GEORGE C. MARSHALL SPACE FLIGHT CENTER

PR-P&VE-S-66-8

MONTHLY PROGRESS REPORT

STRUCTURES DIVISION

(August 1, 1966 - August 31, 1966)

SATURN IB

I. S-IVB Stage

S-IVB Panel Flutter Test

The pressure survey model, for the first phase of the S-IVB stage panel flutter test, has been delivered to the AEDC wind tunnel facility. Model installation and check-out are underway to meet the scheduled test date of September 19, 1966.

The necessary S-IVB skirt segment modification and test box fabrication for the second phase of the S-IVB stage panel flutter test has been completed by the Manufacturing Engineering Laboratory. Preparations are being made for the loads test of the skirt specimen. The wind tunnel test is now scheduled for the week of October 3, 1966.

II. General

A. AS-206 Nose Cap Ejection Assemblies

Twenty-two nose cap ejection assemblies were calibrated and prepared for shipment for use on the AS-206 vehicle. The spring rate for these assemblies was in the 430 #/in range instead of the desired 480 #/in range; however, these rates are acceptable. All the springs (170) were calibrated to select 22 assemblies with optimum spring rates. From the remaining 148 springs, calibrated assemblies will be prepared for the AS-208 and AS-210 vehicles.

B. Real Time Bending Moment Display

A real time Uprated Saturn I vehicle bending moment display at MSFC was requested in support of the MSC command module tension tie problem. The bending moment will be displayed during prelaunch for vehicles AS-202

through AS-212. The display system has been implemented at Computation Laboratory.

SATURN V

I. S-IC Stage

Structural Qualification Test

All major structure components of the S-IC stage SA-501 - 503 configuration, have been fully qualified for man rating (Safety factor of 1.4 on design loads) by the successful completion of the oxidizer tank assembly test, July 29, 1966. Instrumentation data showed that the specimen sustained stresses in the ultimate range (50,000 PSI). Inelastic buckling appeared imminent in the forward skirt and in the S-II interstage. The forward skirt had no permanent deformation and can be used for the S-IC, S-II interface test. Load peaking at the S-IC, S-II interface was not as severe as calculated by the contractors.

The oxidizer tank assembly has been removed from the Load Test Annex and the short lox tank, which contains the sculptured Y-ring, installed.

II. S-II Stage

A. LH_2 Tank Cracking Problem

S&ID presented to MSFC at a meeting on August 18, 1966, the findings regarding the cause of the extensive numbers of cracks which are being found in every S-II stage. While it was evident that a great amount of effort had been expended by S&ID, it appeared that the cause of the problem is still very nebulous.

An important decision reached in the meeting was the concession by S&ID that the CBTT would be used to test the mechanically fastened doubler design to ultimate pressure. S&ID was reluctant to subject the CBTT to ultimate pressure since the required pressure will exceed the previous test pressures employed in the original CBTT program.

B. Establishment of Allowable Pressure Cycling for S-II-F and S-II-1

On August 19, 1966, a meeting was held between P&VE and S&ID to establish the allowable pressure cycling for S-II-F and S-II-1. Since firm technical data was not available, the following decisions represented the best subjective opinions:

1. S-II-F:

   The LH_2 tank may be pressurized at KSC a total of 36 times,

no more than 6 of these may exceed 10 psig. If this limit is reached the tank must be inspected.

2. S-II-1:

The LH_2 tank may be pressurized at MTF a total of 80 cycles; however, no pressure cycles are allowed above 10 psi before verification of the patch. Inspection will be required if the above limits are reached.

C. Debonding of Adhesive Patches on S-II-3

The pneumostatic pressure proof test of the S-II-3 stage was aborted at 30 psig during a planned pressurization to 35 psig. This decision was made from an evaluation of the strain data which indicated that the bonded patches at S&ID station 722 were becoming debonded. Subsequent ultrasonic inspection confirmed that the patches were debonding. All bonded patches on the S-II-3 have been removed and mechanical doubler repairs are planned as replacements.

D. Bolted Patch Concept

S&ID and P&VE have agreed that it is feasible to proceed with installation of the bolted patch design on S-II-1 and S-II-3 even though detailed analysis and qualifying test data is not now available. It is the opinion of Structures Division that the design is reasonable enough to justify the risk of proceeding with rework prior to getting substantiating data. This concept is to apply only the external doubler plus the foil seal without using the "strong-back" ring frame reinforcement. This approach appears possible since a reduced factor of safety is applicable and since the skin thickness is greater than required.

E. Rehydrostat of the S-II-4 Forward Bulkhead/#6 Cylinder Assembly

The rehydrostat test of the S-II-4 forward bulkhead, #6 cylinder assembly was performed successfully on August 23, 1966, to the required proof pressures. The bolted doubler repair with the ring frame beef-up ("strong-back") was incorporated, including extensive strain instrumentation. A preliminary evaluation shows that the ring frame beef-up only reduced the load at the patch area by 10%. Further evaluation is continuing.

F. Interface Hole Problem: S-II-2

An inspection of the bolt holes in the S-II mating flange of the S-II/S-IVB interface shows that practically all of the holes have been elongated or otherwise damaged. The exact cause has not been established. Timely action will be required in the disposition of this problem since the mating S-IVB stage may also be impacted. Evaluation of the problem is proceeding.

G. Vibration Specifications

As a result of S-II-T firing data, revised aft skirt vibration specifications were generated. These specifications will apply to any components which have not previously been qualified to the original criteria. Also, as a result of S-II-T and S-IC-T firing data, revised thrust structure vibration specifications were generated. These specifications will be utilized in any future thrust structure component qualification program.

H. High Force Test Program

Acoustic testing of the aft interstage/thrust complex of the S-II stage at Wyle Laboratories has been completed. Failures occured on mounting brackets for the 3 batteries in the aft interstage. The specimens have been removed from the chamber and are being prepared for mechanical vibration testing.

III. S-IVB Stage

High Force Test Program

The S-IVB high force dynamic test program has been completed at Thiokol, Wasatch, Utah. There were no failures as a result of the test program and preliminary data evaluation indicates that the stage is structurally adequate to withstand anticipated dynamic environments.

IV. Instrument Unit

200/500S-3 Instrument Unit

The cold plates that were removed from 200/500V for use in the 200/500S-3 structural tests were proof loaded to 1.4 limit loads. They withstood these loads satisfactorily, and were installed in the structural test unit. Testing has been delayed because of instrumentation problems. These problems are being resolved.

Cabling from the strain gage bridge completion blocks to the digital data system was begun. Also, the installation of cold plate test fixtures was continued. Some questions were raised as to the necessity of the cold plate center swivel mount for this series of tests. IBM contends that they are required to increase the instrument unit's resistance to panel buckling. Also, IBM specified a maximum gap of 0.005 inch between the ST-124 mounting pads and I.U. when one pad is firmly bolted in place before pulling the other two pads into place. This will require shimming, and possibly machining of the ST-124 pads. Test preparations

for the 200/500S-3 structural qualification have been completed to the extent that a preliminary test run to 60% load was scheduled for the afternoon of August 26. These tests have been delayed approximately 10 days because of instrumentation problems. If the preliminary test run indicates that all systems are working properly, the first test condition will be performed August 29 or 30. It is still possible that the four test conditions can be completed by September 15.

ENGINES

I. F-1 Engine

Vibration Loads

Vibration loads for the F-1 engine turbopump struts and the center engine alignment struts have been determined for the condition of instant release. These loads are generally lower than the design vibration loads which were based on engine cutoff transients. These vibration loads will be combined with the other applicable loading to determine the structural adequacy of these struts for the instant release condition.

II. J-2 Engine

Exploding Bridgewire Initiator

An analysis of the Thiokol TX-346-1 exploding bridgewire initiator for the J-2-SE solid propellant gas generator using an omniseal indicates the initiator body has large margins of safety for proof and burst tests.

APOLLO APPLICATIONS PROGRAM

I. Payload Module Experiment Rack

Preliminary Design

The preliminary design of the rack structure was completed on schedule. Layouts with sufficient dimensions to order longlead materials were supplied to Manufacturing Engineering Laboratory. The payload module experimental rack design is progressing on schedule. A layout of the payload module docking structure, which is independent from existing LEM hardware, but, providing all docking features required, has been submitted for design approval.

II. Experiment Carrier Rack (ECR)

Critical load conditions

An impact study of the existing experiment carrier rack utilizing

ATM loading has been completed. Several of the rack members were critical. A decision has been made to design the rack to the combined critical conditions of all known payloads (PM, ATM, & PT).

III. Apollo Telescope Module

A. Vibration Specifications

ATM assembly vibration specifications were submitted and the preliminary dynamic loads study was completed. Since these interface (rack-to-ATM) loads are considerable less that the CPM interface loads, no design changes to the rack will be required.

B. Vibration Analysis

A preliminary vibration analysis has been completed to determine the first bending mode frequency of the ATM in its docked configuration. The analysis model included the rack, ATM ascent stage, and the command and service modules. This frequency was determined to be 10 cps and will be compared with the forcing frequencies induced by vehicle maneuvers to see if they are coupled.

IV. S-IVB Spent Stage

A. Acoustic Response Study of Micrometeoroid Shield

A parametric acoustic response study of the proposed S-IVB spent stage micrometeoroid shielding has been completed. In this study the maximum acoustic response loads and resulting micrometeoroid shielding deflections were determined for various shielding materials, skin gages and support concepts. These loads and deflections will be used in a weight optimization study for the proposed shield.

B. Spent Stage Experiment Support Module

The design of a meteoroid test tank to be used by Materials Division at Tullahoma, Tennessee was initiated. The tank is 36" in diameter and will allow testing of flame propagation of the S-IVB stage internal insulation when hit by meteoroids.

ADVANCED TECHNOLOGY

I. Titanium Crossbeam

The test set-up for testing the titanium crossbeam was completed with the exception of the instrumentation. The crossbeam will be subjected to a 40% load for 1 hour. After releasing the load, the crossbeam will

be inspected for visible cracks. Two days after the application of the 40% load, a complete dye penetrant check will be made. If the beam has not cracked to a degree that requires further testing, instrumentation will be added and the specimen will be loaded as per the test plan.

II. Nuclear Ground Test Module

Three thrust structure concepts are being investigated and have been given to Strength Analysis for sizing of elements. Physical properties and other data of recommended insulation materials have been requested from Materials Division.

III. Air Lock Design

Flexible and folding type doors and hatch designs are being investigated. Preliminary concepts have been completed and are being evaluated. A rigid type door and hatch design has been completed.

IV. Shroud Separation

Investigation into separation joint designs has been reactivated. Mechanical and gas-operated separation systems will be included with the pyrotechnic systems. Possible design requirements for a contaminent free separation on the Voyager shroud may be forthcoming and special emphasis will be given to suitable systems.

V. Bonded Honeycomb Cylinders

A problem of dimples in the inner face sheet has arisen during fabrication of the 60" panels. The dimples vary in depth from .0005" to .003". The cause of the dimples is unknown; however, thermal stresses and "trash" have been eliminated as possible causes.

VI. Cryogenic Test Tank #3 (105.0 inch dia.)

The manhole cover insulation assembly was blown off during the detanking operation following the insulation space test. The damage was apparently caused by a large gas leak from the manhole cover seal. Steps have been taken to replace the seal and repair the jacket and insulation.

VII. Contract NAS8-11397, The Martin Company, Cryogenic Insulation Research

The final test of this contract has been completed and the final report draft is nearing completion (est. completion date is Sept. 1). Using the results of previous tests, the final insulation performance was 50% improved over earlier results.

for G. A. Kroll
Chief, Structures Division

GEORGE C. MARSHALL SPACE FLIGHT CENTER

R-P&VE-M-66-8

MONTHLY PROGRESS REPORT

AUGUST 1, 1966 THROUGH AUGUST 31, 1966

SATURN IB

I. S-IB Stage

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

Efforts have continued in the development and evaluation of high-temperature, unfired, ceramic insulations for application in radiant thermal environments. Current efforts are directed toward development and evaluation of an insulation having a high zirconium oxide (ZrO_2) content. The high-temperature stability of ZrO_2 was the principal factor in its selection as the primary ingredient. The formulation of the most promising insulation to date was reported earlier. The exact composition, which has been designed as ZA-82 insulation, is as follows:

Ingredient	Parts by Weight
ZrO_2 "A" fibers	40
ZrO_2 powders (-325 mesh)	40
Asbestos fibers	20
Ludox HS-30 (pH-6.6)	210

ZA-82 is prepared, applied, and dried by the same techniques used for the FTA insulations.

The insulation capabilities of ZA-82 have been evaluated in radiant heating environments. For the initial evaluation, 0.350-inch thicknesses of the insulation were applied to stainless steel substrates of 0.040-inch thickness which were overlaid with expanded metal. The cured specimens were exposed to a radiant heat flux of 24 Btu/ft^2-sec for 180 seconds. The average back face temperature rise was 206°F (114°C). This compares to 323°F (178°C) for FTA-442A and 327°F (182°C) for M-31. To further evaluate the insulating qualities of ZA-82, 0.300-inch thicknesses of the insulation were applied to stainless steel honeycomb sandwich substrates of the type used on the S-IB and S-IC heat shields and exposed to radiant heat fluxes of 24 and 40 Btu/ft^2-sec. The results of these tests are compared to results of comparable tests on FTA-442A and M-31 in the following tabulation.

Back face temperature rise after 145 seconds of exposure to radiant heat of three insulations:

	Temperature Rise, °F (°C) Insulation		
heat flux	ZA-82	FTA-442A	M-31
24 Btu/ft^2-sec	110° (61)	115° (64)	189° (105)
40 Btu/ft^2-sec	242° (134)	404° (224)	494° (274)

These data indicate a superior insulating capability for the ZA-82 insulation when compared to FTA-442A and M-31, especially at the higher heat flux.

B. Development of Hazardous Gas Detection (HGD) Systems for Saturn Launch Complexes

Activities have continued in the assembly, qualification and delivery of hazardous gas detection (HGD) systems for use at Saturn launch complexes at the John F. Kennedy Space Center (KSC). Delivery of the second HGD unit to complex 34 was made during the countdown demonstration test, propellant loading and launch of AS-202.

The HGD unit for complex 37 is more than 95 percent complete; therefore, the schedule will be met.

SATURN V

I. S-IC Stage

A. Developmental Welding

Studies have continued on the susceptibility to cracking of weldments of various aluminum alloys. A number of weld restraint tests were evaluated, and none were found to be suitable for producing cracks in weldments in aluminum alloys in plate thicknesses of 1/2-inch or greater. Emphasis in these studies has been directed toward assessing the crack susceptibility of weldments in aluminum alloys of 1/8 and 1/4 inch thickness. During this reporting period "Houldcraft" restraint specimens were fabricated and evaluated for the welding of 5086, 7002, 5456, and 1100 aluminum alloys in thicknesses of 1/8 and 1/4 inch. Presently, circular patch specimens are being evaluated for 7039 and 6061 aluminum alloys.

Studies have been initiated to determine the weldability of X-7106, 2219, and 2014 aluminum alloys in 1/8-inch thickness by the electron-beam welding process. The intent of this program is to establish optimum electron-beam welding techniques. The study will include investigation of relative merits of single pass versus two pass welds. Sufficient data will be generated on mechanical and metallurgical properties to establish firmly

significant design data. One important objective of this program is to determine a nondestructive testing technique which can be used to properly evaluate electron beam welds. Presently, there is no established technique or standard for acceptance of electron beam welds.

B. Study of Corrosion and Cleaning Procedures

1. Stress Corrosion Studies

Investigations have continued into the stress corrosion susceptibility of the 7000 series aluminum alloys. Specimens of aluminum alloy 7039 in the -T61 and -T64 tempers have been exposed for 168 and 144 days respectively to alternate immersion in salt water and exposure to the atmosphere and to the local atmosphere. Failures occurred in both environments on specimens stressed as little as ten ksi in the short transverse direction of grain growth.

Carpenter Custom 455 stainless steel, aged at 1000°F (538°C) and 1100°F (593°C) was found resistant to stress corrosion cracking in the alternate immersion environment. Additional specimens of this alloy aged at 1150°F (621°C) and stressed to various loads up to 100 percent of the yield strength are currently being tested. No failures have occurred after 135 days exposure.

Specimens of aluminum alloys X2021 and X7007 were stressed in all three directions and exposed in the alternate immersion tester and to the local atmosphere. Preliminary tests for the threshold stress levels for X2021-T8E3 (90 days) and X7007-T6E136 (180 days) in the alternate immersion tester are complete. These test data indicate that the threshold stress level for X7007-T6E136 in the short transverse grain direction is below 10 ksi (15 percent of the yield strength), and less than 51 ksi (75 percent of the yield strength) for the longitudinal grain direction. The only failures encountered in the local atmosphere are two specimens stressed in the short transverse grain direction at 20 ksi (30 percent of the yield strength). These failures occurred after 62 and 112 days of exposure. The results of tests on specimens of X2021-T8E31 indicate that the threshold stress level for the short transverse grain direction is below 25 ksi (40 percent of the yield strength) and below 45 ksi (75 percent of the yield strength) in the long transverse and longitudinal grain directions. However, severe general surface corrosion may be interfering with the test results. Additional tests on these alloys are planned to obtain a more accurate threshold level.

2. Study of the Corrosion Susceptibility of Hydraulic Actuators

Exposure to a salt spray environment has continued on representative specimens of two different designs of the S-IC stage hydraulic actuators. The actuator fabricated from 7079-T6 has been exposed for 351 days without failure, and the actuator made from 7075-T73 has been exposed for 321 days without failure. Recommendations have been made that all actuators be painted with an epoxy primer and epoxy topcoat in order to reduce the possibility of stress corrosion cracking.

3. Study of Corrosion Inducing Characteristics of Fluoro-Finder FL-50 Penetrant

Tests have been conducted to determine corrosive effects of Fluoro-Finder FL-50 penetrant on 2219-T87 aluminum alloy and 321 stainless steel alloy. This penetrant is produced by Testing Systems, Incorporated. After a 90-day exposure test this penetrant had no corrosive effect on 321 stainless steel. On the aluminum alloy some slight attack was noted after 24 hours exposure, but this did not increase significantly after 90 days. No appreciable corrosion problem should occur if this product is used correctly on 2219-T87 alloy.

C. Investigation of Various Paints and Paint Primers

A search has been initiated for information on fluorescent paints which could be used to paint an American flag on the liquid oxygen tank of S-IC stages. A military specification written for an epoxy zinc rich primer for steels has been reviewed and additional information has been requested from an approved source for the primer. The primer is reported to offer excellent corrosion protection to structural steels in a seacoast environment.

D. Investigation of Fasteners and Fastener Materials

Efforts have continued in the evaluation of cold worked A-286 for use in MC-125 sleeves. An investigation was made on a 1/4-inch diameter tubing assembly which had been torqued to 150 percent of the specified torque value. Examination of this overtorqued assembly revealed that the "B" nuts were deformed, apparently due to overtorque causing the nut to "bottom-out" on the union. This force did not affect adversely the A-286 sleeve which was still characterized by a consistent micro-hardness. The results of this investigation indicate that the A-286 sleeves are acceptable for application at -423°F (-253°C). This material is now being included in the applicable specifications for MC sleeves.

