PROPULSION AND VEHICLE
ENGINEERING LABORATORY

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MONTHLY PROGRESS REPORT

For Period

March 1, 1966, Through March 31, 1966

FOR INTERNAL USE ONLY

GEORGE C. MARSHALL SPACE FLIGHT CENTER
HUNTSVILLE, ALABAMA

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

MSFC - Form 1262 (September 1961)

PROPULSION AND VEHICLE ENGINEERING LABORATORY

MPR-P&VE-66-3

MONTHLY PROGRESS REPORT

(March 1, 1966, Through March 31, 1966)

Advanced Studies Office
Vehicle Systems Division
Propulsion Division
Structures Division
Materials Division

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GEORGE C. MARSHALL SPACE FLIGHT CENTER

PR-P&VE-A-66-3

MONTHLY PROGRESS REPORT
ADVANCED STUDIES OFFICE
(March 1, 1966, Through March 31, 1966)

SATURN V

I. Saturn V/Voyager

A status review of the Saturn V/Voyager shroud size study was presented to JPL personnel on March 3, 1966. The final review is scheduled to be presented to JPL the week of April 18, 1966. This will allow a report of the study results to be given at the Voyager Quarterly Review meeting scheduled for the week of April 25, 1966, at NASA Headquarters.

II. LEM Lunar Logistics

Work is continuing on the feasibility of incorporating a LEM/Truck in the Apollo spacecraft configuration. Weight tables were generated which indicate the weights associated with each spacecraft maneuver, from translunar injection to lunar touchdown. The size of a lunar base which may be built up by such a mission is presently under consideration.

Effort is also continuing on the extension of the above study which involves refueling of the Saturn V Lunar Logistics configuration in earth orbit by Saturn IB tanker vehicles. Work completed on this study includes the following: (1) estimates of payload requirements in earth orbit for different net payloads on the LEM/Truck delivered to the lunar surface; (2) estimates of tanker requirements for refueling the Saturn V S-IVB stage; and (3) some conceptual design on the Saturn IB propellant tankers. This study is scheduled to be completed by July 1966.

Efforts continued on the feasibility study of transporting two LEM/Truck vehicles on a Saturn V launch vehicle. A configuration drawing was furnished R-P&VE-S for shroud weight calculations. Vehicle weight summaries were prepared and furnished R-AERO-X for performance, control, and load analyses.

APOLLO APPLICATIONS PROGRAM

I. Earth Orbital

A. S-IVB Workshop (AS-209 Design)

A draft of the Experiment Development Proposal prepared by MSFC was reviewed and coordinated with MSC on March 7, 1966, and a preliminary copy submitted to NASA Headquarters on March 22, 1966. A presentation was given to the Manned Space Flight Experiments Board (MSFEB) on March 21, 1966. Currently, assistance is being given to NASA Headquarters in preparing a work statement on contracted studies for an air lock slice.

Mass characteristics and preliminary configuration of the AS-209 S-IVB Workshop were published for both 14-day and 30-day missions, incorporating the proposed McDonnell air lock slice, in memorandum R-P&VE-AA-66-43, "Preliminary Mass Characteristics of the S-IVB Orbital Workshop," dated March 10, 1966, and in memorandum R-P&VE-AA-66-53, "Preliminary Mass Characteristics of the S-IVB Orbital Workshop (30-Day Mission Lifetime)," dated March 22, 1966.

B. Advanced Orbital Systems

Study plans and task directives have been prepared for a four-month in-house study to define a logical evolutionary program for S-IVB stage utilization in orbit. An Advanced Systems Office/NASA Headquarters work statement for a nine-month contract study, to begin in July 1966, is being reviewed.

C. Extra Vehicular Engineering Activity (EVEA)

Advanced Systems Office work statements have been reviewed for planned EVEA contract studies to be initiated by July 1966. These studies include experiment definition, satellite and material recovery,

material and personnel maneuvering and rescue support activities. This Office is providing in-house design support and panel member support for some of these studies. Plans and technical directives have been developed for a five-month in-house study on EVEA equipment concepts and designs.

II. Lunar Surface

A. Local Scientific Survey Module (LSSM)

The mid-term presentations of the LSSM studies were held at Bendix on March 7-8, 1966, and at Boeing on March 9-10, 1966. Data were presented on primary study configurations and on several minimum-cost mobility systems. Bendix demonstrated their Mobility Test Article (MTA), and the compatibility of their LSSM Mock-up with an astronaut in a hard suit.

Results of the Army Corps of Engineers' study of a lunar conversion of the M-274 Army "Mule" vehicle show the vehicle to be very similar to the LSSM.

B. Advanced Systems Contracts

A status review of the Mobility Systems Comparison and Evolution Study (MOBEV) was held at Bendix on March 7, 1966. Detailed plans for the MOBEV methodology were presented.

A meeting was held with LMSC personnel to discuss the transportation systems to be utilized in the Mission Modes and Systems Analysis (MIMOSA) program. Launch vehicles with capabilities (injected into a translunar trajectory) of 95 K, 125 K, 150 K, and 200 K pounds are being considered. LMSC is also considering both lunar orbit rendezvous and direct landing modes, delivering three to seven men to the lunar surface with a six-month rotation time.

The contractor orientation for the Early Lunar Shelter study was held on March 8, 1966. The Garrett Corporation presented an outline of effort proposed for the six-month study.

III. Apollo Applications Program Integration

A two-week in-house study to establish the feasibility of integrating 16 selected experiments into the LEM Lab and CSM Experiment Pallet was performed in connection with Dr. Mueller's request that MSFC and MSC concern themselves with the need for developing all the proposed AAP experiment carriers. Results indicate that, individually, any of the experiments could be placed in either the LEM Lab or CSM Experiment Pallet; however, groups of experiments are considered more adaptable to the LEM Lab since certain cases exceed the capabilities of the CSM Experiment Pallet. The 16 experiments used were suggested by NASA Headquarters as representative and approved experiments.

Eighty-five selected AAP experiments from the AAP Experiments Data Bank have been catalogued and distributed to individuals involved in AAP experiment integration activity. Data on additional experiments will be catalogued and distributed during the next three months.

A computer program, which will provide a means of establishing the feasibility of experiment grouping for selected AAP flights, is being developed. A simplified version of this program is expected to be operational by early May 1966.

Modifications required to the S-IVB stage for performing the AAP synchronous missions SA-507, 509, and 513, the AAP polar mission SA-210, and the rendezvous mission SA-211 and 212 were presented to General Jones, NASA Headquarters, on March 14, 1966. Modifications reported ranged from none for the Apollo-type synchronous mission profiles to changes in the slosh baffling, tank pressurization, engine pneumatic control, auxiliary propulsion system, and to the Instrument Unit environmental control and gas supply systems for profiles requiring two restarts of the S-IVB stage. Maximum weight increase (2000 pounds) and cost ($5.35 million) were reported for the mission profiles requiring two restarts. It was emphasized that an immediate decision is required to incorporate necessary modifications in the S-IVB stage.

NUCLEAR ROCKET PROGRAM

I. Nuclear Ground Test Module Program

Two detailed studies of propulsion module radiation environment were completed. One study considers the engine system leakage to be represented as leakage from a number of point sources. Detailed

neutron and gamma leakage data through 18 spherical sector area strips 10 feet from the core center have been obtained. The second study provides detailed environment data for 70 small volume elements in the lower bulkhead region, supplementing information which has been released in memorandum R-P&VE-AV-66-21. Preliminary copies of both study results were distributed to the Nuclear Ground Test Module Task Force at the mid-term meeting, March 23, 1966.

II. Nuclear Flight Safety

Some final trajectories were obtained for the in-house launch azimuth study, and preliminary safety analyses are being performed.

Final reports from the funded contractor effort have still not been received. The contractor now has distribution scheduled for the week of April 18, 1966. A draft of the follow-on work statement was reviewed by NASA Headquarters and revisions were incorporated. Initiation of the follow-on effort is expected in late April 1966.

MISCELLANEOUS

I. Interplanetary Fly-by Studies

Efforts are continuing in the in-house investigation to define a feasible spacecraft configuration for a Mars/Venus fly-by mission.

Work statements were prepared for two 8-month study efforts to determine the feasibility of modifying the S-II and S-IVB stages for use as orbital injection stages for manned planetary fly-by missions. The work statements were approved by the Advanced Systems Office and forwarded to the Purchasing Office for submission to the study contractors: NAA for the S-II stage; DAC for the S-IVB stage. These studies will support the 12-month mission analysis contract which will investigate manned Mars and Venus fly-by missions using Saturn/Apollo systems with minimum modifications.

II. De-orbit Capsule Study (DOC)

Preliminary conceptual capsule designs of various shapes and sizes, both telescopic and non-telescopic, were generated to determine the minimum size DOC that would be compatible with certain primary missions.

These capsules are being investigated to determine volume, weight, size, shape, and trade-offs of each based on rescue and retrieval restraints. A de-orbit capsule, which has variable volume (telescopic capability) and can be packaged in the CSM Experiment Pallet, is being investigated for use on these recovery missions. Capsule packaging inside the LEM adapter is still being considered. The first phase of the study is scheduled for completion by August 30, 1966.

III. Reusable Orbital Transport (ROT) Studies

A cursory investigation has been completed to determine new designs and preliminary concepts regarding sled slippers for a ground accelerator. To reduce the high friction during low ground acceleration speeds, an air bearing track has been suggested and is being investigated in more detail.

IV. LEM Descent Shelter

A study was initiated to investigate the feasibility of utilizing the LEM descent stage as a lunar shelter capable of supporting three men 14 days. Expandable structure is used inside the descent stage after certain assemblies, sub-assemblies, and interface structure are removed. Two concepts were considered: (1) keep the main cruciform and other primary descent structure intact; (2) remove portions of the cruciform structure and still allow the descent stage to support the ascent stage for lunar lift-off. The first study concept, leaving the descent stage intact, has been completed; study of the second concept is still in progress.

V. Lunar Backpack Jumper

Efforts are continuing in the preliminary definition and conceptual design of a propulsive system to serve as an "assist" unit to enable an astronaut to jump short distances on the lunar surface. Several parametric studies, including jump distance, burn time (including retro), propellant requirement, and optimum thrust-to-weight ratio, have been completed. A survey of storable propellants for the system has been completed. Detailed system design is now underway and should be completed by May 1966.

VI. Film Return Capsule Study

An in-house study has been initiated to determine the most promising conceptual design of a capsule capable of returning photographic films from either lunar or earth orbit. An investigation has been undertaken to define

the camera types and film sizes (length and width) that would be used in space for photographing regions of the moon or earth. The scheduled completion date for this initial study phase is August 15, 1966.

VII. Advanced Engine Characteristics

A six-month in-house study has been initiated to investigate the desirability of incorporating variable area ratio into advanced engine design concepts. Possible payload advantages will be determined for intermediate payload range vehicles with advanced engines in the first and second stages. The scheduled completion date for this study is September 1966.

VIII. Vehicle Performance and Design Handbook

The compilation in handbook form of computer programs necessary for conducting performance and design studies on advanced vehicles is proceeding satisfactorily. The following programs were completed during the third quarter, FY 1966:

(1) Vehicle flight loads computer program

(2) Ground wind load analysis program

(3) Mass characteristics

(4) Miscellaneous mass characteristics

(5) Propellant utilization program tandem tank (S-IC)

Charles L. Barkey

Erich E. Goerner
Chief, Advanced Studies Office

GEORGE C. MARSHALL SPACE FLIGHT CENTER

R-P&VE-V-66-3

MONTHLY PROGRESS REPORT
VEHICLE SYSTEMS DIVISION

(March 1, 1966, through March 31, 1966)

SATURN IB

I. S-IB Stage

A. S-IB CALIPS Checkout Console

Delivery of the S-IB Stage CALIPS checkout console has been rescheduled for June 1966 because of console modifications and procurement lead times.

B. Installation of the Linear Shaped Charge (LSC) Assemblies

To eliminate future occurrences of end adapters pulling off on installation of the LSC assemblies, a request was made that all adapter assemblies be subjected to a pull test of 35 $\pm$ 5 pounds and inspected per drawing 60C110209 prior to use on flight vehicles.

II. S-IVB Stage

A. Pneumatic Ground Support Equipment (GSE)

The LH2 supply valve of the gas heat exchanger, model DSV-4B-438, has consistently given trouble at launch complex (LC) 34. Douglas Aircraft Company (DAC) is currently testing valves from several manufacturers to obtain a replacement. Tests have started on a Parker valve which was used on the S-IV stage and therefore, has a history of use and reliability. The heat exchanger valve will be replaced as soon as a valve has been qualified for this use. It is believed that this replacement will take place before the SA-203 launch.

B. Auxiliary Propulsion System (APS) Servicer

An Engineering Change Proposal (ECP) which would relax the required flow measuring system accuracy in the acceptance test procedure from $\pm$0.5 percent to $\pm$1.5 percent has been reviewed and its approval recommended to the Saturn IB Vehicle Ground Support Equipment Office.

C. S-IVB Heat Exchanger Requirements

Interface Revision Notices (IRN's) reflecting facilities design requirements for the S-IVB Heat Exchanger were prepared against level "A" Interface Control Documents (ICD's) 65ICD8604 and 65ICD9404. These IRN's should provide criteria which will eliminate the back pressure problem (experienced on SA-201) on future launches.

D. DSV-4B-1862, Environmental Protective Kit

The environmental protective kit was flight tested on the super guppy during the transportation of the S-IVB dynamic stage to the west coast. The kit performed satisfactorily and limited the maximum negative internal-ambient pressure differentials to 0.02 p.s.i. for the LH_2 tank and 0.08 p.s.i. for the lox tank. These pressure differentials, which are well within allowable limits, were reached during a high rate of descent that exceeded normal descent rates.

E. DSV-4B-400, Battery Handling Kit

DAC has shipped the first unit of the battery handling kit to Kennedy Space Center (KSC). Although DAC proof loaded the kit, it was not bought off by MSFC. Therefore, it must be load tested at KSC before MSFC buyoff.

F. S-IVB/IU Stage Umbilicals

The following umbilical equipment was shipped to KSC on Friday, March 11, 1966:

S-IVB aft umbilical, model DSV-4B-355 (one each).

S-IVB aft umbilical, model DSV-4B-353 (two each).

S-IVB forward umbilical, model DSV-4B-354 (two each).

IU umbilical, 11M00004-1 (two each).

IU electrical eject plate, 11M00229 (three each).

III. Instrument Unit

A. Ground Support Cooling Unit (GSCU)

The first Chrysler Airtemp built GSCU's, units 9 and 10, successfully completed acceptance tests and were delivered to KSC for use on LC 37B. Due to long lead times involved, deviations were approved to use some commercial grade components in place of military grade. These will be replaced with military grade components as soon as they become available.

B. IU Pneumatic Console

Engineering change request (ECR) VMS-IB-009 was initiated to modify the ST-124M purge circuit to prevent high pressure backflow into the purge circuit and subsequently to the ST-124M housing.

IV. General

A. AS-201 Mass Characteristics

Mass characteristics flight evaluations for AS-201 were completed. Chrysler Corporation Space Division (CCSD) Michoud, performed a parallel effort for this vehicle. Since this effort was performed satisfactorily, the vehicle flight evaluation of Mass Characteristics for all subsequent IB flights will be transferred to CCSD.

B. SA-202 Launching Information

Launching Information for SA-202 has been completed and submitted to the Industrial Operations Test Office for transmittal to KSC.

C. SA-203 Flight Sequence

The Saturn IB/SA-203 Flight Sequence requirements, drawing number 10M30153 Revision A, was released. The flight sequence requirements contained in this document consist of four parts:

1. The SA-203 Primary Sequence of Events from liftoff through the completion of the hydrogen experiment.

2. The alternate sequences which will be used during the hydrogen experiment if the failure mode, which is noted with each alternate sequence, occurs.

3. Liquid hydrogen experiment special requirements which will affect the programming of the Launch Vehicle Digital Computer.

4. Supplemental data for parts.

D. Prelaunch Test and Checkout Requirements and Specifications and Criteria

A review of SA-203 Prelaunch Test Specifications and Criteria has been initiated and will be complete early next period. First priority has been assigned to resolving "redline" difficulties with complete document review called for as soon as possible.

E. SA-204 and SA-205 Flight Sequence

The Saturn IB/SA-204 and SA-205 Flight Sequence was revised to reflect a time differential change from 6 seconds to 3 seconds between S-IB stage inboard engines cutoff and outboard engines cutoff.

F. Subsystem Reports

Saturn IB Functional Integration Subsystem Reports revisions for SA-202 and SA-204 has been completed.

G. Contamination Studies

Final coordination of the contamination level was accomplished. Approval of the IRN's against the level "B" ICD's is expected to begin prior to April 1, and will include concurrence of cognizant MSFC organizations.

H. Main Stage Propellant Fill Flowrates

Engineering Change Requests (ECR) and Interface Revision Notices (IRN) against the level "B" ICD's were prepared and approved by Research and Development Operations to establish propellant flow tolerances for the Saturn IB/V vehicles. This was requested by KSC to provide some latitude in handling system fluctuations that do not alter the acceptability of the ground system.

I. Mechanical Interface Control Program

The "A" level interface control documents transfer to CCSD is virtually completed. Preliminary work continues on the system definition and problem areas of the S-IB, S-IVB, and IU nitrogen system.

J. CALIPS Checkout Circuit Development Test

1. CALIPS breadboard was transported to Astrionics Laboratory, February 22, 1966. Preliminary testing began March 4, 1966, and continued up to March 18, 1966. At that time, due to unavoidable circumstances, the testing was temporarily discontinued. This problem arose when the vehicle in which the breadboard was housed had to be returned to the Army Missile Command. The problem of housing the breadboard has been resolved and testing resumed.

2. A review of all GSE CALIPS switch circuitry concerning the overall test is being conducted to determine if any operational conflicts exist between the electrical support equipment and the MGSE.

K. Hazardous Gas Analyzer (HGA)

1. The HGA used for the AS-201 vehicle (LC-34) will be transferred by KSC over to LC 37B for the SA-203 vehicle launch. This unit will be updated to the present design configuration during the month of April 1966.

2. The second unit, scheduled for LC 34 (SA-202), will be built to the current HGA design. This unit is scheduled for delivery to KSC on July 31, 1966.

L. Technical Information Handbook

The SA-203 Technical Information Handbook has been prepared and distributed.

SATURN V

I. S-IC Stage

A. Propellant Dispersion System (PDS) Documentation

1. The PDS documentation was reviewed and disapproved. Additional dimensioning was required to locate clamps and ordnance tees correctly and the routing/location of the confined detonating fuse assemblies and exploding bridgewire detonators for the S&A device was incorrect.

2. The technical review of the ordnance installations for the S-IC-504 was completed. Twenty-six out of forty-six installation drawings were disapproved and comments have been documented.

B. S-IC GSE Hydraulic Performance Tester

The integration test requirements/specifications document, D5-15401-31, was reviewed and approved subject to changing the hydraulic power unit capability requirement from "3500 p.s.i.g." to 2500 p.s.i.g." which more adequately reflects the capability of the system to be tested.

C. RJ-1 and Sodium Nitrite Servicer

Negotiations were concluded to convert servicers, serial number (S/N) 001 and S/N 002, to the exclusive use of RJ-1/ethylene glycol. Servicer, S/N 001, was sent to Hayes International Corporation, Birmingham, Alabama, for the necessary work.

D. Umbilicals

1. Acceptance testing of the intertank umbilical reconnect assembly (Merritt Island Launch Area (MILA) 1 unit) at Michoud Assembly Facility (MAF) was successfully completed on March 7, 1966, and the assembly was delivered to MSFC Test Laboratory. There was no recurrence of the locking mechanism problems experienced during the previous acceptance testing.

2. The disconnect arm assembly (65B80668-1) provided by ECP 0041C was received at MSFC on March 17, 1966, and installed on the forward umbilical carrier assembly designated for MILA. Shipment of the forward umbilical to KSC was made on March 22, 1966.

3. Aft umbilicals 1 and 2 for MILA were shipped from MSFC to KSC on March 23, 1966. Aft umbilical 3 for MILA is presently in shipping at MAF and will be shipped directly to KSC.

II. S-II Stage

A. Umbilicals

1. Work was initiated to provide adapter couplings for use on the S-II stage facilities checkout vehicle as a backup for the propellant fill and drain couplings.

2. The bellows assembly for the A7-64 and A7-65 propellant disconnects failed during proof pressure testing. Two new bellows assemblies were delivered to North American Aviation, Space and Information Systems Division (NAA, S&ID), by Flexonics Corporation for the A7-64 and A7-65 propellant disconnects. Testing of the propellant disconnects is in progress.

B. S-II Pneumatic Consoles

ECR VMS-V-026, dated February 7, 1966, has been approved by Configuration Control Board (CCB) action. This ECR provides regulator and relief valve settings, orifice resizing, and orifice deletions in the S7-41A, B, and D consoles. These changes will bring the console pneumatic outputs in compliance with the revised S-II stage fluid requirements document (13M50097), dated September 10, 1965.

C. S-II Interlocks and Automatic Sequence

1. Inasmuch as there are differences between the interlocks and automatic sequence for MTF and those planned for KSC. S&ID and Propulsion and Vehicle Engineering Laboratory personnel will review the interlocks and automatic sequence for both locations to determine if they can be made more compatible with each other.

2. In addition, Mississippi Test Operations has requested an independent analysis by MSFC to determine adequate interlocks for the S-II-T stage.

3. MSFC is to initiate action to get the S-II-501 automatic sequence (which is presently event oriented) changed to a time oriented sequence to simulate the launch sequence at KSC.

D. Relocation of Range Safety Decoders

A study is being made to determine the feasibility of relocating the range safety decoder from container 221 to container 223 in the S-II stage forward skirt area.

E. S-II Insulation Pullout Plug

SK10-8279 has been delivered to the Development Shop for test hardware fabrication. Testing of this design will begin at the MSFC Test Laboratory swing arm facility in approximately 6 - 8 weeks.

F. S-II-F Modification

1. All mechanical and electrical installations have been completed at KSC. Documentation reflecting the as-built configuration on the following items has been updated and preliminary copies have been transmitted to S&ID:

SK10-1991, revision C, Simulated J-2 Engine.

SK10-8269, revision A, Heat Shield Curtain Installation.

SK10-8275, revision A, Electrical Equipment Container.

2. All the required documentation necessary for S-II-F stage aft environmental conditioning systems verification test has been completed and sent to KSC.

G. S-II Stage Control Weights

The revision to the S-II stage control weights was transmitted to Saturn V Program Office. The purpose of the revised control weights is to give the S-II project manager more freedom in making mandatory changes to the S-II stage without adversely affecting cost or schedules.

III. S-IVB Stage

A. Pneumatic GSE

The fluid requirements for the SDF have been revised. The equipment will be modified to meet the new requirements of DAC's

drawing 1B45011, revision B. This equipment includes the pneumatic consoles, models DSV-4B-432 and DSV-4B-433, and the S-IVB space flight simulator auxiliary console.

B. Auxiliary Propulsion System (APS)

Final approval has been recommended on change order 715 to contract NAS7-101 to the S-IVB stage manager. DAC will supply the following equipment in accordance with this change order:

DSV-4B-472, APS Propellant Transfer and Interconnect Kit, Fuel.

DSV-4B-473, APS Propellant Transfer and Interconnect Kit, Oxidizer.

DSV-4B-493A, APS Checkout Accessory Kit.

DSV-4B-1874, APS Instrumentation and Checkout Kit, Fuel.

DSV-4B-1875, APS Instrumentation and Checkout Kit, Oxidizer.

C. S-II/S-IVB Hydraulic Servicer

D5-15402-6, "Phase II Integration Test Requirements/Specifications for the S-II/S-IVB Hydraulic Servicer for Mobile Launcher 1," was reviewed and approval was recommended.

D. Umbilicals

1. Qualification testing of the debris valve is scheduled for completion on May 7, 1966, at DAC.

2. IU and S-IVB stage umbilicals to support 500F vehicle checkout were shipped to KSC on March 22, 1966.

IV. Instrument Unit

A. IU Pneumatic Console

1. ECR VMS-V-015 pertaining to the IU pneumatic console inlet pressure valve failure was prepared. This ECR modifies the valve by relocating the backup ring.

2. ECR VMS-V-047 pertains to the ST-124M purge circuit within the IU pneumatic console and has been initiated to prevent high pressure backflow due to failure of check valves in the purge vent system.

B. IU Ground Support Cooling Unit

1. Unit 11 completed acceptance tests and was shipped to MSFC on March 18, 1966, for use on the Saturn V SDF. Unit 1, which was temporarily being used at SDF, is being returned to building 4619 for continuation of IU tests by the Propulsion Division.

2. D5-15402-5, "Integration Test Requirements/Specifications for the IU Ground Support Subsystem Checkout Local Control," was reviewed and approval recommended.

C. Flow Control Valve Box

EIC 901N, "Unit Flow Control, Ground Support Water/Methanol Coolant," was reviewed and approval recommended.

V. General

A. Mechanical Interface Program

The R&D ICD matrix charts for SA-501 have been completed.

B. Mechanical Automation Breadboard (MAB)

The majority of equipment is now in place and the interconnect cables, tubing, and pipe lines are laid out on the trays. The hydraulic facility and servo system are the outstanding missing items.

C. S-II Stage Simulator

1. All items of stage hardware have arrived from NAA.

2. The floor equipment, consoles, boards, and tank are mounted and interconnect tray installation has started.

D. Instrument Unit

1. The cooling unit was delivered to the SDF without additional heat load equipment. An electric water heater was used to load and check out the unit.

2. The IU pneumatic console has been installed and is being connected to the GN_2 facility.

E. Saturn V Launch Vehicle Design Reference Mission Sequence

The initial release of the Saturn V Launch Vehicle Design Reference Mission Sequence (drawing number 10M30601) was made for use

in analysis and planning purposes. Presented is a launch vehicle mission sequence covering the time period from stages arrival at KSC through end of launch vehicle mission.

ADVANCED TECHNOLOGY

I. Vehicle Systems Engineering

A. MSFC In-Flight Experiments

1. Experiment number 2, Thermal Control Coating - The Astrionics Laboratory was requested by memorandum R-P&VE-DIR-66-88, dated March 15, 1966, to determine the attitude control requirements to conduct this experiment on launch vehicle AS-206.

2. Experiments numbers 5, 6, and 7 - The proposed structural support configuration SK10-8276 has been completed; however, the tank location is still questioned. A proposed configuration, SK10-8277, for the propellant transfer system, and for the pressurization and vent system, SK10-8278, has been started.

B. LEM Descent Stage Experiment Carrier

A request was submitted to Resources Management Office for negotiation of a study contract to determine the feasibility of using a Phase II LEM Descent Stage as a carrier for MSFC In-Flight Experiments 3 through 7.

C. In-Flight Experiment Carrier

The Phase B Study Plan for this experiment carrier was revised to reflect latest center comments. The revised plan will be presented to MSFC participating activities on April 1, 1966.

D. Saturn V Ordnance Camera Ejection System

The feasibility test hardware drawings have been completed and the hardware is being fabricated.

E. In-Flight Sterilization of Mars Probes

A paper was presented by Dr. Fisher at the AIAA/AAS Stepping Stones to Mars Meeting held in Baltimore. The paper was presented to give some engineering concepts for in-flight sterilization which would have the advantage of relieving the stringent and expensive requirements for terminal sterilization of space probes within the contaminated earth environment.

F. Lunar Excursion Module (LEM) Laboratory Separation Study

A preliminary report of a study to develop criteria for orbital separation of a fully loaded, inactive LEM from a Spent S-IVB stage has been completed. However, a supplement to the report is in progress which broadens the scope of the study to include the impact of retromotor misalignment.

G. "Man-System Task Analysis for Lunar Surface Experiments" Contract NAS8-20095

1. Three primary subjects completed pressure suit familiarization exercises and re-enacted a continuous lunar surface scientific mission under pressurized conditions. The scientific tasks simulated were gravimetry, magnetometry, seismometry, surveying, photography, driving, loading and unloading equipment. The outcome of this Phase I section of the study will be a list of Human Factors Design Guidelines for Lunar Surface Scientific Instruments. This list will serve to support Research Projects Laboratory in its scientific instruments development program.

2. By invitation of the Office of Advanced Research and Technology, the whole impact of physiological measurements as part of a study of man's functional capabilities in a spacesuit was studied. A general consensus of required measurements and recommended ones was reached with other investigators in this field at a meeting at the Manned Spacecraft Center (MSC). Another meeting is scheduled for this summer to finalize standard guidelines for all workers making energy measurements upon a pressure-suited subject. Such measurements are planned under Phase II of this study (Contract NAS8-20095).

H. Reduced Gravity Simulation

1. Analytical studies have been conducted to determine if the fidelity of simulated tasks under water is improved or unchanged in performing the tasks at depths greater that 30 feet.

2. Design data are being produced for obtaining components approaching neutral buoyancy under water for reduced gravity simulated tests.

I. Conical Shaped Charge Study

Layouts defining the stage modifications necessary for replacing the present linear shaped charge destruct systems with bidirectional charges have been completed. Minor revisions may be required to the S-IC stage layout to relocate the confined detonating fuse routing to the fuel tank.

J. Review and Evaluation of Test Results for Low Amplification Brackets

The vibrational test results from an aluminum and a beryllium bracket were evaluated. The brackets were loaded with two Exploding Bridgewire (EBW) firing units and subjected to sine sweep excitation in three mutually perpendicular axes. In comparing the transmissibility for these brackets, the beryllium bracket was shown to have less transmissibility in the major resonant frequency. Also, the beryllium had a shift upward of the major resonant frequency.

K. Engineering Documentation

1. One Qualified Parts List (QPL), 1 10M series drawing, 13 new and revised 50M series drawings, and 12 Engineering Orders (EO's) were released into the Saturn system during this reporting period.

2. There were 157 Interface Control Documents (ICD's) input forms processed and 146 drawings sent to the repository.

3. There were 99 official documentation packages released this reporting period. These packages included 189 new drawings, 185 revised drawings, 357 Drawing Release Lists/Engineering Parts Lists (DRL/EPL's) and 206 EO's. In addition, the following advanced released for procurement documents were processed: 1 DRL/EPL, 1 drawing and 22 EO's.

John O. Aberg

GEORGE C. MARSHALL SPACE FLIGHT CENTER

PR-P&VE-P-66-3

MONTHLY PROGRESS REPORT

PROPULSION DIVISION

(March 1, 1966 Through March 31, 1966)

SATURN IB

I. S-IB Stage

A. S-IB Stage Outboard Engines Cut Off Early on AS-201 Flight

The outboard engines cut off early when the fuel depletion sensors gave a premature signal. The fuel depletion sensors indicated an uncovered condition earlier than expected, although there was a significant amount of fuel remaining. If this condition is not rectified, and future flights repeat the characteristics of S-IB-1, the outboard engines can be expected to cutoff when the fuel depletion sensors are armed (one to one and one-half seconds early), which would cause an increase in residuals to 3,000 - 4,500 pounds. Recommendations have been submitted to prevent a re-occurrence of the abnormality.

B. Retro and Ullage Motors Performance Satisfactory on AS-201 Flight

Preliminary evaluation of the data obtained from the retro and ullage motors on AS-201 indicate all motors performed as required. Performance parameters for each motor were within motor specification and no abnormalities were detected in the data. The data are being evaluated in detail.

C. S-IB-5 Static Tested at MSFC

A short duration static test of the S-IB-5 stage was successfully conducted. Before the test, the thrust OK pressure switches (three per engine) were removed and X-rayed to determine if they

were contaminated. As a result, one switch was replaced. Also, all the engines were retrofitted with the new LOX pump shaft seal to reduce leakage during start. During post-test checkout, the main fuel valve on engine position No. 5 was leaking slightly and was, therefore, replaced. A full duration test was scheduled for March 30, 1966.

D. S-IB-5 Hydraulic System Contaminated

Fluid samples taken from positions 1 and 4 contained contamination particles having the appearance of sand, but since most of the particles were less than 10 microns in size, it is believed that they are of some other substance. The particles are being identified. The systems on positions 1 and 4 were recleaned by purging. Fluid sampling of the systems on positions 2 and 3 will follow.

E. Hydraulic Package GN_2 Leakage Problem Investigated

Investigation of the GN_2 precharge leakage problems encountered on S-IB-4 was completed. Tests showed that the GN_2 leakage occurred only at low temperatures. Severe leakage occurred at 0° F. As a result, it was recommended that the precharge be applied at 40° F minimum, and that the precharge rate not exceed 250 psi/min. A CCP was prepared to change to a sleeveless accumulator for S-IB-13 and subsequent. This change will eliminate the static seal through which the GN_2 leaks into the liquid side of the accumulator.

F. Base Thermal Analysis Reviewed

Based on recent wind tunnel test results a significant increase in base convective heating for S-IB-203 and subsequent was predicted, due to rerouting of the turbine exhaust gas ducts. A base thermal analysis was performed to determine the effect of this more severe environment on base thermal protection. The study revealed the heat shield and curtain temperatures to be higher than previous predictions, but still within design limits. The study also showed two inboard engine hat bands to exceed the design limits previously specified by the engine contractor. Based on recent analytical and experimental data, however, the stage contractor has agreed to relax these limits so that no additional thermal protection is required. It is, therefore, concluded that the S-IB-203, and subsequent, base thermal design is compatible with the new base thermal environment.

G. Additional Firing Program on H-1 500 V Igniters Completed

The purpose of this program was to investigate the pressure delay time obtained during previous testing which indicated no delay time between the application of the firing pulse until maximum pressure was obtained. These tests have shown that previous results were in error and were caused by the oscilloscope trigger signal being delayed for up to eight milliseconds.

II. S-IVB Stage

A. LH_2 Tank Pressure Decayed on S-IVB-201

The LH_2 tank pressure decay apparently was due to sloshing. S-IVB-203 also exhibited an LH_2 tank pressure decay during static firing that was caused by a combination of very small ullage and high pressurant velocities. The phenomenon of S-IVB-203 is apparently an anomalous situation where an increase in pressurant flowrate results in tank pressure decay. These problems are being investigated.

B. Preliminary Flight Prediction Tapes for S-IVB-202 and 203 Completed

These tapes have been furnished for use in checking the computability of MSFC computer programs with the stage contractor's tape format.

C. S-IVB-203 Acceptance Firing Discrepancy Investigated

Before the S-IVB-203 stage acceptance firing, the J-2 gas generator control valve orifice was changed to reduce the fuel turbine inlet temperature during engine starts. Engine tests by the engine contractor showed that the change would reduce the temperature with a small reduction in fuel pump stall margin. The S-IVB-203 acceptance firing showed, on the other hand, that while the temperature was reduced, there was a significant and more than anticipated reduction in the stall margin. The stage contractor requested that tests be performed to verify the results obtained on the S-IVB-203 stage acceptance firing. When a problem is identified, other tests and analyses will be performed to determine a solution.

D. S-IVB-204 Acceptance Test Firing Completed Successfully

On March 18, the S-IVB-204 stage was successfully acceptance fired. The test was terminated after 451.2 seconds of mainstage by the facility computer when the LOX level reached 1880 pounds. This test was the first test successfully fired on the first attempt. Preliminary data analyses indicate the start transients and side loads were normal. Post-firing inspection revealed metal chips in the LOX pump. The engine was inspected for cracks on the first stage turbine wheel. No cracks were discovered, and the engine was considered to be flight worthy.

E. MSFC S-IVB Battleship Firing Conducted

Two battleship firings were attempted. The first test was a successful 412-second mainstage firing. Cutoff was given by the fuel tank depletion redline observer. The second attempt was made with a 200-second duration scheduled. The test was automatically terminated after approximately two seconds of mainstage when the J-2 engine gas generator combustion temperature exceeded the cutoff value of 2000° F. Data are being analyzed to determine the cause of the high temperature.

F. Training for MSFC LH_2 Experiment Flight Control Personnel Initiated

A three day introductory course familiarized the personnel with flight planning to date, the phenomena expected in the LH_2 tank of S-IVB-203, the TV picture the personnel will be viewing, and flight control procedures. This introductory course was attended by approximately twenty persons from I-MO-F, DAC, R-P&VE-P, and R-AERO-D. The S-IVB-203 static firing yielded data valuable for simulation training and for comparison with LH_2 Experiment data for postflight data analysis.

G. Auxiliary Propulsion System (APS) Tested at MSFC

The APS was fired in the altitude cell for a full 4 1/2-hour mission profile test. Preliminary results indicate that the engine O/F ratios differed from those noted during sea level tests at the DAC Sacramento Facility. Neither hardware damage nor any apparent problem was noted.

H. Component Qualification Test Program

Documentation review including specifications and test procedures was completed on nineteen components of the formal Qualification Test Program. The documentation review portion of this program is 58% complete. Three formal qualification tests were started; all have failed as follows:

F-13 Swing Check Valve - failed pressure drop.
I-8 Internal Vent Duct - failed vibration test.
H-17 Bellows - failed cycle tests.

I. First Subscale Firing in S-IVB Retro Motor Contamination Test Program Completed

Results obtained were generally good, although some data was lost due to instrumentation failure and lack of electronic equipment to retrieve the data from magnetic tape. Contamination of the paint samples was detected; however, the size and depth of the craters has not been determined. Data from this firing is being evaluated.

A second motor firing was attempted, but resulted in a hang fire (non-ignition) of the motor. Investigation into the cause of the failure revealed that the plastic bag igniter burst when subjected to the vacuum environment of the test cell allowing the igniter pellets to fall out of the motor nozzle. The electrical squib fired but without the igniter pellets the energy released was not enough to ignite the propellant grain. Small holes were punched in a second bag igniter to eliminate this problem on the next firing attempt.

J. S-IVB Accumulator Reservoir Assembly Tested

Proof pressure, relief valve, functional, low temperature, and high temperature tests were completed. The life cycle test is in progress with 2000 of the 9000 cycles completed. All other tests were completed satisfactorily except the low pressure relief valve test in which the maximum allowable inlet pressure at rated flow was exceeded.

K. S-IVB Workshop

A parametric study of radiator area and weight requirements have been completed. Various waste heat rejections, inlet temperatures, and orbit positions were considered. In all cases a weight-optimized fin/tube configuration was studied. The study results are

not sufficient to completely define radiator operational performance; however, the results are very useful for estimating radiator area and weight requirements for various workshop concepts.

III. Instrument Unit

A. I. U. Component Qualification Test Program

1. Coolant Pump, TRW with Brushless DC Motor - Unit 1: Thermal vacuum and service life tests completed. Unit 2: Qualification tests not started.

