and nonvolatile gum residue tests were conducted in accordance with methods outlined in ASTM D-381: *Standard Test Method for Gum Content in Fuels by Jet Evaporation*. According to MIL-T-6396 and MIL-T-27422, Stoved Gum Residue of bladder materials should be less than 20 mg/100 ml, and the Nonvolatile Gum Residue should be less than 60 mg/100 ml. These tests were performed by Texas Oiltech Labs. Both inner liner rubber materials met this requirement without any problem.

Table 19. Inner Liner Rubber, D471 Results – Fluid Aging in JP8+100 @ 225°F

METSS ID		ΔV (%)	ΔM (%)	Initial Hardness	Final Hardness
HNBR	3 Days	8.27	4.21	78	75
	28 Days	6.51	3.88	78	75
ECO	3 Days	8.45	3.83	75	71
	28 Days	0.24	0.66	75	66

Table 20. Inner Liner Rubber, Tensile Properties – Fluid Aging in JP8+100 @ 225°F

Material ID		Unaged		Fluid Aged	
		Tensile Strength (psi)	Elongation @ Break (%)	ΔTensile Strength (%)	ΔElongation @ Break (%)
HNBR	3 Days	3676	2529	-22.4	-12.7
	28 Days			-69.8	-65.6
ECO	3 Days	1518	1155	-28.5	-22.7
	28 Days			-46.6	-41.7

Table 21. Inner Liner Rubber, Tear Properties – Fluid Aging in JP8+100 @ 225°F

Material ID		Unaged (lb/in)	%Change After Aging
HNBR	3 Days	156.1	-29.0
	28 Days		-29.0
ECO	3 Days	206.4	-36.3
	28 Days		-52.1

Table 22. Inner Liner Rubbers – Stoved and Nonvolatile Gum Residue

Material ID	Stoved Gum Residue (mg/100 ml)	Nonvolatile Gum Residue (mg/100 ml)
HNBR	< 1.0	24.8
ECO	1.1	30.9

4.4 Fabric Reinforced Inner Liners

Once the performance of final inner liner rubber formulations was verified, fabric reinforced inner liner materials were prepared for testing and evaluation. The inner liners were prepared by calendaring the uncured HNBR and ECO rubber materials onto both sides of a nylon reinforcing fabric through contact in the nip of a pair of calendaring rolls. The nylon fabric used in the construction of the inner liner had a density of 3.8 oz/yd^2, made from 210 denier fibers in a 78 by 61 plain weave construction. The nylon fabric was treated with resorcinol formaldehyde latex to promote adhesion to the rubber prior to the calendaring process. The final fabric reinforced laminates were cured into 0.080 inch thick sections in a press at 350°F to support the inner liner testing and evaluation portions of the program.

D471 fluid aging experiments were performed on the fabric reinforced inner liner materials to evaluate the effect of the fabric reinforcement on volume swell and weight gain. The results of the 28 day fluid aging experiments in JP8+100 are presented in Table 23.

Fabric inner liner strength was determined in accordance with methods outlined in Federal Test Method Standard No. 191, Method 5100. The results are presented in Table 24. Both samples were tested before and after aging in JP8+100 at 225°F for 3 and 28 days. The military performance specifications provide for a maximum change in fabric inner liner strength of 20% change after 3 days of fuel aging. Both inner liner materials performed well in this test.

The tensile properties of the fabric reinforced inner liner materials were also determined after high temperature humidity exposure for a period of 30 days at 95% RH and 160°F. Tensile properties were determined using methods provided in ASTM D751: *Standard Test Methods for Coated Fabrics*. Allowable property changes after high temperature humidity exposure are ±45% tensile, and ±30% elongation. The results are presented in Table 25, with both samples performing very well.