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

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

F. Evaluation of Commercial Adhesives

Studies are continuing as outlined below to evaluate, develop, or qualify new adhesives for use in the Saturn Program.

Investigation of the Effect of Humidity and Cure Cycles on the Bond Strength of Narmco 7343/7139 Adhesives

An attempt was made to determine the effect on bond strength of exposure to atmospheric humidity of aluminum adherends bonded with Narmco 7343/7139 adhesive. The approach was to expose the adherends, coated with adhesive to the humid conditions for periods up to one hour, in increments of fifteen minutes, prior to bonding the specimens. The test conditions were 84°F (29°C) and 57 percent relative humidity. The cure cycle was three days at room temperature plus 6 hours at 140°F (60°C). Test results indicate that bond strength decreased with increase on exposure to the humidity. These tests will be repeated for confirmation.

The accepted cure cycle for 7343/7139 has been 2 days at room temperature followed by 24 hours at 160°F (71°C). Several additional cycles based upon lower temperatures have been evaluated. Preliminary conclusions from these test data indicate that a 24-hour cure at 140°F (60°C) may be superior to a 24-hour, 160°F (71°C) cure. Further evaluation is underway.

G. Development and Evaluation of Potting Compounds

Studies conducted over the past several months have shown that introduction of siloxane groups into the chemical structures of epoxy and urethane polymers provides a means for improving the dielectric properties of the resin systems which are employed as potting and conformal coating compositions. Work is being continued on epoxysilane, urethanesiloxane and urethanesilazane polymers.

1. Epoxysilane Polymers

Approximately 25 grams of 1,4-bis(epoxypropylpropoxydimethylsilyl)-benzene were prepared by the reaction of allylglycidylether with an intermediate described previously, 1,4-bis(hydrogendimethylsilyl)benzene. The process was carried out in ether solution at 25°C in the presence of 0.1 percent by weight of chloroplatinic acid hexahydrate. The diepoxide was obtained in 92 percent yield. Since the compound could not be distilled at 200°C/0.05 torr, it was purified by treatment with activated carbon.

A resilient, tough crosslinked elastomer was produced by curing the diepoxide for 4 hours at 100°C with 10 weight percent of triethylene-tetramine. The dielectric properties of the material are being compared with those of the resin formed by curing diglycidyl ether of Bisphenol A under duplicate conditions.

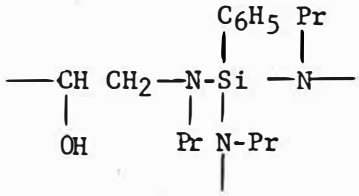
2. Epoxy Polymer Cured with Silazanes

The polymerization of pure Bisphenol A diglycidyl ether with tris(N-propylamino)phenylsilane was described previously as yielding a tough, water white polymer suitable for potting formulations. This was an example of the introduction of the silazane group into the cured epoxy

resin structure via the curing agent, a process which was studied in the previous contract with the Hughes Aircraft Company (NAS8-5499, completed August 1966) and is being studied further in the new contract with Monsanto Research Corporation, (NAS8-20402, initiated June 1966).

The sample mentioned above, which has been stored for about three months, has become soft in the outer portions indicating that some kind of depolymerization or degradation process has occurred in the surface layer. This curing system has been withdrawn from the contract program with Monsanto (NAS8-20402) until the problem has been solved.

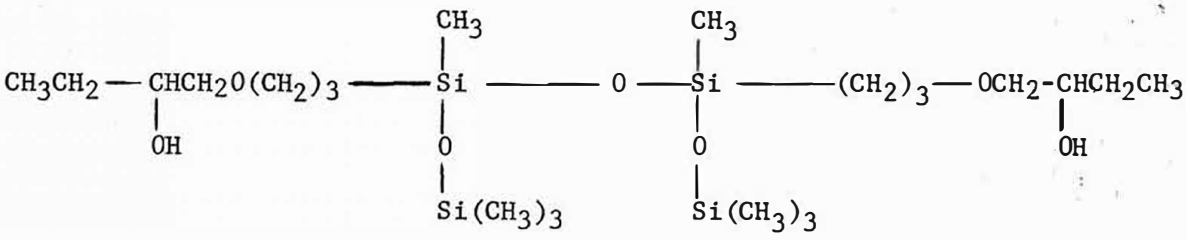
The results of preliminary experiments indicate that the degradation process did not eliminate propylamine from the structure, and further, that the gross product was not affected on standing in water at 25°C for 24 hours or boiling water for 8 hours. It appears that depolymerization or chain scission results from reactivity of the grouping,



Further tests are in progress to identify the agent (possibly water, oxygen or light) which initiates the degradation. A series of trisilazanes will be prepared in which the alkyl group adjacent to the Si-N bond is progressively larger in order to provide the steric shielding necessary to retain stability.

3. Siloxaneurethane Polymers

The siloxane diol described in the July report



has been prepared in approximately a 200 gram quantity by Grignard reduction of the corresponding diepoxide. An attempt was made to extend the chain length of the diol to increase the ratio of polyether to carbamate moieties in the cured polymer. Chain extension of the diol would theoretically increase low temperature resilience, and minimize the number of highly polar carbamate groups which contribute to deleterious dielectric properties. The diol was allowed to condense with bis(N,N-dimethylamino)-dimethylsilane in a 10:1 molar ratio at 25°C. Dimethylamine was evolved

rapidly and the diol approximately doubled in viscosity. Infrared analysis indicated formation of the Si-O bonds, which are expected to be formed in the condensation. Following characterization of the extended diol, isocyanate prepolymers will be prepared employing toluene-2,4-diisocyanate.

4. Polyurethane Conformal Coating

On August 5 we were apprised of serious component failures on printed circuit boards. Astrionics Laboratory has recently utilized glass components having a 10 mil wall thickness. The conformal coatings normally acceptable for these applications have contributed to fracture of the components during thermal cycling. It was agreed that the most immediate solution would be reformulation of the prepolymer and curing agent to give coatings of lower moduli or tensile strength, thus providing more resilience to absorb the thermal cycling stresses.

H. Investigation of Materials for Low Noise, Miniature Slip Rings

The purpose of this project is to develop or locate materials of low wear and noise characteristics for slip rings to operate for extended periods of time at low amplitude oscillations.

A set of experimental slip rings consisting of 24K electroplated gold rings and NEY-ORO 28A brushes was operated at 25 milliamperes brush current and 0.15 degrees DA oscillation. The rings were operated for 200 hours. The noise level stabilized at about 25 microvolts, average, and about 70 microvolts, (peak to peak) on peaks. The position of the noise peaks appeared to correspond to the maximum velocity portion of the driving waveform, which would be expected in the low amplitude displacement region. This test is continuing.

I. 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 ground support equipment (GSE) 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 have been made on commercial additives in the Shell Four Ball wear tester and the Falex lubricant tester. The Shell Four Ball wear test is a relative measure of the lubricating ability of the fluids. The Falex tester is also a relative measure of the lubricating ability of the fluid. The Falex test measures bearing load and resulting wear produced by forces on a rotating pin and set of vee-blocks. A successful test is 3.0 hours in duration with wear measured in the number of loading gear teeth required to maintain the 100 pound load. Results of these screening tests of RJ-1 lubricant additives are being published. Four of these additives have been selected for testing in the RJ-1 pump simulator.

Testing is continuing on the simulated ground support hydraulic pump. The pump consists of nine steel cylinders in a bronze cylindrical housing. These cylinders have swivel bronze shoes which are attached to one end and rest against a swivelling wear plate. This plate is driven by an electric motor at 1,750 rpm. The fluid being tested (RJ-1 or RJ-1 and additive) is forced by a small pressure through the pistons and small openings in the center of the wear shoes forming a lubricating film between the shoe surfaces and the wear plate surface. A static load is applied to the center of the shoes (on a holding plate). This load can be varied manually during the test. The motor torque and static load are measured on load cells and recorded on a Sanborn instrument. Several tests have been made on RJ-1 and RJ-1 with 1000 ppm of Esso additive WS 5412. The results of these tests that the Esso additive, when used in a concentration of 1000 ppm, permits a significant increase in the static load limit which may be applied to the test fixture.

J. 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

Tensile test specimens of PH 14-8Mo stainless steel have been prepared and heat treated to the SRH 950 and SRH 1050 conditions. Evaluation testing of these specimens is in process at cryogenic temperatures.

2. Evaluation of Titanium Alloy 8Al-1Mo-1V Duplex Anneal

Smooth sheet specimens of 8Al-1Mo-1V duplex annealed titanium alloy of 0.050-inch and 0.10-inch thick thickness have been tested at low temperatures. The notched specimens have not been fabricated.

3. Evaluation of Aluminum Alloy 7039

Test specimens for evaluating the -T61 and -T64 tempers of 7039 aluminum alloy have been fabricated in thicknesses ranging from 0.250 inch through 2.0 inches. These specimens will be tested at 27°C (80°F), -196°C (-320°F), and -253°C (-423°F).

II. Contract Research

During this report period, Saturn-related supporting research activities have continued in the fields of technology with the contractors and under contract numbers listed below.

A. Polymer Research, Development, and Testing

1. University of Florida, NAS8-20247
2. Narmco Research and Development, NAS8-5053, NAS8-11958
3. Peninsular Chem Research, Inc., NAS8-5352
4. Midwest Research Institute, NAS8-11338
5. Battelle Memorial Institute, NAS8-11837
6. Bell Aerosystems Company, NASw-1317

B. Development of Cryogenic and High Temperature Insulation Material

1. Goodyear Aerospace Corporation, NAS8-11747
2. IIT Research Institute, NAS8-11333
3. United Aircraft Corporation, NAS8-20089

C. Analytical Methods Development

Beckman Instruments, Incorporated, NAS8-11510

D. Assessment and Evaluation of Blast Hazards

1. Edwards Air Force Base, Government Order H-61465
2. National Bureau of Mines, Government Order H-76708

E. Development of Materials for Special Purpose Electrical Equipment

IIT Research Institute, NAS8-5351

F. Nondestructive Testing Techniques

1. North American Aviation, Incorporated, NAS8-11733
2. R. W. Benson and Associates, Incorporated, NAS8-20208

III. S-II Stage

A. Investigation of S-II-T Rib and Stringer Cracks

Analysis has continued on several cracked ribs and stringers retrieved from the S-II-T stage debris. The principal purpose of this study is to identify any material characteristics which could contribute to the cracking problem. This relatively limited purpose is necessitated by the fact that the S-II-T cracks may have resulted during vehicle failure and, therefore, would not necessarily be representative of those found on flight hardware.

Efforts during this month were concentrated on metallographic and fractographic studies. The fracture surfaces were generally ductile; however, unusual network patterns were observed on all the fractures studied. These patterns could be the result of a brittle sub-grain network; or, according to some fractographers, they could be caused by the presence of a conversion coating. Both possibilities are currently being evaluated.

Metallographic studies revealed no irregularities other than a substantial amount of sub-grains. Positive identification of sub-grains and associated micro-structure is being attempted with thin foil transmission studies. The outcome of these efforts will dictate future work in this area.

B. S-II Stage, Project Management, Materials

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

During the hydrostatic test of S-II-503 cracks were discovered in the fitting welds of the No. 3 and No. 5 liquid hydrogen feed lines. With some difficulty, the cracks were repaired by manual welding. On the basis of a rehydrostatic test and a pneumostatic test to 30 psig the weld repairs were apparently satisfactory. Subsequent to a second pneumostatic test of the stage to 35 psig, a crack reappeared in the No. 5 feed line fitting weld. The stage contractor (S&ID) currently proposes to insert bolts through the fittings in an attempt to mechanically attach the feed line to the mounting fitting. The reappearance of structural cracking problems on repeated pressurization cycles of the stage hardware casts serious doubt on the integrity of the stages particularly since the hydrostatic and pneumostatic tests are intended to establish the acceptability of the hardware.

Subsequent to the aforementioned pneumostatic test to 35 psig, two additional vertical rib ends were found to be cracked by dye penetrant inspection over the chemical conversion coating. Thus, it becomes increasingly evident that S&ID is no closer to an identification of the source of the cracking problem than they were when the cracks were first discovered.

Because of a hydrogen embrittlement problem with the H-11 steel originally specified for the bolts at the S-II/S-IVB interface, it was recommended that A-286 alloy bolts with a tensile strength of 200 ksi be used. There was some concern about the proper bolt diameter, but the Structures Division has decided that 5/16-inch diameter bolts are appropriate for AS-501 through -503 and that 7/16-inch diameter bolts should be used for AS-504 and subsequent vehicles.

The S-II stage contractor recently has informed this Center that the aerodynamic heating of the interstage caused by protuberances during the S-IC boost necessitates the addition of approximately 400 pounds of cork insulation to the exterior of the interstage to keep the structure at temperatures below 250°F (121°C). Also, S&ID has indicated the necessity of adding cork insulation to the interior of the interstage to keep the structure from melting during the dual plan separation sequence. The installation of such cork insulation on S-II-501 would have to be done at the John F. Kennedy Space Center.

IV. S-IVB Stage

A. Developmental Welding

1. Efforts have continued in the development of weld repair procedures for 3/8-inch thick, 2014-T6 aluminum alloy plate. Weldments have been prepared using various combinations of 2319 and 4043 filler wires and automatic and manual techniques. In each weld combination a maximum of ten weld repairs will be evaluated. Many specimens have been prepared for tensile testing and a portion of the metallurgical examination has been completed. Sufficient testing has not been completed to permit development of valid conclusions.

2. In order to more firmly establish the characteristics of 2014-T6 weldments, a study has been initiated into the effects of weld heat input on weld strength. These studies will include metallurgical and mechanical property determinations at specified time periods after the welding operation. Tests will be made to compare welds made with normal weld heat input to welds of excessive heat input. The time intervals for testing are established to determine the effects of natural aging for the different heat input levels. Together with these principal studies a cursory study will be made to determine the effects of successive heat (fusion) passes upon a normal weld.

B. Investigation of the Failure of S-IVB LOX Suction Line Bellows

Three failures of S-IVB LOX suction line bellows during component qualification testing prompted an examination of the unit from the S-IVB Battleship Stage. This component had undergone approximately 5400 seconds of firing time. The qualification units had failed in bellows convolutes with failure attributed to fatigue. The Battleship Stage duct was X-rayed to determine placement of the bellows within the shroud. No shroud-bellows contact was observable in the three bellows sections present in the duct. Dimensional measurements were taken through weep holes in the bellows section that failed in the qualification units. The shroud was then removed from this section and the bellows inspected. No evidences of rubbing or manufacturing defects was observed. It was then discovered that the manufacturer had instituted a configuration change, and that the Battleship unit was not representative of the qualification components. Studies of the Battleship unit will be continued in the event a decision is made to return to this design configuration.

C. Study of Materials Problems Attendant to the S-IVB Workshop Program

1. Materials Testing in Vacuum

The purpose of this project is to determine the vacuum compatibility of materials contained within the liquid hydrogen tank of the S-IVB stage. Materials will be evaluated as to weight loss and the outgassing constituents will be identified for possible toxic products. This project has recently been expanded to include testing in a 5 psia atmosphere.

Tests are continuing on Dynatherm D-65, the flame-retardant coating for the S-IVB insulation, and D-65A, the primer coat for D-65.

Four samples of D-65A primer were prepared by brush coating four aluminum plates 3 inches by 5 inches and air drying for 48 hours. Two of the samples were placed in separate stainless steel test chambers, evacuated to 100 microns and backfilled with oxygen (O_2). This operation was repeated three times with a final filling to 5 psia O_2. The samples were heated to 120°F (49°C) for 24 hours, and then the chamber gas was subjected to gas chromatographic analysis, which indicated only the presence of nitrogen (N_2) and oxygen (O_2).

Samples of D-65 were prepared by spray coating two 0.010 inch layers of D-65 on an aluminum substrate and air drying for 48 hours. One of the samples was placed in a test chamber at 5 psia O_2 and heated to 120°F (49°C) for 48 hours. A second sample was placed in a vacuum of 5×10^{-5} torr for 48 hours prior to being placed in a test chamber with 5 psia O_2 and heated to 120°F (49°C) for 48 hours.

Analysis of the first sample indicated 806 parts per million tetrahydrofuran and 13 parts per million cyclohexanone. Analysis of the second sample indicated less than 10 parts per million of tetrahydrofuran and no trace of cyclohexanone.

Dynatherm is normally a mixture of 80 percent base material and 20 percent solvent. The solvent is a mixture of 90 percent tetrahydrofuran and 10 percent cyclohexanone. The results of the analyses indicate that the coating is not completely cured by the 48-hour air-dry, but that exposure to vacuum for 48 hours completely removes the remaining solvents. These tests definitely indicate that proper curing of this coating will reduce the outgas products below the toxic level.

2. Evaluation of Materials as Sealants for Dynatherm D-65

Four 3 by 5-inch aluminum specimens were coated with D-65 and air dried for approximately two weeks. These specimens were weighed and subjected to 212°F (100°C) for 48 hours. After the heat soak, the specimens were reweighed and revealed a weight loss of approximately 11 percent.

Two of the specimens were spray coated with Kel-F-800 coating from Spraylon Manufacturing Company. These coated samples were air dried for two weeks and then submerged in water for 24 hours along with the uncoated samples. The specimens were removed from the water, blotted, and weighed. The uncoated specimens gained approximately 20 percent of the dried sample weight, whereas the specimens coated with Kel-F gained only one percent. All four specimens lost all adhesion after immersion, thus, in the event that a seal coat is required for the D-65, this Kel-F overcoat would merit further study.

3. Investigation of the Adhesion of Dynatherm D-65

A series of samples was prepared to evaluate the adhesion of Dynatherm D-65 coating that has been proposed as a fire-retardant on the 3D foam insulation of the spent stage S-IVB Workshop.

The first series of samples consisted of 2014 aluminum strips, 2 inches x 10 inches x 0.125 inch which had been acid-etched and coated with Narmco 7343 impregnated glass cloth. Separate specimens were prepared with the exposed Narmco 7343 surface primed as follows before application of the D-65 coating:

a. Unprimed
b. Primed with 10 percent Narmco 7343 in toluene
c. Primed with Goodyear G-207 primer
d. Primed with 3M Company XC-2901 primer
e. Primed with Dynatherm D-65A

Half of the samples were installed in a liquid hydrogen (LH_2) cryostat and were loaded in tension to approximately 100 pounds. The cryostat was filled with LH_2. Within minutes the tension had increased to 2000-2500 pounds. The remaining samples were loaded to 2500 pounds after being installed in the cryostat, the LH_2 was added and after three minutes additional load was applied until metal failure occurred in a pin hole area. This required about 11,000 pounds total load. The samples were visually examined after returning to ambient conditions and no indications of any cracks or other failures were evident. A low power microscopic examination failed to detect micro-cracks.