2. Pre-flight Heat Exchanger (Hamilton Std) - Qualification tests completed.

3. Sublimator, Water Accumulator and Orifice GN_2 Regulator (Hamilton Std) - Qualification tests completed.

4. Water Shut-off Valve (Marotta) - Qualification tests completed.

B. Saturn IB and V Environmental Control System

1. I. U./S-IVB Cooling System Test - A comprehensive review of all I. U./S-IVB Cooling System and component tests is being prepared.

2. Sublimator Testing - Testing of Sublimators SN 3', SN 5' and SN 7' at AEDC in Tullahoma, Tenn. was completed. Tests of several additional sublimators are anticipated. Sublimator testing at MSFC was discontinued due to the lack of a pump capable of handling Methanol/Water at the required flowrates.

3. Prototype Flight Pump Test - The prototype flight pump was operated in the I. U./S-IVB Cooling System Mockup at off-design power supply levels. The resulting data will be used in selecting a device to switch to a redundant pump in case of failure of the primary flight pump.

4. No. 2 Gas Bearing Heat Exchange Qualification Tests - Thermal and flow tests at -100° F and Burst Test were successfully conducted.

C. Gas Bearing Gas Supply System Mounting Panel Vibration Tested

After successfully completing vibration in two axes, the outlet nipple of the high pressure fitting (20M42053) broke off during vibration in the third axis. Vibration testing will continue upon receipt of modified high pressure fitting.

D. Analysis of Gas Bearing Supply System Completed

The analysis based upon preliminary data is complete and a computer program was written. The analysis is being modified to include the orifice in the ST-124 Stabilizer and new data on the loss of gas bearing pressure. A test setup is being made to provide orifice data for the program.

E. AS-203 Gas Bearing Heat Exchanger (GBHE) Tested

A development Test was completed to determine flow and thermal characteristics on 203 GBHE.

F. Gas Bearing Heat Exchanger Qualification

The qualification test of GBHE was completed.

G. Coolant Pump Development

Pump S/N 002 performed satisfactorily on Vehicle SA-201 after slightly over 1000 hours operation during acceptance tests and vehicle checkout. The suppression diode circuitry on pump S/N 004 was modified after an investigation performed on pump S/N 005 as a follow-up on the diode failures occurring previously. Pump S/N 004 subsequently completed thermal vacuum testing and a 500-hour service life test. Temperatures during 30 VDC operation in a vacuum enviroment exceeded the most desirable operating conditions, and means of decreasing this operating temperature are being investigated.

A decision was made to change the rotor material of pump S/N 005 from Alnico VI to Alnico VIII. Tests have shown the pump performance with an Alnico Rotor is improved, with greater efficiency and improved reliability expected as results of the change. Pumps S/N 006, 007, and 008 were delivered during March.

The backup Coolant Pump Breadboard was tested with performance requirements. The first backup pump is scheduled for shipment during April. Changes were initiated to provide remote starting capability and to relocate the electrical connector on the instrument unit coolant pump assembly.

H. Gas Bearing Panel Assembly Qualified

Efforts to qualify the gas bearing panel assembly by formal testing have stopped because of the inability to adequately simulate the proper environments. The assembly is considered qualified, based upon component qualification tests.

SATURN V

I. S-IC Stage

A. F-1 Engine

1. R&D Engine Tests at EFL

Seventy-nine tests were conducted, and a total duration of 5,254.1 seconds was accumulated. Twenty-five of these tests were full duration runs (150 seconds or more), thereby increasing the total number of full duration tests to date to 478. Eleven of the tests were terminated prematurely; twenty-one were ignition only tests; and the other twenty-two tests ran for the programmed duration.

2. Production Engine Tests at EFL

Six tests were conducted, and a total duration of 641.6 seconds was accumulated. All of the tests were successfully conducted as intended. Three tests were conducted at full duration (150 seconds or more). The total number of production engine tests conducted to date is 128 with an accumulated duration of 9,705 seconds.

3. R&D Engine Tests at MSFC

Four tests were conducted on engine F-4T2. The objectives of these tests were to checkout the new thrust measurement system in the F-1 test stand and to study high NPSH. Each of these tests were conducted for a mainstage duration of 40 seconds. Additional tests will be conducted to obtain data for the NPSH studies.

4. Engine Component Qualification Tests

Twenty-eight components have been subjected to complete qualification tests. Fifteen components are presently in various phases of qualification testing, with five of these scheduled to be completed this period. A re-design of the high voltage igniters is

being considered because of its failure to meet all static discharge requirements. Testing of the liquid level detector is being stopped because it may be deleted from the engine.

5. Thrust OK Pressure Switch Contamination Investigated

Since the thrust OK pressure switches on the H-1 and J-2 engines were found to be contaminated, similar switches for S-IC-2 and spares were X-rayed to determine if they too were contaminated. No evidence of contamination was found in these switches for S-IC-1.

6. Gas Injection Effects on Engine Performance Investigated

The results of tests conducted to determine the effects of inert gas injection on the F-1 and H-1 engine performance and operation were evaluated. The maximum injection rates tested were 2.5% (H-1) and 3.2% (F-1) by volume in the LOX suction ducts and 1.5% (H-1) and 1.8% (F-1) by volume in the fuel suction ducts. A significant decrease in performance was observed when the injection rates into the F-1 LOX suction duct were above 2.5%. No other significant changes in engine performance were noted. Helium and nitrogen gases were used in these tests.

7. Staggered Ignition Achieved on S-IC-1

Using individual start signals to each engine, the targeted 1-2-2 ignition sequence, with very slight deviation, was achieved during the second static test firing of S-IC-1. The starting times used on this test were based on the data obtained from the previous test. It was demonstrated that the desired 1-2-2 ignition sequence for launch can be accomplished using individual start signals based on the engine and the stage acceptance tests.

8. Requirements for F-1 Engine Cocoon Conditioning System Established

Orifice sizes in the stage systems were determined. A salient feature of the study is the establishment of a minimum pressure-temperature relationship required at the stage umbilical.

B. S-IC Propellant Loading Monitor Program Completed

The computer program calculates fuel density from fuel temperature measurements during countdown and projects fuel density for liftoff. Corresponding propellant loading values are determined for the projected density. It will initially be used for S-IC-F propellant loading tests at KSC.

C. S-IC-2 Schedule Changed

S-IC-2 is now scheduled for delivery to Test Laboratory on May 2, 1966 and acceptance testing on May 26, 1966. This is a slippage of six weeks from the original schedule. The acceptance testing will consist of one scheduled test of approximately 125-seconds. All future S-IC stage acceptance tests will consist of one scheduled long duration run instead of the previously planned two runs (1 short and 1 long). This change was made to achieve compatibility with the S-II and S-IVB stage programs that contractually require only one acceptance test.

D. Aerodynamic Heating of Propellant Dispersion Ordnance Analyzed

Aerodynamic heating analyses were conducted to estimate temperatures of the S-IC Propellant Dispersion Ordnance (P. D. O.) for SA-501, 502, and 503. A maximum temperature of 140° F was estimated for the lead sheath enclosing the explosive of the linear shaped charge. This temperature is well below the maximum allowable temperature of 220° F.

E. Combined Detonative Fuse (CDF) and Exploding Bridgewire (EBW) Initiator Performance Comparability Tests Completed

Preliminary results indicate that performance of the two types of initiators are comparable.

F. S-IC LOX Vortex Formation Studied

Consideration is being given to extending the standpipe on the center engine LOX Suction Line approximately 34 inches farther into the LOX tank to facilitate earlier cutoff of the center engine. This is being done to alleviate structural problems associated with late engine cutoff. An experimental program will be conducted to determine the effects of extending the standpipe on causing a vortex to enter the standpipe. Buildup of the experimental apparatus has started and tests should start approximately April 20.

G. Component Qualification Test Program Status Reported

1. LOX Pre-Valve (AiResearch) - All qualification tests are completed; however, the valve is not recommended for flight use.

2. LOX Vent Valve, 10" (Parker) - Vibration tests on one unit were completed. One unit failed, (actuator bellows) during life cycle tests. Design changes are being made and qualification tests should restart in April.

3. LOX Pressure Volume Compensator - Outboard, (Flexonics) - Life cycle and vibration testing were successfully completed.

II. S-II Stage

A. J-2 Engine

1. LOX Pump Turbine Problem Investigated

Cracks were found in the LOX pump first stage turbine wheel of production engines 2038 (vehicle 501) and 2027 (MSFC). These cracks were caused by a critical vibration mode of the turbine wheel. An inspection procedure to locate other cracked turbine wheels was initiated. New thick turbine wheels, which will eliminate the cracking problem, are scheduled for vehicles 504., S-IB-208 and subs.. An investigation is being conducted to determine a positive solution for the engines in the ear lier stages.

2. J-2 Start Tank Subjected to Simulated Altitude Testing

Tests are being conducted on the vent and relief valve before installing it on the start tank. The tests completed during this reporting period are flow, cracking, leakage and low temperature.

3. J-2S Solid Propellant Gas Generator (SPGG) Used During Engine Tests

Two J-2 engine firings were made in which the SPGG was used for engine starting. The engine contractor indicated the starting characteristics were good, and future tests will be made at other flow-rates of the gas generator to define the start-box limitations. The contractor is investigating the effects of temperature on the gas generator propellant performance. Temperatures between the range of 0 to 100° F are presently being investigated.

The contractor has also suggested that the EBW initiator for the J-2S SPGG application may not be able to withstand the high pressures (5500 - 6000 psi) expected in the gas generator. This problem is being investigated.

B. Battleship Program Completed

Battleship testing was completed with a successful firing on March 15, 1966. Test duration was 349 seconds, and cutoff was initiated automatically by the LH_2 low level cutoff sensors. Continuation of S-II battleship program was reassessed, and the battleship vehicle will not be subjected to future testing at this time but will be held on standby until tests of the S-II-T are successful.

C. LN_2 Loading Test on S-II-T Stage

On March 26, the S-II All-Systems test stage was tanked with LN_2. This was the first time cryogenics have been loaded in a flight-type S-II stage. The loading test was successfully accomplished. An LH_2/LN_2 tanking test is scheduled. This test will provide considerable information on subsystems performance (recirculation, thrust chamber chill, etc.) as well as a crucial test of the LH_2 tank insulation system.

D. S-II-T Component Qualification Status Reviewed

Propulsion System Components	Reviewed	139
	Qualified	83
	Acceptable for S-II-T	41
	Unqualified	15

The 15 unqualified components with potential failures during qualification testing have been reviewed, and based on the latest status of development and partial qualification test results, the components were considered acceptable for S-II-T firing, unless major failures occur during qualification testing and stage checkout.

E. Protuberance Heating to LH_2 Tank Insulation Estimated

Inflight temperatures of the LH_2 tank wall insulation were estimated based on the MSFC maximum heating trajectory for SA-501, 502, and 503. A maximum temperature of 310° F was predicted for the outer seal layer in locations not affected by protuberances. Maximum temperatures up to 540° F were estimated for localized areas affected by protuberance heating. The results of these analyses will be used to determine whether or not thermal protection material will be required.

F. S-II MARK IX Computer Program Modified

A revised S-II PU system simulation was incorporated into the MARK IX computer program. The S-II stage program nomenclature change was completed except for one subroutine. In preparation for the impending static test program, two static test predictions were made. The predictions differ in the ratio of the tanking mixture.

III. S-IVB Stage

A. New Operational Procedure for S-IVB/Saturn V to Relax Mission Constraints

An operational procedure was recommended for the propellant management system that will eliminate orbital vehicle positioning and time constraints to guarantee restart for 501. The only propulsion system constraint remaining is that at least 15 minutes must transpire between S-IVB first cutoff and restart.

B. In-House Synchronous Orbit Study Initiated for AS-507

This study will determine minimum modifications to the launch vehicle required to accomplish an unmanned synchronous orbit mission. Ground rules for the study are:

1. Fully loaded S-IVB coasts for nine minutes after separation from S-II stage.
2. First S-IVB burn is initiated at end of nine minutes coast and terminated at injection into Hohmann transfer orbit.
3. S-IVB coasts in Hohmann transfer orbit for 5.5 hours.
4. Reignite the S-IVB at end of 5.5 hours coast to circularize in synchronous orbit.
5. No further S-IVB propulsion system requirements after end of second burn.
6. Mission APS propellant requirements must be within the present system capacity.

C. J-2 Engine Thermal Environments from Firings of Ullage Motors and O_2/H_2 Burner Defined

This environment is for the 72-pound ullage motor scarf angle turning the plume centerline 3°, and heating rates are approximately one-half those for the plume centerline turning 10° previously presented.

SPECIAL STUDIES

I. Investigation of Freon E-3 as Working Fluid in Thrust Vector Control Systems Low Temperatures (-150° F)

A low temperature hydraulic system using Freon E-3 as the liquid is being assembled. The pump mounting brackets and adapter designs are completed, and components are being fabricated. The test setup is being fabricated and is approximately 15% complete.

II. Zero Leakage Program

A. Investigation of Brazed and Welded Tube Connectors - Proof test was performed on all 1/4-inch specimens. Two thin-wall welded connectors leaked appreciably during proof test. These connectors were replaced by new hardware.

B. Storage Test (18 month) of MF Fittings - The specimens will remain in storage until June 1, 1967. After a final leakage test, the specimens will be returned to MSFC for metallographic inspection.

C. Leakage Test to Determine Surface Finish Required for MF Fittings - Testing was completed.

D. Leakage Test of MC Fittings with A286 Sleeves at LH_2 Temperature - Proof test and room temperature leakage tests were completed with all specimens. Two 3/8-inch fittings leaked excessively.

III. Spin Heat Splitter

The theoretical analysis is completed, and theoretical performance curves were plotted for water, liquid nitrogen, and liquid hydrogen. An experimental apparatus was designed and is being fabricated.

IV. 60-Foot Parabolic Antenna

A preliminary thermal analysis was completed for two conceptual antenna designs. Because of extremely close contour tolerances, the thermal expansion resulting from temperature gradients must be kept to a minimum. The first design, a truss support structure, was discarded in favor of a second design tubular configuration. The antenna reflector design is not final; therefore, only the support structure was considered. Beryllium is specified as the antenna material. Due to the high thermal conductivity of Beryllium,

temperature gradients will be minimized. The thermal analysis showed that the radial gradient could be held to less than 5° F.

V. Investigation of LH_2 Recirculation System Vapor Entrainment

This study is an investigation of vapor entrainment observed in LH_2 recirculation systems while flowing subcooled liquid. Recent tests indicate that this vapor could be caused by contamination of LH_2 with helium used for pressurization. Preliminary analysis indicates that a significant quantity of H_2 vapor (sufficient to explain existing test data) can result from small quantities of helium in the flowing LH_2 as saturation conditions are approached. Although conclusive data is not presently available, no apparent subcooled boiling occurs during tests when GH_2 is used for system gas requirements instead of helium.

VI. Superinsulation

Installation of the 105-inch diameter tank was completed and the second series of tests are being performed. Preliminary results indicate better thermal performance for this test. A boil-off value of 2.0 lb/hr or 2.4% per day was obtained for the last test. The second 105-inch diameter tank was wrapped with Linde type insulation. A vacuum jacket of mylar-lead-mylar, 2.8 mils thick, was applied over the Linde insulation.

ADVANCED PROPULSION AND TECHNOLOGY

I. 350K High Pressure LH_2 Pump

The pump failed again at 31,000 rpm. Data indicate that rigid shaft frequency oscillations persisted beyond their resonant speeds and caused the pump bearing failure. A series of fixes were agreed upon for an early April pump test. At 31,000 rpm, the pump operates at 75% of its design speed and at approximately 67% of the 5500 psia design discharge pressure.

II. Pump Inducer Development Program

Testing was resumed at the Component and Sub-Systems Test Division after several weeks of delay. The present program is anticipated to be conducted for approximately one year and is aimed at development of pump inducers for the F-1, 350K, and other advanced

engines. A telemetry system that will transmit the inducer and impeller blade loading data is also being designed and developed.

III. Filtration Mechanics and Sampling Techniques Study

The DECOTO contamination monitor was examined. This device is intended to indicate the contamination level of an oil by monitoring the degree of frictional resistance in a spool-type valve caused by contaminant jamming the clearance between the spool and sleeve. A bank of valves is used to indicate the sensitivity to several ranges of contaminant sizes. Attempts will be made to examine its performance quantitatively by comparison with other contamination level measurement methods. Work is also continuing on the HIAC counter. New calibration factors are being applied to achieve better correlation with the ARP-598 particle counting method. Silting Index units are being evaluated and compared on the basis of repeatibility of a sample, sensitivity to changes in levels, and ease of operation.

PUBLICATIONS

I. "Qualification of Gemini SE-7 Engines as the Saturn S-IVB Stage Ullage Control Thruster," Unclassified, TMX-53400, by Donald Pryor, Dated February 24, 1966; Published March 18, 1966.

II. "AS-203 LH_2 Experiment Handbook for Flight Control Personnel," Unclassified, by C. D. Arnett

III. "Vibration Test of S-II Stage LH_2 Outboard Heat and Recirculation Lines," Unclassified, IN-P&VE-P-66-5, by H. Bergeler, E. Rudder, R. Allen and H. Hammac. Dated March 23, 1966; Published April 4, 1966.

H. G. Paul
Chief, Propulsion Division

GEORGE C. MARSHALL SPACE FLIGHT CENTER

PR-P&VE-S-66-3

MONTHLY PROGRESS REPORT

STRUCTURES DIVISION

(March 1, 1966 Through March 31, 1966)

SATURN IB

I. S-IB Stage

During the S-IB 70-inch LOX tank qualification test a failure occurred in skin panel 10 at 120 percent of design limit load. Skin panel 10 was not considered to be the most critical panel for this condition. The area of failure is being investigated and evaluated at the present. It appears that the test fixture may not have simulated the proper stiffness at the forward skirt ring frame.

II. S-IVB Stage

The Super Guppy flight test schedule was finalized with Douglas Aircraft Company. The S-IVB dynamic stage was flown to Huntington Beach, California on March 20, where Douglas will reduce the data acquired during the flight. This data will be evaluated to qualify the aircraft as a mode for transporting the S-IVB stage.

III. General

A. SA-201

A preliminary flight evaluation for POGO stability was completed for the SA-201 flight.

B. SA-206

A study to determine the POGO stability of the SA-206 vehicle was completed using revised vehicle weights data.

A torsional vibration analysis for the Saturn IB, SA-206A vehicle was completed for eight flight times during first stage burning and for the empty condition only of the upper stages (memorandum R-P&VE-SLA-66-6).

C. Differential Pressures

Differential pressures on the spacecraft, instrument unit, and S-IVB forward skirt of SA-202, 204, 207, and 205 have been published. Data is contained in memorandums R-P&VE-SLL-66-14, R-P&VE-SLL-66-15, R-P&VE-SLL-66-16, and R-P&VE-SLL-66-17, respectively.

D. POGO Stability Study

A study to determine the POGO stability of the S-IVB/203 was completed using a detailed structural J-2 engine model as supplied by Rocketdyne.

A study to determine the POGO stability of the Saturn 207/S-IVB was initiated.

E. 200/500-II Instrument Unit

Dismantling of the 200/500-II instrument unit test setup has been completed except for the portion of the test setup that will be left in place pending the delivery of the second instrument unit in June 1966, for continuation of the structural qualification test program.

F. S-IC-T-14

An evaluation of test data has been completed for longitudinal loads on the S-IC-T-14 booster stage.

I. S-IC Stage

A. Strength Analysis

During the first test of the S-IC-S lower assembly for the cutoff condition, the load cylinder applying the load to the crossbeam bottomed-out at 132 percent of design limit load. The Boeing Company retested this area to 140 percent on March 22, 1966, for the same condition and all the lower assembly structure is now considered qualified.

The heat shield was tested to 158 percent of design limit load on March 17, 1966. The test consisted of loading nine heat shield panels on the fuel tank thrust structure assembly, to simulate a design limit pressure of 2.52 psig. In addition, loads simulating the effects of the heat shield curtain were applied in two areas. The data is being evaluated at this time.

B. S-IC-S Fuel Tank-Thrust Structure-Intertank

The structural qualification test program on the S-IC thrust structure-fuel tank-intertank assembly has been completed. Test phases completed this month were: 1) qualification of the slow release fittings on the thrust structure; 2) qualification of the heat shield for max q load conditions; and 3) retest of flight cutoff conditions. The flight cutoff condition was retested, because during the initial test the hydraulic cylinder bottomed-out at approximately 130 percent load. Since the center engine affects only the crossbeam, it was decided qualification could be completed by applying only the center engine thrust load. The stress levels recorded on the crossbeam for this supplemental test agreed with those of the earlier tests through 130 percent load.

The S-IC thrust structure fuel tank intertank assembly is now fully qualified for all major design conditions, except ultimate pressure in the fuel tank. The major tests performed on this specimen have been as follows:

a. 99.9 percent ground wind
b. Rebound with 99 percent ground wind
c. Max $q\alpha$
d. Cutoff
e. Static firing holddown conditions

C. 1/4-Scale Intertank

The 1/4-scale intertank was tested to failure on March 10, 1966. The specimen failed at 150 percent of combined axial load, bending moment, and shear in a cryogenic environment. This concludes all of the intertank scale testing in the S-IC program. The 1/8 and 1/4-scale tests prove that subscale testing of larger structures is feasible, and if conducted properly, can produce very beneficial data.

D. General

The Boeing Company has analyzed the S-IC forward skirt and S-II aft interstage to determine the approximate load distribution at the interface, station 1541. Using the "COSMOS" computer program, which has proved reliable in previous S-IC analysis and testing, a 30 percent peaking load or 30 percent above uniform load exists just above the S-IC forward skirt umbilical and access panel. A coordination meeting to discuss the problem and plan future action is planned for the week of March 28, 1966.

A review of Flexonics' design analyses of the S-IC pressure volume compensators was completed. The analyses were disapproved because previous suggested improvements had not been incorporated.

II. S-II Stage

A. S-IC/S-II Interface

The Boeing Company has completed a study of the load distribution across the S-IC/S-II interface. Their analysis indicates a nonlinearity of load distribution of 30 percent. The peak loading occurs in the vicinity of the access door hardpoints. Local stringer beefup will be required on S-II-1 and subsequent stages.

B. S-II Recirculation Bypass Line

Testing of the S-II recirculation bypass line was completed with a line supported by two added support brackets passing all required testing. The additional brackets will be required on S-II-T and all flight stages.

C. Vibration Analysis

A lateral vibration analysis for two fill conditions of the S-II stage in the static test stand has been completed.

D. Panel Buckling Tests

The S-II stage panel buckling tests for the S-II-4 configuration, 2020-T6 aluminum extrusion stringer in the thrust structure have been completed.

E. LOX Bulkhead

An S-II stage aft LOX bulkhead was hydrostatically tested to its ultimate design pressure loads on March 2, 1966. The sump bolts failed in tension at 96.5 p.s.i.g. The test structurally qualified the bulkhead to the factor of safety listed below:

Sump bolts	1.4
Parent metal in the dollar region	1.36
Weld in the dollar region	1.26
(Analytical extrapolation)	1.55
Parent metal (near apex) of gore	1.35
Weld at waffle area to membrane	1.36
Parent metal (near equator) of gore	1.46
Parent metal of waffle	1.47
Meridional weld	1.45

The subject test demonstrates the structural integrity of the bulkhead to its design parameters. The analytical design objective of the bulkhead was a factor of safety of 1.31.

The methods of sealing the S-II aft LOX bulkhead dollar reinforcement were tested during the ultimate hydrostatic testing. The fix using the aluminum foil seal over two doublers did not leak during the test. The "gunked" bolt fix began leaking at 80 p.s.i.g.

F. General

Because of the multiplicity of failures already experienced in the instrumentation container test program, it has been recommended that the program be expanded to include all containers, not just the few

representative samples currently programmed. Change Order 351 should be implemented immediately and made mandatory for qualification of S-II-1.

Continuing failures on the 207 instrument container have necessitated redesign. The redesigned container will be evaluated under the CCN-13 program (flight configuration canisters). Responsibility for the S-II component qualification program was transferred from S&ID's Reliability Department to their Engineering Department, but this has not brought the necessary changes in test philosophy. The program continues to be extremely weak in the area of test setup. There is no attempt being made to simulate vehicle mounting conditions for the component tests.

Aerojet-General will fully instrument the S-II-3 aft LOX bulkhead to demonstrate the stress wave technique. Stress waves were detected during the ultimate test of the aft LOX bulkhead and several weld cracks have been located during the subsequent inspections. No correlation of the cracks with the monitored stress waves has been made at this time.

S&ID is performing a shear lag analysis of the GSE adapter ring, static firing skirt, and aft skirt of the S-II-T stage to determine the criticality of a four point support during static firing at MSFC.

III. S-IVB Stage

A. S-IVB/SA-500F Damper System

Design configurations for the wind load alleviation system for the SA-500F vehicle are now being finalized. Major problem areas were as follows:

1. Mounting of hydraulic cylinders

2. Attachment of system to the S-IVB interstage

Preliminary investigation has indicated that the interstage ring station 2569 will require beefup. Work is being coordinated with Douglas for the proposed beefup.

B. SA-503 LOX Tank Crack

The S-IVB/SA-503 LOX bulkhead experienced a failure during hydrostatic testing. Douglas has proposed a mechanical double shear lap joint to repair the area in an effort to keep the SA-503 in its proper sequence in the schedule. The Douglas proposed repair was reviewed and approved on March 16, 1966. As of this date the repair has been made on the SA-503 and hydrostatic testing is scheduled for March 28, 1966.

To prove the structural integrity of the repair, Douglas plans to test the repair on the following hardware:

1. 5-1/2 foot dome test fixture

2. The LOX tank remaining from the original hydrostatic test tank.

C. Hydrostatic Test Hardware

As a result of the concern for the structural integrity of the S-IVB tankage design and since the S-IVB tankage design has never been proven to 140 percent of limit pressures, it has been decided to remove the SA-504 tankage from the production line and make it available for a complete program of hydrostatic testing. The planned testing will include limit, ultimate, and failure test phases. Tentative plans are to complete testing by August 1966.

D. Common Bulkhead Failure Investigation

On March 3, 1966, Douglas presented a status review of their investigation of the S-IVB common bulkhead structural test failure. The investigation revealed that the failure probably initiated in the aft face sheet to lower ring circumferential weld, propagated in both directions, and caused both face sheets to sever around the entire circumference of the bulkhead. The exact location of the initial failure was not pinpointed.

Douglas stated that the reverse pressure on the bulkhead attained before it failed was an ullage pressure of 36.7 p.s.i.d. The existing margins of safety as shown by the tests are as follows:

Effectivity	S-IC Cufoff Max. Vent Setting in LH_2 Tank, LOX Tank Vented		Prelaunch Max. Vent Setting in LH_2 Tank LOX Tank Vented	
	Pressure (psid)	Factor	Pressure (psid)	Factor
SA-201 & SA-202	-30.2	1.15	-28.0	1.24
SA-203	-32.8	1.06	-28.3	1.23
SA-204 and Subs.	-27.2	1.28	-25.0	1.39
SA-501 and Subs.	-26.1	1.33	-23.1	1.50

The required factor of safety for both the prelaunch and end of first stage boost is 1.40.

E. Saturn V/Voyager

The Saturn V instrument unit is capable of withstanding the loads given in the following memorandums:

R-P&VE-SLR-66-13, Saturn V Voyager (42' payload) and Saturn V Voyager (22' payload) Inflight Structural Loads Produced by Vibration, February 17, 1966.

R-P&VE-SLR-66-15, Longitudinal Dynamic Loads for Saturn V/Voyager with 42-Foot Shroud, February 23, 1966.

Additionally, the Saturn V instrument unit capability envelope has been prepared for use in Saturn V/Voyager studies.

F. General

Douglas letter A2-860-KABC-4.43.0-L-511, "Effect of Severe Aerodynamic Environment on the S-IVB/IB and S-IVB/V Aft Skirt and Aft Interstage," dated March 4, 1966, in response to NASA letter I-V-S-IVB-L-66-59, "Effect of Severe Aerodynamic Environment on the S-IVB/V Aft Skirt and Interstage," dated February 2, 1966, has been reviewed by the Vibration and Acoustics Branch. This letter delineates Douglas' position on the effect of the severe aerodynamic environment on the S-IVB/V aft skirt and aft interstage skin panels.

Essentially, Douglas believes that the MSFC acoustic environment predictions as given in memorandum R-AERO-AU-66-8, "Maximum anticipated fluctuating pressure environment in the region of the S-IVB/S-II interstage for the Saturn V vehicle," dated January 24, 1966, are overly conservative and that the skin panels cannot withstand this environment. The Vibration and Acoustics Branch agrees with the former conclusion, but still maintains that the skin panels can withstand this environment. It has been determined that no fix will be required by MSFC.

IV. Instrument Unit

Preliminary test plans for IBM to complete the Saturn V instrument unit qualification test program have been reviewed and forwarded to R-P&VE-SJ. However, a decision has been made to complete this testing inhouse and plans are now being formulated for this program.

A combined heat/load test was run on a simulated Saturn IB/V instrument unit panel. The panel failed at approximately 1.90 times ultimate cutoff load, 400 degrees Fahrenheit outer face temperature, and 40 degrees Fahrenheit [illegible]t, tentatively showing that the structure is adequate for a high temperature condition.

V. General

A. J-2 Engine

At the J-2 quarterly review meeting at Canoga Park, Rocketdyne presented stress analysis data which indicated that the stud drive fuel turbine wheel failure which occurred in November 1965, could be attributed to nonsynchronous whirl of the wheel because of flexible bearing supports. This whirl causes a relatively low amplitude alternating stress to be superimposed upon the high steady state stress caused by the wheel spin speed and thermal gradient. Material fatigue testing with a high steady state stress had been performed which showed that the combined calculated steady state and alternating stresses could cause fatigue failure of the wheel. A more rigid bearing support has been incorporated and a reduction of the nonsynchronous whirl has been measured.

Failures of LOX turbine wheels at MSFC and S&ID are under investigation. Cracking and severe rubbing between the first stage wheel and the second stage stator has been observed.

B. F-1 Engine

An analog response analysis for determining the loads in the actuator during engine gimbaling checkout was completed. Maximum loading was found to be approximately 39,000 pounds, compared to an allowable load of 85,000 pounds.

C. SA-501

A study to determine the POGO stability of the Saturn V, SA-501 was completed using Langley Research Center's 1/10-scale model damping data.

A study which determined the effects of varying engine fuel suction flow rate to thrust cross coupling transfer function gains on the POGO stability of the Saturn V, SA-501 vehicle was completed.

An analysis has been initiated to determine the allowable thrust buildup envelope of second stage ignition for the SA-501 vehicle.

An analysis to determine the ground wind gust response and the von Karman excitation of the Saturn V facilities vehicle was completed including a damper system between the LUT and vehicle.

A study to determine the POGO stability of the S-IVB stages for the Saturn V, SA-501 S-IVB stage was completed.

D. SA-504

Longitudinal force at maximum thrust for the SA-504 vehicle has been supplied to R-P&VE-SJ. This data reflects an increase in thrust (memorandum R-P&VE-SLL-66-13).

A study to determine the POGO stability of the S-IVB stages for the Saturn V, SA-504 S-IVB stage was completed.

E. LH_2 Pump

Two front bearing failures of the Pratt and Whitney Aircraft developmental LH_2 pump have occurred at about 31,000 r.p.m. Pratt and Whitney has attributed the failure to a dynamic mode where the rotating parts act as a rigid body supported on flexible bearing supports. More rigid bearing supports are to be incorporated before the next run.

F. Heat and Loads Test of 36-Inch Honeycomb Panel

Heat and loads test of 36-inch honeycomb panel was completed. Panel failed at 1720 lb/in. axial compressive load, 1890 pound average bracket loads, and 400°F temperature. A test report is presently being prepared and will be available for distribution about July 1, 1966.

G. Impedance Trailer

The Structures Division impedance trailer has been moved from Brown Engineering Company to building 4619. This marked the NASA acceptance of the trailer, and dynamic testing utilizing its data acquisition capabilities is scheduled in the near future.

H. Solid Motor Test

The second 260-inch solid motor test in Dade County, Florida, was instrumented by the Vibration and Acoustics Branch. Six radiometers, eight vibration, and 22 acoustic measurements were installed. Data from all except eight of the measurements was successfully acquired.

I. Nuclear Engine

Vibration, acoustic, and strain measurements have been established in the measuring program for cold flow and hot firings of nuclear ground test modules. These instrumentation requirements will be used in estimating nuclear engine development costs for NASA Headquarters.

J. Rocket Sled Test

The fourth successful rocket sled firing was conducted at Holloman Air Force Base, New Mexico, on March 10, 1966. This was the first firing with the flexible panel on board the sled. The instrumentation physically survived the test and a cursory review of the 'quick look' data indicates that all channels contain usable data.

K. Saturn V Workshop Experiment

The Saturn V instrument unit is capable of withstanding the loads given in memorandum R-P&VE-SLL-66-5, "Preliminary Structural Loads Analysis Including Preliminary Structural Limits for the Saturn V Workshop Experiment," January 25, 1966.

L. Reverse Pressure Tests of Model Bulkheads

Setup and instrumentation have been completed for a reverse pressure bulkhead. However, test has been delayed because of a data system printer malfunction. The printer is expected to be repaired and bulkhead tested before April 8, 1966.

APOLLO APPLICATIONS PROGRAM

I. Orbital Experiment Compartment

A brief study was initiated to select tanks and tank support systems for MSFC experiments 5, 6, and 7 when these experiments are carried in a stripped LEM descent stage. Also initiated was an investigation into structural systems for an experiment rack for possible MSFC development that would be a back-up for the LEM descent stage used as an experiment rack, and capable of being used with or without a LEM ascent stage. To date, one concept has been developed and a structural weight estimate for it completed. A one-day visit was made to the Manned Spacecraft Center to acquaint personnel there with MSFC experiments 5, 6, and 7.

II. Lunar Shelter Study

Complete data for the preliminary sizing of the two revised expandable/inflatable shelters was received from the Strength Analysis Branch. The two new expandable concepts, along with two rigid structure shelter concepts were completed, and copies furnished to Strength Analysis Branch for preliminary sizing.

III. LO_2 Tanker Docking Structure

The Vibration and Acoustics Branch has employed a more detailed dynamics program to check the results of the limited original docking dynamics program. The predicted loads of the original program proved to be excessively high. More recent predicted loads imposed on the docked tankers by the subsequent docking of the S-II stage compare favorably with the intertanker docking loads for the symmetric case. The unsymmetrical impacts are presently being investigated.

IV. Apollo Shroud For A Single LEM Payload

Two concepts with associated weights and design considerations were preparad for an Apollo shroud for a single LEM payload. The design variables included guide rail separation, pyrotechnic separation, honeycomb construction, and skin-ring-stringer construction.

V. LEM Experiment Compartment

Structural sizing has been completed on a redesigned carrier vehicle to be used in the LEM experiment compartment.

Analysis and sizing has also been completed on two concepts of a shroud/nose cone to be used in the Saturn IB single LEM payload experiment.

VI. LFV Reusable Strut Study

The originally designed reusable strut concept was compressed in a tensile test machine, and the stroke-force characteristic was found to be undesirable. An effort to improve the condition with a minimum change modification has been initiated.

VII. Vibration Environments

Vibration environments for Voyager have been submitted for inclusion in the study proposal. Environments and/or test procedures have been submitted for numerous other AAP items, such as the lunar cryogenic storage tank, AAP experiments 3-7, and nuclear ground test module.

ADVANCED TECHNOLOGY

I. Design Studies

A. Nuclear Ground Test Module

Structural sizing has been completed on one concept of the nuclear ground test module to be used in testing the NERVA engine.

B. Modular Nuclear Stage Study

Preliminary design of the supports for the tank were completed by the support contractor. Main effort was spent on the tension support system.

C. Manned Flying Systems

Drawings of the Bendix Corporation's reusable strut development concept were reviewed and Bendix's representatives were informed of the resulting comments. A trip to Bell Aerosystems Company was planned to discuss the various strut concepts under study by the Bendix Corporation.

D. Vibration Qualification Tests Damage Criteria Study

The test damage criteria study on a typical duct was performed to correlate stress levels that would result when the duct is subjected to the Saturn V vibration qualification test levels. Normally, stress levels are not measured to this extent. Data analyses from the strain gages indicate the qualification test maximum stress levels due to vibration that did not exceed 20 percent of the yield strength of the material.

E. Nitrogen Tetroxide Cylinder

The design of a cylinder to contain nitrogen tetroxide has been made and drawings have been released to manufacturing. This work was in support of Materials Division.

F. Beryllium Instrument Unit

Preliminary designs of four different test panels have been made and material ordered. The material order was for 19 sheets of various thicknesses for these and future designs.

II. Design Research

A. High Temperature Structures

The final report covering general design considerations for the structure of a reusable re-entry vehicle is now complete. This report, entitled "Design Guide for High Temperature Structure," is available for distribution to interested parties. With the completion of this report, no further design effort on hot structures is anticipated until renewed interest in the Reusable Orbital Transport is evident.

B. Meteoroid Shielding Study

An analytical investigation was conducted to determine meteoroid protection requirements for the proposed Lunar Flying Vehicle and a Lunar Shelter Concept. Extrapolated Pegasus data was used for the penetrating flux. The study indicates tremendous weight penalty for an exposure time of 180 days. Review of useful documents to establish a "Meteoroid Protection Design Guide" is continuing.

C. Semi-Toroidal Tank Study

The weight tables and graphs showing the optimum weight bulkhead contours were completed, and the drawing that defines the optimum weight configuration was finished. The draft of the final study report is about 80 percent complete.

D. Wind Alleviation Design

The design of the wind alleviation damping system is in progress. Drawings for the structure have been released for fabrication. Requirements for the hydraulic system have been prepared and sent to manufacturing. Test requirements are being coordinated. The design is scheduled to be fabricated, tested, and shipped to the launch site by early May 1966.

E. 124 Huckbolt Blind Fastener

The huckbolt blind fastener tests were completed. A series of these specimens were subjected to sine excitation at the resonant frequency to evaluate the fastener's tension characteristics. Two specimens were evaluated at 100 g's for 60 minutes without structural degradation or failure. The third specimen was evaluated at 200 g's and failed after 6.4 minutes. The specimens were returned to R-P&VE-SVA for detailed inspection.

F. Nitrogen Tetroxide Container

An analysis has been completed and the drawings signed on a nitrogen tetroxide shipping container and the results forwarded to R-P&VE-SA.

G. Multicell Test Tank

Data evaluation on a limited basis has been completed on strain gauge information received from the multicell tank pressure tests.

III. Structural Experiments

A. Cryogenic Test Tanks

1. 105-Inch Test Tank

The tank struts have been modified such that the machining necessary for the installation of the Goodyear vacuum jacket may be accomplished in the subassembly stage.

2. 70-Inch Test Tank

A concept has been developed for supporting the tank in a manner similar to what is presently being considered in the Orbital Experiment Compartment Tests. This support system will eliminate the pinned joints employed on the present tank supports.