Seam strength testing was performed according to Federal Test Method Standard No. 601, Method 8011 to determine the ability of the fabric reinforced inner liner materials to bond to themselves. The adhesion strength of the cured inner liner rubber to the nylon fabric was also evaluated, using methods outlined in ASTM D 413: *Adhesion to Flexible Substrates (Rubber to Fabric).* Both sets of measurements were performed before and after aging in JP8+100 at 225ºF for 3 and 28 days. The performance specifications require no less than 6 lbs/inch seam strength or, for seam strength, that

tension failure of samples containing seams perpendicular to the direction of load occur in the rubber and not at the seam. The results of the seam strength testing and adhesion testing of the gum inner liners to the nylon are presented in Tables 26 and 27 respectively. Both of the inner liner materials demonstrated excellent performance with respect to both tests, even after 28 days of immersion in JP8+100 at 225°F. However, the adhesion of the HNBR rubber material to the treated nylon fabric passed by only a slight margin.

The low temperature flexibility of the fabric reinforced inner liner was determined using methods outlined in ASTM D2137: *Standard Test Methods for Rubber Property-Brittleness Point of Flexible Polymers and Coated Fabrics.* This test entails striking cantilever samples cooled to -65°F and -40°F and examining the samples for evidence of delaminating or cracking. The results of this test are reported on a pass/fail basis for 5 replicate tests. Samples were tested before and after 3 days of aging in JP8+100 at 225°F. The results reported in Table 28 indicate the number of passes out of each of the five tests for each test condition. The HNBR fabric reinforced inner liners materials performed exceptionally well, even at -65°F. However, the while the ECO inner liners may perform adequately at -40°F, they demonstrate a substantial loss in flexibility at -65°F. While this may not be an issue with JP8+100, it may pose a problem with low operating temperature fluids.

Table 23. Inner Liner D471 Results – Fluid Aging in JP8+100 @ 225°F

METSS ID	ΔV (%)	ΔM (%)	Initial Hardness	Hardness (28 days)
HNBR	7.66	3.97	83	83
ECO	6.75	1.49	82	81

Table 24. Fabric Inner Liner Strength – Fluid Aging in JP8+100 @ 225°F

Material ID		Unaged		Fluid Aged	
		Tensile Strength (psi)	Elongation @ Break (%)	ΔTensile Strength (%)	ΔElongation @ Break (%)
HNBR	3 Days	3457	48.6	-8.9	-20.4
	28 Days			-5.6	-23
ECO	3 Days	3688	48.9	-3.9	-17.3
	28 Days			-35.3	-16.8

Table 25. Fabric Inner Liner Strength – 30 Days, 95% RH, 160°F

Material ID	Unaged		Fluid Aged	
	Breaking Strength (psi)	Elongation @ Break (%)	ΔTensile Strength (%)	ΔElongation @ Break (%)
HNBR	2482	158.3	5.0	0.76
ECO	2423	39.4	-3.5	-7.0

Table 26. Seam Strength of Inner Liner Materials – Before and After Fluid Aging

Material ID		Unaged Peel Strength (lb/in)	After Aging Peel Strength (lb/in)
HNBR	3 Days	42.17	31.58
	28 Days		26.42
ECO	3 Days	106	73.29
	28 Days		23.83

Table 27. Inner Liner Adhesion to Nylon – Before and After Fluid Aging

Material ID		Unaged Peel Strength (lb/in)	After Aging Peel Strength (lb/in)
HNBR	3 Days	8.54	6.9
	28 Days		7.04
ECO	3 Days	Rubber tore	Rubber tore
	28 Days		14.63

Table 28. Low Temperature Flexibility

Material ID	Test Temperature	Unaged	After 3 Days in JP8+100 @ 225°F
HNBR	-40°F	5/5 pass	5/5 pass
	-65°F	5/5 pass	5/5 pass
ECO	-40°F	3/5 pass	5/5 pass
	-65°F	1/5 pass	0/5 pass

4.5 Inner Liner with Barrier Material

Three additional tests were performed on the fabric reinforced inner liner materials coated with the Nycote® 7-11 nylon fuel vapor barrier layer. These tests included vapor barrier testing, adhesion of the nylon coating to the inner liner material and stress aging.