A second series of test specimens was prepared using the 2 inches x 10 inches x 0.125 inch aluminum strips with pieces of 3D foam material (2 inches x 6 inches) bonded on each side of the center area. The foam was covered with urethane impregnated glass cloth and allowed to cure. Prior to application of the D-65 coating, the impregnated glass surface was primed with the primer systems listed above. These samples were installed in the cryostat, preloaded with 2500 pounds tension, submerged in LH_2, allowed to soak about three minutes and then pulled to failure. The failures were again in a pin-hole area, and occurred at about 11,000 pounds total load. These specimens were examined at ambient conditions and a fine crack was evident at the metal surface between the two foam pieces. This cracking was caused by the tensile loading after liquid hydrogen addition, and there was no indication of any failure between the D-65 coating and the glass reinforced urethane coating on the 3D foam. The conditions used for this test are much more severe than would ever be encountered in flight conditions of the S-IVB vehicle.

Two other test specimens were made by bonding two 3D foam blocks to metal plates of 0.125 inch thickness with Lefkoweld 109 adhesive. The 3D foam blocks were recessed in the center section to completely enclose the metal plates when bonded together. The exterior surface of the samples were covered with glass cloth reinforced polyurethane and one sample was

primed with XC-3901 primer. Dynatherm D-65 was then applied to the two specimens. The samples were placed in the cryostat, LH_2 was added, and the specimens were soaked in the LH_2 for approximately 5 minutes. The specimens were removed, and were examined after warming to room temperature. There was no evidence of cracks, debonding, or failure of any kind at any place on the two samples.

These tests have not shown any indication of failure of the D-65 coating as applied to the glass cloth-urethane seal on the 3D foam. Since there was no failure, advantages that could be attributed to the use of the primers were not evident.

4. Investigation of the Flammability of Materials in Gaseous Oxygen

Activities have continued in the development of a test method for assessing the flammability and compatibility of materials in an atmosphere of oxygen when contacted by an electrically heated wire.

In addition to the above flammability tests, an evaluation was made of various top coats for D-65 materials that would protect it from the water wash now contemplated for the S-IV Spent Stage.

Materials evaluated were (1) 3D foam, (2) 3D foam coated with Dynatherm D-65, (3) 3D foam coated with Dynatherm D-65 and Ram Chemical 5-MJG-3-4 and (4) 3D foam coated with Dynatherm D-65 and Kel-F-800.

The tentative results of these evaluations indicate that Kel-F-800 or Ram Chem. No. 4 can be used as top coats for D-65 with no detrimental flammability problems. Before a top coat is selected, low temperature tests and hypervelocity impact tests will be made to fully establish the acceptability of a top coat. One important observation made from this limited study is the flame retarding property imparted to the 3D insulation (based on these tests) by the D-65. Without the D-65, the 3D insulation ignited and burned completely while the coated samples usually only charred.

5. Simulated Micrometeoroid Tests of S-IVB Materials

Testing and materials evaluation have continued in the study of the effects of simulated micrometeoroid impacts on materials used in the S-IVB Stage.

An additional program was started to study the "ballistic limit" of the 3D insulation. Experiments completed to date indicate that the insulation will burn when punctured with a 3/32-inch diameter aluminum pellet at velocities of 22,700 feet per second. Tests are planned in which an insulation sample 3-4 feet in diameter will be impacted with a hypervelocity particle to ascertain if the generated flame will extinguish or continue to burn across the ribs of the S-IVB Spent Stage.

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. Auxiliary Propulsion System Oxidizer Tankage

a. Testing of two APS (Auxiliary Propulsion Systems) shot-peened tanks has been completed at the Langley Research Center and the third tank of this three-tank program is being returned to the stage contractor for use in the flight program. The two tested tanks were exposed to N_2O_4 at 110°F (43°C) and a skin stress of 90 KIPS. One tank was pressurized to failure after thirty days exposure to these conditions, and the second tank was pressurized to failure after sixty days exposure. Failure of the tanks occurred at 800 psi and 745 psi respectively. The design burst pressure of these tanks is 550 psi, and the maximum skin stress these tanks will experience in service is about 55 KIPS. Thus, shot-peened tanks are considered flightworthy for Saturn V/S-IVB APS applications.

b. Numerous APS tanks of the type originally intended to be used for Saturn V/S-IVB APS applications have failed in the Apollo program, presumably because of stress corrosion. Bell Aerosystems undertook a rather extensive program to solve the problem, and the reported conclusions of this program are that (a) 6Al-4V titanium is satisfactory for APS tank applications, (b) tank failures were caused by N_2O_4 having a low NO content in accordance with requirements of MIL-P-26539A, (c) inhibited N_2O_4 in accordance with MSC-PPD-2 eliminated the stress corrosion problem of tank failures, and (d) shot peening of tanks is not required to eliminate the stress corrosion problem. This division does not agree that shot peening of APS tanks can be eliminated at this time because there are not sufficient data to guarantee structural integrity of unpeened APS tanks for S-IVB applications.

2. Orbital Workshop, Materials

Personnel from this division visited Douglas Aircraft Company, Incorporated (DAC) to review the work which has been done relative to the D-65 overcoating for the internal insulation of S-IVB-209. As a result of this meeting it was determined that there is no evidence of any toxicity problem and that there is no evidence that the D-65 coating will crack and flake off during the life of the stage. Nevertheless, DAC has expressed concern over the use of the material for reasons that remain unclear.

Efforts on the part of Propulsion Division to design mechanical-type seals for the ports and vents in the stage are apparently going to be terminated because of the impracticality of the approach. The sealing

of the stage to create a habitable environment, therefore, is still a completely open item. This division is attempting to identify some materials that might be foamed or cast in place for sealants.

The thermal control coating for the exterior of the stage is still unresolved because of the mutually exclusive "requirements" for low absorbtivity - emissivity coatings and a precise knowledge of the what the absolute values will be in orbit. The problem is caused by the contamination from staging rockets that cannot be simulated accurately in ground tests. This division has and continues to propose certain experiments to illucidate the problem in orbit and is seeking support and financing for these experiments.

3. The following documents were reviewed:

a. Dynatherm D-4327 Application to S-IVB-202 LOX Tank

b. Analysis of Equipment Attachment Fittings, S-IVB Spent Stage

c. Use of HL-20 and HL-21 Hi-LOK Fasteners in the Aft Skirt Separation Ring, S-IVB Vehicle

d. Fluid Specifications

e. Stress Corrosion Susceptibility of Engine Actuators.

V. Instrument Unit

General Corrosion Studies

An Avco Products cold plate which had been filled with inhibited methanol-water solution and pressurized to 50 pounds for six months was examined for corrosion. No visual corrosion was evident.

All general corrosion tests of magnesium lithium alloys LA141 and LAZ933 continue with no appreciable changes. The samples, which are galvanically coupled to magnesium alloys, have not shown as much corrosion as was expected. Some severe corrosion has been noted on several of the samples coupled to the stainless steel alloys. Stress corrosion tests continue in the local atmosphere; however, no additional failures have occurred. Outdoor exposure tests are continuing also on various electroplated coatings on these alloys with no appreciable changes noted during the last 30 days.

ADVANCED RESEARCH AND TECHNOLOGY

I. Contract Research

Supporting research contract activities have continued in the areas of technology and with the contractors as specified below:

A. Polymer Development and Characterization

1. Southern Research Institute, NAS8-20190
2. Hughes Aircraft Company, NAS8-5499
3. Goodyear Aerospace Corporation, NAS8-11070
4. W. R. Grace Company, NASw-924
5. National Bureau of Standards, Government Order H-92120

B. Adhesives Development

1. Narmco Research and Development, NAS8-11068
2. Monsanto Research Corporation, NAS8-11371, NAS8-20402, NAS8-20406

C. Developmental Welding

1. Southwest Research Institute, NAS8-20160
2. The Boeing Company, NAS8-20156

D. Alloy Development

American Machine and Foundry Company, NAS8-11168

E. Physical and Mechanical Metallurgy

1. Aluminum Company of America, NAS8-5452
2. Syracuse University, NAS8-11345
3. Battelle Memorial Institute, NAS8-20029

F. Composite Material Development and Testing

1. Harvey Aluminum, Incorporated, NAS8-11508
2. Aeronca Manufacturing Company, NAS8-5445
3. North American Aviation, Incorporated, NAS8-11108
4. Melpar, Incorporated, NAS8-11322
5. Douglas Aircraft Company, NAS7-429
6. Mitron, Research and Development Corporation, NAS8-20609

G. Lubricants and Lubricity

Midwest Research Institute, NAS8-1540

H. Corrosion in Aluminum and Steel

1. Aluminum Company of America, NAS8-5340, NAS8-11226, NAS8-20396
2. National Bureau of Standards, GO-H2151A
3. Northrop Corporation, NAS8-20333
4. Tyco Laboratories, Inc., NAS8-20297
5. Kaiser Aluminum and Chemical Company, NAS8-20285

I. Explosion Hazards and Sensitivity of Fuels

1. Stanford Research Institute, NAS8-20220
2. Air Reduction Company, NAS8-20078

J. Synergistic Effects of Nuclear Radiation, Vacuum, and Temperature on Materials

1. General Dynamics Corporation, NAS8-18024
2. Hughes Aircraft Company, NAS8-20210

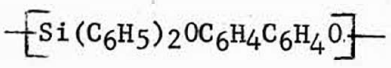
K. Instrument Development

1. Battelle Memorial Institute, NAS8-11891
2. Canadian Commercial Corporation, NAS8-20529

II. General - In-House

A. Development of High Temperature Resistant Polymers

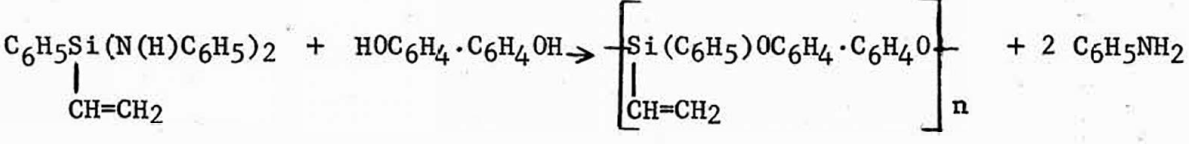
Studies have continued on aryloxysilane polymer structures, particularly Polymer A,



Several materials of closely related structure have been found to possess favorable combinations of properties, especially stability at relatively high temperatures. The current work includes synthesis of new structures, appraisal of crosslinking agents and procedures, and further studies of the physical properties of some of the products previously described.

1. Crosslinking Studies

In order to introduce vinyl side chains into a structure of the Polymer A type, dianilinophenylvinylsilane was prepared by the reaction of aniline and phenylvinyldichlorosilane under conditions previously described. The product, boiling at 201°C (394°F), 1.05 torr was obtained in 76 percent yield, and was judged by gas chromatographic analysis to be 98 percent pure. The polymer was produced by melt condensation (0.1 torr, 240°C (464°F), 6 hours) of the dianilinophenylvinylsilane with p,p'-biphenol:



The product softened at 72-77°C (162-171°F). After curing with two percent by weight of azobisisobutyronitrile at 232°C (450°F) during 15 hours, the softening point was increased to 200°C (329°F), indicating that crosslinking had occurred. Differential thermal analysis (DTA) revealed that the polymer was stable to rapid heating (10°C/min (18°F/min)) in nitrogen to 425°C (797°F).

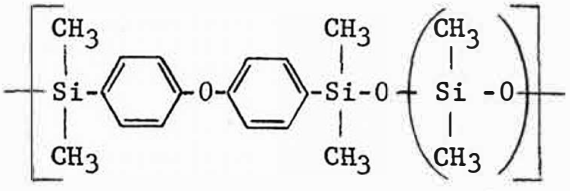
Copolymerization of the polymer possessing the vinyl side groups with styrene in the presence of peroxide yielded a thermoplastic material softening at 86-92°C (187-198°F) and thermally stable in the DTA measurement to 500°C (932°F). Thus, it appears that the styrene copolymerized with the vinyl branches to form longer branches without crosslinking. Surprisingly, the styrene did not appear to adversely affect the thermal resistance and actually may have improved its short time thermal stability.

Activities have continued in the development of means of testing the effectiveness of curing agents for Polymer A compositions filled with glass cloth. The flexural strength of the laminates is determined by use of a modification of the ASTM D790-63 Flexural Test device. The first results obtained at room temperature indicate that Polymer A, cured with trianilinophenylsilane, has high adhesion for glass fiber, and values of flexural strength are obtained that are comparable to those of epoxy formulations.

2. Investigation of Physical Properties

The results of measurements of the dielectric properties of an early sample of Polymer A indicate that the material may compare favorably with the best dielectric polymers now available, namely, Teflon and polyethylene. While these results are very promising, a definitive conclusion must await further evaluation of new samples which possess improved mechanical properties and which are formed with more appropriate molding equipment.

A sample of alkylaryloxysilane polymer,



has been subjected to thermal stability tests by differential thermal analysis (DTA) and differential scanning calorimetry (DSC). By DTA it was found that the material was stable to rapid heating (10°C/min (18°F/min)) in an atmosphere of nitrogen to 500°C (932°F). By DSC it was observed that T_g (the temperature at which the material changes from a rubber to a brittle, glass-like, material is 60°C (140°F). These data indicate that the polymer exhibits a remarkable combination of properties.

Through an informal cooperative arrangement between Midwest Research Institute, Rock Island Arsenal, and George C. Marshall Space Flight Center, personnel of the Rock Island Arsenal rubber group attempted to vulcanize the alkylaryloxysilane polymer. The trials with the peroxide systems normally employed for curing dimethylsilicone elastomers were unsuccessful. It appears that utility of this interesting polymer will depend on the development of an effective curing system.

B. Development and Characterization of Phosphonitrilic Polymer

The projected synthesis of substituted phenylphosphonitrilic chloride tetramers was continued with the completion of the study of the reaction of anhydrous HCl with 2,4,6,8-tetraphenyl-2,4,6,8-tetrakisdimethylaminophosphonitrile.

$$P_4N_4Ø_4(N(CH_3)_2)_4 + 8\ HCl \longrightarrow (ØPNCl)_4 + 4\ DMA\cdot HCl.$$

The oily product obtained from the reaction, after separation of amine hydrochloride salt, could not be induced to crystallize. Dissolution of the oil in glacial acetic acid followed by additional treatment of the resulting solution with anhydrous HCl failed to effect separation of any further amounts of amine salt. Examination of the IR spectrum of the oily product isolated from the reaction revealed the presence of a weak absorption band at 980 cm^{-1} which indicated that the product was still contaminated with unregenerated amide. Although the experiment could not be termed a complete success by any means, the techniques to be used in arylation of the PN ring were firmly developed for subsequent reactions.

Therefore, emphasis was reshifted to the preparation of a non-geminally substituted $(RPNCl_2)_4$, where $R = NR'_2$, isomer which could be arylated via the Grignard reaction to give a mixture of isomers capable of subsequent replacement of the NR'_2 groups by halogen atoms that the reaction of $(PNCl_2)_4$ with 8 equivalents of dimethylamine gave atbest a 50 percent yield of product which contained 1-3 percent of geminally substituted isomers.

To this end the reaction between $(PNCl_2)_4$ and N-methylcyclohexylamine was reinvestigated. The choice of this particular amine was based on the report in the literature (K. John, et al., J. Am. Chem. Soc., 83, 2608 (1961)) that reaction of $(PNCl_2)_4$ with excess N-methylaniline gave a tetrasubstituted derivative in which one amine group is bonded to each phosphorous atom. The presence of but a single line in the nuclear magnetic resonance (N.M.R.) spectrum of the compound (H_3PO_4 as reference) suggests only one phosphorous environment for the molecule. The completely reduced derivative of the N-methylaniline was used for this study because of the greatly increased basicity and concomittant shorter reaction times. It was also assumed that there was no difference in the size of the two amine molecules and hence the same steric factors would dictate the formation of only non-geminally substituted derivatives when using N-methylcyclohexylamine.

In a typical run 0.4 mole N-methylcyclohexylamine in 250 milliliters (ml) of tetrahydrofuran (THF) was cooled to -10°C (-14°F) and maintained at this temperature with stirring while treated dropwise with a solution of $(PNCl_2)_4$ 0.05 mole) in 125 ml. of THF. After stirring for 24 hours at room temperature the separated amine salt was removed by filtration (95 percent yield). The filtrate was concentrated in vacuo to an oil which solidified upon trituration with acetonitrile, (93 percent yield), melting range 115-117°C (239-243°F). An analytical sample was recrystallized

from acetonitrile-benzene, melting range 117-118°C (243°F -244°F). The narrow melting point range suggests the presence of only a single isomer. Elemental analyses agreed closely with the theoretical values.

The arylation reaction with the Grignard reagent was carried out as shown below:

$$P_4N_4Cl_4(N(CH_3)\ (C_6H_{11}))_4 + 4\ \emptyset MgBr \longrightarrow P_4N_4\emptyset_4(N(CH_3)(C_6H_{11}))_4 + 4\ MgBrCl.$$

A dibutyl ether solution of the amide derivative (0.03 mole) was heated to 100°C (212°F) and filtered to remove a trace of insoluble material. The filtrate was maintained at 100°C (212°F) and treated dropwise with stirring with a dibutyl ether solution of ØMgBr (0.12 mole). After stirring for two hours at this temperature, the resulting mixture was filtered and the filtrate concentrated in vacuo. The resulting oil could not be induced to solidify. The IR spectrum of the oil indicated that phenyl substitution had occurred and that displacement of the amine groups had not taken place. The weight of the oil represented a 96 percent yield - the weight of the MgBrCl indicated quantitative conversion to the tetraphenyl derivative. Characterization of the oil was limited to infrared spectral data.

Regeneration of the arylated tetra substituted amide was attempted in the following manner:

$$P_4N_4\emptyset_4(N(CH_3)(C_6H_{11}))_4 + 8\ HCl \longrightarrow (\emptyset PNCl)_4 + 4\ \text{amine salt}.$$

The oily mixture of isomers was dissolved in chlorobenzene, the resulting solution heated to reflux (132°C (270°F)) and maintained at this temperature while treated with anhydrous HCl for two hours. At the end of this time the mixture was cooled and the solid which had separated removed by filtration (13 g., 70 percent).

The filtrate was concentrated in vacuo after having been washed with water to remove any residual amine salt and dried over anhydrous magnesium sulfate. The residual oil was triturated with cyclohexane and the resulting solid separated by filtration. The product is soluble in benzene, carbon tetrachloride and insoluble in petroleum ether. The infrared spectrum (CCl_4) of the solid shows tetrameric PN ring absorption and the near complete disappearance of the PNR_2 absorption band at 980 cm^{-1} indicating displacement of this group by halogen atoms. If this compound is shown to be a $(\emptyset PNCl)_4$ isomer, then a new route to the preparation of tetrameric arylsubstituted phosphonitrilic chlorides has been developed.

C. Investigation of Materials for Electrical Contacts in Vacuum

Development work and qualification testing have continued on low resistivity brush materials for possible application in the environment of space.

As reported earlier, the niobium diselenide ($NbSe_2$) based brush materials appear more promising for the projected applications than the previously developed molybdenum disulfide (MoS_2) based materials. Initial tests have shown that single phase $NbSe_2$ brushes have low wear rates and minimal commutator damage; however, the $NbSe_2$ brushes have a relatively high coefficient of friction and a susceptibility to chipping and/or splitting along the planes of preferred crystallite orientation. At present, attempts are being made to overcome these deficiencies by the use of selected additives, such as MoS_2, silver, graphite fibers, and the diselenides of tungsten, tantalum, and molybdenum.