The 70-inch diameter test tanks will be modified to a configuration similar to tankage proposed for orbital experiments 5, 6, and 7. This will allow studies on insulation application procedures in advance of actual hardware. Alternate insulation attachments will be investigated for the helium-purged NRC-2 insulation. The basic objective will be to reduce the tediousness of currently required assembly techniques.

B. Cryogenic Burst Tests

Two rejected hemispherical cryogenic burst specimens were received. These specimens will be used for checkout of the cryogenic burst test facility systems. Checkout test will be run about May 1, 1966.

OTHER

Mobility Test Article (MTA)

Preliminary negotiations with two bidders on the Lunar Gravity Simulator Study RFQ were attended. These discussions resulted in an MSFC request that the proposal which received the highest technical rating be resubmitted with a work scope reduced so as to meet available funds.

G. A. Kroll
Chief, Structures Division

GEORGE C. MARSHALL SPACE FLIGHT CENTER

R-P&VE-M-66-3

Monthly Progress Report

Materials Division

March 1, 1966 Through March 31, 1966

SATURN IB

I. S-IB Stage

A. Investigation of Materials for Thermal Insulation in the Aft Area of the S-IB Stage

The objective of this program is to develop unfired ceramic insulations with bulk densities below 60 lb/ft^3 which can be applied to metal honeycomb substrates for protection against primarily radiant heat fluxes of 40 Btu/ft^2-sec. Specifically, a replacement is being sought for M-31, the composition presently used for insulating the horizontal heat shield of the S-IB stage. The replacement is necessitated by the withdrawal from the commercial market of the primary ingredient of M-31. The material being sought must have insulating qualities at least equal to those of M-31, and its adherence to metal substrates must be superior to M-31.

As reported earlier, insulations composed of various inorganic fillers and opacifiers bonded with a colloidal silica sol are being investigated as replacements for M-31. Based upon preliminary evaluations, two insulations, designated FTA-442A and FTA-532A, were selected for further evaluation. Both insulations contain Fiberfrax and asbestos fibers as the fillers, pigmentary potassium titanate (PKT) as the opacifier, and Ludox HS30 colloidal silica as the binder. The exact formulations are given in the following table:

TABLE 1

Composition of Experimental FTA Insulations

Material	Parts by Weight FTA-442	FTA-532A
Fiberfrax fibers	40	50
PKT	40	30
Asbestos fibers	20	20
Ludox HS30 (pH-6.6)	420	420

The FTA insulations are prepared and applied by the same general procedures as used for M-31 except the silica sol is gelled to prevent silica migration during drying of the insulations. Gelation is accomplished merely by lowering the pH of the silica sol to a point at which the sol becomes unstable and a silica gel is formed. The prevention of silica migration results in the formation of a strong insulation with a uniform structure. This prevention of silica migration also enhances the insulation-substrate adhesion which is related directly to the strength of the interior of the insulation.

The FTA-442A and FTA-532A insulations have been characterized as to bulk density, water absorption, refractoriness, specific heat, mechanical strength, adhesion to metal honeycomb sandwich substrates, and insulating capability in radiant heating environments. The characteristics of these FTA insulations are compared to those of M-31 in the following table:

TABLE 2

Characteristics of Experimental FTA Insulations Compared to Those of M-31 Insulation

	FTA-442A	FTA-532A	M-31
Bulk density (lbs/ft^3)	61	59	55
Water absorption (percent)	44	45	69
Melting point (°F)	2400	2400	2200
Adherence to honeycomb substrate flatwise tensile strength (psi)	61	49	13
Modulus of Rupture (psi)	1535	1288	670
Insulating capability Back-face temperature rise (°F) after 145 seconds exposure to a radiant heat flux of			
24 Btu/ft^2-sec	115	180	189
40 Btu/ft^2-sec	404	419	494

In summary, two insulations, FTA-442A and FTA-532A, have been developed as potential replacements for M-31, the material now used on the S-IB stage. Although these insulations are slightly heavier than M-31, they have insulating qualities and adhesion characteristics superior to M-31.

Future efforts in this study will include further evaluation of the FTA-442A and FTA-532A insulations, and the development and evaluation of other insulations potentially useful as replacements for M-31.

B. Monitoring of Gas Leaks in the S-IB/S-IVB Interstage Compartments

The mass spectrometer hazardous gas detection system which was assembled for support of tanking tests on the Saturn I at Launch Complex 37B was moved to Launch Complex 34 for support of AS-201 and was used during tanking tests in September and November 1965 at Launch Complex 34. With slight modifications to the sampling valves, the system was used for support of Countdown Demonstration Tests (CDDT) on February 6-7 and February 9, 1966. Sample lines were installed in the S-IB engine compartment, the S-IVB aft interstage, and the Instrument Unit (IU). During the first CDDT, the equipment functioned satisfactorily but on the second CDDT the instrument failed just after LOX loading due to a malfunction of a vacuum gauge. The gauge was replaced by a more rugged gauge to prevent failure.

At approximately T-4:26 hours in the terminal count, a vacuum transducer in the hazardous gas detection system (HGDS) indicated an instantaneous perfect vacuum. The automatic range-seeking feature of this transducer, in trying to sense this false indication, shut down the HGDS mass spectrometer. Since a perfect vacuum did not exist, the vacuum transducer over-pressure protection circuit would not allow manual re-start from the HGDS blockhouse remote control panel.

After the launch, the HGDS was restarted and operated by manually resetting the range-seeking switch on the vacuum transducer. Since the HGDS operated normally in all aspects after having undergone the shock and vibration of the launch, the cause of malfunction was not an internal failure of the HGDS.

An analysis of the failure indicates that an external spurious signal caused a negative bias of the vacuum transducer signal which normally ranges from 0 to 100 millivolts. On the present prototype HGDS used on AS-201, the failure can be corrected by overriding the over-pressure protection circuit of the vacuum transducer. On subsequent models of the HGDS, the transducer signal level will be 0 to 10 volts and, therefore, will not be affected by spurious external signals.

Work on building the HGDS for Complexes 34 and 37B is progressing normally and is expected to meet schedules.

In addition, this division has been requested to support the loading tests on 500-F. This will require construction and operation of an HGDS by June 1, 1966. Necessary equipment for this unit has been ordered.

C. Investigation of the Failure of MF Fitting Springs

Investigations have continued on the failed 17-7 PH stainless steel wave springs used on MF flared tube fittings. Annealed springs were heat treated to RH950, RH1050, TH1050, and TH1100. All of these springs failed within five days except the springs heat treated to the TH1100 condition, which have been in test 39 days without failure. Tests also are being made with this alloy in the various heat treatment conditions using flat type tensile and round threaded-end tensile specimens exposed to alternate immersion in salt water and exposure to the atmosphere. Only one failure has occurred in the sheet tensile specimens after 49 days of exposure but all of the threaded-end tensile specimens stressed to 140 ksi in the transverse direction failed within 11 days. None of the specimens stressed in the longitudinal direction have failed after 26 days of exposure. Other materials are being tested for possible spring application. Inconel 718 has been in test for 27 days without failure. Gold plated Berylco No. 25 springs failed after 30 and 38 days exposure and one bare Berylco spring failed after 14 days exposure in the salt spray. Metallurgical examination of the Berylco springs indicated that these failures were not due to stress corrosion cracking.

D. Investigation of Failure of S-IB LOX Tank

A Saturn I-B 70 inch light-weight LOX tank failed prematurely during qualification testing at the Michoud Assembly Facility. A meeting was attended at the facility during which personnel of Chrysler presented the results of their analysis. The primary failure consisted of skin buckling below a point of maximum compressive loading. No design test or material irregularities that could have caused the failure were identified. Chrysler is proposing to install a non-stiffening patch and resume the tests. Particular attention will be devoted to a possible canting effect at the circumferential skin welds.

SATURN V

I. S-IC Stage

A. Developmental Welding

Investigations have continued in an attempt to establish the susceptibility to cracking of welds in various aluminum alloys. Cruciform specimens of welds on one inch plate 2219-T87 and 2014-T6 aluminum have been prepared and are undergoing natural aging. To date, difficulty has been encountered in developing a test which will cause reproducible cracking in thick sections.

Because of harmful gas fumes emitted during welding of experimental alloys X-2021 and X-7007, it was necessary to install exhaust ducts near the welding torches prior to proceeding with weld development work on these alloys. The exhaust ducts have been installed and the facility has been inspected and approved by the local safety office. Thus, developmental welding will be initiated in the near future on aluminum alloys X-2021 and X-7007.

Studies have continued into the cause and methods of eliminating porosity in welds in 2219 aluminum alloy. Phase I of this program consists of evaluation of bead-on-plate welds with partial and full penetration, with and without filler wire. Phase II provides for a study of comparable parameters except square butt joint geometry will be used and an additional parameter, i.e. mechanized versus manual cleaning, will be introduced. Welding is being accomplished in the horizontal position using the GTA process. All welding under Phase I has been completed. Radiographic inspection and metallographic examination are being used to determine weld quality.

Electron beam weldments have been made in 1/4-inch plate 2014 and 2219 aluminum alloys in an attempt to evaluate the weldability of alloys with minimized influence of other factors such as filler wire, etc. Radiographic examination of the weldments revealed longitudinal weld cracks in the 2014 while the 2219 welds appeared sound. Metallographic examination is being used to verify these results and to determine the nature of the cracking. Tensile specimens are being fabricated to determine the properties of the welds.

All welding operations have been completed on the program to evaluate the weldability of aluminum alloy X-7106 using the GTA process. Testing of tensile specimens at cryogenic temperatures continues to be delayed because of testing problems and higher priority work.

B. Investigation of the Low Temperature Mechanical Properties of Engineering Alloys

Testing has continued in the study of the low temperature mechanical properties of structural alloys. The status of this program is as follows:

1. Evaluation of PH 14-8Mo Stainless Steel Sheet

Specimens from both air and vacuum melts of PH 14-8Mo stainless steel have been heat treated and initial tests have been evaluated. Erratic material behavior thought to be due to improper heat treatment caused by a malfunction in the furnace temperature controls, has delayed this program. New tests will be made as soon as properly heat treated specimens become available.

2. Evaluation of Aluminum Alloys X-2021 and X-7007

Specimen fabrication from one-inch thick plate of aluminum alloys X-2021 and X-7007 and their weldments is nearly complete. It is anticipated that the testing of these alloys at cryogenic temperature will be initiated during the next report period.

3. Evaluation of M-45 Aluminum Casting Alloy

Fatigue testing has continued on specimens of M-45 aluminum casting alloy. Due to the time dependent nature of these tests there are no significant results to report at this time.

C. Study of Corrosion and Cleaning Procedures

1. Stress Corrosion Studies

Investigations have continued into the stress corrosion aspects of various alloys having potential or immediate application as structural materials in Saturn vehicles. The status of these investigations is as follows:

Studies are being continued to determine the threshold stress levels of 7106-T6351, 7139-T6351, and 7039-T61 aluminum alloys. Specimens of these alloys were stressed to 20 ksi, 15 ksi, and 10 ksi in the short transverse direction and 75 percent of the yield strength in the longitudinal and long transverse directions. No failures have occurred to alloys 7106-T6351 and 7139-T6351 after 69 days exposure and to alloy 7039-T61 after 11 days exposure in the alternate immersion tester and outside atmosphere at this Center. Specimens of alloy 7039-T74 will be introduced into the test schedule in the near future.

Studies are continuing on the evaluation of the effect of aging time on the stress corrosion resistance of 2024-T6 alloy. Current tests have been completed after 90 days exposure in the alternate immersion tester. Mechanical properties of the specimens that did not fail are being obtained. Analysis of these test results will dictate the requirements for future work on this alloy.

Stress corrosion studies of round and flat tensile specimens of ELI grade Ti-6Al-4V are continuing in the alternate immersion tester. No failures have occurred after 299 days of exposure. Specimens are being fabricated to provide for stress corrosion tests in salt water on pre-cracked specimens. Tests also are continuing in an attempt to determine the stress corrosion susceptibility of this alloy in N_2O_4 (nitrogen-tetroxide). Specimens are being fabricated for testing this alloy at stress levels of 20 to 100 percent of the yield strength.

Stress corrosion tests are continuing on specimens of Carpenter Custom 455 Stainless Steel in two heat conditions (aged at 1000°F (538°C) and 1100°F (593°C)). The alloy aged at 1000°F (538°C) was found to be resistant to stress corrosion cracking at loads up to 100 percent of the yield strength based on 180 days of exposure in the alternate immersion tester. The test of this alloy aged to 1100°F (593°C) is continuing. No failures have occurred after 140 days in the alternate immersion tester.

A testing program has been initiated to determine the stress corrosion characteristics of two high strength weldable aluminum alloys (X2021-T8E31 and X7007-T6E136) being developed under contract NAS8-5452. The stress corrosion characteristics of both alloys are being studied in all three grain directions in the alternate immersion tester and ambient atmosphere of this Center. Failures have been encountered in X7007-T6E136 stressed in the short transverse direction to 30 percent of the yield strength (20 ksi) in 27 to 30 days and in 28 days stressed to 23 percent of the yield strength (15 ksi) in the alternate immersion tester. This test has been in progress for 36 days. No failures have occurred in the atmosphere. The X2021-T8E31 specimens have been in test for 19 days with no failures in either environment.

Exposure to a salt spray environment has continued on representative specimens of two different designs of S-IC stage hydraulic actuators. The actuator fabricated from 7079-T6 aluminum alloy has been exposed for 220 days and the actuator fabricated from 7075-T73 has been exposed for 200 days. There is no evidence of cracks in either actuator as yet.

2. Investigation of Corrosion Protective Coatings for Aluminum to Steel Diffusion Bonded Joints

Materials evaluation has continued on coatings of potential use for protecting stainless steel to aluminum diffusion bonded joints against corrosion. Two coating materials Kel-F-800, and a fluorochemical lacquer Kel-F FX-703 are being evaluated.

Specimens were coated with the fluorocarbon materials to a thickness of 1.8 to 2 mils and were subjected to a salt spray environment (five percent NaCl) and a cold shock test. The cold shock test consisted of cycling between liquid nitrogen and room temperature water. After alternately cold shocking and exposure to the salt spray for 72 hours, the coatings were still intact with no apparent loss of adhesion. There were only two or three very small spots of corrosion. This amount of corrosion was considerably less than other sections of aluminum which had been treated with Alodine.

D. Study of Heat Treatments for M-45 Aluminum Alloy Castings

Activities have continued in the determination of a heat treatment cycle which will enhance the strength while maintaining optimum stress corrosion resistance of aluminum alloy M-45 castings. Samples of this

alloy were solution heat treated at 980-1000°F (527-538°C) for 16-72 hours and quenched in cold water. The solution heat treated samples were aged in the temperature range 325-400°F (163-204°C) for 6-72 hours. Tensile specimens were machined from this aged material, and mechanical property tests are in process on the aged material. Additional samples of M-45 were solution heat treated at 1000°F (538°C) for 24 hours, quenched in cold water, and aged in the temperature range 325-400°F (163-204°C). This material is being machined into tensile specimens for stress corrosion evaluations.

E. Investigation of the Materials Aspects of Joining Metallic Composites

Work has continued on the investigation of various joining techniques applicable in the forming of metallic composite materials. These investigations have included diffusion bonding and various solders and soldering techniques.

1. Study of Diffusion Bonding of Various Metals

During this report period a second attempt was made to prepare a diffusion bonded titanium lapshear specimen. The procedure used was as follows:

a. Clean specimen (HNO_3 and HF)

b. Abrade bonding surfaces of the specimen

c. Place specimen in stainless steel retort and seal weld

d. Evacuate retort after placing retort between ceramic platens in the furnace

e. Heat furnace to 1750°F (954°C) and maintain temperature for six hours at a pressure of approximately 400 psi.

Unfortunately, during the heating and bonding cycle one of the connections in the vacuum pump developed a severe leak causing the retort to lose its vacuum. This caused the titanium specimen to become heavily oxidized. However, subsequent micro analysis indicated a good diffusion bond. This procedure will be repeated during the next reporting period with every effort directed towards insuring that an adequate vacuum is maintained.

2. Evaluation of Solders for Joining Composite Materials

In the previous report it was noted that flatwise tension strength of soldered aluminum sandwich samples were found to be unsatisfactorily low. It was concluded that the low strength and inconsistent test results were due to insufficient solder foil thickness. Therefore, the foil thickness was increased to 0.008 inch with the surprising result

of approximately doubling the flatwise tensile properties as previously reported, that is 240 psi (an average of four specimens ranging from 185 to 303 psi in flatwise tensile). It should be noted, however, that considerable solder was found segregated on the lower face of the composite, which was the result of solder from the upper face flowing to the lower by the force of gravity. More work is planned during the next reporting period to rectify this condition.

F. Investigation of Fasteners and Fastener Materials

Materials testing and evaluation are continuing on the study and characterization of various fasteners and fastener materials used, or of potential use, in launch and space vehicles.

1. Material Evaluation

Continued evaluation of Carpenter's Custom 455 stainless steel. Specimens from 0.090 and 0.034-inch sheet and 13/16-inch bar, aged at 1150°F (621°C), are presently being evaluated.

2. Mechanical Fasteners

Work is in process to establish the cause of forging failures in beryllium bolts being produced under contract NAS8-20158, "Investigation of Advanced High Strength Alloys Used in Weight Reduction of the Saturn V Thrust Structure," with the Standard Pressed Steel Company. To date a successful head forging process has not been established, and all materials show evidence of longitudinal cracks at the hot forged head area; however, thread rolling of the beryllium material presents no problem. Material from Brush, Berylco, Beryllium Metals, and General Astrometals has been used in the investigation.

G. Study of the Compatibility of Various Engineering Materials with Propellants

Eleven miscellaneous materials were evaluated for compatibility with liquid oxygen in accordance with MSFC-SPEC-106A. Data generated from these tests were forwarded to cognizant design personnel and other interested groups.

Activities have continued in the cooperative test program designed to qualify and correlate various liquid oxygen impact sensitivity tests being made by stage and engine contractors. During this report period one hundred individual impact tests were made at each of six energy levels on three of the standard samples. These tests were made to demonstrate the reproducibility of impact tests at this Center when made in accordance with MSFC-SPEC-106A. A review of the test data indicates that a high degree of reproducibility is obtained. An attempt is being made, through application of statistical analysis techniques, to place confidence levels to all data generated in the cooperative liquid oxygen sensitivity test program.

H. Evaluation of Commercial Adhesives

Continuous evaluation of adhesive systems for Saturn applications is in progress. Since polyurethane resins exhibit good engineering properties at cryogenic temperatures, there is interest in all aspects of processing and behavior of these systems.

The Narmco 7343/7139 adhesives system on aluminum lapshear tensile specimens was subjected to various open face layup times. Conditions under observation ranged from 50 percent humidity at 80°F (27°C) to 80 percent humidity at 110°F (43°C), while time of exposure ranged from 0.5 hours to 3.0 hours. Adherends tested were both unprimed and primed with G-207 primer. Results confirmed earlier studies which showed that the relative humidity should not appreciably exceed 50 percent at a layup temperature of 80°F (27°C), and the relative humidity should be controlled to below 50 percent if layup temperature rises above 80°F (27°C).

Aluminum lapshear specimens bonded with the Narmco 7343/7139 adhesive system were subjected to accelerated aging conditions of 98-100°F (37-38°C) at 100 percent humidity over a period of one to thirty-five days. One series was primed with 3M Company's silane type primer XC-3901, another series with Goodyear's G-207 and a third series was unprimed. As a result of the accelerated aging, progressive bond deterioration was evident in all three series, although this effect was much less noticeable in specimens tested at cryogenic temperatures.

A number of primer systems for AF-111 film adhesive were evaluated in a T-peel configuration of aluminum adherends. These included EC-2320, M-602, and HT-424 A/B primer systems. No primer effected any notable increase in bond strength over unprimed specimens. A similar series of tests was run using unetched aluminum adherends which had been subjected only to vapor degreasing before bonding. Very poor results revealed that solvent degrease is probably insufficient surface preparation for aluminum adherends bonded with AF-111.

Investigation of the Narmco 7343/7139 adhesive system is continuing with studies on the effect of curing agent concentrations, the effectiveness of different mixing procedures and other parameters which may be responsible for bond strength variations.

I. Development and Evaluation of Potting Compounds

The objective of this program is the development of transparent, curable resin systems that exhibit good dielectric properties and are either inherently flexible or may be filled to yield a coefficient of thermal expansion approaching that of ceramics and other materials used in electronic circuitry. As to dielectric properties, the resins are required to meet MSFC-SPEC-202, "Specification for Elastomeric Conformal

Coating Compounds," and/or MSFC-SPEC-393, "Specification for Elastomeric Conformal Coating Compound for Printed Circuit Boards." Urethane-siloxane and epoxy-siloxane copolymers are being investigated as possible materials for this application.

1. Epoxy-Silane Copolymers

The projected synthesis of polymer precursors containing the silarylene bridging group and terminal epoxy groups has continued with the attempted synthesis of bis(4,4'-vinyldimethylsilylphenyl)-ether by a two-step *in situ* condensation of bis(4,4'-bromophenyl)-ether, magnesium, and vinyldimethylchlorosilane. A viscous, water-white liquid boiling at 105-108°C/0.03 torr has been isolated from the reaction mixture and tentatively characterized as the desired compound by infrared analysis. Epoxidation of this material should produce the desired epoxy polymer precursor. In other polymer systems the diphenylether backbone has proved to increase the toughness and flexibility characteristics. It is hoped to introduce these advantages into the epoxy-silane polymer system.

The epoxidation of the olefinic silphenylene materials has been attempted using trifluoroperacetic acid. The peracid is prepared *in situ* from trifluoroacetic anhydride and 90 percent hydrogen peroxide. The epoxidation is carried out in the presence of a large excess of anhydrous, finely-divided sodium carbonate to act as scavenger for the trifluoroacetic acid generated during the reaction. Formation of the epoxide rings occurs readily, but is accompanied by esterification of the epoxide. Modification of this procedure by using the more basic potassium carbonate scavenger and maintaining the reaction temperature at 0°C is expected to minimize side reactions of the epoxy ring.

2. Urethane Polymers

The synthesis of urethane-silazane polymers has continued with attempted preparation of 1,4-bis(N-methylaminodimethylsilyl)-benzene by condensation of 1,4-bis(chlorodimethylsilyl)-benzene with methyl amine gas at 0°C. The optimum conditions have not been established to isolate this monomer due to polymerization tendencies. The meta-isomer of this species is believed more amenable to synthesis and work will proceed to prepare this material.

An additional silicon-containing olefin has been synthesized for incorporation into the epoxy-silane polymer program. An epoxidation technique is being employed which successfully produces the epoxy ring with some concomitant cleavage.

Future efforts will be directed toward optimization of the epoxidation reaction variables to minimize side reactions of the epoxy ring. The synthesis of 1,3-bis(N-methylaminodimethylsilyl)-benzene will be attempted for incorporation into a urethane-silazane polymer system.

J. Investigation of the Lubricating Characteristics of Fuels and Hydraulic Oils

The engine fuel, RJ-1, will be used as the hydraulic fluid for the servo-actuators of the S-IC stage. Concern has arisen over the life of the GSE (Ground System Equipment) hydraulic pump because of the poor lubricating properties of RJ-1. As a result, emphasis has been placed on evaluating RJ-1 lubricating additives.

Screening tests are being made on commercial additives at constant temperature in the Shell Four Ball Wear Tester. The Shell Four Ball Wear Test is a relative measure of the lubricating ability of the fluids. As shown in Table 3, the additives L 1722, L 1588, and L 1721 have been tested in this reporting period.

TABLE 3

SHELL FOUR BALL WEAR TEST DATA
(Temp. = 75°F (24°C), RPM = 1,800)

ADDITIVE	CONCENTRATION IN RP-1 FUEL	LOAD	SCAR DIAMETER (MM)
L 1722	1,000 ppm	5 KG	0.519
		10 KG	0.743
		20 KG	0.988
L 1588	300 ppm	5 KG	0.523
		10 KG	0.628
		20 KG	1.058
L 1588	1,000 ppm	5 KG	0.510
		10 KG	0.756
		20 KG	1.149
L 1721	300 ppm	5 KG	0.504
		10 KG	0.887
		20 KG	1.250

Additional tests on the lubricant additives have been made on the Falex lubricant tester. The Falex tester is another relative measure of the lubricating ability of a fluid. The test measures bearing load and resulting wear produced by forces on a rotating pin and set of vee-blocks. It has been established that three hours of testing is required to establish wear life with wear measured in the number of gear teeth required to maintain a 100-pound load. Table 4 shows the fluids that have been tested on the Falex Lubricant Tester during this reporting period.

TABLE 4

FALEX LUBRICANT TEST DATA

ADDITIVE	CONCENTRATION	NUMBER OF TEETH	DURATION
L 1722	1,000 ppm	Galled	15 Minutes
L 1588	300 ppm	Galled	12 Minutes
L 1588	1,000 ppm	36	3.0 Hours
L 1721	300 ppm	Galled	12 Minutes

K. Evaluation of Materials for Miniature Slip Ring Assemblies

Miniature slip rings are employed to transmit electrical information across the gimbal axes in inertial platforms. These slip rings must have long life and extremely low noise levels. This program is directed toward the development of materials for the slip ring base (body), slip ring, brush block, and brush which possess the desired properties of long wear life, low noise, and resistance to contamination.

Since it is necessary to know instantaneous displacement as a function of time in order to determine noise as a function of ring position, a device was constructed to permit displacement to be displayed on an oscilloscope or on a high speed multi-channel recorder, along with the noise waveform. Installation of this LVDT (linear variable differential transformer) has been accomplished. The system is capable of resolving amplitude variations from 0.01 degrees to 5.0 degrees and frequency to 30 cycles. Testing of experimental capsules will be resumed.

L. Investigation of the Failure of a LOX Flowmeter Prevalve

The LOX prevalve flowmeters were removed from S-IC/501 after a 45-second static firing to determine the cause of the erratic performance experienced during the firing. Metallographic studies revealed areas of corrosion pitting on the surface of the 6061 aluminum housing which contains the tungsten carbide bearing. Although cracks were detected in the shaft bearing, it was not determined whether these cracks resulted from the electric discharge method for cutting the bearing or whether they were present prior to cutting. Spectrographic analysis revealed aluminum on the galled surfaces of both the shaft bearing and the ring bearings which indicates that Al_2O_3 contributed to the galling action. No trace of aluminum was found on the nongalled areas of the bearings. The manufacturer has modified two assemblies in an effort to correct the problem. The modifications consisted of introducing coolant passages on the upstream side of the shaft bearing and in the shaft. One of the shaft bearings was moly-coated and the other was modified to incorporate Teflon rubbing surfaces.

II. Contract Research

The status of the several Saturn-funded research contracts under the technical supervision of this division is described below. In keeping with the previous policy of this division, this report presents the contract research effort in terms of a quarterly review. A brief statement of the scope of work is given, together with the present status of the program.

A. Polymer Research, Development, and Evaluation

1. Contract Title: Study of Age Deterioration of Gasket Materials Installed in Simulated Launch Vehicle Hardware
   Contract Number: NAS8-20247

Contractor: University of Florida, Engineering Experiment Station
Contract Cost: $12,210.00
Contract Duration: 60 Months
Negotiated: June 28, 1965
Responsible Individual: J. T. Schell

Purpose of Project:

This project is designed to obtain statistically useful test data regarding the effects of semitropical environmental conditions (i.e., temperature, humidity, oxidation, fungus growth, etc.) as a function of time on a variety of gasket materials used or considered for use in launch vehicles. To accomplish the stated purpose, test specimens of a wide variety of gasket materials are installed in simulated launch vehicle hardware and exposed to the semitropical environment. Sample swatches of material are exposed similarly. Periodic examinations and tests are made on these gasket materials by the contractor. As failureis noted in any given material, it is removed from the test group, and new materials, based upon the availability of new products or modifications to existing formulations, are introduced into the test program.

Technical Status:

No activity by the contractor was required under the terms of this contract since the inspection of the exposed components made in October 1965, which was described in the previous report. The next scheduled inspection will be conducted in late April of the current year.

As a direct result of this program, the permissible shelf life of elastomeric items has been increased from two quarters to eight quarters and the permissible installed life increased from 12 quarters to 16 quarters.

This program will continue with no redirection anticipated during the contract period.

2. Contract Title: Development of Structural Foams for Cryogenic Applications
Contract Number: NAS8-11406
Contractor: Hughes Aircraft Company
Contract Cost: $126,882.00
Contract Duration: 21 Months
Negotiated: June 30, 1964
Responsible Individual: J. M. Stuckey

Purpose of Project:

The contract was directed toward the synthesis and development of halocarbon plastic foams which could be used as structural materials at cryogenic temperatures and which could be used in contact with liquid oxygen. Major emphasis was directed toward producing uniform, essentially

closed cell foams in densities of approximately two pounds per cubic foot and not greater than ten pounds per cubic foot. Efforts were directed toward developing foams having structural properties which equal or exceed those of the best commercially available polyurethane foams of the same densities. Both rigid and flexible foams have applications; however, emphasis was directed toward development of a rigid foam for cryogenic temperature usage. Efforts also were expended toward the development of a foamed-in-place material. Consideration was given to materials that could be employed to attach or hold foams in desired locations.

Technical Status:

This contract was directed toward the production of low density foams from commercially available halocarbon polymers that are compatible with liquid oxygen, and the development and synthesis of new halocarbon monomers and polymers for foamed-in-place materials. In the study of the production of foams from commercially available materials, a method of dry blending resins with nucleating materials such as boron nitride and chlorotrifluoroethylene resin (CTFE) was developed. Foams were made from various resins using both extrusion and static foaming techniques. Oven foaming of TFE Teflon, using CTFE resin both as a nucleating and foaming agent, was studied using a number of different heating and cooling cycles. Foams produced from these commercially available liquid oxygen-compatible resins by either static or extrusion foaming techniques were relatively non-uniform and heavy, weighing approximately 30 lb/ft^3 or more. This technique appears restrictive to samples having very limited cross sections due to difficulties encountered both in introducing heat to the samples and dissipating heat from the foamed material. Synthetic foams were studied using both FEP and TFE resins in combination with hollow glass microballoons. Foams produced by this technique that have adequate structural integrity are quite heavy, weighing approximately 80 lb/ft^3.

A number of halocarbon monomers and polymers based on polyurethane and epoxy monomers were synthesized for use in the production of a foam-in-place material. The most attractive foam resulting from this program was a brittle, low strength, fairly uniform material weighing approximately 3 lb/ft^3 that was produced by the condensation of a fluorinated novolac prepolymer with formaldehyde, but its high hydrogen content rules out any possibility that it could be used for liquid oxygen applications.

3. Contract Title: Development of Improved Gasket Materials for Liquid Oxygen Applications
   Contract Number: NAS8-5053
   Contractor: Narmco Research and Development, A Division of Whittaker Corporation

Contract Cost: $362,650.00
Contract Duration: 51 Months
Negotiated: June 21, 1962
Responsible Individual: J. E. Curry

Purpose of Project:

This program provides for the continued development of a unique cryogenic gasketing concept based upon a glass-fabric fluorocarbon plastic laminated construction, which is characterized by chemical inertness to liquid oxygen and unusual compressive properties. Broader simulated service testing will be conducted on flat gaskets and other seals of rectangular cross section.

A primary goal of the current contract modification is to determine the feasibility of incorporating this unique system of reinforcement into non-rectangular cross sections of interest, including O-rings, chevron and lip seals, and valve seats.

Technical Status:

The survey of basic seal configurations which can be fabricated by the laminating process developed earlier for flat gaskets has continued.

Chevron seals have been produced satisfactorily by only minor tooling changes and variations in the layup sequence used for flat gaskets. The only departures from the flat gasket layup technique arise during the final encapsulation step, where special techniques must be employed to pre-form the Teflon FEP to avoid surface wrinkling and folds.

Further comparison of winding and layup techniques for O-ring fabrication has indicated that winding techniques may be preferred only for small diameters and cross sections. In other cases, the pre-forming and encapsulation of flat laminates is much easier from a manufacturing point of view.

The unique reinforcement provided to Teflon by partially-wetted glass fabrics continues to show considerable promise, and has been adapted to circular and other non-rectangular seal cross sections of interest. As another processing goal, the fabrication of an actual seal for a large Saturn LOX prevalve will be attempted.

4. Contract Title: Optimization of the Performance of a Polyurethane Adhesive System Over the Temperature Range of -423° to +200°F
Contract Number: NAS8-11958
Contractor: Narmco Research and Development Division of Whittaker Corporation
Contract Cost: $129,986.00

Contract Duration: 10 Months
Negotiated: May 14, 1965
Responsible Individual: L. M. Thompson

Purpose of Project:

The purpose of this contract is to determine what factors contribute to the wide variations in bond strength which are known to exist in the polyurethane adhesive systems and to determine the best methods for eliminating or minimizing these conditions. The primary effort is directed toward defining the specific adhesive components, procedures and conditions which will consistently give acceptable bond strengths over the temperature range between -423°F and +200°F and that will also give a predictable mean bond strength with a known minimum variation.

Technical Status:

The contractor has continued the study of various cure procedures, catalyst concentrations, environmental conditions, component purities, mixing techniques, primers, and adherend cleaning procedures in an attempt to identify and characterize those factors contributing to the bond strength variations of the polyurethane adhesive system. Results were obtained which indicated that component purities, standard cleaning procedures, laboratory environments and different lots of adhesive and catalyst were not the causative factors of observed strength variations. The following factors directly influenced the bond strengths and variation: activator ratio, mixing technique, storage humidity, adhesive thickness, cure conditions and primer usage. The best results to date were found during a primer screening evaluation using the following techniques:

a. The use of a silane primer (Dow Corning Z-6020)
b. A stoichiometric catalyst ratio
c. A controlled bondline thickness
d. A specified minimum cure cycle
e. Improved mixing technique.

A major result achieved during this study was a significant improvement of the urethane adhesive bond strength at room temperature and above. Several types of primers were evaluated but best results were obtained using the silane type primers similar to the materials used as fiberglass finishes. Since the urethane adhesives are very strong at cryogenic temperature and relatively deficient above ambient conditions, a 100 percent increase in this area is highly significant. This increase was obtainable with several primers, most of which were the silane types.

A concentrated examination of primers, mixing techniques, adhesive additives, and cure conditions will be continued to obtain fully defined criteria for use of this polyurethane adhesive to achieve optimum results.

5. Contract Title: Development of Vulcanizable Elastomers Suitable for Use in Contact with Liquid Oxygen
Contract Number: NAS8-5352
Contractor: Peninsular Chem Research, Incorporated
Contract Cost: $251,589.00
Contract Duration: 37 months
Negotiated: May 10, 1963
Responsible Individual: W. E. Hill

Purpose of Project:

The ultimate goal of this research effort is the development of at least one vulcanizable elastomer which will be compatible with liquid oxygen and will possess useful elastomeric properties at the low temperatures of interest. The term "compatibility" as used herein means that the material must not show any evidence of chemical reactivity toward liquid oxygen when subjected to the tests specified in MSFC-SPEC-106. During prior modifications to this contract, it has been established that the fluorocarbon polymers containing $-CH_2-CF_2-$ and $-C_2F_4-$ linkages demonstrate the greatest promise of achieving the overall program goals. Further efforts will be made to incorporate these linkages into fluorocarbon materials by the homopolymerization of substituted fluorovinyl ethers and by copolymerization with fluoro-olefins and diolefins.

Various synthetic routes to fluorinated polyesters and polyethers will be attempted as suitable synthetic routes to the required monomers are developed.

Technical Status:

Copolymerization of $CH_2=CF_2$ or $CF_2=CF_2$ with fluoroalkylvinyl ethers continues to yield materials with good elastomeric properties. Each of these olefins has yielded tough elastomers with $CF_3OCF=CF_2$ and with $CF_3CF_2OCF=CF_2$. The ether, $(CF_3O)_2-C=CF_2$, gave a brittle polymer with $CF_2=CF_2$ but yielded a tough flexible film with $CH_2=CF_2$. Polymerization of $(CF_3O)_2\ C=CF_2$ with CF_3NO gave a small amount of brittle polymer, in contrast to the $CF_3OCF=CF_2/CF_3NO$ copolymer, which shows some degree of toughness. In order to extend the investigation of polymer systems to include heteroatoms in the polymer chain, a study of fluorocarbon aldehyde polymers has been initiated.

Copolymerization of fluoroalkylvinyl ethers with fluorinated olefins has proved a promising route to materials which may ultimately meet the requirements of this research effort. Many of the monomers utilized in this and other routes investigated were newly synthesized in this program. A relationship between molecular structure and glass transition temperature is being recognized and defined for certain classes of polymers.

Within the near future, polymers of fluoroalkylvinyl ethers with alkyl groups to C_5 will be available for polymerization studies. Quantitative determinations of polymer properties at low temperatures and of compatibility with liquid oxygen will be made. Exploratory investigations of other polymer systems will continue with copolymer studies of CF_3CHO. Synthesis of $CF_3OCF{=}CFOCF_3$ will be completed and preparation of copolymers will be attempted.

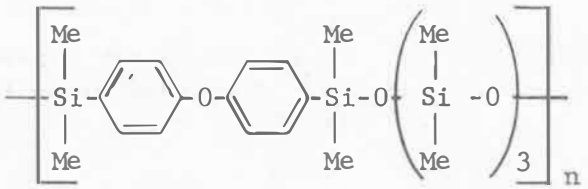
6. Contract Title: Synthesis and Evaluation of New High Temperature Polymers for Coating Application
Contract Number: NAS8-11338
Contractor: Midwest Research Institute
Contract Cost: $157,390.00
Contract Duration: 25 Months
Negotiated: June 25, 1964
Responsible Individual: J. D. Byrd

Purpose of Project:

The purpose of this project is to develop, synthesize, and evaluate new organic and semi-organic polymers with useful film-forming properties which are stable to high temperature, high vacuum, radiation, and the environment of space. This program emphasizes the evaluation of two known, recently developed polymers and also includes the exploration of new ideas which will lead to the development of new thermally resistant polymeric materials. The evaluation of these polymers as coating materials also will be emphasized.

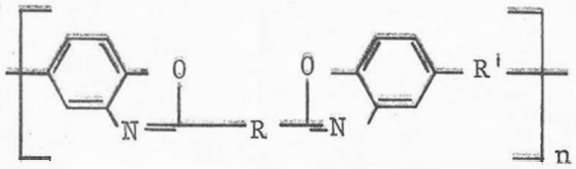
Technical Status:

A modification of the poly(arylenesiloxane) which was reported in the previous report has shown superior properties. This new polymer has the following structure:



This polymer is stable to 500°C and has good radiation stability compared to a silicone resin used in the coating now being applied to the Saturn S-IVB stage. It is soluble, readily obtained in high molecular weights and has good film-forming properties. In bulk form it is a tough elastomeric material. Under ultraviolet radiation, changes in reflectance of the specimens did not indicate any particular dependence of stability on structure in the phenylenesiloxane series. Spectra and thermal stability of the polymers are also reported.