METSS performed permeation measurements on each of the inner liner materials with and without the nylon barrier film to see if either of the candidate materials demonstrated inherent resistance to fuel vapor permeation. Permeation measurements were performed using methods outlined in MIL-T-6396, Section 4.6.12. According this specifications, the room temperature permeation rate of JP8+100 should be less than 0.025-fl.oz./ft²/day for each of the replicates tested (this is a pass/fail test). Permeation results are presented in Table 29. The Nycote® vapor barrier coating provides a definite benefit.

The adhesion strength of the Nycote® vapor barrier coating to the HNBR and ECO inner liner materials was determined before and after aging in JP8+100 at 225ºF for 3 and 28 days. All measurements were performed using methods outlined in ASTM D 413: *Adhesion to Flexible Substrates (Rubber to Fabric).* The results are presented in Table 30. In all cases, the adhesion of the Nycote® to the inner liner materials is less than the 6 lbs/inch required to be consistent with the other fuel cell peel strength requirements. Poor adhesion of the Nycote® to the high temperature inner liner materials could result in delamination of the remaining composite cell structure. While this is a critical issue, based on discussion with fuel cell manufacturers, this result is not uncommon for the configuration tested. In existing fuel cell manufacturing operations there are adhesive agents that promote the bonding of the Nycote® layer to subsequent rubber layers. As such, this is not considered a significant problem with the HNBR and ECO inner liner materials developed under this program as this issue is commonly addressed in current fuel bladder production operations.

Finally, stress aging testing of the fabric reinforced inner liner materials (with barrier coating) was conducted to support MIL-T-24722B qualification. This test involves exposing folded samples (10 each) to JP8+100 for 7 days at 160°F and examining samples for evidence of blistering, cracking, separation, or other material failure. This is a pass/fail test, with no evidence of degradation of the samples permitted after 7 days of aging. None of the ECO samples exhibited evidence of degradation after stress aging testing. One out of ten HNBR samples demonstrated slight evidence of surface cracking during stress aging, with the remaining nine samples showing no evidence of degradation.

Table 29. Permeation of JP8+100 Through Inner Liner Materials

METSS ID	Diffusion Rate (oz/ft^2-24hrs)	
	Uncoated	With Nycote®
HNBR	0.0617 fail	0.0042 pass
ECO	0.0017 pass	0.0044 pass

Table 30. Nycote® Adhesion to Inner Liner Materials – Before and After Fluid Aging

Material ID		Unaged Peel Strength (lb/in)	After Aging Peel Strength (lb/in)
HNBR	3 Days	0.40	3.27
	28 Days		3.31
ECO	3 Days	1.14	0.19
	28 Days		0.69

4.6 Composite Wall and Fuel Cell Testing

Once the inner liner materials are qualified, the remaining structure of the fuel cell remains unchanged, so substitution of the new high temperature resistant inner liner materials into existing fuel cell designs should not affect the performance of the rest of the fuel cell design. As such, the replacement inner liner materials may be qualified by inference. The same goes for the fully constructed fuel cell, provided the inner line material demonstrates high temperature resistance to JP8+100. However, to verify this, METSS arranged additional testing to be supported by the fuel cell manufacturing community.

In support of this effort, METSS provided a number of samples of both inner liner materials and uncured rubber gums to a commercial fuel cell manufacturer to support sample preparation efforts and testing and evaluation efforts. Testing and evaluation efforts were to include testing of the composite samples by the fuel cell manufacturer as well as METSS. The test jigs required to support internal testing efforts were constructed by METSS in accordance with the guidelines provided in the military performance specifications.