The $NbSe_2$-MoS_2 system yields materials of adequate strength and electrical conductivity in the composition range of 0-40 volume percent MoS_2. However, these materials have not demonstrated properties superior to single-phase $NbSe_2$. The system $NbSe_2$-tantalum diselenide ($TaSe_2$) also yields materials with adequate mechanical and electrical properties but, again, no improvement in brush operating characteristics has been noted.

A single sample of $NbSe_2$-silver (Ag) has been hot pressed and tested in the D.C. motor-generator set. This material did not chip during the initial test, so this system is believed to merit further study.

Tests are being set up to check out the coefficient of friction in vacuum of the following materials: 100 percent $NbSe_2$, 85 MoS_2-15 Ag, 60 $NbSe_2$-40 MoS_2, 80 $NbSe_2$-20 MoS_2, and 80 $NbSe_2$-20 TaS_2. In addition the following materials have been prepared and will be tested: 60 $NbSe_2$-40 $TaSe_2$, 70 $NbSe_2$-30 $TaSe_2$, 90 $NbSe_2$-10 Ag and 90 $NbSe_2$-10 Ag.

Further measurements are being made to fully characterize the electrical properties of the hot-pressed brush materials. Determinations were made of the resistivity as a function of temperature of four samples of 2.5 micron MoS_2-Ag and one sample of 4 micron MoS_2-Ag. All of the materials tested were characterized by metallic conduction and one sample consisting of 70 percent 4 micron MoS_2-30 percent silver had a resistivity at 100°K of 8.1×10^{-6} ohm-cm.

Determinations were made of the Seebeck coefficient for 22 materials of varying composition, pressing temperature and pressing pressure. In general the 4 micron MoS_2 indicated a lower and unchanging thermoelectric voltage while the 2.5 micron MoS_2 was higher and varied as a function of composition. Pure 2.5 micron MoS_2 had a Seebeck voltage that was positive, which would be expected of a P-type semiconductor. As the percent silver is increased, the Seebeck voltage becomes negative, indicating N-type doping, which agrees with the postulated conduction mechanism of these materials. However, Seebeck voltage for 100 percent 4 micro MoS_2 was also positive, and larger than the 2.5 micron MoS_2 value, which was not expected. These results are still being analyzed.

D. Investigation of the Dielectric Properties of Materials

1. Flat Cable Tests

Because Mylar flat cables currently are used in launch vehicles and spacecraft, a study has been made to determine the changes in electrical properties produced by exposure to the environment of space. Mylar flat cables were exposed to ultraviolet radiation both at an environmental pressure of 10^{-9} torr and in air. Periodic measurements were made of the dielectric constant, dissipation factor, and conductivity throughout the run. The experimental data indicate that under the conditions of the test and for the exposure times used Mylar flat cable is not adversely affected by ultraviolet radiation in air or in vacuum.

2. Teflon Tubing and RJ-1 Fuel

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 tubing and additives to the hydraulic fluid. 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.

Tests were made to determine the relative effectiveness of Shell ASA-3 and Esso WS-3966 anti-static (conductivity) additives when added to RJ-1 in various concentrations. Both additives increased the conductivity of RJ-1, exponentially, producing the greatest increase in conductivity per unit mass of additive at the smaller concentrations, up to 300 ppm, and producing less improvement as the concentration approached 1000 ppm. However, the ASA-3 produced a conductivity about one order of magnitude higher than the WS-3996 for all concentrations.

The voltage gradient was varied also for tests on both ASA-3 and WS 3996. Over a wide range of voltage gradients no particular variation was noted in conductivity, although values were more uniform at higher gradients.

E. Investigation of Nuclear Environmental Effects on Materials

1. Study of the Effect of Charged-Particle Irradiation of Polymers

A study has been initiated into the effects of charged-particle radiation on the mechanical properties of polymeric materials. Much data have been obtained on the effects of gamma radiation but very little information is available on charged-particle effects. The ultimate goal of this program is to obtain sufficient data to determine whether a correlation exists between radiation damage produced by electron and gamma radiations.

Ninety-six specimens of thin film materials were tested to determine changes in ultimate tensile strength and elongation produced by irradiation with one Mev electrons. Standard tensile specimens of Mylar (2 mil), Teflon (5 mil), H-Film (2 mil), and Tedlar (2 mil) were irradiated to fluences of approximately 10^{14}, 10^{15}, and 10^{16} electrons cm^{-2} and then analyzed with an Instron tensile tester.

It has been shown that H-film is less affected by neutron or gamma ray bombardment than Teflon, Mylar or Tedlar. However, under electron radiation both Mylar and Tedlar retained significant strength at a fluence of 1×10^{16} whereas H-film and Teflon suffered total degradation.

2. Vacuum Tensile Tests

A program has been initiated to determine in-situ the effects on the tensile properties of materials after prolonged exposure to vacuum and temperature. It is necessary that the vacuum-temperature effects on the tensile properties be isolated in order to determine the effects of radiation on the tensile properties of materials in a space simulated pressure-temperature environment.

Mechanical property tests were made in vacuum on 27 specimens of Mylar and 45 specimens of Tedlar after exposure to reduced pressures of 3×10^{-7} to 5×10^{-8} torr for periods of 24, 48, and 96 hours. Two thicknesses of each material were evaluated to determine if specimens of different thicknesses behave the same in a vacuum environment. The data from these tests are being evaluated.

F. Study of the Compatibility of Lubricants with MIL-H-5606 Hydraulic Fluid

Tests are continuing to determine if Dow Corning DC-4 (silicone compound) lubricant and technical petrolatum (VV-P-236) are compatible with hydraulic fluid. A testing device was designed and fabricated whereby four samples of the same lubricant could be tested at one time resulting in more reliable data. This is obtained by fastening 4 screened cups each containing approximately 4 grams of the lubricant to a four spoke wheel. All four cups are submerged in the hydraulic fluid and the 6-inch diameter wheel is rotated by a directly coupled 1 rpm motor. The hydraulic fluid can be heated by placing the container on an electric hot-plate. The samples are carefully weighed before and after test to check the weight loss or weight gain. Three tests have been run with DC-4 for one hour and 45 minutes duration each at room temperature. The test data show an average increase in weight of 7 percent possibly indicating some adsorption of hydraulic fluid. Three tests were also run with DC-4 for one hour and 45 minutes duration each at 128°F (53°C). These test data also showed an increase in weight of approximately 8 percent.

G. Development of Direct Current Motors for Use in the Environment of Space

Materials are being evaluated at extreme temperatures and low pressures for use in direct current (d.c.) motors designed for operation in the space environment.

During the reporting period one test was made with the motor generator set. This test was made with the field currents set at 1.5 amperes, and motor armature at 40 volts giving a speed from 650 to 700 rpm. Brushes and bearing retainers employed in this test were niobium diselenide-silver (90 percent - 10 percent) and polyimide silver respectively. The longest continuous operating period during the test was 7 hours with accumulated test time of 21 hours. Heavy sparking was observed at the commutator brush interface during the first 5 hours of operation. The test was then stopped, heat applied to the motor overnight and then restarted the following day. The set was operated at elevated temperatures for 7 hours during which time only light sparking was observed. The next 7-hour test period was begun with heat applied to the system as the test was begun. Again light sparking occurred. The final test period was a 2-hour test in air at ambient temperature. This test was stopped when a ring scar appeared on the commutator.

During vacuum testing no chipping was observed on the brushes. However, the brushes did chip as they were forcefully removed from their holders at the conclusion of the test. Since the brushes remained intact during testing, it would indicate that the silver has added to the strength of the brush. Future testing will include brushes of various percentages of niobium diselenide and silver with the aim of obtaining an optimum composition of niobium diselenide-silver brush.

H. Determination of Physical Properties of Materials by Nondestructive Techniques

1. Internal Friction Measurements

The internal friction of metals is being investigated to determine if this property is sensitive to the microstructural changes produced by the stress corrosion process.

Internal friction measurements have been made on fifty specimens of 7079-T651 aluminum alloy in air and in a vacuum. Both 1/8-inch and 3/16-inch diameter specimens machined in the short transverse or longitudinal directions were tested. The greatest energy losses were obtained when the short transverse specimens were subjected to both stress and the salt solution, whereas only small losses were measured when the specimens were exposed to the corrosive environment alone.

2. Electrical Conductivity

Electrical conductivity studies are being made to determine whether surface conductivity changes can be used to indicate the progress of the stress corrosion process in aluminum.

Recent efforts have been directed toward the measurement of conductivity using a high frequency eddy current instrument. Several specimens which had been exposed to stress corrosion were tested in the frequency range of 100 KHz to 6 MHz with the result that large conductivity changes were noted. Further measurements are being made to define the relationship between high frequency conductivity and time of exposure to stress corrosion.

I. Study of Materials Problems Associated with Advanced Manned Missions

Apollo Telescope Mount

Vacuum compatibility tests were made on two materials which are being considered for the Apollo Telescope Mount, and for the Electromagnetic Radiation Experiment.

It was reported that spectral requirements for the cameras for both these applications will require the camera film to be exposed to vacuum. Samples of Kodak TRI-X panchromatic film were exposed to a pressure of 10^{-8} torr for 48 hours to determine if there was any embrittlement of the emulsion backing, or any substantial change in the gelatinous portion of the emulsion. Visual examination revealed only a slight change in the stiffness of the backing.

Another sample of Kodak TRI-X was subjected to a weight loss determination at 10^{-6} torr up to 310°F (154°C). The major portion of the weight loss, 1.4 percent, occurred during pumpdown and may be attributed to loss of water vapor. Temperature to 310°F (154°C) over the next 19 hours yielded only 1 percent additional weight loss, and indicates the material is quite stable. However, no attempt was made to determine any possible vacuum effects on the sensitivity of the film.

The second material tested was a 90 percent bismuth powder, 10 percent epoxy bonded solid proposed as a shield material for the gamma ray spectrometer experiment. The sample was evacuated to 2×10^{-5} torr at 82°F (28°C) for 2.5 hours and then heated to 320°F (160°C) for 3 hours. No weight loss (0.05 milligram sensitivity) was noted.

J. Study of Material for the Nuclear Ground Test Module

In-house and contractual studies are being pursued to develop the materials technology required to support the Nuclear Ground Test Module (GTM) Program. Specifically, the areas of cryogenic insulation, valve seals, transducer materials, gimbal and bearing lubricants, and induced neutron activation are being actively investigated.

A contract (NAS8-18024) has been initiated with the General Dynamics Corporation, Forth Worth, Texas to evaluate the effects of radiation and cryogenic temperature on the mechanical properties of selected cryogenic insulations, adhesives, and vapor barriers. In addition, the structural integrity of two insulation systems will be determined after exposure to acoustic, cryogenic temperature, and radiation stresses.

Work has been initiated to determine the coefficient of thermal conductivity from room temperature to 20°F (-7°C) for a specimen of laminated corkboard which is a candidate insulation system for the GTM. In addition, the coefficient of linear thermal expansion is being determine at cryogenic temperatures for corkboard. These data will be used in the analysis of the thermal and structural performance of this insulation system.

Currently, efforts are being made to obtain drawings of the cryogenic valves and transducers which conceivably could be used on the GTM. When these drawings become available, they will be analyzed to determine the types of materials involved. Recommendations then will be made as to the acceptability of these materials for use in a radiation environment and, when appropriate, replacement materials will be specified. Qualification testing of these components will be conducted under contract NAS8-18024 and will be coordinated with personnel of the Quality and Reliability Assurance Laboratory and the Propulsion Division of this laboratory.

K. Documentation Review

The following specifications, documents, or reports were reviewed and comments were forwarded, where appropriate, to responsible individuals or organizations:

1. Douglas Process Specification, DPS-32333, "Common Bulkhead Aft Toe Weld Doubler Installation"

2. Douglas Process Specification, DPS-32332, "Jamb Weld Doubler Installation, LOX and LH_2 Tank Assemblies"

3. Douglas Process Specification - -41006, "Aluminum Alloys, Preparation for Welding"

4. Douglas Process Specification DPS 14052, "Mechanical MIG-GMA and TIG Fusion Welding Aluminum, Saturn Assemblies"

5. S&ID SPEC-MB00170-040, "Inconel 718 Alloy Sheet, Strip, and Plate: Consumable Electrode, Vacuum Melted, Solution Annealed at 1950°F"

6. S&ID SPEC-MB0170-012, "Inconel 718 Alloy Bar, Forgings and Forgings Stock: Consumable Electrode, Vacuum Melted, Solution Annealed at 1800°F"

7. S&ID SPEC-MB0170-041, "Inconel 718 Alloy Bar, Forging and Forging Stock, Consumable Electrode, Vacuum Melted, Solution Annealed at 1950°F"

8. MSFC-PROC-195A, dated 8-10-66, "Cleanliness Level Requirements and Inspection Methods for Determining Cleanliness Level of Gas Bearing Gas Supply and Slosh Measuring System"

L. 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. Lubricants and Lubricity
4. High and low temperature resistant polymers
5. Stress corrosion on structural alloys.

J. E. Kingsbury

MONTHLY PRODUCTION REPORT
MATERIALS DIVISION
AUGUST 1, 1966 THROUGH AUGUST 31, 1966

I. Radiographic Inspection

One hundred and fifty miscellaneous parts, components and test specimens were inspected radiographically during this report period.

II. Photography

	Negatives	Prints	Other
Engineering Photography	124	476	
Metallography and Fractography	123	776	
Miscellaneous Photography Processing, Copywork, etc.			31

III. Metallurgical and Metallographic Testing and Support Services

A. Analysis of the failure of an S-II actuator component has been completed. This work was initiated at the request of Astrionics Laboratory. The anchor end of the actuator failed after eight months of intermittent operation at the Astrionics Laboratory. Although the actual number of cycles experienced by the actuator is not known, it is believed by Astrionics personnel that the component had undergone several thousand more cycles than the design limit. "Beach markings" on the fractured surface identified the mode of failure as fatigue, and this finding was verified as high cycle fatigue with the aid of the electron microscope.

B. The weld joint design and weld procedures for fabrication of component parts for the Instrument Unit package were evaluated.

C. A study was made on the joining of tin-plated nickel-200 alloy leads of a capacitor bank as part of the Instrument Unit package.

D. Aluminum alloy 1100 was mechanically and thermally processed to produce annealed stock of various thicknesses, 0.010 to 0.014 inch, for ME Laboratory for a S-IVB application.

E. Assistance was provided to R-AERO for materials selection for torsion arm assembly to determine angle of attack - dynamic aerodynamic damping relationships. A maraging steel was recommended on the basis of a requirement for a material of high torsion modulus, high yield strength, and high endurance limit.

F. The Boeing Engineering Change Procedure ECP0088, "Replacement of Fasteners on Flow Lines for Fuel Prevalves and Fuel Emergency Drain Valves," was reviewed. The fastener which is under consideration for replacement is the 137148-1 locknut, specified as 50FA-420 by the Standard Pressed Steel Company. A review of the components indicated that the specified 303 stainless steel should be cold worked rather than annealed, and that the Standard Pressed Steel locknut was adequate for the application.

IV. Spectrographic Analysis

Eight hundred and sixty-two spectrographic determinations were made on forty-one samples and six hundred and forty-seven standard determinations were made.

V. Infrared Analyses

Samples of thirteen experimental polymers, four lubricants or lubricant additives and seven miscellaneous materials were analyzed qualitatively by infrared techniques during this report period.

VI. Chemical Analyses

	Determinations
methanol-water mixture for	
sodium benzoate	11
water samples for	
heravalent chromium	6
RP-1 fuel samples for	
moisture	10
water reaction	4
RJ-1 fuel additive for	
carbon	3
hydrogen	3
phosphorus	3
sulfur	3
experimental polymers for	
carbon	44
hydrogen	44
silicon	10
nitrogen	14
chlorine	4
gas samples for	
oxygen	37
nitrogen	34
helium	8
hydrogen	14
carbon monoxide	2
moisture	7
atmospheric contamination specimens for	
acetic acid	2
fluorides	4
chromatographic analyses	94

VII. Physico Chemical Analyses

	Determinations
density of RP-1 fuel	26
pH of methanol-water mixture	11
specific resistance of methanol water mixture	11
heat of combustion of RP-1 fuel	15
flash point of	8
RP-1 fuel	8
gear box oil	6
distillation range of RP-1	4
fire point of gear box oil	2

VIII. Rubber and Plastics

	Items
molded and extruded	216
cemented	14
potted	16
coated	38
fabricated	10

IX. Electroplating and Surface Treatment

	Items
cleaned	57
salt spray tested	18
electroplated	46

X. Development Shop Production

A. A total of 4,462 man-hours, direct labor, was utilized during this period for machining, fabrication, and welding.

B. Eight hundred and eighty-five man-hours, approximately 19.8 percent of the total man-hours, were devoted to productive effort of a non-routine nature and applied to the work orders listed below.

1. 3000 psi Injector Body

Components for the 3000 psi injector body are ready for assembly and final machining.

2. Chamber Section-Hydro Coil Lined

One hydro coil-lined chamber section is near completion and another is being modified.

3. Diaphragm dies

Dies for fabrication of experimental diaphragms are complete and delivered.

4. S-IVB Fuel Tank Motion Study Components

Components for the S-IVB fuel tank motion study are complete and delivered.

5. Thermal Sensor Experiment Assembly

The thermal sensor assembly has been completed.

6. Motor Torque Gauge and Brace

Fabrication of the components for the motor torque and brace is near completion.

XI. Miscellaneous

A. Sixteen aluminum alloy items, two hundred items of stainless steel, twenty-five items of nickel alloy and one piece of copper were heat treated during this report period.

B. Applied MLF-I lubricant to twelve S-IC test stand bearing plates.

C. Thirty-two silicone rubber seals for flat cable conductors were molded for Astrionics Laboratory. These seals will be evaluated as possible replacements for Neoprene seals which have proved to be unsatisfactory for use at the temperatures encountered.

D. Spray coated three stainless steel plates 20 inches x 50 inches with Emralon 310 Teflon dispersion for Computation Laboratory. These plates are used to support the tilted lunar map for the SMK-23 simulator. Without this coating on the backup plates, vibration in the map causes the MOLAB simulator to appear to jump 4 to 5 feet off of the surface. The coating reduces the friction and eliminates this problem.

E. Eight each thermogravimetric analyses and differential scanning calorimetry tests and fifty-three differential thermal analyses were made during this report period.

XII. Publications Issued

A. "An Integrating-Sphere Reflectometer for the Determination of Absolute Hemispherical Spectral Reflectance," Gene A. Zerlaut and A. C. Krupnick, AIAA Journal, 4, No. 7, 1227-1232, July 1966.

B. "Automatic High-Pressure Gas-Sampling System for Gas Chromatography," by W. M. Langdon, V. R. Ivanuski, R. E. Putscher, H. J. O'Neill, and A. C. Krupnick, Journal of Gas Chromatography, 269-270, July 1966.

