Cleared: January 12th, 1972
Clearing Authority: Air Force Materials Laboratory

EXPLORATORY EVALUATION OF FILAMENT-WOUND COMPOSITES FOR TANKAGE OF ROCKET OXIDIZERS AND FUELS

M. J. Sanger, R. Molho, W. W. Howard

FOREWORD

This report was prepared by the Chemical and Structural Products Division, Von Karman Center, Aerojet-General Corporation, Azusa, California, under USAF Contract No. AF 33(615)-1671. The contract was initiated under Project No. 7381, Task No. 738101. The work was administered under the direction of the Air Force Materials Laboratory, Research and Technology Division, Mr. T. J. Reinhart, Jr., Project Engineer.

This report covers work conducted from June 1964 to September 1965 and is submitted in fulfillment of the contract. The manuscript was released by the authors in October 1965 for publication as an RTD technical report.

The study was conducted under the direction of M. J. Sanger, Program Manager, and the supervision of R. W. Buxton, Head, Applications Section. Others who cooperated in the study and in the preparation of this report were W. T. Cox, Chief Engineer, Chemical and Structural Products Division; S. B. Fabeck, Manager, Composite Structures Department; F. J. Darms, Head, Analysis Section; R. N. Hanson, Development Engineer; M. Segimoto, Resin Chemist; J. Creedon, Adhesives Chemist; R. Molho, Design Engineer; and W. W. Howard, Development Engineer. This report is catalogued by Aerojet-General as Report No. 3078.

This technical report has been reviewed and is approved.

W. P. Conrardy

W. P. CONRARDY, Chief
Materials Engineering Branch
Materials Applications Div
AF Materials Laboratory

ABSTRACT

This work was undertaken to provide information on materials and fabrication techniques for the design of filament-wound tankage for storable propellants, and to define a space experiment for validating the recommended materials and techniques.

Resins and liner materials were studied, under accelerated exposure conditions, in the unreinforced form and as composites. Fabrication techniques were evaluated, and dynamic tests of subscale tankage were conducted.

The environmental tests indicated that the materials and fabrication techniques selected for the tankage produced satisfactory filament-wound containers for storable propellants. A technique developed for bonding a metallic liner to the filament-wound chamber provided a solution to the problem of strain compatibility under pressure cycling at optimum strain levels for glass-filament-reinforced structures.

Designs were developed for a prototype tank, and analysis showed that a spheroidal configuration with a length-to-diameter ratio of 1.0 was acceptable from the standpoint of space limitations and weight economy. A space experiment was defined for use in determining the efficiency of a filament-wound storable propellant tank over a period of 1 year in an earth-orbit environment.

TABLE OF CONTENTS

TABLE OF CONTENTS (cont.)

ILLUSTRATIONS

ILLUSTRATIONS (cont.)

TABLES

TABLES (cont.)

I. SUMMARY

The major objectives of this program were to evaluate and select filament-winding resins and liner materials compatible with the specified propellants: unsymmetrical dimethylhydrazine (UDMH) and hydrazine in a 50/50-wt% mixture (AeroZINE 50 propellant), nitrogen tetroxide (N_2O_4), pentaborane (B_5H_6), and chlorine trifluoride (ClF_3).

The work included the establishment of fabrication procedures for subscale tanks for use in environmental testing with these propellants. Additional objectives were the design and analysis of a prototype propellant container and the design of a planned space experiment to evaluate the performance of subscale tankage.

None of the resins evaluated in Phase I demonstrated complete compatibility with the propellants for extended exposures, but some resin systems were found that had moderate resistance. A novolac type of epoxy resin was selected for use in the subscale tankage because of its favorable physical properties and chemical stability.

The chemical compatibility and permeability characteristics of materials suitable for liners of filament-wound tankage were studied in Phase II. Of the polymeric-type materials, the fluorocarbons were found to be the most compatible with rocket fuels and oxidizers. Films of these materials, however, were permeable to propellants and thus would not provide complete separation of fluids and tank structure. Stainless steel foils of the 300 series showed good compatibility with the propellants and were adaptable to manufacturing techniques for tankage liners.

Composites of glass filaments and the more promising resin systems were exposed to propellants at room temperature and at $100^{\circ}F$ in Phase III. More severe degradation of the composites occurred than was observed when the resin systems alone were tested. This condition emphasized the need for a barrier type of liner.

Additional emphasis was concentrated on the properties of metallic liner materials in view of these findings. A novel concept for obtaining strain

compatibility between a metal liner and the filament-wound case was developed and proved feasible by uniaxial and pressure-cycle strain tests. It involved adhesive bonding of the metal-foil liner to the glass-reinforced overwrap.

Chemical-adhesive bonding of metal-liner segments as an alternative to welding was also studied in Phase III and was found to have definite merit for further investigation. This procedure would be very useful in the fabrication of large prototype tanks.

Composites of resin and glass filaments were also exposed to a simulated space environment of heat and vacuum in Phase III, and very little degradation in properties was observed.

The long-term performance of filament-wound tankage containing rocket fuels and oxidizers was determined in Phase IV. Minor difficulties were encountered with two tanks containing ClF_3 because of corrosion at weld areas; an improvement in weld design corrected the problem. Subscale tanks containing AeroZINE 50, N_2O_4, and ClF_3 were in good condition after 4 months of exposure and thermal cycling.

Phase IV pressure-cycling tests of subscale tanks in which the bonded-metal-liner concept was used demonstrated the validity of this technique. The life obtained substantially exceeded the target requirement of 50 cycles.

Three optional designs of a prototype tank were outlined in Phase V. Analysis of these designs showed that a spheroidal tank with a length-to-diameter (L/D) ratio of 1.0 most closely satisfied the requirements of fabrication feasibility and space limitations.

A space experiment was designed in Phase VI to provide for evaluation of the performance of a propellant-filled subscale tank in an orbital mission. Equipment and fabrication details were generated to permit a properly instrumented tank to be produced from commercially available items and incorporated in a space vehicle.

II. INTRODUCTION

Inherent weight savings and fabrication advantages have resulted in increasing applications of filament-wound structures to aerospace vehicles. A logical extension of the use of the low weight-to-strength ratio characteristic of glass-filament-reinforced products is in the fabrication of tankage for storable propellants in a space environment.

The work under this contract was undertaken to provide the Air Force with information on materials and fabrication techniques for use in the design of filament-wound tankage for storable propellants. It was performed by the Chemical and Structural Products Division of Aerojet-General Corporation in a six-phase, 15-month program of material and composite studies, design effort, and simulated aerospace testing. The information obtained will permit the design and fabrication of structures for propellant storage in space at an altitude of approximately 600 miles for periods up to 1 year.

This final report presents a detailed summary of all work performed in Phases I through VI. In addition, conclusions and recommendations related to program results are presented for guidance in future work on filament-wound tankage for storable propellants.

III. PHASE I - STUDY OF RESIN MATERIALS

The objective of this study was to determine the resin system having maximum compatibility with the specified fluids and satisfactory processing characteristics to permit fabrication of subscale and prototype tankage. The propellants of interest are AeroZINE 50, N_2O_4, B_5H_6, and ClF_3.

A. LITERATURE SURVEY

The information available in the literature was rather limited with regard to the compatibility of resin-binder systems with the propellants of interest, but the investigators apparently believe unanimously that the common resin binders would not withstand direct contact with the foregoing corrosive materials for long periods. Variations in degree of resistance between resins do exist, however; Aerojet workers found (Reference 1) that the cyclic and novolac epoxies, phenolics, and blended compositions have a moderate degree of resistance under ambient-temperature conditions. The novolac epoxies, because of their higher cross-link density than the bisphenol-A type, appear to have good compatibility with AeroZINE 50. Systems cured with Methyl nadic anhydride (MNA) and boron trifluoride monoethylamine (BF_3-400) have also shown some promise in compatibility with the hydrazine-type fuels.

In the study reported in Reference 1, very few of the resin systems withstood more than a few hours of exposure to N_2O_4. The Boeing Company found that an epoxy-resin/glass matrix supporting a 5-mil Teflon film showed a very rapid reduction in flexural strength from the permeation of N_2O_4 through the film (Reference 2). Phenol formaldehyde and melamine formaldehyde resins may be promising candidates for binders for filament-wound tankage containing N_2O_4 because of their known resistance to oxidizing media. However, processing problems and a tendency toward crazing, both of which are characteristic of these resins, may prevent their acceptance.

Although little information is available on the compatibility of resin compositions with pentaborane, it has been observed that this fuel reacts with any organic compound containing a reducible functional group. The rate of reaction depends on the type and concentration of these functional groups. It has been reported that pentaborane forms shock-sensitive mixtures with a number of organic compounds, including polysulfides, ethers, esters, ketones, and chlorinated ethers (References 3 and 4).

Chlorine trifluoride is known to be a very corrosive oxidizer and will cause the detonation of many polymers and organic materials (Reference 5). There are instances of a violent incendiary reaction when an epoxy compound comes in contact with ClF_3 (Reference 6). A completely impermeable liner is obviously required for filament-wound tankage containing ClF_3.

B. PRELIMINARY SCREENING PROGRAM

Although the literature survey indicated that organic resin systems will not withstand continuous exposure to rocket oxidizers and fuels, it is desirable that maximum compatibility be obtained to provide some degree of protection for filament-wound structures in case of spillage or minor permeation of propellant through the liner.

The candidate resin systems are identified and their compositions are given in Table 1. The forms in which they are available, and the probable processing techniques for each, are shown in Table 2.

A 2-in.-dia disk was cast from each resin system, the Barcol hardnesses were determined, and each disk was then cut into two pieces and spot-tested by immersion in AeroZINE 50 and N_2O_4 for 2 days at 70°F. At the end of this exposure, the specimens were removed, were visually examined, and the Barcol hardnesses were again determined. The propellant-exposure results are shown in Table 3. Figure 1 shows the specimens after exposure. Inasmuch as most of the candidate resin systems have very high viscosities, considerable difficulty was encountered in the casting of smooth specimens; the deep pock marks evident in the photographs were present in the original specimens and were not due to attack by the immersion medium. Surface erosion is obvious, however, on the less-resistant resin systems.

The results of these spot tests confirmed, in general, the information on compatibility of resins with AeroZINE 50 and N_2O_4 that had been reported in the literature. The novolac epoxies and polyepoxides, as represented by RS-4, -5, -6, and -11 (see Table 1), showed good resistance to either one or both of the propellants. RS-10, a phenolic resin, had good resistance to both propellants but RS-12, another phenolic, had a disappointing performance. The blending of novolac epoxies with bisphenol-A, to achieve improved processing, severely reduced the propellant resistance of these systems. The high viscosity of the novolac epoxies will probably require the use of a preimpregnation (prepreg) technique for application of the resin to the glass filaments.

TABLE 1
RESIN SYSTEMS USED IN SCREENING STUDY

Resin System	Composition[1] Ingredient	Parts by Weight	Resin Type	Source
RS-1	Epon 1031	50.00	Novolac epoxy	Shell Chemical Company
	Epon 828	50.00	Bisphenol-A epoxy	Shell
	MNA	90.00	-	-
	BDMA	0.55	-	-
RS-2	Epon 1031	50.00	Novolac epoxy	Shell
	DER 322	50.00	-	-
	BF_3-400	1.00	-	-
RS-3	Epon 1031	50.00	Novolac epoxy	Shell
	DER 332	50.00	Bisphenol-A epoxy	Dow Chemical Company
	BDMA	0.25	-	-
RS-4	DEN 438	75.00	Novolac epoxy	Dow
	DER 332	25.00	Bisphenol-A epoxy	Dow
	BF_3-400	2.00	-	-
RS-5	DEN 438	49.50	Novolac epoxy	Dow
	MNA	50.50	-	-
	BDMA	0.25	-	-
RS-6	Kopox 170	75.00	Polyepoxide	Koppers Company
	DER 332	25.00	Bisphenol-A epoxy	Dow
	BF_3-400	2.00	-	-
RS-7	Laminac 4173	100.00	Polyester	American Cyanamid Company
	MEK peroxide	2.00	-	-
	DMA	0.025	-	-
RS-8	Cymel 431	100.00	Melamine formaldehyde	American Cyanamid
RS-9	Cymel 430	100.00	Melamine formaldehyde	American Cyanamid
RS-10	Resin No. 46	100.00	Phenol formaldehyde	U.S. Polymeric Chemicals, Inc.
RS-11	DEN 438	100.00	Novolac epoxy	Dow
	BF_3-400	2.00	-	-
RS-12	Resin No. 36	100.00	Phenol formaldehyde	U.S. Polymeric
RS-13	Resin No. 91LD	100.00	Phenol formaldehyde	Cincinnati Testing Laboratories
RS-14	Resin No. L-70	100.00	Styrene-butene-vinyl toluene	Emerson & Cuming, Inc.
RS-15	Kopox 170	100.00	Polyepoxide	Koppers
	BF_3-400	3.00	-	-

(1) Abbreviations:

	Definition	Use
MNA	Methyl nadic anhydride	Curing agent
BDMA	Benzyldimenthylamine	Accelerator
BF_3-400	Boron trifluoride monoethylamine	Latent curing agent
MEK peroxide	Methyl ethyl ketone peroxide	Curing agent
DMA	Dimethyl aniline	Accelerator

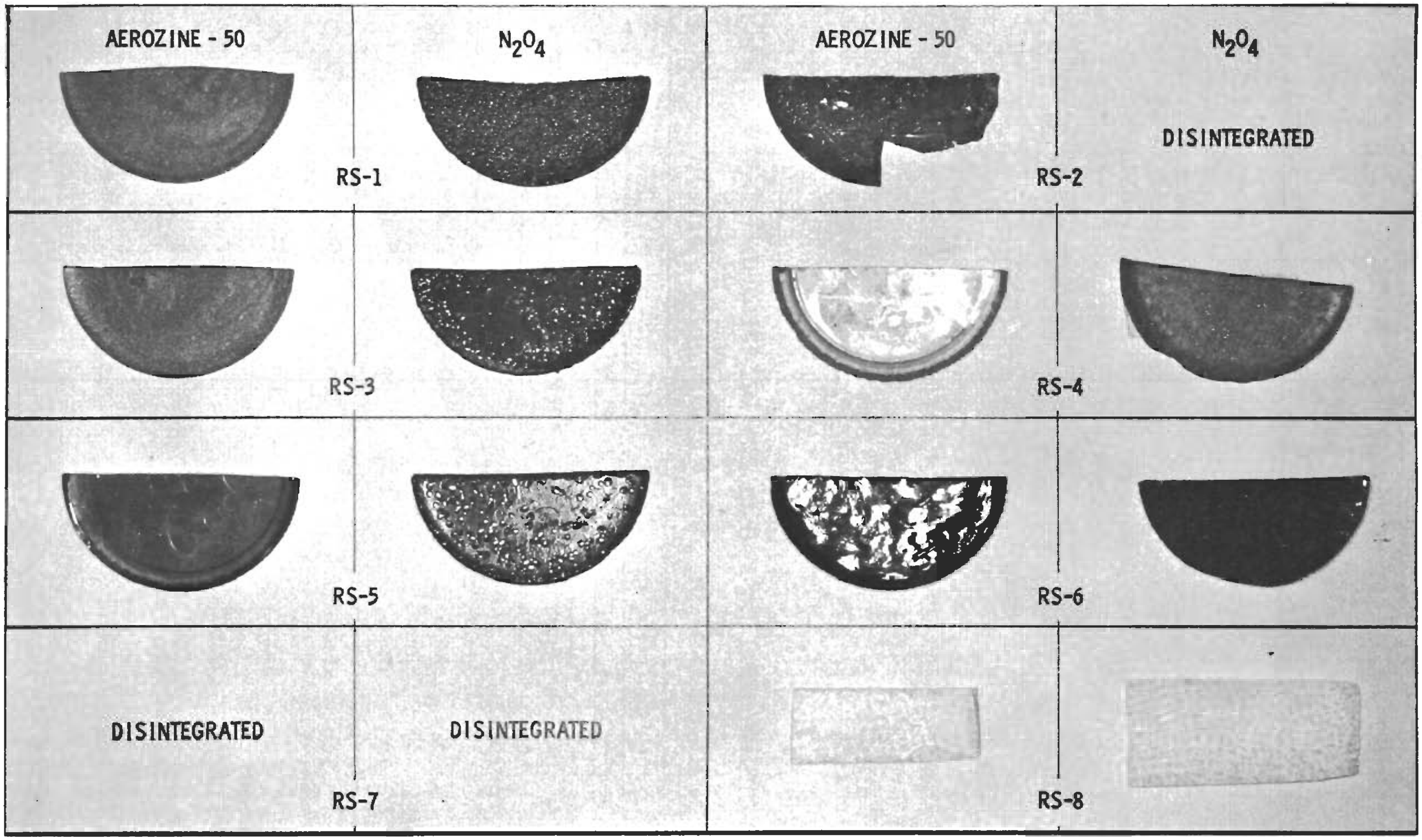
TABLE 2
PROCESSABILITY OF CANDIDATE RESIN SYSTEMS

Resin System	Form Available	Processing Technique
RS-1	Viscous fluid	Prepreg, too viscous for in-process application
RS-2	Solid	Prepreg, solvent solution application
RS-3	Viscous fluid	Prepreg, too viscous for in-process application
RS-4	Viscous fluid	Prepreg, too viscous for in-process application
RS-5	Slightly viscous fluid	Satisfactory for in-process application
RS-6	Viscous fluid	Prepreg, too viscous for in-process application
RS-7	Nonviscous fluid	Satisfactory for in-process application
RS-8	Solid	High-pressure molding process
RS-9	Solid	High-pressure molding process
RS-10	Solvent system	Prepreg, venting required
RS-11	Viscous fluid	Prepreg, too viscous for in-process application
RS-12	Solvent system	Available as prepreg only
RS-13	Solvent system	Available as prepreg only
RS-14	Nonviscous fluid	Satisfactory for in-process application
RS-15	Viscous fluid	Prepreg, too viscous for in-process application

TABLE 3
RESIN-MATERIAL SCREENING, TWO-DAY EXPOSURE(1)

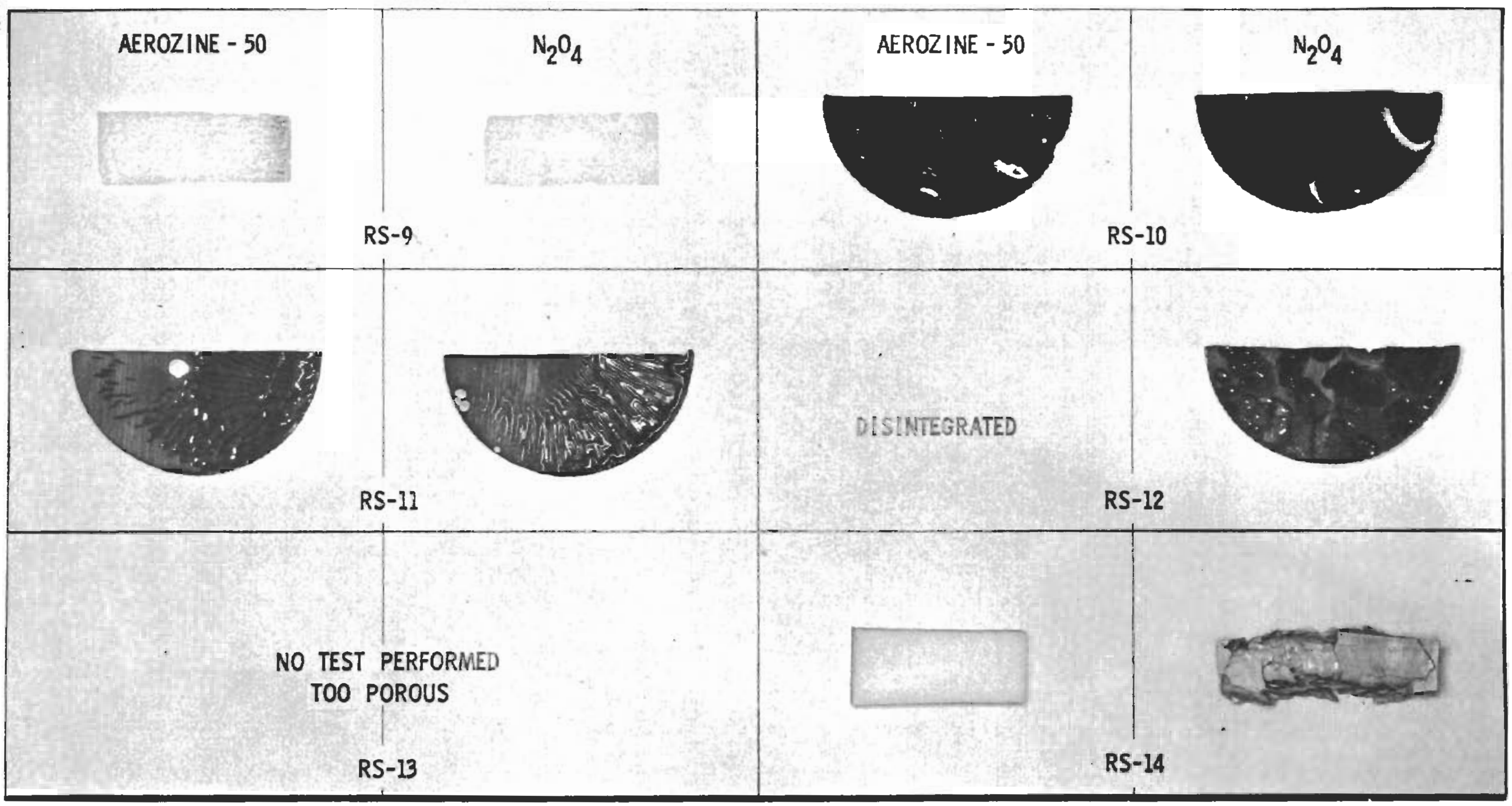
		Condition After Exposure			
		In AeroZINE 50		In N_2O_4	
Resin System	Original Barcol Hardness	Barcol	Appearance	Barcol	Appearance
RS-1	37	41	Rough surface	33	Severely degraded
RS-2	Too Brittle	Too Brittle	Rough surface	Completely degraded	
RS-3	38	43	Rough surface	34	Severely degraded
RS-4	38	32	Good	40	Degraded
RS-5	33	33	Good	28	Slight surface attack
RS-6	41	33	Good	28	Slight surface attack
RS-7	28	Completely degraded		Completely degraded	
RS-8	Too Brittle	Too Brittle	Good	Too Brittle	Good
RS-9	Too Brittle	Too Brittle	Good	Too Brittle	Discolored
RS-10	50	32	Good	54	Good
RS-11	36	31	Good	38	Rough surface
RS-12	34	Completely degraded		20	Soft, mushy
RS-13	Porous	Too porous to test		Too porous to test	
RS-14	67(2)	62(2)	Good	Completely degraded	

(1) At 70°F in AeroZINE 50 and N_2O_4.
(2) Hardness readings made with "D" Durometer.



a. RS-1 Through RS-8

Figure 1. Compatibility of Resin Systems with AeroZINE 50 and Nitrogen Tetroxide (N_2O_4), Appearance After 48-Hour Exposure at Room Temperature



864-690

b. RS-9 Through RS-14

Figure 1---Concluded

The melamine formaldehyde resins, RS-8 and -9, were outstandingly compatible with the propellant but were too brittle for hardness determinations and of doubtful processability as resin binders for filament winding.

The five most promising resin systems as indicated by the spot tests (RS-4, -5, -6, -10, and -11) were cast into 1/8-in. slabs, and flexural-test specimens were prepared for further screening. Specimens were exposed to N_2O_4 and AeroZINE 50 for 7 days at 100^oF; RS-10 was not included in this test because of severe blistering during casting. The remaining four systems decomposed completely during N_2O_4 exposure.

All five resin systems were then exposed for 7 days, at 70^oF, to AeroZINE 50 and N_2O_4. Barcol hardness, flexural strength, and weight determinations were made before and after exposure. The results are summarized in Table 4, and the specimens are shown in Figure 2. This screening study shows that RS-4, -6, and -11 (the novolac epoxies, a polyepoxide, and blends with bisphenol-A epoxies) have the highest degree of compatibility with AeroZINE 50. The RS-6 combination of Kopox 170 with DER 332, cured with BF_3-400, showed fairly good resistance to N_2O_4 even though the flexural strength had dropped by 50%. The phenolic system, RS-10, also was in fairly good condition, although the specimens could not be tested for flexural strength because warpage had occurred during casting. The rest of the resin systems were severely degraded by exposure to N_2O_4.

These tests indicated that RS-4 and -11, two resin systems of similar composition, have the highest degree of compatibility with AeroZINE 50, whereas two other systems, RS-6 and -10, were most resistant to N_2O_4.

C. LONG-TERM EXPOSURE TESTING

After preliminary screening of 14 candidate resin systems, four were subjected to long-term exposure to AeroZINE 50 and N_2O_4 in equipment designed for this program. The equipment included 33 stainless steel cylinders (12 in. long by 3 in. ID) and four constant-temperature baths (16 in. wide by 26 in. long by 12 in. deep). An exposure cylinder is shown in Figure 3, and a constant-temperature bath is shown in Figure 4.

The exposure specimens were 3 by 1/2 by 1/8-in. coupons. The resin system and specimen number were inscribed on each specimen. Five specimens of each resin system, separated by 3/16-in.-thick Teflon spacers, were banded

TABLE 4
RESIN-MATERIAL SCREENING, SEVEN-DAY EXPOSURE(1)

Resin System	Original(2) Barcol Hardness	Original(2) Flexural Strength, psi	After Exposure(2) Barcol Hardness	After Exposure(2) Flexural Strength, psi	After Exposure(2) Weight Change, %	After Exposure(2) Visual Condition
			Exposure in AeroZINE 50			
RS-4	30	15,700	34	16,200	+0.42	No change
RS-5	35	15,860	Major portion of material dissolved			
RS-6	42	10,710	41	5,470	+0.36	Good
RS-10	56	No test(3)	31	No test(3)	+9.40	Faded, no change
RS-11	30	16,280	34	15,540	+0.42	No change
			Exposure in Nitrogen Tetroxide			
RS-4	30	15,700	Major portion of material dissolved			
RS-5	33	15,860	-	Degraded	+5.70	Severely pitted
RS-6	41	10,710	46	4,920	+0.81	Good
RS-10	54	No test(3)	56	No test(3)	+1.39	Slightly faded, good
RS-11	30	16,280	-	Degraded	-	Severely pitted

(1) At 70°F in AeroZINE 50 and N_2O_4.
(2) Property values are averages of three tests.
(3) Specimens too distorted for flexural-strength test.

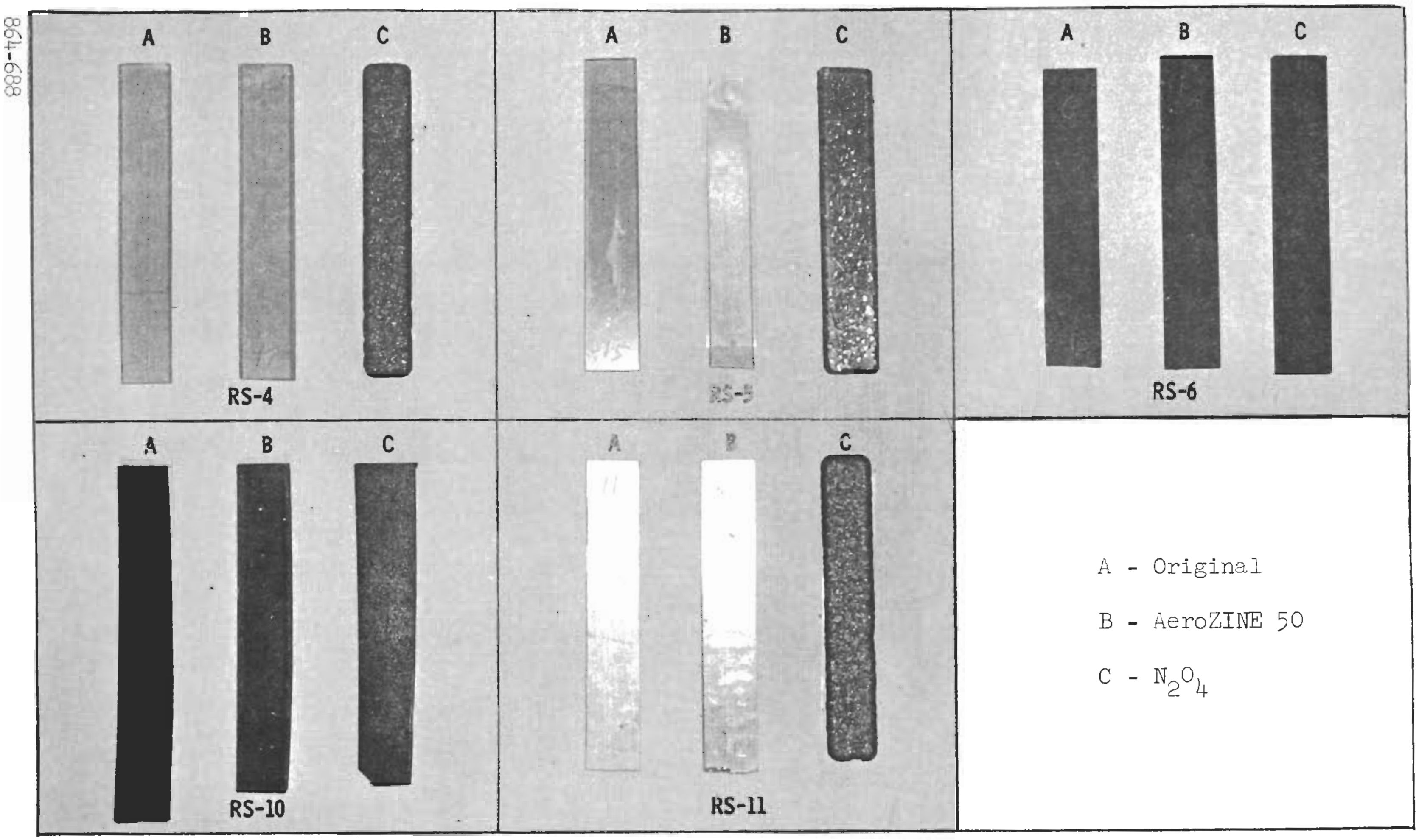


Figure 2. Compatibility of Resin Systems with AeroZINE 50 and N_2O_4, Condition Before and After 7-Day Exposure at $70^{\circ}F$

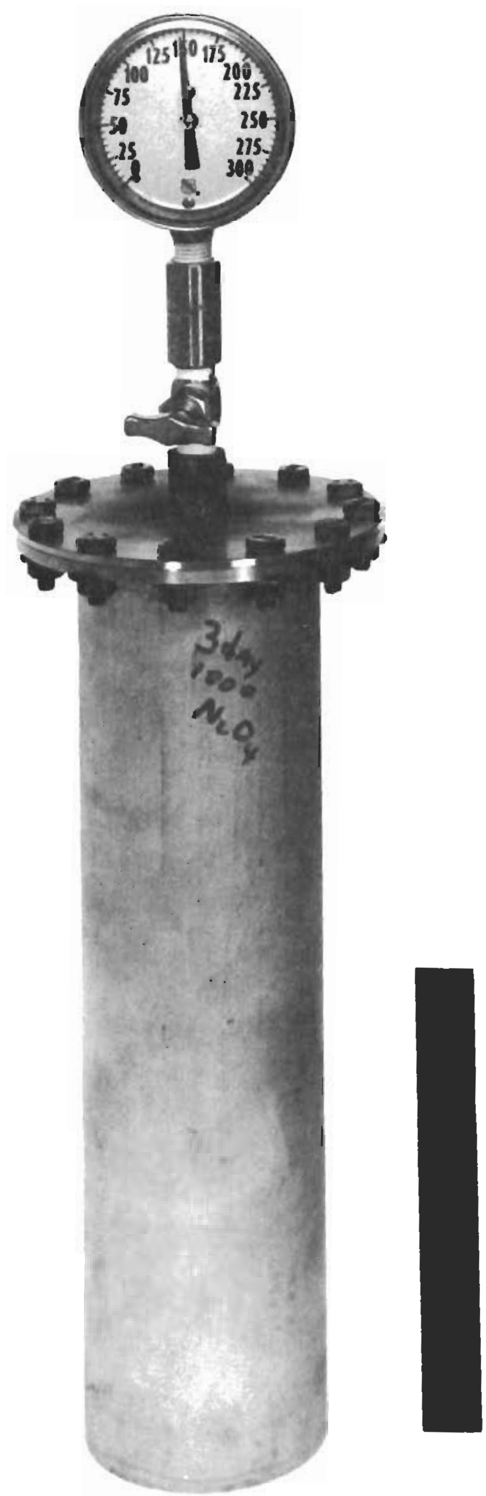


Figure 3. Propellant-Exposure Container

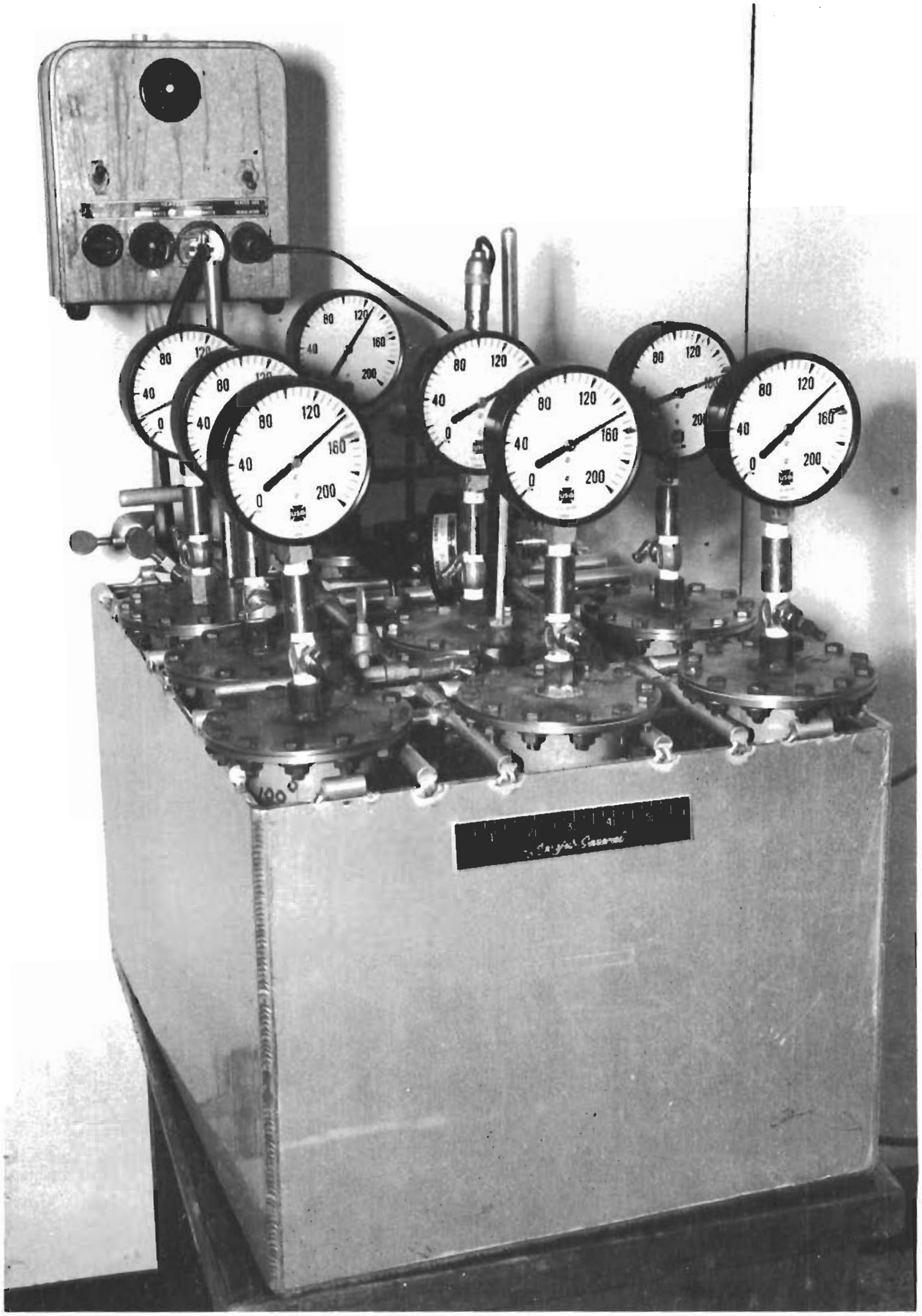


Figure 4. Constant-Temperature Bath with Propellant Containers

together with aluminum wire, and an identification tag of aluminum foil was attached to each bundle (see Figure 5).