Numerous modifications of the poly(benzoxazole) structure failed to provide polymers with the required solubility characteristics.



Polymers were prepared where R is $-(CH_2)_3-$ and $-(CF_2)_3$ and where R' is a bond only, $-C(CH_2)_2-$, $-SO_2-$, or $-O-$. None of these combinations improved the solubility to a suitable extent. Attempts to cast films from some of these polymers resulted in films that were dark and adhered poorly to test specimens.

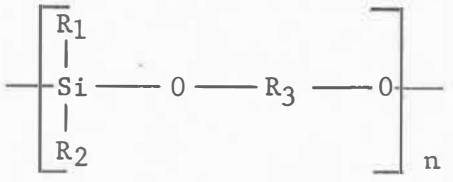
A number of new poly(arylenesiloxanes) have been prepared. Some of these materials have interesting properties which suggest their potential use in high temperature coatings. A number of polybenzoxazole structural variants have been prepared, but none have had the film-forming attributes desired for coating resins.

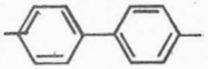
Major emphasis will now be placed on the accelerated production and coatings evaluation of poly(arylenesiloxanes).

7. Contract Title: Process Development and Pilot-Plant Production of Silane Polymers of Diols
   Contract Number: NAS8-11837
   Contractor: Battelle Memorial Institute
   Contract Cost: $95,000.00
   Contract Duration: 15 Months
   Negotiated: November 27, 1964
   Responsible Individual: J. D. Byrd

   Purpose of Project:

The purpose of this project is to provide engineering and scientific services for the process development and pilot plant production of polymers having the general structure



where $R_1 = R_2 = C_6H_5$ and $R_3 =$ 

Polymers of this type have been prepared in the laboratories of this Center, and they appear to have useful properties which merit the more detailed evaluation of larger quantities of specific compositions. Thus, the goal of this program shall be the development of a suitable process for making larger quantities of specific polymers of this type and to make certain detailed evaluations of these materials.

Technical Status:

More than 100 pounds of Polymer A, poly4,4'-(bisoxybiphenylene)-diphenylsilane, have been prepared to date. Pilot-scale samples of Polymer A having molecular weights ranging from about 40,000 to 85,000 are available. A procedure for raising the molecular weight and upgrading the mechanical properties of Polymer A has been discovered and successfully applied to pilot-scale lots of the polymer.

Polymer A has been shown to be processable as a conventional thermoplastic, and to possess properties making it potentially suitable for use as a structural resin. The polymer has been converted into film and sheet forms by both solvent-casting and compression molding procedures.

Typical properties of Polymer A having a molecular weight of 49,000-84,000, are shown below:

Flexural Strength, psi	3,000-10,000
Tensile Strength, psi	3,000-8,000
Heat Deflection Temperature °C (264 psi)	95-108
Impact Strength, Notched Izod, ft-lbs/inch of notch	0.18-0.43
Thermal Breakdown Temperatures °C (by TGA)	>400
Percent Elongation	<5

Solubility and chemical reactivity studies have shown that Polymer A is not readily subject to hydrolysis under neutral, acidic, or basic conditions, but can show sensitivity to such reagents upon prolonged exposure. The polymer is soluble in a variety of organic solvents from which it can be fabricated into films and coatings.

Further work will be done on model and polymeric species to obtain Polymer A analogues which can be chemically cross-lined in a controlled fashion. The successful attainment of this goal will enhance the development of adhesives and elastomers from these resin systems. Suitable quantities of these curable materials will also be prepared on a pilot plant scale.

B. Development of Cryogenic and High Temperature Insulation Material

1. Contract Title: Development of Materials and Materials Application Concepts for Joint Use as Cryogenic Insulation and Micrometeorite Bumpers
Contract Number: NAS8-11747
Contractor: Goodyear Aerospace Corporation
Contract Cost: $462,694.00
Contract Duration: 26 Months
Negotiated: June 30, 1964
Responsible Individual: J. M. Stuckey

Purpose of Project:

The contractor is directed to develop an easily installed externally applied lightweight insulation capable of providing the necessary thermal and micrometeorite protection on launch vehicles. The insulation must meet the following requirements:

a. The average equilibrium heat leak for space environment (Q) shall be approximately 0.25 Btu/ft^2/hr.

b. The complete system shall weigh less than 0.5 lb/ft^2.

c. The composite concept shall be capable of preventing or instantaneously sealing a penetration of a particle in the mass range of 10^{-1} to 10^{-5} grams impacting at a velocity of 30,000 ft/sec or greater.

d. It shall be capable of reliably performing its function under conditions of pre-launch, launch, and space flight when applied externally to a launch or space vehicle.

e. It shall be capable of being reliably applied to the external surface of launch vehicles which are nine feet in diameter or larger.

f. The concept shall prevent condensation of air or oxygen within the system during pre-launch operations.

g. It shall be capable of withstanding surface temperatures up to 400°F for short times during the aerodynamic heating portion of the flight.

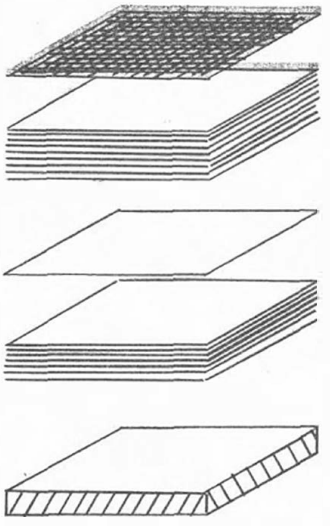
h. There shall be no adverse effects from changes in tank size due to flight loads or shrinkage during cryogenic fueling.

i. The insulation shall not be susceptible to damage if reasonable care is taken in handling.

j. The concept shall allow reliable application to tanks with irregular surface protuberances such as lines and instrument tunnels.

Technical Status:

The basic insulation concept now being evaluated in this program, as it evolved through the developmental work reported earlier, consists of three separate sections:



Style 181 glass fabric impregnated with polyurethane

Tape-wrapped aluminized Mylar (1/4 mil) with alternating foam layers

MAAM (Mylar-aluminum-aluminum-Mylar) vacuum bag

Evacuated ground hold section; aluminized Mylar-foam spacers 0.4-inch total thickness

Tank wall

The vacuum-bagged ground hold section is evacuated to provide insulation during ground hold conditions. Spontaneous evacuation of the outer multilayer section occurs during ascent. A low-density foam spacer between the outer multilayer section and the fiberglass outer bumper that was used in some of the previous studies has now been deleted.

Studies have been initiated with a 11-inch diameter flat plate calorimeter to evaluate the effectiveness of the sealed ground hold section during repetitive cycling. A helium purge was maintained through the space surrounding the calorimeter during these tests, and gradual deterioration of the insulation effectiveness occurred due to inward permeation of the helium purge gas. The "k" (thermal conductivity) of one ground hold test section increased during twelve tests from approximately 0.04 to 0.11 Btu/in/hr/ft^2/°F. Helium leakage into the evacuated sealed ground-hold section as represented by this test is probably not too serious with respect to mission requirements, but it does indicate the difficulties that will be encountered when attempting to use this concept on large flight vehicles. The effectiveness of several variations of this insulation concept for micrometeoroid protection has been evaluated by hypervelocity impact tests. These tests have shown that this insulation will prevent damage to the outer wall of cryogenic fuel tanks when impacted by particles weighing 17 and 70 mg traveling at speeds up to approximately 22,500 ft/sec.

The insulation has been installed on a 30-inch diameter end-guarded cylindrical calorimeter. The 1/4-mil aluminized Mylar radiation shields were applied as a 1/2-inch wide tape using a filament winding technique. The overall thickness of the insulation on the tank was approximately 2 inches, and the weight was approximately 0.5 lb/ft^2.

Tests are planned with an insulated 30-inch diameter cylindrical calorimeter to evaluate the performance of the installed insulation under simulated outer space conditions, for ground-hold conditions, and for simulated aerodynamic heating during launch. Additional high velocity impact tests are planned to more fully evaluate the insulation as a protecting shield against micrometeoroid penetrations. Additional tests will be made with the 6-inch diameter space calorimeter and the 11-inch diameter flat plate ground calorimeter in order to further optimize this insulation.

2. Contract Title: Development of Lightweight Cryogenic Insulation Systems
Contract Number: NAS8-11761
Contractor: Goodyear Aerospace Corporation
Contract Cost: $349,865.00
Contract Duration: 20 Months
Negotiated: June 30, 1964
Responsible Individual: J. M. Stuckey

Purpose of Project:

The contractor was directed toward the development, fabrication, and experimental evaluation of advanced and improved lightweight insulation systems for cryogenic liquid propellant tanks of launch vehicles. The systems developed by the contractor were demonstrated to be capable of providing lightweight, efficient, and highly reliable thermal insulation when applied externally to cryogenic propellant tankage of launch vehicles and subjected to simulated operational conditions. The program consisted of the following five phases and included liquid hydrogen tests on the six most promising concepts:

Phase I - Literature survey of launch vehicle insulation systems
Phase II - Advanced insulation systems design and determination
Phase III - Insulation system fabrication
Phase IV - Liquid hydrogen tankage tests
Phase V - Evaluation of test results

The requisite insulation materials were designed to meet the following requirements:

a. The insulation material must be of low density and a low thermal conductivity. Design goals shall be a weight of less than 0.4 lb/ft^2 and a thermal conductivity of less than 0.15 Btu/hr/ft^2/°F/in.

b. The insulation material must be reliably hermetically sealed.

c. The attachment of the hermetically sealed insulation must give reliable service under pre-launch and launch operation.

d. The system must maintain structural integrity and reliability when subjected to the following environments:

Vibration - 6.0 g's at 20-150 cps
Acceleration - 6.6 g's maximum
Surface temperature - 400°F (204°C) for short times.

e. The system must be capable of insulating and sealing around protuberances and hardware penetrating the tank walls such as propellant feed lines and instrumental lines.

Shortly after the contract was initiated, the contractor was instructed to concentrate efforts on the development of additional information on the dual seal insulation developed by this Center. Later, the contract was extended in order that two additional sets of dual seal insulation panels could be fabricated, installed on a 6-foot by 8-foot tank, and evaluated for structural integrity by subjecting them to liquid hydrogen fill and drain cycles.

Technical Status:

Since this contract has expired, a summary of the total program accomplishments is given below:

The dual seal insulation system was evaluated, and engineering data obtained to qualify the materials and concepts and to achieve lower insulation weight and more reliable performance on large boosters by the following tests:

(1) Mechanical and thermal property tests of new materials

(2) Vibration and aerodynamic heating tests on insulation panels attached to oval-shaped tanks containing cryogenic fluids

(3) Measurements of the apparent thermal conductivity of selected dual seal insulation concepts by means of flat plate calorimeter tests

(4) Structural verification and heat transfer measurements on a large oval-shaped guarded calorimeter tank filled with liquid hydrogen and cycled through multi-fill ground-hold conditions, including a simulated ascent heating cycle.

The results of this investigation indicated that the dual seal insulation concept is an effective and reliable means of providing

thermal protection to the exterior of liquid hydrogen tanks of large booster vehicles. The thermal effectiveness of the sealed, cryopumped sublayer portion of the insulation system when tested under liquid hydrogen tankage conditions showed thermal conductivities approaching values theoretically predicted for this system. At ambient test conditions, thermal conductivities as low as 0.10 to 0.15 Btu/in/hr/ft^2/°F were obtained.

In the course of the program, four sets of dual seal insulation panels were fabricated and evaluated by liquid hydrogen tests on a large oval-shaped guarded calorimeter tank having an insulation surface area of approximately 100 square feet. These tests showed that under ground-hold conditions the thermal effectiveness of the system was not degraded by repetitive liquid hydrogen fill and drain cycles.

Insulation panels fabricated and bonded to test tanks in this program confirmed that the dual seal insulation can be installed on the sides of liquid hydrogen tanks in the weight range of 0.44 to 0.50 lb/ft^2. Further reduction in weight for this insulation is feasible. The weight of the insulation could be reduced by approximately 0.04 to 0.06 lb/ft^2 by bonding the Mylar core of the sealed cell sublayer portion directly to the tank wall.

3. Contract Title: Research Study on Development of Lightweight Thermal Insulation Materials for Rigid Heat Shields
   Contract Number: NAS8-11333
   Contractor: IIT Research Institute
   Contract Cost: $221,852.00
   Contract Duration: 27 Months
   Negotiated: June 25, 1964
   Responsible Individual: V. F. Seitzinger

   Purpose of Project:

The purpose of this project is to develop lightweight ceramic insulation materials having potential applicability for protecting the exposed structural members of launch vehicles from the engine exhausts. The requisite insulations must be suitable for use in a thermal environment (primarily radiant) of 40 Btu/ft^2-sec and simultaneous mechanical environments, which consist of a vibrational environment of 90 g's acceleration in the first case, and random vibrational levels of up to 2000 cps in the second case. Insulation materials developed under this program must have a bulk density of not greater than 60 lbs/ft^3.

The contractor is directed to select for laboratory investigation candidate materials which demonstrate the greatest promise of fulfilling the objective of this program. Candidate materials should have demonstrated high thermal efficiency and good thermal and mechanical shock resistance and must be capable of application to metal substrates that include both the sheet steels and the general class of materials described as "honeycomb."

Materials are required for insulation in both radiant and convective heating environments; therefore, it is expected that no single material will satisfy all requirements. Initiallly, emphasis shall be directed toward the development of a thermal insulation suitable for application in radiant heating environments. Such materials must have a high reflectivity over the wavelength range of1.0 to 2.4 microns. Ideally, the target material shall consist of a highly reflective porous ceramic material with a melting point of approximately 2500°F (1371°C). The materials development effort shall include the general class of ceramic materials described as "foamed ceramics" or modifications thereto. The incorporation of ceramic fibers as foam additives to improve mechanical characteristics shall be investigated. Both chemically bonded castable-type and prefired foams shall be investigated. Suggested materials for investigation include mullite, alumina, zirconia, and zircon. Binder systems to be considered for the chemically bonded foams shall include the soluble silicates, metal phosphates, metal oxychlorides, and modifications thereto. Efforts shall be directed toward advancing the state-of-the-art of foaming techniques. New foaming systems shall be investigated as a means of obtaining controlled foam density and micropore size with uniform thickness.

Technical Status:

A large portion of the work during this period has been directed toward improving the adherence of phosphate-bonded zircon foams cast on metal substrates. The effects of various additives, varying drying conditions, and different base coats on the interfacial bond strength were investigated. Migration of the phosphate binder has been the primary factor limiting foam-substrate adhesion. Methods employed to prevent binder migration and improve interfacial bonding include increasing the amount of monaluminum phosphate binder with accompanying increases of setting agents to obtain low-temperature bond-set characteristics, and the use of setting agents to accelerate the hardening of the foams.

Materials investigated as setting agents included hydrated aluminum oxide ($Al_2O_3 \cdot 3H_2O$), aluminum hydroxide ($Al(OH)_3$), and zinc oxide (ZnO). The $Al_2O_3 \cdot 3H_2O$ and ZnO were effective in lowering the curing temperature and retarding binder migration. $Al(OH)_3$ caused floating, resulting in foams with poor structural and adherence characteristics. Calcium oxide (CaO), magnesium oxide (MgO), and "plaster of paris" were also evaluated as "setting" additives. Although all three of these additives were effective in shortening the setting time, only CaO and "plaster of paris" improved interfacial bond strength. Results to date show that the phosphate-bonded zircon foams having high interfacial bond strengths have a striated micropore structure while the foams that have the desirable fine micropore structure, while the foams that have the desirable fine micropore structure have lower interfacial bond strenths. Experiments are in progress which will provide a better understanding of the role of these additives, so that phosphate-bonded zircon foams with high interfacial bond strength and fine micropore structure can be prepared.

Optimized potassium silicate-bonded zircon foams developed under this program are being evaluated under a radiant heat flux of 40 Btu/ft^2-sec and simultaneous mechanical vibrations which simulate the mechanical environment experienced by a full-size heat shield panel of the Saturn S-IC stage. The test panels consist of foams (1/2 inch x 12 inches x 12 inches) cast on stainless steel plates (0.093 inch x 13 inches x 13 inches). The 1/2-inch strip of bare metal around the edges of the plate is used for attaching the panel to the vibration exciter. An optimized foam was subjected to the above test conditions for 5 minutes. The back face temperature of the panel at the end of test was 204°C (400°F). Although the foam was still bonded well to the plate after the test, there was some interfacial debonding around the edges of the foam. It is believed that most of the debonding was due to warpage of the steel plate during test.

Phosphate-bonded zircon foams which can be cast onto metal substrates have been developed. The phosphate-bonded zircon foams have flexural strengths far higher than the corresponding silicate-bonded foams. Although the phosphate-bonded foam-metal substrate bond strengths have not yet been optimized, significant improvements have been obtained through various additives and curing conditions. Silicate-bonded zircon foams cast on metal substrates have withstood 5 minutes exposure to a radiant heat flux of 40 Btu/ft^2-sec and simultaneous random vibrational levels up to 2000 cps.

In the immediate future, emphasis will be directed toward optimizing the phosphate-bonded zircon foams with respect to mechanical strength, substrate adhesion, micropore structure, and insulating capability. Promising foams will then be evaluated under a radiant heat flux of 40 Btu/ft^2-sec and a simultaneous mechanical vibration of 90 g's acceleration. Evaluation of optimized potassium silicated-bonded zircon foams under a radiant thermal environment of 40 Btu/ft^2-sec and a simultaneous dynamic environment which simulates the Saturn S-IC heat shield environment will be continued.

4. Contract Title: Development of Alkali Metal Peroxide and Superoxide Blown Ceramic Foams
   Contract Number: NAS8-20089
   Contractor: United Aircraft Corporation, Hamilton Standard Division
   Contract Cost: $29,422.00
   Contract Duration: 9 Months
   Negotiated: June 17, 1965
   Responsible Individual: V. F. Seitzinger

   Purpose of Project:

The primary objective of this program is to develop a technique for preparing ceramic bodies that have a low density (not greater than 60 lbs/ft^2), low thermal conductivity, high reflectivity, and low emissivity and that are capable of maintaining structural integrity at

temperatures up to 1649°C (3000°F). The technique is based upon the development of a method of forming ceramic foams having uniform pore size and distribution, using oxygen (O_2) as the foaming agent and curing at a low temperature. The initial phase of the program will consist of the necessary exploratory work to develop foamed ceramic bodies utilizing alkali metal peroxides and superoxides as the foaming agents. During the latter phase of the program, foamed ceramics developed will be characterized as to thermal conductivity, thermal shock resistance, density, melting point, reflectance, emittance, flexural strength, and compressive strength.

Technical Status:

The major portion of the work during this period has been directed toward developing techniques for producing ceramic foams having a closed cell structure. Previous work has indicated that slurry compositions possessing a thixatropic character would act to entrain the blowing agent and tend to produce a closed cell structure. This approach was investigated thoroughly. "Baymal," colloidal alumina was used as a foam additive to impart a thixotropic character to the slurry. Properties of the initial formulations verified that this is a promising approach. However, the as-cast foams hardened so rapidly that longitudinal cracks developed in the foam structure before the blowing process was complete. Additions of ammonium dihydrogen phosphate ($NH_4H_2PO_4$) were effective in retarding the set time and eliminated this problem. This foaming technique coupled with optimized mixing procedures has been effective in reducing the open pore volume from 65 to 30 percent, and it is believed that the open pore volume could be reduced even further. However, this phase of the program was terminated to allow adequate time for characterizing the optimized foams. Because of the difficulty in obtaining a closed cell foam structure, the program was extended at no additional cost for two months.

Although a completely closed cell foam structure was not achieved by using a thixotropic slurry, considerable progress was made toward this goal. Foams prepared by this technique exhibit uniform pore size and structure.

The contractor is preparing the final report on this program which will include detailed data on the thermal conductivity, thermal shock resistance, infrared reflectivity, emissivity, melting point, and flexural and compressive strengths of the foams prepared.

C. Analytical Methods Development

1. Contract Title: Research and Development of Analytical Methods and Fabrication of Test Equipment for Determination of Hydrocarbon Contamination
   Contract Number: NAS8-2681
   Contractor: IIT Research Institute, Chicago, Illinois
   Contract Cost: $445,349.00
   Contract Duration: 42 Months

Negotiated: May 22, 1962
Responsible Individual: A. C. Krupnick

Purpose of Project:

This contract is directed toward development of analytical methods and subsequent design and fabrication of test equipment for determining the concentration of condensable hydrocarbons in compressed gases, and the detection and quantitative analysis of organic contaminants on launch vehicle surfaces, such as tanks, valves, transfer lines, etc., and degreasing solvents.

Technical Status:

The continuous condensable hydrocarbon analyzer prototype unit has been delivered. Based on the final report submitted by IITRI, the prototype instrument provides for continuous monitoring of contaminating levels of moisture, condensable hydrocarbons, and halocarbons in high-pressure gas streams such as helium, air, or nitrogen. The monitor can be calibrated automatically and is simple to operate. Audio and visual alarms indicate contamination levels above 0.2 parts per million.

The moisture contamination is monitored by an integrated Beckman moisture analyzer, the condensable hydrocarbon contamination is monitored by two hydrogen flame detectors, and the halocarbon contamination is monitored by a General Electric halocarbon leak detector.

The performance factors tested for the hydrocarbon and halocarbon analyzers were as follows:

a. Linearity of response
b. Sensitivity
c. Stability
d. Relative response
e. Interferences

The performance results are as follow:

a. The analyzers are linear for contamination levels of 0.05 to 0.5 ppm.
b. The analyzers are sensitive at 0.2 ppm.
c. Stability is satisfactory over an 8-hour period.
d. The use of squalane and Kel-F 10 as calibration compounds provides satisfactory relative response.
e. The observed interferences indicate that fluorosilicones are 1.5 times more sensitive to detection by the halocarbon analyzer than is Kel-F 10.

Delivery of this prototype constitutes completion of this contract; thus, future reports will not reference this program.

2. Contract Title: Development of Techniques and Instrumentation for Detection of Hydrogen Gas
Contract Number: NAS8-11510
Contractor: Beckman Instruments, Inc., Fullerton, California
Contract Cost: $229,337.00
Contract Duration: 38 Months
Negotiated: June 29, 1963
Responsible Individual: A. C. Krupnick

Purpose of Project:

This contract is directed toward the development of techniques and instrumentation for (1) rapid quantitative detection of hydrogen in oxygen, nitrogen, inert gases, or mixtures of these, and (2) the detection and quantitative determination of the rate of hydrogen flow, in other gases, past a given point. This work is being pursued in two separate phases: (1) the development of a hydrogen detector based on the polarographic principle, and (2) development of a hydrogen detector based on ultrasonic attenuation by hydrogen gas.

Technical Status:

Activities directed toward the polarographic hydrogen detector were devoted to the development of the silver-palladium-coated polystyrene membrane and further characterization of the detector performance.

A barrier film was installed in the polarographic detector in a reversed fashion and was found to have slightly better hydrogen diffusion characteristics. The change and improvement was attributed to the electrochemical reduction of the oxides during the reactivation process of the electrode. Thus, it is believed that palladium oxide is present on the surface of the barrier film and that it affects the disassociation kinetics of hydrogen. The presence of the oxide has been confirmed by electron diffraction elimination.

The detector cartridge was subjected to further tests and it was found that the heater circuit performance was degraded by an oscillation in the potentiostatic loop of the system. The oscillations endured for an undesirable length of time and were caused by the heater wire element stray capacitance. The system was analyzed and the necessary corrective action defined.

Development of the prototype polarographic hydrogen detection is considered to be 90 percent complete. The only problem remaining at this time is that of the barrier film. Studies are in process to find methods of circumventing the decrease in response caused by the catalytic action of the palladium oxide surface.

Future work on the development of the polarographic hydrogen detector will be devoted to further checkout of the system and to the development and evaluation of silver-palladium membranes. The development work will be pursued on the basis of establishing the nature of the oxides on the palladium surface and to define and institute corrective action. In addition, a prototype unit will be delivered sans barrier film to this Center for field evaluation.

Efforts on the development of the acoustical hydrogen detector were devoted to the experimental investigation of the detector performance with the acoustical calibration unit and fabrication of multilayered acoustical transducers. The preliminary testing of the calibration unit employing the phase shift method was corrected with the attenuation method. The correlation showed that at least part of the phase shift measured, thought to be due to the relaxation effect, could have been due to the phase shift of the acoustical standing wave. The standing wave phase shift occurs because of the change in acoustical impedance of the system with gas pressure.

Fabrication of multilayered transducers has been initiated, and a number of tests conducted. The transducers fabricated to date consist of three material layers: (1) the active crystal, (2) a quarter wave of some fluid media covering the entire interface of the active crystal, and (3) a quarter wave polystyrene layer bonded to the active crystal as well. The transducers fabricated in this manner have shown positive results though inconsistent to the extent of efficiency.

All further efforts on the acoustical hydrogen detector will be devoted to the design and development of a multilayered acoustical transducer.

D. Assessment and Evaluation of Blast Hazards

1. Contract Title: Study of Blast Hazards of Rocket Propellants
Government Work Order: H-61465
Contractor: 6583rd Test Development Group, EAFB, California
Contract Cost: $2,297,667.00
Contract Duration: 44 Months
Negotiated: June 7, 1963
Responsible Individual: W. A. Riehl

Purpose of Project:

A reliable philosophy with adequate experimentally derived backup data is required for predicting that credible damage potential which may be experienced accidentally from an explosion of cryogenic or storable propellants during launch operations of space vehicles. Despite scaled experimental explosions of Jupiter, Atlas, Centaur, and Saturn propellants, no methods exist for predicting the probable potential for such accidental occurrences or explosions. This task provides for

the design, execution, and investigation of controlled experiments and determination of the feasibility of numerically extrapolating these experimental techniques, results, and functional parameters to possible explosions of full-scale vehicles.

This project provides for a comprehensive test and data analyses program directed specifically toward producing definitive siting criteria with respect to blast hazards for liquid-propelled vehicle launch and test sites. A series of over 300 tests is proposed with propellant systems liquid oxygen /RP-1 fuel (LOX/RP-1) and liquid hydrogen (LOX/LH_2) propellant systems at scales of 200, 1,000, and 5,000 pounds of combined propellant per test. Various tankage configurations are to be tested with various failure modes, shock and squib initiation, and various delay times from tank rupture to initiation.

Technical Status:

The high velocity impact tests using Nitrogen tetroxide/ Aerozine 50 (N_2O_4/A-50) LH_2/LOX, RP-1/LOX, and chlorine trifluoride (CTF)/aluminum polyurethane has been completed and all data reduced. The results of the tests are summarized in the following tabulation.

Type	Target Configuration	Propellant Weight, Lb.	Terminal Yield Percent, TNT Equivalency
N_2O_4/A-50	Flat Wall	200	15
N_2O_4/A-50	Flat Wall	1000	23
N_2O_4/A-50	Flat Wall	1000	21
N_2O_4/A-50	Deep Hole	200	37
N_2O_4/A-50	Deep Hole	1000	34
LH_2/LOX	Flat Wall	200	121
LH_2/LOX	Deep Hole	200	163
RP-1/LOX	Flat Wall	200	21
RP-1/LOX	Flat Wall	200	20
RP-1/LOX	Deep Hole	200	57
RP-1/LOX	Deep Hole	200	77
CTF/Solid	Deep Hole	200	0.6

In the CTF/solid tests, 48 pounds of the total of 60 pounds of solid propellant was collected after the tests. This explains the low yield. A statistical analysis was made of the terminal data from these tests. The conclusions of these analyses is listed as follows:

a. For all propellant combinations, the deep hole target gives significantly larger yields than the flat wall target.

b. For N_2O_4/A-50, a variation in impact velocity from approximately 340 fps to approximately 580 fps did not significantly affect the yield.

c. For N_2O_4/A-50, a slight tendency existed for the yield to decrease with increasing propellant weight.

d. There were no significant differences between the yields from RP-1/LOX and N_2O_4/A-50.

The 100-foot drop tower for the simulated missile fallback tests has been completed and propellant loading systems are in final stages of checkout.

The second half of the replicate tests of the 200-pound RP-1/LOX confinement-by-the-missile test series has been completed and the data generated is being reduced and analyzed. Efforts of the immediate future will be directed toward continuing the 200-pound propellant weight tests in the bulkhead failure mode.

2. Contract Title: Determination of Flammability of Specific Propellant Combinations Under Varying Environmental Conditions
Government Order Number: H-76708
Contractor: Bureau of Mines, Pittsburgh, Pennsylvania
Contract Cost: $165,000.00
Contract Duration: 25 Months
Negotiated: June 8, 1964
Responsible Individual: C. F. Key

Purpose of Project:

This project provides for an experimental determination of the flammability limits, minimum ignition energies, quenching distances, and spontaneous ignition temperature for vapors from various mixtures of fuels and oxidizers with and without the presence of air and/or other diluents of interest.

Technical Status:

a. Limits of Flammability

Limits of flammability of 1,1,1-trichloroethane were determined in air at 100° and 200°C (212° and 392°F) to complete the data for this fuel at 1 atmosphere pressure. Similar flammability determinations were made for the 1-chlorobutane-oxygen-nitrogen system at 25°C (77°F) and 3 initial pressures (760, 100 and 50 torr); also for the 1,2-dichloroethane-oxygen-nitrogen system at 25°C (77°F) and 50 torr and at 100° and 200°C (212° and 392°F) at 760 torr. Table 4 summarizes the lower and upper limit-of-flammability data obtained to date in air and oxygen atmospheres for n-hexane and the above 3 fuels. In general, the range of flammability tends to increase with increasing temperature and pressure and is noticeably greater in oxygen than in air. It is also evident that the range of flammability for the halogenated hydrocarbons is greater than for n-hexane in an oxygen atmosphere.

b. Minimum Energy and Quenching Distances

Flat plate quenching distance and ignition energies were determined for 1-chlorobutane and 1,2-dichloroethane at 198°C (388°F). The results are summarized in tables 5 and 6. The mixtures investigated are the same as those previously investigated at 88°C (190°F); it has not been demonstrated that these mixtures correspond to the most easily ignitable mixtures at this higher temperature.

The neat mixtures of either fuel with oxygen have quenching distances between 0.012 and 0.016 inch and minimum ignition energies approximating 10 microjoules. Thus, these chlorinated hydrocarbon vapors at 198°C (388°F) are ignited as easily as the unchlorinated parent hydrocarbons are at ambient temperature. The dependence of quenching distance and minimum ignition energy upon pressure appears to be the same as for the parent fuels; the effect of nitrogen and helium dilution is also similar to that for the parent fuels at ambient temperature.

c. Spontaneous Ignition Temperature (S.I.T.'s)

Minimum S.I.T.'s were determined for n-hexane and 1-chlorobutane at subatmospheric pressures in atmospheres of oxygen, air and equilibrium mixtures of nitrogen tetroxide and nitrogen dioxide. A summary of the values obtained is shown in table 7, which also includes for purposes of comparison the data reported previously at atmospheric pressure. The reduced test pressures indicated in this table are the pressures at the time of ignition. In these determinations, there is an appreciable pressure rise upon fuel injection and the subsequent vaporization and heating of the fuel. Thus, the initial test pressures were adjusted downward prior to injection such that the ignitions or nonignitions occurred at the desired pressure conditions. For the two fuels employed here, the effect of reduced pressure on minimum S.I.T. was more noticeable in atmospheres of air or nitrogen tetroxide than in oxygen, and particularly when the pressure was reduced from 100 to 50 torr.

TABLE 4

Summary of Limits of Flammability Data

Combustible	Temperature °C	Pressure mm Hg	Lower Limit O_2	Lower Limit Air	Upper Limit O_2	Upper Limit Air
1,1,1-Trichloroethane	25	760	6.6	6.8	16.5	
	100	760	5.5	6.3	56.8	13.0
	200	760	4.1	5.9	59.9	14.3
n-Hexane	25	760	--	1.2	--	7.4
	25	100	1.6	2.0	39.8	8.6
	25	50	1.5	1.1	37.5	7.5
	100	760	--	1.22	--	9.0
	200	760	--	1.14	--	26.5

Table 4 (Continued)

Summary of Limits of Flammability Data

Combustible	Temperature °C	Pressure mm Hg	Lower Limit O_2	Lower Limit Air	Upper Limit O_2	Upper Limit Air
1-Chlorobutane	25	760	1.85	--	--	11.0
	25	100	2.25	--	53.5	8.8
	25	50	2.3	2.5	51.5	8.8
1,2-Dichloroethane	25	50	5.3	5.7	58.5	15.7
	100	760	4.0	4.5	67.5	17.3
	200	760	3.5	3.9	68.9	21.2

TABLE 5

Quenching Gap and Ignition Energy Data for Mixtures Containing 1-chlorobutane at 198°C, In a One Liter Bomb, with 1/2-inch Vycor Flanges

Mixture	Pressure mm Hg	Quenching Gap, inches	Ignition Energy, mj
20.0% C_4H_9Cl + 80.0% O_2	760	.012	.008
	190	.036	.159
	48	.155	2.1
11.6% C_4H_9Cl + 46.4% O_2 + 42.0% N_2	760	.018	.012
	190	.060	.393
	48	.290	4.18
11.6% C_4H_9Cl + 46.4% O_2 + 42.0% He	760	.020	.042
	190	.075	.659
	48	.310	5.56
8.0% C_4H_9Cl + 32.0% O_2 + 60.0% N_2	760	.028	.038
	190	.100	.635
	48	.375	6.51
8.0% C_4H_9Cl + 32.0% O_2 + 60.0% He	760	.032	.115
	190	.130	1.47
	48	.475	15.2
5.0% C_4H_9Cl + 20.0% O_2 + 75.0% N_2	760	.075	.297
	190	.255	2.77
5.0% C_4H_9Cl + 20.0% O_2 + 75.0% He	760	.142	2.36
	190	.450	17.9

TABLE 6

Quenching Gap and Ignition Energy Data for Mixtures Containing 1-2 Dichloroethane at 198°C, In a One Liter Bomb, with 1/2-inch Vycor Flanges

Mixture	Pressure mm Hg	Quenching Gap, Inches	Ignition Energy, mj
28.0% $C_2H_4Cl_2$ + 72.0% O_2	760	.016	.011
	190	.064	.189
	48	.220	3.72
16.7% $C_2H_4Cl_2$ + 42.9% O_2 + 40.4% N_2	760	.026	.022
	190	.110	.646
	48	.420	6.38
16.7% $C_2H_4Cl_2$ + 42.9% O_2 + 40.4% He	760	.030	.044
	190	.130	1.09
	48	.525	16.5
11.9% $C_2H_4Cl_2$ + 30.5% O_2 + 57.5% N_2	760	.047	.087
	190	.172	1.14
11.9% $C_2H_4Cl_2$ + 30.5% O_2 + 57.5% He	760	.062	.396
	190	.235	3.50
7.6% $C_2H_4Cl_2$ + 19.4% O_2 + 73.0% N_2	760	.130	.921
	190	.400	10.5
7.6% $C_2H_4Cl_2$ + 19.4% O_2 + 73.0% He	760	.187	7.17
	190	.600	No Ignition

TABLE 7

Minimum Spontaneous Ignition Temperatures in Oxygen, Air and Nitrogen Tetroxide (NO_2*)[1] Atmospheres at Various Pressures

		Minimum S.I.T., °C			
		Pressure, mm Hg			
Combustible	Oxidant	760	400	100	50
n-Hexane	Oxygen	218	234	260	268
	Air	234	244	268	296
	NO_2*	216	238	316	364
1-Chlorobutane	Oxygen	235	238	246	252
	Air	240	246	264	302
	NO_2*	248	272	318	362

[1] NO_2* = equilibrium mixture of N_2O_4 and NO_2.

3. Contract Title: TOFLOX Synthesizer Studies
Contract Number: NAS8-20099
Contractor: Air Reduction Company, Murray Hill, New Jersey
Contract Cost: $53,550.00
Contract Duration: 8 Months
Negotiated: May 27, 1965
Responsible Individual: Harold Perkins

Purpose of Project:

This division is conducting tests to determine blast hazards associated with the primary rocket propellant combinations being used in the Saturn V vehicle. These propellants, RP-1, liquid oxygen (LOX), and liquid hydrogen (LH_2), exhibit high TNT equivalences when extensively mixed before ignition. It is believed that an appropriate additive, trioxygen difluoride (O_3F_2), will cause hypergolic ignition upon contact, thus preventing extensive mixing of the unreacted propellant and resulting in a lessened TNT equivalency. The contractor is to fabricate an O_3F_2 synthesizer and an O_3F_2/LOX mixing pilot plant capable of producing 25-gallon quantities of 0.05 percent O_3F_2/LOX (TOFLOX). This equipment is to be operated at this Center.

Technical Status:

During the program, the synthesizer was fabricated and qualified. The pure O_3F_2 is prepared by the glow discharge reaction produced when premixed oxygen and fluorine (60-40 percent), are introduced at approximately 20 torr into each of five, LOX-cooled, electrode reactors where the electrodes are maintained at a nominal 2,500 volts.

The synthesizer developed under this contract employs five reactors to produce sufficient neat O_3F_2 to yield 25 gallons of TOFLOX in approximately six hours. Once the O_3F_2 is transferred into the LOX mix tank, the reactors may begin to produce another batch of O_3F_2. The mix tank stirs O_3F_2 and LOX until chemical analysis indicates a desired concentration is attained. This time is nominally six hours for 0.10 percent solution.

The reactor has been delivered and installed at this Center and personnel of this division have been trained in operation of the system.

This contract is complete and will not be referenced in future reports.

E. Development of Materials for Special Purpose Electrical Equipment

Contract Title: Development of Materials for Slip-Ring Assemblies
Contract Number: NAS8-5251
Contractor: IIT Research Institute

Contract Cost: $234,580.00
Contract Duration: 38 Months
Negotiated: March 5, 1963
Responsible Individual: J. C. Horton

Purpose of Project:

Miniature slip-rings are employed to transmit electrical information across the gimbal axes in inertial platforms. These slip-rings must have long life and extremely low noise level. This contract is directed toward the development of materials for the slip-ring base (body), slip ring, brush block, and brush which possess the desired properties of long wear life, low noise, and resistance to contamination.