C. Morgan, W. R.: Low Temperature Mechanical Properties of Berylco Nickel 440 Strip, IN-P&VE-M-66-4, August 2, 1966.

J. E. Kingsbury

GEORGE C. MARSHALL SPACE FLIGHT CENTER

PR-P&VE-S-66-8

MONTHLY PROGRESS REPORT

STRUCTURES DIVISION

(August 1, 1966 - August 31, 1966)

SATURN IB

I. S-IVB Stage

S-IVB Panel Flutter Test

The pressure survey model, for the first phase of the S-IVB stage panel flutter test, has been delivered to the AEDC wind tunnel facility. Model installation and check-out are underway to meet the scheduled test date of September 19, 1966.

The necessary S-IVB skirt segment modification and test box fabrication for the second phase of the S-IVB stage panel flutter test has been completed by the Manufacturing Engineering Laboratory. Preparations are being made for the loads test of the skirt specimen. The wind tunnel test is now scheduled for the week of October 3, 1966.

II. General

A. AS-206 Nose Cap Ejection Assemblies

Twenty-two nose cap ejection assemblies were calibrated and prepared for shipment for use on the AS-206 vehicle. The spring rate for these assemblies was in the 430 #/in range instead of the desired 480 #/in range; however, these rates are acceptable. All the springs (170) were calibrated to select 22 assemblies with optimum spring rates. From the remaining 148 springs, calibrated assemblies will be prepared for the AS-208 and AS-210 vehicles.

B. Real Time Bending Moment Display

A real time Uprated Saturn I vehicle bending moment display at MSFC was requested in support of the MSC command module tension tie problem. The bending moment will be displayed during prelaunch for vehicles AS-202

through AS-212. The display system has been implemented at Computation Laboratory.

SATURN V

I. S-IC Stage

Structural Qualification Test

All major structure components of the S-IC stage SA-501 - 503 configuration, have been fully qualified for man rating (Safety factor of 1.4 on design loads) by the successful completion of the oxidizer tank assembly test, July 29, 1966. Instrumentation data showed that the specimen sustained stresses in the ultimate range (50,000 PSI). Inelastic buckling appeared imminent in the forward skirt and in the S-II interstage. The forward skirt had no permanent deformation and can be used for the S-IC, S-II interface test. Load peaking at the S-IC, S-II interface was not as severe as calculated by the contractors.

The oxidizer tank assembly has been removed from the Load Test Annex and the short lox tank, which contains the sculptured Y-ring, installed.

II. S-II Stage

A. LH_2 Tank Cracking Problem

S&ID presented to MSFC at a meeting on August 18, 1966, the findings regarding the cause of the extensive numbers of cracks which are being found in every S-II stage. While it was evident that a great amount of effort had been expended by S&ID, it appeared that the cause of the problem is still very nebulous.

An important decision reached in the meeting was the concession by S&ID that the CBTT would be used to test the mechanically fastened doubler design to ultimate pressure. S&ID was reluctant to subject the CBTT to ultimate pressure since the required pressure will exceed the previous test pressures employed in the original CBTT program.

B. Establishment of Allowable Pressure Cycling for S-II-F and S-II-1

On August 19, 1966, a meeting was held between P&VE and S&ID to establish the allowable pressure cycling for S-II-F and S-II-1. Since firm technical data was not available, the following decisions represented the best subjective opinions:

1. S-II-F:

   The LH_2 tank may be pressurized at KSC a total of 36 times,

no more than 6 of these may exceed 10 psig. If this limit is reached the tank must be inspected.

2. S-II-1:

The LH_2 tank may be pressurized at MTF a total of 80 cycles; however, no pressure cycles are allowed above 10 psi before verification of the patch. Inspection will be required if the above limits are reached.

C. Debonding of Adhesive Patches on S-II-3

The pneumostatic pressure proof test of the S-II-3 stage was aborted at 30 psig during a planned pressurization to 35 psig. This decision was made from an evaluation of the strain data which indicated that the bonded patches at S&ID station 722 were becoming debonded. Subsequent ultrasonic inspection confirmed that the patches were debonding. All bonded patches on the S-II-3 have been removed and mechanical doubler repairs are planned as replacements.

D. Bolted Patch Concept

S&ID and P&VE have agreed that it is feasible to proceed with installation of the bolted patch design on S-II-1 and S-II-3 even though detailed analysis and qualifying test data is not now available. It is the opinion of Structures Division that the design is reasonable enough to justify the risk of proceeding with rework prior to getting substantiating data. This concept is to apply only the external doubler plus the foil seal without using the "strong-back" ring frame reinforcement. This approach appears possible since a reduced factor of safety is applicable and since the skin thickness is greater than required.

E. Rehydrostat of the S-II-4 Forward Bulkhead/#6 Cylinder Assembly

The rehydrostat test of the S-II-4 forward bulkhead, #6 cylinder assembly was performed successfully on August 23, 1966, to the required proof pressures. The bolted doubler repair with the ring frame beef-up ("strong-back") was incorporated, including extensive strain instrumentation. A preliminary evaluation shows that the ring frame beef-up only reduced the load at the patch area by 10%. Further evaluation is continuing.

F. Interface Hole Problem: S-II-2

An inspection of the bolt holes in the S-II mating flange of the S-II/S-IVB interface shows that practically all of the holes have been elongated or otherwise damaged. The exact cause has not been established. Timely action will be required in the disposition of this problem since the mating S-IVB stage may also be impacted. Evaluation of the problem is proceeding.

G. Vibration Specifications

As a result of S-II-T firing data, revised aft skirt vibration specifications were generated. These specifications will apply to any components which have not previously been qualified to the original criteria. Also, as a result of S-II-T and S-IC-T firing data, revised thrust structure vibration specifications were generated. These specifications will be utilized in any future thrust structure component qualification program.

H. High Force Test Program

Acoustic testing of the aft interstage/thrust complex of the S-II stage at Wyle Laboratories has been completed. Failures occured on mounting brackets for the 3 batteries in the aft interstage. The specimens have been removed from the chamber and are being prepared for mechanical vibration testing.

III. S-IVB Stage

High Force Test Program

The S-IVB high force dynamic test program has been completed at Thiokol, Wasatch, Utah. There were no failures as a result of the test program and preliminary data evaluation indicates that the stage is structurally adequate to withstand anticipated dynamic environments.

IV. Instrument Unit

200/500S-3 Instrument Unit

The cold plates that were removed from 200/500V for use in the 200/500S-3 structural tests were proof loaded to 1.4 limit loads. They withstood these loads satisfactorily, and were installed in the structural test unit. Testing has been delayed because of instrumentation problems. These problems are being resolved.

Cabling from the strain gage bridge completion blocks to the digital data system was begun. Also, the installation of cold plate test fixtures was continued. Some questions were raised as to the necessity of the cold plate center swivel mount for this series of tests. IBM contends that they are required to increase the instrument unit's resistance to panel buckling. Also, IBM specified a maximum gap of 0.005 inch between the ST-124 mounting pads and I.U. when one pad is firmly bolted in place before pulling the other two pads into place. This will require shimming, and possibly machining of the ST-124 pads. Test preparations

for the 200/500S-3 structural qualification have been completed to the extent that a preliminary test run to 60% load was scheduled for the afternoon of August 26. These tests have been delayed approximately 10 days because of instrumentation problems. If the preliminary test run indicates that all systems are working properly, the first test condition will be performed August 29 or 30. It is still possible that the four test conditions can be completed by September 15.

ENGINES

I. F-1 Engine

Vibration Loads

Vibration loads for the F-1 engine turbopump struts and the center engine alignment struts have been determined for the condition of instant release. These loads are generally lower than the design vibration loads which were based on engine cutoff transients. These vibration loads will be combined with the other applicable loading to determine the structural adequacy of these struts for the instant release condition.

II. J-2 Engine

Exploding Bridgewire Initiator

An analysis of the Thiokol TX-346-1 exploding bridgewire initiator for the J-2-SE solid propellant gas generator using an omniseal indicates the initiator body has large margins of safety for proof and burst tests.

APOLLO APPLICATIONS PROGRAM

I. Payload Module Experiment Rack

Preliminary Design

The preliminary design of the rack structure was completed on schedule. Layouts with sufficient dimensions to order longlead materials were supplied to Manufacturing Engineering Laboratory. The payload module experimental rack design is progressing on schedule. A layout of the payload module docking structure, which is independent from existing LEM hardware, but, providing all docking features required, has been submitted for design approval.

II. Experiment Carrier Rack (ECR)

Critical load conditions

An impact study of the existing experiment carrier rack utilizing

ATM loading has been completed. Several of the rack members were critical. A decision has been made to design the rack to the combined critical conditions of all known payloads (PM, ATM, & PT).

III. Apollo Telescope Module

A. Vibration Specifications

ATM assembly vibration specifications were submitted and the preliminary dynamic loads study was completed. Since these interface (rack-to-ATM) loads are considerable less that the CPM interface loads, no design changes to the rack will be required.

B. Vibration Analysis

A preliminary vibration analysis has been completed to determine the first bending mode frequency of the ATM in its docked configuration. The analysis model included the rack, ATM ascent stage, and the command and service modules. This frequency was determined to be 10 cps and will be compared with the forcing frequencies induced by vehicle maneuvers to see if they are coupled.

IV. S-IVB Spent Stage

A. Acoustic Response Study of Micrometeoroid Shield

A parametric acoustic response study of the proposed S-IVB spent stage micrometeoroid shielding has been completed. In this study the maximum acoustic response loads and resulting micrometeoroid shielding deflections were determined for various shielding materials, skin gages and support concepts. These loads and deflections will be used in a weight optimization study for the proposed shield.

B. Spent Stage Experiment Support Module

The design of a meteoroid test tank to be used by Materials Division at Tullahoma, Tennessee was initiated. The tank is 36" in diameter and will allow testing of flame propagation of the S-IVB stage internal insulation when hit by meteoroids.

ADVANCED TECHNOLOGY

I. Titanium Crossbeam

The test set-up for testing the titanium crossbeam was completed with the exception of the instrumentation. The crossbeam will be subjected to a 40% load for 1 hour. After releasing the load, the crossbeam will

be inspected for visible cracks. Two days after the application of the 40% load, a complete dye penetrant check will be made. If the beam has not cracked to a degree that requires further testing, instrumentation will be added and the specimen will be loaded as per the test plan.

II. Nuclear Ground Test Module

Three thrust structure concepts are being investigated and have been given to Strength Analysis for sizing of elements. Physical properties and other data of recommended insulation materials have been requested from Materials Division.

III. Air Lock Design

Flexible and folding type doors and hatch designs are being investigated. Preliminary concepts have been completed and are being evaluated. A rigid type door and hatch design has been completed.

IV. Shroud Separation

Investigation into separation joint designs has been reactivated. Mechanical and gas-operated separation systems will be included with the pyrotechnic systems. Possible design requirements for a contaminent free separation on the Voyager shroud may be forthcoming and special emphasis will be given to suitable systems.

V. Bonded Honeycomb Cylinders

A problem of dimples in the inner face sheet has arisen during fabrication of the 60" panels. The dimples vary in depth from .0005" to .003". The cause of the dimples is unknown; however, thermal stresses and "trash" have been eliminated as possible causes.

VI. Cryogenic Test Tank #3 (105.0 inch dia.)

The manhole cover insulation assembly was blown off during the detanking operation following the insulation space test. The damage was apparently caused by a large gas leak from the manhole cover seal. Steps have been taken to replace the seal and repair the jacket and insulation.

VII. Contract NAS8-11397, The Martin Company, Cryogenic Insulation Research

The final test of this contract has been completed and the final report draft is nearing completion (est. completion date is Sept. 1). Using the results of previous tests, the final insulation performance was 50% improved over earlier results.

for G. A. Kroll
Chief, Structures Division

GEORGE C. MARSHALL SPACE FLIGHT CENTER

R-P&VE-M-66-8

MONTHLY PROGRESS REPORT

AUGUST 1, 1966 THROUGH AUGUST 31, 1966

SATURN IB

I. S-IB Stage

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

Efforts have continued in the development and evaluation of high-temperature, unfired, ceramic insulations for application in radiant thermal environments. Current efforts are directed toward development and evaluation of an insulation having a high zirconium oxide (ZrO_2) content. The high-temperature stability of ZrO_2 was the principal factor in its selection as the primary ingredient. The formulation of the most promising insulation to date was reported earlier. The exact composition, which has been designed as ZA-82 insulation, is as follows:

Ingredient	Parts by Weight
ZrO_2 "A" fibers	40
ZrO_2 powders (-325 mesh)	40
Asbestos fibers	20
Ludox HS-30 (pH-6.6)	210

ZA-82 is prepared, applied, and dried by the same techniques used for the FTA insulations.

The insulation capabilities of ZA-82 have been evaluated in radiant heating environments. For the initial evaluation, 0.350-inch thicknesses of the insulation were applied to stainless steel substrates of 0.040-inch thickness which were overlaid with expanded metal. The cured specimens were exposed to a radiant heat flux of 24 Btu/ft^2-sec for 180 seconds. The average back face temperature rise was 206°F (114°C). This compares to 323°F (178°C) for FTA-442A and 327°F (182°C) for M-31. To further evaluate the insulating qualities of ZA-82, 0.300-inch thicknesses of the insulation were applied to stainless steel honeycomb sandwich substrates of the type used on the S-IB and S-IC heat shields and exposed to radiant heat fluxes of 24 and 40 Btu/ft^2-sec. The results of these tests are compared to results of comparable tests on FTA-442A and M-31 in the following tabulation.

Back face temperature rise after 145 seconds of exposure to radiant heat of three insulations:

	Temperature Rise, °F (°C) Insulation		
heat flux	ZA-82	FTA-442A	M-31
24 Btu/ft^2-sec	110° (61)	115° (64)	189° (105)
40 Btu/ft^2-sec	242° (134)	404° (224)	494° (274)

These data indicate a superior insulating capability for the ZA-82 insulation when compared to FTA-442A and M-31, especially at the higher heat flux.

B. Development of Hazardous Gas Detection (HGD) Systems for Saturn Launch Complexes

Activities have continued in the assembly, qualification and delivery of hazardous gas detection (HGD) systems for use at Saturn launch complexes at the John F. Kennedy Space Center (KSC). Delivery of the second HGD unit to complex 34 was made during the countdown demonstration test, propellant loading and launch of AS-202.

The HGD unit for complex 37 is more than 95 percent complete; therefore, the schedule will be met.

SATURN V

I. S-IC Stage

A. Developmental Welding

Studies have continued on the susceptibility to cracking of weldments of various aluminum alloys. A number of weld restraint tests were evaluated, and none were found to be suitable for producing cracks in weldments in aluminum alloys in plate thicknesses of 1/2-inch or greater. Emphasis in these studies has been directed toward assessing the crack susceptibility of weldments in aluminum alloys of 1/8 and 1/4 inch thickness. During this reporting period "Houldcraft" restraint specimens were fabricated and evaluated for the welding of 5086, 7002, 5456, and 1100 aluminum alloys in thicknesses of 1/8 and 1/4 inch. Presently, circular patch specimens are being evaluated for 7039 and 6061 aluminum alloys.

Studies have been initiated to determine the weldability of X-7106, 2219, and 2014 aluminum alloys in 1/8-inch thickness by the electron-beam welding process. The intent of this program is to establish optimum electron-beam welding techniques. The study will include investigation of relative merits of single pass versus two pass welds. Sufficient data will be generated on mechanical and metallurgical properties to establish firmly

significant design data. One important objective of this program is to determine a nondestructive testing technique which can be used to properly evaluate electron beam welds. Presently, there is no established technique or standard for acceptance of electron beam welds.

B. Study of Corrosion and Cleaning Procedures

1. Stress Corrosion Studies

Investigations have continued into the stress corrosion susceptibility of the 7000 series aluminum alloys. Specimens of aluminum alloy 7039 in the -T61 and -T64 tempers have been exposed for 168 and 144 days respectively to alternate immersion in salt water and exposure to the atmosphere and to the local atmosphere. Failures occurred in both environments on specimens stressed as little as ten ksi in the short transverse direction of grain growth.

Carpenter Custom 455 stainless steel, aged at 1000°F (538°C) and 1100°F (593°C) was found resistant to stress corrosion cracking in the alternate immersion environment. Additional specimens of this alloy aged at 1150°F (621°C) and stressed to various loads up to 100 percent of the yield strength are currently being tested. No failures have occurred after 135 days exposure.

Specimens of aluminum alloys X2021 and X7007 were stressed in all three directions and exposed in the alternate immersion tester and to the local atmosphere. Preliminary tests for the threshold stress levels for X2021-T8E3 (90 days) and X7007-T6E136 (180 days) in the alternate immersion tester are complete. These test data indicate that the threshold stress level for X7007-T6E136 in the short transverse grain direction is below 10 ksi (15 percent of the yield strength), and less than 51 ksi (75 percent of the yield strength) for the longitudinal grain direction. The only failures encountered in the local atmosphere are two specimens stressed in the short transverse grain direction at 20 ksi (30 percent of the yield strength). These failures occurred after 62 and 112 days of exposure. The results of tests on specimens of X2021-T8E31 indicate that the threshold stress level for the short transverse grain direction is below 25 ksi (40 percent of the yield strength) and below 45 ksi (75 percent of the yield strength) in the long transverse and longitudinal grain directions. However, severe general surface corrosion may be interfering with the test results. Additional tests on these alloys are planned to obtain a more accurate threshold level.

2. Study of the Corrosion Susceptibility of Hydraulic Actuators

Exposure to a salt spray environment has continued on representative specimens of two different designs of the S-IC stage hydraulic actuators. The actuator fabricated from 7079-T6 has been exposed for 351 days without failure, and the actuator made from 7075-T73 has been exposed for 321 days without failure. Recommendations have been made that all actuators be painted with an epoxy primer and epoxy topcoat in order to reduce the possibility of stress corrosion cracking.

3. Study of Corrosion Inducing Characteristics of Fluoro-Finder FL-50 Penetrant

Tests have been conducted to determine corrosive effects of Fluoro-Finder FL-50 penetrant on 2219-T87 aluminum alloy and 321 stainless steel alloy. This penetrant is produced by Testing Systems, Incorporated. After a 90-day exposure test this penetrant had no corrosive effect on 321 stainless steel. On the aluminum alloy some slight attack was noted after 24 hours exposure, but this did not increase significantly after 90 days. No appreciable corrosion problem should occur if this product is used correctly on 2219-T87 alloy.

C. Investigation of Various Paints and Paint Primers

A search has been initiated for information on fluorescent paints which could be used to paint an American flag on the liquid oxygen tank of S-IC stages. A military specification written for an epoxy zinc rich primer for steels has been reviewed and additional information has been requested from an approved source for the primer. The primer is reported to offer excellent corrosion protection to structural steels in a seacoast environment.

D. Investigation of Fasteners and Fastener Materials

Efforts have continued in the evaluation of cold worked A-286 for use in MC-125 sleeves. An investigation was made on a 1/4-inch diameter tubing assembly which had been torqued to 150 percent of the specified torque value. Examination of this overtorqued assembly revealed that the "B" nuts were deformed, apparently due to overtorque causing the nut to "bottom-out" on the union. This force did not affect adversely the A-286 sleeve which was still characterized by a consistent micro-hardness. The results of this investigation indicate that the A-286 sleeves are acceptable for application at -423°F (-253°C). This material is now being included in the applicable specifications for MC sleeves.