Hardness, weight, and volume measurements were made on each specimen before incorporation in the bundle. The bundles were then immersed in the propellant fluid in the exposure cylinders, and the cylinders were pressurized with helium and were placed in the constant-temperature baths for the specified time and temperature. At the end of the exposure period, the specimen bundles were removed from the propellants, decontaminated, and tested for hardness, weight, volume, and flexural strength.

The following test methods of the American Society for Testing Materials (ASTM) were used: D792-50 for weight and volume, E110-55T for hardness, and D790-61 for flexural strength.

The original properties of the resin systems are shown in Table 5. It was considered advisable to include RS-15, a 100% polyepoxide, in the long-term exposure to permit its evaluation without dilution by a bisphenol-A type of resin.

The test results are summarized in Table 6. Progressive deterioration is very evident. Although the degradation was greater in the oxidizer (N_2O_4) than in the fuel (AeroZINE 50), the loss in strength was also great when these resin systems were exposed to the fuel at 100°F. Figures 6 and 7 show the condition of resin specimens after the 30-day exposure in the propellants.

In AeroZINE 50 at 60°F the RS-6 system, a combination of a polyepoxide and a bisphenol-A type of resin, had fairly good properties after the 30-day exposure. After exposure to AeroZINE 50 at 100°F for this same period, however, RS-6 suffered a substantial loss in flexural strength and hardness and was severely eroded. The influence of the polyepoxide in RS-6 was evident in the N_2O_4 exposure, at both 60 and 100°F, inasmuch as the specimen retained fairly good flexural strength despite considerable weight and volume loss.

The phenolic resin system, RS-10, showed low resistance to AeroZINE 50; all specimens dissolved on exposure. RS-10 retained good flexural strength during short exposures to N_2O_4. It also retained its strength when exposed to N_2O_4 at 60°F for 30 days, but was severely eroded and had a very high weight and volume loss at 100°F in N_2O_4 for this longer period.

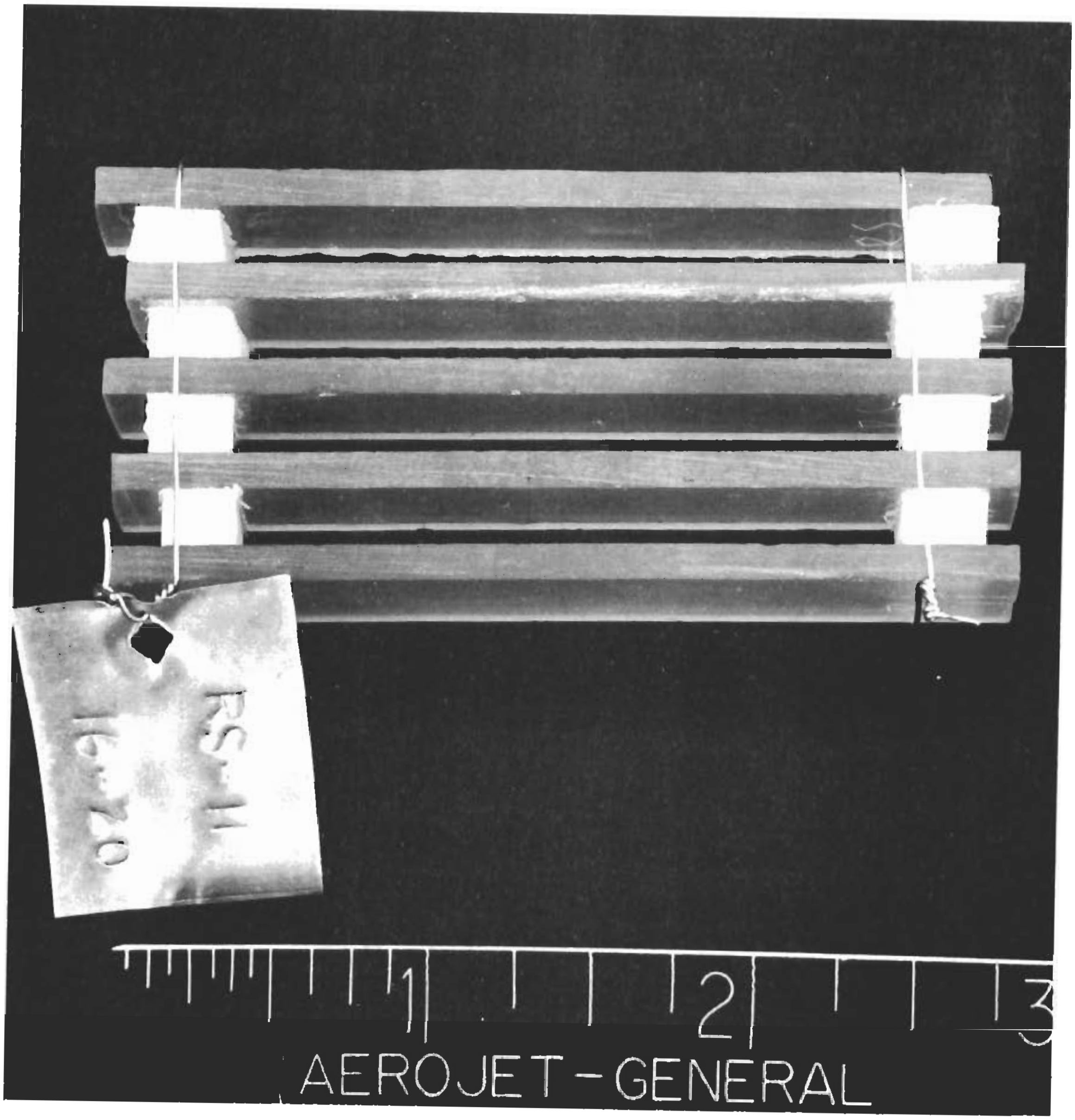


Figure 5. Specimen Bundles

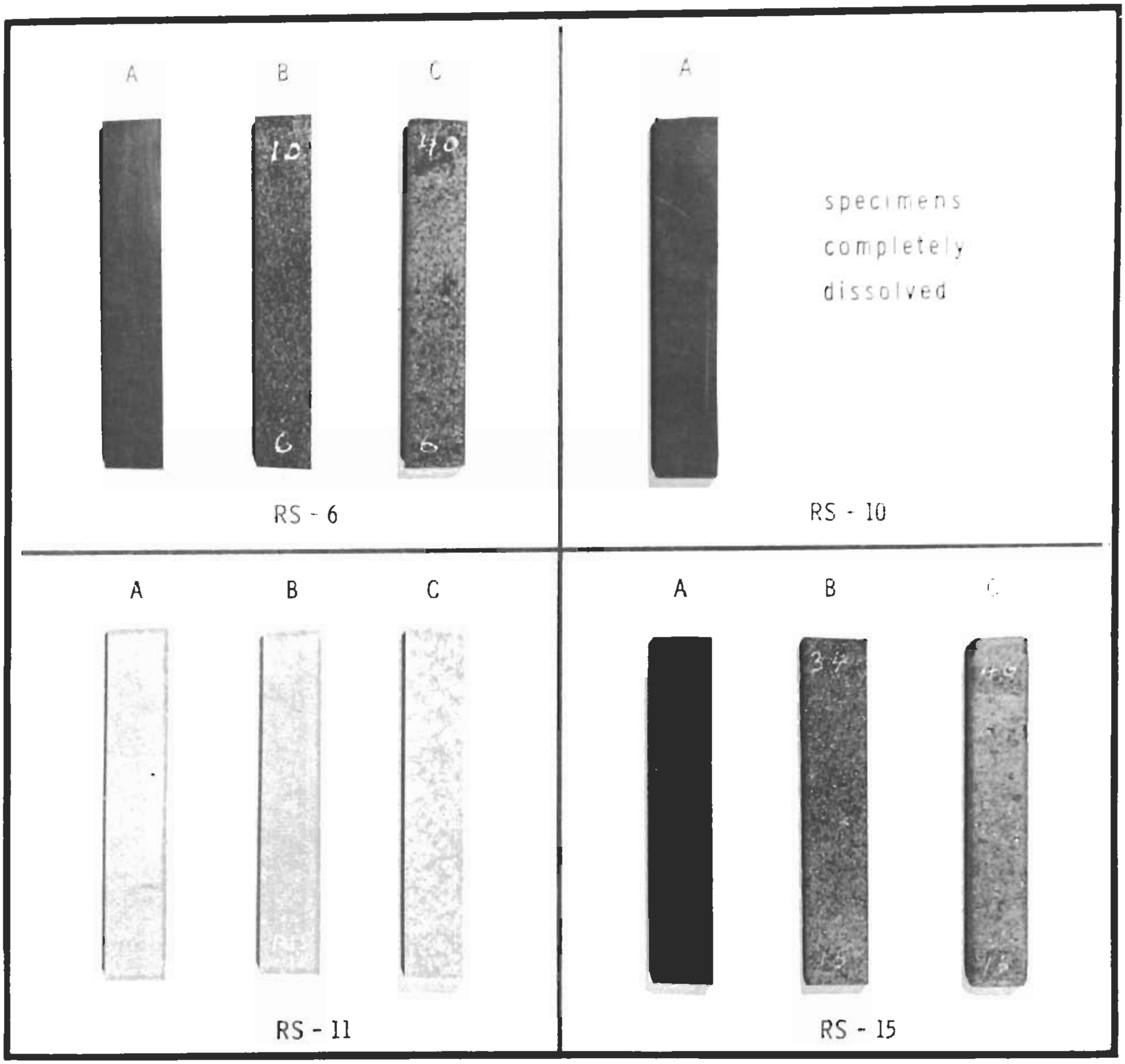
TABLE 5
ORIGINAL PROPERTIES OF RESIN SYSTEMS

RS-6			RS-10			RS-11			RS-15		
Barcol Hardness	Flexural Strength psi	Specific Gravity	Barcol Hardness	Flexural Strength psi	Specific Gravity	Barcol Hardness	Flexural Strength psi	Specific Gravity	Barcol Hardness	Flexural Strength psi	Specific Gravity
38	11,011	1.28	40	14,597	1.24	36	11,016	1.24	44	9,633	1.29
38	10,444	1.28	40	12,803	1.24	36	8,287	1.24	41	5,250	1.29
38	13,701	1.27	37	14,574	1.24	36	12,604	1.24	44	6,941	1.29
40	9,643	1.28	36	11,063	1.24	36	8,066	1.24	43	5,455	1.29
39	14,668	1.28	40	18,271	1.24	36	12,631	1.24	44	7,701	1.29
37	12,114	1.28	38	11,500	1.24	34	10,866	1.24	44	6,176	1.29
40	10,246	1.28	40	17,872	1.24	34	13,139	1.24	45	4,627	1.29
38	(5,486)[1]	1.28	38	18,553	1.24	34	11,299	1.24	45	6,307	1.29
38	10,216	1.28	38	17,703	1.24	35	12,918	1.24	43	5,053	1.29
37	12,139	1.28	39	13,944	1.28	35	10,027	1.24	44	-	-
Average											
38	11,576	1.28	39	15,088	1.24	35	11,085	1.24	44	6,349	1.29

(1) Defective specimen, not used in calculation of average.

TABLE 6
PROPERTIES OF RESIN SYSTEMS AFTER EXPOSURE TO PROPELLANTS

Resin System	Propellant	Exposure Time Days	Exposure Temp °F	Weight Change, %	Volume Change, %	Flexural Strength psi	Flexural Strength % Retention	Hardness Barcol	Hardness Pts. Change	Appearance
RS-6	(Kopox 170/DER 332/BF_3-400)					11,576 (control)		38 (control)		
	Aerozine-50	7	60	+0.4	+0.8	11,060	96.0	45	+7	Good - no crazing
	Aerozine-50	30	60	+1.2	+1.4	5,432	47.0	44	+6	Good - slight erosion
	Aerozine-50	90	60	+2.6	+2.9	7,696	66.3	42	+4	Good - slight pitting
	Aerozine-50	7	100	+2.4	+3.0	9,264	80.4	40	+2	Good - slight crazing
	Aerozine-50	30	100	+5.2	+6.2	2,794	24.1	28	-10	Fair - slight erosion
	Aerozine-50	90	100	+5.3	+6.5	4,094	35.4	19	-19	Poor - severe erosion
	N_2O_4	7	60	+0.6	+0.7	8,860	76.7	46	+8	Good - no crazing
	N_2O_4	30	60	+1.1	+1.6	3,005	26.0	44	+6	Good - no crazing
	N_2O_4	90	60	-0.9	-1.0	5,394	46.7	42	+4	Fair - severe crazing
	N_2O_4	3	100	+1.1	+1.3	2,839	24.5	40	+2	Good - no crazing
	N_2O_4	10	100	+1.5	+1.1	2,959	25.5	46	+8	Fair - slight erosion
	N_2O_4	30	100	-17.9	-18.2	3,786	32.7	47	+9	Fair - slight erosion
RS-10	(U.S. Poly 46 - Phenol Formaldehyde)					15,088 (control)		39 (control)		
	Aerozine-50	7	60	All specimens completely dissolved						
	Aerozine-50	30	60	All specimens completely dissolved						
	Aerozine-50	90	60	All specimens completely dissolved						
	Aerozine-50	7	100	All specimens completely dissolved						
	Aerozine-50	30	100	All specimens completely dissolved						
	Aerozine-50	90	100	All specimens completely dissolved						
	N_2O_4	7	60	+2.0	+1.5	8,623	57.2	47	+8	Good - no crazing
	N_2O_4	30	60	-6.2	-7.2	9,557	63.3	48	+9	Good - slight crazing
	N_2O_4	90	60	-21.4	-18.3	7,220	47.8	44	+5	Poor - severe erosion
	N_2O_4	3	100	-2.6	-49	6,666	44.2	52	+13	Good - slight crazing
	N_2O_4	10	100	-11.5	-10.5	6,468	42.8	54	+15	Fair - slight crazing
	N_2O_4	30	100	-65.6	-66.2	3,688	24.5	Too rough to test		Poor - severe erosion
RS-11	(DEN 438/BF_3-400)					11,085 (control)		35 (control)		
	Aerozine-50	7	60	+0.6	+0.2	10,742	97.0	40	+5	Good - no crazing
	Aerozine-50	30	60	+1.0	+0.3	10,497	94.5	43	+8	Good - no crazing
	Aerozine-50	90	60	+2.2	+1.5	9,244	83.5	40	+5	Good - slight pitting
	Aerozine-50	7	100	+1.7	+0.9	10,323	93.2	40	+5	Good - no crazing
	Aerozine-50	30	100	+4.5	+4.3	3,757	34.9	25	-10	Fair - severe crazing
	Aerozine-50	90	100	+9.8	+9.4	5,110	47.6	19	-16	Fair - slight blistering
	N_2O_4	7	60	+1.4	0.0	7,957	73.4	42	+7	Good - no crazing
	N_2O_4	30	60	+2.3	+0.4	2,402	21.7	36	+1	Poor - severe crazing
	N_2O_4	90	60	+2.1	-0.2	1,567	14.2	19	-16	Poor - severe crazing
	N_2O_4	3	100	+2.6	-1.9	2,017	18.2	35	0	Good - dark color
	N_2O_4	10	100	+3.7	+0.4	2,070	18.7	40	+5	Poor - severe crazing
	N_2O_4	30	100	-52.5	-54.2	1,939	17.5	Too rough to test		Poor - severe erosion
RS-15	(Kopox 170/BF_3-400)					6,349 (control)		44 (control)		
	Aerozine-50	7	60	+0.8	+0.2	11,097	175.0	49	+5	Good - no crazing
	Aerozine-50	30	60	+1.2	+0.2	5,350	84.2	44	0	Good - slight erosion
	Aerozine-50	90	60	+3.2	+2.3	7,695	121.0	41	-3	Good - slight pitting
	Aerozine-50	7	100	+2.4	+1.1	7,603	120.0	44	0	Good - slight crazing
	Aerozine-50	30	100	+6.0	+7.1	4,707	74.4	31	-13	Fair - severe erosion
	Aerozine-50	90	100	+9.6	+10.0	4,236	67.0	22	-22	Poor - severe erosion
	N_2O_4	7	60	+0.8	-0.7	7,511	118.5	50	+6	Good - no crazing
	N_2O_4	30	60	+0.8	-0.2	3,733	58.8	49	+5	Good - no crazing
	N_2O_4	90	60	-1.1	-1.0	4,182	66.0	50	+6	Good - slight pitting
	N_2O_4	3	100	+0.7	-2.3	4,477	70.6	47	+3	Good - no crazing
	N_2O_4	10	100	+1.4	+0.4	3,294	52.0	52	+8	Fair - slight crazing
	N_2O_4	30	100	-8.5	-9.7	4,408	69.5	55	+11	Fair - slight erosion

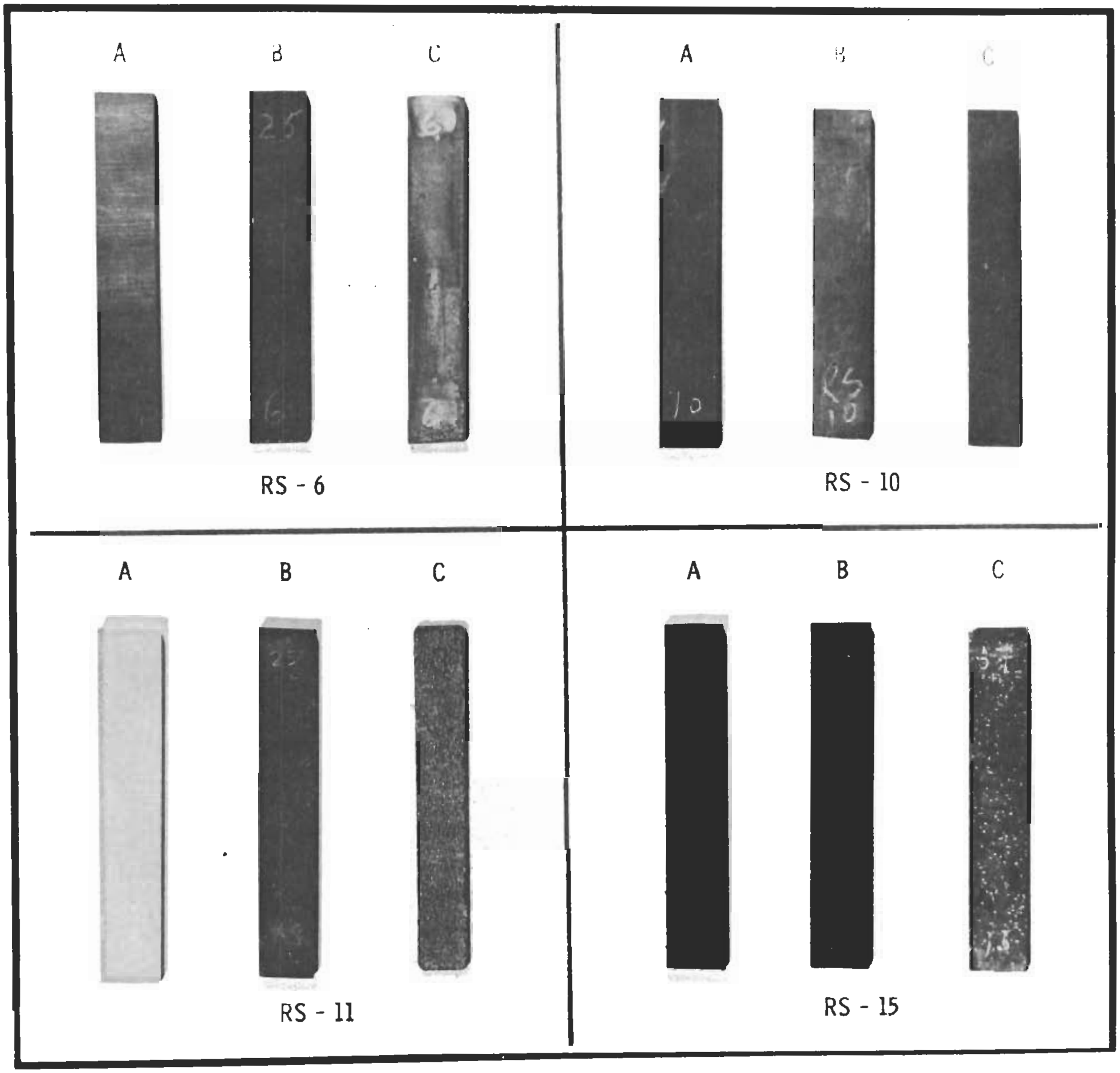


A - Original

B - One Month in AeroZINE 50 at 60°F and 150 psi

C - One Month in AeroZINE 50 at 100°F and 150 psi

Figure 6. Compatibility of Resin Systems with AeroZINE 50, Condition Before and After 1-Month Exposure at 60 and 100°F



A - Original

B - One Month in N_2O_4 at 60^oF and 150 psi

C - One Month in N_2O_4 at 100^oF and 150 psi

Figure 7. Compatibility of Resin Systems with N_2O_4, Condition Before and After 1-Month Exposure at 60 and 100^oF

The novolac resin system, RS-11, demonstrated good resistance to AeroZINE 50 at 60°F. At 100°F in AeroZINE 50 it retained fairly good flexural strength but showed severe crazing. In N_2O_4 it was severely degraded at both exposure temperatures.

The polyepoxide system, RS-15, was the most resistant to both AeroZINE 50 and N_2O_4. Although severe erosion was noted after exposure to AeroZINE 50 for 30 days at 100°F, the flexural-strength retention was good. The low initial flexural strength indicates that the RS-15 system was not completely cured. The activation effect of a chemically basic type of material is shown by the increase in RS-15 flexural strength after the 7-day exposure to AeroZINE 50.

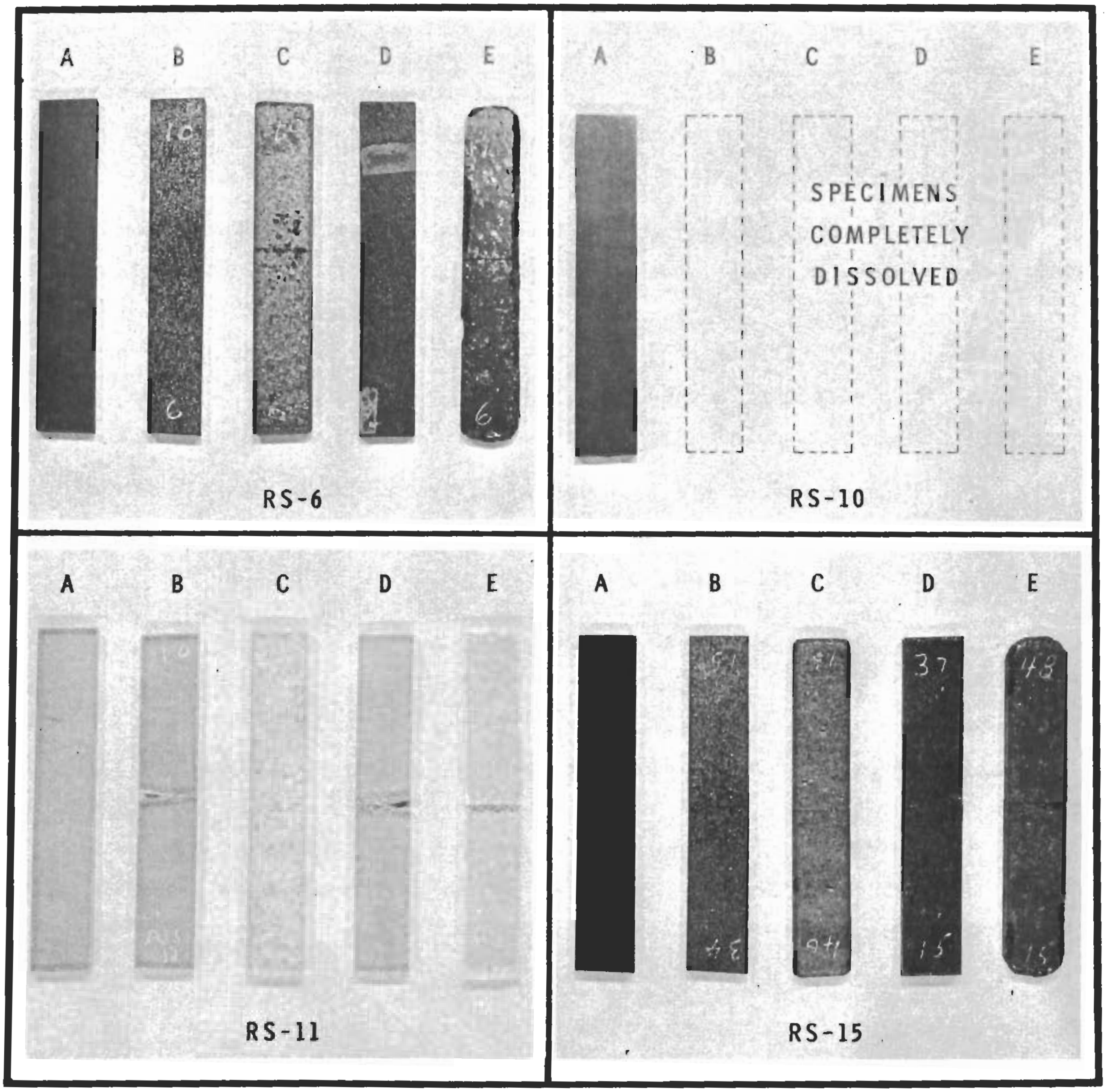
The 90-day tests of the four candidate systems showed that although they had lost considerable strength during this severe long-term exposure to propellants, they still retained a fair degree of integrity. Figures 8 and 9 show their appearance after exposure to AeroZINE 50 and N_2O_4, respectively.

D. CONCLUSIONS

The results of the exposure tests of resin systems confirmed observations by earlier investigators that organic materials, such as epoxy and phenolic resins, do not have a high degree of compatibility with rocket fuels and oxidizers. It was found however, that the novolac and polyepoxy resins resist the effects of these propellants for a limited period and could be expected to tolerate spillage and short-term contact.

It may be noted that RS-6 - the combination polyepoxy, bisphenol-A, epoxy-type, resin system - retained fairly good physical properties after the 90-day exposure to AeroZINE 50 at 60°F. The specimens, however, were severely eroded when the fuel exposure was conducted at 100°F. Exposure to N_2O_4 at 60°F for 90 days produced considerable crazing of RS-6 and a 53% loss in flexural strength; when exposed at 100°F in N_2O_4, RS-6 was slightly eroded and lost considerable weight after 30 days. The addition of DER 332 to Kopox 170 reduced the resistance of the Kopox 170 to N_2O_4 without appreciably improving the properties on exposure to AeroZINE 50 propellant.

The phenol formaldehyde resin system, RS-10, appeared to crumble and disintegrate when exposed to AeroZINE 50. It eroded badly even in N_2O_4, although its flexural strength remained fairly high. It was severely eroded by



A - Original

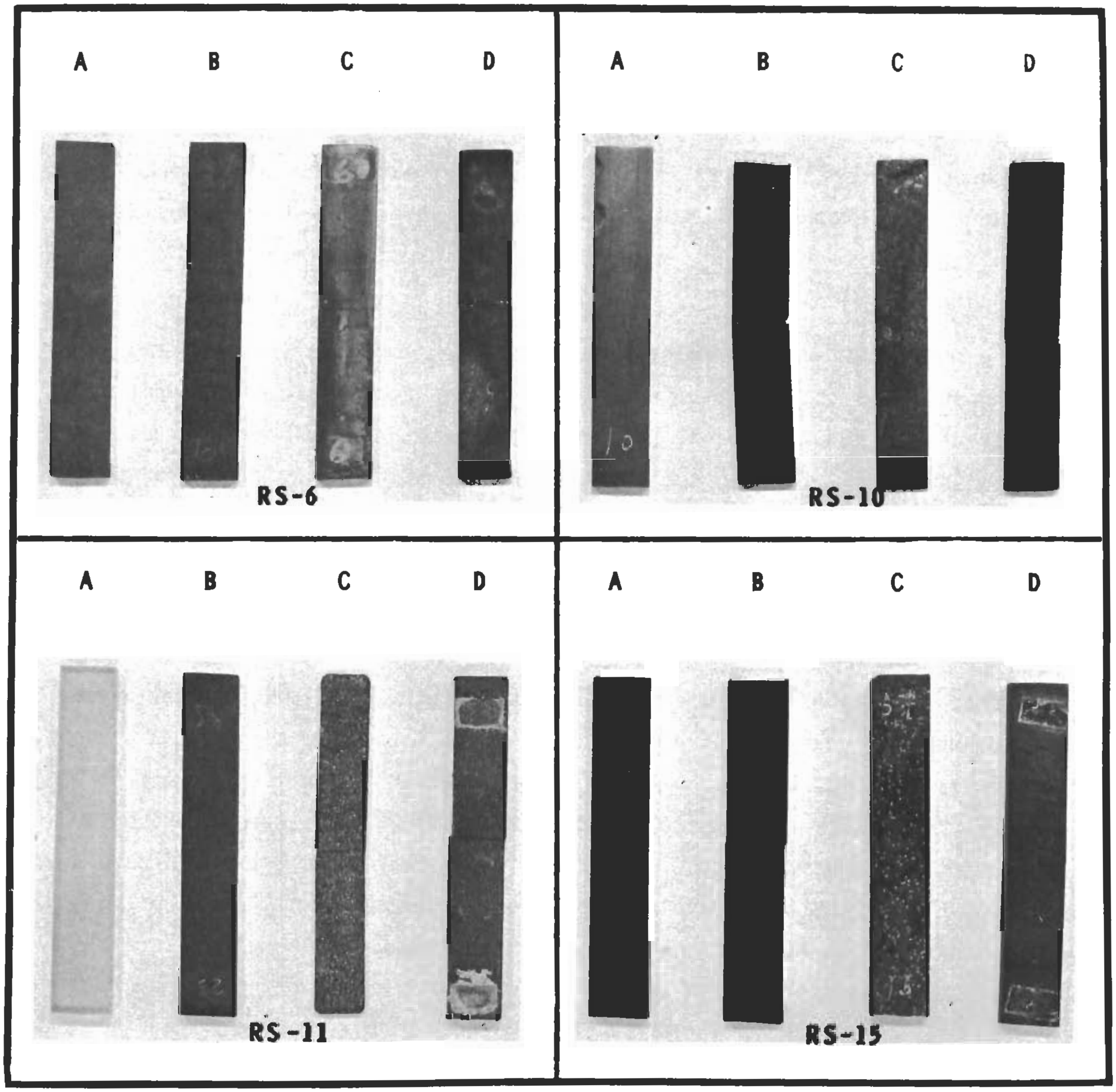
B - One Month in AeroZINE 50 at 60°F and 150 psi

C - One Month in AeroZINE 50 at 100°F and 150 psi

D - Three Months in AeroZINE 50 at 60°F and 150 psi

E - Three Months in AeroZINE 50 at 100°F and 150 psi

Figure 8. Compatibility of Resin Systems with AeroZINE 50, Condition Before and After 3-Month Exposure at 60 and 100°F



A - Original

B - One Month in N_2O_4 at 60°F and 150 psi

C - One Month in N_2O_4 at 100°F and 150 psi

D - Three Months in N_2O_4 at 60°F and 150 psi

Figure 9. Compatibility of Resin Systems with N_2O_4, Condition Before and After 1-Month and 3-Month Exposures

N_2O_4 and suffered a substantial weight loss after 30 days at 100°F and 90 days at 60°F. Because of the unfavorable results in both fuel and oxidizer, RS-10 was not considered a suitable candidate for the subscale chambers.