After preparing and supplying numerous samples to support the construction of composite wall samples for internal and external testing and evaluation, METSS was finally forced to suspend all efforts related to this task due to lack of directed support from the fuel cell manufacturing community and sufficient program funding to continue to support the materials development efforts. However, while supporting these efforts, it was determined that a set of modified HNBR and ECO formulations should be developed to support production operations that require longer processing times or lower cure temperature materials.

The target cure temperature for the HNBR formulation was 250°F. To achieve this, METSS reformulated the HNBR inner liner material utilizing a sulfur cure system to replace the higher temperature (350°F) peroxide cure system as originally designed. The sulfur cure HNBR formulation is presented in Table 31. Additional testing was performed on the revised HNRB formulation to verify performance against JP8+100. Longer processing times were required for the ECO formulation. The original ECO formulation was cured via a triazine compound (Zisnet FP-T) which had an accelerator (DPG) added, which resulted in premature curing during storage. A new ECO formulation was developed that did not contain an accelerator (Table 32).

Table 31. Sulfur Cure HNBR Formulation

Material	Parts by Weight
HNBR	100
N762 carbon black	55
PlastHall P7068	5
Zinc Oxide	5
Stearic acid	1
Naugard 445	1
Vanox MTI	0.5
Sulfur (325 mesh)	1.5
Altax MBTS	1.5
TMTM	0.2
Total	170.7

Table 32. Unaccelerated ECO Formulation

Material	Parts by Weight
ECO	100
N550 black	60
TP95	5
Calcium carbonate	5
Naugard 445	1
Vanox MTI	0.5
Zisnet FP-T	0.9
Vulcalent EC	0.9
Aflux 42	1
Total	174.6

5.0 SUMMARY

The results of this program demonstrate several classes of rubber that are suitable for high temperature fuel cell applications with an upper used temperature of at least 225°F, specifically in the presence of JP8+100. Of these materials, HNBRs and ECOs represent the most cost effective classes of materials demonstrating high temperature resistance to JP8+100.

The performance of specific formulations based on each of these systems was demonstrated against the program requirements and the applicable military performance specifications for fuel cell materials. A specific peroxide-cured inner liner material HNBR formulation for calendared bladder systems is provided in Table 17, while a triazine cured ECO compound formulation is provided in Table 18. A sulfur-cured HNBR counterpart is provided in Tables 31. An revised ECO compound is presented in Table 32 to support longer processing times.

Summary data for the HNBR and ECO inner liner materials evaluated under this program are presented in Tables 33 and 34, respectively. The only limitation noted in the performance of these inner liner materials was poor bonding with the Nycote® vapor barrier coating. However, this did not affect the performance of the coating and, as noted in the discussion, this issue is commonly dealt with during commercial fuel cell manufacturing. The primary difference between the ECO and the HNBR inner liner materials is low temperature performance capability. The HNBR systems demonstrated low temperature flexibility down to -65°F while the ECO materials demonstrated marginal performance, even at -40°F.

Program efforts related to the development of liquid systems to support the production of high temperature fuel cell bladders by spraying demonstrated limited results during the materials development trials. Industry interest is needed to direct and support these efforts so they can be targeted at and supported by existing manufacturing operations.

The inner liner materials developed under this program can be provided as ready to calendar gums or fully calendared (but uncured) inner liner materials to support full qualification trails and manufacturing efforts.