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

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

F. Evaluation of Commercial Adhesives

Studies are continuing as outlined below to evaluate, develop, or qualify new adhesives for use in the Saturn Program.

Investigation of the Effect of Humidity and Cure Cycles on the Bond Strength of Narmco 7343/7139 Adhesives

An attempt was made to determine the effect on bond strength of exposure to atmospheric humidity of aluminum adherends bonded with Narmco 7343/7139 adhesive. The approach was to expose the adherends, coated with adhesive to the humid conditions for periods up to one hour, in increments of fifteen minutes, prior to bonding the specimens. The test conditions were 84°F (29°C) and 57 percent relative humidity. The cure cycle was three days at room temperature plus 6 hours at 140°F (60°C). Test results indicate that bond strength decreased with increase on exposure to the humidity. These tests will be repeated for confirmation.

The accepted cure cycle for 7343/7139 has been 2 days at room temperature followed by 24 hours at 160°F (71°C). Several additional cycles based upon lower temperatures have been evaluated. Preliminary conclusions from these test data indicate that a 24-hour cure at 140°F (60°C) may be superior to a 24-hour, 160°F (71°C) cure. Further evaluation is underway.

G. Development and Evaluation of Potting Compounds

Studies conducted over the past several months have shown that introduction of siloxane groups into the chemical structures of epoxy and urethane polymers provides a means for improving the dielectric properties of the resin systems which are employed as potting and conformal coating compositions. Work is being continued on epoxysilane, urethanesiloxane and urethanesilazane polymers.

1. Epoxysilane Polymers

Approximately 25 grams of 1,4-bis(epoxypropylpropoxydimethylsilyl)-benzene were prepared by the reaction of allylglycidylether with an intermediate described previously, 1,4-bis(hydrogendimethylsilyl)benzene. The process was carried out in ether solution at 25°C in the presence of 0.1 percent by weight of chloroplatinic acid hexahydrate. The diepoxide was obtained in 92 percent yield. Since the compound could not be distilled at 200°C/0.05 torr, it was purified by treatment with activated carbon.

A resilient, tough crosslinked elastomer was produced by curing the diepoxide for 4 hours at 100°C with 10 weight percent of triethylene-tetramine. The dielectric properties of the material are being compared with those of the resin formed by curing diglycidyl ether of Bisphenol A under duplicate conditions.

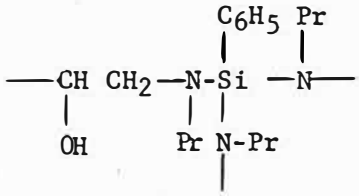
2. Epoxy Polymer Cured with Silazanes

The polymerization of pure Bisphenol A diglycidyl ether with tris(N-propylamino)phenylsilane was described previously as yielding a tough, water white polymer suitable for potting formulations. This was an example of the introduction of the silazane group into the cured epoxy

resin structure via the curing agent, a process which was studied in the previous contract with the Hughes Aircraft Company (NAS8-5499, completed August 1966) and is being studied further in the new contract with Monsanto Research Corporation, (NAS8-20402, initiated June 1966).

The sample mentioned above, which has been stored for about three months, has become soft in the outer portions indicating that some kind of depolymerization or degradation process has occurred in the surface layer. This curing system has been withdrawn from the contract program with Monsanto (NAS8-20402) until the problem has been solved.

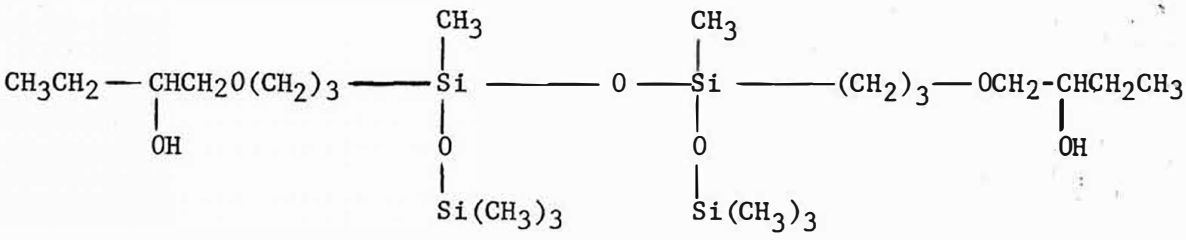
The results of preliminary experiments indicate that the degradation process did not eliminate propylamine from the structure, and further, that the gross product was not affected on standing in water at 25°C for 24 hours or boiling water for 8 hours. It appears that depolymerization or chain scission results from reactivity of the grouping,



Further tests are in progress to identify the agent (possibly water, oxygen or light) which initiates the degradation. A series of trisilazanes will be prepared in which the alkyl group adjacent to the Si-N bond is progressively larger in order to provide the steric shielding necessary to retain stability.

3. Siloxaneurethane Polymers

The siloxane diol described in the July report



has been prepared in approximately a 200 gram quantity by Grignard reduction of the corresponding diepoxide. An attempt was made to extend the chain length of the diol to increase the ratio of polyether to carbamate moieties in the cured polymer. Chain extension of the diol would theoretically increase low temperature resilience, and minimize the number of highly polar carbamate groups which contribute to deleterious dielectric properties. The diol was allowed to condense with bis(N,N-dimethylamino)-dimethylsilane in a 10:1 molar ratio at 25°C. Dimethylamine was evolved

rapidly and the diol approximately doubled in viscosity. Infrared analysis indicated formation of the Si-O bonds, which are expected to be formed in the condensation. Following characterization of the extended diol, isocyanate prepolymers will be prepared employing toluene-2,4-diisocyanate.

4. Polyurethane Conformal Coating

On August 5 we were apprised of serious component failures on printed circuit boards. Astrionics Laboratory has recently utilized glass components having a 10 mil wall thickness. The conformal coatings normally acceptable for these applications have contributed to fracture of the components during thermal cycling. It was agreed that the most immediate solution would be reformulation of the prepolymer and curing agent to give coatings of lower moduli or tensile strength, thus providing more resilience to absorb the thermal cycling stresses.

H. Investigation of Materials for Low Noise, Miniature Slip Rings

The purpose of this project is to develop or locate materials of low wear and noise characteristics for slip rings to operate for extended periods of time at low amplitude oscillations.

A set of experimental slip rings consisting of 24K electroplated gold rings and NEY-ORO 28A brushes was operated at 25 milliamperes brush current and 0.15 degrees DA oscillation. The rings were operated for 200 hours. The noise level stabilized at about 25 microvolts, average, and about 70 microvolts, (peak to peak) on peaks. The position of the noise peaks appeared to correspond to the maximum velocity portion of the driving waveform, which would be expected in the low amplitude displacement region. This test is continuing.

I. 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 ground support equipment (GSE) 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 have been made on commercial additives in the Shell Four Ball wear tester and the Falex lubricant tester. The Shell Four Ball wear test is a relative measure of the lubricating ability of the fluids. The Falex tester is also a relative measure of the lubricating ability of the fluid. The Falex test measures bearing load and resulting wear produced by forces on a rotating pin and set of vee-blocks. A successful test is 3.0 hours in duration with wear measured in the number of loading gear teeth required to maintain the 100 pound load. Results of these screening tests of RJ-1 lubricant additives are being published. Four of these additives have been selected for testing in the RJ-1 pump simulator.

Testing is continuing on the simulated ground support hydraulic pump. The pump consists of nine steel cylinders in a bronze cylindrical housing. These cylinders have swivel bronze shoes which are attached to one end and rest against a swivelling wear plate. This plate is driven by an electric motor at 1,750 rpm. The fluid being tested (RJ-1 or RJ-1 and additive) is forced by a small pressure through the pistons and small openings in the center of the wear shoes forming a lubricating film between the shoe surfaces and the wear plate surface. A static load is applied to the center of the shoes (on a holding plate). This load can be varied manually during the test. The motor torque and static load are measured on load cells and recorded on a Sanborn instrument. Several tests have been made on RJ-1 and RJ-1 with 1000 ppm of Esso additive WS 5412. The results of these tests that the Esso additive, when used in a concentration of 1000 ppm, permits a significant increase in the static load limit which may be applied to the test fixture.

J. 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

Tensile test specimens of PH 14-8Mo stainless steel have been prepared and heat treated to the SRH 950 and SRH 1050 conditions. Evaluation testing of these specimens is in process at cryogenic temperatures.

2. Evaluation of Titanium Alloy 8Al-1Mo-1V Duplex Anneal

Smooth sheet specimens of 8Al-1Mo-1V duplex annealed titanium alloy of 0.050-inch and 0.10-inch thick thickness have been tested at low temperatures. The notched specimens have not been fabricated.

3. Evaluation of Aluminum Alloy 7039

Test specimens for evaluating the -T61 and -T64 tempers of 7039 aluminum alloy have been fabricated in thicknesses ranging from 0.250 inch through 2.0 inches. These specimens will be tested at 27°C (80°F), -196°C (-320°F), and -253°C (-423°F).

II. Contract Research

During this report period, Saturn-related supporting research activities have continued in the fields of technology with the contractors and under contract numbers listed below.

A. Polymer Research, Development, and Testing

1. University of Florida, NAS8-20247
2. Narmco Research and Development, NAS8-5053, NAS8-11958
3. Peninsular Chem Research, Inc., NAS8-5352
4. Midwest Research Institute, NAS8-11338
5. Battelle Memorial Institute, NAS8-11837
6. Bell Aerosystems Company, NASw-1317

B. Development of Cryogenic and High Temperature Insulation Material

1. Goodyear Aerospace Corporation, NAS8-11747
2. IIT Research Institute, NAS8-11333
3. United Aircraft Corporation, NAS8-20089

C. Analytical Methods Development

Beckman Instruments, Incorporated, NAS8-11510

D. Assessment and Evaluation of Blast Hazards

1. Edwards Air Force Base, Government Order H-61465
2. National Bureau of Mines, Government Order H-76708

E. Development of Materials for Special Purpose Electrical Equipment

IIT Research Institute, NAS8-5351

F. Nondestructive Testing Techniques

1. North American Aviation, Incorporated, NAS8-11733
2. R. W. Benson and Associates, Incorporated, NAS8-20208

III. S-II Stage

A. Investigation of S-II-T Rib and Stringer Cracks

Analysis has continued on several cracked ribs and stringers retrieved from the S-II-T stage debris. The principal purpose of this study is to identify any material characteristics which could contribute to the cracking problem. This relatively limited purpose is necessitated by the fact that the S-II-T cracks may have resulted during vehicle failure and, therefore, would not necessarily be representative of those found on flight hardware.

Efforts during this month were concentrated on metallographic and fractographic studies. The fracture surfaces were generally ductile; however, unusual network patterns were observed on all the fractures studied. These patterns could be the result of a brittle sub-grain network; or, according to some fractographers, they could be caused by the presence of a conversion coating. Both possibilities are currently being evaluated.

Metallographic studies revealed no irregularities other than a substantial amount of sub-grains. Positive identification of sub-grains and associated micro-structure is being attempted with thin foil transmission studies. The outcome of these efforts will dictate future work in this area.

B. S-II Stage, Project Management, Materials

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

During the hydrostatic test of S-II-503 cracks were discovered in the fitting welds of the No. 3 and No. 5 liquid hydrogen feed lines. With some difficulty, the cracks were repaired by manual welding. On the basis of a rehydrostatic test and a pneumostatic test to 30 psig the weld repairs were apparently satisfactory. Subsequent to a second pneumostatic test of the stage to 35 psig, a crack reappeared in the No. 5 feed line fitting weld. The stage contractor (S&ID) currently proposes to insert bolts through the fittings in an attempt to mechanically attach the feed line to the mounting fitting. The reappearance of structural cracking problems on repeated pressurization cycles of the stage hardware casts serious doubt on the integrity of the stages particularly since the hydrostatic and pneumostatic tests are intended to establish the acceptability of the hardware.

Subsequent to the aforementioned pneumostatic test to 35 psig, two additional vertical rib ends were found to be cracked by dye penetrant inspection over the chemical conversion coating. Thus, it becomes increasingly evident that S&ID is no closer to an identification of the source of the cracking problem than they were when the cracks were first discovered.

Because of a hydrogen embrittlement problem with the H-11 steel originally specified for the bolts at the S-II/S-IVB interface, it was recommended that A-286 alloy bolts with a tensile strength of 200 ksi be used. There was some concern about the proper bolt diameter, but the Structures Division has decided that 5/16-inch diameter bolts are appropriate for AS-501 through -503 and that 7/16-inch diameter bolts should be used for AS-504 and subsequent vehicles.

The S-II stage contractor recently has informed this Center that the aerodynamic heating of the interstage caused by protuberances during the S-IC boost necessitates the addition of approximately 400 pounds of cork insulation to the exterior of the interstage to keep the structure at temperatures below 250°F (121°C). Also, S&ID has indicated the necessity of adding cork insulation to the interior of the interstage to keep the structure from melting during the dual plan separation sequence. The installation of such cork insulation on S-II-501 would have to be done at the John F. Kennedy Space Center.

IV. S-IVB Stage

A. Developmental Welding

1. Efforts have continued in the development of weld repair procedures for 3/8-inch thick, 2014-T6 aluminum alloy plate. Weldments have been prepared using various combinations of 2319 and 4043 filler wires and automatic and manual techniques. In each weld combination a maximum of ten weld repairs will be evaluated. Many specimens have been prepared for tensile testing and a portion of the metallurgical examination has been completed. Sufficient testing has not been completed to permit development of valid conclusions.

2. In order to more firmly establish the characteristics of 2014-T6 weldments, a study has been initiated into the effects of weld heat input on weld strength. These studies will include metallurgical and mechanical property determinations at specified time periods after the welding operation. Tests will be made to compare welds made with normal weld heat input to welds of excessive heat input. The time intervals for testing are established to determine the effects of natural aging for the different heat input levels. Together with these principal studies a cursory study will be made to determine the effects of successive heat (fusion) passes upon a normal weld.

B. Investigation of the Failure of S-IVB LOX Suction Line Bellows

Three failures of S-IVB LOX suction line bellows during component qualification testing prompted an examination of the unit from the S-IVB Battleship Stage. This component had undergone approximately 5400 seconds of firing time. The qualification units had failed in bellows convolutes with failure attributed to fatigue. The Battleship Stage duct was X-rayed to determine placement of the bellows within the shroud. No shroud-bellows contact was observable in the three bellows sections present in the duct. Dimensional measurements were taken through weep holes in the bellows section that failed in the qualification units. The shroud was then removed from this section and the bellows inspected. No evidences of rubbing or manufacturing defects was observed. It was then discovered that the manufacturer had instituted a configuration change, and that the Battleship unit was not representative of the qualification components. Studies of the Battleship unit will be continued in the event a decision is made to return to this design configuration.

C. Study of Materials Problems Attendant to the S-IVB Workshop Program

1. Materials Testing in Vacuum

The purpose of this project is to determine the vacuum compatibility of materials contained within the liquid hydrogen tank of the S-IVB stage. Materials will be evaluated as to weight loss and the outgassing constituents will be identified for possible toxic products. This project has recently been expanded to include testing in a 5 psia atmosphere.

Tests are continuing on Dynatherm D-65, the flame-retardant coating for the S-IVB insulation, and D-65A, the primer coat for D-65.

Four samples of D-65A primer were prepared by brush coating four aluminum plates 3 inches by 5 inches and air drying for 48 hours. Two of the samples were placed in separate stainless steel test chambers, evacuated to 100 microns and backfilled with oxygen (O_2). This operation was repeated three times with a final filling to 5 psia O_2. The samples were heated to 120°F (49°C) for 24 hours, and then the chamber gas was subjected to gas chromatographic analysis, which indicated only the presence of nitrogen (N_2) and oxygen (O_2).

Samples of D-65 were prepared by spray coating two 0.010 inch layers of D-65 on an aluminum substrate and air drying for 48 hours. One of the samples was placed in a test chamber at 5 psia O_2 and heated to 120°F (49°C) for 48 hours. A second sample was placed in a vacuum of 5×10^{-5} torr for 48 hours prior to being placed in a test chamber with 5 psia O_2 and heated to 120°F (49°C) for 48 hours.

Analysis of the first sample indicated 806 parts per million tetrahydrofuran and 13 parts per million cyclohexanone. Analysis of the second sample indicated less than 10 parts per million of tetrahydrofuran and no trace of cyclohexanone.

Dynatherm is normally a mixture of 80 percent base material and 20 percent solvent. The solvent is a mixture of 90 percent tetrahydrofuran and 10 percent cyclohexanone. The results of the analyses indicate that the coating is not completely cured by the 48-hour air-dry, but that exposure to vacuum for 48 hours completely removes the remaining solvents. These tests definitely indicate that proper curing of this coating will reduce the outgas products below the toxic level.

2. Evaluation of Materials as Sealants for Dynatherm D-65

Four 3 by 5-inch aluminum specimens were coated with D-65 and air dried for approximately two weeks. These specimens were weighed and subjected to 212°F (100°C) for 48 hours. After the heat soak, the specimens were reweighed and revealed a weight loss of approximately 11 percent.

Two of the specimens were spray coated with Kel-F-800 coating from Spraylon Manufacturing Company. These coated samples were air dried for two weeks and then submerged in water for 24 hours along with the uncoated samples. The specimens were removed from the water, blotted, and weighed. The uncoated specimens gained approximately 20 percent of the dried sample weight, whereas the specimens coated with Kel-F gained only one percent. All four specimens lost all adhesion after immersion, thus, in the event that a seal coat is required for the D-65, this Kel-F overcoat would merit further study.

3. Investigation of the Adhesion of Dynatherm D-65

A series of samples was prepared to evaluate the adhesion of Dynatherm D-65 coating that has been proposed as a fire-retardant on the 3D foam insulation of the spent stage S-IVB Workshop.

The first series of samples consisted of 2014 aluminum strips, 2 inches x 10 inches x 0.125 inch which had been acid-etched and coated with Narmco 7343 impregnated glass cloth. Separate specimens were prepared with the exposed Narmco 7343 surface primed as follows before application of the D-65 coating:

a. Unprimed
b. Primed with 10 percent Narmco 7343 in toluene
c. Primed with Goodyear G-207 primer
d. Primed with 3M Company XC-2901 primer
e. Primed with Dynatherm D-65A

Half of the samples were installed in a liquid hydrogen (LH_2) cryostat and were loaded in tension to approximately 100 pounds. The cryostat was filled with LH_2. Within minutes the tension had increased to 2000-2500 pounds. The remaining samples were loaded to 2500 pounds after being installed in the cryostat, the LH_2 was added and after three minutes additional load was applied until metal failure occurred in a pin hole area. This required about 11,000 pounds total load. The samples were visually examined after returning to ambient conditions and no indications of any cracks or other failures were evident. A low power microscopic examination failed to detect micro-cracks.