The novolac resin system, RS-11, retained fairly good flexural strength when exposed to AeroZINE 50 at both 60 and 100°F, although considerable erosion was evident after long exposures. Its flexural strength was substantially reduced on exposure to N_2O_4, but severe erosion did not occur except on the 30-day exposure at 100°F.

The polyepoxy resin system, RS-15, although low in original flexural strength, retained this property very well after exposure to the propellants. As compared with RS-11, however, its actual strengths were somewhat lower and it eroded somewhat more when exposed to AeroZINE 50. In resistance to N_2O_4, RS-15 was substantially better than any other resin system evaluated. On a hand-bending test, RS-15 specimens appeared to be somewhat brittle. This property is, of course, important to consider in the expansion and contraction of filament-wound structures under thermal and pressure cycling conditions.

After 90 days of exposure testing, it was concluded that a high degree of resistance to rocket fuels and oxidizers cannot be expected of the types of resins used in filament-winding applications. To ensure a high degree of structural integrity in filament-wound tankage for storable propellants, an impervious type of liner is therefore required. This study of resin materials has shown, however, that a novolac epoxy resin system, RS-11, and a polyepoxide resin system such as RS-15 do have moderate resistance and could tolerate minor spillage and exposure to the propellants.

IV. PHASE II - STUDY OF LINER MATERIALS

A. LITERATURE SURVEY

The literature indicates that major effort has been devoted to a search for elastomeric and plastic liner materials that are compatible with rocket fuels and oxidizers. Several materials have been found useful for various applications involving contact with the less corrosive propellants. After long exposure and exposure at higher-than-ambient temperatures, however, the polymeric materials exhibit swelling, loss of strength, and propellant absorption. The investigators who have made these studies concur in their findings that no organic lining material is completely impermeable to the propellants. For containment of the more corrosive fluids such as N_2O_4, pentaborane, and ClF_3, a corrosion-resistant metallic liner is indicated. The literature survey is briefly summarized below, and the recommended liner materials are indicated for each propellant.

1. AeroZINE 50

Fluorocarbons (e.g., Teflon and Kynar) resist attack by AeroZINE 50 for long periods, but are permeable to this fluid (Reference 1). Kel-F was also found to be resistant, but showed stress cracking after long exposures (Reference 1). Resin-cured butyl compounds have been found compatible with AeroZINE 50, but swell slightly after long exposures and are permeable (Reference 5). Diamine nylons have shown excellent resistance (Reference 7). Polyethylene and polypropylene absorb AeroZINE 50, but no chemical attack was observed (Reference 7). Ethylene propylene rubber and cis-1,4-polybutadiene rubber compounds showed good compatibility with hydrazine-type fuels (Reference 5). Most aluminums, stainless steels, nickel, Chromel, and Monel are compatible with hydrazine-type propellants (Reference 8). Aluminum-Teflon laminates (Reference 9) and aluminum-butyl rubber laminates are reported to withstand attack by AeroZINE 50, and to show no permeability to it (Reference 5).

2. Nitrogen Tetroxide

Although Teflon TFE (tetrafluoroethylene) and FEP (fluorinated ethylene polymer) are the most compatible of the plastic materials when exposed to N_2O_4, they absorb and expel it by outgassing when exposed to a vacuum (Reference 7). These plastic films are also very permeable to N_2O_4

(Reference 1). Aluminum alloys 1100, 2024, and 5052, and American Iron and Steel Institute (AISI) Type 303, 304, and 347 stainless steel (SS) were rated as good in N_2O_4 service (Reference 8). Other investigators, however, do not consider Al 2024 as acceptable when the water content in N_2O_4 is greater than 0.2% (References 10, 11, and 12). The stainless steels are resistant regardless of the water content (Reference 11).

3. Pentaborane

Of the nonmetallic materials, Teflon, Kel-F-5500, and Viton A and B are considered compatible with pentaborane (References 3 and 4). The following have been found compatible (References 3 and 4): 302 SS, 304 SS, 321 SS, 347 SS, and 18-8 SS; aluminum alloys 5052-S, 6061-T6, 7075-T6, 2024-T3, 3003-H14, and 356-T6; and Monel, nickel, magnesium, titanium, copper, brass, and Hastelloy.

4. Chlorine Trifluoride

No nonmetallic materials are recommended for use with ClF_3 because of its extremely corrosive nature (Reference 6). The compatible metals include the 300 series stainless steels; aluminum alloys 356, 1100, 2024, 5052, 6061, 6063; and chromium-plated steel, copper, nickel, Monel, K-Monel, Rene 41, nickel-base Superalloy, and indium (Reference 13). In one study no evidence of stress corrosion was found on any of the exposed metals either by visual examination or dye-penetrant inspection (Reference 6); it was also found that passivation by ClF_3 was unnecessary for reducing the corrosion of properly cleaned metals. An Aerojet study of metals compatible with hydrazine N_2O_4, B_5H_9, ClF_3, and liquid fluorine concluded that " ... the metal approaching complete compatibility with all propellants under consideration is Type 347 stainless steel ..." (Reference 14).

B. EXPERIMENTAL PROGRAM

1. Corrosion Testing of Metallic Materials

Metallic liners appear to be required for the more corrosive rocket fluids. Very thin linings are necessary, however, in order to achieve maximum weight economy in filament-wound tankage. The lining must tolerate the expansion and contraction of the tankage during temperature and pressure cycling. It must also satisfy the design requirements of glass-filament-wound structures.

Inasmuch as the resin studies indicated that metallic liners are required for storable propellants, the corrosion resistance of these metals is very important. To substantiate results reported in the literature, a study was therefore undertaken to determine the resistance of the candidate metallic liner materials to N_2O_4 corrosion. Two specimens of each of the metals (2 by 7/8 by 0.012 in.) were weighed and were then immersed in N_2O_4 at 160°F for 163 hours. At the end of this period, they were reweighed and examined for evidence of corrosion and discoloration. The corrosion rate, in mils per year, was calculated from the weight-loss measurements.

In general, the results confirm the observations reported in the literature. The Teflon peeled away from the aluminum in the laminated construction; this peeling would be very unsafe if it occurred in a propellant tank. Dr. A. Church of Swedlow, Inc. advised in a private communication, however, that the normal procedure in the fabrication of metal liners is to expose the metal side of the aluminum-Teflon laminate to the fuel. He stated that a coating, such as Teflon, over the metal liner increased the service life of the liner. The corrosion rate of Al 2024 was definitely measurable and indicates the presence of moisture in the N_2O_4; a slight discoloration on the surface of the alloy was further evidence of corrosion. The high corrosion rate of the Monel metal in this test would rule out its use for N_2O_4 containment, but Al 1100, 304 SS, and 347 SS appear to be acceptable for liner use. The test results are shown in Table 7.

2. Compatibility Testing, Polymeric Films

Although nonmetallic materials cannot be considered as candidates for tank liners for the more corrosive propellants, it was believed that they might be useful as laminates with metal foils. Compatibility tests of Teflon, Kynar, polyethylene, and propylene films were made to determine the effect of exposure to AeroZINE 50, N_2O_4, and ClF_3 on the strength properties and permeability of the polymeric liner materials (see Table 8).

The compatibility of these materials (along with a butyl rubber compound) to ClF_3 was studied in a brief spot test. Approximately 1 to 2 cc of liquid ClF_3 was allowed to flow across the test material. The polyethylene and polypropylene immediately ignited on contact and burned fiercely until they were consumed. The butyl rubber did not ignite, but it was badly eroded and became very brittle.

TABLE 7

LINER-MATERIAL SCREENING, CORROSION OF METALS BY N_2O_4

	Thickness, in.	Time, hours	Temp, °F	Corrosion Rate mils/year	Appearance of Specimens
Aluminum-Teflon laminate	0.002/0.004	163	160	None	Teflon peeled
Al 1100-0	0.015	163	160	None	No change
Al 2024-T3	0.015	163	160	0.5	Slight discoloration
304 SS	0.015	163	160	Negligible	No change
347 SS	0.015	163	160	Negligible	No change
Monel	0.015	163	160	4.03	Slight discoloration

TABLE 8

PROPERTIES OF LINER MATERIALS AFTER EXPOSURE TO PROPELLANTS

Liner Material	Propellant	Exposure: Time Days	Exposure: Temp °F	Tensile Strength: psi	Tensile Strength: % Retention	Elongation: %	Elongation: % Retention	Hardness: "B" Duro	Hardness: Pts. Change	Appearance
L-1	(Teflon)			3,439 (control)		292 (control)		83 (control)		
	Aerozine-50	7	100	3,037	88.2	235	80.5	81	-2	Good - no change
	Aerozine-50	30	100	4,436	119.0	344	118.0	82	-1	Good - no change
	N_2O_4	7	100	2,854	83.0	262	89.8	83	0	Good - no change
	N_2O_4	30	100	4,127	120.0	244	83.5	75	-8	Good - no change
	Chlorine trifluoride	7	100	3,563	104.0	284	97.2	90	+7	Good - no change
	Chlorine trifluoride	30	100	3.873	112.8	276	94.5	90	+7	Good - no change
L-2	(Kynar)			5,933 (control)		24 (control)		91 (control)		
	Aerozine-50	7	100	4,800	81.0	15	62.5	89	-2	Good - no change
	Aerozine-50	30	100	6,533	110.0	258	100+	90	-1	Good - no change
	N_2O_4	7	100	4,943	83.2	264	100+	88	-3	Fair - discolored
	N_2O_4	30	100	6,867	115.0	12	50	80	-11	Poor - severely discolored
	Chlorine trifluoride	7	100	6,100	102.7	91	100+	95	+4	Good - no change
	Chlorine trifluoride	30	100	5,733	96.6	30	125.0	94	+3	Good - no change
L-3	(Polyethylene)			1,835 (control)		450 (control)		75 (control)		
	Aerozine-50	7	100	2,200	126.0	603	132.0	75	0	Good - no change
	Aerozine-50	30	100	2,142	117.0	577	124.0	60	-15	Good - no change
	N_2O_4	7	100	All specimens too badly degraded to test						
	N_2O_4	30	100	All specimens too badly degraded to test						
L-4	(Polypropylene)			4,258 (control)		430 (control)		94 (control)		
	Aerozine-50	7	100	2,845	66.8	*		93	-1	Good - no change
	Aerozine-50	30	100	4,894	115.0	20	4.6	94	0	Fair - slightly brittle
	N_2O_4	7	100	All specimens too badly degraded to test						
	N_2O_4	30	100	All specimens too badly degraded to test						

*Less than 10% elongation.

Preliminary screening tests indicated that the fluoropolymers, and also ethylene and propylene plastic films, have moderate resistance to rocket fuels. Teflon TFE and Kynar (polyvinylidene fluoride) reportedly have good resistance to both AeroZINE 50 and N_2O_4, whereas Kel-F (polychlorotrifluorethylene) has shown severe stress-corrosion cracking when exposed to them (Reference 1). Polyethylene and polypropylene were found to absorb AeroZINE 50, but no chemical attack was observed (Reference 7). Although cured elastomeric compounds, such as butyl and ethylene-propylene rubber, have been found to be fairly resistant to AeroZINE 50, the extraction of organic curing agents, antioxidants, and softeners by the propellant would create serious contamination problems. The elastomeric materials are also known to be permeable to both AeroZINE 50 and N_2O_4 (Reference 5).

Laminates of a plastic film and a metal foil have been given some consideration as liners (Reference 5). The metal foil acts as a barrier for the propellant, and the plastic film serves as a carrier for the foil. The laminate-type liner presents two problems however: (a) If the plastic film (e.g., Teflon) is used on the side next to the propellant, fuel or oxidizer permeation causes the film to peel away from the foil, and (b) no satisfactory joining procedure has been found to bond the laminate sections together or to the metal bosses of the tankage. Various types of folding designs have been tried but without success (Reference 2).

Chemical compatibility studies were made on the following candidate liner materials: Teflon, Kynar, polyethylene, and polypropylene. Tensile and permeability specimens were immersed in the propellants for 7- and 30-day periods at $100^{\circ}F$. Tensile, elongation, and hardness measurements were made in accordance with the following ASTM test methods: D638-58T for tensile strength and elongation, and D1706-59T for hardness (Durometer). The test results, summarized in Table 8, are discussed below.

a. Exposure to AeroZINE 50 Propellant

All four liner materials were compatible with this fuel for 7- and 30-day exposures at $100^{\circ}F$. Polypropylene showed some evidence of embrittlement after 30-day immersion, but the tensile-strength retention was good. Polyethylene was softened as a result of a 30-day exposure, but suffered no loss in tensile strength.

b. Exposure to N_2O_4

The fluorocarbon polymers, Teflon and Kynar, demonstrated good compatibility with N_2O_4 at 100°F, although the Kynar turned brown. The discoloration disappeared within a few days after the test. The results of elongation tests of the Kynar specimens were very erratic and thus were not meaningful for comparisons. The oxidizer caused severe degradation of both the polyethylene and polypropylene films.

c. Exposure to ClF_3

The exposure of Teflon and Kynar to ClF_3 for 7 and 30 days at 100°F produced slight hardening of the films, but their tensile strengths were not reduced. In the dismantling of one of the exposure containers, slight residual pressure forced a small amount of ClF_3 over the flat Teflon gasket; this produced immediate breakdown, and a section of the gasket was consumed. The incident demonstrates the extremely corrosive nature of ClF_3 under dynamic conditions.

d. Permeability Testing

Permeability tests were made on the liner materials before and after various periods of exposure to AeroZINE 50 at 100°F. After several modifications of test equipment and procedures, a method was developed that produced reasonably consistent results. The test equipment and procedure are described below, and the equipment is shown in Figures 10 and 11.

The test samples consisted of 2-in.-dia disks stamped from sheet stock. They were clamped between the mating surfaces of the two sections of the test fixture shown in Figure 10. A small portion of the test fluid (e.g., AeroZINE 50) was placed in the longer portion of the test chamber before the disk was clamped in place.

A glass U-tube trap was connected to the section containing the supporting screen. The test chamber was placed in a vertical position with the U at the bottom side. In this manner the disk was covered with the liquid phase of the test fluid. A pressure line was connected to the upper fitting and helium at a pressure of 150 psig was applied to the test fluid. A vacuum line was attached to the free arm of the U-tube and all air was pumped out. A Dewar flask of liquid nitrogen was placed so that the entire radius of the U

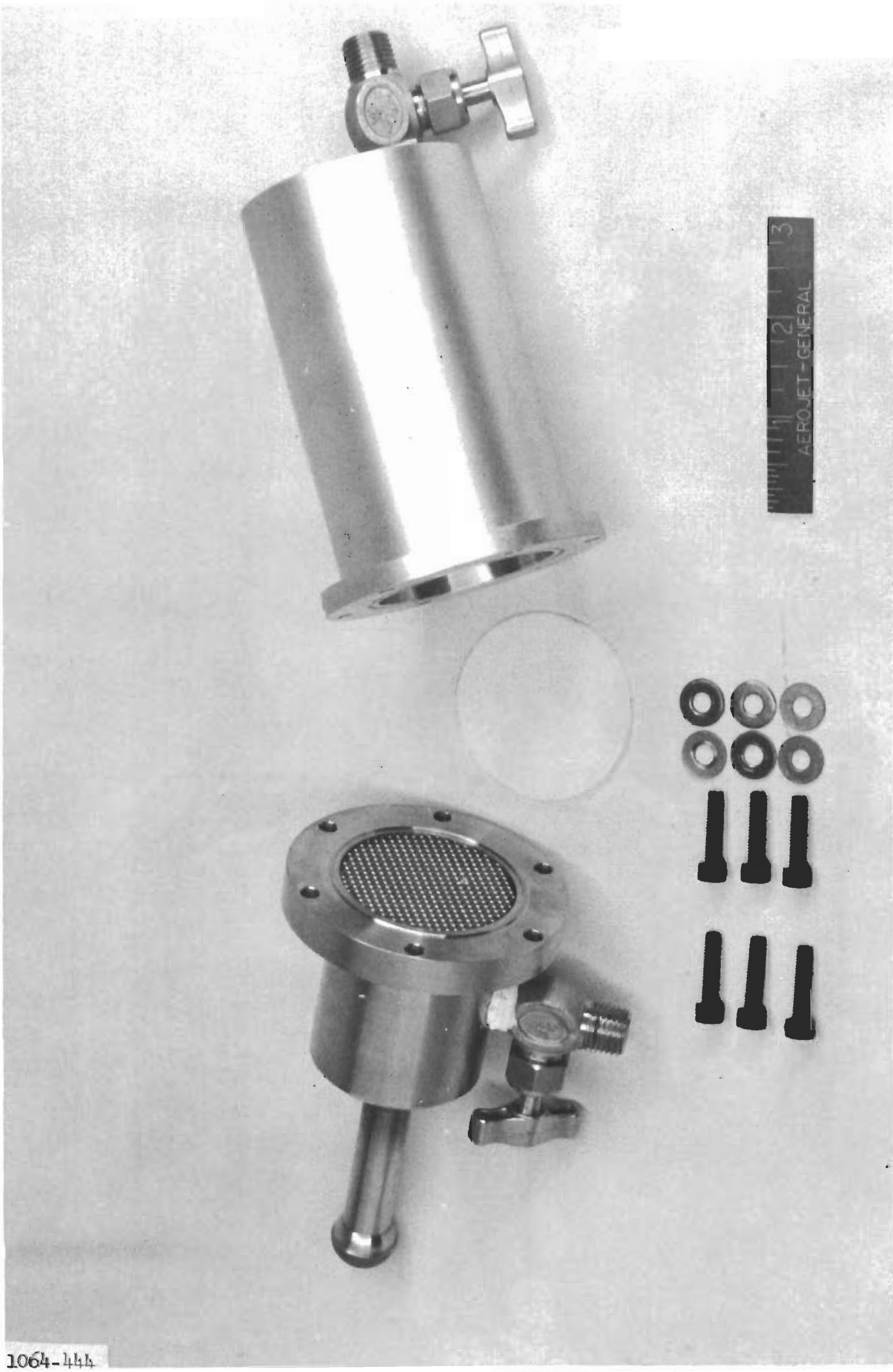


Figure 10. Permeability Cell

Figure 11. Sampler for Permeability Cell

and approximately 1 in. of the arms were immersed. Stopcocks on each end of the U trap permitted easier control of moisture condensation and air contamination. After the exposure, the stopcocks were closed and the trap was removed. Fifty microliters of n-butyl alcohol were added to wash down the condensed AeroZINE 50 from the walls of the tube. A portion of the solute was introduced into a chromatograph. During this part of the test, determinations were made at 194°F using a 6-ft, 1/4-in. column of 5% quadrol on a Teflon substrate. The area contained under the resulting peak was compared with a calibration curve prepared with measured quantities of AeroZINE 50 in the aliquot. The total quantity of AeroZINE 50 was then determined, and this value was reduced to units of milligrams per square inch per hour.

This method was used to determine the permeation of AeroZINE 50 through a 10-mil Teflon film for a total of 32 hours. The cumulative amount passing through the film is plotted against time in Figure 12. The values fall so nearly on a straight line that it is safe to postulate that the permeability rate is directly proportional to time. Extrapolation of this curve to any given time should be valid for use in predicting the amount of AeroZINE 50 that would permeate through the film.

Permeation to the extent indicated by these studies would cause degradation and weakening of the resin binder used in the filament-wound case. This degradation would be particularly rapid at higher temperatures such as 158°F (i.e., the boiling point of AeroZINE 50 and the temperature to which the subscale chambers were to be cycled in Phase IV).

It can be concluded that a barrier-type liner, such as a metallic foil, is required for long-term reliability of filament-wound tankage. The proven corrosion resistance of stainless steel to all common liquid propellants is a further argument in favor of a metallic liner.

3. Metallic-Type Liner Materials

Metallic liner materials, particularly the aluminum and stainless steel alloys, are known to be resistant to the corrosive effects of N_2O_4, AeroZINE 50, pentaborane, and ClF_3 (References 4, 6, and 11). In a recent report, Al 1100 was shown to have good corrosion resistance to ClF_3 (Reference 15). A liner fabricated from Al 1100 could probably be used for the containment of ClF_3 as well as AeroZINE 50 and N_2O_4. However, discussions with

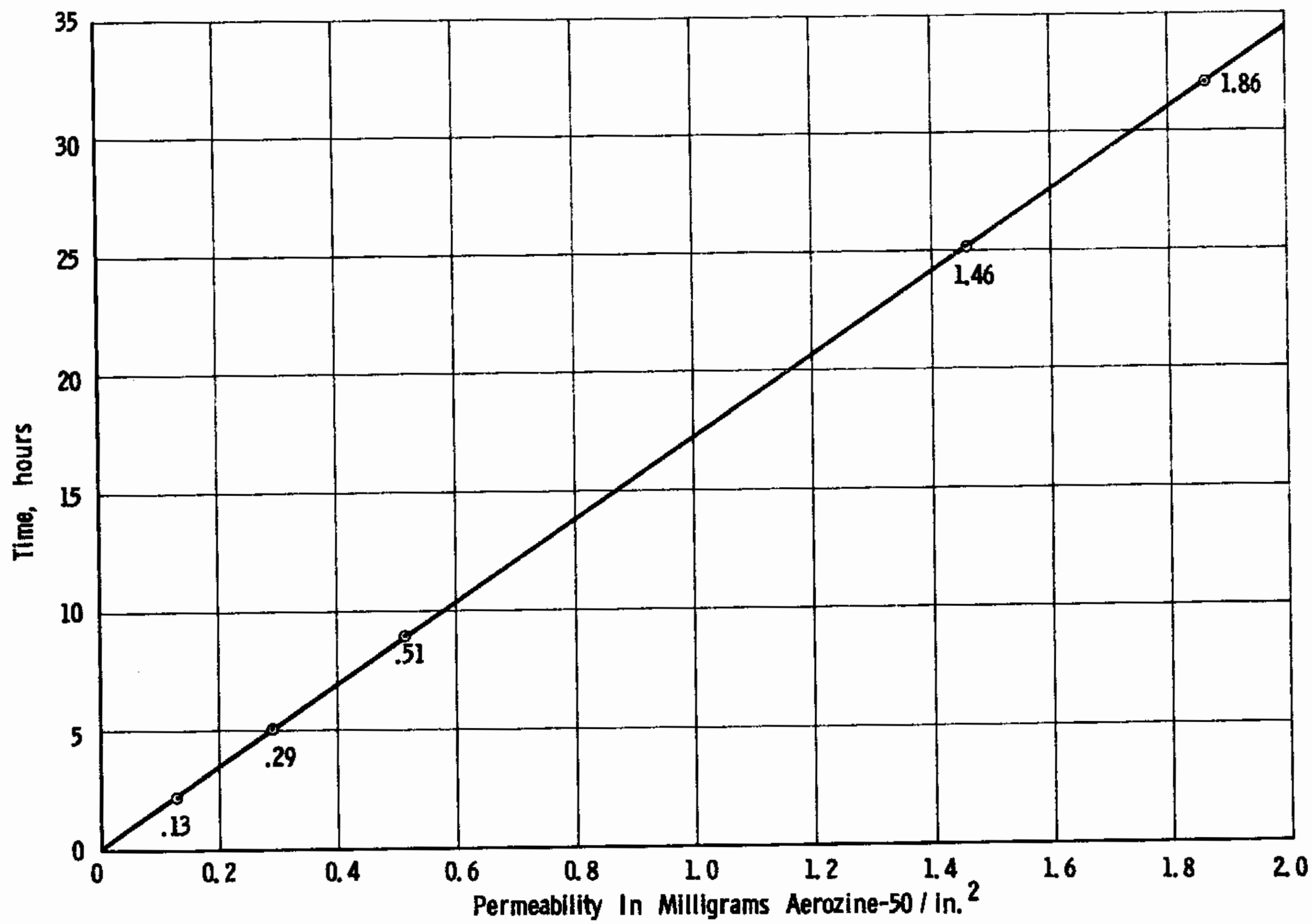


Figure 12. Permeability of 0.010-in. Teflon Film to AeroZINE 50 at 150 psig

welding specialists indicate that weldments of the softer grades of aluminum are difficult to fabricate and are also subject to porosity.

The choice of a metallic liner for filament-wound tankage requires consideration of corrosion resistance, physical properties, and fabrication feasibility. Tensile tests were made on candidate metal foils to determine their mechanical-strength properties. The results are shown in Table 9. The yield strength and ultimate tensile strength show very little variation with foil thickness. The elongations increase with thickness. The more promising metal foils appear to be Al 1100-0, 304 SS, and 347 SS.

The optimum design-strain level for a glass-reinforced filament-wound container is considered to be approximately 2.0%. To achieve this with a metal-lined tank, some type of pattern in the metal or other novel fabrication technique is required. A study of several pattern designs was conducted in a concurrent program at Aerojet's Von Karman Center, and two designs were selected as most promising in meeting the design requirements.

The patterns were cast in room-temperature-vulcanizing silicone-rubber molds, transferred to forming dies of a low-melting alloy, and impressed on the metal foils by a rubber pad in a hydraulic press. Biaxial-stretching tests were then made in a special fixture to determine their extensibility characteristics.

The two most promising designs, a crossing pattern and an "S" pattern, were impressed on 0.005-in.-thick 304 SS foil. They were then subjected to a biaxial-stretching test at strain levels of 1.0 to 3.0%. After being stretched to each level, the specimens were released and the return was measured across each axis. The following results were obtained:

	Crossing Pattern, %		S Pattern, %	
	A Axis	B Axis	A Axis	B Axis
Stretched to	1	0.75	1.0	1.5
Returned to	0	0	0	0
Stretched to	1.5	1.0	1.35	1.5
Returned to	1.0	0.5	0	1.15
Stretched to	2.0	1.5	2.0	2.65
Returned to	1.5	1.0	1.5	2.65
Stretched to	2.5	1.5	2.5	3.3
Returned to	1.5	1.0	2.0	3.0
Stretched to	3.0	2.0	-	-
Returned to	2.0	1.5	-	-

TABLE 9
TENSILE PROPERTIES OF METAL FOILS

Material	Thickness in.	Yield Strength psi	Ultimate Tensile Strength psi	Modulus x 10^6	Elongation in 2 in., %
Al 1100-0	0.0065	5,424	11,923	-	31.1
Al 1100-0	0.0083	5,310	11,332	6.2	35.9
Al 1100-0	0.0120	5,783	13,586	10.0	37.2
Al 1100-0	0.0162	5,834	13,476	10.6	36.5
302 SS	0.0010	70,400	102,043	37.8	15.3
304 SS (annealed)	0.0030	50,336	95,651	38.5	29.7
304 SS (annealed)	0.0050	41,360	89,920	39.7	46.2
347 SS (annealed)	0.0030	47,507	85,853	37.7	21.7
347 SS (annealed)	0.0062	44,607	94,006	43.1	31.9

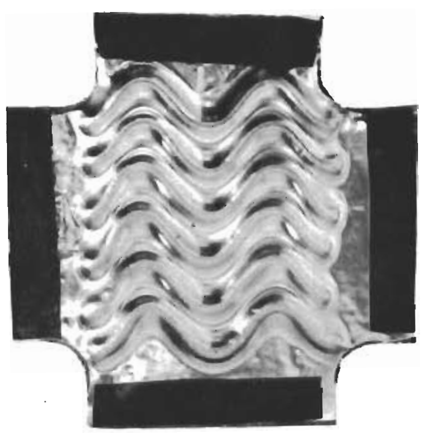
These results indicate that the crossing pattern is somewhat more satisfactory than the S pattern in recovery. Neither showed buckling or cracking in the pattern area. Figure 13 shows these patterns before and after stretching. Small cracks developed in the unpatterned area of each panel at the cutout point as a result of the stretching test. This study indicates that a metallic liner in which these patterns are incorporated would tolerate the strain level required in the optimum design of filament-wound tankage. Difficulty in obtaining wrinkle-free patterns encountered in this study may indicate a potential problem area in liner fabrication, because the detection of minor flaws in patterns would be very difficult. The tooling required to produce these patterns would also be complicated and costly.

C. CONCLUSIONS

Polymeric liner materials are considered too permeable to the propellants of interest to permit their use in filament-wound tankage for long-term storage of rocket oxidizers and fuels. Metals, particularly aluminum alloys and stainless steels, have satisfactory corrosion resistance for this type of service.

Crossing Pattern Before Testing

Crossing Pattern After Testing



"S" Pattern Before Testing

"S" Pattern After Testing

Figure 13. Biaxial Strain Testing of Liner Patterns

V. PHASE III - EVALUATION OF COMPOSITES

This phase was undertaken to establish the composites to be used in the fabrication of subscale tankage for environmental-exposure testing. Inasmuch as the resins studied in Phase I are used in combination with glass filaments in filament-wound tankage, it was necessary to determine how composites of these materials react to propellants and to a space environment. Accordingly, the promising resin systems from the Phase I study were used in the impregnation of S-HTS glass roving and the fabrication of Naval Ordnance Laboratory (NOL) rings and filament-wound flat panels. Specimens of these composites were then exposed to the propellants of interest and to a simulated space environment of heat and vacuum.

An additional objective of Phase III was to establish the most effective method of combining a metal liner with a glass-filament-wound case to provide separation of the propellant and the resin/glass composite. Phase I and II studies of resins and polymeric liner materials had definitely established the necessity for a barrier-type liner. To allow a complete explanation of this important problem, an investigation of bonded, metal-lined, filament-wound tankage was authorized as a substitute for pentaborane-exposure studies. This investigation covered the fabrication and testing of flat bonded panels, followed by pressure-cycle testing of subscale tankage in Phase IV.

A further objective of the work in Phase III was determination of the feasibility of using chemical-adhesive bonding as an alternative to welding, which has limitations for the joining of large foil segments, in the fabrication of metal-foil-lined tankage.

A. LITERATURE SURVEY

A survey was conducted to determine the available information on the effect of a space environment on materials that may be used in filament-wound tankage for storable propellants. Considerable data exist on the outgassing of materials exposed to the hard vacuum and radiation of space. The effect of these conditions on the physical properties of materials, however, has not been extensively studied.

It was reported (Reference 16) that, after an exposure of 500 hours to ultraviolet (2 pyrons) and a vacuum of 4.4×10^{-7} torr (mm Hg), an epoxy-resin

(Shell Epon 815) glass-reinforced laminate lost 0.83 wt%; the maximum temperature reached was 319°F. Under the same conditions, a phenolic resin showed a weight loss of 1.30%; the maximum temperature reached was 342°F. A polyester resin lost 0.78 wt% and increased in temperature to 288°F under the same exposure conditions. As a result of this ultraviolet and hard-vacuum exposure, the flexural strength of the epoxy laminate dropped from 95,300 to 86,300 psi, and that of the phenolic laminate dropped from 77,040 to 70,100 psi. The flexural strength of the polyester laminate increased from 72,170 to 74,200 psi.

According to Ringwood (Reference 17), the vacuum of space has a rather pronounced effect on many plastics. In some cases it degrades the material by volatilizing components essential to the optimum performance properties. In others, it improves properties by the same volatilization mechanism wherein impurities are outgassed. The author also states that, at 10^{-6} torr, epoxies, polyesters, and phenolics require temperatures between 100 and 200°C (212 and 392°F), depending on the formulation, to lose 10 wt% per year. Some modified epoxy systems, however, have a similar loss at temperatures as low as 50°C (122°F).

It has also been reported that thermoplastic materials have the least weight loss in a vacuum. The rate of loss for epoxy resins is about an order of magnitude greater, and that for reinforced phenolics is about 2 orders of magnitude greater (Reference 18).

According to Brownlee and McNally, the combined effects of high vacuum, ultraviolet radiation, and elevated temperatures are not significant for most structural-plastic materials. In tests they reported, epoxy and phenolic laminates showed no significant degradation after 500 hours of exposure to a radiant energy flux in the range from 2500 to 5800 angstroms at a temperature of 300°F (Reference 19).

The Battelle Memorial Institute, however, states in a recent report (Reference 20): "... more experimental effort is still needed in studying combined effects of radiation and other environments. Although much is known concerning the loss of material due to evaporation and sublimation, very little is known concerning the effects of these material losses on the physical strength and electrical characteristics."

B. FABRICATION FOR EXPOSURE TESTING

The following resin systems were selected for evaluation as resin/glass composites for exposure to the propellants and to a vacuum environment:

RS-10 - U.S. Polymeric No. 46 (phenol formaldehyde)

RS-11 - DEN 438/BF_3-400 (novolac epoxy)

RS-15 - Kopox 170/BF_3-400 (polyepoxide)

Inasmuch as these systems were too high in viscosity to permit in-process impregnation, they were preimpregnated on S-HTS glass roving by an outside vendor. They performed satisfactorily in this method of processing.

The prepreg rovings were then used to fabricate NOL rings and flat panels. The NOL-ring specimens of each system were prepared under the following conditions: winding assembly temperature 125°F, spool tension 4 lb, winding tension 15 lb, 110 turns, and a cure of 2 hours at 200°F, 2 hours at 250°F, and 12 hours at 300°F. The resin contents were as follows: 20.5 wt% for RS-10, 19.8% for RS-11, and 18.8% for RS-15. The flat panels were all prepared with a winding tension of 5 lb, 12 turns/in., 15 to 16 layers 0.125 in. thick, and a vacuum-bag cure of 2 hours at 200°F, 2 hours at 250°F, and 12 hours at 300°F.