Table 33. Summary Data - HNBR Formulation

Test Standard	Test Description	Test Requirement	HNBR Results
ASTM D471 - *Effect of Liquids*	Tested after aging - 28 days in JP8+100 @ 225°F	Volume Swell, < 10 %	3.0 %
		Mass Absorption, <5 %	3.88 %
		Hardness Change, max ± 10	-3
ASTM D412 - *Tensile Strength*	Retention of properties, before and after aging 3 days in JP8+100 @ 225°F	>50 %	77.6 %
ASTM D624 - *Tear Strength*	Retention of properties, before and after aging 3 days in JP8+100 @ 225°F	>50 %	71.0 %
ASTM D381 - *Nonvolatile Gum Residue*	tested cured inner liner material in JP8+100	< 60 mg/100ml	24.8
ASTM D381 - *Stoved Gum Residue*	tested cured inner liner material in JP8+100	< 20 mg/100ml	<1.0
FED-STD-191, method 5100 - *Fabric Inner Liner Strength*	Retention of properties, before and after aging 3 days in JP8+100 @ 225°F	>80 %	91.9 %
ASTM D751 - Standard Test *Properties in Tension*	Retention of properties, before and after aging 30 days @ 160°F, 95% RH	Change ± 45 %	+5.0 %
FED-STD-601, method 8011 - *Seam Adhesion*	Tested after aging - 28 days in JP8+100 @ 225°F	Peel Strength, >6 lbs/in	26.4
ASTM D413 - *Inner Liner Adhesion to Fabric*	Tested after aging - 28 days in JP8+100 @ 225°F	Peel Strength, >6 lbs/in	7.04
MIL-T-6396E Section 4.6.12 - *Permeability with Nycote®*	Permeation test, 8 days at RT	<0.0250 oz/ft^2-24hrs	0.0042
FED-STD-601, method 8011 - *Inner Liner Adhesion to Sealant*	Tested after aging - 28 days in JP8+100 @ 225°F	Peel Strength, lbs/in	3.31
ASTM D3127 - *Low Temperature Brittleness*	Tested after aging - 3 days in JP8+100 @ 225°F	No evidence of cracking at -40°F	Pass
MIL-T-24722B - *Stress Aging*	Tested after aging - 7 days in JP8+100 @ 160°F	No evidence of cracking	Pass

Table 34. Summary Data - ECO Formulation

Test Standard	Test Description	Test Requirement	ECO Results
ASTM D471 - *Effect of Liquids*	Tested after aging - 28 days in JP8+100 @ 225°F	Volume Swell, < 10 %	0.24 %
		Mass Absorption, <5 %	0.66 %
		Hardness Change, max ± 10	-9
ASTM D412 - *Tensile Strength*	Retention of properties, before and after aging 3 days in JP8+100 @ 225°F	>50 %	71.5 %
ASTM D624 - *Tear Strength*	Retention of properties, before and after aging 3 days in JP8+100 @ 225°F	>50 %	63.7 %
ASTM D381 - *Nonvolatile Gum Residue*	tested cured inner liner material in JP8+100	< 60 mg/100ml	30.9
ASTM D381 - *Stoved Gum Residue*	tested cured inner liner material in JP8+100	< 20 mg/100ml	1.1
FED-STD-191, method 5100 - *Fabric Inner Liner Strength*	Retention of properties, before and after aging 3 days in JP8+100 @ 225°F	>80 %	96.1 %
ASTM D751 - Standard Test *Properties in Tension*	Retention of properties, before and after aging 30 days @ 160°F, 95% RH	Change ± 45 %	-3.5 %
FED-STD-601, method 8011 - *Seam Adhesion*	Tested after aging - 28 days in JP8+100 @ 225°F	Peel Strength, >6 lbs/in	23.8
ASTM D413 - *Inner Liner Adhesion to Fabric*	Tested after aging - 28 days in JP8+100 @ 225°F	Peel Strength, >6 lbs/in	14.63
MIL-T-6396E Section 4.6.12 - *Permeability with Nycote®*	Permeation test, 8 days at RT	<0.0250 oz/ft^2-24hrs	0.0044
FED-STD-601, method 8011 - *Inner Liner Adhesion to Sealant*	Tested after aging - 28 days in JP8+100 @ 225°F	Peel Strength, lbs/in	0.69
ASTM D3127 - *Low Temperature Brittleness*	Tested after aging - 3 days in JP8+100 @ 225°F	No evidence of cracking at -40°F	Pass
MIL-T-24722B - *Stress Aging*	Tested after aging - 7 days in JP8+100 @ 160°F	No evidence of cracking	Pass