A second series of test specimens was prepared using the 2 inches x 10 inches x 0.125 inch aluminum strips with pieces of 3D foam material (2 inches x 6 inches) bonded on each side of the center area. The foam was covered with urethane impregnated glass cloth and allowed to cure. Prior to application of the D-65 coating, the impregnated glass surface was primed with the primer systems listed above. These samples were installed in the cryostat, preloaded with 2500 pounds tension, submerged in LH_2, allowed to soak about three minutes and then pulled to failure. The failures were again in a pin-hole area, and occurred at about 11,000 pounds total load. These specimens were examined at ambient conditions and a fine crack was evident at the metal surface between the two foam pieces. This cracking was caused by the tensile loading after liquid hydrogen addition, and there was no indication of any failure between the D-65 coating and the glass reinforced urethane coating on the 3D foam. The conditions used for this test are much more severe than would ever be encountered in flight conditions of the S-IVB vehicle.

Two other test specimens were made by bonding two 3D foam blocks to metal plates of 0.125 inch thickness with Lefkoweld 109 adhesive. The 3D foam blocks were recessed in the center section to completely enclose the metal plates when bonded together. The exterior surface of the samples were covered with glass cloth reinforced polyurethane and one sample was

primed with XC-3901 primer. Dynatherm D-65 was then applied to the two specimens. The samples were placed in the cryostat, LH_2 was added, and the specimens were soaked in the LH_2 for approximately 5 minutes. The specimens were removed, and were examined after warming to room temperature. There was no evidence of cracks, debonding, or failure of any kind at any place on the two samples.

These tests have not shown any indication of failure of the D-65 coating as applied to the glass cloth-urethane seal on the 3D foam. Since there was no failure, advantages that could be attributed to the use of the primers were not evident.

4. Investigation of the Flammability of Materials in Gaseous Oxygen

Activities have continued in the development of a test method for assessing the flammability and compatibility of materials in an atmosphere of oxygen when contacted by an electrically heated wire.

In addition to the above flammability tests, an evaluation was made of various top coats for D-65 materials that would protect it from the water wash now contemplated for the S-IV Spent Stage.

Materials evaluated were (1) 3D foam, (2) 3D foam coated with Dynatherm D-65, (3) 3D foam coated with Dynatherm D-65 and Ram Chemical 5-MJG-3-4 and (4) 3D foam coated with Dynatherm D-65 and Kel-F-800.

The tentative results of these evaluations indicate that Kel-F-800 or Ram Chem. No. 4 can be used as top coats for D-65 with no detrimental flammability problems. Before a top coat is selected, low temperature tests and hypervelocity impact tests will be made to fully establish the acceptability of a top coat. One important observation made from this limited study is the flame retarding property imparted to the 3D insulation (based on these tests) by the D-65. Without the D-65, the 3D insulation ignited and burned completely while the coated samples usually only charred.

5. Simulated Micrometeoroid Tests of S-IVB Materials

Testing and materials evaluation have continued in the study of the effects of simulated micrometeoroid impacts on materials used in the S-IVB Stage.

An additional program was started to study the "ballistic limit" of the 3D insulation. Experiments completed to date indicate that the insulation will burn when punctured with a 3/32-inch diameter aluminum pellet at velocities of 22,700 feet per second. Tests are planned in which an insulation sample 3-4 feet in diameter will be impacted with a hypervelocity particle to ascertain if the generated flame will extinguish or continue to burn across the ribs of the S-IVB Spent Stage.

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. Auxiliary Propulsion System Oxidizer Tankage

a. Testing of two APS (Auxiliary Propulsion Systems) shot-peened tanks has been completed at the Langley Research Center and the third tank of this three-tank program is being returned to the stage contractor for use in the flight program. The two tested tanks were exposed to N_2O_4 at 110°F (43°C) and a skin stress of 90 KIPS. One tank was pressurized to failure after thirty days exposure to these conditions, and the second tank was pressurized to failure after sixty days exposure. Failure of the tanks occurred at 800 psi and 745 psi respectively. The design burst pressure of these tanks is 550 psi, and the maximum skin stress these tanks will experience in service is about 55 KIPS. Thus, shot-peened tanks are considered flightworthy for Saturn V/S-IVB APS applications.

b. Numerous APS tanks of the type originally intended to be used for Saturn V/S-IVB APS applications have failed in the Apollo program, presumably because of stress corrosion. Bell Aerosystems undertook a rather extensive program to solve the problem, and the reported conclusions of this program are that (a) 6Al-4V titanium is satisfactory for APS tank applications, (b) tank failures were caused by N_2O_4 having a low NO content in accordance with requirements of MIL-P-26539A, (c) inhibited N_2O_4 in accordance with MSC-PPD-2 eliminated the stress corrosion problem of tank failures, and (d) shot peening of tanks is not required to eliminate the stress corrosion problem. This division does not agree that shot peening of APS tanks can be eliminated at this time because there are not sufficient data to guarantee structural integrity of unpeened APS tanks for S-IVB applications.

2. Orbital Workshop, Materials

Personnel from this division visited Douglas Aircraft Company, Incorporated (DAC) to review the work which has been done relative to the D-65 overcoating for the internal insulation of S-IVB-209. As a result of this meeting it was determined that there is no evidence of any toxicity problem and that there is no evidence that the D-65 coating will crack and flake off during the life of the stage. Nevertheless, DAC has expressed concern over the use of the material for reasons that remain unclear.

Efforts on the part of Propulsion Division to design mechanical-type seals for the ports and vents in the stage are apparently going to be terminated because of the impracticality of the approach. The sealing

of the stage to create a habitable environment, therefore, is still a completely open item. This division is attempting to identify some materials that might be foamed or cast in place for sealants.

The thermal control coating for the exterior of the stage is still unresolved because of the mutually exclusive "requirements" for low absorbtivity - emissivity coatings and a precise knowledge of the what the absolute values will be in orbit. The problem is caused by the contamination from staging rockets that cannot be simulated accurately in ground tests. This division has and continues to propose certain experiments to illucidate the problem in orbit and is seeking support and financing for these experiments.

3. The following documents were reviewed:

a. Dynatherm D-4327 Application to S-IVB-202 LOX Tank

b. Analysis of Equipment Attachment Fittings, S-IVB Spent Stage

c. Use of HL-20 and HL-21 Hi-LOK Fasteners in the Aft Skirt Separation Ring, S-IVB Vehicle

d. Fluid Specifications

e. Stress Corrosion Susceptibility of Engine Actuators.

V. Instrument Unit

General Corrosion Studies

An Avco Products cold plate which had been filled with inhibited methanol-water solution and pressurized to 50 pounds for six months was examined for corrosion. No visual corrosion was evident.

All general corrosion tests of magnesium lithium alloys LA141 and LAZ933 continue with no appreciable changes. The samples, which are galvanically coupled to magnesium alloys, have not shown as much corrosion as was expected. Some severe corrosion has been noted on several of the samples coupled to the stainless steel alloys. Stress corrosion tests continue in the local atmosphere; however, no additional failures have occurred. Outdoor exposure tests are continuing also on various electroplated coatings on these alloys with no appreciable changes noted during the last 30 days.

ADVANCED RESEARCH AND TECHNOLOGY

I. Contract Research

Supporting research contract activities have continued in the areas of technology and with the contractors as specified below:

A. Polymer Development and Characterization

1. Southern Research Institute, NAS8-20190
2. Hughes Aircraft Company, NAS8-5499
3. Goodyear Aerospace Corporation, NAS8-11070
4. W. R. Grace Company, NASw-924
5. National Bureau of Standards, Government Order H-92120

B. Adhesives Development

1. Narmco Research and Development, NAS8-11068
2. Monsanto Research Corporation, NAS8-11371, NAS8-20402, NAS8-20406

C. Developmental Welding

1. Southwest Research Institute, NAS8-20160
2. The Boeing Company, NAS8-20156

D. Alloy Development

American Machine and Foundry Company, NAS8-11168

E. Physical and Mechanical Metallurgy

1. Aluminum Company of America, NAS8-5452
2. Syracuse University, NAS8-11345
3. Battelle Memorial Institute, NAS8-20029

F. Composite Material Development and Testing

1. Harvey Aluminum, Incorporated, NAS8-11508
2. Aeronca Manufacturing Company, NAS8-5445
3. North American Aviation, Incorporated, NAS8-11108
4. Melpar, Incorporated, NAS8-11322
5. Douglas Aircraft Company, NAS7-429
6. Mitron, Research and Development Corporation, NAS8-20609

G. Lubricants and Lubricity

Midwest Research Institute, NAS8-1540

H. Corrosion in Aluminum and Steel

1. Aluminum Company of America, NAS8-5340, NAS8-11226, NAS8-20396
2. National Bureau of Standards, GO-H2151A
3. Northrop Corporation, NAS8-20333
4. Tyco Laboratories, Inc., NAS8-20297
5. Kaiser Aluminum and Chemical Company, NAS8-20285

I. Explosion Hazards and Sensitivity of Fuels

1. Stanford Research Institute, NAS8-20220
2. Air Reduction Company, NAS8-20078

J. Synergistic Effects of Nuclear Radiation, Vacuum, and Temperature on Materials

1. General Dynamics Corporation, NAS8-18024
2. Hughes Aircraft Company, NAS8-20210

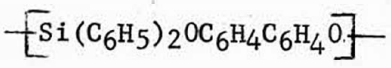
K. Instrument Development

1. Battelle Memorial Institute, NAS8-11891
2. Canadian Commercial Corporation, NAS8-20529

II. General - In-House

A. Development of High Temperature Resistant Polymers

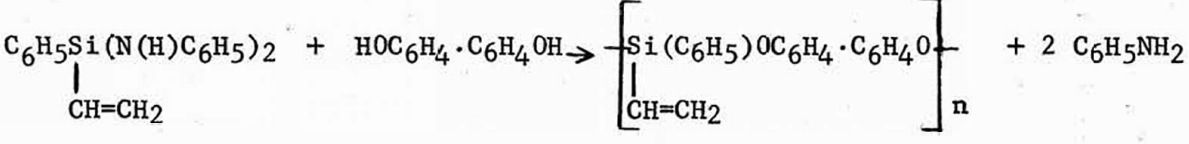
Studies have continued on aryloxysilane polymer structures, particularly Polymer A,



Several materials of closely related structure have been found to possess favorable combinations of properties, especially stability at relatively high temperatures. The current work includes synthesis of new structures, appraisal of crosslinking agents and procedures, and further studies of the physical properties of some of the products previously described.

1. Crosslinking Studies

In order to introduce vinyl side chains into a structure of the Polymer A type, dianilinophenylvinylsilane was prepared by the reaction of aniline and phenylvinyldichlorosilane under conditions previously described. The product, boiling at 201°C (394°F), 1.05 torr was obtained in 76 percent yield, and was judged by gas chromatographic analysis to be 98 percent pure. The polymer was produced by melt condensation (0.1 torr, 240°C (464°F), 6 hours) of the dianilinophenylvinylsilane with p,p'-biphenol:



The product softened at 72-77°C (162-171°F). After curing with two percent by weight of azobisisobutyronitrile at 232°C (450°F) during 15 hours, the softening point was increased to 200°C (329°F), indicating that crosslinking had occurred. Differential thermal analysis (DTA) revealed that the polymer was stable to rapid heating (10°C/min (18°F/min)) in nitrogen to 425°C (797°F).

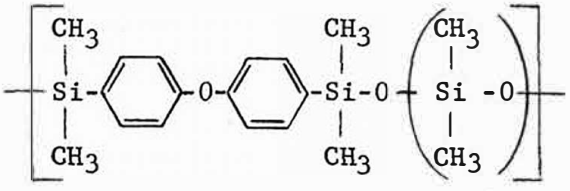
Copolymerization of the polymer possessing the vinyl side groups with styrene in the presence of peroxide yielded a thermoplastic material softening at 86-92°C (187-198°F) and thermally stable in the DTA measurement to 500°C (932°F). Thus, it appears that the styrene copolymerized with the vinyl branches to form longer branches without crosslinking. Surprisingly, the styrene did not appear to adversely affect the thermal resistance and actually may have improved its short time thermal stability.

Activities have continued in the development of means of testing the effectiveness of curing agents for Polymer A compositions filled with glass cloth. The flexural strength of the laminates is determined by use of a modification of the ASTM D790-63 Flexural Test device. The first results obtained at room temperature indicate that Polymer A, cured with trianilinophenylsilane, has high adhesion for glass fiber, and values of flexural strength are obtained that are comparable to those of epoxy formulations.

2. Investigation of Physical Properties

The results of measurements of the dielectric properties of an early sample of Polymer A indicate that the material may compare favorably with the best dielectric polymers now available, namely, Teflon and polyethylene. While these results are very promising, a definitive conclusion must await further evaluation of new samples which possess improved mechanical properties and which are formed with more appropriate molding equipment.

A sample of alkylaryloxysilane polymer,



has been subjected to thermal stability tests by differential thermal analysis (DTA) and differential scanning calorimetry (DSC). By DTA it was found that the material was stable to rapid heating (10°C/min (18°F/min)) in an atmosphere of nitrogen to 500°C (932°F). By DSC it was observed that T_g (the temperature at which the material changes from a rubber to a brittle, glass-like, material is 60°C (140°F). These data indicate that the polymer exhibits a remarkable combination of properties.

Through an informal cooperative arrangement between Midwest Research Institute, Rock Island Arsenal, and George C. Marshall Space Flight Center, personnel of the Rock Island Arsenal rubber group attempted to vulcanize the alkylaryloxysilane polymer. The trials with the peroxide systems normally employed for curing dimethylsilicone elastomers were unsuccessful. It appears that utility of this interesting polymer will depend on the development of an effective curing system.

B. Development and Characterization of Phosphonitrilic Polymer

The projected synthesis of substituted phenylphosphonitrilic chloride tetramers was continued with the completion of the study of the reaction of anhydrous HCl with 2,4,6,8-tetraphenyl-2,4,6,8-tetrakisdimethylaminophosphonitrile.

$$P_4N_4Ø_4(N(CH_3)_2)_4 + 8\ HCl \longrightarrow (ØPNCl)_4 + 4\ DMA\cdot HCl.$$

The oily product obtained from the reaction, after separation of amine hydrochloride salt, could not be induced to crystallize. Dissolution of the oil in glacial acetic acid followed by additional treatment of the resulting solution with anhydrous HCl failed to effect separation of any further amounts of amine salt. Examination of the IR spectrum of the oily product isolated from the reaction revealed the presence of a weak absorption band at 980 cm^{-1} which indicated that the product was still contaminated with unregenerated amide. Although the experiment could not be termed a complete success by any means, the techniques to be used in arylation of the PN ring were firmly developed for subsequent reactions.

Therefore, emphasis was reshifted to the preparation of a non-geminally substituted $(RPNCl_2)_4$, where $R = NR'_2$, isomer which could be arylated via the Grignard reaction to give a mixture of isomers capable of subsequent replacement of the NR'_2 groups by halogen atoms that the reaction of $(PNCl_2)_4$ with 8 equivalents of dimethylamine gave atbest a 50 percent yield of product which contained 1-3 percent of geminally substituted isomers.

To this end the reaction between $(PNCl_2)_4$ and N-methylcyclohexylamine was reinvestigated. The choice of this particular amine was based on the report in the literature (K. John, et al., J. Am. Chem. Soc., 83, 2608 (1961)) that reaction of $(PNCl_2)_4$ with excess N-methylaniline gave a tetrasubstituted derivative in which one amine group is bonded to each phosphorous atom. The presence of but a single line in the nuclear magnetic resonance (N.M.R.) spectrum of the compound (H_3PO_4 as reference) suggests only one phosphorous environment for the molecule. The completely reduced derivative of the N-methylaniline was used for this study because of the greatly increased basicity and concomittant shorter reaction times. It was also assumed that there was no difference in the size of the two amine molecules and hence the same steric factors would dictate the formation of only non-geminally substituted derivatives when using N-methylcyclohexylamine.

In a typical run 0.4 mole N-methylcyclohexylamine in 250 milliliters (ml) of tetrahydrofuran (THF) was cooled to -10°C (-14°F) and maintained at this temperature with stirring while treated dropwise with a solution of $(PNCl_2)_4$ 0.05 mole) in 125 ml. of THF. After stirring for 24 hours at room temperature the separated amine salt was removed by filtration (95 percent yield). The filtrate was concentrated in vacuo to an oil which solidified upon trituration with acetonitrile, (93 percent yield), melting range 115-117°C (239-243°F). An analytical sample was recrystallized

from acetonitrile-benzene, melting range 117-118°C (243°F -244°F). The narrow melting point range suggests the presence of only a single isomer. Elemental analyses agreed closely with the theoretical values.

The arylation reaction with the Grignard reagent was carried out as shown below:

$$P_4N_4Cl_4(N(CH_3)\ (C_6H_{11}))_4 + 4\ \emptyset MgBr \longrightarrow P_4N_4\emptyset_4(N(CH_3)(C_6H_{11}))_4 + 4\ MgBrCl.$$

A dibutyl ether solution of the amide derivative (0.03 mole) was heated to 100°C (212°F) and filtered to remove a trace of insoluble material. The filtrate was maintained at 100°C (212°F) and treated dropwise with stirring with a dibutyl ether solution of ØMgBr (0.12 mole). After stirring for two hours at this temperature, the resulting mixture was filtered and the filtrate concentrated in vacuo. The resulting oil could not be induced to solidify. The IR spectrum of the oil indicated that phenyl substitution had occurred and that displacement of the amine groups had not taken place. The weight of the oil represented a 96 percent yield - the weight of the MgBrCl indicated quantitative conversion to the tetraphenyl derivative. Characterization of the oil was limited to infrared spectral data.

Regeneration of the arylated tetra substituted amide was attempted in the following manner:

$$P_4N_4\emptyset_4(N(CH_3)(C_6H_{11}))_4 + 8\ HCl \longrightarrow (\emptyset PNCl)_4 + 4\ \text{amine salt}.$$

The oily mixture of isomers was dissolved in chlorobenzene, the resulting solution heated to reflux (132°C (270°F)) and maintained at this temperature while treated with anhydrous HCl for two hours. At the end of this time the mixture was cooled and the solid which had separated removed by filtration (13 g., 70 percent).

The filtrate was concentrated in vacuo after having been washed with water to remove any residual amine salt and dried over anhydrous magnesium sulfate. The residual oil was triturated with cyclohexane and the resulting solid separated by filtration. The product is soluble in benzene, carbon tetrachloride and insoluble in petroleum ether. The infrared spectrum (CCl_4) of the solid shows tetrameric PN ring absorption and the near complete disappearance of the PNR_2 absorption band at 980 cm^{-1} indicating displacement of this group by halogen atoms. If this compound is shown to be a $(\emptyset PNCl)_4$ isomer, then a new route to the preparation of tetrameric arylsubstituted phosphonitrilic chlorides has been developed.

C. Investigation of Materials for Electrical Contacts in Vacuum

Development work and qualification testing have continued on low resistivity brush materials for possible application in the environment of space.

As reported earlier, the niobium diselenide ($NbSe_2$) based brush materials appear more promising for the projected applications than the previously developed molybdenum disulfide (MoS_2) based materials. Initial tests have shown that single phase $NbSe_2$ brushes have low wear rates and minimal commutator damage; however, the $NbSe_2$ brushes have a relatively high coefficient of friction and a susceptibility to chipping and/or splitting along the planes of preferred crystallite orientation. At present, attempts are being made to overcome these deficiencies by the use of selected additives, such as MoS_2, silver, graphite fibers, and the diselenides of tungsten, tantalum, and molybdenum.