Specimens were cut from these composites for evaluation before and after exposure to AeroZINE 50 and N_2O_4 at two temperatures and to hard vacuum at two temperatures. Satisfactory samples were obtained from the RS-11 and -15 systems. The filament-wound flat panels produced from the S-HTS glass roving preimpregnated with RS-10 delaminated during the cure and were not acceptable for testing. The NOL-ring samples made from the RS-10 prepreg were satisfactory. Specimens were cut from these composites for the following tests:

	Specimen	Test Method
Horizontal shear	1-in. section of NOL ring	WS 1028-A (U.S. Navy)
Short-span flexural strength	3-in. by 1-in. panel	ASTM D790-59T
Long-span flexural strength	4-in. by 1-in. panel	ASTM D790-59T
Hardness (Barcol)	All	Manufacturer's instructions
Weight change	All	ASTM D-792-50

C. PROPELLANT EXPOSURE

The filament-wound composites of the three resin systems were exposed to AeroZINE 50 and N_2O_4 as follows:

Resin System	Propellant	Temp, °F	Exposure Periods
RS-10	AeroZINE 50	60	1 week
RS-11	AeroZINE 50	60	1 week, 1 month, 3 months
RS-15	AeroZINE 50	60	1 week, 1 month, 3 months
RS-10	AeroZINE 50	100	1 week
RS-11	AeroZINE 50	100	1 week, 1 month, 3 months
RS-15	AeroZINE 50	100	1 week, 1 month, 3 months
RS-10	N_2O_4	60	1 week, 1 month, 3 months
RS-11	N_2O_4	60	1 week
RS-15	N_2O_4	60	1 week, 1 month, 3 months
RS-10	N_2O_4	100	1 week, 1 month, 3 months
RS-11	N_2O_4	100	1 week
RS-15	N_2O_4	100	1 week, 1 month, 3 months

The results of the propellant-exposure tests of the composites are summarized in Table 10. The composites were degraded much more than are the resins alone. In fact, on exposure to N_2O_4 for 7 days at 100°F, 30 days at 60°F, and 30 days at 100°F, all specimens delaminated so much that tests could not be performed. This severe degradation is believed to have resulted from the combined effect of the lower density of composites and the breakdown of the coating on the glass filaments.

Comments on the appearance of resin/glass-filament composites are given in Table 11. Comparisons of the data presented in Tables 10 and 11 show that composites of RS-11 (the novolac epoxy) and RS-15 (the polyepoxy type) are superior to those of RS-10 (a phenolic system) in resistance to both AeroZINE 50 and N_2O_4. The RS-10 system was softened considerably and showed a substantial weight gain on exposure to AeroZINE 50 for short periods.

Exposure produced a substantial loss in specimen hardness. This was particularly evident when the exposure temperature was 100°F.

In retention of shear strength after AeroZINE 50 exposure the RS-11 and -15 systems were approximately equivalent, although RS-11 showed somewhat higher values after the exposure.

TABLE 10
PROPERTIES OF RESIN/GLASS-FILAMENT COMPOSITES AFTER EXPOSURE TO PROPELLANTS(1)

Resin System	Exposure: Time days	Exposure: Temp °F	Exposure: Propellant	Horizontal Shear: Weight Change %	Horizontal Shear: Barcol Change	Horizontal Shear: Shear Strength psi	Horizontal Shear: Shear Strength % Ret	Long-Span Flexure: Weight Change %	Long-Span Flexure: Barcol Change	Long-Span Flexure: Flexural Strength psi	Long-Span Flexure: Flexural Strength % Ret	Short-Span Flexure: Weight Change %	Short-Span Flexure: Barcol Change	Short-Span Flexure: Flexural Strength psi	Short-Span Flexure: Flexural Strength % Ret
RS-10	[U.S. Poly 46 (Phenol Formaldehyde)/S-HTS Glass]														
	-	-	-	-	68 (C)	3,795 (C)	-	-	-	-	-	-	-	-	-
	7	60	AeroZINE 50	+2.82	-29	3,514	92.3	NT(DS)	-	-	-	-	-	-	-
	7	100	AeroZINE 50	+6.80	-13	3,460	91.5	NT(DS)	-	-	-	-	-	-	-
	7	60	N_2O_4	NT(SD)	-	-	-	NT(DS)	-	-	-	-	-	-	-
	7	100	N_2O_4	NT(SD)	-	-	-	NT(DS)	-	-	-	-	-	-	-
	30	60	N_2O_4	NT(SD)	-	-	-	NT(DS)	-	-	-	-	-	-	-
	30	100	N_2O_4	NT(SD)	-	-	-	NT(DS)	-	-	-	-	-	-	-
RS-11	(DEN 438/BF_3-400/S-HTS Glass)														
	-	-	-	-	71 (C)	8,797 (C)	-	-	73 (C)	167,786 (C)	-	-	73 (C)	5,933 (C)	-
	7	60	AeroZINE 50	+0.63	-4	6,081	69.2	+0.34	+3	148,748	88.5	+0.40	-2	5,977	101.0
	7	100	AeroZINE 50	+0.87	-39	5,102	58.2	+3.11	-27	54,686	32.5	+3.22	-31	2,444	41.3
	30	60	AeroZINE 50	+0.97	-8	4,018	45.7	+0.80	-4	145,845	86.6	+1.05	-7	5,414	91.4
	30	100	AeroZINE 50	+1.09	-20	2,786	31.7	+1.91	-25	73,475	43.8	+1.49	-21	3,467	58.0
	90	60	AeroZINE 50	+1.93	-16	3,040	34.6	+1.90	-19	82,302	49.0	+1.76	-17	3,317	53.7
	90	100	AeroZINE 50	+0.99	-38	2,765	31.5	+1.39	-27	60,862	36.2	+1.20	-29	3,045	50.3
	7	60	N_2O_4	-0.37	-2	5,706	65.0	+2.73	-8	27,947	16.6	+2.10	-1	3,411	57.7
	7	100	N_2O_4	NT(SD)	-	-	-	NT(SD)	-	-	-	NT(SD)	-	-	-
RS-15	(Kopox 170/BF_3-400/S-HTS Glass)														
	-	-	-	-	70 (C)	7,135 (C)	-	-	65 (C)	118,066 (C)	-	-	64 (C)	4,399 (C)	-
	7	60	AeroZINE 50	+1.27	-15	4,450	62.4	+1.66	-13	86,879	73.7	+1.65	-12	3,684	84.0
	7	100	AeroZINE 50	+1.98	-41	2,826	39.7	+2.27	-23	50,121	42.5	+2.25	-21	2,559	58.2
	30	60	AeroZINE 50	+1.45	-17	3,109	43.7	+1.56	-9	82,326	69.6	+1.33	-7	3,168	72.2
	30	100	AeroZINE 50	+1.06	-18	2,408	33.7	+1.58	-22	66,635	56.3	+0.85	-19	3,221	73.2
	90	60	AeroZINE 50	+0.97	-4	3,337	46.8	+1.11	-14	70,350	59.5	+1.09	-10	2,908	66.1
	90	100	AeroZINE 50	+0.62	-30	2,016	28.2	+0.45	-19	65,078	55.0	+0.40	-20	2,766	62.8
	7	60	N_2O_4	+3.25	Soft	1,588	22.2	+4.44	-6	30,525	25.9	+1.84	+5	3,435	78.2
	7	100	N_2O_4	NT(SD)	-	-	-	NT(SD)	-	-	-	NT(SD)	-	-	-
	30	60	N_2O_4	NT(SD)	-	-	-	NT(SD)	-	-	-	NT(SD)	-	-	-
	30	100	N_2O_4	NT(SD)	-	-	-	NT(SD)	-	-	-	NT(SD)	-	-	-
	90	60	N_2O_4	NT(SD)	-	-	-	NT(SD)	-	-	-	NT(SD)	-	-	-
	90	100	N_2O_4	NT(SD)	-	-	-	NT(SD)	-	-	-	NT(SD)	-	-	-

(1) Values reported on controls are averages of ten tests; values reported on exposures are averages of five tests. (C) = control. % Ret = Retention, %. NT = no test. (SD) = specimens delaminated. (DS) = defective specimens.

TABLE 11
APPEARANCE OF RESIN/GLASS COMPOSITES AFTER EXPOSURE TO PROPELLANTS

Resin System	Type of Specimen(1)	Propellant	Temp °F	Appearance: 30-Day Exposure	Appearance: 90-Day Exposure
RS-11	HS	AeroZINE 50	60	Fair, slight swelling	Fair, slight swelling
RS-15	HS		60	Same	Same
RS-11	LSF		60	Same	Same
RS-15	LSF		60	Fair, slight delamination	Fair, slight delamination
RS-11	SSF		60	Fair, slight swelling	Fair, slight swelling
RS-15	SSF		60	Same	Fair, slight delamination
RS-11	HS		100	Fair, slight delamination	Good, no delamination
RS-15	HS		100	Good, no delamination	Fair, slight delamination
RS-11	LSF		100	Fair, slight delamination	
RS-15	LSF		100	Fair, serious delamination	
RS-11	SSF		100	Fair, slight delamination	
RS-15	SSF	AeroZINE 50	100	Same	Fair, slight delamination
RS-10	HS	N_2O_4	60	Poor, complete delamination	Poor, complete delamination
RS-15	HS		60	Poor, considerable delamination	Same as above
RS-10	LSF		60	No test, defective specimen	No test, defective specimen
RS-15	LSF		60	Poor, complete delamination	Poor, complete delamination
RS-10	SSF		60	No test, defective specimen	No test, defective specimen
RS-15	SSF		60	Poor, complete delamination	Poor, complete delamination
RS-10	HS		100	All specimens completely delaminated	
RS-15	HS		100		
RS-10	LSF		100		
RS-15	LSF		100		
RS-10	SSF	N_2O_4	100	All specimens completely delaminated	

(1) HS = horizontal shear, LSF = long-span flexural, SSF = short-span flexural.

The flexural-strength retention of RS-11 was somewhat superior to that of RS-15 on short-term exposures to AeroZINE 50. However, in the 90-day exposure, and particularly at the higher temperature, the RS-15 system demonstrated greater resistance. The flexural strengths after exposure were in most cases higher for RS-11 than for RS-15.

The percentage weight changes after the immersion tests were relatively low and rather inconsistent. These values cannot be considered as accurate indices of degradation, because the decontaminated specimens were washed in water and were then dried to permit physical testing. This treatment could not be controlled accurately enough to produce meaningful weight readings.

D. VACUUM EXPOSURE

The filament-wound composites of the three resin systems were exposed to a vacuum of 10^{-5} to 10^{-7} torr for 7, 30, and 90 days at each of the following temperatures: 75 and 200°F. Table 12 presents the test results. The exposure for 1 week at 75°F produced very little effect. A very slight decrease in weight was shown by horizontal-shear specimens of RS-10. This type of resin (phenol formaldehyde) is known to give off small amounts of water vapor during vacuum exposure. A slight drop in the horizontal shear strength of the novolac resin system was the only change in strength properties.

The change in physical properties was also very minor in a 200°F exposure for 7 days. The horizontal-shear strength of the RS-10 and -11 composites dropped slightly. The long-span and short-span flexural strengths of the RS-11 and -15 composites increased slightly, as did their hardness; this was no doubt due to an increase in their state of cure. The 0.85% weight loss shown by the horizontal-shear specimens of RS-10, although rather low, would be approximately 4.5% based on the resin content of the composite. A comparable value for the RS-11 and -15 composites would be approximately 0.51 and 0.6%, respectively, based on the resin content.

Thirty-day exposures did not produce a major change in strengths. At 75°F the shear strength of all systems showed a slight drop, although the flexural strength remained high. At 200°F, however, the shear-strength retention of all resin systems was higher than at 75°F. The weight losses for the resins after the longer exposure were no higher than for the shorter period, indicating that the volatile constituents had disappeared in the early part of the exposure.

TABLE 12
PROPERTIES OF RESIN/GLASS-FILAMENT COMPOSITES AFTER EXPOSURE TO VACUUM(1)

Resin System	Exposure Time days	Exposure Temp °F	Horizontal Shear: Weight Change %	Horizontal Shear: Barcol Change	Horizontal Shear: Shear Strength psi	Horizontal Shear: Shear Strength % Ret	Long-Span Flexure: Weight Change %	Long-Span Flexure: Barcol Change	Long-Span Flexure: Flexural Strength psi	Long-Span Flexure: Flexural Strength % Ret	Short-Span Flexure: Weight Change %	Short-Span Flexure: Barcol Change	Short-Span Flexure: Flexural Strength psi	Short-Span Flexure: Flexural Strength % Ret
RS-10	[U.S. Poly 46 (Phenol Formaldehyde)/S-HTS Glass]													
	-	-	-	68 (C)	3,795 (C)	-	-	-	-	-	-	-	-	-
	7	75	-0.15	+2	4,408	116.0	NT(DS)	-	-	-	-	-	-	-
	30	75	-0.15	+8	2,770	73.0	NT(DS)	-	-	-	-	-	-	-
	7	200	-0.85	0	3,169	83.7	NT(DS)	-	-	-	-	-	-	-
	30	200	-0.83	+1	3,948	104.0	NT(DS)	-	-	-	-	-	-	-
	90	75	-0.29	+4	3,434	90.5	NT(DS)	-	-	-	-	-	-	-
	90	200	-1.03	-2	3,821	101.0	NT(DS)	-	-	-	-	-	-	-
RS-11	(DEN 438/BF_3-400/S-HTS Glass)													
	-	-	-	71 (C)	8,797 (C)	-	-	73 (C)	167,786 (C)	-	-	73 (C)	5,933 (C)	-
	7	75	0	-2	7,692	87.5	-0.04	+4	171,604	102.0	-0.05	+4	5,836	98.4
	30	75	0	0	7,059	80.5	-0.05	+1	165,184	98.5	-0.05	-1	6,995	118.0
	7	200	-0.10	-5	7,823	89.2	-0.07	+1	189,363	107.0	-0.07	0	6,428	108.0
	30	200	-0.05	-3	7,712	87.8	-0.06	0	161,700	96.3	-0.08	0	6,430	108.5
	90	75	0	-3	8,261	94.0	-0.04	+2	178,739	106.4	-0.04	+2	6,553	106.4
	90	200	-0.06	0	7,583	86.4	-0.06	0	188,826	112.7	-0.07	+2	6,580	111.0
RS-15	(Kopox 170/BF_3-400/S-HTS Glass)													
	-	-	-	70 (C)	7,135 (C)	-	-	65 (C)	118,066 (C)	-	-	64 (C)	4,399 (C)	-
	7	75	0	0	7,291	102.0	-0.06	+5	126,917	107.0	-0.06	+2	4,699	107.0
	30	75	0	+4	5,735	80.5	-0.07	+2	118,975	101.0	-0.07	-2	4,713	107.6
	7	200	-0.15	0	7,594	106.0	-0.20	+6	129,517	110.0	-0.20	+4	5,044	114.0
	30	200	-0.20	+1	7,527	105.6	-0.15	+6	124,160	105.0	-0.17	+3	5,040	114.0
	90	75	0	0	6,771	95.0	-0.07	+3	130,450	110.0	-0.07	+2	4,871	110.8
	90	200	-0.11	+4	7,476	105.0	-0.12	+7	132,235	112.5	-0.13	+5	4,964	113.0

(1) Vacuum held to 10^{-6} to 10^{-7} torr on 75°F exposures. Vacuum held to 10^{-5} to 10^{-7} torr on 200°F exposures. Values reported for controls are averages of ten tests and for exposures are averages of five tests. % Ret = Retention, %. (C) = control. NT(DS) = no test (defective specimens).

Test methods: Weight, ASTM D-792-50; hardness (Barcol), manufacturer's instructions; horizontal shear, Weapon Specification 1028-A U.S. Navy; flexural strength, ASTM D790 - 59T.

The results of 90-day exposures show that, even at 200°F, a vacuum produces very little degradation of phenolic and epoxy resin/glass-filament composites. The only significant change was a weight loss by RS-10; this was to be expected on the basis of the known loss of moisture from phenol formaldehyde resins after heating. The 1.03% weight loss for RS-10 after 90 days at 200°F was 5.0 wt% based on the resin content of the composite. The weight loss of the RS-11 horizontal-shear specimens after this exposure period was 0.30% on the same basis; for RS-15 the value was 0.58%. The weight losses of these specimens increased very little after the first 7 days of exposure, indicating that the volatiles disappeared during the early part of the heating cycle and that very little resin breakdown occurs.

The flexural strengths of the RS-11 and -15 systems increased measurably during the 90-day exposure. This may be interpreted to mean that the volatile constituents are actually minor contaminants and that, after their removal, the resin continues cross-linking that produces higher strengths.

These exposure tests indicate that the effects of vacuum in a space environment on resin/glass composites are minimal. It can therefore be assumed that vacuum effects can be ignored in the design of filament-wound space structures.

Comparison of the candidate resin systems, in the form of composites, for compatibility with rocket propellants and resistance to the heat and vacuum of space indicated no great margin of difference between the novolac epoxy and the polyepoxide (RS-11 and -15, respectively). The RS-15 system was superior in high-temperature resistance. The flexural strengths of RS-11 were higher even after some of the propellant-exposure tests, however, indicating that RS-11 produces a tougher composite that should have less tendency to craze in filament-wound constructions than RS-15. In view of the decision to use an impermeable type of liner, the structural integrity of the tank structure assumes greater importance. After consideration of these factors and consultation with the Research and Technology Division (RTD) Project Engineer, it was decided to use RS-11 in the fabrication of subscale chambers for Phase IV.

E. STRUCTURAL COMPATIBILITY OF METAL-FOIL/GLASS COMPOSITES

It has been reported that a metallic liner did not perform satisfactorily in a filament-wound tank because of creasing and cracking during pressure cycling (Reference 9). A recent discussion, however, with J. Barber of the Lewis Research Center, National Aeronautics and Space Administration, indicated that this would not occur if the metallic liner were adhesively bonded to the case. If substantiated, this approach could be of significant value in the fabrication of metal-lined filament-wound tankage.

The use of a metal liner creates a compatibility problem under pressure-cycling conditions because of the difference in properties between the filament-wound epoxy/glass composite and the liner. Specifically, the composite has a modulus approximately one-fourth that of stainless steel. An efficient composite must operate at a biaxial-strain level of from 1-1/2 to 3%. The metal liner (e.g., stainless steel), however, is capable of reaching only 0.3 to 0.5% strain before plastically yielding. This normally results in buckling during pressure cycling. The problem should be controllable if the adhesive-bonding concept described above is valid. The bonding of the liner to the overwrap should prevent the liner from assuming buckling-mode shapes.

A study of the structural compatibility of metal-foil/glass-filament composites was conducted to determine the validity of the bonding concept. Aluminum and stainless steel foils were bonded to epoxy-coated glass and Dacron fabrics, and 1-in.-wide strips of the panels were subjected to uniaxial cyclic strain tests at various levels of strain. The specimens were then examined for evidence of buckling, and the percentage set in the measured section was determined.

The compositions of the laminates and the results obtained are shown in Table 13. A 2-1/2% strain level was reached without buckling when the metal was bonded directly to the glass fabric. However, when a layer of rubber was included as a sandwich between the metal and glass fabric, such as in Specimens Al-7 and Al-8, buckling and even cracking occurred. In some cases the stretching exceeded the elongation of the glass fabric, and failure of the glass occurred. When epoxy-coated 181 glass fabric was used, however, the elongation could be extended to 2-1/2%.

TABLE 13

RESULTS OF CYCLE TESTS, UNIAXIAL STRETCHING OF METAL-FOIL/EPOXY/GLASS LAMINATES[1]

Spec. No.	Foil	Glass Fabric	Adhesive	Sandwich	Spec Width in.	Cycling Condition: Elongation, %	Cycling Condition: Cycles	Test Results: % Set	Test Results: Appearance
Al-1	Al 1100	143	None	None	0.96	2	27	0	Good, no failure
Al-2	↓	143	None	↓	0.96	2-1/4		0	Glass failure
Al-3	↓	143	None	↓	0.97	2	27	0	Good, no failure
Al-4	↓	143	Y-9141	↓	0.95	2	36	0	Good, no failure
Al-5	↓	181	Y-9141	↓	1.00	2-1/2	25	0	Good, no failure
Al-6	↓	143	Urethane	None	0.99	2-1/4	25	0	Glass failure
Al-7	↓	181	Y-9141	Buna N (0.030 in. thick)	1.02	2	21	0	Foil buckled and cracked
Al-8	Al 1100	181	Y-9141	Buna N (0.090 in. thick)	1.00	2	25	0	Foil buckled, no cracking
SS-1	347 SS	143	Y-9141	None	0.95	2	25	0	Good, no failure
SS-2	↓	143	None	↓	1.00	2	27	0	Good, no failure
SS-3	↓	181	↓	↓	1.00	2	25	0	Good, no failure
SS-4	↓	181	↓	↓	1.00	2-1/2	25	1	Good, no failure
SS-5	↓	181	↓	↓	1.00	3	1	-	Glass failure
SS-6	↓	181	None	↓	1.00	3-3/4	1	-	Glass failure
SS-7	347 SS[2]	181	Y-9141	↓	1.00	3-3/4	1	-	Glass failure
SS-8	347 SS[2]	143	↓	↓	0.96	2	27	1	Good, no failure
SS-9	347 SS[2]	143	↓	↓	0.96	2-1/4	27	1	Good, no failure
SS-10	347 SS	143	Y-9141	None	0.98	2-1/2	1	-	Glass failure

(1) Foil thickness, 0.006 in. throughout. Glass fabric, two plies throughout.
(2) Roll-seam weld in center of specimen.

The effect of a roll-seam weld on the structural compatibility of the laminate was evaluated with Specimens SS-8, SS-9, and SS-10. The laminate was successfully stretched to 2-1/4%, but the glass failed at 2-1/2%. Obviously, the slight offset produced by the weld did not cause separation of the metal foil from the epoxy-coated glass fabric. The use of 181 glass fabric in this test would have permitted elongation to 2-1/2%, as for the unwelded metal specimens.

These results show that the metal actually undergoes compression when bonded to a rigid supporting member. This compression prevents buckling and separation from the glass-fabric substrate, even at a strain level of 2-1/2%. In practice, this effect should preserve the integrity of a metallic liner for filament-wound tankage.

In order to determine the ultimate elongation that can be tolerated by metal foils when bonded to a semirigid substrate, composites were made with resin-coated Dacron 2353 fabric (three plies) as the backup material, using Y-9141 adhesive (Minnesota Mining and Manufacturing (3M) Company). One-inch strips of these laminates were then subjected to a uniaxial-stretching test to 2-1/2 and 3% strain. The results were as follows:

Spec. No.	Foil: Type	Foil: Thickness in.	Cycling Condition: Elongation, %	Cycling Condition: Cycles	Test Results: % Set	Test Results: Appearance
SS-11	347 SS	0.006	2-1/2	27	2-1/2	No failure, bowed specimen
SS-12	347 SS	0.006	3	27	3	No failure, bowed specimen
Al-9	Al 1100	0.006	3	27	3	No failure, very slight buckle

As noted above, the 347 SS foil withstood 3% stretching without separation from the substrate. The foil did take a set and was bowed on removal from the clamping jaws. The aluminum-foil laminate also withstood 3% stretching, although a slight buckle appeared at the edge of the clamping area that may have been due to the clamp pressure at this point. The aluminum-foil-laminate specimen was slightly bowed after removal from the clamps, although not to the same degree as the stainless steel laminate.

A panel was fabricated from 0.006-in.-thick Al 1100 bonded to an epoxy-resin-coated Dacron fabric. It was subjected to stretching in the biaxial test fixture to a 2% strain level. Although the laminate withstood this treatment satisfactorily, the clamps pulled out of the tabs after one cycle and it was necessary to discontinue the test. Laminates of aluminum, stainless steel, and Teflon-aluminum were fabricated with reinforced tabs. Biaxial cycling tests were attempted on these laminates but without success because of breaking of the welded ends on the cables of the test fixture. These tests were discontinued in favor of the pressure-cycling tests on subscale tanks.

F. ADHESIVE BONDING OF METALLIC LINER SEGMENTS FOR FABRICATION OF FILAMENT-WOUND TANKAGE

1. General Considerations

Work was done in accordance with a contract provision that particular consideration be given to "the creation of new or novel approaches to the liner, fabrication techniques for the liner, and techniques for combining the liner and the glass reinforced tank so that strain compatibility is achieved, both during internal loading and thermal cycling." Fabrication techniques for metallic liners were developed that appear to ensure the necessary compatibility with the filament-wound supporting structure of the tankage. Resistance-roll-seam welding of the metallic-foil liner segments and bonding of the liner to the case produced a satsifactory tank for the containment of rocket oxidizers and fuels.

The fabrication of full-scale tankage for storable propellants will require some modification of the procedures that have been developed for the subscale tanks. It will of course be desirable to maintain the thin lining for large tanks in order to provide maximum weight economy, but, the handling of metallic foils in thicknesses of 0.0062 in. and in diameters of 56 to 64 in. will require special techniques. Pressure forming of hemispherical sections of this size may not be within the scope of available equipment. Other methods of fabricating the metallic liner, such as spinning and welding of gore segments, may be considered, but each has definite limitations and undesirable features.

A fabrication method for the liner that would involve laying segments over a removable mandrel would permit the handling of thin foils. Although techniques are available for welding these segments together, the reliability of some types of weldments is questionable. However, if the segments could be bonded together and to the metallic bosses of the tank by adhesive bonding, the fabrication techniques would be simplified.

Although most common adhesives are not resistant to corrosive propellants, some were found in these studies that have moderately good resistance. In addition, the area of the adhesive exposed to the propellant on bonded joints is very low. By the use of wide lap joints, the leak path could be made as long as required for any given service condition.

2. Experimental Program

A preliminary study was conducted to determine if the concept of adhesive bonding had merit. Several adhesive formulations were prepared with Kopox 170 as the base resin. The Kopox 170 was compounded with aluminum powder and a high-silica filler to produce optimum flow properties and maximum shear strength. The experimental adhesives were brushed on cleaned 2024-T3 aluminum panels on a 1/2-in. overlap and were cured at a pressure of 25 psi. The following formulations were tested for lap-shear strength before and after exposure to AeroZINE 50 and N_2O_4:

	Base Resin		Curing Agent		Filler		Thickener	
Adhesive	Type	Parts by Weight	Type	Parts by Weight	Type	Parts by Weight	Type	Parts by Weight
KC-1	Kopox 170	100.0	BF_3-400	3.0	Alcoa 101	100.0	None	-
KC-2	Kopox 170	100.0	BF_3-400	3.0	None	-	None	-
KC-3	Kopox 170	100.0	BF_3-400	3.0	Alcoa 101	100.0	Cab-O-Sil	4.0
KC-4	Kopox 170	100.0	BF_3-400	3.0	Alcoa 101	85.0	Cab-O-Sil	7.0

The results obtained with these adhesive formulations are shown in Table 14. The adhesives do not produce as high shear strengths as some of the commercial metal adhesives. The retention of shear strength after immersion in propellants was creditable, however, and indicates that this concept has definite merit.

TABLE 14
PROPERTIES OF ADHESIVE-BONDED METAL PANELS AFTER
EXPOSURE TO PROPELLANTS(1)

Adhesive	Exposure Conditions	Lap-Shear Strength, psi	Comments
KC-1	Controls	1429 1256 1567 Av 1417	Poor wetting, aluminum powder settled out
KC-1	3 days in N_2O_4 at 75°F	1582 1128 1329 Av 1346	Dark flash on bonded area
KC-2	Controls	1476 1077 1210 Av 1254	Too fluid, more body required
KC-2	3 days in N_2O_4 at 75°F	1333 1345 667 Av 1115	Dark flash on bonded area
KC-3	Controls	1555 1760 1580 Av 1632	Slightly low in flow properties
KC-4	Controls	1591 1500 Av 1546	Fair on flow, reduction in Cab-O-Sil indicated
KC-4	3 days in N_2O_4 at 75°F	1658 1242 Av 1450	Dark flash on bonded area
KC-4	3 days in AeroZINE 50 at 75°F	1381 1268 Av 1299	No change in color of flash on bonded area

(1) Cure cycle for Adhesives KC-1 and KC-2 = 2 hours at 200°F, plus 4 hours at 250°F, plus 12 hours at 350°F. Cure cycle for KC-3 and KC-4 = 1 hour at 250°F plus 16 hours at 350°F.

A second group of adhesive compositions was formulated and evaluated for the bonding of metal-liner segments. Aluminum 2024-T3 was also used as the adherend in this study of lap-shear strength. Aluminum 1100-0 has been reported as having good resistance to AeroZINE 50, N_2O_4, and ClF_3 (Reference 13). It cannot be satisfactorily welded by conventional techniques in thin foils, however, and thus was not used in the fabrication of subscale tankage. Al 1100-0 is therefore a good candidate to consider for use in adhesive bonding. For this study, Al 2024-T3 was used because of its availability in 16-gage panels.

Lap-shear specimens using the adhesive formulations shown in Table 15 were fabricated and tested before and after the 7 days of exposures to AeroZINE 50 or N_2O_4 at 100°F.

The shear-test results are given in Table 16. Kopox 171, a lower-viscosity polyepoxy resin of the same composition as Kopox 170, was used in this series because of its greater adaptability to adhesive work. Although some of the shear-strength values for the Kopox 171 adhesives are not as high as those obtained with Kopox 170, adhesive KC-7 was outstanding in original values and after exposure to AeroZINE 50. Because its resistance to N_2O_4 was low, however, KC-7 could not be considered a satisfactory candidate for service in both propellants. KC-5 was low in original shear strength, but was fair in resistance to N_2O_4. KC-6 and KC-8 were low in both original shear strength and strength after exposure to propellants.

A third group of adhesives, shown in Table 17, was evaluated for shear strength before and after the following exposures: in AeroZINE 50 - 7 days at 100°F, 30 days at 100°F; in N_2O_4 - 7 days at 100°F, 30 days at 60°F. The results are presented in Table 18. KC-9, an unpigmented Kopox 171 cured with Z Catalyst, demonstrated excellent resistance to both AeroZINE 50 and N_2O_4 in short and long exposures. The use of Z Catalyst, a blend of MDA (methylene dianiline) and MPDA (meta phenylenediamine) appears to enhance the chemical resistance of this resin appreciably. The addition of filler, as in KC-10, obviously caused a deterioration in this adhesive. KC-12, the adhesive based on Kel-F-KX633 dispersion appeared very promising in original shear-strength values. After 7 days of exposure to N_2O_4 at 100°F, the shear strength of KC-12 was substantially lower than that of KC-9, but could be rated fair in comparison with other adhesives in this series.

TABLE 15
COMPOSITION AND CURE OF ADHESIVE CANDIDATES (KC-5 THROUGH KC-8)

Adhesive	Composition: Material	Composition: Parts by Weight	Cure
KC-5	Kopox 171[1]	100.00	2 hours at 300°F plus 18 hours at 350°F
	Alcoa 101[2]	75.00	
	Cab-O-Sil[3]	5.00	
	BF_3-400	3.00	
		183.00	
KC-6	Kopox 171	100.00	Same
	Antimony trioxide	20.00	
	BF_3-400	3.00	
		123.00	
KC-7	Zytel 61[4]	100.00	30 min at 350°F
	Methyl alcohol	170.00	
	Chloroform	40.00	
		240.00	
	To this is added		
	Kopox 171	30.00	
	Antimony trioxide	5.00	
	Dicyandianiline	1.50	
		36.50	
KC-8	DEN 438	100.00	2 hours at 300°F plus 18 hours at 350°F
	Antimony trioxide	20.00	
	BF_3-400	3.00	
		123.00	

(1) Koppers Company polyepoxy resin.
(2) Aluminum Company of America aluminum powder.
(3) Cabot Company silica filler.
(4) E.I. du Pont de Nemours & Company nylon powder.

TABLE 16

RESULTS OF ADHESIVE-BONDING STUDY (KC-5 THROUGH KC-8)

Original Properties		After 7 Days of Exposure at 100°F: In AeroZINE 50			In N_2O_4		
Specimen No.	Shear Strength, psi	Specimen No.	Shear Strength, psi	Condition	Specimen No.	Shear Strength, psi	Condition
Specimen KC-5 (Kopox 171/Alcoa 101/Cab-O-Sil/BF_3-400)							
KC-5-1	909	KC-5-IA-1	624	Poor flaked adhesive film	KC-5-IN-1	596	Fair metal dark under adhesive
KC-5-2	1027	KC-5-IA-2	140		KC-5-IN-2	803	
KC-5-3	710	KC-5-IA-3	425		KC-5-IN-3	350	
KC-5-4	936	KC-5-IA-4	627		KC-5-IN-4	335	
KC-5-5	1060	KC-5-IA-5	Failed		KC-5-IN-5	505	
Average	924		363			518	
Specimen KC-6 (Kopox 171/Antimony Trioxide/BF_3-400)							
KC-6-1	680	KC-6-IA-1	Failed	Poor flaked adhesive film	KC-6-IN-1	Failed	Poor adhesive film dissolved
KC-6-2	412	KC-6-IA-2	Failed		KC-6-IN-2	Failed	
KC-6-3	218	KC-6-IA-3	Failed		KC-6-IN-3	Failed	
KC-6-4	300	KC-6-IA-4	131		KC-6-IN-4	879	
KC-6-5	Failed	KC-6-IA-5	Failed		KC-6-IN-5	Failed	
Average	302						
Specimen KC-7 (Kopox 171/Zytel 61/Antimony Trioxide/Dicyandianiline)							
KC-7-1	3975	KC-7-IA-1	3676	Good no change from original specimens	KC-7-IN-1	Failed	Poor adhesive film dissolved completely
KC-7-2	3771	KC-7-IA-2	3725		KC-7-IN-2	Failed	
KC-7-3	4035	KC-7-IA-3	3660		KC-7-IN-3	Failed	
KC-7-4	3762	KC-7-IA-4	3718		KC-7-IN-4	Failed	
KC-7-5	3775	KC-7-IA-5	3663		KC-7-IN-5	Failed	
Average	3864		3688				
Specimen KC-8 (DEN 438/Antimony Trioxide/BF_3-400)							

Adhesive KC-8 failed to bond metal panels together

TABLE 17
COMPOSITION AND CURE OF ADHESIVE CANDIDATES (KC-9 THROUGH KC-14)

Adhesive	Composition: Material	Composition: Parts by Weight	Cure: Time and Temp	Cure: Pressure, psi
KC-9	Kopox 171[1] Z Catalyst[2]	100.00 25.00 125.00	2 hours at 150°F plus 2 hours at 300°F	14.7 (vacuum bag)
KC-10	Kopox 171 Alcoa 101[3] Cab-O-Sil[4] Z Catalyst	100.00 50.00 5.00 25.00 180.00	Same	14.7 (vacuum bag)
KC-11	Epon 422[5]	100.00	Same	14.7 (vacuum bag)
KC-12	Kel-F-KX633 dispersion[6]	100.00	30 min at 500°F, plus 15 min at 400°F, plus 30 min at 500°F	25 (hydraulic press)
KC-13	Kynar dispersion[7] 45% Solids	100.00	10 min at 320°F, raise to 550°F, hold 30 min at 550°F, plus 15 min at 320°F, plus 30 min at 500°F	25 (hydraulic press)
KC-14	Teflon-FEP 100-A film[8]	-	45 sec at 550°F plus 5 sec dwell at 550°F	50 to 100 (induction heater)

(1) Koppers Company polyepoxy resin

(2) Shell Chemical Company blend of methylene dianiline and meta phenylenediamine.