Technical Status:

Operation of slip-rings in a vacuum environment has begun following successful operation in air. Completion of the test apparatus was followed by an extensive evaluation of the vacuum system to insure that no backstreaming oil vapors were reaching the test rings. Once this requirement was satisfied testing on standard gold slip ring-gold alloy brushes with special vacuum compatible insulations was initiated. Two run-in tests were completed at a pressure of $2X10^{-7}$ torr with no lubricant applied. Each test was run for 280 hours continuous rotation at 200 RPM. As expected, noise levels rose to one millivolt within a few hours and to more than 1.0 volt in 60 hours. The test apparatus is designed to permit deposition of various lubricants during rotation of the rings and with brush current flowing. This will permit determining the effectiveness of the lubricant and its influence on the electrical behavior of the contact.

Testing of hard gold overlays in an inert atmosphere is continuing. Rings plated with two different types of hard gold overlay were tested for 300 hours at 200 RPM with 25 milliamperes of current flowing. Noise levels remained below 100 microvolts throughout both tests. However, a fluffy black powder formed on one ring set (as has been reported in inertial platform slip rings). Although it did not interfere with proper operation, the debris was collected for analysis. Emission spectrographic analysis revealed that no metallic debris was present. The analysis for organic constituents is in progress.

Effects to improve the quality of the electro-deposited gold are continuing in two areas. Ultrasonic agitation of the bath during plating has resulted in a smooth uniform plate. This material is presently being evaluated. Hardening of the gold plate by the addition of up to 10 percent noble metal ions has begun with platinum added to a nominal 24 K soft gold plating bath.

Slip rings composed of 24 K gold rings with a 0.0005-inch hard gold overlay operating against gold alloy brushes have operated up to 500 hours with noise levels less than 100 microvolts. The noise level indicates a threshold effect, depending on oscillatory amplitude, being linearly dependent on amplitude up to 0.4 degrees, and independent of amplitude beyond this point. Surface lubrication with a 1 percent oil, 99 percent freon solution has improved both wear and noise characteristics on both experimental and commercial slip-ring assemblies and has enabled operation up to 10 times required life time.

Evaluation of the effects of lubrication on slip-rings in vacuum will be carried out with the slip-rings operating to determine the effect on both wear and on electrical noise. Evaluations will be continued to determine the identity and source of the black deposits.

F. Nondestructive Testing Techniques

1. Contract Title: Development of Nondestructive Testing Techniques for Honeycomb Heat Shields
Contract Number: NAS8-11733
Contractor: North American Aviation, Incorporated
Contract Cost: $215,271.00
Contract Duration: 27 Months
Negotiated: June 29, 1964
Responsible Individual: W. N. Clotfelter

Purpose of Project:

The objectives of this project are: (1) the development of techniques and instrumentation for the nondestructive evaluation of composite materials in which all instruments, probes, detectors, are located on one side of the material only; (2) the modification of these nondestructive techniques to include the capability of measuring bond quality as well as the determination of no-bond conditions; (3) the establishment of system calibration procedures and the ultrasonic energy response characteristics as a function of various inherent mechanical defects in the material being evaluated.

Technical Status:

The first phase of the program was completed satisfactorily during the first year of the contract. Techniques and instrumentation have been developed which can be used to determine bond or no-bond conditions from a single side of honeycomb cored sandwich material. An impedance technique proved best for the evaluation of composites having HRP honeycomb cores. A new "eddy-sonic" technique has been developed that is best for inspecting composites of the dual-seal type. This involves vibration of the thin metal surface of the insulation with an electromagnetic transducer. The resulting acoustic waves travel throughout the composite and are reflected from the skin. Changes in this

reflected energy indicate debonds. Several techniques are being reviewed for possible application to bond quality measurements.

Current work involves correlation of bond degradation and the ultrasonic measurement of adhesive bond properties. Also, vibrational studies have been conducted using adhesive bond joints in various excitation modes. The vibrational modes proved highly complex and extensive mathematical analyses were required to define their parameters. Preliminary results show that the parameters indicative of bond quality may be masked by other factors. However, the analysis is continuing in an effort to separate these parameters.

Two very effective systems have been developed for the nondestructive evaluation of composite honeycomb materials. An impedance technique is the best all round method, it can be used to locate debonds on both the HRP type composite and the dual-seal insulation from a single side. However, the eddy-sonic technique is much better for dual seal insulation. Vibrations can be induced into the composite with no mechanical contact. This is a very important feature since the insulation can be damaged. A single scanning and recording system has been developed that can be used with either technique described above. The accomplishments described above are the result of many transducer and electronic circuit developments and modifications.

Techniques for the measurement of adhesive bond quality are being studied. Complete impedance and eddy-sonic systems will be delivered to this Center. The eddy-sonic system will be improved by the development of the capability for determining the interface at which a debond occurs. Other work will be directed toward the development of an "air-coupled" technique to eliminate contacting transducers.

2. Contract Title: Development of Nondestructive Methods for Determining Residual Stress and Fatigue Damage in Metals
Contract Number: NAS8-20208
Contractor: R. W. Benson Associates
Contract Cost: $98,008.00
Contract Duration: 14 Months
Negotiated: July 9, 1965
Responsible Individual: W. N. Clotfelter

Purpose of Project:

The object of this program is to develop a nondestructive technique for evaluating residual stress in structures and components after completion of manufacturing. The need for this technique stems from the intensive use of high strength aluminum alloys, in their highest strength temper, in launch vehicles. The design application

of these materials, coupled with the factor of unknown residual stresses, results in added danger of stress corrosion to susceptible materials. After review of applicable nondestructive testing techniques, a select few will be evaluated in detail. Major emphasis will be placed on high strength aluminum alloys with some minor effort devoted toward selected steels.

Because of the potential relationship between stress corrosion cracking and fatigue cracking, limited attempts will be made to determine whether fatigue faults can be the initiation point for stress corrosion failure.

Technical Status:

Numerous ultrasonic techniques have been investigated for the measurement of residual stress. These include birefringence, surface wave velocity, and the refraction of ultrasonic waves. Birefringence and surface wave velocity techniques have been applied successfully to laboratory specimens.

Conventional ultrasonic birefringence measurements are made by observing the change in polarization of shear waves. These measurements are relative and not suitable for the absolute measurement of stress in a sample. In another technique two receiving crystals are used to measure principal components of vibration simultaneously. A rate of the amplitude along the direction of particle motion to the perpendicular amplitude is obtained. This ratio is the tangent of the angle of polarization. If the frequency of operation is chosen properly, it results in an absolute measurement of stress.

It has been demonstrated that absolute changes in uniaxial stresses can be measured with high accuracy by the use of ultrasonic shear waves propagating through aluminum.

To accomplish the results indicated above, improved transducers have been developed. These transducers represent a real advancement in the state-of-the-art.

The simultaneous application of shear and surface waves is planned. The objective is to obtain a velocity standard for the particular alloy being evaluated. Thus, corrections may be applied to surface wave velocity changes to obtain more accurate stress measurements. An indication of stress distribution may then be obtained by using a range of surface wave frequencies.

G. Investigation of Corrosion Phenomena

1. Contract Title: Development of a Rapid Stress Corrosion Test for Aluminum Alloys
   Contract Number: NAS8-20285
   Contractor: Kaiser Aluminum and Chemical Corporation

Contract Cost: $33,690.00
Contract Duration: 12 Months
Negotiated: March 1, 1966
Responsible Individual: T. S. Humphries

Purpose of Project:

This contract is directed toward development and qualification of a rapid test method (not to exceed approximately one week) for determining the susceptibility to stress corrosion of aluminum alloys. The validity of the rapid test method shall be demonstrated through correlation of test results with data from conventional, relatively long term, stress corrosion tests.

The alloys and alloy tempers to be used in the development of the test technique shall include but not necessarily be limited to the following:

Alloys	Tempers
2024	-T3, -T4, -T6, -T8
2219	-T31, -T37, -T4, -T6, -T81, -T87
7075	-T6, -T73

Test specimens of other alloys and heat treatments may be incorporated into the development program subject to mutual agreement between the contractor and the contracting officer's representative.

Using test specimens of the stipulated alloys, the contractor shall proceed with the development and qualification of a rapid stress corrosion test method suitable for use in the laboratory. In the development of the requisite test procedure, the contractor may use various chemical solutions in various concentrations, induced current, and other such controllable environmental parameters as may be appropriate. To prove the validity of the rapid test technique, the contractor shall test the stipulated materials by the developed technique and by means of more conventional long-term tests. One conventional test shall consist of intermittent immersion of stressed specimens of the alloys in a three and one-half percent solution of sodium chloride. The test device shall provide for automatic repetitive cycles in which the stressed specimens are immersed in the corrodent for 10 minutes and withdrawn and held in the atmosphere for 50 minutes. As a part of the qualifying test procedure, the contractor shall test, by the developed technique and by the means specified above, material in the short transverse direction of grain orientation.

Technical Status:

Inasmuch as this contract was awarded only recently, there is no specific progress to report at this time.

2. Contract Title: Development of an Accelerated Stress Corrosion Test for Ferrous and Nickel Alloys
Contract Number: NAS8-20333
Contractor: Northrop Corporation, Norair Division
Contract Cost: $46,300.00
Contract Duration: 12 Months
Negotiated: March 17, 1966
Responsible Individual: T. S. Humphries

Purpose of Project:

This contract is directed toward development and qualification of an accelerated test method for evaluating the susceptibility to stress corrosion cracking of ferrous and nickel alloys. The preferable test period should be 30 to 90 days and in any case should not exceed 180 days. The reliability of the accelerated test method shall be demonstrated through correlation of test results with published data, service experience, and long-term exposure such as seacost and industrial atmospheres. The requisite method should be easy to implement, be safe, and have the capability of testing numerous specimens simultaneously without involving elaborate or an excessive amount of equipment.

A variety of alloys shall be used in the program to indicate the versatility of the test method for use in evaluating the stress corrosion characteristics of the various classes of ferrous and nickel alloys. The alloys to be used in the development of the test technique shall include but not necessarily be limited to the following:

AM-355 CRES (fully hardened SCT 1000 and SCT 850)
17-4 PH CRES
304 CRES
410 CRES
18 Nickel Maraging Steel
H-11 Steel
Vascojet 1000
4340 Steel
Inconel 718

Using test specimens of the alloys agreed upon, the contractor shall proceed with the development and qualification of an accelerated stress corrosion test method suitable for use in the laboratory. In the development of the test procedure, the contractor may use various chemical solutions in various concentrations, various stress levels, and other controllable test parameters as may be appropriate for inducing stress corrosion cracking in a reproducible manner in a relatively short time. To prove the validity of the accelerated test technique, the contractor shall test the chosen materials by means of the developed technique and by more conventional long-term tests such as a seacoast atmosphere.

Technical Status:

Inasmuch as this contract was awarded only recently, there is no specific progress to report at this time.

3. Contract Title: Investigation of the Directional Effects in the Stress Corrosion of Aluminum Alloys
Contract Number: Government Order H-2151A
Contractor: National Bureau of Standards, Washington, D. C.
Contract Cost: $59,000
Contract Duration: 12 Months
Negotiated: October 15, 1965
Responsible Individual: D. B. Franklin

Purpose of Project:

The purpose of this program is to develop an explanation of the cause of stress corrosion in high strength aluminum alloys and to identify the reasons for the large differences in susceptibility to stress corrosion associated with the directional effects of grain orientation. The primary emphasis in this study is directed toward identifying and correlating the metallurgical factors which affect stress corrosion associated with grain orientation, i.e., short transverse, long transverse, and longitudinal. The alloys to be studied in this program include 7075-T6, 7075-T73, 2219-T37, and 2219-T87.

Technical Status:

Plate material and extrusions of each alloy and temper included in this program have been obtained and a detailed investigation is being made of the metallurgical characteristics of each sample material. A study is also underway to determine the experimental techniques to be used in the program. The mechanical properties of both notched and unnotched specimens are being determined. All of these studies are aimed at establishing the required information as to tensile properties and specific metallurgical characteristics of the material being studied and to provide a basis for the experimental investigations which will be required.

III. S-II Stage

A. Evaluation of Heat Shield Insulation Materials for Use in the S-II Stage

The effect of heat and vacuum on the insulating capabilities of highly reflective ceramic insulations is being studied indirectly by determining the change in their reflectance resulting from subjection to various heat-vacuum treatments. Some of the data obtained for use in

determining the contamination factors of the heat-vacuum furnace have been reduced. The data show that for the standard alumina samples the reduction in reflectance, resulting from furnace contamination, is as high as 28 percent at some temperatures. However, at other temperatures, there is very little contamination. The insulation currently used on the S-IB stage, M-31, has been tested at 500, 600, 700, 800, 900, and 1000°C (932, 1112, 1292, 1472, 1652, and 1832°F). The data have not been reduced as yet.

B. Development and Evaluation of Materials of Potential Use as S-II Stage Tank Wall Insulation

Experimental development has continued on improved lightweight insulation for cryogenic propellant tanks. As reported previously, this program has resulted in a new concept of insulation known as dual seal or sealed cell insulation. Recent efforts have been directed toward the following:

1. Primer and surface preparation evaluation studies for the outer aluminum foil vapor barrier

2. Evaluating the effects of various debond sizes, locations and distributions

3. Development of in-process and field repair techniques for defective or damaged areas

4. Further evaluation of the overall insulation concept for thermal and structural integrity.

During this report period a dual seal insulation panel was installed on the curved face of a 6-foot by 6-foot guarded calorimeter tank and was subjected to seven liquid hydrogen fill and drain cycles. Test data showed that thermal conductivity of the insulation varied from approximately 0.17 to 0.23 Btu/in/hr/ft^2/°F. The data showed no significant increase in thermal conductivity or structural deterioration of the insulation during the seven liquid hydrogen tests. Equipment is being obtained to simulate aerodynamic heating of the insulation while maintaining liquid hydrogen in the tank.

The sealed cell portion of the insulation was applied to a 24-inch diameter tank. This included insulation of both bulkhead areas and a section of the fill line. This tank has been subjected to four liquid hydrogen fill and drain cycles, and there was no visible evidence of structural deterioration of the insulation after these liquid hydrogen tests on this configuration.

Fabrication and evaluation of complete panels and individual insulation layers with specific defects and modified materials and fabrication techniques is continuing. Information is sought on the effects of these parameters by destructive pressure testing under varying time and temperature conditions. A radiant heating facility has been devised which effectively duplicates the S-II thermal profile over a small panel area. Adhesive AF-126 was evaluated with EC-2320 primer for the HRP (heat resistant phenolic) core to aluminum foil outer bond line. With this system, failure was noted in every case at the primer-aluminum interface. In similar tests which are incomplete at this time, Kapton polyimide film, bonded with HT-424 film adhesive to the HRP outer core, is being evaluated as a face sheet material.

Consideration is being given to a program involving the application of dual seal insulation to an S-IC fuel tank, followed by LH_2 (liquid hydrogen) fill and drain cycling tests. A detailed proposal for this effort is being formulated with other laboratories of this Center.

C. Development of Nondestructive Inspection Techniques for Common Bulkheads and Insulation

Impedance methods have been shown to be effective for nondestructively evaluating many types of composite materials when access is limited to a single side. In fact, these techniques are the best available for locating debonds in composites of the type used in common bulkheads. However, the requirement of having a liquid couplant limits the scan rate. Rather long time periods are necessary for a complete nondestructive evaluation of a large bulkhead. Thus, faster techniques are highly desirable. Air coupled techniques have shown considerable promise using sonic and lower ultrasonic frequencies. Work in this area has recently been resumed. A loud speaker was used as the source of sound and a microphone served as a receiver. Maximum penetration of a one-inch thick honeycomb panel was obtained with sound frequencies near three thousand cycles per second. However, debonds could be detected only when frequencies near 21 KCPS were used. This is as would be expected since the lower frequencies penetrate too well. The major difficulty in this area of air coupled honeycomb testing appears to be the establishment of uniform sound fields.

D. Developmental Welding

The study of the weld repairability of 2014-T6, 1/8-inch thick, using 2319 and 4043 filler metals and the automatic GTA process has continued. All welding has been completed using up to ten weld repairs. Tensile testing of these weldments has been completed and metallographic examination remains to be completed. Comparable evaluations, using manual welding, have been completed. Test data are being analyzed.

E. S-II Stage, Project Management (Materials)

Efforts are continuing on the coordination and resolution of problem areas related to the materials aspects of the S-IVB stage of Saturn. During this report period, these activities have included the following:

The S-II-T stage was successfully tanked with LN_2 (liquid nitrogen) at the Mississippi Test Facility. The hydrogen tank was filled to the 30 percent level and LN_2 was maintained in the tank for about 18 hours. One crack about 18 inches long was found in the sidewall insulation when the LN_2 was detanked.

Sections of the 2020 alloy stringers have been received from the Tulsa Division of the Stage Contractor for metallurgical examination. The contractor has experienced cracking of the stringers due primarily to installation techniques. Studies by this division will be concentrated on some anomalous properties of the extruded stringers.

The stage contractor (S&ID) has appointed Mr. Mel Cole to coordinate all of the welding activities for the S-II stage. Formerly, the welding responsibilties at S&ID were divided among seven organizations.

S&ID has completed qualification of the rigid heat shield although Structures Division, R-P&VE-S, intends to run some additional qualification tests as part of the High Force Program.

IV. S-IVB Stage

A. Development of Expulsion Bladders for the S-IVB Auxiliary Propulsion System

Activities have continued in this division on the development of molding techniques and designs for hemispherical bladders of potential use in the expulsion of the oxidizer and fuel from the tanks of the S-IVB auxiliary propulsion system.

A bladder crease tester, designed primarily for earlier use in evaluating films for cryogenic expulsion applications, is being modified in an effort to obtain reproducible data on candidate storable propellant bladder materials at room temperature. Several novel bladder configurations are under development for study on this equipment. These configurations are judged intuitively to be less susceptible to three-cornered fold damage.

The joint NASA/USAF nitroso terpolymer bladder development effort is now being negotiated as an Air Force contract which will be jointly supervised by personnel of this branch and the USAF Materials Laboratory. Final informal discussions pertaining to the request for proposal were held during a visit to the Air Force installation by the representative of this branch on March 2.

B. Investigation of Materials Problems Related to the S-IVB Workshop Program

1. Investigation of the Effects of Reduced Pressure on Internal Tank Materials

The liquid hydrogen tank of the S-IVB stage has been considered for use as a manned workshop. Thus, it is important to know the outgassing characteristics of the internal cryogenic insulation. Simultaneous weight loss and partial pressure determinations were made on samples of the polyurethane foam, Adiprene L-100, and Lefkoweld 109. The data confirm the previous measurements which indicated that the observed weight loss was due to desorption of adsorbed gases.

2. Study of the Effects of Thermal Shock on S-IVB Tank Insulation

Dynatherm D-65 is being considered as a passivating coating on the S-IVB liquid hydrogen (LH_2) tank insulation because simulated micrometeorite tests have shown the internal S-IVB insulation to be flammable. During filling of the LH_2 tank the passivating coating would be subjected to severe thermal shock and it was necessary to ascertain if the coating would crack or chip during filling. Therefore, thermal shock tests in LH_2 were made on samples of S-IVB LH_2 tank insulation with 2 one-mil thick sprayed coatings of Dynatherm D-65. Three sets of two samples each were immersed in LH_2 for 15 minutes. Each set of samples was inspected visually afterwards. There was no evidence of chipping, cracking, or flaking of the D-65 coating after immersion in liquid hydrogen.

3. Study of Materials Problems Attendant to Development of a Life Support Atmosphere in S-IVB Tanks in Space

Activities have continued in the study of materials problems attendant to the use of the hydrogen tanks of spent S-IVB stages as manned chambers in space. This work includes theoretical considerations and experimental testing of each of the following areas:

a. Study of the Effects of Meteoroid Punctures

Testing has continued at the Arnold Engineering Development Center (AEDC) in the study of the effects of simulated micrometeoroid impact and puncture on S-IVB tank wall materials and insulation in contact with various mixtures of air, oxygen, and other gases. Preliminary conclusions based on test results to date indicate that high velocity particle impact causes the insulation to burn in 5 psia oxygen with or without the protective coating and similar impacts in approximately 50/50 mixtures of oxygen and nitrogen indicate that the tendency of the overcoated insulation to ignite is reduced somewhat. Additional tests should be conducted to further investigate this quenching effect if this mixture is contemplated for use.

b. Flammability of Materials in Gaseous Oxygen

An apparatus has been designed to study the flammability of materials under conditions approximating space cabin atmosphere. This apparatus has provisions for testing material in both pure O_2 and O_2/inert gas atmospheres at reduced pressure. Ignition energy will be supplied by an electric spark source. Fabrication of component parts and procurement of support sub-systems have been initiated.

c. Investigation of Hydrogen Diffusion Into and Subsequently From Insulation

Experimentation was initiated to determine whether hydrogen will diffuse into and from S-IVB insulation when this material is brought into contact with liquid hydrogen and then removed from the hydrogen. These studies have been delayed because of difficulties with the residual gas analyzer used to monitor the hydrogen concentration. It is anticipated that data from this study will be available during the coming report period.

C. Development of Brush Materials for Use in the S-IVB Auxiliary Hydraulic Pump Motor

Continued difficulty with brush life in the auxiliary hydraulic pump motor operating in a helium environment has resulted in the decision to operate with dry air supplied from an on-board tank supply. This produces a weight penalty and a reduction in reliability since loss of the air supply for any reason will seriously limit the operational life of the motor. This division has been requested to develop brush materials capable of operating for 500 hours in a helium environment, in order to utilize the stage helium supply and eliminate the need for the air tank supply.

One test was made on brushes composed of 70 percent molybdenum disulfide and 30 percent silver pressed at 4,000 psi. The brushes were run at 2 psi helium pressure at 5,000 rpm and 2.0 amperes. As in previous tests, one brush ran quite well, while the other was unstable from the start and eventually began arcing. New springs will be installed and the test will be run again.

D. S-IVB Stage, Project Management (Materials)

Efforts are continuing in the coordination and resolution of problem areas related to the materials aspects of the S-IVB stage of Saturn. During this report period, these activities have included the following:

1. S-IVB All-Systems Stage

Catastrophic failure of the common bulkhead occurred prematurely during qualification testing of the shortened stage which is generally referred to as the "basketball." The stage contractor's analysis revealed that the failure was caused by separation of the weld which joins the common bulkhead T-ring to the aft dome.

2. S-IVB-503

A ten-inch-long crack occurred in the parent metal adjacent to the seal weld on the LOX side of the common bulkhead of the S-IVB-503 stage. Investigations revealed that numerous attempts had been made to weld the structure where the crack occurred. The structure was degraded by excessive heats of welding; thus, no further attempts will be made to repair this defect by welding. The crack will be repaired by sealing bolted doublers over the damaged area. This repair technique will be qualified by testing a similar repair to failure on the LOX tank of the hydrostatic test stage and by hydrostatic testing of the flight stage to 1.05 percent yield.

3. S-IVB-204

Minor corrosion was found in the LH_2 (liquid hydrogen) tank of S-IVB-204. Evidence of rust was noted on the LH_2 vent line and clamp, the helium bottle strap assemblies, and the Marman coupling. The vent line and clamp were cleaned and passivated with Pasa-Jel 101, the helium bottle strap assemblies were solvent cleaned, and two small stains which remained after cleaning were coated with FR primer. This corrosion was caused by moisture which was trapped in recessed areas of the tank after final cleaning. To guard against recurrence of this problem, purging of these areas with dry air or dry nitrogen is now required after cleaning.

4. Reviewed and commented on 172 specifications for inclusion in the S-IVB contract.

V. Instrument Unit

A. Corrosion Studies of Magnesium Lithium Alloys

All general corrosion and protective coating studies are continuing on magnesium-lithium alloys. Tests of both dissimilar metal couples and single panels with various protective treatments have been in progress for over 100 days. Dow 17 anodize plus epoxy paint continues to be the best protective treatment for both LA141 and LAZ933 alloy. All of the LA141 specimens stressed to 100 percent of the yield strength have been removed from test after 115 days exposure to 100 ppm salt solution. There were no stress corrosion failures; however, both bare and coated samples were very heavily attacked by general surface corrosion. Electropotential measurements of the AZ31B magnesium alloy have been taken in 100 ppm and 3-1/2 percent salt solution. The results of these tests are being evaluated. Additional work has been conducted with thicker electroplated coatings of nickel, cadmium, and gold. These coatings have not shown any promise in preventing corrosion on these magnesium lithium alloys.

VI. J-2 Engine, Project Management (Materials)

A. A meeting was held to discuss a recent failure of J-2 Engine LOX pump turbine wheel. The wheel was found to have a crack almost all the way around the hub and several radial cracks coming from the fir trees at the rim where the blades are attached. The cause of failure was not discussed in detail, other than in some previously cracked wheels, the cause was attributed to fatigue, and this wheel looks similar to the previously cracked specimens.

B. Representatives of the Quality and Reliability Assurance Laboratory submitted a sample of an in place tubing weld from J-2 Engine 2026 for examination by this division. The particular section submitted is a modification being made in the thrust chamber purge system and is a close-out weld, which, in accordance with present procedure, is difficult to purge with inert gas while the weld is being made. The weldment was split open and the internal surface examined. There was oxide scale on the weld metal and heat affected zone which did not appear to be adhering tightly to the surface. The two halves of the split tubing were immersed in LN_2 (liquid nitrogen) to see if the shock would cause any of the scale to flake off. Several particles of black scale, some as large as one-half mm (500 microns) were found in the bottom of the container after the LN_2 had boiled off. This type of condition could cause contamination of the engine system.

I. Contract Research

The status of the several advanced research and technology contracts under the technical supervision of this division is described below. In keeping with the previous policy of this division, this report presents the contract research effort in terms of a quarterly review. A brief statement of the scope of work of each contract is given together with the present status of the program.

A. Polymer Development and Characterization

1. Contract Title: Study of Polymers Containing Silicon-Nitrogen Bonds
   Contract Number: NAS8-20190
   Contractor: Southern Research Institute
   Contract Cost: $75,227.00
   Contract Duration: 12 months
   Negotiated: May 4, 1965
   Responsible Individual: J. D. Byrd

   Purpose of Project:

This contract is directed toward the synthesis, characterization, and evaluation of a class of polymers known as silazanes. These polymeric materials contain recurring silicon-nitrogen linkages in polymer chains with the molecular configurations being either linear or cyclic. This contract also provides for a study of the use of silicon-nitrogen compounds in the preparation of polymer structures which cannot be made through other routes, and polymers are studied which contain silicon-nitrogen units lined to other chemical entities in a polymer chain. These compounds are generally characterized by their resistance to elevated temperatures and, thus, may be of importance in several aspects of launch vehicle development.

Technical Status:

The ability of polymeric silphenylene ethers to retain compressibility, as measured with a durometer, under adverse conditions was studied. Low-temperature compressibility was improved when phenyl groups attached to the chain were replaced by methyl and when the position of substitution in the silphenylene ring was changed from *para* to *meta*. The best silphenylene for low-temperature compressibility was almost equal to a methyl silicone rubber, and was superior to Viton A. A silphenylene elastomer was superior to both Viton A and methyl silicone rubber in retention of compressibility after exposure to gamma radiation and after being heated at 350 °C (662°F), but it was inferior to both in tensile properties.

A foamed silphenylene elastomer was prepared by cross-linking a polymeric silphenylene amine with pentaerythritol. An elastomer was formed also by the reaction of terephthalic acid with hexamethylcyclotrisilazane, but it was highly sensitive to moisture and was soft enough to flow at 200°C (392°F).

Attempts to prepare difunctional polymers containing silicon, nitrogen, and phosphorus in the main chain resulted in cyclization with elimination of silicon.

The reaction of a tetraaminosilane with pentaerythritol is being studied in an attempt to prepare double-chain silaspirane polymers, but difficulty is being encountered because of extremely slow reactions and lack of solubility of pentaerythritol in inert solvents.

Attempts to prepare a silicon-nitrogen-aluminum monomer according to published data may have been successful.

If successful, this monomer will be used in attempts to prepare polymers which contain a silicon-nitrogen-aluminum repeating chain units.

In a study of the nature of silicon - nitrogen bonds, it was found that aminosilanes undergo rapid amine exchange even at room temperature.

Polymeric silphenylene ethers have been prepared which have low temperature compressibility properties similar to methyl silicone rubber. However, the silphenylene polymers withstand elevated temperatures better than the silicone rubber. Highly elastomeric foamed rubber having a silazane linkage has been prepared. More knowledge on the nature of the silicon-nitrogen bond has been gained.

Plans for the immediate future include attempts to prepare polymers with repeating Si-N-Al units, spirane polymers containing silicon-nitrogen bonds and further studies of reactions between organic isocyanates and silazane monomers.

2. Contract Title: Development of Improved Potting and Encapsulating Compounds for Space Applications
   Contract Number: NAS8-5499
   Contractor: Hughes Aircraft Company
   Contract Cost: $186,940.00
   Contract Duration: 36 months
   Negotiated: June 29, 1963
   Responsible Individual: W. J. Patterson

   Purpose of Project:

The primary objective of this research effort is the development of improved potting and encapsulating materials suitable for embedding

electronic modules and for use on printed circuit boards. It is desirable that the potting compounds not be adversely affected by sterilization techniques or by various parameters of the space environment. The main efforts of the research program are directed toward a synthesis program of new polymeric materials in an effort to develop the following target properties:

a. Linear coefficient of thermal expansion: 10-20 x 10^{-6} in/in°C
b. Dielectric constant: maximum of 3.0
c. Temperature stability: -55°C to 150°C (-67°F to 302°F)
d. Transparency
e. Adhesion: 50 psi (minimum)
f. Water absorption: maximum of 0.5 percent
g. Low temperature flexibility: -55°C (-67°F)

In addition, the potting compound shall meet the following electrical requirements: Maximum power factor of 0.01; minimum dielectric strength of 500 volts per mil, minimum volume resistivity of 10^{16} ohm-centimeters at 70°F (21°C); minimum surface resistivity of 10^{12} ohms per square centimeter at 70°F (21°C); and a minimum insulation resistance of 10^{-5} megohms. The synthesis effort designed to meet these requirements consists primarily of development of new epoxy-siloxane and urethane-siloxane copolymers. The materials resulting from these approaches are expected to exhibit more nearly optimum combinations of the required properties. The use of filler systems shall be studied concurrently with the resins developed in an effort to reduce the coefficient of thermal expansion characteristics.

Technical Status:

The projected synthesis of epoxy-siloxane polymers has continued with successful isolation of pure 1,3-bis(epoxypropylphenyl)-tetramethyldisiloxane as confirmed by elemental analysis of the synthesized compound. The compound is considerably less viscous than the standard diglycidyl ether of bisphenol A and is capable of accepting a higher percentage of filler while retaining a pourable viscosity. The diepoxide has been cured in combination with a standard epoxy resin precursor to yield a high strength solid characterized by the following electrical properties at one kilocycle:

Dielectric constant: 3.24
Dissipation factor: 0.007
Volume resistivity 7 x 10^{13} ohm-centimeters

An N-methylamiline-formaldehyde polymeric adduct has cured conventional epoxy resin prepolymers to high strength solids whose dielectric constants vary only from 4.09 to 4.17 over a temperature range of 25°C to 150°C (77°F to 302°F).

Siloxane-containing polyols derived from 1,4-bis (hydroxymethyl)-diphenyl-oxide have cured standard urethane prepolymers into flexible transparent coating materials that are slightly resilient at -78°F (-61°C).

This program has produced a new epoxy-siloxane polymer precursor that has significant utility as a potting compound. Polymeric adducts have been developed which upgrade the relevant electrical and physical properties of epoxy polymers. Urethane-siloxane polymers have been cured into coatings which possess good low-temperature resilience.

Plans for the immediate future include attempts to increase the aromatic content of the siloxane moieties in urethane-siloxane polymers to allow greater compatibility of the non-polar siloxane groups with the highly polar urethane linkage. A more highly functional epoxy-siloxane monomer will be developed to provide increased reactivity with the curing systems now being used. Trifunctional and polymeric silazane curing agents will be prepared for evaluation as epoxy curing systems.

3. Contract Title: Evaluation of Structural Reinforced Plastics at Cryogenic Temperatures
   Contract Number: NAS8-11070
   Contractor: Goodyear Aerospace Corporation
   Contract Cost: $318,673.00
   Contract Duration: 36 months
   Negotiated: June 29, 1963
   Responsible Individual: J. T. Schell

   Purpose of Project:

The contractor is directed to make further studies aimed at improvement of shear and bearing stress measurement methods in reinforced plastics and to assess the validity of the data obtained by such measurements. These studies may be extended also to other areas wherin deficiencies are found during other related aspects of this program. Further studies on engineering models or shapes may be needed to verify the data obtained by the optimized test techniques. Concurrently, a literature and industrial survey shall be made to compile and consolidate the recommendations of other authorities as to resin-reinforcement combinations which show promise for cryogenic applications. The basic engineering and thermal properties shall be measured for the most promising of these systems using the optimized techniques developed under earlier phases of this program. Data obtained shall be verified by studies on engineering models. A limited study shall be made of new advancements in composite materials that may be revealed by the industrial survey.

Technical Status:

Satisfactory test specimens and procedures for the measurement of key physical and thermal properties at cryogenic temperatures have

been developed. The tensile modulus, ultimate tensile strength and elongation, notched-to-unnotched tensile ratio, compressive strength, flexural strength, bearing strength, interlaminar shear strength, thermal expansion and thermal conductivity were determined by test methods developed during this program. These results have been subjected to a statistical analysis which has demonstrated the reproducibility of the data and has vindicated the test procedures which were developed. Two additional epoxy systems and one polyester system are being evaluated by the techniques developed earlier in this program using the Polaris weapons system materials as a model reinforcement system.

Significant progress has been made toward more efficient utilization of reinforced plastics in structural members for use at cryogenic temperatures. The test methods developed during this program have been verified by application to structural members. It was established that the percent-voids in a laminate has a distinct effect on the shear properties of the laminate. Preliminary data indicates that the curing system used has a decided effect on the low temperature properties of epoxy resin systems.

Two additional reinforcement systems, Owens Corning Fiberglass ECK-37 filaments and silicon carbide whiskers for interlaminar supplemental reinforcement, will be evaluated.

4. Contract Title: Research on the Synthesis and Evaluation of a New Class of Inorganic, Linear, Double-Chain Ladder-Type Polymers
   Contract Number: NASw-924
   Contractor: W. R. Grace and Company
   Contract Cost: $148,360.00
   Contract Duration: 24 months
   Negotiated: April 23, 1964
   Responsible Individual: L. R. Moffett, Jr.

   Purpose of Project:

This contract provides for a research effort directed toward the synthesis and evaluation of a new class of semiorganic, linear, double-chain, ladder-type polymers, consisting primarily of tetrameric phosphonitrilic rings linked together in a fashion that will not permit the formation of consecutive carbon-to-carbon bonds in the backbond chains. Such polymers should be expected to possess thermal and oxidative resistance in the range of 400°C to 1000°C (752°F to 1832°F). The program will include the following: (1) development of a satisfactory synthesis for the desired phosphonitrilic tetramer, (2) isolation and identification of each of its four geometrical isomers, (3) synthesis of double-chain, ladder-type polymers from appropriate derivatives, (4) synthesis of isomeric derivatives which can be used to prepare polymers, and (5) description of the polymers and the measurement of their chemical and physical properties.

Technical Status:

Efforts have been directed primarily toward the preparation of methyl phosphonitrilic chloride tetramers through several synthetic approaches. Initially, the alkylation through Grignard reagents of tetrakisdimethylamide derivatives of tetrameric phosphonitrillic chloride was investigated. However, only partial alkylation of the P-N ring system could be achieved in this manner, and it bacame necessary to explore other synthetic methods. Subsequently, the direct reaction of methyltetrachlorophosphorane with ammonium chloride in refluxing chlorobenzene was examined. From this reaction crystalline products could be obtained in very low yield, whose infrared spectra showed the presence of both trimeric and tetrameric species. Furthermore, these methyl substituted derivatives were found to be unstable as evidenced by changes in their infrared spectra which suggested rather rapid hydrolysis of the PCl groups to POH compounds. This hydrolysis was apparently followed by cleavage to methyl phosphonic acid.

Additionally, considerable effort has been directed toward the chemical and spectral examination of polymers derived from the β-trans isomer of $(\text{ØPNNH}_2)_4$ in order to determine the degree of ladder-type polymer present. During this period reagents have been sought that will selectively react with the terminal NH_2 groups on the amide polymer and not with the bridging NH groups. For example, it was attempted to react salicylalhehyde, acetyl acetone, and picrylchloride with amide polymers derived from β-trans-$(\text{ØPNNH}_2)_4$. The products of these reactions are in the process of being separated and will subsequently be examined by nuclear magnetic resonance in order to precisely determine the NH/NH_2 ratio present in the polymer.

The contractor has succeeded in preparing polymers of 20,000 number average molecular weight, representing 40 tetramer units, from the controlled thermal deamination of the β-trans isomer of 2,4,6,8-tetraphenyl-2,4,6,8-tetraamido-phosphonitrile. Previously, chloroform-soluble polymers were obtained by thermal deamination at 240-260°C (464-500°F) of β-trans-$(\text{ØPNNH}_2)_4$. These polymers were composed of only 23 tetramer units and had number average molecular weights of 12,000. The direct synthesis of $(CH_3PNCl)_4$ from CH_3PCl_4 and NH_4Cl has been accomplished with some degree of success. A method for carrying out this synthesis has been developed, but isolation and characterization of the product is still troublesome. Two products, <u>cis</u>-$(CH_3PNCl)_3$ and a non-geminally substituted $(CH_3PHCl)_4$ have been isolated. Definite indications of hydrolytic instability of methylphosphonitrilic cyclics have been obtained.

Future work will be concentrated on the complete characterization of the polymers obtained from the thermal deamination of β-trans$(\text{ØPNNH}_2)_4$. Some insight as to the degree of ladder-type polymer present will hopefully be obtained through both chemical and spectral examination of the products obtained from the reaction of the polymer with compounds that will selectively react with the terminal $-NH_2-$ groups. Additionally, refinement of the method for the preparation of $(CHPNCl_2)_4$ will be investigated.

5. Contract Title: Investigationof Addition-Polymerization of Self-Sealants
Contract Number: Government Work Order H-71461
Contractor: Arnold Engineering Development Center
Contract Cost: $57,000.00
Contract Duration: 24 months
Negotiated: March 4, 1964
Responsible Individual: L. R. Moffett, Jr.

Purpose of Project:

Under this government work order, the U. S. Air Force Materials Laboratory investigated self-sealing materials which function by base catalyzed addition polymerization. These materials would be used between inner and outer walls of spacecraft and, in principle, would seal micrometeoroid punctures by undergoing spontaneous chemical reaction in the vicinity of the puncture to yield a solid plug capable of retaining the capsule atmosphere.

This program was restricted to a study of α-cyanosorbic acid derivatives which show promise for the stated application.