The $NbSe_2$-MoS_2 system yields materials of adequate strength and electrical conductivity in the composition range of 0-40 volume percent MoS_2. However, these materials have not demonstrated properties superior to single-phase $NbSe_2$. The system $NbSe_2$-tantalum diselenide ($TaSe_2$) also yields materials with adequate mechanical and electrical properties but, again, no improvement in brush operating characteristics has been noted.

A single sample of $NbSe_2$-silver (Ag) has been hot pressed and tested in the D.C. motor-generator set. This material did not chip during the initial test, so this system is believed to merit further study.

Tests are being set up to check out the coefficient of friction in vacuum of the following materials: 100 percent $NbSe_2$, 85 MoS_2-15 Ag, 60 $NbSe_2$-40 MoS_2, 80 $NbSe_2$-20 MoS_2, and 80 $NbSe_2$-20 TaS_2. In addition the following materials have been prepared and will be tested: 60 $NbSe_2$-40 $TaSe_2$, 70 $NbSe_2$-30 $TaSe_2$, 90 $NbSe_2$-10 Ag and 90 $NbSe_2$-10 Ag.

Further measurements are being made to fully characterize the electrical properties of the hot-pressed brush materials. Determinations were made of the resistivity as a function of temperature of four samples of 2.5 micron MoS_2-Ag and one sample of 4 micron MoS_2-Ag. All of the materials tested were characterized by metallic conduction and one sample consisting of 70 percent 4 micron MoS_2-30 percent silver had a resistivity at 100°K of 8.1×10^{-6} ohm-cm.

Determinations were made of the Seebeck coefficient for 22 materials of varying composition, pressing temperature and pressing pressure. In general the 4 micron MoS_2 indicated a lower and unchanging thermoelectric voltage while the 2.5 micron MoS_2 was higher and varied as a function of composition. Pure 2.5 micron MoS_2 had a Seebeck voltage that was positive, which would be expected of a P-type semiconductor. As the percent silver is increased, the Seebeck voltage becomes negative, indicating N-type doping, which agrees with the postulated conduction mechanism of these materials. However, Seebeck voltage for 100 percent 4 micro MoS_2 was also positive, and larger than the 2.5 micron MoS_2 value, which was not expected. These results are still being analyzed.

D. Investigation of the Dielectric Properties of Materials

1. Flat Cable Tests

Because Mylar flat cables currently are used in launch vehicles and spacecraft, a study has been made to determine the changes in electrical properties produced by exposure to the environment of space. Mylar flat cables were exposed to ultraviolet radiation both at an environmental pressure of 10^{-9} torr and in air. Periodic measurements were made of the dielectric constant, dissipation factor, and conductivity throughout the run. The experimental data indicate that under the conditions of the test and for the exposure times used Mylar flat cable is not adversely affected by ultraviolet radiation in air or in vacuum.

2. Teflon Tubing and RJ-1 Fuel

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 tubing and additives to the hydraulic fluid. 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.

Tests were made to determine the relative effectiveness of Shell ASA-3 and Esso WS-3966 anti-static (conductivity) additives when added to RJ-1 in various concentrations. Both additives increased the conductivity of RJ-1, exponentially, producing the greatest increase in conductivity per unit mass of additive at the smaller concentrations, up to 300 ppm, and producing less improvement as the concentration approached 1000 ppm. However, the ASA-3 produced a conductivity about one order of magnitude higher than the WS-3996 for all concentrations.

The voltage gradient was varied also for tests on both ASA-3 and WS 3996. Over a wide range of voltage gradients no particular variation was noted in conductivity, although values were more uniform at higher gradients.

E. Investigation of Nuclear Environmental Effects on Materials

1. Study of the Effect of Charged-Particle Irradiation of Polymers

A study has been initiated into the effects of charged-particle radiation on the mechanical properties of polymeric materials. Much data have been obtained on the effects of gamma radiation but very little information is available on charged-particle effects. The ultimate goal of this program is to obtain sufficient data to determine whether a correlation exists between radiation damage produced by electron and gamma radiations.

Ninety-six specimens of thin film materials were tested to determine changes in ultimate tensile strength and elongation produced by irradiation with one Mev electrons. Standard tensile specimens of Mylar (2 mil), Teflon (5 mil), H-Film (2 mil), and Tedlar (2 mil) were irradiated to fluences of approximately 10^{14}, 10^{15}, and 10^{16} electrons cm^{-2} and then analyzed with an Instron tensile tester.

It has been shown that H-film is less affected by neutron or gamma ray bombardment than Teflon, Mylar or Tedlar. However, under electron radiation both Mylar and Tedlar retained significant strength at a fluence of 1×10^{16} whereas H-film and Teflon suffered total degradation.

2. Vacuum Tensile Tests

A program has been initiated to determine in-situ the effects on the tensile properties of materials after prolonged exposure to vacuum and temperature. It is necessary that the vacuum-temperature effects on the tensile properties be isolated in order to determine the effects of radiation on the tensile properties of materials in a space simulated pressure-temperature environment.

Mechanical property tests were made in vacuum on 27 specimens of Mylar and 45 specimens of Tedlar after exposure to reduced pressures of 3×10^{-7} to 5×10^{-8} torr for periods of 24, 48, and 96 hours. Two thicknesses of each material were evaluated to determine if specimens of different thicknesses behave the same in a vacuum environment. The data from these tests are being evaluated.

F. Study of the Compatibility of Lubricants with MIL-H-5606 Hydraulic Fluid

Tests are continuing to determine if Dow Corning DC-4 (silicone compound) lubricant and technical petrolatum (VV-P-236) are compatible with hydraulic fluid. A testing device was designed and fabricated whereby four samples of the same lubricant could be tested at one time resulting in more reliable data. This is obtained by fastening 4 screened cups each containing approximately 4 grams of the lubricant to a four spoke wheel. All four cups are submerged in the hydraulic fluid and the 6-inch diameter wheel is rotated by a directly coupled 1 rpm motor. The hydraulic fluid can be heated by placing the container on an electric hot-plate. The samples are carefully weighed before and after test to check the weight loss or weight gain. Three tests have been run with DC-4 for one hour and 45 minutes duration each at room temperature. The test data show an average increase in weight of 7 percent possibly indicating some adsorption of hydraulic fluid. Three tests were also run with DC-4 for one hour and 45 minutes duration each at 128°F (53°C). These test data also showed an increase in weight of approximately 8 percent.

G. Development of Direct Current Motors for Use in the Environment of Space

Materials are being evaluated at extreme temperatures and low pressures for use in direct current (d.c.) motors designed for operation in the space environment.

During the reporting period one test was made with the motor generator set. This test was made with the field currents set at 1.5 amperes, and motor armature at 40 volts giving a speed from 650 to 700 rpm. Brushes and bearing retainers employed in this test were niobium diselenide-silver (90 percent - 10 percent) and polyimide silver respectively. The longest continuous operating period during the test was 7 hours with accumulated test time of 21 hours. Heavy sparking was observed at the commutator brush interface during the first 5 hours of operation. The test was then stopped, heat applied to the motor overnight and then restarted the following day. The set was operated at elevated temperatures for 7 hours during which time only light sparking was observed. The next 7-hour test period was begun with heat applied to the system as the test was begun. Again light sparking occurred. The final test period was a 2-hour test in air at ambient temperature. This test was stopped when a ring scar appeared on the commutator.

During vacuum testing no chipping was observed on the brushes. However, the brushes did chip as they were forcefully removed from their holders at the conclusion of the test. Since the brushes remained intact during testing, it would indicate that the silver has added to the strength of the brush. Future testing will include brushes of various percentages of niobium diselenide and silver with the aim of obtaining an optimum composition of niobium diselenide-silver brush.

H. Determination of Physical Properties of Materials by Nondestructive Techniques

1. Internal Friction Measurements

The internal friction of metals is being investigated to determine if this property is sensitive to the microstructural changes produced by the stress corrosion process.

Internal friction measurements have been made on fifty specimens of 7079-T651 aluminum alloy in air and in a vacuum. Both 1/8-inch and 3/16-inch diameter specimens machined in the short transverse or longitudinal directions were tested. The greatest energy losses were obtained when the short transverse specimens were subjected to both stress and the salt solution, whereas only small losses were measured when the specimens were exposed to the corrosive environment alone.

2. Electrical Conductivity

Electrical conductivity studies are being made to determine whether surface conductivity changes can be used to indicate the progress of the stress corrosion process in aluminum.

Recent efforts have been directed toward the measurement of conductivity using a high frequency eddy current instrument. Several specimens which had been exposed to stress corrosion were tested in the frequency range of 100 KHz to 6 MHz with the result that large conductivity changes were noted. Further measurements are being made to define the relationship between high frequency conductivity and time of exposure to stress corrosion.

I. Study of Materials Problems Associated with Advanced Manned Missions

Apollo Telescope Mount

Vacuum compatibility tests were made on two materials which are being considered for the Apollo Telescope Mount, and for the Electromagnetic Radiation Experiment.

It was reported that spectral requirements for the cameras for both these applications will require the camera film to be exposed to vacuum. Samples of Kodak TRI-X panchromatic film were exposed to a pressure of 10^{-8} torr for 48 hours to determine if there was any embrittlement of the emulsion backing, or any substantial change in the gelatinous portion of the emulsion. Visual examination revealed only a slight change in the stiffness of the backing.

Another sample of Kodak TRI-X was subjected to a weight loss determination at 10^{-6} torr up to 310°F (154°C). The major portion of the weight loss, 1.4 percent, occurred during pumpdown and may be attributed to loss of water vapor. Temperature to 310°F (154°C) over the next 19 hours yielded only 1 percent additional weight loss, and indicates the material is quite stable. However, no attempt was made to determine any possible vacuum effects on the sensitivity of the film.

The second material tested was a 90 percent bismuth powder, 10 percent epoxy bonded solid proposed as a shield material for the gamma ray spectrometer experiment. The sample was evacuated to 2×10^{-5} torr at 82°F (28°C) for 2.5 hours and then heated to 320°F (160°C) for 3 hours. No weight loss (0.05 milligram sensitivity) was noted.

J. Study of Material for the Nuclear Ground Test Module

In-house and contractual studies are being pursued to develop the materials technology required to support the Nuclear Ground Test Module (GTM) Program. Specifically, the areas of cryogenic insulation, valve seals, transducer materials, gimbal and bearing lubricants, and induced neutron activation are being actively investigated.

A contract (NAS8-18024) has been initiated with the General Dynamics Corporation, Forth Worth, Texas to evaluate the effects of radiation and cryogenic temperature on the mechanical properties of selected cryogenic insulations, adhesives, and vapor barriers. In addition, the structural integrity of two insulation systems will be determined after exposure to acoustic, cryogenic temperature, and radiation stresses.

Work has been initiated to determine the coefficient of thermal conductivity from room temperature to 20°F (-7°C) for a specimen of laminated corkboard which is a candidate insulation system for the GTM. In addition, the coefficient of linear thermal expansion is being determine at cryogenic temperatures for corkboard. These data will be used in the analysis of the thermal and structural performance of this insulation system.

Currently, efforts are being made to obtain drawings of the cryogenic valves and transducers which conceivably could be used on the GTM. When these drawings become available, they will be analyzed to determine the types of materials involved. Recommendations then will be made as to the acceptability of these materials for use in a radiation environment and, when appropriate, replacement materials will be specified. Qualification testing of these components will be conducted under contract NAS8-18024 and will be coordinated with personnel of the Quality and Reliability Assurance Laboratory and the Propulsion Division of this laboratory.

K. Documentation Review

The following specifications, documents, or reports were reviewed and comments were forwarded, where appropriate, to responsible individuals or organizations:

1. Douglas Process Specification, DPS-32333, "Common Bulkhead Aft Toe Weld Doubler Installation"

2. Douglas Process Specification, DPS-32332, "Jamb Weld Doubler Installation, LOX and LH_2 Tank Assemblies"

3. Douglas Process Specification - -41006, "Aluminum Alloys, Preparation for Welding"

4. Douglas Process Specification DPS 14052, "Mechanical MIG-GMA and TIG Fusion Welding Aluminum, Saturn Assemblies"

5. S&ID SPEC-MB00170-040, "Inconel 718 Alloy Sheet, Strip, and Plate: Consumable Electrode, Vacuum Melted, Solution Annealed at 1950°F"

6. S&ID SPEC-MB0170-012, "Inconel 718 Alloy Bar, Forgings and Forgings Stock: Consumable Electrode, Vacuum Melted, Solution Annealed at 1800°F"

7. S&ID SPEC-MB0170-041, "Inconel 718 Alloy Bar, Forging and Forging Stock, Consumable Electrode, Vacuum Melted, Solution Annealed at 1950°F"

8. MSFC-PROC-195A, dated 8-10-66, "Cleanliness Level Requirements and Inspection Methods for Determining Cleanliness Level of Gas Bearing Gas Supply and Slosh Measuring System"

L. 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. Lubricants and Lubricity
4. High and low temperature resistant polymers
5. Stress corrosion on structural alloys.

J. E. Kingsbury

MONTHLY PRODUCTION REPORT
MATERIALS DIVISION
AUGUST 1, 1966 THROUGH AUGUST 31, 1966

I. Radiographic Inspection

One hundred and fifty miscellaneous parts, components and test specimens were inspected radiographically during this report period.

II. Photography

	Negatives	Prints	Other
Engineering Photography	124	476	
Metallography and Fractography	123	776	
Miscellaneous Photography Processing, Copywork, etc.			31

III. Metallurgical and Metallographic Testing and Support Services

A. Analysis of the failure of an S-II actuator component has been completed. This work was initiated at the request of Astrionics Laboratory. The anchor end of the actuator failed after eight months of intermittent operation at the Astrionics Laboratory. Although the actual number of cycles experienced by the actuator is not known, it is believed by Astrionics personnel that the component had undergone several thousand more cycles than the design limit. "Beach markings" on the fractured surface identified the mode of failure as fatigue, and this finding was verified as high cycle fatigue with the aid of the electron microscope.

B. The weld joint design and weld procedures for fabrication of component parts for the Instrument Unit package were evaluated.

C. A study was made on the joining of tin-plated nickel-200 alloy leads of a capacitor bank as part of the Instrument Unit package.

D. Aluminum alloy 1100 was mechanically and thermally processed to produce annealed stock of various thicknesses, 0.010 to 0.014 inch, for ME Laboratory for a S-IVB application.

E. Assistance was provided to R-AERO for materials selection for torsion arm assembly to determine angle of attack - dynamic aerodynamic damping relationships. A maraging steel was recommended on the basis of a requirement for a material of high torsion modulus, high yield strength, and high endurance limit.

F. The Boeing Engineering Change Procedure ECP0088, "Replacement of Fasteners on Flow Lines for Fuel Prevalves and Fuel Emergency Drain Valves," was reviewed. The fastener which is under consideration for replacement is the 137148-1 locknut, specified as 50FA-420 by the Standard Pressed Steel Company. A review of the components indicated that the specified 303 stainless steel should be cold worked rather than annealed, and that the Standard Pressed Steel locknut was adequate for the application.

IV. Spectrographic Analysis

Eight hundred and sixty-two spectrographic determinations were made on forty-one samples and six hundred and forty-seven standard determinations were made.

V. Infrared Analyses

Samples of thirteen experimental polymers, four lubricants or lubricant additives and seven miscellaneous materials were analyzed qualitatively by infrared techniques during this report period.

VI. Chemical Analyses

	Determinations
methanol-water mixture for	
sodium benzoate	11
water samples for	
heravalent chromium	6
RP-1 fuel samples for	
moisture	10
water reaction	4
RJ-1 fuel additive for	
carbon	3
hydrogen	3
phosphorus	3
sulfur	3
experimental polymers for	
carbon	44
hydrogen	44
silicon	10
nitrogen	14
chlorine	4
gas samples for	
oxygen	37
nitrogen	34
helium	8
hydrogen	14
carbon monoxide	2
moisture	7
atmospheric contamination specimens for	
acetic acid	2
fluorides	4
chromatographic analyses	94

VII. Physico Chemical Analyses

	Determinations
density of RP-1 fuel	26
pH of methanol-water mixture	11
specific resistance of methanol water mixture	11
heat of combustion of RP-1 fuel	15
flash point of	8
RP-1 fuel	8
gear box oil	6
distillation range of RP-1	4
fire point of gear box oil	2

VIII. Rubber and Plastics

	Items
molded and extruded	216
cemented	14
potted	16
coated	38
fabricated	10

IX. Electroplating and Surface Treatment

	Items
cleaned	57
salt spray tested	18
electroplated	46

X. Development Shop Production

A. A total of 4,462 man-hours, direct labor, was utilized during this period for machining, fabrication, and welding.

B. Eight hundred and eighty-five man-hours, approximately 19.8 percent of the total man-hours, were devoted to productive effort of a non-routine nature and applied to the work orders listed below.

1. 3000 psi Injector Body

Components for the 3000 psi injector body are ready for assembly and final machining.

2. Chamber Section-Hydro Coil Lined

One hydro coil-lined chamber section is near completion and another is being modified.

3. Diaphragm dies

Dies for fabrication of experimental diaphragms are complete and delivered.

4. S-IVB Fuel Tank Motion Study Components

Components for the S-IVB fuel tank motion study are complete and delivered.

5. Thermal Sensor Experiment Assembly

The thermal sensor assembly has been completed.

6. Motor Torque Gauge and Brace

Fabrication of the components for the motor torque and brace is near completion.

XI. Miscellaneous

A. Sixteen aluminum alloy items, two hundred items of stainless steel, twenty-five items of nickel alloy and one piece of copper were heat treated during this report period.

B. Applied MLF-I lubricant to twelve S-IC test stand bearing plates.

C. Thirty-two silicone rubber seals for flat cable conductors were molded for Astrionics Laboratory. These seals will be evaluated as possible replacements for Neoprene seals which have proved to be unsatisfactory for use at the temperatures encountered.

D. Spray coated three stainless steel plates 20 inches x 50 inches with Emralon 310 Teflon dispersion for Computation Laboratory. These plates are used to support the tilted lunar map for the SMK-23 simulator. Without this coating on the backup plates, vibration in the map causes the MOLAB simulator to appear to jump 4 to 5 feet off of the surface. The coating reduces the friction and eliminates this problem.

E. Eight each thermogravimetric analyses and differential scanning calorimetry tests and fifty-three differential thermal analyses were made during this report period.

XII. Publications Issued

A. "An Integrating-Sphere Reflectometer for the Determination of Absolute Hemispherical Spectral Reflectance," Gene A. Zerlaut and A. C. Krupnick, AIAA Journal, 4, No. 7, 1227-1232, July 1966.

B. "Automatic High-Pressure Gas-Sampling System for Gas Chromatography," by W. M. Langdon, V. R. Ivanuski, R. E. Putscher, H. J. O'Neill, and A. C. Krupnick, Journal of Gas Chromatography, 269-270, July 1966.

C. Morgan, W. R.: Low Temperature Mechanical Properties of Berylco Nickel 440 Strip, IN-P&VE-M-66-4, August 2, 1966.

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