(3) Aluminum Company of America aluminum powder.

(4) Cabot Company silica powder.

(5) Shell structural adhesive.

(6) Minnesota Mining & Manufacturing Company trifluorochloroethylene.

(7) Pennsalt Chemicals Company vinylidene fluoride.

(8) E. I. du Pont de Nemours & Company fluorinated ethylene propylene (FEP) copolymer.

TABLE 18
RESULTS OF ADHESIVE-BONDING STUDY (KC-9 THROUGH KC-14)

Adhesive	Before Exposure: Shear Strength psi	Before Exposure: Failure Type[1]	After Exposure, In AeroZINE 50, 7 Days at 100°F: Shear Strength psi	Failure Type	30 Days at 100°F: Shear Strength psi	Failure Type	After Exposure, In N_2O_4, 7 Days at 100°F: Shear Strength psi	Failure Type	30 Days at 60°F: Shear Strength psi	Failure Type
KC-9 (Kopox 171/Z Catalyst)	2024	A-M	2417	A-M	2387	C-A	2324	A-M	1950	C-A
KC-10 (Kopox 171/Alcoa 101/Cab-O-Sil/Z Catalyst)	1239	A-M,C-A	Failed during exposure			-	804	A-M	556	C-A
KC-11 (Epon 422)	806	C-A	Failed during exposure			-	Failed during exposure			-
KC-12 (Kel-F-KX633 dispersion)	2412	C-A	-	-	-	-	525	A-M,C-A	-	-
KC-13 (Kynar dispersion)	1939	C-A	-	-	-	-	Failed during exposure			-
KC-14 (Teflon-FEP 100-A film)	384	A-M	-	-	-	-	Failed during exposure			-

(1) Failure code: A-M = adhesive-to-metal failure. C-A = cohesive-to-adhesive failure.

Both KC-13 (the Kynar dispersion) and KC-14 (the Teflon FEP 100-A film adhesive) failed to pass the 7-day test at 100°F in N_2O_4 and were not evaluated further. Higher bonding pressures might improve the bond obtained with these adhesives, but high bonding pressures are not considered practical for use in joining metal segments of liners.

In a further study of adhesive bonding, a series of formulations containing various types of flexibilizers for Kopox 171 were evaluated for compatibility with the propellants. Their compositions and cures are shown in Table 19. In this series, various combinations of Kopox 171 and Kel-F dispersions were evaluated. Epon 871, a Shell Chemical Company polyepoxy resin, was also checked for adhesion. Amoco AI(10), a new Amoco Chemicals Corporation polyimide resin, was also evaluated as a solution in DMF (dimethyl formamide) and in combination with Kopox 171.

The results of the shear tests before and after exposure to propellants are presented in Table 20. Only the more promising adhesive candidates in this series were subjected to the exposure tests. KC-21 (a combination of Kopox 171 and the polyimide resin) and KC-22 (a combination of Kopox 171 and Kel-F primer with resin dispersion) looked very promising. It appears that the use of Kel-F-KX643, the primer, is necessary to produce the best results. In KC-21, the addition of Amoco AI(10), the polyimide resin, to Kopox 171, improved the original shear strength of the Kopox 171 system. However, the shear strength of KC-21 was not as high as that of KC-9, the undiluted Kopox resin system, after exposure to the propellants.

The use of Epon 871 as a flexibilizer for Kopox 171 in KC-18 and -19 was not effective; the original shear strength of the Kopox 171 adhesive system was drastically reduced. Various levels of Kel-F-KX633 dispersion in Kopox 171 were also not of any benefit, as indicated by the results for KC-15, -16, and -17. Although the addition of Amoco AI(10) to Kopox 171 was effective in improving shear strength, the use in KC-20 of this resin alone did not produce a satisfactory adhesive.

In view of the promising shear-strength results for KC-9 and -22, they were subjected to peel-adhesion and uniaxial cyclic strain tests. Sheets of 0.006-in.-thick foils of Al 1100-0 were bonded together with the candidate adhesives, and these laminates were affixed to a semirigid substrate of epoxy

TABLE 19
COMPOSITION AND CURE OF ADHESIVE CANDIDATES (KC-15 THROUGH KC-23)

Adhesive	Composition: Material	Composition: Parts by Weight	Cure: Time and Temp	Cure: Pressure, psi
KC-15	Kopox 171 (1) Kel-F-KX633 (2) Z Catalyst (3)	100.00 5.00 25.00 130.00	2 hours at 150°F plus 2 hours at 300°F	14.7 (vacuum bag)
KC-16	Kopox 171 Kel-F-KX633 Z Catalyst	100.00 10.00 25.00 135.00	Same	Same
KC-17	Kopox 171 Kel-F-KX633 Z Catalyst	100.00 20.00 25.00 145.00	Same	Same
KC-18	Kopox 171 Epon 871 (4) Z Catalyst	100.00 10.00 25.00 135.00	Same	Same
KC-19	Kopox 171 Epon 871 Z Catalyst	100.00 20.00 25.00 145.00	Same	Same
KC-20	Amoco AI(10) (5) 30% solution in dimethylformamide	-	30 min at 300°F plus 30 min at 400°F	25 (press)
KC-21	Kopox 171 Amoco AI(10) Z Catalyst	100.00 30.00 25.00 155.00	30 min at 150°F plus 2 hours at 250°F	Same
KC-22	Kopox 171 Kel-F-KX643 Kel-F-KX633 Z Catalyst	100.00 10.00 10.00 25.00 145.00	Same	Same
KC-23	Kel-F-KX643 Kel-F-KX633	50.00 50.00	30 min at 500°F plus 15 min at 400°F, plus 30 min at 500°F	None

(1) Koppers Company polyepoxy resin.
(2) Minnesota Mining & Manufacturing Company trifluorochloroethylene.
(3) Shell Chemical Company blend of MDA and MPDA.
(4) Shell polyepoxy resin.
(5) Amoco Chemicals Corporation polyimide resin.

TABLE 20

RESULTS OF ADHESIVE-BONDING STUDY (KC-15 THROUGH KC-23)

	Before Exposure		After Exposure for 7 Days at 100°F			
			In AeroZINE 50		In N_2O_4	
Adhesive	Shear Strength psi	Failure Type(1)	Shear Strength psi	Failure Type	Shear Strength psi	Failure Type
KC-15	[Kopox 171 (100.00)/Kel-F-KX633 (5.00)/Z Catalyst (25.00)]					
	1069	A-M	-	-	-	-
KC-16	[Kopox-171 (100.00)/Kel-F-KX633 (10.00)/Z Catalyst (25.00)]					
	1165	A-M	-	-	-	-
KC-17	[Kopox 171 (100.00)/Kel-F-KX633 (20.00)/Z Catalyst (25.00)]					
	1236	A-M	946	C-A	1046	C-A
KC-18	[Kopox 171 (100.00)/Epon 871 (10.00)/Z Catalyst (25.00)]					
	681	A-M	-	-	-	-
KC-19	[Kopox 171 (100.00)/Epon 871 (20.00)/Z Catalyst (25.00)]					
	517	A-M	-	-	-	-
KC-20	[Amoco AI (10), 30% solution in dimethylformamide]					
	All specimens separated when removed from press.					
KC-21	[Kopox 171 (100.00)/Amoco AI (10) (30.00)/Z Catalyst (25.00)]					
	2500	C-A	1902	A-M	1847	C-A
KC-22	[Kopox 171 (100.00)/Kel-F-KX633 (10.00)/Kel-F-KX643 (10.00)/Z Catalyst (25.00)]					
	2264	C-A	2081	A-M	2116	A-M
KC-23	[Kopox 171 (100.00)/Z Catalyst (25.00)/Kel F-KX633 (50.00)/Kel-F-KX643 (50.00)]					
	208	A-M	-	-	-	-

(1) Failure code: A-M = adhesive-to-metal failure. C-A = cohesive-to-adhesive failure.

coated Dacron fabric with Narmco 7343/7139 adhesive. One-inch-wide strips of these composites were tested for T-peel adhesion and uniaxial cyclic strain. The results of the T-peel tests (conducted at 75°F and a head speed of 0.5 in./min) were as follows:

Adhesive	T-Peel Adhesion, lb/in.
KC-9-1	13.2
KC-9-2	12.2
KC-9-3	14.3
	Av 13.2
KC-22-1	3.5
KC-22-2	3.0
KC-22-3	3.3
	Av 3.3

The results of the strain tests (performed at 75°F and a head speed of 0.05 in./min) were as follows:

Adhesive	No. of Cycles	Strain Level, %	Permanent Set, %	Remarks
KC-9	30	2.0	1.0	No separation
KC-9	30	2.0	1.0	No separation
KC-9	30	2.5	1.0	No separation
KC-9	30	3.0	2.0	No separation
KC-22	30	2.0	1.0	No separation
KC-22	30	2.0	1.0	No separation
KC-22	4	2.5	-	Metal cracked
KC-22	30	2.5	1.0	No separation
KC-22	5	3.0	-	Metal cracked
KC-22	12	3.0	-	Metal cracked

These tests indicate that the KC-9 adhesive system is superior to KC-22 with regard to T-peel adhesion and uniaxial cyclic strain. The addition of Kel-F dispersions apparently did not flexibilize the Kopox 171 resin as intended. Both adhesives were satisfactory at the 2.0% strain level, however. The encouraging performance of KC-9 in the bonding of aluminum foil indicates that further exploration of this adhesive for the joining of liner segments is justified.

G. LOW-TEMPERATURE ADHESIVES

Because the thermal cycling of subscale tanks containing ClF_3 in Phase IV was to include a temperature reduction to -110°F, the properties of several candidate adhesives were studied at this temperature to ensure satisfactory performance.

Laminates of 0.0065-in.-thick 347 SS foil bonded to resin-coated 181 glass fabric by the candidate adhesives were tested for adhesion at +75 and -110°F. The values obtained (at a head speed of 0.5 in./min) were:

	T-Peel Adhesion, lb/in.	
Adhesive	75°F	-110°F
Y-9141	5.0	3.0
AF-111	4.0	2.0
Narmco 7343/7139	14.0	10.0

These results indicate that the Narmco 7343/7139 should perform satisfactorily at -110°F, whereas the performance of Y-9141 and AF-111 may be questionable.

The room-temperature adhesion of Y-9141 was only 5.0 lb/in., which appears to be lower than normal for this adhesive and may have been due to softening of the film by the epoxy resin used for impregnation of the glass-fabric substrate. The effect of pre-curing the adhesive before bonding to the resin-coated substrate was therefore studied, and uniaxial cyclic strain tests of the bonded panels were included.

The 0.0065-in.-thick 347 SS foil was thoroughly cleaned with acid and was coated and cured as noted below.

	Adhesive		Pre-cure	
Specimen	Identification	Type	Time, min	Temp, °F
S/S-19	Y-9141	Resin-coated film	60	300
S/S-20	Narmco 7343/7139	Urethane paste	30	250
S/S-21	3M 3512 A, B, and C	Urethane paste	30	250

Four plies of Dacron 2353, impregnated with a DER 332 (100.00), Epicure 855 (80.00) resin system, were impressed on the pre-cured adhesive-coated metal foil and the specimens (S/S-19, -20, and -21) were cured (at a pressure of 25 psi) for 30 min at 150°F plus 60 min at 250°F.

T-peel adhesion and uniaxial strain tests were made on 1-in.-wide strips of these composites at +75 and -110°F. Strain tests were not conducted on the S/S-19 specimens at -110°F because the T-peel adhesion test showed there was no bond between the 347 SS foil and the coated Dacron at this temperature. The test results are presented in Table 21.

The adhesion was higher at 75°F for this series of specimens than for those that had been assembled and cured in one operation, but at -110°F was lower than that of the one-shot cured specimens. The relative standing of the Y-9141 and Narmco 7343 adhesives remained the same as before. The uniaxial cycling test results indicated the superiority of the two urethane adhesives at low temperatures.

On the basis of these and the earlier tests, the Narmco 7343/7139 urethane adhesive was selected for use in the fabrication of the subscale tanks for ClF_3. It was considered that this adhesive should preserve the bond between the stainless steel liner and the filament-wound case during thermal cycling at -110°F.

H. CONCLUSIONS

The propellant-exposure tests performed in Phase III produced major degradation at resin/glass-filament composites and emphasized the need for complete separation of the propellant and the filament-wound case. The results of the vacuum-exposure tests indicated that epoxy/glass-filament composites satisfactorily resist deterioration by vacuum and heat.

Bonding and uniaxial-strain tests of metal-foil/fabric laminates demonstrated a concept that could provide a solution to the structural-compatibility problems of metal-lined filament-wound tankage.

Adhesive-bonding studies indicated that the technique had promise in the fabrication of metallic liners for filament-wound tankage.

TABLE 21
RESULTS OF ADHESION AND UNIAXIAL CYCLIC STRAIN TESTS OF ADHESIVES

Specimen	T-Peel Adhesion[1], lb/in.		Uniaxial Strain, 30 Cycles at 2% Strain[2]					
			Load, lb		Set, %		Condition of Specimens	
	75°F	-100°F	75°F	-110°F	75°F	-110°F	75°F	-110°F
S/S 19 (0.0065-in. 347 SS/Y-9141 adhesive/2353 Dacron impregnated with DER 332/Epicure 855 resin system)								
-1	16	0	525		2	No test	Satisfactory	No test
-2	18	0	500		2	No test	Satisfactory	No test
Average	17		512		2			
S/S 20 (0.0065-in. 347 SS/7343 adhesive/2353 Dacron impregnated with DER 332/Epicure 855 resin system)								
-1	14	5	400	950	2	2	Satisfactory	Satisfactory
-2	16	6	400	925	2	2	Satisfactory	Satisfactory
Average	15	5.5	400	938	2	2		
S/S 21 (0.0065-in. 347 SS/3512 A,B,&C adhesives/2353 Dacron impregnated with DER 332/Epicure 855 resin system)								
-1	10	1	450	850	2	2	Satisfactory	Satisfactory
-2	10	1	450		2		Satisfactory	
Average	10	1	450		2			

(1) T-peel adhesion tests performed at a head-travel speed of 0.5 in./min.
(2) Uniaxial strain tests performed at a head-travel speed of 0.05 in./min.

VI. PHASE IV - FABRICATION AND EVALUATION OF SUBSCALE CHAMBERS

The objectives of Phase IV were (a) to determine the long-term performance of subscale tankage upon exposure to propellants, and (b) to evaluate materials and fabrication techniques under conditions simulating those expected of filament-wound propellant tankage in service.

A. PROPELLANT-ENVIRONMENT TESTING

The material-evaluation studies conducted in Phases I, II, and III indicated the necessity for a barrier-type liner in filament-wound tankage. Although resin systems having a moderate degree of resistance to the propellants of interest were found, they would not retain satisfactory structural integrity if exposed to these propellants for long periods. In the form of resin/glass composites, these resins were particularly vulnerable to attack. Organic liner materials, such as plastic films, are permeable to the propellants and thus would not protect the filament-wound case. It was therefore concluded that a metallic liner with an overwrap of glass filament and a propellant-resistant resin system were needed to provide the reliability required of filament-wound tankage in a space environment.

1. Fabrication

Type 347 SS was chosen for the liners of subscale tankage on the basis of good corrosion resistance to all the propellants, favorable physical properties, and ease of welding. Liners for the subscale tanks were fabricated in the following sequence of operations:

a. Bosses were machined from 347 SS bar stock.

b. Hemispherical segments were pressure-formed by a plug and ring process from 0.0062-in.-thick 347 SS foil.

c. The formed segments were stress-relieved by heat treatment.

d. The segments were trimmed to the required dimensions and openings were cut for the bosses.

e. The hemispherical segments were joined to the bosses by resistance-roll-seam welding.

f. The segments with bosses attached were joined together at the girth by resistance-roll-seam welding on a lap joint.

The fabricated liner (Part No. 17754 9) is shown schematically in Figure 14 and photographically in Figure 15. After fabrication, each liner was checked for leaks with the aid of a helium mass-spectrometer leak detector.

The metal liners were then reinforced on the inside by DMM plaster cast to a thickness of 1 in. The plaster and liner were heated for 24 hours at 250°F to cure the plaster. The outside surface of the liner was cleaned with a 50/50-vol% mixture of fuming nitric acid and water, with 0.02 wt% of hydrofluoric acid.

The cleaned liner was coated with Y-9141 adhesive for the AeroZINE 50 and N_2O_4 tanks. The liners of the ClF_3 tanks were coated with Narmco 7343/7139 adhesive.

The adhesive-coated liners were filament-wound with S-HTS roving preimpregnated with RS-11, a novalac epoxy resin system. Each tank was given a cure of 2 hours at 200°F, plus 2 hours at 250°F, plus 10 hours at 300°F. Following the cure, the plaster was washed out with acetic acid. Figure 16 shows the subscale tank (Part No. 17550) schematically.

2. Evaluation

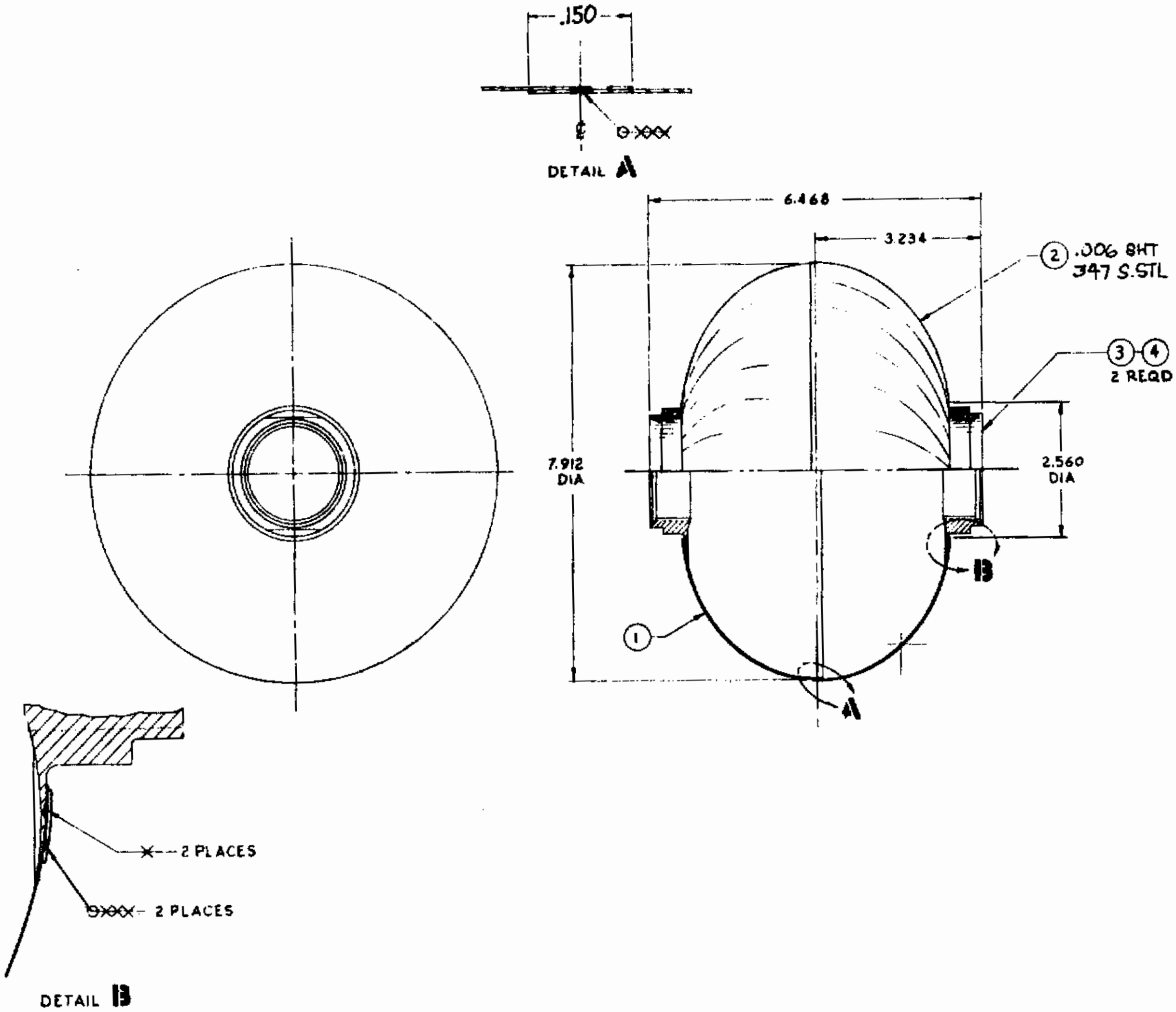
Each of these subscale tanks was solvent-cleaned and fitted with a filling tube and a pressure gage as shown in Figure 17. The tanks for N_2O_4 and ClF_3 were carefully passivated. Each tank was half filled with propellant and was pressurized to 150 psig with nitrogen.

These tanks were then placed in separate environmental chambers and were temperature-cycled every 4 hours as follows:

Propellant	Temp, °F Maximum	Temp, °F Minimum
AeroZINE 50	158	18
N_2O_4	70	12
ClF_3	53	-110

a. Results After One Month of Exposure

One subscale tank was removed from each environmental chamber after 1 month of temperature cycling and exposure. After decontamination,



Part Number 177549

Item No.	Part No.	Description	Material
1	-3	Shell	.006 Type 347 S. Stl.
2	-5	Shell	.006 Type 347 S. Stl.
3	177419-3	Boss	Type 347 S. Stl.
4	177419-3	Boss	Type 347 S. Stl.

Figure 14. Metallic Liner, 8-in. Oblate-Spheroid Tank (Schematic)

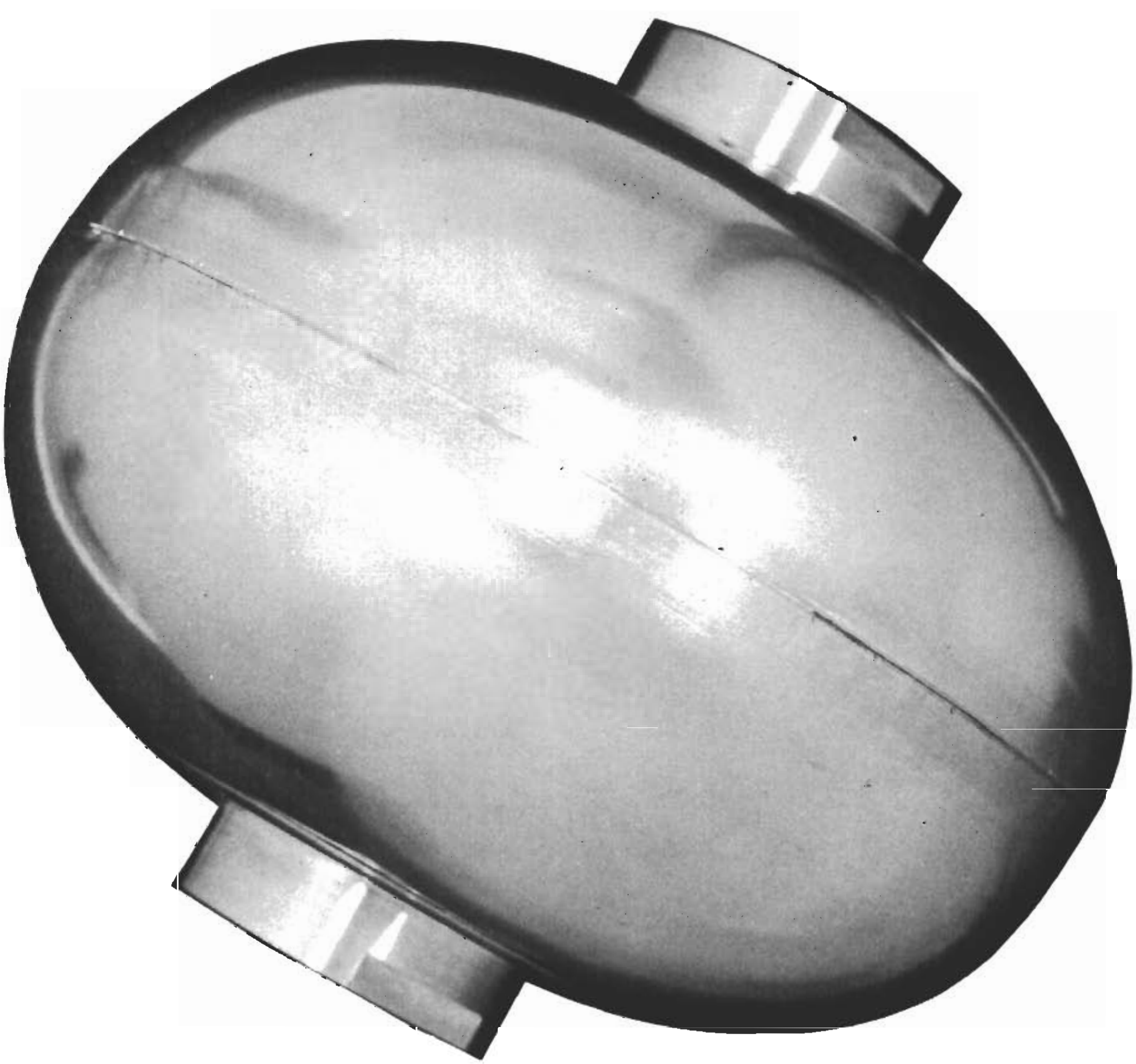


Figure 15. View of Welded Liner for Subscale Oblate-Spheroid Tank

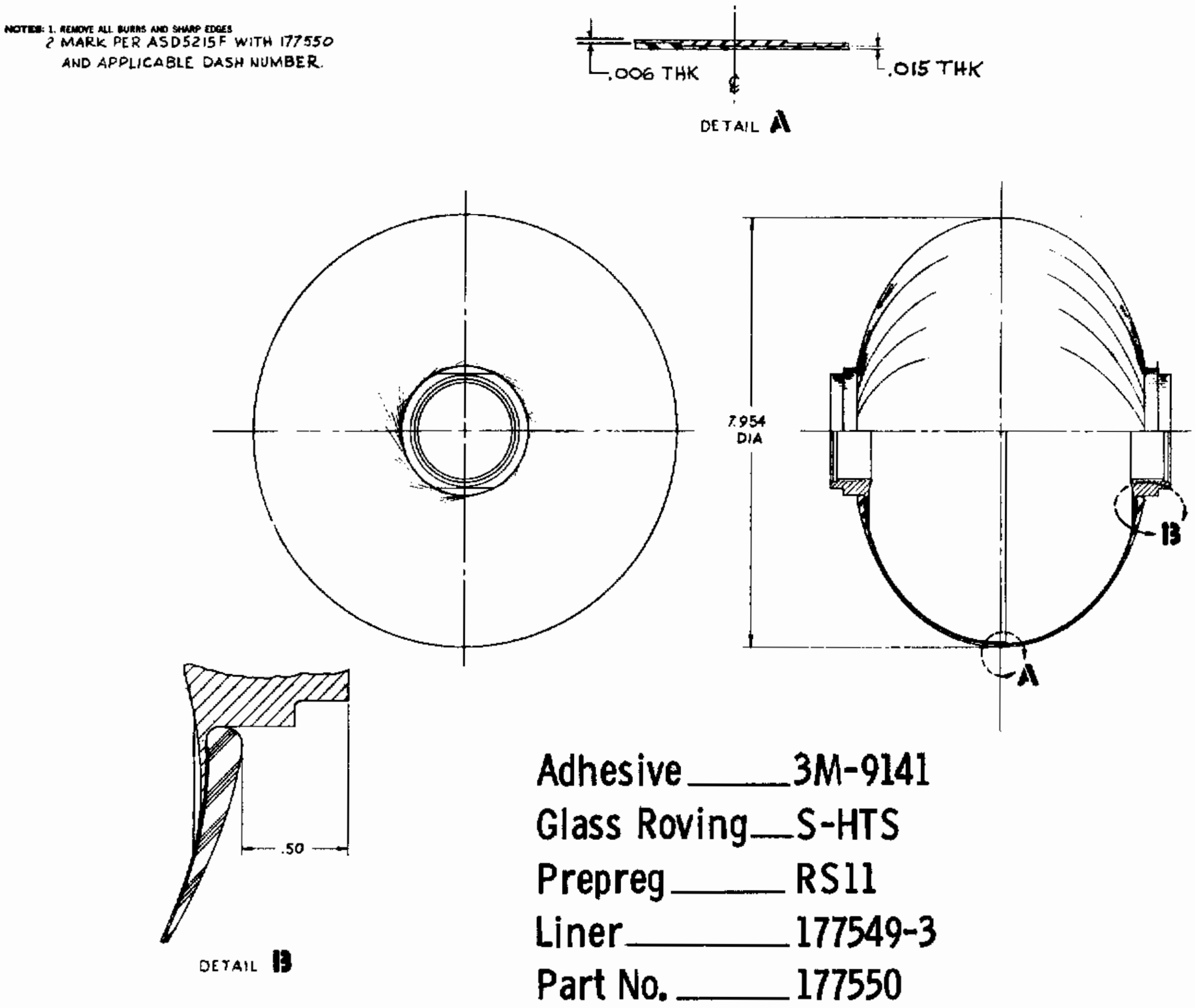


Figure 16. Subscale Oblate-Spheroid Tank (Schematic)

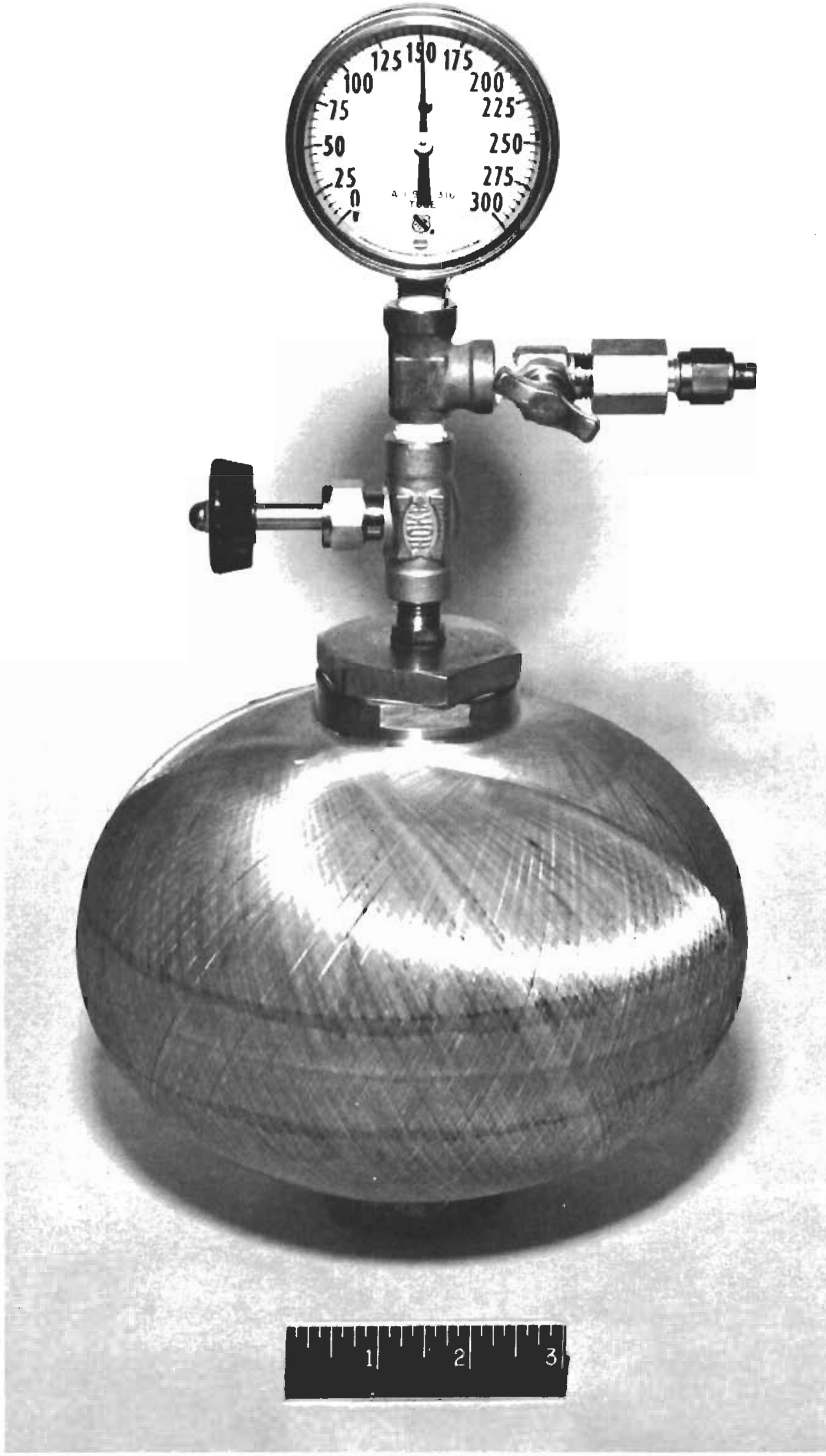


Figure 17. Pressurized Subscale Tank for Environmental Testing

helium leak tests were made, each tank was pressure-cycled 25 times between zero and 150 psig, and a second helium leak test was made to determine if pressure cycling had weakened the structures. Each tank was then instrumented and pressurized to the burst point. The liners were examined for corrosion and any other effects of the propellant and thermal cycling. Burst tests were also made on unexposed control tanks at the same time.