Technical Status:

In an effort to prepare a diester of α-cyanosorbic acid for evaluation as a self-sealant, the preparation of 1,4-butanediol, bis(α-cyanosorbate) was attempted through the reaction of α-cyanosorbyl chloride with 2 molar equivalents of 1,4-butanediol. However, only material of polymeric character was isolated from the reaction. The α-cyanosorbyl chloride was prepared through the reaction of the acid with excess phosphorous trichloride.

Personnel of the Air Force have accomplished the synthesis of a number of alkyl esters of α-cyanosorbic acid. These studies have shown that the base catalyzed addition polymerization of these esters is not a particularly effective self-sealing concept in a space simulated environment. Evaluation tests with the dynamic meteroid simulator have shown that application of such systems as ester/triethylamine as micormeteoroid puncture sealants results in virtually complete loss of pressure; practically no sealing action is obtained. Furthermore, other systems such as diisocyanate/polyfunctional amines, have effected instantaneous seals with neglibible loss of pressure.

Inasmuch as this work order terminated on March 15, 1966, no additional work will be initiated on the use of alkyl-α-cyanosorbate esters as self-sealants.

6. Contract Title: Investigation of the Behavior of Polymeric Materials at Cryogenic Temperatures
Contract Number: Government Order Number H-92120

Contractor: National Bureau of Standards, Cryogenic Engineering Laboratory
Contract Cost: $110,000.00
Contract Duration: 21 months
Negotiated: January 23, 1965
Responsible Individual: W. J. Patterson

Purpose of Project:

The objective of this program is the investigation and elucidation of three aspects of the low temperature behavior of high molecular weight polymers.

The first problem area of interest is the adaptation of low temperature differential thermal analysis (DTA) techniques for the precise difinition and study of low temperature transitions in polymers. After appropriate refinement of this technique, it shall be employed as an investigative tool to study rate dependent and environmental effects on polymer transitions. Attempts also shall be made to define more rigorously the theoretical and practical significance of these transitions.

The low temperature dynamic properties of selected polymers of interest shall be studied by investigation of the residual elastomeric characteristics of the polymers at low temperatures. These investigations shall be made through use of a ball rebound instrument developed by the contractor. This ball rebound instrument also shall be used as an adjunct to the low temperature DTA tests for the study of low temperature transition phenomena.

The third subtask involves a study of methods for minimizing the glassy state thermal expansion coefficients of selected polymers. Novel fillers and/or techniques will be investigated for incorporating fillers and slected orientation in polymers.

Technical Status:

The NBS ball rebound apparatus has been modified to allow the dropping ball to strike the sample surface vertically. This has removed the uncertain error in measuring the rebound angle and errors incurred due to changing force vectors affecting the angular rebound of the ball as the minimum resilience of the sample is approached. This vertical drop technique is combined with the light beam and photo cell detector, a high speed counter, and an automatic printer to complete the apparatus. Resilience measurements taken from this apparatus are believed fairly accurate and amenable to interpretation. It was concluded that rotation of the sample was not feasible since any attempt to test a fresh area of the sample with each drop of the ball would incorporate more error into the resilience readings than it would remove. Resilience measurements

have been made on neoprene, isoprene, and polyurethane samples, and data have been gathered to represent regions of minimum resilience and glass transition coefficient of the polymers tested.

The outstanding contribution of the program has been the development of a highly automated resilience tester which may be expected to give meaningful estimates of glassy state transitions and minimum resilience, as well as providing an experimental link between the theories on dynamic properties of viscoelastic materials at cryogenic temperatures and phenomena observed in these materials.

The projected program calls for modification of the DTA apparatus for analysis of polymer specimens at pressures of 10,000 psi. The pressure dependence of the various endotherms and exotherms corresponding to polymer transitions will be observed. Attempts will be made to provide highly oriented samples for expansion coefficient measurements. Polymers which are characterized by unique cryogenic properties will be investigated by means of the resilience apparatus.

7. Contract Title: A Study of Teflon Bladder Design Criteria for Use in the Expulsion Propellant Tanks of the Apollo-Saturn and Lunar Orbiter Vehicles
Contract Number: NASw-1317
Contractor: Bell Aerosystems Company
Contract Cost: $450,000.00
Contract Duration: 9 1/2 months
Negotiated: October 1, 1965
Responsible Individual: L. R. Moffett, Jr.

Purpose of Project:

The overall program objectives of this contract are to provide the most direct rational means of achieving tank assembly and expulsion device qualification, and to provide a basis for assessing safety margins. These goals will be accomplished through the following list of specific program objectives:

(1) Derive methods and data to enable analysis and evaluation of bladder designs with respect to mechanical performance and life and also with respect to expulsion efficiency.

(2) Determine the margins of safety of the existing designs for the Teflon bladder tank programs for the Command Module, Service Module, Lunar Excursion Module, Saturn V/S-IVB Stage and Lunar Orbiter.

(3) If any of the designs are inadequate, determine the design changes necessary to achieve the required margins of safety.

(4) Increase overall knowledge pertaining to bladder quality control by reviewing and documenting processing, assembly and test procedures and history.

(5) Determine reliable parameters for evaluating bladder quality.

Technical Status:

Failure modes for Teflon FEP/TFE laminated bladder material have been classified as follows: (1) rolling of buckled crease; (2) small V, L, and U-shaped breaks through one or both layers; (3) punctures and scratches; (4) folding followed by biaxial tension; (5) rolling of buckled crease followed by biaxial tension; (6) thin, straight-line ruptures; (7) twist, and (8) extensive delamination with breaks in the Teflon FEP layer.

It has been observed that a substantial amount of damage to Teflon bladders originates from severe folds which appear in the bladder as a result of a partial or complete expulsion of propellants. It has been determined that at least four types of buckled folds can occur during the collapse of a bladder under external pressure: buckled fold (general buckling), buckled crease (local buckling), double buckled fold, and reentrant buckled fold.

Repetitive buckling tests indicate early failure of the FEP in laminates, but no failure in the TFE. The FEP in 6 mil laminates survives from 200 to 250 cycles when the initial fold diameter is twice the thickness of the material. Indications are that the TFE survives 10,000 cycles.

Horizontal Plexiglass tank tests have been made to be used for investigating bladder twist and expulsion efficiency. Initial observations using a standard command module oxidizer bladder of 9 mil thickness with a 6 mil center section have revealed several significant items:

(a) Severe buckling and some crease rolling is unavoidable during assembly. The folding operation, however, can be controlled to minimize buckles in the hemispherical section, although this is accomplished at the expense of increasing the number of buckles in the cylindrical section which is less susceptible to failure.

(b) Initial twist of 3-3/8 inches on the circumference developed on the first filling operation; there was no recovery as the bladder was filled to capactiy. Freon TF was the fluid within the tank.

(c) True stresses in 6 mil laminates rise sharply below a radius of curvative of 0.008 inch.

(d) The outer fibers in plastic bending develop about the same stress magnitudes whether the laminates have equal or unequal proportions of FEP and TFE.

(e) Bond line stresses in bending are very low for laminates with equal and unequal FEP and TFE proportions.

Use of vertical Plexiglass tank to observe bladder tests has resulted in the development of the following information:

(a) Identification of the critical vibration environments for the bladders.

(b) Observation and determination of the liquid and bladder response frequency.

(c) Estimation of the number of motion cycles (buckling, folding, rolling, etc.) that the bladders of each of the Apollo tanks will experience during qualification tests.

(d) Estimation of the fluid pressure acting on the bladders of each of the Apollo tanks.

This type of information is needed to establish design load criteria for the various Apollo tanks. Plans are being formulated for forthcoming Plexiglass tank tests to obtain further experimental data to include slosh observation and to determine the effects of the direction of the vibration input (X axis, Y axis, etc.).

It is visualized that the vibration load criteria, in conjunction with laboratory endurance data in such modes as rolling of double folds, repetitive buckling, etc., will enable evaluation of existing Apollo bladders. Furthermore, if the endurance data show any trend with material thickness, then the most suitable thicknesses may be indicated for each bladder.

Several important recommendations have been made to other contractors involved in the mainstream Apollo program:

(a) Rolling of double fold tests was recommended to evaluate the bladders received that are under specified thickness. This test method was adopted, and endurance data are being collected to include the entire range of bladder thicknesses received. The interpretation of the results will include reference to the bladder motion cycles estimated for each tank program in vibration.

(b) LEM tank project personnel have been advised of the relative severity of the LEM vibration schedules as compared to those of other tanks.

(c) Apollo Command Module tank project personnel have been advised that twist damage is severe and is likely to cause failure if twist exceeds 20 degrees. No control presently exists to limit twist to acceptable magnitude.

Major accomplishments by the contractor during this period have included the overall classification of the failure modes for FEP/TFE laminated bladder material. Additionally, Plexiglass tank tests have afforded invaluable information on the expulsion efficiency and damage incurred by the bladders under specific conditions. Vertical tank tests have been made for studying stress-strain in plastic bending for TFE and FEP laminates. These studies have allowed several important conclusions to be drawn from biaxial stress-strain data at various ratios of biaxial stress.

Future plans include the completion of horizontal tank design criteria, vertical tank design criteria, the establishment of quality inspection techniques, and continuing coordination with other groups concerned with this problem area.

B. Adhesives Development

1. Contract Title: Development of Structural Adhesives Suitable for Use with Liquid Oxygen
Contract Number: NAS8-11068
Contractor: Narmco Research and Development Division of Whittaker Corporation
Contract Cost: $407,166.00
Contract Duration: 39 months
Negotiated: June 29, 1963
Responsible Individual: W. E. Hill

Purpose of Project:

The purpose of this program is the synthesis and development of adhesives which will be useful for structural applications at the temperature of liquid oxygen and which will be compatible with liquid oxygen. These features are observed with some highly fluorinated and chlorinated materials, and the synthetic program is directed toward highly halogenated polymers.

Technical Status:

Synthesis of highly fluorinated monomers and polymers is continuing, with emphasis on preparation of vulcanizable prepolymers.

Effort is being made to endow a polymer system with useful engineering properties while retaining the LOX compatibility properties of polymers prepared earlier in this program. Because some of the processes developed to yield these materials are unique and may have other applications, progress reports originated under this program are classified "Confidential." Further details are omitted to avoid the need for classifying this report.

2. Contract Title: Development of Improved Semiorganic Structural Adhesives for Elevated Temperature Applications
Contract Number: NAS8-11371
Contractor: Monsanto Research Corporation, Boston Laboratory
Contract Cost: $259,260.00
Contract Duration: 26 months
Negotiated: June 29, 1964
Responsible Individual: W. T. Patterson

Purpose of Project:

The contractor is directed toward the synthesis and development of semiorganic polymers that are useful as structural adhesives at elevated temperatures of at least 600°F (316°C). The desired adhesive shall be characterized by a minimum shear tensile strength of 1,500 psi. Curing of the adhesive may be accomplished through coordinate bonding or by a conventional crosslinking mechanism. As a secondary objective, it is essential that bonding and curing of the adhesive be accomplished at temperatures not exceeding 350°F (177°C), and that curing be accomplished over a wide range of temperatures (50-100°F (-253°C)) using a minimum bonding pressure. It is desirable also that the new adhesives have useful strengths at cryogenic temperatures to -423°F (-253°C), i.e., minimum shear tensile strength of 1,000 psi and metal T-peel of five pounds per inch of width. Surface preparation and bonding techniques shall be simple and easily adaptable to manufacturing and assembly processes by technicians having little or no knowledge or experience in plastic technology.

Technical Status:

Synthesis efforts have continued to prepare chelate polymers having modifications of the 5,5'-methylene-bis(salicylaldehydebutylimine) group in order to increase polymer tractability and toughness. Efforts to incorporate siloxane and fluorinated alkyl bridging groups between the ligands have not produced usable polymer systems. A family of compounds typified by 1,4-bis(hydroxydimethylsilyl)-benzene has found significant utility, both for crosslinking the zinc and/or titanium chelate polymers and as a means of introducing substituents in the polymer backbone of the polychelatotitanosiloxanes.

The bidentate acetylacetonato-ligand attached to the titanium atom of the chelatotitanosiloxane polymers has been replaced by 1,4-dihydroxyanthraquinone by way of a ligand exchange reaction. This bis-bidentate

ligand efficiently crosslinks two prepolymer chains together at the titanium site.

At present three polymer systems are being optimized for service as structural adhesives. Type I is a copolymer of poly (5,5'-methylene-bis-(salicyl-aldehydebutylimine)-dibutoxytitanium) and poly (5,5'-methylene-bis-(salicylaldehydebutylimine) zinc) crosslinked with 1,4-bis(hydroxydimethylsilyl)-benzene. Type II is poly(5,5'-methylene-bis(8-hydroxyquinolino)-dibutoxytitanium) crosslinked with 1,4-bis(hydroxydimethylsilyl)-benzene. Type III is a copolymer of 1,4-bis(hydroxydimethylsilyl)-benzene, 1,4-bis-(hydroxydimethylsilyl)-biphenyl, and dibutoxydiacetylacetonatotitanium which is crosslinked by 1,4-dihydroxyanthraquinone in a ligand exchange reaction.

The systhesis effort has produced at least three polymer systems for evaluation testing. Polymer types I, II, and III are soluble in pyridine-like solvents in the uncured state. In the cured state, types I and II soften above 300°C (572°F) while type III softens at 250°C (482°F). The adhesion to aluminum substrates as determined on lap-shear specimens is 500-700 psi for types I and II, and 1000-1300 psi for type III polymers.

The three most promising types of polymer systems will be studied in terms of reducing the curing time, temperature, and pressure required to form an adhesive bond. Attempts will be made to adapt the best adhesive system to either a supported film adhesive or a two-part kit consisting of liquid prepolymer and curing agent.

C. Developmental Welding

1. Contract Title: Development of Welding Techniques and Filler Metals for High Strength Aluminum Alloys"
Contract Number: NAS8-20160
Contractor: Southwest Research Institute
Contract Cost: $79,940.00
Contract Duration: 9 months
Negotiated: July 27, 1965
Responsible Individual: R. A. Davis

Purpose of Project:

The purpose of this project includes fundamental studies of the weldability of the naturally aging aluminum alloy X7106. The contract scope of work has been expanded to study types of biaxial tests for welded structures of three aluminum alloys, X7106, 2219-T87, and 2014-T651. The program can be classified into three phases: (1) Determination of the best laboratory type biaxial test specimen for evaluation of aluminum alloy

weldments, (2) evaluation of biaxial properties of aluminum alloy weldments, X7106, 2219, and 2014, and (3) evaluation of the weldability of X7106 aluminum alloy.

Technical Status:

All welding operations have been completed for the experimental welding studies on the alloy X7106 using both the GMA and GTA welding processes and filler metal types 5456, 5556, and 5180. Specimens of the weldments have been mechanical tested after natural aging periods of 1, 2, 4, and 8 weeks. Results indicate that the DC straight polarity GTA process is better from the weld quality aspect than the GMA process. Of the three filler metals studied, the 5180 has proven superior from the metallurgical structure and mechanical properties aspect. A comparison of the crack susceptibility of the three alloys X7106, 2219, and 2014, using the Houldcraft specimen, indicates that alloy 2219 is the least crack susceptible with X7106 being second.

Testing of biaxial weld specimens is complete. For this phase of the study, the types of specimens used were (1) the MIT biaxial specimens, (2) flat sheet bulge specimens, (3) cylindrical pressure vessel specimens, and (4) the LTV tensile cross specimens. Analysis of the biaxial data obtained from these specimens for determination of the biaxial ultimate strength will be very complex since the strength must be determined from the load and strain measurements. Relating the load-strain measurement to the ultimate strength depends largely on the tangent modulus and "poisson's ratio" in the plastic region which are continuously changing that region.

2. Contract Title: Study of Dissimilar Metal Joining by Solid State Welding
   Contract Number: NAS8-20156
   Contractor: The Boeing Company
   Contract Cost: $98,800
   Contract Duration: 16 months
   Negotiated: May 20, 1965
   Responsible Individual: M. G. Olsen

   Purpose of Project:

The objectives of this program are twofold. The first objective is to study the mechanisms by which dissimilar metal joining of various alloy combinations is accomplished by roll bonding and press bonding and means by which such bonding could be facilitated. The second objective is to determine the characteristics of completed joints between dissimilar metal combinations. The following alloy combinations will be studied.

2219 aluminum alloy to 321 stainless steel
7106 aluminum alloy to 321 stainless steel
2219 aluminum alloy to 5 Al-2.5 Sn titanium alloy
321 stainless steel to 8 Al-1 Mo-IV titanium alloy
321 stainless steel to alloy 718
Inconel to 8 Al-1Mo-1V titanium alloy

The influence of the primary diffusion parameters (time, temperature, and pressure) and of the process variables (surface preparation, atmosphere and intermediate materials) on diffusion couples will be studied for the six dissimilar metal combinations. Metallurgical analysis will be performed to identify constitution of the diffusion zones, and to determine the influence of thickness and hardness of the diffusion zones on the properties of the joint. Additional studies will be made of the effect of material condition (heat treatment and strain hardening) on diffusion bonding. The most promising diffusion bonding techniques will be further identified by metallurgical evaluation, mechanical testing, leak testing, thermal shocking and by corrosion studies.

Technical Status:

A comprehensive state-of-the-art survey has been conducted. The survey consisted of a literature search and a tour to selected organizations. Preliminary experimental work has been conducted on solid state bonding of six dissimilar metal combinations. Press bonded specimens have been prepared to study influence of time, temperature, and pressure parameters on bonding of these materials in the bare condition. Initial results have demonstrated that diffusion bonded joints can be made for all six dissimilar metal combinations in the bare condition.

The results of the test work demonstrated that temperature is the most important factor in controlling the development of the diffusion zone in the alloy combinations, with time having a measurable but much lesser effect. Increasing the weld pressure above that required for intimate contact or initial deformation did not have an observable effect on the diffusion welding of the six material combinations using metal foils and electroplated metals for diffusion aids. Work has also begun on diffusion welding bare material in the cold worked condition using an inert atmosphere.

D. Alloy Development

1. Contract Title: Investigation of Foamed Metals for Launch and Space Vehicle Applications
Contract Number: NAS8-11048
Contractor: Ipsen Industries, Incorporated, Rockford, Illinois

Contract Cost: $143,714
Contract Duration: 29 months
Negotiated: June 29, 1963
Responsible Individual: W. B. McPherson

Purpose of Project:

The purpose of this contract was to develop and evaluate ductile foamed metals for possible application on launch and space vehicles. During the first year of this contract, six foamed metals, nickel, 316 stainless steel, aluminum, titanium, molybdenum, and H-11 steel, were developed and evaluated. During the second year, more extensive development and evaluation were conducted on four foamed metals; aluminum, titanium, stainless steel, and molybdenum. Specific contract objectives include development of ductile foamed metals with controlled densities; determination of the relationship between mechanical properties, densities, and pore size of foamed metals; development of sandwich structures; and investigation of fabrication and forming methods for producing usable shapes from foamed metals.

Technical Status:

The foamed metals studied were produced by suspending a metal powder in a liquid containing cementing and foaming agents. This mixture was then foamed under controlled conditions to produce a uniform cellular structure and subsequently dried to set the cementing agents. The green foamed metal billets were then vacuum sintered. During sintering the cementing and foaming agents were volatilized and driven off as the metal particles were sintered. This process was found to be applicable to most metal or alloy powders which could be sintered. The sintered foamed metals had a uniform cellular structure. The cell size, which ranged between 0.001 to 0.040 inches, could be controlled to some degree by varying the powder particle size and the liquid to solid ratio in the foaming step. Densities were generally reproducible to plus or minus one percent of theoretical.

Sintered foamed metals retain some of the original characteristics of the solid metal; however, their tensile, compressive, and flexural strengths and thermal conductivity are related to their densities. At ambient temperatures, foamed molybdenum, H-11 steel, nickel and stainless steel had tensile strengths ranging from 50 to 2500 psi and flexural strengths between 50 and 5000 psi over the range of densities tested. These properties varied directly with the density of foamed metals. The compressive strengths of foamed H-11 steel and molybdenum, at 2 percent deformation, ranged from 500 to 5000 psi over the density ranges studied. Compressive strengths for foamed nickel and 316 stainless steel were lower, ranging from 200 to 1500 psi over the density range studied. It was noted that the compressive test specimens deformed linearly with load to approximately 1.5 to 2 percent deformation and, in most cases, did not fracture.

Other physical properties such as vibration damping capacity and thermal expansion were also investigated. The vibration damping capacity of foamed metals is greater than that of the solid metal, while the thermal expansion coefficient is essentially unchanged. Foamed metals could be sawed and machined by normal machine shop techniques. Foamed metals were readily joined by vacuum furnace brazing techniques. Brazed sandwich structures, with face sheets of similar and dissimilar wrought metals, were easily fabricated.

It was concluded that the metal powder foaming and sintering process was applicable to many metal or alloy systems. The foamed product, while structurally weaker than wrought material, is capable of withstanding considerable compressive loading and deformation before failure. Foamed metals may have application where low density, low thermal conductivity, vibration damping and compressive plasticity are required while retaining the other basic physical properties offered by wrought metal systems.

The contractor has completed this program.

2. Contract Title: Development of Light Weight Magnesium Alloys for Low Temperature Applications
Contract Number: NAS8-11168
Contractor: American Machine & Foundry Company
Contract Cost: $123,500.00
Contract Duration: 21 months
Negotiated: June 19, 1964
Responsible Individual: H. L. Gilmore

Purpose of Project:

The ultimate objective of this research program is the development of one or more wrought magnesium base alloys with the following properties and/or characteristics:

a. Tensile properties shall equal or exceed the following:

(1) Ultimate tensile strength 45,000 psi at ambient temperature

(2) Yield strength (0.2 percent offset) 35,000 psi at ambient temperature

(3) Percent elongation (2.0 percent offset) 20 percent at ambient temperature

b. The notched/unnotched tensile ratio (k_t=10) shall equal or exceed 1.0 at ambient temperature and shall have as a goal 0.90 at -423°F (-253°C).

c. The weldability of the developed alloys by conventional MIG (metal inert gas) or TIG (tungsten inert gas) techniques shall equal or exceed that of the AZ31B magnesium alloy.

d. The developed alloys shall exhibit weld joint efficiencies of 80 percent or more at ambient temperature.

e. The developed alloys shall be adequately protected from corrosion with an available commercial coating.

Technical Status:

Of the five modifications made on the nominal composition of alloy II-4 (Mg-1Zr-3Th-7Li-6Zn-5Cd-6Ag) prior to scale-up preparations for the 50-pound melts, II-4B (Mg-C_6Cl_6-2Th-8Li-6Zn-2.5-6Ag) met the ambient temperature mechanical property goals required of the program through an optimum combination of rolling temperature and heat treatment. The results of the modification program further indicated that addition of hexachlorobenzene can produce grain refinement without embrittlement and that the brittleness caused by a high temperature solution heat treatment is not permanent; ductility can be restored by proper aging.

Three alloys were selected for scale-up studies, and preparation of the 50-pound melts has been completed. The pilot ingot material has been rolled into sheet at 400°F, 450°F, 500°F, and 750°F (204°C, 232°C 260°C, and 399°C). In all cases, the lower temperature rolling gives the highest and most stable properties. The tensile strength of alloy IA6 (Mg-3Th-9Li-2Zn-1Mn-4Al-4Ag) was increased to 50,6000 psi (at room temperature) by optimizing the rolling temperature, and it was determined that the stability of the alloy could be greatly increased by a rapid quench from the solution heat treating temperature. The best properties achieved with alloy ZLH972 (Mg-9Zn-7Li-2Th) resulted from rolling the material at 450°F (232°C). When properly worked and heat treated, the tensile strength of the alloy is about 43,000 psi or slightly under the goals of the program. Sheets of all three of the above alloys have been welded successfully and are being tested presently. Chemical milling tests and coating studies are underway.

E. Physical and Mechanical Metallurgy

1. Contract Title: Development of a High Strength Aluminum Alloy, Readily Weldable in Plate Thicknesses, and Suitable for Application at -423°F (-253°C)
   Contract Number: NAS8-5452
   Contractor: Aluminum Company of America
   Contract Cost: $191,586.00
   Contract Duration: 44 months
   Negotiated: June 28, 1963
   Responsible Individual: J. H. Hess

Purpose of Project:

This contract is directed toward the development of one or more high strength wrought aluminum alloys suitable for use in structural applications at temperatures from ambient to -423°F (-253°C). The alloy or alloys must be capable of being readily fabricated to plate thicknesses, and must have weldability, by conventional techniques, equal to or greater than that of aluminum alloys 5456 or 2219. The mechanical properties of the developed alloy or alloys must be higher than those of the weldable alloys which are now commercially available and must exhibit low notch sensitivity in both parent metal and weldments at temperatures down to -423°F (-253°C). The program will consist of a literature survey, a preliminary experimental program involving laboratory production and testing of approximately 30 alloys, an advanced evaluation of the six most promising alloys selected from the preliminary experimental program, and plant fabrication of two selected alloys in two plate thicknesses. Optimum thermal treatments will be determined for the selected alloys, weldability studies will be made, accelerated stress-corrosion tests will be performed, and a thorough mechanical property evaluation will be made.

Technical Status:

The technical status of this program is essentially the same as presented in the last report. The additional test data which were obtained since that time do not significantly change the results which were summarized in that report.

During this reporting period, the contract was modified to permit a fourteen-month continuation. During this term of the contract, efforts will be directed toward uprating the properties of the alloys X2021 and X7007, which were developed earlier in the program. For alloy X2021, the role of the cadmium and tin additions in promoting age-hardening will be established, and the relationship of varying amounts of these elements to the known harmful effects of cold working will be investigated. Studies will be made to determine the effects of varying amounts of cadmium and tin and dispersoid forming elements such as manganese on the quench sensitivity of thick sections of this alloy. In order to increase the weld joint efficiency of X2021, various experimental filler alloy compositions will be investigated and evaluated.

A concentrated effort will be made to improve the short transverse direction stress corrosion cracking resistance of X7007. Investigations will include a study of the effects of dispersoid forming elements such as manganese, chromium, tantalum, and columbium on this property. Additional studies of the effect of silver addition will also be made.

In addition to the investigations of the basic alloy modifications described above, the engineering properties of the current compositions and tempers of the alloys will be determined. Properties to be determined are shear strength, bearing strength, modulus of elasticity in tension and compression, fatigue strength and fracture toughness. In addition, physical properties of both alloys will be determined, including density, melting range, electrical conductivity, thermal conductivity, and thermal expansion.

2. Contract Title: Evaluation of High Strength Fasteners and Fastener Materials for Space Vehicles
Contract Number: NAS8-11125
Contractor: Standard Pressed Steel Company
Contract Cost: $188,044
Contract Duration: 24 months
Negotiated: November 4, 1963
Responsible Individual: R. R. Rowe

Purpose of Project:

The purpose of this contract was to characterize fasteners and fastener materials for space vehicles. The experimental work included (1) a survey to determine fastener requirements for space vehicle construction and to determine design criteria for proper fastener utilization, (2) evaluation of a selected group of 25 fastener classes in each of three sizes through standard and special test techniques, (3) development of standard testing techniques for fasteners that permit evaluation of fasteners with greater reliability for space vehicle use, (4) evaluation of ten new materials or processes that indicate promise in achieving higher strength fasteners, and (5) fabrication of the three most promising alloys into three fastener configurations in two sizes. Aluminum, iron, nickel, and titanium base fasteners will be included in the evaluation at -423°F (-253°C), ambient, and 1600°F (871°C) or to the maximum useful temperature of the respective fastener.

Technical Status:

This contract has been completed. Since the last period, all phase III (Evaluation of Potential High Strength Materials as Fasteners) fasteners have been fabricated and evaluated. The fasteners were made from 5Al-2.5Sn ELI Ti, U-212, Inconel 718, Waspaloy (220 ksi) and AF-1753 materials in two configurations (tension and semi-blind) and two diameters. The evaluation tests consisted of (1) tensile (material and fastener) at -253°C, -196°C, 21°C (-423°F, -320°F, 70°F), and maximum elevated service temperature, (2) angle block tensile at -253°C, -196°C, and 21°C, (-423°F, -320°F, 70°F), (3) stress durability at 21°C (70°F), (4) tension impact at -253°C, -196°C, and 21°C (-423°F, -320°F, 70°F), (5) double shear at -253°C, 21°C, (-423°F, 70°F), and maximum elevated service temperature, (6) semi-blind grip preload,

(7) stress rupture at maximum elevated service temperature for 1, 10, and 100 hours, (8) stress relaxation at one preload level and maximum elevated service temperature, (9) nut reusability at 21°C (70°F), (10) torque vs. induced load, and (11) mechanical properties after environmental exposure during stress relaxation tests. Accelerated salt spray and seacoast environmental corrosion tests were made under maximum preload condition for each fastener assembly.

Twenty-five classes of various structural fasteners were characterized to determine performance under various installation and test conditions. Ten new high strength materials, 18 Ni maraging steel, Unitemp 212, Inconel 718, 1Al-8V-5Fe Ti, 25 percent cold reduced Waspaloy, 5Al-5Zr-5Sn Ti, 5Al2.5Sn ELI Ti, 30 percent cold reduced L-605 cobalt alloy, AF-1753 nickel alloy, and Udimet 630 nickel alloy, were screened and evaluated as fastener materials and fasteners. Thermal cycling was determined to present no new problems that would decrease fastener performance. Vibration of fastener assemblies at cryogenic temperatures was determined to be less demanding on fastener performance than at ambient temperature. All titanium fasteners studied were found to be sensitive to minimal bending at -196°C (-320°F) except the 5Al-2.5Sn ELI which became sensitive at -253°C (-423°F). Corrosion performance of the fastener-structural vehicle material combinations under various pre-conditioning treatments was determined. Thread rolling sequence (thread rolling after final heat treatment) was established for optimum cryogenic temperature performance. Use of high cold worked fasteners for elevated temperature service was established as undesirable.

Fastener performance under shock loading at cryogenic temperature was determined. Comparisons were made on all the fasteners to establish relative rank for various service conditions or design criteria. A model procurement specification was illustrated and additional research and development areas in fastener testing was categorized.

3. Contract Title: Collection of Material Property Data and Presentation of Said Data in the Form of Material Data Handbooks
   Contract Number: NAS8-11345
   Contractor: Syracuse University
   Contract Cost: $54,976.00
   Contract Duration: 17 Months
   Negotiated: June 26, 1964
   Responsible Individual: M. G. Olsen

   Purpose of Project:

The purpose of this project is to collect, analyze, and present technical data in a single source for 14 metal alloys in the form of data handbooks. In addition to mechanical and physical property values, information on the following will be collected and analyzed: stress-strain curves, heat treatment procedures, effect of radiation,

vacuum and combined vacuum-solar environments on properties, resistance to corrosion in various media, surface treatments for various environments, alloy stability, forming and machining techniques, joining techniques, metallography and applicable specifications.

Technical Status:

The collecting and screening of information on the alloys to be covered is continuing. The first drafts of manuscripts for aluminum alloys 5456, 6061, and 7075, type 301 stainless steel have been completed. Handbooks on aluminum alloys 2014 and 2219 are completed. Drafts on the remaining alloys to be covered are in various stages of development.

4. Contract Title: Study of Hydrogen Embrittlement of Various Alloys
   Contract Number: NAS8-20029
   Contractor: Battelle Memorial Institute
   Contract Cost: $42,150.00
   Contract Duration: 12 Months
   Negotiated: June 24, 1965
   Responsible Individual: J. R. Lowery

Purpose of Project:

The purpose of this study is to investigate the susceptibility of selected structural alloys to hydrogen embrittlement which may result from various cleaning, etching, and electroplating procedures. As a part of the basic investigation, appropriate processing procedures to relieve hydrogen embrittlement once it has been induced in the selected alloys are to be explored.

The materials to be studied under this program include the following:

Alloy	Strength Level, psi
6Al-4V titanium	160,000
AISI H-11 steel	260,000
AISI 4130 steel	180,000
18 nickel maraging steel	260,000
Inconel 718	180,000
U-212 steel	180,000
Rene' 41	200,000
Waspaloy	260,000
17-7 PH stainless steel	200,000
17-4 PH stainless steel	210,000
AISI 8740 steel	180,000
AM-355 stainless steel	190,000
AISI 4340 steel	260,000
AISI 410 stainless steel	180,000

Technical Status:

The literature and industrial survey have been completed and the final report of the survey has been received.

Phase one of the experimental program, which is a preliminary study of the sensitivity of these alloys to hydrogen embrittlement, has been completed with the exception of alloys U-212 steel, AM-355 and 410 stainless steel. AM-355 stainless steel and U-212 steel are still being evaluated and the 410 stainless steel, being furnished by the government, should be processed and ready for evaluation in the near future. In this initial screening study, specimens of the various alloys were cathodically charged with hydrogen (the charging electrolyte was composed of four percent by weight of sulfuric acid at a current density of 8 milliamperes per square inch) while being stressed in tension at approximately 80 percent of their yield strength. The following alloys sustained the stress for a period of 200 hours or more and were considered to be insensitive to hydrogen embrittlement under these conditions:

Ti-Al-4V
Rene' 41
Waspaloy
Inconel 718

The alloys that failed by delayed brittle fracture in less than 200 hours under the stated charging condition and tensile stress were considered to be sensitive. Additional specimens of these sensitive alloys were tested further by decreasing the severity of cathodic charging (under constant tensile stress) in an effort to determine the minimum hydrogen content required for embrittlement and to rate the alloys on the basis of relative sensitivity under continuous charging. Hydrogen analyses will then be conducted on specimens charged under these limiting conditions. The following alloys were found to be sensitive under the above conditions and are listed in order of their increasing sensitivity:

17-7 PH Stainless Steel
AISI E8740 steel
18 Nickel maraging steel
AISI Type H-11 steel
17-4 PH stainless steel
AISI 4130 steel
AISI 4340 steel

Chemical analyses of these materials are being made. Hydrogen-entry rate experiments have been initiated. In order to evaluate further the alloy sensitivity and to rate the alloys (both sensitive and insensitive, as determined in the first part of this phase) on the basis of relative

hydrogen-absorption as well as relative embrittlement susceptibility. Preliminary tests on six of the alloys under study indicated that there is considerable scatter in the results but there is a definite indication that the materials absorb hydrogen at different rates. No attempt will be made to correlate the hydrogen entry-rate with susceptibility to delayed brittle failure until the entry rates of more of the materials have been determined.

Phase two, a study of the embrittling effects of hydrogen resulting from cleaning, pickling, and electroplating processes, has been initiated and the results of sustained load tests indicate that 4340 steel was severely embrittled by conventional cadmium plating. Other plating systems and alloys are presently in test.

Phase three, a study of hydrogen relief methods, will be initiated in the near future.

F. Composite Material Development and Testing

1. Contract Title: Development of Ultrahigh Strength, Low Density Aluminum Plate Composites
Contract Number: NAS8-11508
Contractor: Harvey Aluminum, Incorporated
Contract Cost: $278,731
Contract Duration: 38 Months
Negotiated: June 29, 1963
Responsible Individual: W. R. Morgan

Purpose of Project:

The objective of this research program is to develop methods for producing and to establish techniques for joining aluminum alloy sheet and plate composites reinforced with continuous steel wires. The required composites are to be produced in thicknesses of 0.250-inch and 0.750-inch thick plates and 0.040-inch sheet having a minimum tensile strength of 175,000 psi and a maximum density of 0.144 pound per cubic inch (3.986 grams per cubic centimeter). The aluminum alloy to be used in this research effort will be 5456 (or approved equivalent), and the reinforcement shall be of small diameter (not greater than 0.010-inch) steel wire of approximately 600,000 psi tensile strength.

Technical Status:

Results indicate that hot pressing is the most economical method for producing plate, and that hot rolling appears to be the best method for producing sheet or tape modules to be used in the production of formed shapes. The investigation of bonding has been divided into two areas: aluminum to aluminum and aluminum to steel wire. The best results with aluminum to aluminum bonding have been obtained by diffusion bonding the aluminum matrix at 482°C (900°F) and a pressure of 14,000 psi.

The most efficient bond between the aluminum matrix and the steel wire appears to be simply a mechanical bond. Three plates have been made (0.250 inch thick, 12 inches wide and 96 inches long) composed of 0.009-inch diameter NS355 wire and aluminum alloy 2024 matrix.

Results of screening tests on the first plate are given below:

Test Temperature	Condition	UTS (psi)
Room	As fabricated	150,000
Room	SHT, CW 2%, Aged	159,000
Room	SHT, CW 2%, Aged, CW 2%	161,000
-73°C (-100°F)	SHT, Aged	158,000
-129°C (-200°F)	SHT, Aged	173,000
-196°C (-320°F)	SHT, Aged	210,000

SHT - solution heat treated
CW - cold worked

V-notched charpy impact strength of sub-size specimens increased from 12 foot/pounds at room temperature to 18 foot-pounds at -196°C (-320°F). Notched/unnotched tensile ratios of about 0.95 were obtained using notched tensile specimens with a stress concentration factor K_t of 6. One small composite sample containing 49 volume percent of beryllium wire as the reinforcement and 51 volume percent of aluminum alloy 2024 as the matrix was produced and tested. Room temperature ultimate tensile strength of 87,500 psi was achieved.

Preliminary tests indicate that the most promising technique for joining plate material is as follows:

a. Etch out the aluminum matrix with sodium hydroxide to expose the steel wire on the ends of the two pieces of plate to be joined.

b. Clean the exposed wires with nitric acid.

c. Vacuum infiltrate the exposed wire on each piece with an alloy composed of 95 percent zinc, 4 percent aluminum and 1 percent copper.

d. Heat the two pieces to be joined in a jig, exerting pressure on the ends until a 3/4-inch overlap is obtained.

e. Hot press to size and remove excess zinc alloy.

Resistance welding appears to be a suitable technique for joining sheet composite material.

2. Contract Title: Development of High Strength Brazed Aluminum Honeycomb Sandwich Composites for Both Brazed and Cryogenic Temperature Applications
Contract Number: NAS8-5445
Contractor: Aeronca Manufacturing Corporation
Contract Cost: $205,000.00
Contract Duration: 36 Months
Negotiated: June 29, 1963
Responsible Individual: F. P. LaIacona

Purpose of Project:

This contract is directed toward the development of high strength, brazed aluminum honeycomb sandwich structure for use at moderately elevated and cryogenic temperatures in space or launch vehicles. These objectives are to be accomplished by joining heat treatable facing and core alloys by fluxless brazing. Phase I of the follow-on contract will continue the comprehensive literature survey. In Phase II, a selection of four additional, most promising braze alloy systems will be investigated, other than those systems selected previously. Emphasis

will be placed on procuring and investigating welded, diffusion bonded or any other type of bonding method for core material fabrication. Brazing methods will be investigated and evaluated, and it is expected that this will require the development of new tooling concepts based on faster quenching rates than are presently possible. Feasibility data will be established for joining the brazed specimens by fusion welding techniques. Phase III will be directed towards evaluation of the two most promising brazed alloys from the alloys screened. Non-destructive testing techniques will be included in this investigation. Brazed sandwich structures developed from this phase of work will be evaluated as required. Finally, at least two sections of a one-eighth segment of a sphere approximately 16 feet in circumference will be produced as an assurance that the materials and techniques developed are suitable for manufacturing adaptations.