The results are presented in Table 22 and Figure 18. The subscale tanks after burst testing are shown in Figure 19. The appearance of the liners and tanks after propellant exposure is described below.

(1) AeroZINE 50 Tank

This tank was in good condition at the end of the 1-month exposure. No liner corrosion occurred. The thermal cycling did not affect the strength of the filament-wound case; the burst strength was 1300 psig, which was higher than that of either of the control tanks. The liner burst with a vertical split parallel to the glass filaments. The welded areas did not fail. The burst had caused the bosses to be offset from each other by approximately 2 in., probably because of the restraining effect of the metal liner on the normal burst pattern.

(2) N_2O_4 Tank

This tank was also in good condition after exposure. There was no evidence of liner corrosion. The burst strength (1160 psig) was approximately the same as that of the controls. The same boss offset was noted as for the tank above.

(3) ClF_3 Tank

This tank developed a small leak after 28 days of exposure. The propellant was removed and the tank was decontaminated. Examination showed no obvious liner breakdown. The leak was located at the weld line at the edge of the flange on the boss. The combined effects of the corrosive propellant, thermal cycling, and pressure had apparently produced a failure at the weld. This weld appears to be a weak point in this particular group of liners. The weakness was corrected in the fabrication of a new group of liners by the addition of a doubler at the weld area and by moving the roll-seam weld 1/16 in. back from the edge of the boss flange.

TABLE 22
RESULTS OF BURST TESTS OF SUBSCALE TANKS[1]
30-DAY EXPOSURE, ENVIRONMENTAL-TEST PROGRAM

Pressure psig	Increase in Tank Height at Boss, in.			
	Control Tanks		Experimental Tanks After Exposure	
	No. 1	No. 2	Aerozine 50	N_2O_4
200	0.050	0.053	0.040	0.040
400	0.105	0.110	0.096	0.078
600	0.164	0.168	0.150	0.120
800	0.194	0.198	0.174	0.150
1000	0.223	0.220	0.183	0.165
1100	-	0.230 (burst)	-	-
1160	-	-	-	0.175 (burst)
1200	0.245 (burst)	-	0.200	-
1300	-	-	0.213 (burst)	-

(1) Fabrication details for control and experimental tanks: Liner, 347 SS; glass filament, S-HTS 20-end roving prepreg; resin binder, RS-11 (DEN 438/BF_3-400); adhesive on liner, 3M-Y 9141; tension on tape, 8 lb; number of turns of tape per revolution, 248; number of revolutions, 1.

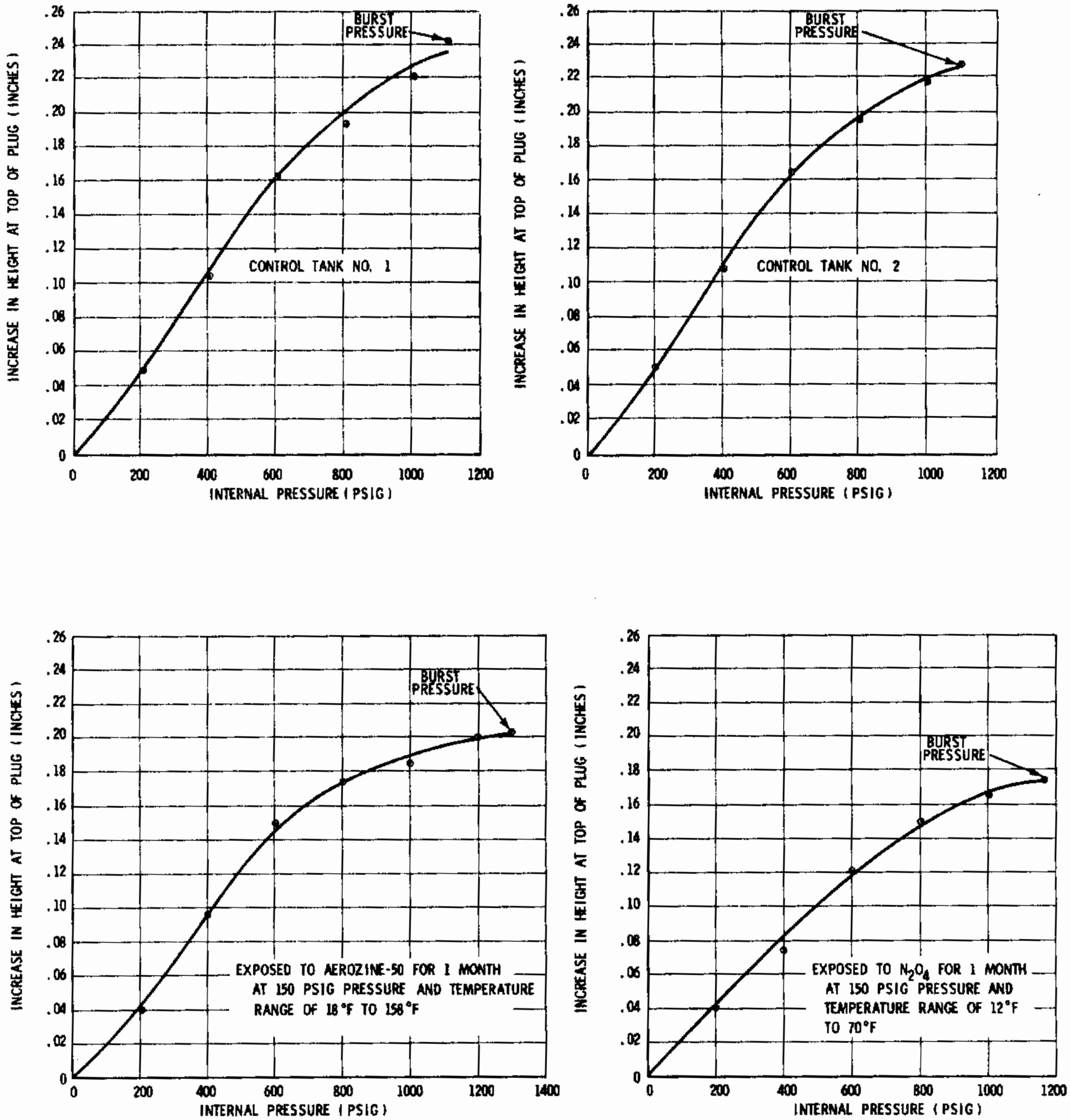


Figure 18. Results of Burst Tests of Subscale Tanks

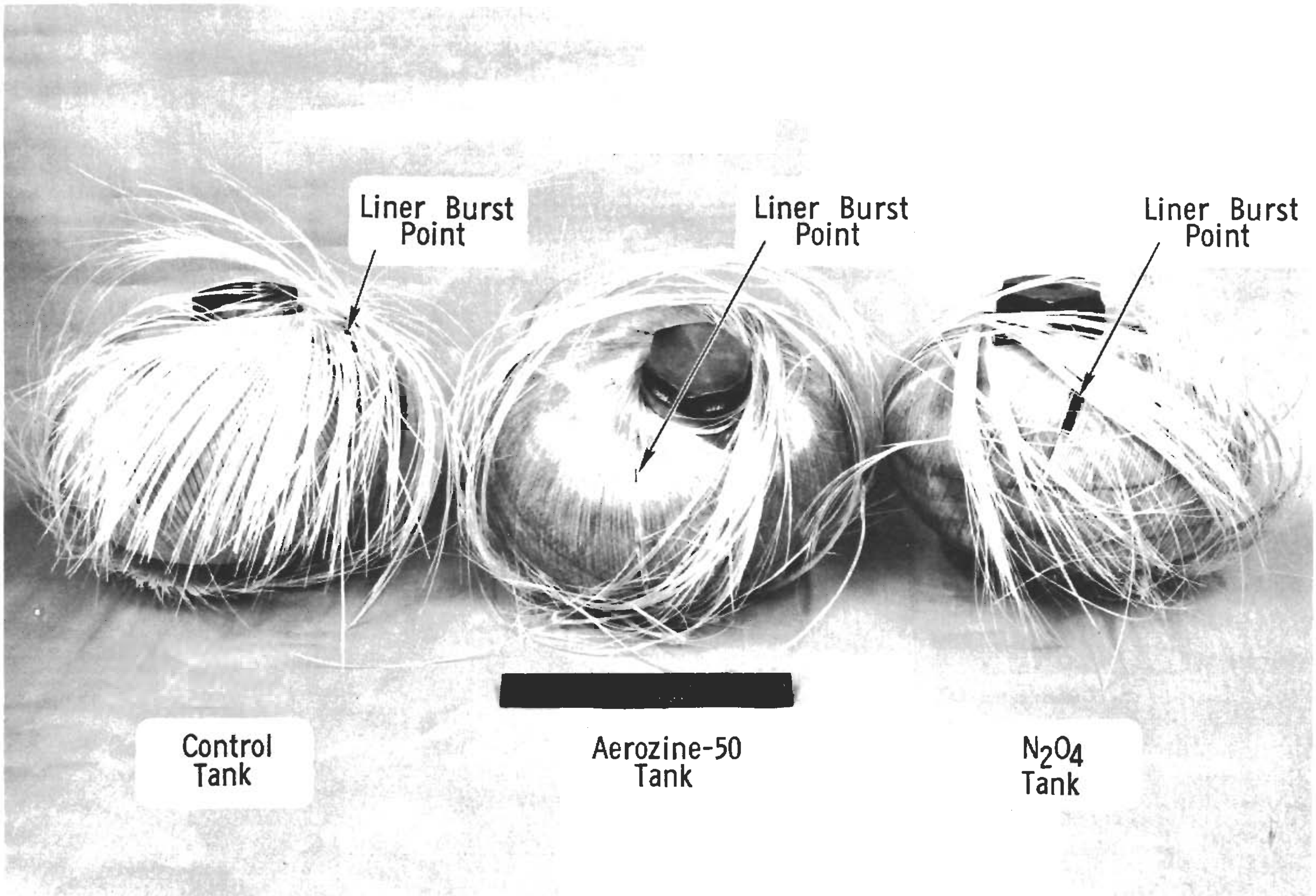


Figure 19. Subscale Tanks After Burst Testing

Figure 20 shows this tank after removal from the test. The leaking ClF_3 had dissolved the glass filaments and resin binder for approximately 1/2 in. around the leak in the liner, but no major catastrophic degradation of the filament-wound case had occurred. This indicates the value of the material-compatibility studies and the selection of a corrosion-resistant resin system.

The leaking tank was replaced by another subscale tank of the same fabrication group. The second tank also developed a leak, after 20 days of exposure. It is known that carbides can be precipitated at the grain boundaries when stainless steels are welded or used in or above the critical high-temperature range. When this occurs, the metal becomes susceptible to intergranular corrosion in the presence of certain corrosive media (Reference 21). This effect, plus the reduction in liner thickness for this particular group of liners, apparently accounted for the failure of the two ClF_3 tanks. One of the original subscale tanks containing ClF_3 functioned satisfactorily.

b. Results After Four Months of Exposure

A second subscale tank was removed from each of the three environmental chambers after 4 months of temperature cycling and propellant exposure. These tanks are shown in Figure 21. After decontamination, the tanks were leak-checked and found to be satisfactory. They were then pressurized 25 times between zero and 150 psig, and all passed a second leak test. Following this, they were instrumented and pressurized to the burst point. The results are presented in Table 23 and Figures 22, 23, and 24, and are discussed below.

(1) AeroZINE 50 Tank

This tank was in good condition at the end of the exposure period. There was no evident liner corrosion and no reduction in the strength of the filament-wound chamber.

(2) N_2O_4 Tank

This tank was also in good condition. There was no evident liner corrosion, and the burst strength (1200 psig) was equal to that of the control tanks.

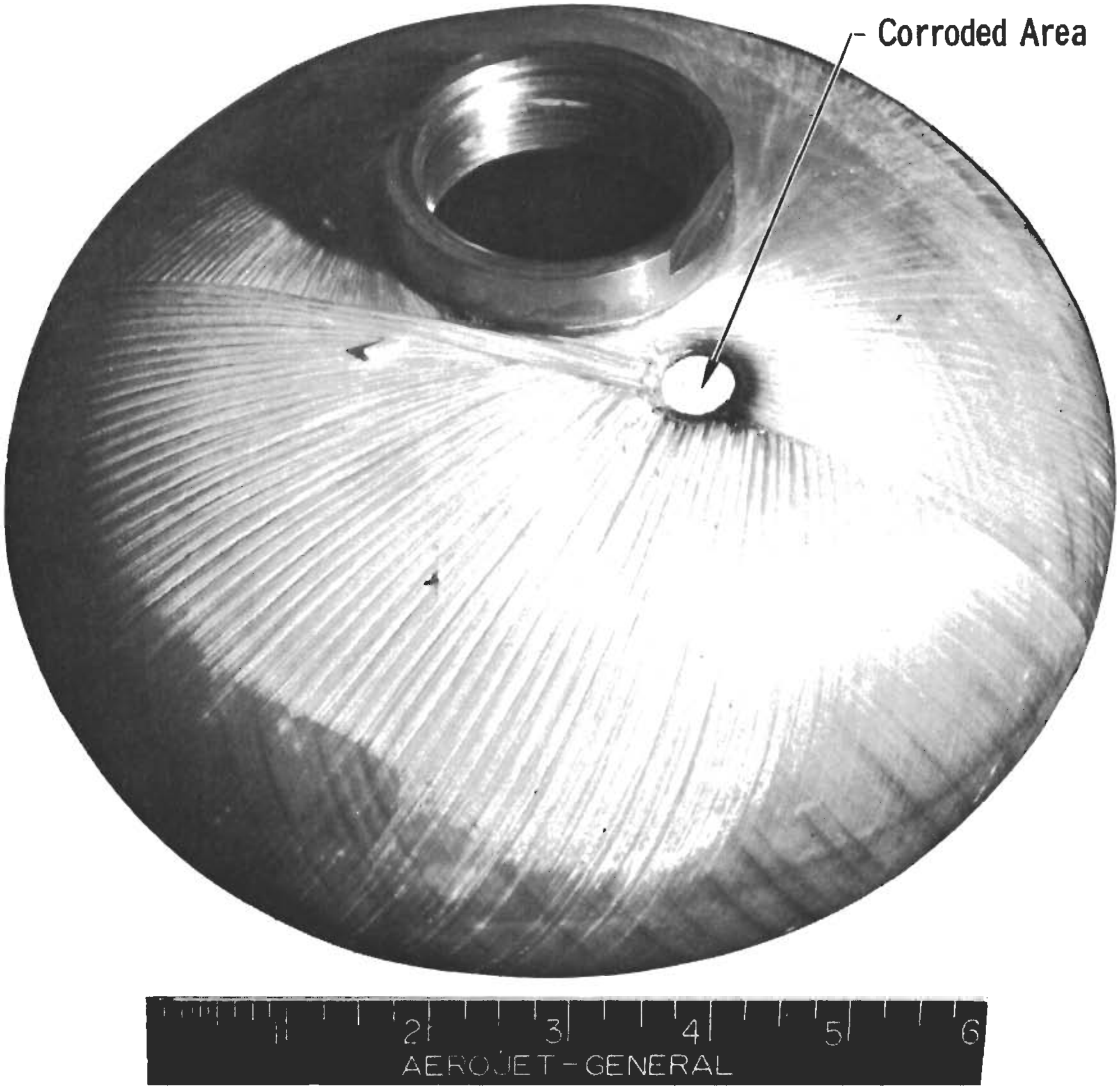


Figure 20. Subscale Tank After Exposure to Chlorine Trifluoride (ClF_3) for 28 Days at +53 to -110°F

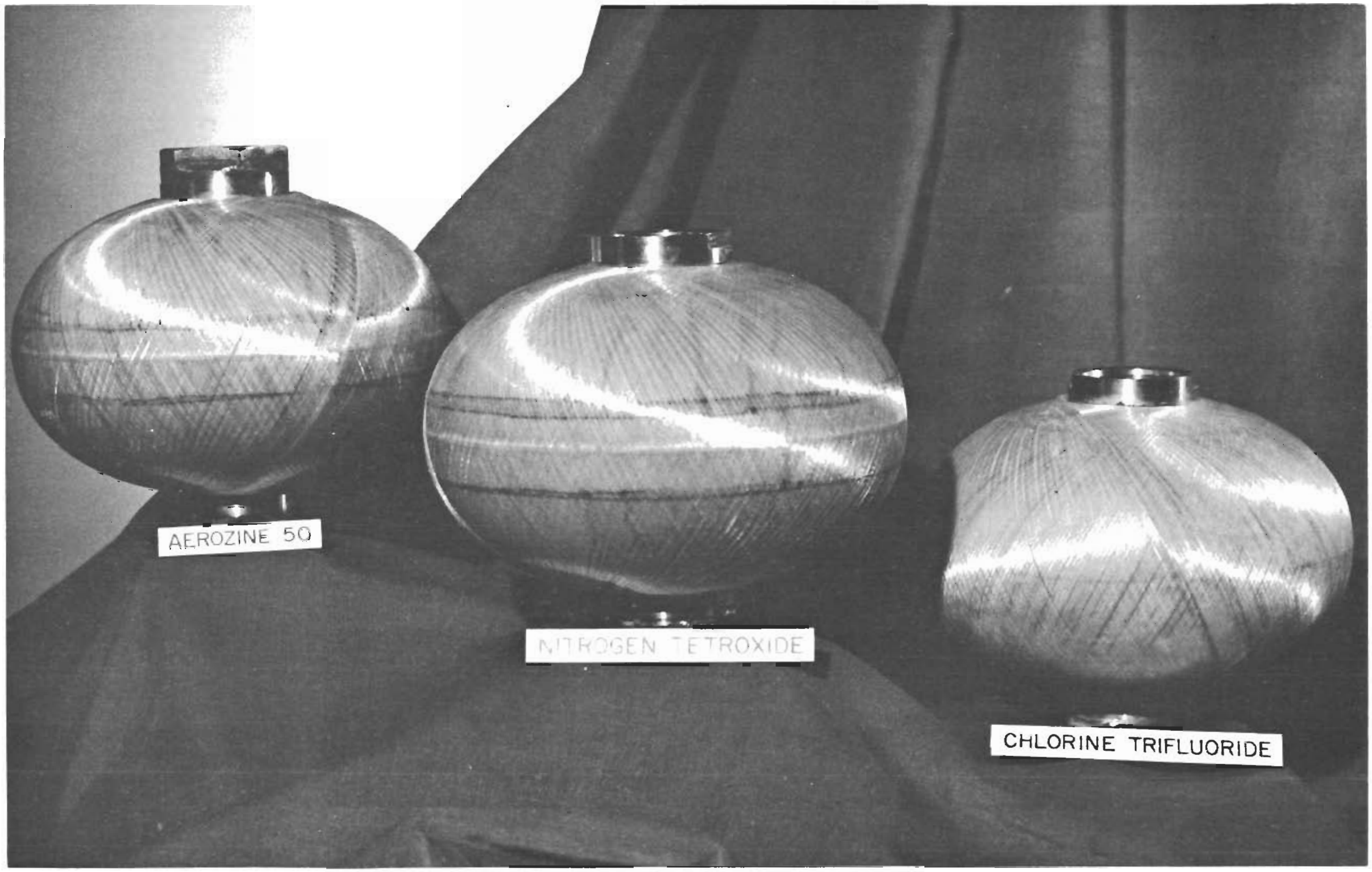


Figure 21. Subscale Tanks After 4-Month Exposure to Propellants

TABLE 23
RESULTS OF BURST TESTS OF SUBSCALE TANKS[1]
120-DAY EXPOSURE, ENVIRONMENTAL-TEST PROGRAM

Pressure psig	Dimensional Increases, in., After 120-Day Exposure					
	AeroZINE 50		N_2O_4		ClF_3	
	Height	Girth	Height	Girth	Height	Girth
100	0.010	0.000	0.020	0.000	0.030	0.000
200	0.030	0.000	0.050	0.000	0.050	0.000
300	0.070	0.000	0.080	0.000	0.080	0.005
400	0.100	0.000	0.130	0.000	0.120	0.010
500	0.125	0.000	0.150	0.000	0.130	0.020
600	0.150	0.10	0.160	0.000	0.140	0.050
700	0.170	0.020	0.170	0.000	0.150	0.070
800	0.185	0.050	0.180	0.005	0.170	0.100
900	0.200	0.120	0.200	0.010	0.180	0.125
1000	0.210	0.145	0.210	0.040	0.200	0.140
1100	0.220	0.170	0.230	0.080	0.250	0.180
1200	0.230	0.200	0.235	0.120	-	-
1220	0.235	0.210	-	-	-	-

(1) Tank-fabrication details: Liner, 0.0062-in.-thick 347 SS; glass filament, S-HTS 20-end roving prepreg; resin binder, RS-11 (DEN 438/BF_3-400); adhesive on liner, 3M-9141; tension on tape, 8 lb; number of turns of tape per revolution, 248; number of revolutions, 1.

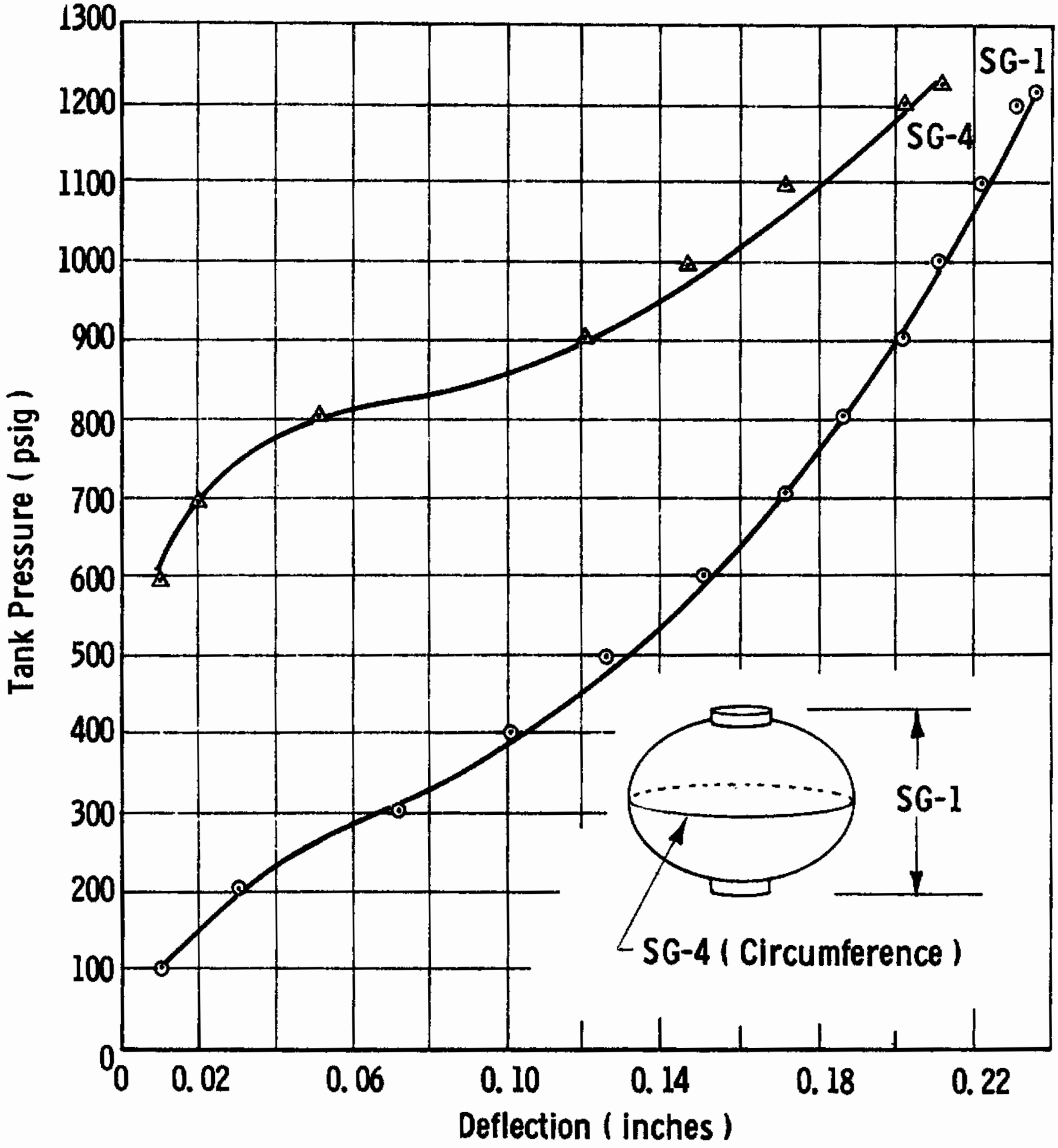


Figure 22. Deflection of Tank During Burst Tests
After 4-Month Exposure to AeroZINE 50

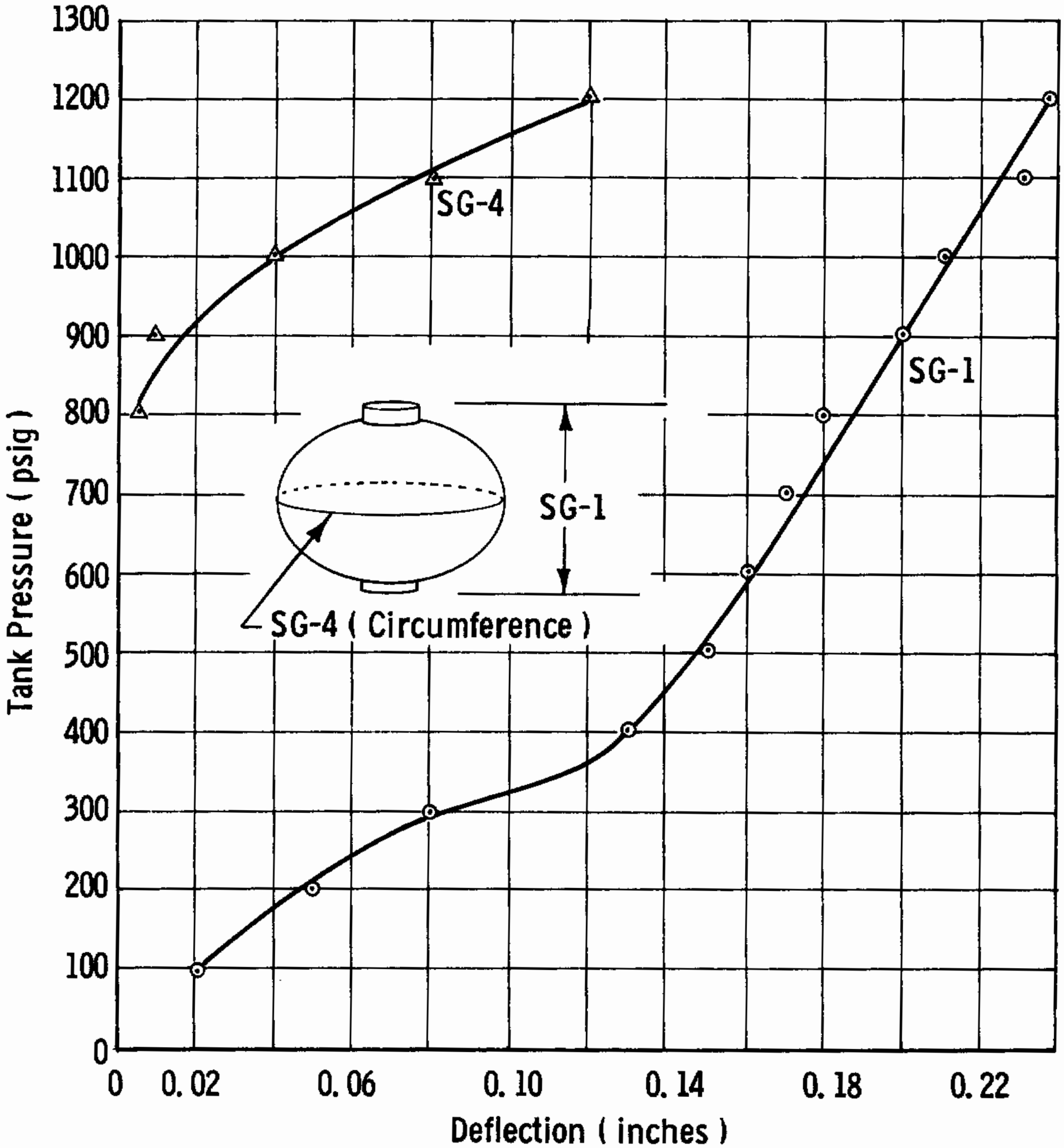


Figure 23. Deflection of Tank During Burst Test After 4-Month Exposure to N_2O_4

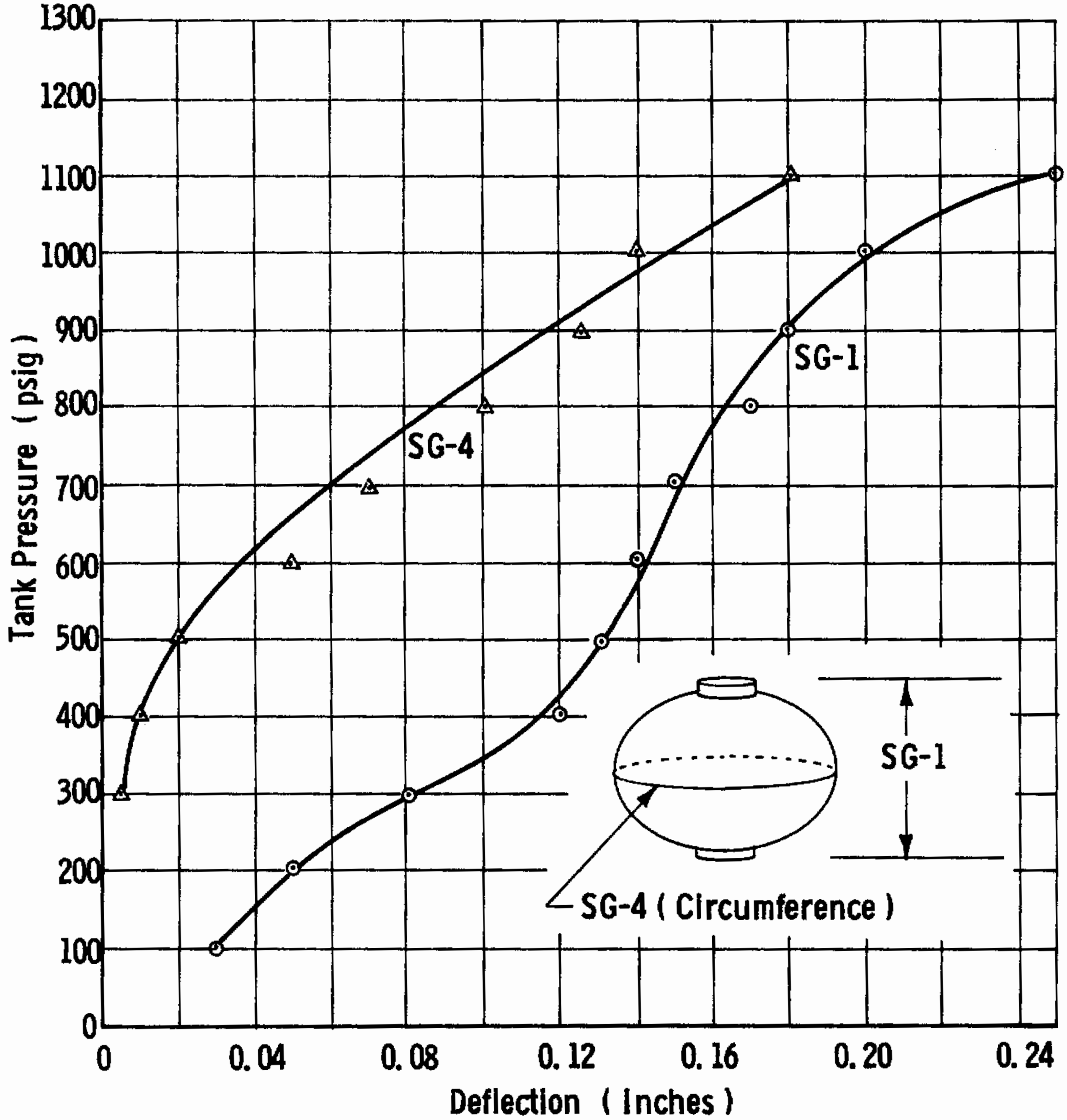


Figure 24. Deflection of Tank During Burst Test After 4-Month Exposure to ClF_3

(3) ClF_3 Tank

This tank was also in good condition. Examination of the weld areas disclosed no evidence of the corrosion that had produced leaks in two earlier tanks. The burst strength of 1100 psig, although slightly lower than that of the tanks exposed to AeroZINE 50 and N_2O_4, was still equal to that obtained on one of the control tanks.

c. Summary of Results

The results of the 1-month and 4-month environmental testing program demonstrate the favorable capabilities of metal-lined filament-wound tankage for liquid-propellant storage. Although a corrosion problem was initially encountered with two of the tanks containing ClF_3, it is believed that the improved welding design, used on the replacement tanks, satisfactorily solved this problem. The fact that one tank containing ClF_3 was thermally cycled for 4 months and showed no evidence of corrosion confirmed this belief. The results obtained with the AeroZINE 50, N_2O_4, and ClF_3 tanks indicate that long-term space storage of these propellants is feasible and practical. A third set of subscale tanks containing each of the propellants has been thermally cycled for more than 5 months, and there has been no visible evidence of deterioration.

B. PRESSURE-CYCLE TESTING

The Phase I, II, and III material studies established that a barrier-type liner was required for filament-wound tankage to contain corrosive liquid propellants. It was therefore decided to emphasize the study of metallic liners. A program was agreed upon and initiated that involved a transfer of effort from compatibility and subscale-chamber testing in pentaborane to a study of metallic-liner fabrication and testing.

The major problem expected in the use of a metallic liner in filament-wound tankage was structural compatibility with the resin-coated glass-filament case throughout the pressure and thermal cycling encountered in service. Difficulties of this type have been reported by other investigators (References 2 and 9). Patterned-type liners have been considered as a possible solution, but a less complicated technique involves the bonding of a smooth metallic liner inside the filament-wound case. Uniaxial and biaxial testing of adhesively bonded metallic foils indicated that this concept had merit. A decision was therefore made to evaluate the bonded-metal concept in filament-wound subscale tanks.

The contract provided that "at least eight 8-in. oblate spheroids incorporating the most promising patterned metal liner concepts or other promising configurations shall be fabricated using an extensible filament in order to permit simulation of the strain conditions in full-scale chambers. These tanks shall be fatigue-tested by pressure cycling with water under sufficient pressure to simulate the strain conditions in the full-scale tank. Each tank will be removed and boro-scope inspected and helium-leak tested at intervals to determine if the pressure cycling has caused breakdown of the metallic liner. Tanks shall be subjected to fatigue cycling with the objective of obtaining 50 fatigue cycle tests with no apparent liner damage." A linear-strain level of 2% for fatigue cycling was adopted as the optimum design level for full-scale tanks.

1. Fabrication of Test Tanks

The liners for the test tanks were fabricated in the same manner as those for the environmental-test chambers. The hemispherical segments were pressure-formed and welded to the bosses and to each other by resistance-roll-seam welding. Dye-penetrant and helium mass-spectrometer leak tests were used to check each liner for leaks.

The extensible filament selected to permit simulation of the strain conditions in full-scale chambers was Du Pont Dacron 1100-250-52. A semi-flexible resin system was used to provide for pressure cycling under higher-than-normal strain levels.

The metallic liners were acid-cleaned to provide a good surface for bonding. Primer (Minnesota Mining EC 2320) was then applied to the liners and 1/2 hour was allowed for drying. A layer of 3M AF-120 film was placed over the primed surface to bond the liner to the case. The following wrap pattern was used in the winding over the liner:

Number of strands in tape	3
Number of turns of tape per revolution	144
Number of revolutions	2

The calculated composite thickness (t_c) for this wrap pattern was 0.0326 in.

The three strands of Dacron 1100-250-52 were in-process impregnated with the following resin system: Dow DER 332 (100.00 parts by weight) and Jones-Dabney Epicure 855 (50.00 parts by weight). The filament-wound tanks were rotated continuously for approximately 12 hours at room temperature to allow the resin to gel. The first chamber was then cured for 1 hour at 150°F, plus 2 hours at 250°F.

Very low tension was used on the Dacron filaments during the winding of the first test tank (A-1), and no supporting mandrel was used inside the liner. Because slight buckling of the liner had occurred during winding, a soluble plaster mandrel (0.5 to 0.75 in. thick) was cast inside the liner of the second and all succeeding test chambers. A cure involving a minimum of 14 hours at 250°F was used for liners containing the mandrel, and the cure time for the filament-wound chambers was increased to 1-1/2 hours at 150°F plus 2-1/2 hours at 250°F. The plaster mandrel prevented further liner buckling when no tension was used on the filaments. For Tank A-3, approximately 10 lb of tension was used to apply more pressure on the adhesive; this unfortunately caused rather severe liner buckling, and a minimum tension was used in the winding of the remainder of the tanks.

2. <u>Fatigue Testing, First Series of Subscale Tanks</u>

Each subscale tank was tested in accordance with contract provisions that they should be fatigue-tested by pressure cycling with water under sufficient pressure to simulate the strain conditions in the full-scale tank. The volume of each chamber was measured and was multiplied by $(1.02)^3$ to obtain the volume required to produce a 2% linear-strain level. The original circumference at the girth of the oblate spheroid was measured by means of a pi tape.

The test tank was then expanded by forcing the required volume of water into the tank. A pump was used for the first tank. For succeeding tanks, the water was introduced into a pressure accumulator and was activated by pneumatic pressure. Each tank was pressure-cycled to the 2% linear-strain level at a rate of 2 min/cycle until failure. The time of failure was shown by water seepage through the filament-wound case.

The test results are summarized in Table 24, and are analyzed below.

TABLE 24
RESULTS OF PRESSURE-CYCLE TESTING OF SUBSCALE TANKS

Tank	Test Condition	Strain Level, %		Cycles per Min	Cycling Pressure, psig		Volume, cu cm			Circumference, in.			Cycles to Failure	Condition of Liner
		Linear	Volume		Initial	Final	Initial	Final	Increase	Initial	Final	Increase		
A-1	Fixed volume	2.0	6.1	0.5	460	400	3115	3218	103	25.21	25.35	0.14	27	Stretched and creased at edge of boss flange
A-2	Fixed volume	2.0	6.1	0.5	445	400	3145	3250	105	25.25	25.39	0.14	25	Stretched and creased at edge of boss flange
A-3	Fixed volume	2.0	6.1	0.5	450	400	3122	3189	67	25.27	25.30	0.03	20	Buckled and creased at edge of boss flange
A-4	Fixed volume	2.0	6.1	0.5	450	360	3080	3194	114	25.30	25.40	0.10	118	3-in. buckle 0.5 in. from edge of boss flange
A-5	Fixed volume	2.0	6.1	0.5	440	350	3059	3126	67	25.28	25.32	0.04	113	Very slightly creased at edge of boss flange
A-6	Fixed pressure	2.0	6.1	0.5	445	445	3075	3186	111	25.26	25.32	0.06	56	3-in. buckle at edge of boss
A-7a	Fixed volume	1.0	3.0	0.5	520	480	3065	3096	31	25.16	25.24	0.08	50	No failure
A-7b	Fixed volume	2.0	6.1	0.5	1080	1060	3096	3111	46	25.24	25.24	0.00	10	Numerous creases at edge of bosses

a. Tank A-1

This tank was emptied after 5 cycles and examined for liner damage. A small crease had developed in the liner near the edge of the flange on the boss, but no cracks were noted. Pressure cycling was then continued until failure. The pressure required to expand the tank was 460 psig at the start; it dropped to 400 psig at the end of the 10th cycle, after which it remained constant. Examination after failure showed that the liner had stretched extensively between the weld on the boss and the edge of the flange, where repeated creasing had caused a leak to develop (as shown by a dye-penetrant test). The test data are as follows:

Original tank volume	3115 cu cm
Additional volume of water required to produce 2% strain	190 cu cm
Tank volume at end of test	3218 cu cm
Permanent increase in volume	103 cu cm
Original girth	25.21 in.
Final girth	25.35 in.
Girth increase	0.14 in.
Initial pressure	460 psig
Final pressure	400 psig
Cycles to failure	27

b. Tank A-2

This tank was emptied after 25 cycles. Slight bulging of the liner was noted at several points around the edge of the flange on the bosses. The tank was again filled, and pressure cycling was begun. A leak developed when the pressure reached 300 psig. The tank was emptied, and the final volume and girth circumference were measured. This tank was sectioned at the girth, and the filament winding was removed by an acid solution. A dye-penetrant test showed that a crack had developed in a location similar to that found in Tank A-1.

As with Tank A-1, the liner had stretched between the weld on the boss and the edge of the flange. This area is not bonded to the filament-wound case because it is covered by the welding doubler. The stretching effect at the edge of the boss is shown in Figure 25. To reduce the area of

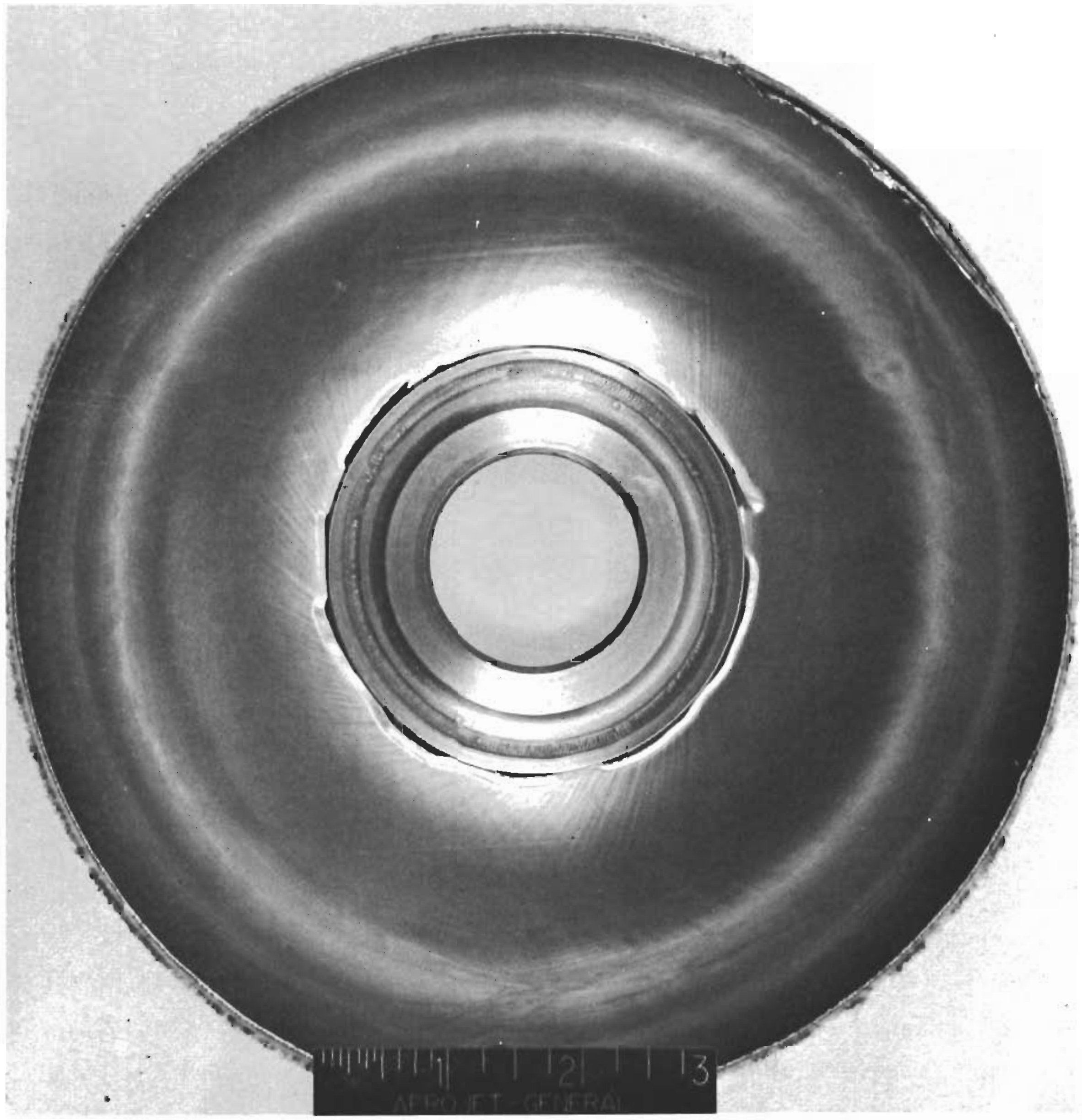


Figure 25. Head Section of Subscale Oblate-Spheroid Tank
After 25 Pressure Cycles to 2% Strain Level

unbonded metal liner, a row of spot welds was made as close to the edge of the doubler as possible on the remaining tank liners. The test data for Tank A-2 are as follows:

Original tank volume	3145 cu cm
Additional volume of water required to produce 2% strain	194 cu cm
Tank volume at end of test	3250 cu cm
Permanent increase in volume	105 cu cm
Original girth	25.25 in.
Final girth	25.39 in.
Girth increase	0.14 in.
Initial pressure	445 psig
Final pressure	400 psig
Cycles to failure	25

c. Tank A-3

In the winding of Tank A-3, the Dacron filament was tensioned to about 10 lb in the hope of increasing the pressure on the surface of the liner and improving adhesion. Although this tank contained 0.5 in. of soluble plaster mandrel as liner support, the liner was severely buckled during the cure. The tank was pressure-cycled until failure occurred without any removals. Examination of the liner revealed that severe creasing had occurred at the bulged areas. However, there appeared to be no major stretching of the metal liner at the bosses as had occurred with Tanks A-1 and A-2. The test data are as follows:

Original tank volume	3122 cu cm
Additional volume of water required to produce 2% strain	192 cu cm
Tank volume at end of test	3109 cu cm
Permanent increase in volume	67 cu cm
Original girth	25.27 in.
Final girth	25.30 in.
Girth increase	0.03 in.
Initial pressure	450 psig
Final pressure	400 psig
Cycles to failure	20

d. Tank A-4

To further reinforce the rewelded area at the edge of the doubler ring on the liner of Tank A-4, two flat rings of resin-coated glass fabric were placed over the AF-120 adhesive film. The edge of the top ring of glass fabric extended about 0.60 in. beyond the edge of the flange on the boss. Minimum tension was used on the Dacron filament during winding. The test data are as follows:

Original tank volume	3080 cu cm
Additional volume of water required to produce 2% strain	185 cu cm
Tank volume at end of test	3194 cu cm
Permanent increase in volume	114 cu cm
Original girth	25.30 in.
Final girth	25.40 in.
Girth increase	0.10 in.
Initial pressure	450 psig
Final pressure	360 psig
Cycles to failure	118

The pressure cycling was interrupted only for an overnight shutdown. After test termination, the liner was examined, and a 3-in.-long buckle about 0.5 in. from the edge of the boss was observed. X-rays did not show a crack in this area but revealed one at the line formed by the roll-seam welding on the boss. The metal had apparently become fatigued as a result of stretching in this area. Tank A-4 is shown in Figure 26.

e. Tank A-5

In addition to spot rewelding at the edge of the doubler on the liner of Tank A-5, reinforced glass-fabric rings were used on the tank. The cycling conditions duplicated those for Tank A-4 as closely as possible. The test data are as follows:

Original tank volume	3059 cu cm
Additional volume of water required to produce 2% strain	183 cu cm
Tank volume at end of test	3126 cu cm
Permanent increase in volume	67 cu cm
Original girth	25.28 in.

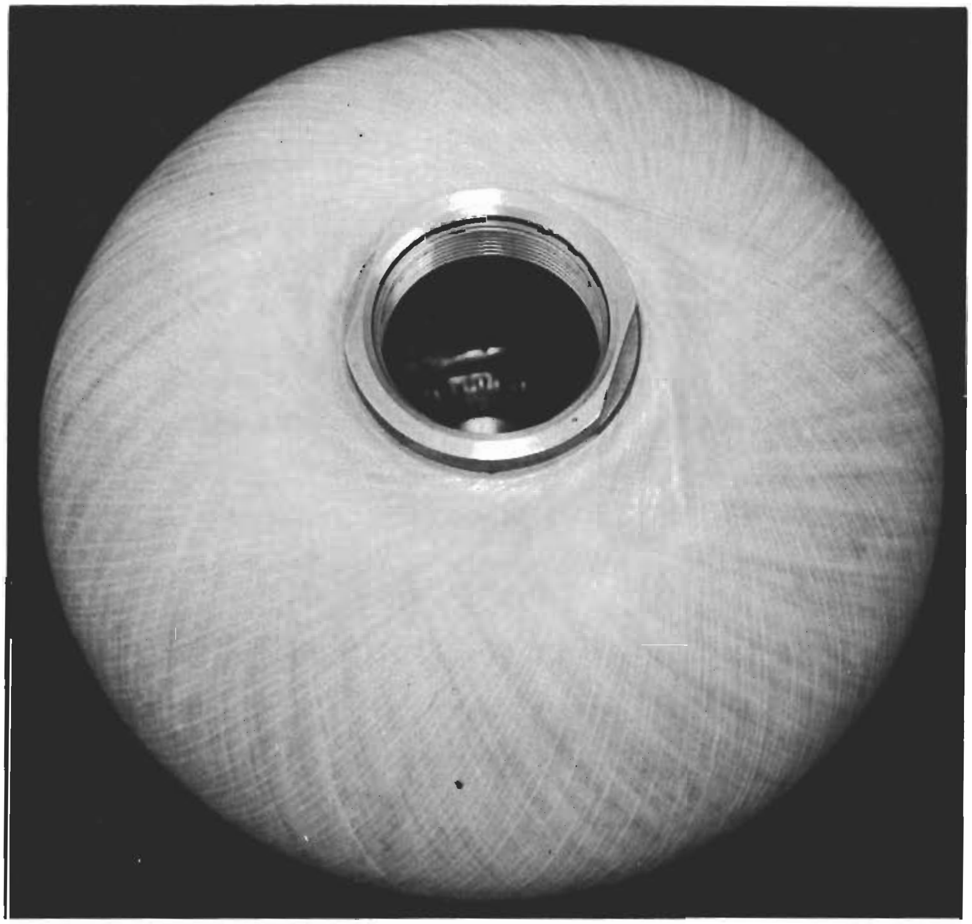


Figure 26. Subscale Oblate-Spheroid Tank After 118 Pressure Cycles to 2% Strain Level

Final girth	25.32 in.
Girth increase	0.04 in.
Initial pressure	440 psig
Final pressure	350 psig
Cycles to failure	113

A leak developed in the pressure line at the end of 3 cycles, and the system was disconnected; the liner was examined and showed no evidence of damage. After repair of the leak, cycling was continued to tank failure. Examination of the liner showed three slightly creased areas at the edge of the flange of the boss. Liner stretching at the edge of the boss that had been found in Tanks A-1 and A-2 was not found in Tanks A-4 and A-5; apparently, the spot welding and glass fabric had adequately reinforced this weak spot.

f. Tank A-6

In the fabrication of Tank A-6 the liner was rewelded and reinforced in the same manner as Tanks A-4 and A-5, and the winding procedure was the same as had been used for previous test tanks. The tank was cycled to a fixed pressure level in contrast to the fixed-volume conditions of the previous tanks. The volume of water required to produce an initial 2% linear-strain level was determined and was introduced into the tank. The pressure required to expand the tank to the 2% strain level was then measured. Following this initial pressurization, water was added to the accumulator and pressure cycling was conducted at the initial pressure level until failure occurred. The test data are as follows:

Original tank volume	3075 cu cm
Volume of water required for 2% strain	184 cu cm
Tank volume at end of test	3186 cu cm
Permanent increase in volume	111 cu cm
Pressure required to produce 2% strain	445 psig
Original girth	25.26 in.
Final girth	25.32 in.
Girth increase	0.06 in.
Original height at bosses	6.47 in.
Final height at bosses	6.66 in.

Permanent increase in height	0.19 in.
Cycles to failure	56

Examination of the liner after failure showed one 3-in.-long crease near the flange of the boss, several small creases at the edge of the flange, and no major areas of separation.

g. Tank A-7

The liner of this tank was rewelded in the same manner as Tanks A-4 and A-5, but was filament-wound with two 20-strand rovings of S-994 HTS glass filament preimpregnated with the Shell 58/68R resin system. Tension of 8 lb was applied to the roving during winding. The tank was cured 1-1/2 hours at 150°F, plus 2-1/2 hours at 250°F, plus 4 hours at 300°F.

The tank was pressure-cycled to a 1% linear-strain level at a fixed volume. Inasmuch as it was filament-wound with glass (a relatively high-modulus filament as compared with Dacron), the pressure requirements were much higher than those for the Dacron-reinforced tank. In fact, a higher pressure was required to produce 1% linear strain in the glass-reinforced tank than was required for 2% linear strain in the Dacron-reinforced tank. Tank A-7 was cycled without failure at 1% linear strain for 50 cycles, and was removed, examined, and reinstalled for pressure cycling at a 2% linear-strain level.

The test data at the 1% strain level are as follows:

Original tank volume	3065 cu cm
Volume of water required for 1% strain	94 cu cm
Tank volume at end of test	3096 cu cm
Increase in volume	31 cu cm
Initial pressure	520 psig
Final pressure	480 psig
Original height of tank	6.47 in.
Final height of tank	6.52 in.
Increase in height	0.05 in.
Original girth	25.26 in.
Final girth	25.24 in.
Girth increase	0.08 in.
Number of cycles at removal	50

The liner was slightly stretched at the edge of the bosses but had no creases. The test data at the 2% strain level are as follows:

Original tank volume	3096 cu cm
Volume of water required to produce a 2% strain	153 cu cm
Tank volume at end of test	3111 cu cm
Increase in volume	15 cu cm
Total increase in volume	46 cu cm
Initial pressure	1080 psig
Final pressure	1060 psig
Original height	6.52 in.
Final height	6.52 in.
Increase in height	None
Number of cycles to failure	10

At the end of the test the liner showed numerous creases at the edges of the bosses and considerable extrusion into the interstices of the filaments. The results of this test demonstrate the difficulty of producing a 2% strain level in a small glass-reinforced tank. The performance of this tank at the 1% strain level was very creditable and serves to support the bonded-metal-liner concept.

3. Fatigue Testing, Second Series of Subscale Tanks

The fact that the pressure-cycling results for two tanks in the first series of subscale test tanks were more than double the target requirements confirmed the value of the bonded-metal-liner concept. The results indicated, however, that almost complete bonding of the liner to the filament-wound case was necessary to achieve satisfactory performance. Theoretically, even longer cycle life should be possible if bonding over the entire outside area of the liner could be accomplished.

The bosses were redesigned to eliminate the doubler ring and thus permit complete bonding of the resin/glass structure to the metallic liner. This modification required thinning of the flanges on the bosses and welding at or near the boss edge. The welding of two stainless steel components necessitates almost complete matching of thicknesses.

The evolution of the welding procedure is shown in Figure 27. The weakness of Weld Design No. 1 was that the liner under the doubler stretched during pressure cycling and cracked after a few cycles. The re-welding of Design No. 1 reduced this stretching problem and resulted in pressure cycles totaling 113 and 118. Weld Design No. 2, in combination with a thinner flange on the boss, was expected to produce an additional improvement because complete bonding to the filament-wound case was accomplished. However, the pressure of the electrode roller reduced the liner thickness at the edge of the flange and resulted in leaks in five of the 15 liners fabricated for the environmental-exposure testing program.

Accordingly, Weld Design No. 3 was used on a replacement group of liners. It incorporated a 0.0065-in.-thick, 347 SS doubler over the liner. In it no liner thinning occurs, because the electrode roller is impressed into the doubler instead of the liner and the roll-seam weld is so near the edge of the doubler that very little unbonded metal liner is exposed. All these liners were successfully leak-tested.

In the fatigue testing of the first series of tanks it was demonstrated that a filament-wound tank with a properly bonded metal-foil liner will tolerate pressure cycling at a 2% linear-strain level for more than 100 cycles. It was desirable, however, to have performance data for the pressure cycling of filament-wound tankage under more severe conditions - e.g., a higher linear-strain level, with glass reinforcement, and for a longer period with Dacron reinforcement. Tanks in a second series incorporating the latest weld design were therefore subjected to additional pressure-cycling tests. The test setup for this series is shown in Figure 28.

a. Fabrication

In the first phase of the second series of pressure-cycling test tanks the liners were overwrapped with 20-end S-HTS glass roving <u>in situ</u>-impregnated with the following resin system: DER 332 (100.00 parts by weight) and Epicure 855 (80.00 parts by weight).

Two revolutions of 144 wraps each were used on the first group of tanks in this series. Earlier pressure-cycle tests were made on Tank A-7 with only one revolution of glass winding. Although this tank survived 50 cycles at a 1% strain level, it failed after 10 cycles when the pressure

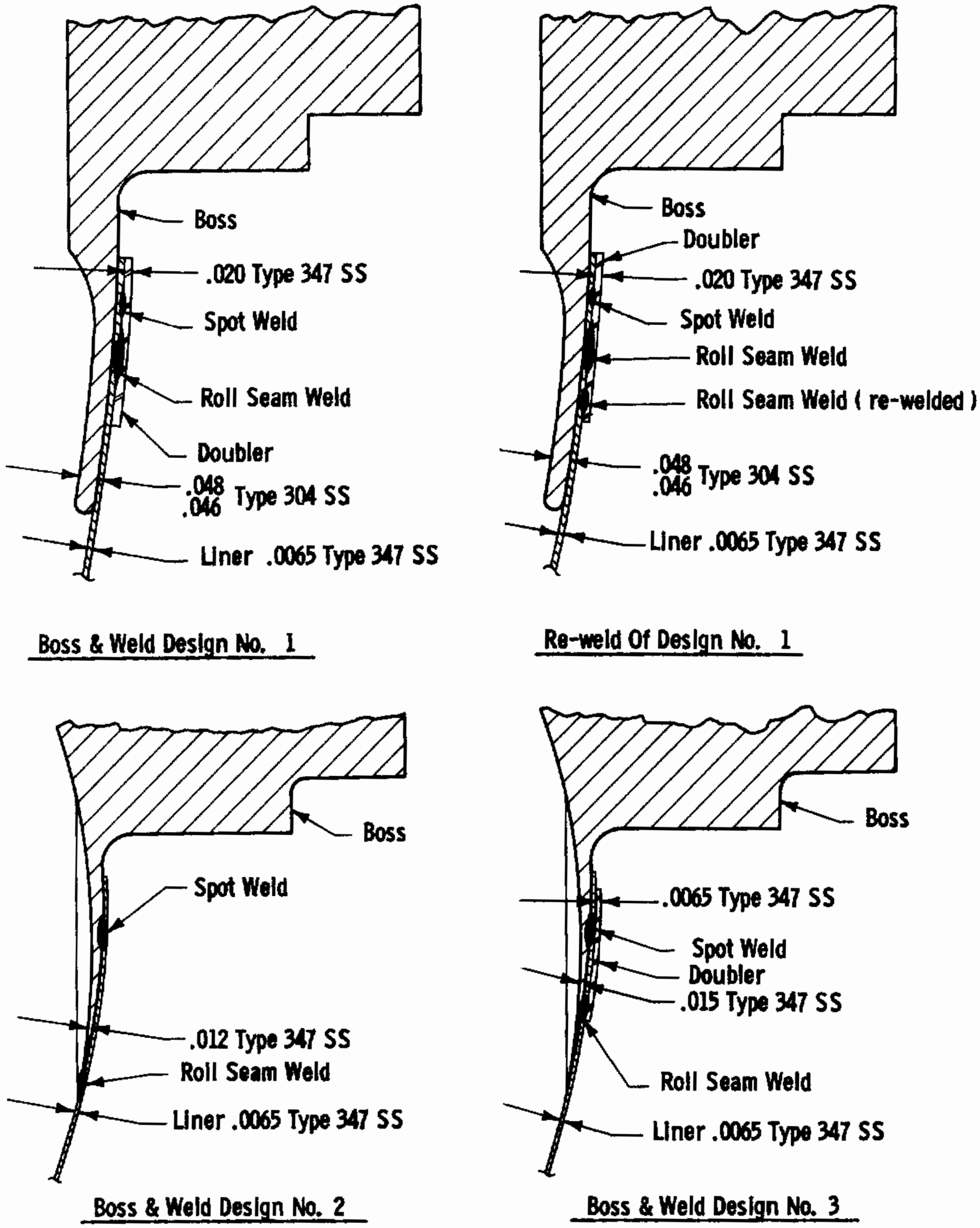


Figure 27. Evolution of Procedure for Welding of Liner to Boss Flange

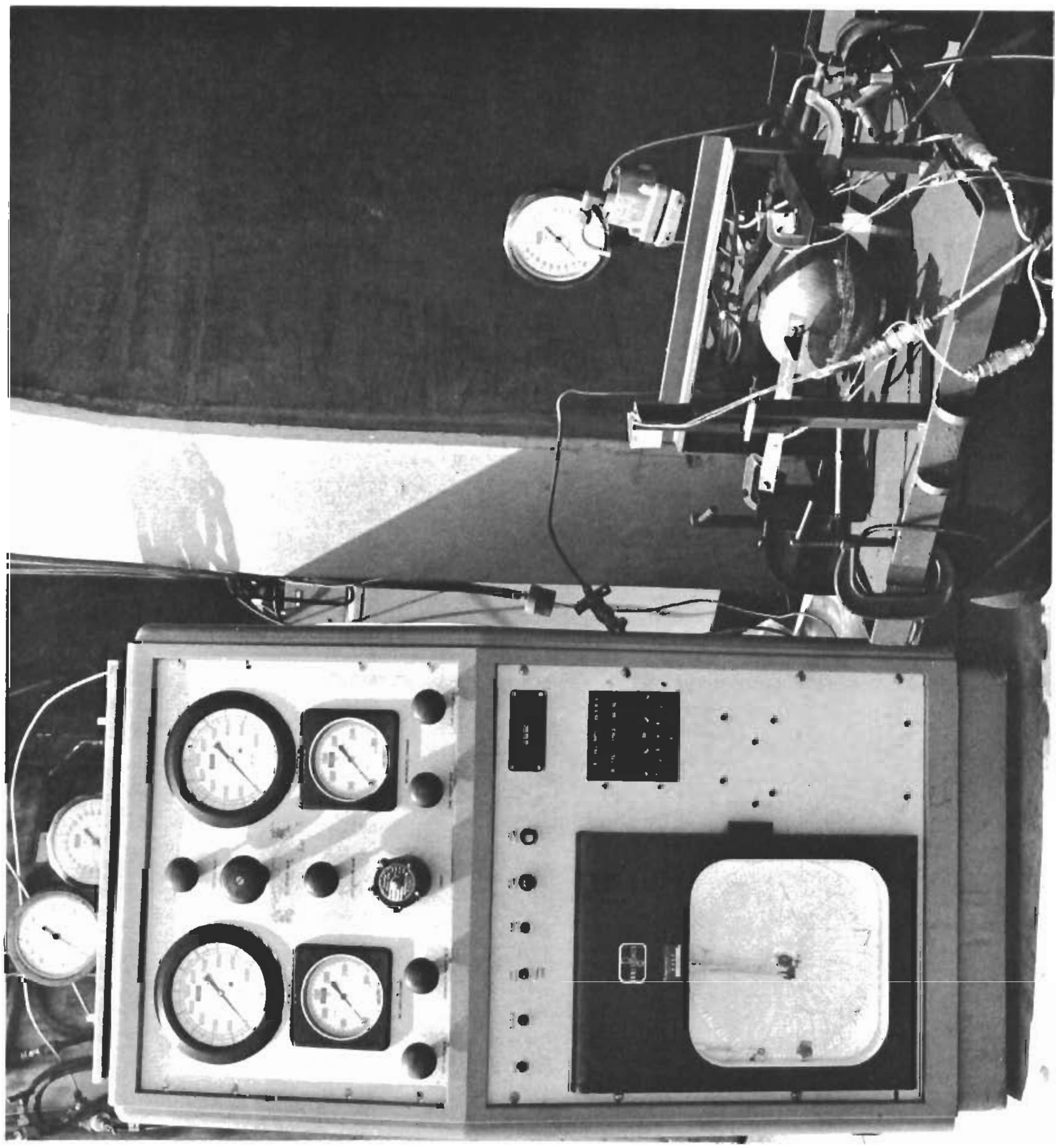


Figure 28. Pressure-Cycle Test Setup for Subscale Tanks

was increased to produce a 2% strain level. It was believed that a tighter wrap and more reinforcement would prevent extrusion of the metal liner through the interstices of the filament winding and improve the cycle life.

b. Testing

(1) Control Tanks

Two control tanks, C-1 and C-2, were instrumented to measure the pressure, height increase, volume increase, and girth increase. They were then pressurized to the burst point. The data for Tank C-1 are plotted in Figures 29, 30, and 31, respectively, as volume expansion, height increase, and girth increase vs pressure.

Figure 29 shows that expansion occurs as a linear function of the pressure. The burst pressure of 3200 psig is considered satisfactory for a glass-filament-reinforced tank of this wrap pattern. The linear-strain level reached by the tank during pressurization is noted on the volume-expansion curve of that figure. This subscale tank (C-1) exceeded 3% linear strain before failure.

The plot of height increase vs pressure in Figure 30 indicates a relationship similar to that of the volume-pressure plot. The pressure values at each linear-strain level were transposed from Figure 29 to the curve in Figure 30 and indicate the height increase at which each level of linear strain is reached.

The increase in girth with pressure (Figure 31) does not follow the same pattern as the other plots. There is very little girth increase during the early stages of pressurization. At approximately 2200 psig, however, the rate of growth of the girth increased sharply and from that point on became essentially a straight-line function.