Technical Status:

The contractor has completed the mechanical testing of the specimens taken from three previously brazed aluminum honeycomb sandwich panels. Tests have been completed by this division on those test samples received for testing at liquid hydrogen temperatures and the data obtained have been reduced and returned to the contractor for consolidation with room temperature data and incorporation into the final report on this effort.

The brazing alloy development program is still in progress. Specimens from the selected braze alloy group were sectioned, etched, and examined following test brazing. The alloys showing the most promise were in the systems Al-Ge-Si-Ag and Al-Ge-Si-Zn groups. Both of these alloy systems showed good strength and appreciable ductility in bend tests. These alloy systems also have shown the most ease of formability under hot rolling conditions. These brazing alloys are being developed wherein their brazing temperatures will be compatible with the solution heat treatment temperature of the 2000 and 7000 series aluminum alloys.

7. Contract Title: Development of High Strength, Low Density Composite Materials for Saturn Applications
   Contract Number: NAS8-11108
   Contractor: North American Aviation
   Contract Cost: $196,386.00
   Contract Duration: 29 Months
   Negotiated: September 27, 1963
   Responsible Individual: F. P. LaIacona

Purpose of Project:

The purpose of this program is to develop composite structural materials in sandwich and/or honeycomb constructions; the composites to be developed from combinations of materials chosen to offer the best possible application in composite form for use as storage or pressure vessels for propellants such as liquid oxygen, liquid hydrogen, liquid

nitrogen, and hydrocarbon fuels. Phase I consists of a comprehensive survey of the literature and appropriate industrial sources. From this study, the contractor shall select the most suitable metals, non-metals, and combinations of both to be used as core and facing materials. Small scale experiments shall be made to demonstrate the feasiblity of producing the selected composites. In Phase II, a minimum of four of the composites developed under Phase I of this program shall be selected and evaluated as to performance. One panel shall be made for each of the four required applications; these panels shall then be subjected to various tests in the temperature range of 212°F (100°C) to -423°F (-253°C).

Technical Status:

Phase II of this contract is presently in progress. Configuration I Composite, a titanium sheet diffusion bonded to very thin ribs, is being tested in a manner other than the conventional flatwise tension-type specimen, that is, in a "T" type fixture which has given more realistic measure of rib-to-face bond strength. The average failure strength in the "T" type specimen tests (72,700 psi) is 60 percent of the tensile strength (121,500 psi) of the facing tension control specimen. Edgewise compression test results were 102,500 psi at room temperature and 130,000 psi at -200°F (-129°C). The average mechanical property test results for configuration II composite, a duplex structure consisting of titanium face sheets diffusion bonded to titanium honeycomb core which in turn is adhesively bonded to a fiberglass honeycomb core and titanium face sheet structure, were 120,000 psi at room temperature and 148,000 psi at -200°F (-129°C) in edgewise compression. The fabrication and joining of configuration III composite, aluminum faces soldered to aluminum honeycomb core, have produced negative results to date. Restrictions regarding money and time have necessitated the discontinuance of future work with this configuration. The original joining method selected in fabricating configuration IV composite, fiberglass honeycomb sandwiched between aluminum faces which are welded to aluminum corrugations, was determined to be unsatisfactory. Therefore, it has been decided that in lieu of ultrasonic welding, resistance welding should be employed. Preliminary tests indicate that the panel corrugations can be successfully resistance welded to the aluminum faces.

4. Contract Title: Investigation of the Effects of Mechanical Stress on Permeability of Engineering Materials to Liquid Hydrogen and Other Propellants Used in Launch Vehicles

Contract Number: NAS8-11322
Contractor: Melpar, Incorporated, Falls Church, Virginia
Contract Cost: $158,000.00
Contract Duration: 26 Months
Negotiated: June 30, 1964
Responsible Individual: J. G. Austin, Jr.

Purpose of Project:

Among the problems associated with the use of liquid propellants in space vehicles is the permeation of these fluids through engineering materials. Prior permeability studies have been based on specimens in a relatively unstressed condition, where, in reality, the material may be used in a highly stressed state. The purpose of this contract is to determine the effects of mechanical stress on permeability rates of fluids through engineering materials. Fluids considered in this study are: liquid nitrogen (LN_2), liquid hydrogen (LH_2), liquid oxygen (LOX), nitrogen tetroxide (N_2O_4), and monomethyl hydrazine (MMH). The engineering materials range from sheet metals and simple polymer diaphragms to complex composite structures of the honeycomb type.

Technical Status:

The second year of investigations on this contract is directed toward an intensive investigation of the effects of mechanical stress on the permeability of engineering materials that are more directly applicable to vehicle engineering. The materials to be studied are composites of the following types: fiberglass reinforced resins, laminates, honeycomb structures, and adhesive bonds. The fluids used as permeating agents are LH_2, LN_2, LOX, and MMH. During the first year of investigation, considerable effort was expended to determine the effects of MMH on the physical properties of the individual materials prior to testing under applied stress. A limited program of compatibility testing has been initiated to determine the effects of MMH on the structural integrity of the composite materials which are to be studied in the second year.

Cryogenic permeation testing of polymeric materials has continued. Permeation tests of adhesive FM-1000 in the form of an aluminum-to-Mylar bond indicate that the GH_2 permeation rate is affected by stresses in the adhesive. Tensile stresses result in increased permeation in comparison with compressive stresses. Nitrogen permeation of FM-1000 bonding aluminum-to-Mylar was not detectable. Both hydrogen and nitrogen permeation rates are greater than $1X10^{-3}$ and $1.4X10^{-3}$ SPU (standard permeability unit, i.e., $cc/cm^2/cm/torr$ at standard temperature and pressure), respectively.

Hydrogen permeation of epoxy glass board is $1X10^{-10}$ SPU; polyester glass board, $7.6X10^{-9}$ SPU. Nitrogen permeation was not detectable through either polyester or epoxy fiberglass reinforced resin.

Tests on Teflon indicate that the departure of permeation rate from an Arrhenius relationship on cooldown results from diaphragm seal bypass. Fluid bypass is caused by material cracking at the compression seal. Reexamination of previously tested diaphragms, including those diaphragms subjected to LN_2 indicates similar materials failure.

Further permeation testing of stressed polymeric materials was attempted. However, diaphragm material failure occurred at the seal by compressive shear during startup.

Tests conducted on the LH_2 two-stage dewar system indicate a LN_2 loss rate of three liters per hour from the inner chamber. Further tests have revealed that the dewar vacuum walls are not leak tight.

The overall program seems to be progressing on schedule. The present status indicates that the objective stated in the original scope of work and amendments will be completed by the contract termination date, August 1966. Particular emphasis is being placed on early completion of the experimental program to allow for an analytical analysis of the stress-permeation data.

5. Contract Title: Development of a System for Prestressing Brittle Materials
Contract Number: NAS7-429
Contractor: Douglas Aircraft Company, Incorporated
Contract Cost: $67,700
Contract Duration: 12 Months
Negotiated: December 7, 1965
Responsible Individual: V. F. Seitzinger

Purpose of Project:

The objective of this project is the development of thermal-shock and impact resistant refractory composite materials of higher tensile load carrying ability than that of conventional brittle refractories. The program is designed to circumvent the inherent low tensile strength of ceramics by taking advantage of their high compressive strength, thereby eliminating or minimizing failure from thermal and mechanical shock. The concept consists of internally prestressing, in compression, a ceramic material reinforced with continuous metal filaments while prestressing at a low temperature. The ceramic will be chemically bonded at 204 to 315°C (400-600°F). The feasibility of the concept and the validity of the theoretical approach for prestressing reinforced chemically bonded ceramics will be determined and an appraisal will be made of the properties of the resulting composite materials.

Technical Status:

The literature survey of properties of reinforcing and matrix materials has been completed. The reinforcing materials for which property data were accumulated include columbium, molybdenum, rhenium, tantalum, and tungsten. Matrix materials were limited to those with melting points above 1927°C (3500°F) which can be potentially consolidated by chemically bonding at low temperatures. Therefore, the literature survey was restricted to alumina (Al_2O_3), thoria (ThO_2),

hafnia (HfO_2), hafnium diboride (HfB_2), hafnium nitride (HfN), hafnium carbide (HfC), zirconium diboride (ZrB_2), zirconium carbide (ZrC), and zirconia (ZrO_2).

Methods have been developed for estimating strength and behavior of prestressed and reinforced ceramics. These analysis techniques have been used to study the effects of various parameters on the ceramic prestress and on the strengths of ceramic composites. "Trade-off" studies have been made of the room temperature ultimate tensile strength-to-density characteristics of various combinations of ceramic and reinforcing materials.

Future efforts will include further optimization studies on the strength properties of these composites at elevated temperatures. Upon completion of the optimization studies, one reinforcement and one matrix material in addition to ZrO_2 will be chosen for the laboratory investigation. The initial phase of the laboratory investigation will consist of the development of a chemical bonding system for the additional matrix, and the determination of the mechanical properties of both matrix systems at room and elevated temperatures.

C. Lubricants and Lubricity

1. Contract Title: Research on Bearing Lubricants for Use in High Vacuum
Contract Number: NAS8-1540
Contractor: Midwest Research Institute
Contract Cost: $391,271.00
Contract Duration: 51 Months
Negotiated: February 23, 1961
Responsible Individual: K. E. Demorest

Purpose of Project:

The purpose of this contract is to develop dry film lubricants having potential applicability on bearings and sliding surfaces in high vacuum. Lubricating films of primary interest are those of ceramics, metals, and cermets. A number of potential lubricants have been selected and are undergoing evaluations. These evaluations are based on film formulation, screening, and wear-life investigations, and frictional behavior investigations in ultrahigh vacuum. Those materials offering the most promise are applied to bearing surfaces, evaluated, and forwarded to this Center for final testing.

Technical Status:

The contractor has completed a previously described test program on the wear-life and friction of various lubricants at light loads and high speeds. The results of the tests, made at normal load of 13.5 psi and a velocity of 765 feet per minute, are summarized in the tabulation below. Four recently introduced, commercially available, dry film lubricants are included along with the developed series of MLF lubricants.

Comparison of Dry Film Lubricants for Space Vehicle Applications

Lubricant	Coefficient of Friction		Wear-Life (hrs) to Probability of Failure	
	70°F (21°C)	400°F (204°C)	10%	95%
MLF-5	0.10	0.15	18 hrs	110 hrs
MLF-6	0.13	0.16	30	115
MLF-7	0.14	0.14	22	70
MLF-8	0.10	0.11	22	120
MLF-9	0,12	0.15	24	200
COMMERCIAL A	0.10	0.18	18	80
COMMERCIAL B	0.10	0.24	23	200
COMMERCIAL C	0.08	0.14	16	150
COMMERCIAL D	0.11	0.17	6	50

In addition, wear-life investigations are being made on new film formulations of molybdenum disulfide and tungsten diselenide using aluminum phosphate as the binding agent. Evaluations of the effect of mating surface roughness and hardness on the friction and wear-life of MLF-5 are being continued. To date, the longest wear-life for MLF-5 has been obtained when the surfaces were maintained at 5 micro-inches, RMS finish.

Several tests have been made on Bar-Temp* lubricated R-4 bearings in a glass ultrahigh vacuum apparatus following the "baseline" test described previously. The environmental pressure reached $2X10^{-10}$ torr before the start of the test but, later, rose to $1.5X10^{-8}$ torr. A partial pressure analysis of the outgassing products of the bearing showed that large amounts of hydrogen, carbon monoxide, and carbon dioxide were being produced by the rotating bearings. The large outgassing load appeared to be indicative of high wear rates; however, the bearing was operating satisfactorily. In the future, additional pumping will be supplied to reduce pressure to the initial value during the test.

Future plans call for the formulation and evaluation of additional solid lubricant films using various lubricant and binder materials. The friction and wear characteristics of these developed lubricants will be determined under the test conditions established during the past year. These solid films will be applied to gears and instrument bearings and their friction and wear characteristics will be investigated.

Additional plans include the completion of a fluid lubricant handbook. Data contained in the handbook will be obtained through standard friction and wear tests and through a literature survey.

H. Corrosion in Aluminum and Steel

1. Contract Title: Investigation of the Stress Corrosion Cracking of High Strength Aluminum Alloys
   Contract Number: NAS8-5340
   Contractor: Aluminum Company of America
   Contract Cost: $160,236
   Contract Duration: 38 Months
   Negotiated: May 6, 1963
   Responsible Individual: J. G. Williamson

   Purpose of Project:

The purpose of this work is to investigate the stress corrosion susceptibility of the various high strength aluminum alloys in the 2000 and 7000 series which are presently being used or considered for use in the Saturn program. Special emphasis is being placed on determining the value of protective coatings and surface treatments as a means of reducing or eliminating stress corrosion cracking of these alloys. Some

*Tradename of Barden Bearing Company solid lubricant bearing cage.

work is being conducted in an effort to develop an understanding of the basic mechanism of stress corrosion cracking and to devise and evaluate a rapid screening method to distinguish between the more stress corrosion resistant tempers of some of the high strength aluminum alloys. Studies are also being conducted to determine the stress corrosion susceptibility of welded joints of some of the high strength weldable aluminum alloys with some consideration being given to the type of stressing jig that should be used for studying such welded joints.

Technical Status:

Stress corrosion studies on unprotected alloys using accelerated test methods have been completed. These tests indicate that aluminum alloys 7075-T73, 2219-T87, 2219-T851, 2219-T62, and 2024-T851 are essentially free of stress corrosion cracking when stressed up to 75 percent of the yield strength. Several unprotected alloys, 7075-T7351, 2219-T87, 2014-T651, X7006-T651, 7079-T651, and 7178-T651, exposed to atmospheric conditions at Point Comfort, Texas, Point Judith, Rhode Island, and New Kensington, Pennsylvania, remain in test. These tests, to date, indicate that alloys 2219-T87 and 7075-T7351 are highly resistant to stress corrosion cracking in all three environments. The New Kensington industrial atmosphere is as severe as the seacoast environment in causing stress corrosion cracking of the X7006-T651, 7079-T651, and 7178-T651, but less severe on 2014-T651.

Studies are continuing on the effect of various coatings in reducing the susceptibility of high strength aluminum alloys to stress corrosion cracking. There have been no changes from the previous reports which indicate that the best protection is being afforded by the combination of either shot peening or metallized 7072 alloy plus an epoxy-polyamide topcoat. Anodic coating has not proven to be effective in preventing stress corrosion cracking. This type of coating, however, provided a marked degree of protection to 2014-T651 alloy in the salt water alternate immersion tests but only slight protection in the three atmospheres. It is believed this may be the result of a greater amount of crazing in the atmospheric specimens which encounter larger fluctuations in temperature. Additional studies will be made in an effort to resolve this difference between the two environments.

Studies on the stress corrosion susceptibility of welds on high strength aluminum alloys are being continued. Various welded joints of four new high strength weldable Al-Zn-Mg alloys (X7002, X7106, 7039, and X7139) have been in test up to ten months. Although the parent metal specimens of all four of these alloys exhibited high resistance to general type corrosion, the as-welded specimens suffered significant losses in tensile strength ranging from 15 to 19 percent after three months and up to 38 percent after six months in the 3.5 percent sodium chloride alternate immersion tester. The post-weld aging results in an improvement of the corrosion resistance of welded specimens, however, the resistance to stress corrosion cracking is adversely affected by the post-weld aging treatment employed. Recent results indicate that removal of the weld bead may reduce the susceptibility of stress corrosion cracking of welded specimens. The

weld bead of 1/8-inch thick X7106-T6 post-weld aged specimens was removed by machining and exposed in the alternate immersion tester. There has been no evidence of failure after approximately 28 days in test. Similar post-weld aged specimens with the bead intact failed after two to five days. It is believed that the removal of the bead may diminish the stress concentration resulting from the notch effect of the weld bead and, therefore, might significantly extend the specimen life.

2. Contract Title: Development of Improved Conversion Coatings for Aluminum Alloys
Contract Number: NAS8-11226
Contractor: Aluminum Company of America
Contract Cost: $53,714
Contract Duration: 2 Years
Negotiated: May 1, 1964
Responsible Individual: R. H. Higgins

Purpose of Project:

The ultimate goal of this project is to develop a chemical conversion coating for application to 2219 aluminum and other high strength aluminum alloys which will withstand at least 168 hours salt spray exposure without any visible signs of corrosion or coating deterioration. The coating shall be free from organic or highly oxidizable material and be applied by non-electrolytic means similar to coatings conforming to military specification MIL-C-5541. Metallurgical and chemical studies will be made of both the alloys in question and the mechanism of protection afforded by the coating.

Technical Status:

The work on this contract has included both chemical and metallurgical examinations of 2219 aluminum alloy surfaces and several chemical conversion coatings. The main emphasis has been directed toward chromate type conversion coatings with Iridite 14-2 (manufactured by Allied Research Products) serving as the control coating process. Several recommendations have been offered for improving the amount of protection afforded by this process which included modifications to the control coating and specific surface preparatory treatments. Considerable effort has been expended toward the attainment of an effective sealing treatment for the chromate coating. A silicate-chromate sealing treatment was found to improve the salt spray resistance of the coating; however, this treatment was found to be detrimental to zinc chromate primer adhesion. Coated panels also were cathodically "sealed" at various current densities in a 5 percent chromic acid solution. Subsequent salt spray exposure showed no real advantage for this treatment. Since initial evaluation of a dichromate sealing solution had shown some slight advantage, the potentialities of this material are being studied using several possible variations in bath make-up.

Investigations involving Iridite bath modifications continue. Coatings formed in 6-10 oz/gal. solutions have shown considerable improvement in the amount of protection afforded; however, the results have not been consistent with different lots of material. Concentrated Iridite baths were found to function by a collective increase of all three major components of the proprietary material. Coating times for a 10 oz/gal. solution in excess of three minutes did not prove advantageous. The variable response of different lots of 2219-T87 alloy to the Iridite chromate conversion coating treatment, with respect to the salt spray resistance, is being investigated. One aspect that might contribute to such variability is the degree of artificial aging that the alloy has received. Results, thus far, indicate that over aging will render the surface more susceptible to surface corrosion; however, the degree of aging also affects mechanical properties and resistance to stress corrosion. These studies are to be continued.

Visual examination of panels exposed for four months at Point Judith, Rhode Island and eight months at New Kensington, Pennsylvania show evidence of corrosion and pitting attack similar to that of panels exposed for short periods in the five percent salt spray. Some variation was also evident between lots of the -T87 temper in test.

I. Explosion Hazards and Sensitivity of Fuels

1. Contract Title: Investigation of the Reactivity of Launch Vehicle Materials with Liquid Oxygen
Contract Number: NAS8-20220
Contractor: Stanford Research Institute
Contract Cost: $79,137.00
Contract Duration: 15 Months
Negotiated: June 23, 1965
Responsible Individual: W. A Riehl

Purpose of Project:

The primary objective of this program is the development of an understanding of the basic physical and chemical factors which contribute to the reactivity of selected materials with liquid oxygen.

Consideration of modes of experimental investigation by the contractor shall include but not necessarily be limited to the following approaches:

The first approach to the problem shall consist of studying the course of free radical reactions between liquid or gaseous oxygen and selected materials at low temperatures; whereas, gas phase oxidation of materials at elevated temperatures apparently takes place via predominantly free radical mechanisms. The nature of the reaction processes involved

in the liquid phase or low temperature gas phase oxidation of materials is largely unknown. Therefore, an analytical and experimental investigation of the mechanism of low temperature oxidation of selected simple hydrocarbons, alcohols, and other classes of compounds shall be carried out to study the identity and stability of the oxygenated reaction intermediates. At least one metal, titanium, shall be included in the study.

As a second approach, oxygenated reaction intermediates shall be prepared indirectly by various methods, possibly including decomposition of unstable compounds (such as azo derivatives) in the presence of low temperature gaseous oxygen. The sensitivity to impact of these oxidation products will be tested in the absence of gaseous or liquid oxygen as one method for determining the importance of oxygenated intermediates in the initiation process.

As a third approach to the problem, impact tests on a few pure compounds selected on the basis of their chemical structure shall be performed to study the dependence of reactivity on chemical structure. This information also shall be correlated with the results of the initial studies.

Finally, as a fourth approach, tests shall be made to study the effects of degree of subdivision on reactivity. This will consist of selecting materials which can be prepared in different states of subdivisions and testing in these various states; in some instances, the size of particles should be decreased just prior to impacting and before oxidation of the freshly exposed surfaces can take place.

Technical Status:

Photolysis of azoisobutane (AIB) at -95°C (-139°F) in several solvents gives an unstable intermediate believed to be di-t-butyl tetraazadiene. In the presence of oxygen, photolysis of AIB leads to oxygen uptake and evolution such as to suggest the presence of an intermediate polyoxide, either di-t-butyl tetroxide or trioxide. The formation of tetraazadiene is unaffected by the presence of oxygen.

Solutions of 0.01 M AIB in pentane were irradiated in the presence of oxygen for 60-68 minutes at -95°C (-139°F), the solutions were then cooled to the freezing point of pentane (-121°C (-186°F)), and the nitrogen evolved from the photolysis and the residual oxygen were removed from the reaction vessel and measured. Rates of decomposition of the intermediates were then measured at specified temperatures ranging from -106°C to -32°C (-159°F to -26°F) by quickly warming a solution from -121°C (-186°F). Evolved oxygen and nitrogen were then measured, and the solution was again warmed and another measurement was taken.

Rate constants for these reactions were calculated in the conventional manner by plotting the log of the nitrogen or oxygen evolution against time. Each of the nitrogen evolution runs gave three points which described a straight line. Oxygen evolution was not linear on this plot and a crudely fitted straight line was used to estimate the rate constant.

The data show that tetraazadiene is a fairly stable species with a half-life at 32°C (-26°F) of about 16 minutes. The Arrhenius parameters for this decomposition were obtained from a plot of log k_1 versus 1/T over the range -52°C to -32°C (-61°F to -26°F). An A-factor of $5X10^{18}$ sec^{-1} and an activation energy of 19 kcal/mol appear reasonable for a unimolecular decomposition of a molecule such as tetraazadiene. Using these parameters, one can calculate that at 25°C (77°F), the half-life would be approximately 0.2 seconds.

Experimental data indicate that oxygen evolution is clearly much faster than nitrogen evolution and only at temperatures below -90°C (-130°F) was it possible to obtain even crude measurements of the rate. This fact in itself is not surprising inasmuch as both the trioxide and the tetroxide were expected to be rather unstable much above this temperature. However, the disturbing aspect of these data is that oxygen uptake and slow evolution have been noted even in experiments where oxygen was admitted after irradiation ceased and free radicals would no longer be expected to be present. Control experiments have established that no oxygen uptake occurs if the irradiated solution is first warmed to 25°C (77°F), then re-cooled to -95°C or -121°C (-139°F or -186°F), and oxygen then admitted. Either the life-time of the free radicals in this system are much longer than was suspected or oxygen is able to complex or react with some metastable species generated by photolysis. The tetraazadiene is of course such a species.

Low temperature electron paramagnetic resonance (EPR) and ultraviolet (UV) spectral studies of the irradiated solutions may help elucidate the nature of the unstable oxygen species being generated in these experiments. Such experiments will be started shortly. Product analyses by nuclear magnetic resonance (NMR) appear to be feasible and will be pursued.

As part of the study of reactivity of launch vehicle materials, the contractor is investigating the conditions and mechanisms required to ignite fuels under impact in liquid oxygen (LOX). It is suspected that free radicals may have an important role in the ignition process; therefore, these studies include an examination of impacted or ground materials with emphasis upon the detection of free radicals by electron paramagnetic resonance spectroscopy (EPS).

It has been shown that mechanical action, e.g. low temperature ball milling or grinding in vacuo degrades macromolecules, probably by rupture of side chains as well as of the main skeleton. Radical concentrations as high as 10^{21} per gram have been found using EPR and the maximum efficiency of this tribochemical reaction may approach 10 percent.

Radical stability varies considerably. Polycaprolactam, for example, produced extremely active fragments: concentrations of 10^{15} per gram, produced at -196°C (-321°F), disappeared after 1-2 minutes. Polystyrene fragments remained unchanged for six days at the same temperature.

Both temperature and atmosphere are important parameters. Gelatin yields radicals which are reasonably stable at -196°C (-321°F). However, these so-called "primary" fragments change to different "secondary" units, also radicals, as the temperature rises above about -126°C (-195°F). In the presence of oxygen, polystyrene radicals convert in a few minutes to peroxide radicals, R-O-O·, which are stable up to 0°C (32°F).

The possible importance of these processes to impact ignition is emphasized by noting that the radicals produced are located at surface layers where they are exposed to rapid, diffusion-independent, chemical reactions. For example, for polymethylmethacrylate fragments, the average surface area per radical is estimated to be 30Å.

To determine if explosion hazards exist or whether ignition takes place between oxygen and degraded polystyrene powders containing exposed free radicals, experiments with ground polystyrene and oxygen at -183°C (-297°F) were conducted. Because of the potential hazards, special methods were employed.

All of the experiments used polystyrene prepared by high speed sawing under liquid nitrogen.

a. Liquid nitrogen was removed by warming the nitrogen-powder mixture in a LOX bath. The nitrogen was evacuated and the pressure was reduced to 10^{-4} torr over the powder. Gaseous oxygen was fed rapidly into the LOX-cooled trap containing the degassed powder. No reaction was observed.

b. The powder was produced under LOX. The grinding was accompanied by ignition at the saw-polymer interface and of particles settling through the LOX. Ignition did not propagate and infrared product analysis was unproductive. No new materials were detected; of course CO_2 and HCHO and other volatile combustion products would not have been detected.

c. Addition, at -183°C (-297°F), of LOX to a mixture of liquid CH_4 and polystyrene powder produced no reaction.

Equipment is being designed and assembled with which to study these materials in an EPR spectrometer. After the techniques for transferring the polymer fragments at -183°C (-297°F) is perfected, the free radical content of these polymers will be determined and a study will be made of their half-lives in the presence of low pressure gaseous oxygen at -183°C (-297°F).

2. Contract Title: Investigation of Reactivity of Titanium and Other Materials with Liquid Oxygen and Nitrogen Tetroxide
Contract Number: NAS8-20078
Contractor: Air Reduction Company, Murray Hill, New Jersey
Contract Cost: $99,919.00
Contract Duration: 14 Months
Negotiated: May 22, 1965
Responsible Individual: C. F. Key

Purpose of Project:

The upper stages of Saturn V launch vehicles are designed to use liquid oxygen as the oxidizer in the main propulsion system and nitrogen tetroxide in the auxiliary propulsion system. The contractor is directed to develop and qualify an adiabatic compression tester suitable for investigating the sensitivity of materials in contact with liquid oxygen. Knowledge derived from this study should permit improved estimates of the likelihood that undesirable reactions will occur under any given set of conditions and may suggest methods for minimizing the reactivity of materials with oxidizers in space vehicles. The development of this adiabatic compression apparatus and correlation of the results with those resulting from evaluation by means of the ABMA Impact Tester may tend to elucidate the kinetics and mechanisms of reaction of liquid oxygen and nitrogen tetroxide with engineering materials. The ABMA Impact Tester is used at the contracting agency to qualify materials for use with liquid oxygen. The tester and test procedure are described in MSFC-SPEC-106A.

In addition, the estimates of reaction probability derived from this study will be of assistance in such problems as evaluating the use of titanium propellant tanks for auxiliary propulsion systems. Use of such tanks would result in substantial weight savings.

Technical Status:

Design, fabrication, and assembly of the required adiabatic compression tester has been completed and initial experimental qualification testing was initiated. During these tests a deficiency was noted in the seal at the end of the compression piston, and major emphasis was directed toward location of a new seal material or design to overcome this problem. A new "Omniseal "O"-ring has been tested which held an

initial pressure of 23,800 psi at liquid oxygen temperature but developed a leak on the second stroke of the piston. The seal was checked at 30,600 psi and leakage was observed. The possibility exists that the seal at the burst diaphragm is leaking and the contractor is fabricating a "blind" chamber to check this possibility.

In addition to the seal problem, damage occurred to the piston-cylinder when titanium was tested in gaseous oxygen at room temperature. The contractor is working on ways to alleviate this problem.

A new piston cavity design is being fabricated. This design will use a static "O"-ring seal rather than dynamic seal presently used.

3. Contract Title: Cryogenic Propellant Fluid Properties Data Evaluation
Government Order Number: H-76797
Contractor: National Bureau of Standards, Boulder, Colorado
Contract Duration: 12 Months
Negotiated: January 25, 1965
Responsible Individual: Harold Perkins

Purpose of Project:

Properties of the various cryogenic propellant fluids are widely reported in the literature. The problem of theoretically evaluating these data for consistency and accuracy is beyond the capabilities or area of purview of most workers requiring standardized properties data for the fluids nitrogen, oxygen, argon, helium, hydrogen, fluorine, and their binary mixtures. The purpose of this program is to critically review the existing literature and evaluate these data for theoretical consistency and accuracy of the various fundamental and calculated properties of the fluids. The pressures and temperatures studied will cover the range of interest to launch vehicle designers.

Technical Status:

This government order expired on January 27, 1966, however, the work that has been expended will be resumed in early 1966 under a new NASA government order to be funded through NASA Headquarters.

a. Thermodynamic Properties of Normal and Para Hydrogen

During the current reporting period, work has been underway on the programming of the new equation of state for p-H_2 for the new p-H_2 data. The programming of the equation at this time was to assist in the development of the equation by providing programming that would allow for constraining the equation to the critical point data.

b. Thermodynamic Properties of Oxygen

During the current reporting period, new data for the density of oxygen became available from experimental measurements. These data include over 1,000 data points for oxygen, liquid, and vapor. The new liquid data are in addition to liquid values previously available from this experimental program. The new vapor measurements are for the temperature range 148° to 300°K with pressures from 30 to 300 atmospheres. The only previous measurements that have been made in this range for the vapor are values by Kamerlingh Onnes and Kuypers (Communs. Phys. Lab. Univ. Leiden No. 169a, 1924). These older data were for temperatures from 156° to 233°K with pressures from 20 to 61 atmospheres. These new data, therefore, extend the available experimental data to significantly higher pressures as well as providing a second source of values which can be expected to be generally more accurate than the older data.

Comparisons have been made between these new data and the older data available in the literature. These comparisons show the agreement for the vapor data of Kamerlingh Onnes and Kuypers and these new data to be within the precision of the older measurements. The ambient temperature data by Michels, Schamp, and de Graaf (Phisica 20, 1209-14, 1954) are in agreement with the new data to within 0.1 percent. The liquid data by van Itterbeek and Verbeke (Cryogenics 1, No. 2, 77-80, 1960) are in agreement with the new liquid data to 0.1 percent, which is the approximate precision of both sets of data. The higher temperature liquid data by Timrot and Borisoglebskii (Inzh, Fiz. Zh, Akad, Nauk Belorussk, SSR 4, No. 1, 3-13, 1961) are not in satisfactory agreement with the new measurement; each isotherm appears to be different from the new measurements by a constant amount. A correction was, therefore, applied to Timrot's and Borisoglebskii's data on the basis of new saturation liquid densities determined by the new measurements. The result of this correction is that Timrot's and Borisoglebskii's data are then made concordant with the new measurements.

The new experimental data, together with selected data from the literature, have now been fitted to an equation of state which represents the experimental data for both the liquid and vapor phases over the temperature range from the triple point to ambient temperatures and with pressures to 350 atmospheres. This equation of state represents all of these data to within the precision of these data with the exception of isotherms in the immediate vicinity of the critical point. Additional tests on this equation of state are now in progress, such as a comparison of vapor pressures determined from the equation of state with vapor pressure data, calculated values of the second virial equation with theoretical values, and the characteristics of the Joule-Thomson inversion curve and calculated values of specific heats.

This equation of state is used in the calculation of a new set of thermodynamic property tables for oxygen and for the construction of temperature-entropy diagrams.

c. Viscosity and Thermal Conductivity of the Cryogenic Fluids

Two manuscripts have been prepared on this task, (1) "The Viscosity and Thermal Conductivity of Dilute Argon Between 90 and 2000°K" by J. M. Hanley, NBS Technical Note (in editorial review), and (2) "Comparison of the Lennard-Jones, Exp. 6, and Kihara Potential Functions Using Viscosity Data of Dilute Argon" by H. J. M. Hanley (submitted for publication).

The literature search for transport properties of oxygen and nitrogen has been completed and the data have been extracted from the pertinent publications and compiled in work notebooks. The viscosity data for nitrogen has now been correlated and optimum parameters selected for the Kihara potential function from these data. Specific heats for nitrogen are now being investigated as a supplementary aid in the correlation of the thermal conductivity data for nitrogen. The oxygen data for viscosity are also being correlated.

A general method was developed for procedures to determine the best intermolecular potential functions as a means of determining the optimum correlation of a given set or sets of data for transport and thermodynamic data. This general method has been developed from the studies that have been made in this task. Literature searches are now underway for transport property data for additional fluids which will be compiled in an extension of this task.

Reference data were evaluated and reported. An updated list of publications is as follows:

a. Olien, N. A. and Hall, L. A., A Bibliography of Experimental Saturation Properties of Cryogenic Fluids, Natl. Bur. Standards Tech. Note No. 309 (Mar 1965).

b. Hust, J. G. and Stewart, R. B., A Vapor Pressure Equation for Oxygen, Natl. Bur. Standards Rept. No. 8753 (Feb 1965).

c. Hust, J. G. and Stewart, R. B., A Compilation of the Property Differences of Ortho and Para Hydrogen or Mixtures of Ortho and Para Hydrogen, Natl. Bur. Standards Rept. No. 8812 (May 1965).

d. Cryogenic Data Center, A Bibliography of References for the Thermophysical Properties of Helium-4, Hydrogen, Deuterium, Hydrogen Deuteride, Neon, Argon, Nitrogen, Oxygen, Carbon Dioxide, Methane, Ethane, Drypton, and Refrigerants 13, 14, and 23, Natl. Bur. Standards Rept. No. 8808 (May 1965).

e. McCarty, R. D., Thermodynamic Properties of Liquid-Vapor Parahydrogen and Liquid-Vapor Oxygen, Natl. Bur. Standards Rept. No. 8883 (Sept. 1965).

J. Synergistic Effects of Nuclear Radiation, Vacuum, and Temperature on Materials

1. Contract Title: Investigation of the Combined Effects of Nuclear Radiation, Cryogenic Temperatures, and Vacuum on Engineering Materials
Contract Number: NAS8-2450
Contractor: General Dynamics Corporation, Fort Worth, Texas
Contract Cost: $1,134,266.00
Contract Duration: 53 Months
Negotiated: November 9, 1961
Responsible Individual: E. C. McKannan

Purpose of Project:

This program is directed toward the development of experimental data on the combined effects of nuclear radiation, vacuum, and cryogenic temperatures on engineering materials. Materials of major interest to this project are primarily organic such as lubricants, plastics, elastomers, and surface coatings. Tests are to be made during exposure to (a) radiation from a nuclear reactor, (b) reduced pressures of 10^{-6} torr, and (c) cryogenic temperatures to determine progressive changes in the physical and mechanical properties of selected materials of potential use in nuclear-propelled vehicles. Tests are to determine weight loss, gas evolution, and physical properties as appropriate to the material and its proposed use.

Technical Status:

As stated previously, the final report covering the second year of materials testing and data evaluation has been published. Mechanical and electrical property measurements have been made on 83 aerospace engineering materials. The current work has been planned to fill in missing data points, to check anomalies, and to complete the characterization of materials evaluated previously. This work requires three weeks of irradiation time with vacuum chambers, liquid nitrogen, and liquid hydrogen dewars. Previously, lists of materials which have been evaluated and the environments for which data are available in the annual reports were given.

The first irradiation exposure of the third year program was made involving static tests in vacuum and in air on specimens at doses to $3X10^{10}$ ergs-gm (c). Static testing means that property measurements were made before and after exposure to radiation but not during the exposure. Control tests on ball bearings running at high speed in motors in vacuum indicated that fluorosilicone oils in ribbon retainers were superior to bearings with plastic retainers.

The second irradiation involved the dynamic testing (in-situ) of plastic films subjected to a dose of $5X10^8$ ergs-gm^{-1} (c). Also, static specimens were exposed to $5X10^8$ and to $1X10^9$ ergs-gm^{-1} (c). The dielectric and cryo-mechanical test systems were irradiated to $1X10^{10}$ ergs-gm^{-1} (c) at LN_2 temperatures. Both of these systems were operated at nearly constant temperatures due to the addition of heat shields where before they were affected by gamma heating.

The third and final irradiation involved the dielectric test system operating at -143°C and 10^{-6} torr with four commonly used dielectric materials.

The Bearing Tester was irradiated in air to a dose of $1X10^{10}$ ergs-gm (c); the run was continued on a postirradiation basis for a total bearing operating time of 500 hours. The motors, containing the following listed lubricants, were operating satisfactorily at the end of the test: Polymer SP-2, OS-124, DC-705, and Kynar (PVF).

Data were obtained from the air irradiation of the thermal conductivity units at nominal dose levels of $5X10^9$, $1X10^{10}$ and $3X10^{10}$ ergs-gm (c). The changes in the thermal conductivity for the foam materials tested at these doses ranged from 2 to 5 percent.

The liquid hydrogen irradiation tests were made and involved irradiation of both the Cryotensile Tester and the Thermal Conductivity Tester. The first test was terminated after a nominal dose of $2X10^{10}$ ergs-gm (c) because of a suspected hydrogen leak; however, tensile testing of the samples was completed before termination of the LH_2 flow. The second irradiation test was completed without incident to the desired dose level of $3X10^{10}$ ergs-gm (c).

The data are being compiled for the final report and the contract will be completed.

2. Contract Title: Development and Validation of a Method for Predicting Neutron Induced Activation in Materials
   Contract Number: NAS8-11160
   Contractor: IIT Research Institute
   Contract Cost: $101,144.00
   Contract Duration: 17 Months
   Negotiated: May 14, 1964
   Responsible Individual: L. K. Zoller

   Purpose of Project:

To support in-house and contractual studies of the effects of high energy nuclear radiation on engineering materials and to evaluate the degradation of materials on spacecraft which carry nuclear power sources or which are exposed to indigenous space radiation, it is necessary

to determine the extent and characteristics of induced radioactivity from nuclear particle (neutrons, protons, electrons, etc.) reactions. In theory, the calculation of induced radioactivity is direct; a lack of fundamental reaction data, however, restricts the calculations to the few elements or isotopes where the reaction probabilities are well defined as a function of the kinetic energy of the bombarding particle and where the isotopic decay mechanisms are well known. To circumvent the lack of fundamental reaction data, this project entails the derivation of theoretical and empirical formula for predicting requisite nuclear reaction data and the assembly of these formulas into a digital computer source program. Furthermore, since the computer program employs interpolation and extrapolation techniques, all aspects of the computer program are to be validated with carefully controlled experimentation. When completed and validated, the computer program will permit calculation of nuclear particle induced radioactivity.

Technical Status:

The computer program has been completed and validated. This program will readily permit the calculation of nuclear particle induced radioactivity as a function of many geometric and operational variables, for all common nuclear particle reactions with all stable isotopes of all elements. The program input is currently complete for neutron induced reactions, (n,γ), (n,α), (n,p), and $(n,2n)$. The computer program will be valuable to the industry at large for such diverse functions as activation analysis (analytical chemistry), experiment planning, and facility siting and operation. The final report of this contract encompasses the nuclear theory upon which the program is based, the experimental validation of the theoretical and empirical analyses, the detailed operating procedures for the computer program, and the listing of the program source deck and "permanent" data input decks.

3. Contract Title: Investigation of the Combined Effects of Space Environmental Parameters on Space Vehicle Materials
   Contract Number: NAS8-20210
   Contractor: Hughes Aircraft Company
   Contract Cost: $96,564.00
   Contract Duration: 12 Months
   Negotiated: June 30, 1965
   Responsible Individual: E. C. McKannan

   Purpose of Project:

   The requirement for operating space vehicles for long periods of time in high altitude terrestrial orbits and on deep space probes necessitates the determination of the detrimental effects of the space indigenous radiation in addition to temperature extremes and ultra-low pressures on space vehicle materials which are employed for their thermal insulating, dielectric, or optical (thermally reflective) properties.

The objective of this program is to obtain pertinent, valid, experimental data for application to engineering design of space vehicles.

Technical Status:

All of the experimental equipment for the program has been designed. The standardized and the available specialized equipment has been assembled and calibrated. The heart of the experiment, a "Lazy-Susan" specimen turntable mounted in a vacuum chamber, has been manufactured. In this vacuum system, a specimen can be exposed to the electron beam and ultraviolet beam while being heated or cooled, and then it can be rotated to another position to be measured. In this manner, twenty-five specimens can be alternately exposed and measured without opening the vacuum system (in-situ). The three types of specimens and measurements are listed in the following tabulation.

SPECIMENS FOR MEASUREMENT OF COMBINED ENVIRONMENTAL EFFECTS

Specimens	Measurement	Property
L. Thermal insulation, multilayer film, and foam	1. Flash-diffusivity	1. Thermal Conductivity
2. Dielectric films	2a. D.C. resistance 2b. Complex-permitivity at x-band	2a. Resistivity 2b. Dielectric constant and dissipation factor
3. Thermal control coatings	3. Diffuse reflectivity	3. Solar absorptance

The thermal diffusivity method depends upon measuring the temperature rise of the back surface of a sample due to the passage of the thermal pulse. If the pulse input time is short compared to the time for passage through the sample, the specimen may be treated mathematically as an infinite slab. A large advantage of the technique is that the heat input need not be known for thermal diffusivity measurements. The technique may be adapted to vacuum systems with minimum effort. The transmission of the energy may be carried out through a suitable window in the chamber wall.

The major problem with this technique is in the amplifier. Many different amplifiers have been used but the combination of a Tektronix RM122 preamplifier and a Tektronix 53/54E low-level differential amplifier gives the best results. Shielding of all wires is most important to remove specimen signal from the flash lamp discharge. The thermal pulse

is generated by a G. E. flash bulb operated at 1000 volts and 85 mfd capacitance. This particular lamp acts as a point source. The sample is encased in a copper housing to eliminate pickup from the flash tube discharge.

Instrumentation for the DC conductivity measurements has been completed. The configuration is in conformity with ASTM specification D 257-61, and the method provides resistivity measurements up to $1X10^{20}$ ohm-cm.

A 2-1/2 inch ID integrating sphere reflectometer has been designed to measure solar absorptance (by integration of spectral reflectance). Monochromatic light in the 0.29 to 2.6 micron range will enter the integrating sphere via a sapphire window in the top chamber cover plate, will strike the specimen surface along the lower edge of the integrating sphere, and after successive internal multireflections, will be picked up on a thermopile sensor. The integrating sphere will be raised and lowered by a pneumatically activated bellows operating on two supporting dowel guide pins. Readout of signals depends upon proper conversion, sufficient amplification of the signal, and differentiation between signal and noise, which is important because the light sources and detectors are inherently noisy and because signal strength in certain regions of the spectrum (notably in the UV) is quite low. The light beam is chopped (13 cps), and a tuned preamplifier-amplifier and tuned band pass filter are employed. The system is read with an integrating digital voltmeter which provides 10^{12} amplification with a noise level reduced to 10^{-11} volts. The source is a 150 watt xenon lamp.

The various elements for making measurements will be assembled into the vacuum system, and preliminary calibration measurements will be made. Following this the test program will be initiated starting with vacuum - UV exposure and continuing with the addition of exposure to 0.9 Mev electrons.

K. Instrument Development

1. Contract Title: Development of Techniques for Measuring Thermal Diffusivity
   Contract Number: NAS8-11891
   Contractor: Battelle Memorial Institute, Columbus, Ohio
   Contract Cost: $49,925.00
   Contract Duration: 12 Months
   Negotiated: June 30, 1965
   Responsible Individual: J. G. Austin, Jr.

   Purpose of Project:

The objective of this program is to develop the apparatus and technique required to measure the thermal diffusivity of materials including metals, plastics, reinforced plastics, ceramics, and composites ranging in

thermal diffusivity from 0.001 to 2 cm^2/sec over a temperature range from 200°C to 2000°C (392°F to 3632°F). The technique will use a pulsed laser as a transient thermal energy source.

Technical Status:

Progress during this report period has included the selection of all components and ordering of all long-lead-time items, and the completion of shop drawings for the final design.

A Consolidated Vacuum Corporation high-vacuum pumping station, less mechanical pump, has been received. This station includes a 4-inch diffusion pump enclosed in a sturdy cabinet. The furnace will be mounted directly on the pump flange with both laser head and detector system mounted in-line on the top of the station. This arrangement will maintain alignment of components and provide easy accessibility.

Design of the furnace enclosure with heater has been completed and fabrication has commenced. The water-cooled enclosure is stainless steel pipe approximately 8 inches long and 5 inches in diameter. A folded tantalum heater, similar in construction to those in current use, is attached directly to one end of the enclosure in order to maintain adequate optical alignment at all temperatures. Two annular conductors with electrical insulation between them function as the heater electrodes as well as the enclosure and cover. Tantalum sheets act as thermal shielding between the heater and the enclosure.

An indium arsenide detector with integral glass Dewar and sapphire window has been ordered along with two sapphire lenses to be used to measure specimen temperature. In view of its short time constant and spectral response, InAs seems to be the most logical choice for a single detector for the temperature range 200 to 2000°C, especially, when most interest is focused on 500°C and above.

The two sapphire lenses will permit a circular area of approximately 3 mm diameter on a 13 mm diameter specimen to be viewed by the detector. Smaller areas on the specimen may be viewed with the system but with a corresponding decrease in detector output. This decrease in detector output will ordinarily be worthy of notice only in the temperature range estimated to be below 350°C (177°F). Sapphire was selected to ensure that all long-wavelength radiation to which the detector is sensitive will be passed and still permit visual alignment of the system.

2. Contract Title: Development of a Micrometeoroid Simulation Device
Contract Number: NAS8-20529
Contractor: Canadian Commercial Corporation
Contract Cost: $259,018.00
Contract Duration: 18 Months
Negotiated: June 25, 1965
Responsible Individual: C. O. Gray

Purpose of Project:

The objective of this project shall be to design, fabricate, deliver, install, and demonstrate a test device suitable for simulating micrometeoroid impacts on a variety of materials and composites or on multilayer structures. The requisite test device shall be complete in all respects except for necessary utilities, such as electrical power, compressed air, cooling water, etc., and shall include a particle acceleration system with control and operating instrumentation, a target holder (as may be required), and all necessary diagnostic equipment and readout instrumentation as required for defining the accelerated particle at the moment just prior to impact in terms of (1) measured momentum, (2) mass, (3) physical state, and (4) velocity. The test device shall be capable of routinely accelerating and impacting an isolated, single, discrete particle of solid matter in the mass range of 0.5 to 0.0001 gram to velocities from 20,000 to at least 50,000 feet per second (6096 to 15,250 meters per second). However, a velocity of 100,000 feet per second (30,480 meters per second) shall be investigated as a velocity design objective.

Technical Status:

The effort during the quarter has been concentrated on the experimental performance testing of the proposed accelerator concept. Firings have been conducted to establish the effect of each of the primary loading variables on the final velocity attained. Comparison of the experimental results and the predictions of earlier internal ballistics analysis has indicated severe discrepancies although some general trends have been predicted correctly. An overall examination of results of experimental firings to date has revealed the most important loading variables. Recent firings to optimize performance have met with more positive results than those conducted in the earlier part of the second quarter.

The temporary experimental facility setup for the firings has provided a valuable test bed for the instrumentation proposed for the final facility which is to be constructed during the next two quarters. The originally proposed instrumentation setup has been simplified overall to provide greater reliability and reduced operating effort. The final test range layout has been established and final drawings are now being prepared.

The primary launcher (light gas gun) design for the facility to be delivered to the NASA has been completed and drawings have been issued for quotations.

Modifications to the flash X-ray coverage on the temporary facility now in use at Computing Devices, have made it no longer possible to make experimental measurements of the in-flight projectile mass. This condition will of course be corrected on the final facility which will provide for three independent measurements of the projectile mass on each firing.

In general, all parts of the program, with the exception of the experimental performance demonstration, are on schedule and no unforeseen difficulties have been encountered which might have affected the program goals.

Four experimental firings were completed in January and February. The results showed no significant increase in performance beyond previous results; therefore, procurement of the facility components has been delayed. Efforts of the immediate future will be directed toward a more intensive theoretical analysis and computer simulation firing to develop a more logical approach for selection of experimental conditions which will isolate the reason for the apparent limitation on performance of firings to date.

II. General - In-House

A. Study of Silicon-Nitrogen Polymers

This program is being conducted to augment work by Southern Research Institute in the same general area under contract NAS8-20190. Progress on the contracted effort is reported elsewhere in this report.

A method has been developed for the preparation of a new silazane compound, trianilinosilane. The method used was a modification of that employed by Anderson (Journal of the American Chemical Society 72, 5802, 1951), to prepare similar compounds with a lower order of amine substitution. The method involves reacting a 1:3 molar ratio of phenyltrichlorosilane and aniline in the presence of triethylamine in benzene solvent. The product is recrystallized from isooctane and has a melting point of 132-134°C (270-273°F). The infrared spectrum of this compound has partially confirmed its structure and other confirmatory analyses are in progress. This compound will be tested as a crosslinking agent for polyaryloxysilanes and other adhesive prepolymers.

A total of 500 grams of bis(methylamino)diphenylsilane has been prepared by the method described by Burks et. al. (contract NAS8-20190, progress report No. 3). Excess methylamine is reacted with dimethyldichlorosilane in a suitable solvent. The product is purified by distillation and has a boiling range of 103-104°C (217-219°F) at 0.2 torr. Its purity was confirmed by gas chromatography and refractive index. This silazane will be used as a curing agent for epoxy adhesives and as a comonomer for the preparation of polyaryloxysilanes. In the latter application, it should yield polymers free of the undesirable aniline decomposition products that are trapped in the polymer by current preparative methods.

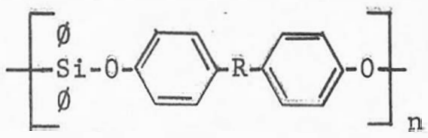
A new silazane compound, phenyltranilineosilane, has been prepared, isolated and characterized. A working quantity of bis(methylamino)diphenylsilane has been prepared. These compounds will both be evaluated as epoxy resin curing agents, and as reactive intermediates in copolymerizations with organic diols. Related monomeric structures will be investigated for possible routes to spirosilazane polymers.

B. Development of High Temperature Resistant Polymers

This effort is being conducted as an adjunct to contracts NAS8-11338 with Midwest Research Institute and NAS8-11837 with Battelle Memorial Institute. The contracted work is reported elsewhere in this report.

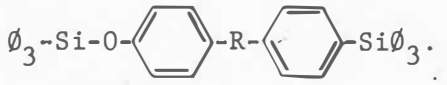
Work has continued on the study of the effect of different diol monomers upon the properties of polyaryloxysilanes. This is accomplished by using different diols which give rise to varying R groups in the

following representation of the basic polymer structure:



A total of nine different R groups were studied. It was found that where R is -O-, $-CH_2-$, $-\underset{Et}{\overset{Et}{C}}=C-$ or $-C(CHCH_3)=C(CHCH_3)-$, polymers of high molecular weight (Mw = 70,000 - 120,000) are obtained. These polymers are tough, clear, semiflexible plastics, and are stable to about 450-500°C (752-932°F).

In connection with the preparation and study of the above polymers, seven new related model compounds also were prepared. These compounds coreesponded to the new polymer structures as shown



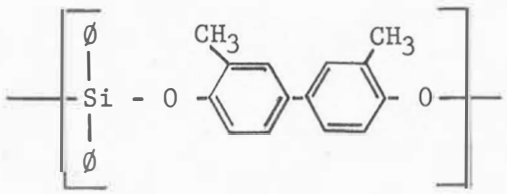
with R groups as shown above.

The preparation and characterization of a number of other model aryloxysilane compounds was reported previously. A limited amount of hydrolytic stability work was done on two of the compounds reported previously, 4,4-bis(triphenylsiloxy)biphenyl (V), and p-phenylphenoxy-triphenylsilane (VII). Rate constants were determined for the hydrolysis of these compounds in media of varying pH.

Compound	pH	$k \times 10^4$ sec^{-1}
V	12.05	28
VII	7.0	0.024
VII	9.0	71.19
VII	12.05	108

Some problems with incomplete solubility were encountered and the accuracy of these constants is subject to question. However, it is indicated that the rate of hydrolysis is quite low, even under these adverse basic conditions.

A new polyaryloxysilane has been prepared in high molecular weight.



This polymer was prepared after the corresponding model compound, 4,4'-bis(triphenylsiloxy) 3,3'-dimethylbiphenyl (VI) was studied and found to be relatively stable to hydrolysis. In fact, no measurable hydrolysis could be observed under the conditions employed to study compounds V and VII. This new polymer is a very tough, semi-flexible, clear solid which offers potentially useful engineering properties. It is also stable to 500°C (932°F) and has a somewhat higher residue (75 percent) at 900°C (1652°F) than does poly(4,4'-bisoxybiphenylene)-diphenylsilane (Polymer A). It is concluded that the methyl substitution on the biphenyl group increases both the hydrolytic and thermal stability of the basic Polymer A structure.

A number of new aryloxysilane polymers have been prepared and characterized. Their average molecular weights have ranged upward to 120,000 and some of them have very useful properties. The materials are generally very tough, semi-flexible, clear plastics which are stable to about 500°C (932°F). A number of new aryloxysilane model compounds have been prepared. Some kinetic studies have been done on the rates of hydrolysis of selected model compounds.

Future work will concentrate on the preparation of a chemically crosslinkable system for polyaryloxysilanes. This will include the study of methyl substituted polymers crosslinked by peroxides, and the use of some trifunctional monomers. Work will also continue on the preparation and study of new polymer structures.

C. Development and Characterization of Phosphonitrilic Polymers

This project is being supplemented by W. R. Grace and Company under contract NASw-924 which is described in the contract research section of this report.

The projected synthesis of alkyl tetrameric phosphonitrilic chlorides was continued with additional investigation of the alkylation of tetrakisdimethylamide derivatives of $(PNCl_2)_4$ with Grignard reagents as outlined below:

$$\underset{\text{I}}{(PNClN(CH_3)_2)_4} + 4\ CH_3MgX \longrightarrow \underset{\text{III}}{P_4N_4(CH_3)_4(N(CH_3)_2)_4} + 4\ MgXCl$$

II IV

In contrast to the alkylation of trisdimethyl derivatives of $(PNCl_2)_3$ which can be accomplished in 87 percent yield, it was determined through a number of experiments that the reaction of I with stoichiometric amounts of II proceeds only to the extent of replacement of two of the ring chlorine atoms with methyl groups even under the forcing conditions:

$$(PNClN(CH_3)_2)_4 + 2\ CH_3MgX \longrightarrow P_4N_4Cl_2(CH_3)_2\ N(CH_3)_2)_4 + 2\ MgXCl$$

Elemental analysis for chlorine in the product agreed closely with the percent composition computed from the imperical formula.

In a typical reaction I was treated with a stochiometric quantity of CH_3MgCl in THF (tetrahydrofuran) and the resulting solution heated at reflux for 72 hours. At the end of this time, the solution was concentrated in vacuo and the residue exhasutively extracted with cyclohexane in a Soxhlet apparatus for 24 hours. Removal of the solvent afforded a slightly turbid oil which was dissolved in benzene and chromatographed on activated alumina. Elution with ether and subsequent concentration in vacuo of the eluates gave a 56 percent yield of clear colorless oil. Vapor phase chromatography showed the presence of at least three components, presumably geometrical isomers.

The projected synthetic approach was found to be further complicated when it was attempted to regenerate the oil to the hexachloro derivative with anhydrous hydrogen chloride:

$$\underset{\text{IV}}{P_4N_4Cl_2(CH_3)_2(N(CH_3)_2)_4} + 8\ HCl \longrightarrow \underset{\text{V}}{P_4N_4(CH_3)_2Cl_6} + 4\ \underset{\text{VI}}{HN(CH_3)_2 \cdot HCl}$$

The introduction of gaseous HCl into a refluxing solution of IV in xylene gave rise to the separation of solid material which was subsequently isolated as a fine white powder, melting point 135-145°C (275-293°F), (melting point of dimethylamine hydrochloride, VI, is 170-171°C (338-340°F). Examination of its infrared spectrum showed an absorption band at 2400 cm^{-1} due to the presence of $-NH^+$. Instead of being regenerated to the hexachloro derivative, as outlined above, IV was forming a hydrochloride salt:

$$P_4N_4Cl_2(CH_3)_2(N(CH_3)_2)_4 + HCl \longrightarrow (P_4N_4(CH_3)_2\ Cl_2\overset{(+)}{\underset{H}{N}}(CH_3)_2)_4Cl^{(-)}$$

Thus, even if complete alkylation of the tetrameric PN ring could be accomplished, it would probably not be possible to convert the tetrakis-dimethylamide derivative to the corresponding tetrachloro derivative for ultimate conversion to ladder-type polymers.

This continued inability to synthesize alkyl tetrameric phosphonitrilic chlorides has necessitated, at least for the present time, the discontinuation of further work in this area. Consequently, further work will be devoted to the preparation of aromatic substituted phosphonitrilic chloride tetramers in an effort to prepare ladder-type polymers with thermal stability exceeding the present limit of 400°C (752°F) now being experienced with tetrameric phenylphosphonitrilic chloride polymers.

To this end, it was decided initially to investigate alkoxy substituted phenyl phosphonitrilic chloride tetramers and the n-butoxyphenyl group was

arbitrarily chosen for initial study. However, the necessary intermediate n-butoxyphenyl phosphorous chloride could not be prepared during an investigation described in a recent Russian publication in which butylphenyl ether was treated with excess phosphorous trichloride in the presence of fused zinc chloride.

An attempt was made to repeat the reaction using anhydrous aluminum chloride as the catalyst. However, the aluminum chloride complexed with the ether and the resulting oxanium complex could not be selectively decomposed to yield the $RPCl_2$ derivative.

Therefore, it was decided to investigate an alkylphenyl substituted type system and for this study the n-butylbromide group was arbitrarily selected. Butylbenzene was prepared through the anhydrous ferric chloride catalyzed reaction of phenylmagnesium bromide with n-butylbromide in ether. Work-up of the reaction mixture gave the desired product in moderate yield.

n-Butylphenylphosphorous dichloride, $RPCl_2$, was prepared in 55 percent yield through the anhydrous aluminum chloride catalyzed reaction of butylbenzene with excess phosphorous trichloride.

The crude product was fractionally distilled through a Vigreoux column and the fraction boiling at 98-100°C (208-212°F)/50 torr was collected. Examination of the infrared spectrum of this material showed the presence of both ortho and para substituted derivatives. Vapor phase chromatography of the distillate indicated that it consisted of 85 percent para substituted product and 15 percent ortho substituted derivative even though its boiling point was over only a 2°C range.

For the preparation of the tetrameric butylphenyl phosphonitrilic chlorides, the phosphorous dichloride was first converted to the corresponding butylphenyltetrachlorophosphorane, $RPCl_4$.

Following the preparation of ammonium chloride *in situ* in chlorobenzene, a chlorobenzene solution of the phosphorane was added rapidly to the stirred refluxing mixture of the ammonium chloride:

$$4\ C_4Hg\text{-}C_6H_5PCl_4 + 4\ NH_4Cl \longrightarrow (C_4Hg\text{-}C_6H_5)_4\ P_4N_4\ Cl_4 + 16\ HCl$$

After 24 hours the evolution of HCl had ceased and the mixture was filtered. Concentration of the filtrate in vacuo left a residue of brownish oil which did not crystallize upon standing over a 48 hour period. The infrared spectrum of the oil showed PN ring absorption at both 1180 and 1300 cm^{-1} indicating the presence of both trimer and tetramer.

It was not known until after the last reaction had been started that the $RPCl_2$ contained a large percentage of ortho derivative. Its presence in the reaction would lead to an abnormally high total number of trimeric and tetrameric geometrical isomers which would require a major undertaking to effect complete separation and identification.

Therefore, the products of this reaction will not be further investigated, and future efforts will be directed toward using mesitylene-(1,3,5-trimethylbenzene) whose reaction with PCl_3 can lead to the formation of only one substituted derivative.

These efforts have established that the synthesis of tetrameric methyl phosphonitrilic chlorides may not be possible by any method presently known. These studies have shown that only partial alkylation of the tetrameric PN ring is effected with Grignard reagent even under forcing conditions. Subsequent efforts to regenerate the partially alkylated derivative to the corresponding hexachloro configuration have led to the formation of the tetrakisamido hydrochloride salt instead.

Future in-house plans call for the evaluation of mesitylene-1,3,5-trimethylbenzene, as the starting material in a projected synthesis of tetrameric phosphonitrilic chlorides for ultimate conversion to high molecular weight polymers possessing thermal stability exceeding the present limit of 400°C (752°F) as experienced with the phenyl derivatives.

D. Investigation of Materials for Electrical Contacts in Space

Electrical contacts in vacuum concerns any device for transferring electrical energy through moving contact surfaces, such as brushes, slip rings, and make-break switches. Standard brush-commutator type machines suffer a severe degradation of performance at high altitudes, principally due to rapid wear of the graphitic carbon brushes. This results from failure of the normal process of lubrication of the contact surfaces. Therefore, this program was initiated to develop electrical brushes for use in a space environment.

A test series has begun to determine the influence of environmental test pressure on wear rate. Brushes of 85 percent molybdenum disulfide-15 percent silver pressed at 4,000 psi will be operated at 2,000 rpm and 1.0 ampere brush current. Pressure will be varied in one decade steps, operating for 100 hours in each range.

Two tests were made on brushes of $NbSe_2$ (niobium diselenide). One set of brushes was vacuum annealed at 1000°C (1837°F) for 24 hours and then brought back to ambient temperature at the rate of 3°C (5.4°F) per minute. These brushes ran for four hours prior to the onset of severe arcing which caused heavy deposits on the commutator. The second set of $NbSe_2$ brushes were hot pressed at 5,500 psi and 760°C (1400°F). These brushes ran for six hours until the coefficient of friction had increased to the point where the magnetic drive slipped.

The microstructure of MoS_2-Ag brush materials varying from 60 MoS_2-40 Ag to 85 MoS_2-15 Ag (volume percent basis) hot pressed at 927°C (1700°F) with 4000 psi mold pressure has been determined for the 4 micron MoS_2 and 0.3-6 micron Ag. The $NbSe_2$ brush materials being developed offer the possibility of operation with brush current densities greater than 30 amperes per square inch under conditions where brush sparking or arcing may occur. These are conditions under which the performance of MoS_2-Ag brush materials is known to be inadequate.

A limited investigation of a two-phase brush material based on $NbSe_2$ will be made. The second phase will be a hexagonal, lamellar structured sulfide or selenide added to reduce the coefficient of friction and apparent brittleness of the $NbSe_2$.

One additional test was made in the test series to determine the optimum spring pressure for the 85 MoS_2-15 Ag composition. Brushes were run at 2,000 rpm at 1.0 ampere brush current for 600 hours with a 12 psi spring installed. The wear was 4.6×10^{-5} inches/hour. However, one spring had dropped to 6 psi at the end of the test and the data is therefore inconclusive. The brushes in this test were used for all of the brush spring pressure tests, and have accumulated 2,959 hours at an average wear rate of 1.7×10^{-5} inches/hour.

E. Investigation of the Dielectric Properties of Materials

Teflon tubing used in the hydraulic system of the S-IC stage accumulates an electrostatic charge due to the high velocity flow of fluid through the filter system. The charge is great enough to cause electrical breakdown of the unfilled Teflon high pressure tubing. Therefore, a study was initiated to determine the effect of high conductivity additives on the electrical conductivity of the hydraulic fluid and the Teflon tubing. If the additives increase sufficiently the conductivity of either the Teflon or the RJ-1 fluid, it is believed that the accumulated charge will be eliminated or reduced to a low level so that electrical breakdown of the tubing will not occur.

The tests to evaluate the conductivity of Teflon tubing with (1) carbon added and (2) with a carbon liner have been completed and a memorandum containing the results has been written. The data indicate that (1) there is no appreciable difference between the conductivity of Teflon tubing with 2 weight-percent carbon added and that of pure Teflon and (2) that Teflon tubing with a carbon liner has a conductivity six orders of magnitude greater than either the pure Teflon or the 2 weight-percent carbon-filled Teflon.

Tests to evaluate the electrical conductivity of RJ-1 containing differing amounts of additives currently are being made.

Recently, a critical need arose for a sensitive method of determining the impurity content of N_2O_4 (nitrogen tetroxide) oxidizer.

Consequently, a study was initiated to investigate the feasibility of using electrical methods for this purpose.

The tests planned for this program have been completed and a memorandum describing the results has been written. Dissipation factor data obtained as a function of frequency indicate peaks which may be associated with the absorption of energy by the impurities in the N_2O_4. However, a quantitative relationship between the number of peaks and the impurity content has not been obtained due to the unavailability of pure N_2O_4 for reference measurements.

F. Investigation of Nuclear Environmental Effects on Materials

1. Study of the Effects of Radiation on Thermal Control Coatings

A test program has been initiated to determine electron and proton induced changes in the optical properties of various thermal control coatings in order to evaluate their suitability for use on spacecraft and launch vehicles. Irradiations in air and vacuum with electrons and in vacuum only with protons are planned. Equipment will be developed to permit in-situ optical property measurements.

The integrating sphere and associated spectrometer system required for in-situ optical measurements are being mated to the AVCO/Tulsa developed optical coupling unit. The complete system should be operational the latter part of May. In the meantime, irradiations are being planned for the specimens of S-13 and 4-93 coatings which presently are being optically evaluated by the Hughes Aircraft Company on a service contract basis.

2. Application of Radiation Induced Defects to Study of Stress Corrosion

Stress corrosion of critical stage hardware currently is a major problem and one which is in need of a solution based on an understanding of the stress corrosion mechanism. Consequently, a test program was formulated to determine what effect point defects, produced by proton bombardment, would have on the susceptibility to stress corrosion of selected metals.

Stress corrosion testing of the longitudinally cut 7079-T6 tensile specimens is continuing. As soon as the results of these tests become available, the data will be analyzed to determine to what extent proton bombardment influences the stress corrosion mechanism.

G. Documentation Review

The following specifications, documents, or reports were reviewed and comments were forwarded, where appropriate, to responsible individuals or organizations:

1. Rocketdyne Specification NA5-260142, "Gimbal Assembly, Helium Inlet"

2. North American Aviation Specification MA0607-002, "Manual Fusion Welding of Magnesium Alloys for Saturn S-II Vehicle"

3. MIL-W-12332 Amend 1, "Welding, Resistance, Spot and Projection for Fabricating Assemblies of Low Carbon Steel"

4. MPD-2000, "Welding Spot (Commercial)"

5. MIL-2672 B, "Aluminum Brazing"

6. MIL-W-6858C, "Welding, Resistance: Aluminum, Magnesium, Non-hardening Steels or Alloys, Nickel Alloys, Heat Resisting Alloys, and Titanium Alloys: Spot and Seam"

7. MC-611, "Rivet, Blind, Mechanically Locked Stan, Self Plugging, 100° Countersunk Head, Monel and Corrosion Resistant Steel, Pull Tool Type"

8. MC-612, "Rivet, Blind, Mechanical Locked Stem, Self-Plugging, 100° Countersunk Head, Aluminum Alloy, Pull Tool Type"

9. MC-612, "Rivet, Blind, Mechanical Locked Stem, Self-Plugging, Protruding Head, Monel and Corrosion Resistant Steel, Pull Tool Type"

10. MC-614, "Rivet, Blind, Mechanical Locked Stem, Self-Plugging, Protruding Head, Aluminum Alloy, Pull Tool Type"

11. MC-640, "Lockbolt Pin, Pan Head, Close Tolerance, Stainless Steel"

12. MC-641, "Lockbolt Pin, 100° Countersunk Head, Close Tolerance, Stainless Steel"

13. MC-642, "Lockbolt Pin, 100° Countersunk Head, Close Tolerance Shank, Alloy Steel or A-286 Stainless Steel, Shear Application"

14. MC-643, "Lockbolt Pin, Pan Head, Close Tolerance Shank, Alloy Steel or A-286 Stainless Steel Shear Application"

15. NAA MA0110--13 rev. G, dated 11-9-65, "Descaling and Passivation of Corrosion and Heat Resistant Alloys"

16. AMS 2410C (For DAC Use), "Silver Plating - Nickel Strike - High Bake"

17. Parker Aircraft Co. EPS2661270. "Spring Replacement Procedure for MF Fittings and Assemblies"

18. Standard Part Drawings

MF873, MF874, MF806, MF821, MF824, MF833, MF837, MF844
MF919, MF1784, MF1804, MF1834, MF1837, MF1919, MF4039

19. Parker Aircraft Co. ES2-90, "Fabrication and Handling of MF4039 Spring Washers"

20. Space and Information Systems Div. MQ0501-008, "Nondestructive Testing Requirements for Materials and Processed Parts"

21. Douglas Aircraft MAS-5734C

22. Rocketdyne NAS-260142, "Gimbal Assembly, Helium Inlet."

H. Literature Survey

Surveys of the pertinent literature have been initiated or are continuing on the following subjects:

1. Radiation effects on engineering materials
2. Vacuum effects on engineering materials
3. Liquid and gaseous hydrogen and oxygen
4. Lubricants and lubricity
5. High and low temperature resistant polymers
6. Stress corrosion on structural alloys
7. Measurement of residual stresses
8. Ultrasonic inspection techniques
9. Refractory metals
10. Metallic fracture surfaces
11. Weld defects in aluminum.

for J. E. Kingsbury

MONTHLY PRODUCTION REPORT
MATERIALS DIVISION
MARCH 1 - 31, 1966

I. One thousand two hundred and forty-three miscellaneous parts, components and test specimens were inspected radiographically during this report period; four parts were inspected by magnetic particle techniques and two items were inspected by dye penetrants.

II. Photography

	Negatives	Prints	Other
Engineering photography	167	1201	
Metallography and fractography	105	743	
Miscellaneous photography, processing, copywork, etc.	34	114	41

III. Metallurgical and Metallographic Testing and Support Services

A. Metallographic studies were completed on samples of 2219 aluminum joined to 321 stainless steel by the co-extrusion process. Metallographically the bond appeared good; however, the samples are being studied further by electron microprobe analysis to check the depth of diffusion.

B. Failure analysis was completed on an S-IC inlet flight supply line that ruptured during pressure cycling tests being made by Propulsion Division personnel. The failed line consisted of an inner teflon hose jacketed with a 300-series stainless steel flex hose for reinforcement. The only irregularity discovered during the study was misalignment of several of the wires in the flex hose. This condition could cause premature failure by virtue of abrasion and/or stress concentration at the point of wire cross-over. It was recommended that future flex hoses be rigidly inspected for wire misalignment before and after testing.

C. A metallographic study has been completed on several gold-plated contact pins at the request of R-QUAL-QET. The plating thickness ranged from 40 to 60 micro inches, which is below the required 60 to 90 micro inches. The core material was found to be Kovar base.

D. Metallurgical study has been completed of braze repairs on 347 stainless steel tubing from F-1 Engine system. This study was made at the request of the Engine Management Office. No cracks were revealed in the braze interface during the examination. The bond appeared to be adequate; however, no metallurgical mixing of the braze material or base material occurred. A well defined line was found at the interface.

E. A metallurgical evaluation of a swaged Resistoflex fitting has been completed at the request of the Propulsion Division, R-P&VE-PAE. The AM 355 stainless steel fitting contained a swaged-in 17-4 stainless

steel tubing. Metallographic examination revealed a good fit between the two components. Carbide networks were found in both materials.

F. Hardness determinations were made on several bronze electrical contact parts at the request of the Astrionics Laboratory.

G. Metallographic studies were completed on a failed worm gear from an experimental RL-10 actuator. The gear failure was caused by a malfunction in the clutch mechanism. No materials defect was noted; however, the gear appeared to be plated rather than surface hardened.

H. Metallurgical examination was completed on a small metallic fragment removed from a jet engine at the request of the Manned Spacecraft Center. The material was identified chemically as being a 300-series stainless steel. Metallographic studies indicated that the fragment was a piece of welded metal tubing about 3/8-inch diameter.

I. Failure analysis was completed on a failed experimental fuel-LOX injector. Metallographic examination showed lack of penetration in the electron beam welded face plate allowing leakage between the LOX and fuel which resulted in an explosion. Erratic depth of weld penetration was evident in the various rings.

J. Failure analysis was completed of a Saturn V hold down separation link that failed during testing at this Center. The failure was attributed to the presence of a previous fracture that propagated during testing. No reason was determined for the pre-crack in the component since the history of the component was unknown prior to the failure.

K. Failure analysis was completed on a failed LN_2 line junction (Parker Aircraft Company design) at the request of the Propulsion Division. Failure of the component was attributed to fatigue.

L. A test program was conducted on material removed from four shear formed LOX tunnels to determine the variance of mechanical properties between tunnels. The variance was sufficient to overlap the properties of magnetically sized tunnels. A report has been prepared reflecting the results of the program.

M. A limited investigation was made to determine the thermal response of 7001 aluminum alloy. Hardness and electrical conductivity measurements were made on 7001 in the T-75 condition. The material was re-solution heat treated and aged in a manner to achieve the same hardness and electrical conductivity values as the original T-75 condition.

IV. Spectrographic Analysis

Three hundred and ninety determinations were made on thirty samples and six hundred and thirty-eight standard determinations were made.

V. Infrared Analysis

Samples of nine miscellaneous polymers, monomers, and chemical compounds were analyzed by infrared techniques during this report period.

VI. Chemical Analysis

	Determinations
methanol-water mixture for	
sodium benzoate content	3
Dow Clene solvent for water	2
Metal samples for	
carbon	14
oxygen	20
nitrogen	30
copper	6
phosphorus	4
tin	3
hydrogen	20
Gas samples for	
nitrogen	28
oxygen	42
hydrogen	32
helium	16
argon	16
carbon dioxide	16
moisture	4
waste water for hexavalent chromium	5

VII. Physico-Chemical Analysis

	Determinations
Density of	
petroleum ether	6
RP-1 fuel	20
tungsten carbide	3
pH of water-methanol solution	3
specific resistance of water-methanol	3
viscosity of petroleum ether	6
surface tension of petroleum ether	6

VIII. Rubber and Plastics

	Items
molded or extruded	105
cemented	2
fabricated	41
coated	23

IX. Electroplating and Surface Treatment

	Items
painted test specimens	34
chemically coated	12
electroplated	17

X. Development Shop Production

A. A total of 5,964 man-hours, direct labor, was utilized during this period for machining, fabricating, and welding.

B. One thousand two hundred and eighty-two man-hours, approximately 21 percent of the total man-hours, were devoted to productive effort of a nonroutine nature and applied to the work orders listed below.

1. Impact Tester

Work on the impact tester is still delayed because of more urgent requests.

2. Butterfly Valve Assembly

The Butterfly Valve Assembly is complete and delivered.

3. F1 - GG Injector Assembly

The F-1 engine injector assembly is complete and delivered.

4. Baffle Assembly Zero "G" Drop Test

Work is in process in three additional baffle assemblies for the Zero "G" drop tower.

5. Radiation Shield and Sample Chamber

Work on the radiation shield and sample chamber is approximately 90 percent complete.

6. LOX and LH_2 Container

Work on the LOX and LH_2 container units is approximately 85 percent complete.

7. P & W Inducer Test Components

Work on the Pratt and Whitney inducer test components is complete except for contour machining.

8. Ultrasonic Specimen Goniometer

Work is planned and material is on order for fabrication of an ultrasonic specimen goniometer.

9. Test Fixture - S-IVB Engine Simulator

Work on the S-IVB Engine Simulator is ahead of schedule.

XI. Miscellaneous

A. Sixty-four miscellaneous components and test items were flame-sprayed during this report period.

B. Seventeen parts were fabricated from lava.

C. Twenty-six steel items, five items of titanium alloy, forty items of nickel alloy and one hundred and four aluminum alloy items were heat treated during this report period.

D. Ten chromatographic analyses were made on five polymer specimens.

E. Four determinations were made of nonvolatile residue contamination in two filters.

F. Specimens from 48 jars of Dow Corning FS-1281 grease were tested for impact sensitivity in contact with liquid oxygen in accordance with MSFC-SPEC-106A.

XII. Publications

TM X-53404, "Galvanic Corrosion of Aluminum Assemblies by Stainless Steel Wire Inserts," by T. S. Humphries and E. E. Nelson, dated March 2, 1966.

TM X-53407, "Low Temperature Mechanical Properties of High Strength A-286 Bolts," by J. W. Montano, dated March 9, 1966.

J. E. Kingsbury