(2) Tank P-1

A third glass-filament-wound tank (P-1) was fabricated in the same manner as the two control tanks. It was then instrumented with linear-motion gages and a transducer and was pressure-cycled at a 2% strain level. A leak developed at the top of the tank after 6 cycles, a much shorter cycle life than had been obtained with the tanks reinforced with Dacron filaments. Examination showed that the liner had stretched excessively at the edge

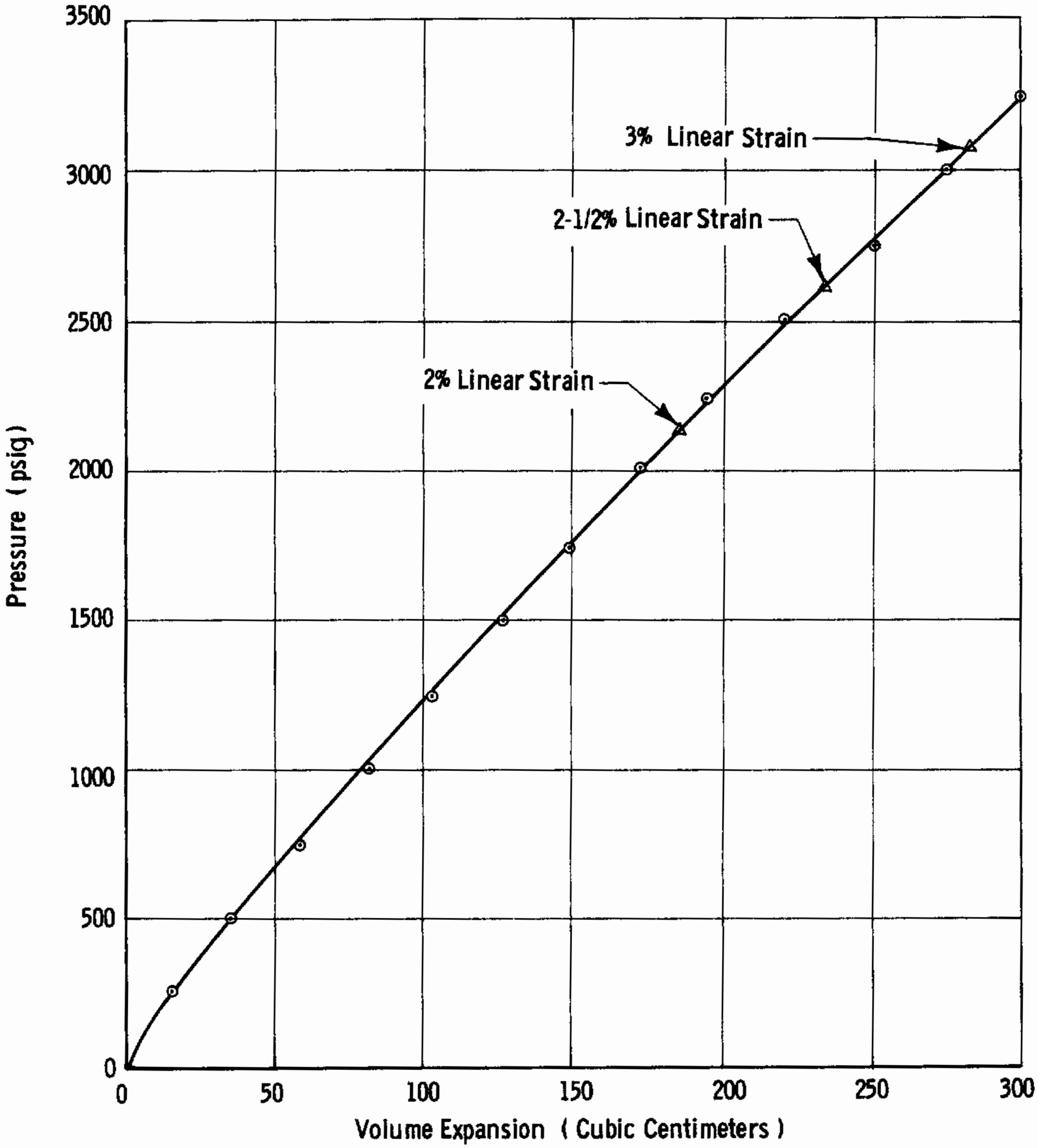


Figure 29. Expansion of Tank C-1 During Burst Test

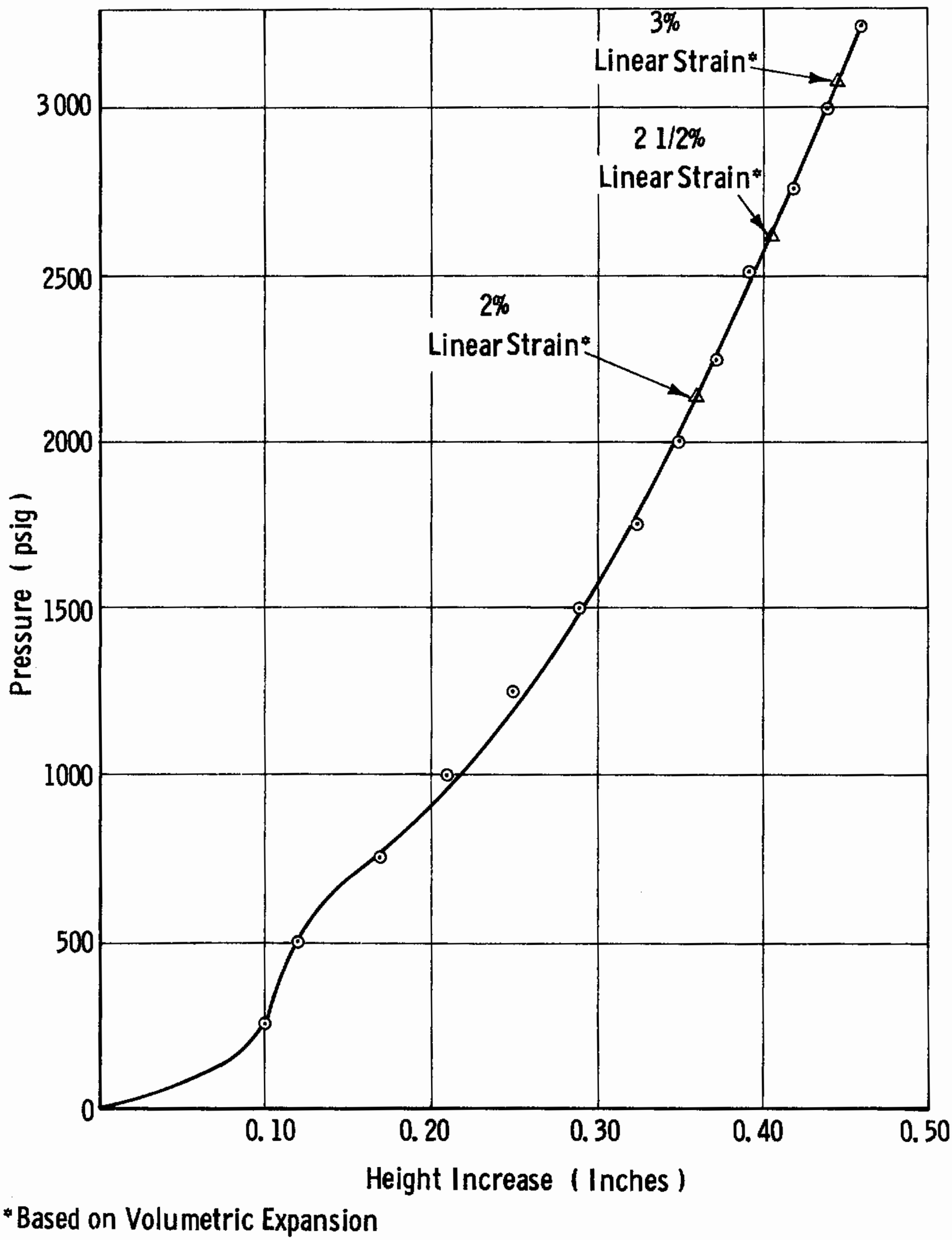


Figure 30. Increase in Height of Tank C-1 During Burst Test

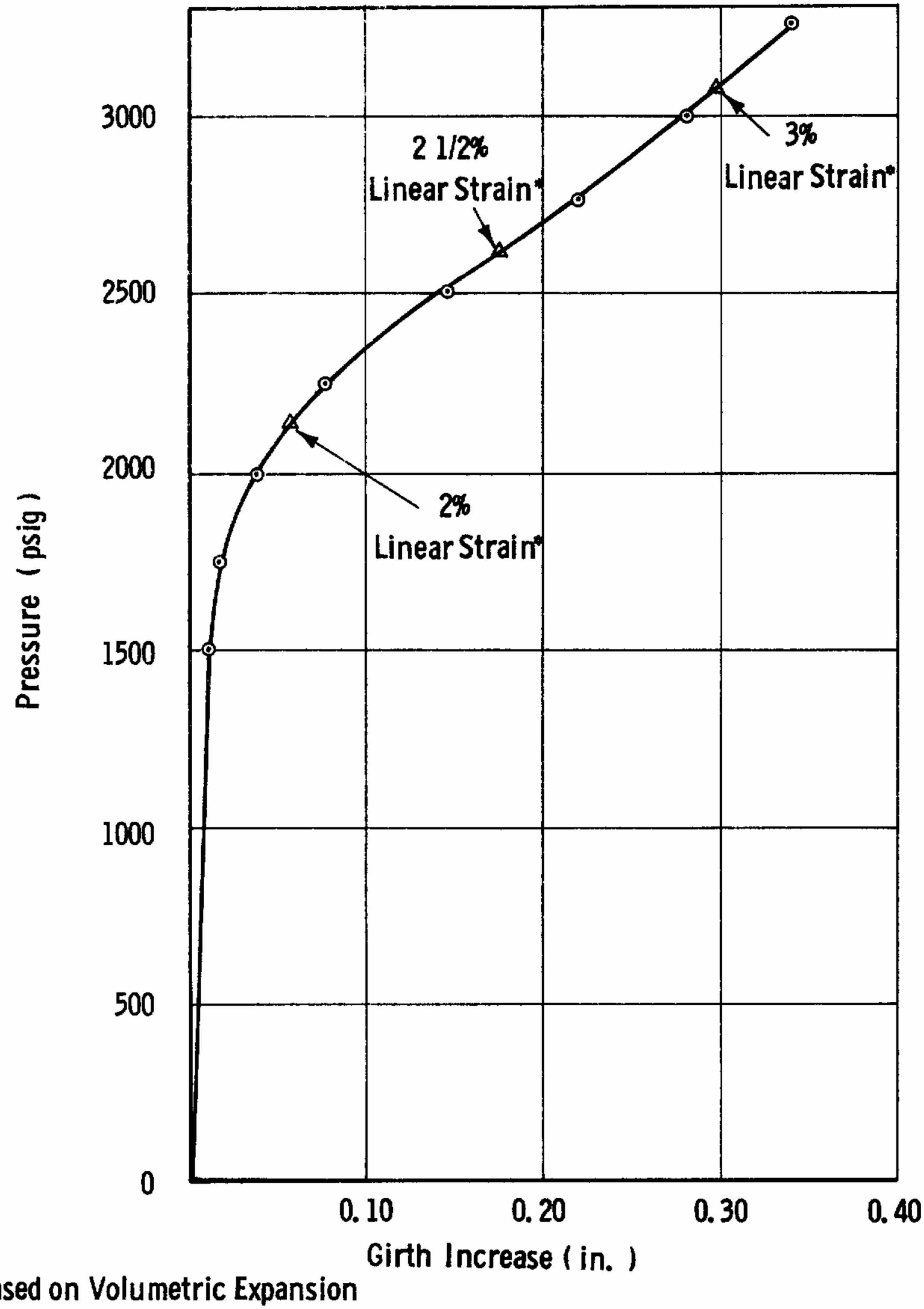


Figure 31. Increase in Girth of Tank C-1 During Burst Test

of the flange on the boss. The use of glass filaments for reinforcement had obviously caused stress concentration at the bosses. In addition, the reduction in thickness of the flange on the boss, which was required for welding improvement, permitted more vertical growth. The logical solution of this problem appeared to be reinforcement of the head area.

(3) Tank P-2

A fourth tank was fabricated in the same manner as the three previous tanks in this series, except that a glass-fabric reinforcing ring was placed over the liner at the boss and extended 0.6 in. beyond the edge of the boss. Although this design had been used in the previous successful pressure-cycling tests, the modification did not improve the pressure-cycle life of Tank P-2, which failed after 6 cycles. The failure occurred in approximately the same location as the P-1 failure.

It was decided to check the effect of the new welding design on the cycle life of the subscale tanks by fabricating a tank with Dacron filaments for reinforcement. The only change from the subscale tanks in the first series was the design of the flange on the boss and the welding; elimination of glass-filament winding as a variable was a logical step.

(4) Tank P-3

A subscale tank was fabricated in the same manner as Tanks A-4 and A-5, which had pressure-life cycles of 118 and 113 at a 2% strain level. Dacron 1100-250-52 filaments were in-process-impregnated with the following resin system: DER 332 (100.00 parts by weight) and Epicure 855 (50.00 parts by weight). The tank was rotated at room temperature for approximately 12 hours to allow the resin to gel. It was then cured for 1-1/2 hours at 150°F plus 2-1/2 hours at 250°F.

Tank P-3 was instrumented to show pressure, height increase, and girth increase. It was pressure-cycled at a 2% strain level until failure. The height increase for this tank was greater than for earlier test tanks and reached 0.350 in. on the 15th cycle. There was a minor increase in the girth, showing a maximum of 0.046 in. at the 15th cycle. The pressure was a maximum of 300 psig at the start of the test; it dropped to 250 psig at the 18th cycle, at which time a leak developed.

In view of the excessive growth in height, additional reinforcement of the head section appeared to be necessary. The thin rings of glass cloth at the boss area did not serve this purpose satisfactorily.

(5) Tank P-4

Another Dacron-filament-reinforced tank was fabricated in the same manner as Tank P-3 except that a two-layer doily, or reinforcing collar, was placed over each boss and extended approximately 1 in. beyond the edge of the flange on the boss. This doily was fabricated from resin-impregnated S-HTS 20-end glass roving. The Dacron filaments were then wound over the doily and the liner.

Tank P-4 was instrumented to show pressure, height increase, and girth increase. It was pressure-cycled at a 2% strain level. The vertical growth was much less than that of Tank P-3, amounting to only 0.209 in. at the 10th cycle as compared with 0.341 in. for Tank P-3. The girth increase of 0.161 in., however, was much greater than that for Tank P-3, which was 0.046 in. at the 10th cycle. These comparisons are shown graphically in Figures 32 and 33, respectively.

The reinforcement of the Tank P-4 head section proved very successful; this tank was pressure-cycled at a 2% strain level for 1304 cycles without failure. Examination of the liner indicated numerous small creases but no major buckling. The tank after testing is shown in Figure 34; the doily reinforcement is shown as a lighter-colored area extending down from the boss. The permanent increase in volume was 165 cu cm, indicating a high permanent set. This compares with 185 cu cm of water required to pressurize this tank to a 2% linear-strain level.

(6) Tank P-5

The technique of using glass-filament doilies for head-section reinforcement on pressure-test tanks was then applied to a glass-filament-wound tank. In the fabrication of Tank P-5, the prefabricated doilies were placed over the 3M AF-120 adhesive film at each boss area. The tank was then wound with one revolution of 20-end S-HTS glass-roving that was in-process-impregnated with the following resin system: DER 332 (100.00 parts by weight) and Epicure 855 (80.00 parts by weight). This resin system had a higher Epicure 855 content and was thus more flexible than the previous resin system

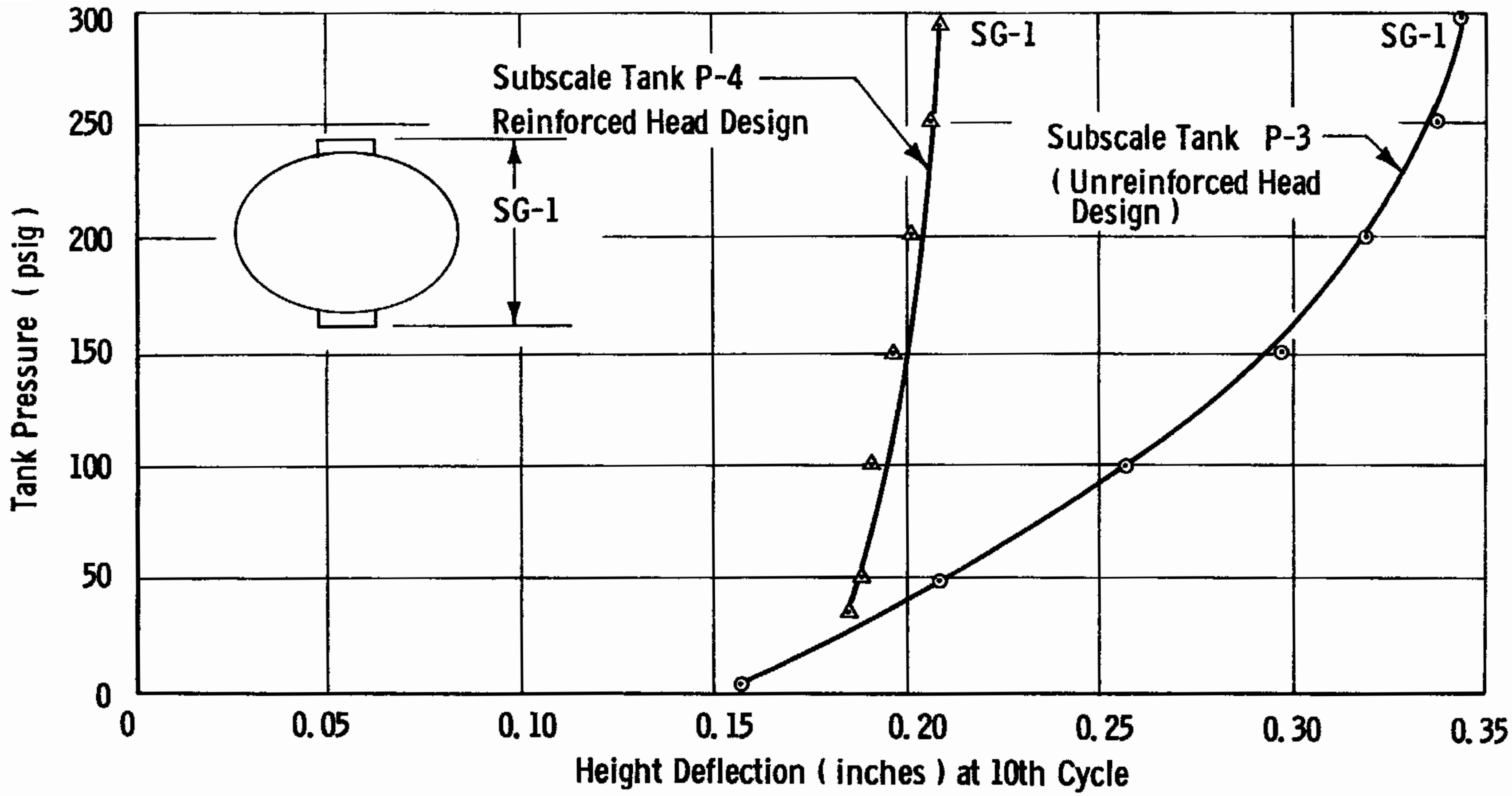


Figure 32. Effect of Head Reinforcement on Height Deflection of Tanks P-3 and P-4

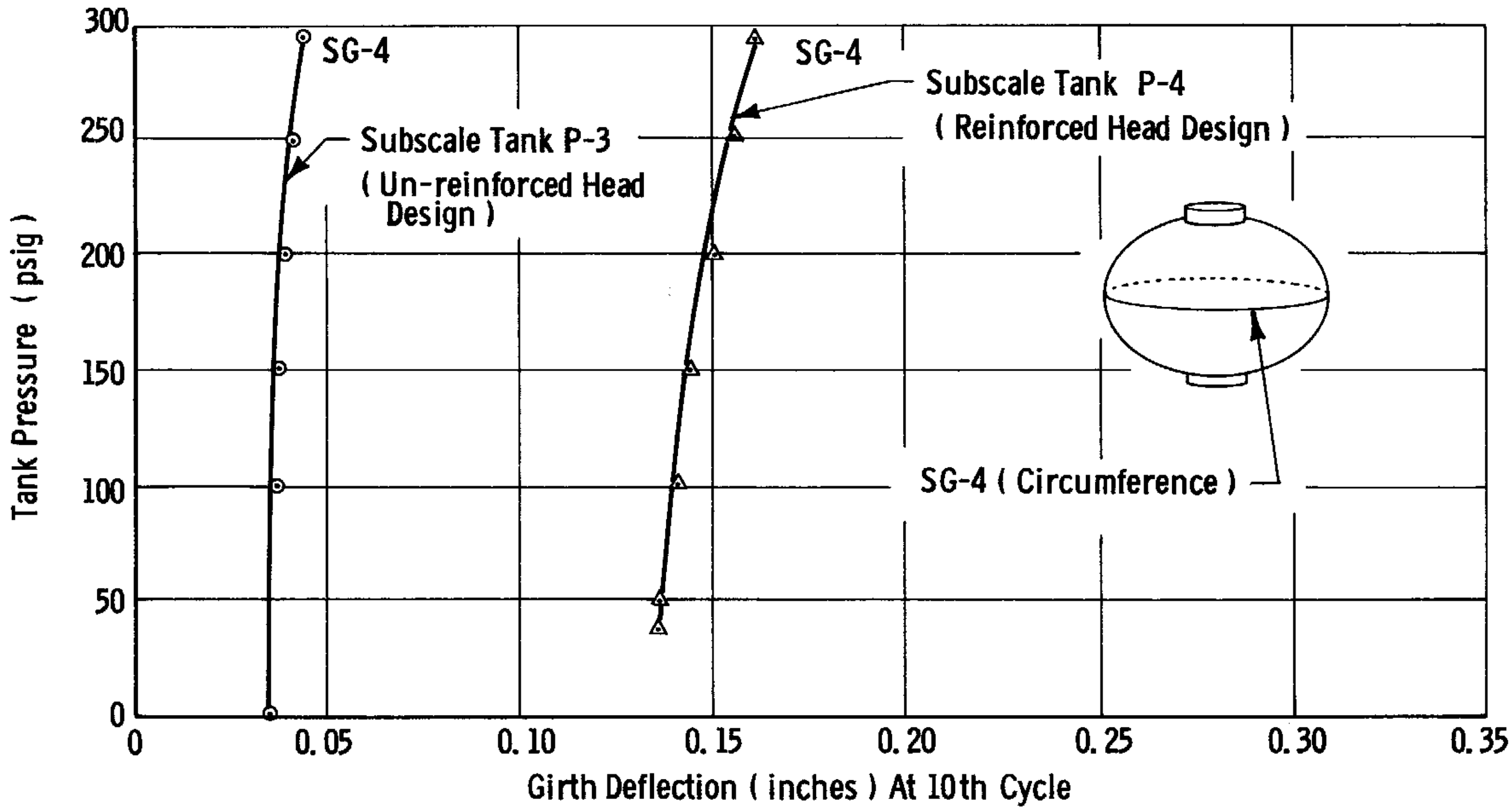


Figure 33. Effect of Head Reinforcement on Girth Deflection of Tanks P-3 and P-4

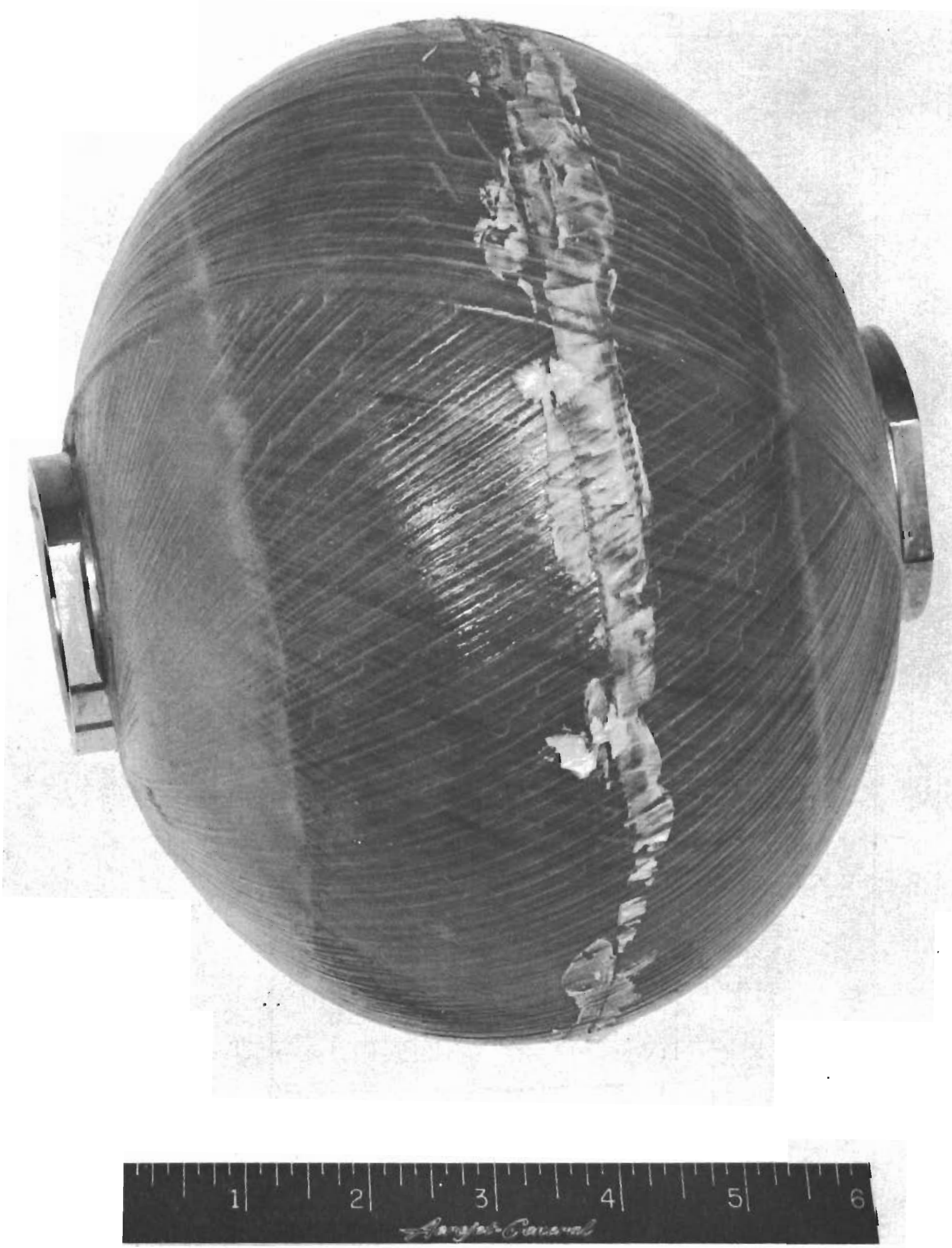


Figure 34. Subscale Tank P-4 After 1304 Pressure Cycles at 2% Strain Level

used. The change was made to avoid resin crazing, which appeared to be imminent on earlier test tanks. A single revolution of glass filament also reduced the pressure requirements and more closely simulated the conditions that would exist on full-size tankage.

Tank P-5 was instrumented to show pressure, height increase, and girth increase. It was set up to be pressure-cycled at a 2% strain level. However, the valve on the city water line that had been utilized to supply the 185 cu cm of water for use in expanding the tank to a 2% linear-strain level (6.1% by volume) was inadvertently left open during the first 2 cycles. The result was excessive pressure on the liner and tank failure on the 2nd cycle at 1750 psig. At this pressure the height increase was 0.375 in. and the girth increase 0.363 in., which indicated that the tank was expanded well beyond the 2% strain level.

(7) Tank P-6

Another glass-filament-wound tank was fabricated with glass-filament doilies for reinforcement of the head sections. The same materials and fabrication procedure as for Tank P-5 were used. Tank P-6 was instrumented to show pressure, height increase, and girth increase, and was pressure-cycled at a 2% linear strain. The linear strain was produced by the addition of 185 cu cm of water, and the tank survived 26 cycles before a leak developed. Pertinent data at various cycles are presented below.

Pressure Cycle	Pressure psig	Dimensional Increase, in.	
		Height	Girth
2	850	0.238	0.120
4	770	0.238	0.137
6	740	0.238	0.140
8	720	0.238	0.160
10	720	0.232	0.160
15	700	0.232	0.180
20	680	0.232	0.185
25	660	0.226	0.185
26	630	0.209	0.185
27	550	0.197	0.185

The permanent increase in volume was 37 cu cm.

Examination of the liner showed severe creases at the edge of the flange on the boss and under the doilies. There were minor corrugations in the liner beyond the doily area.

The doily reinforcement was effective in transferring some of the stress away from the boss area, as shown by the large growth in girth and relatively small growth in height. The fact that the adhesion of the liner to the overwrap did not appear to be very great in the doily area is understandable because of the rather coarse surface between the doily and the metal. In winding, it was difficult to prevent the filaments in the doily from bunching; this would tend to reduce the pressure in these areas as well as the area of contact with the adhesive film. A layer of cross-woven glass fabric under the doily would no doubt improve the adhesion.

The permanent increase in volume of Tank P-6 (37 cu cm) was substantially less than that of the Dacron-filament-reinforced tanks of the A-series and of Tank P-4. This indicates that the liner and case of a non-extensible filament-reinforced tank take much less permanent set than for a tank reinforced with an extensible filament.

c. Analysis of Results, Conclusions

Although the life of the glass-filament-reinforced tanks in the second series was not as long as desired, useful information was obtained and some progress was made. The high cycle life obtained from the Dacron-reinforced tank indicated the potential of a bonded metal liner combined with a properly reinforced head section. This test also indicated a possible need for a modified head contour for a metal-lined tank. Conclusions based on these pressure-cycle studies of bonded, metal-lined tanks can be summarized as follows:

(1) Dacron reinforcement of filament-wound tanks does not put the metal liner under as much compressive stress as glass-fiber reinforcement of the same thickness.

(2) As the compressive stress on the metal liner is reduced, the cycle life of the pressure vessel is increased.

(3) Reinforcement of the head section of the subscale tank by a prefabricated doily reduces the stress on the liner at the boss area and improves the cycle life of the tank.

VII. PHASE V - DESIGN OF PROTOTYPE CHAMBER

The objective of Phase V was to combine the information and data obtained from the Phase I, II, III, and IV studies in a design for a full-scale prototype chamber for the containment of storable propellants in a space environment. Inasmuch as this tankage might be required to transport any one of the propellants of interest, it had to be designed to tolerate the extreme conditions of all of them. An additional requirement was that the ultimate design should effectively utilize the most efficient aspects of filament-wound structures. The design also was required to meet practical fabrication procedures. This section covers the design considerations in detail.

A. DESIGN CRITERIA

The design criteria established for the prototype tank to be used for the containment of storable propellants in a space environment are outlined below.

Propellant	Transition Points, °F at 14.7 psia: Freezing	Boiling
AeroZINE 50	+18	158
N_2O_4	+12	70
ClF_3	-117	53

Tankage-Design Parameters

Shapes	Sphere, oblate spheriod, spheroid with L/D = 1.0
Diameter (inside liner)	
Sphere	56.0 in.
Oblate spheroid	63.5 in.
Spheroid (L/D = 1.0)	52.3 in.
Volume	
Sphere	92,000 cu in.
Oblate spheroid	92,000 cu in.
Spheroid (L/D = 1.0)	92,000 cu in.

Pressure Ratios	
Proof-to-working	1.10
Burst-to-working	1.38
Pressures	
Working	150 psia
Proof	165 psia
Burst	207 psia
Service life	1 year
Temperature extremes	-117 to +158°F
Cyclic loading	
Pressure	0 to 150 psia
Number of cycles	25
Sustained loading	
Pressure	150 psia
Duration	1 year
Materials of construction	
Liner	347 SS
Bosses	347 SS
Glass filaments	S-HTS
Resin matrix	RS-11 (DEN 438/BF_3-400)
Adhesive (liner to composite)	Narmco 7343/7139

B. CONFIGURATION CRITERIA

A spherical design with a diameter of 56 in. was specified in the contract. Such a filament-wound tank was designed and is discussed in Appendix I. Two alternative configurations were also designed and are presented there for consideration; it is believed that they will be found more efficient than the spherical design. The basis for this belief is analyzed in detail in Appendix I and is summarized briefly below.

The application of glass-filament-winding fabrication techniques to a pressurized spherical configuration does not produce an optimized design because of the unidirectional qualitites of the glass filaments and the isotropic force-field conditions in the pressurized sphere. Because the windings in a sphere must be randomized within the limitations of the design and equipment, the orientation of the load-carrying fibers becomes a critical and complex problem; there is also a substantial loss in efficiency due to the constant change required in the wrapping pattern to meet the resulting forces.

For these reasons, an oblate spheroid with balanced-head contours and a single in-plane winding pattern becomes an ideal configuration for use in conjunction with high-strength unidirectional glass filaments. The main objection of designers is the large-diameter major axis necessary to assure the required volume. Another configuration, although not as idealized as the oblate spheroid, is the spheroid with a chamber-length to chamber-diameter (L/D) ratio of 1.0. It approaches a spherical configuration, but utilizes a short cylindrical section in which the unidirectional filaments are oriented to meet the biaxial force field. This is accomplished by a combination of longitudinal and circumferential filaments.

C. WEIGHT COMPARISONS, FILAMENT-WOUND VS ALL-METAL TANKS

It is desirable to have comparative data on the weights of filament-wound tankage and all-metal tankage to facilitate determination of filament-winding qualifications in this area. Weights associated with the three alternative designs are presented below.

	Weight, lb		
Components	Sphere	Oblate Spheroid	Spheroid (L/D = 1.0)
Composite structure	36.29	21.88	24.98
Metal hardware			
Liner	18.12	18.43	18.28
Bosses (2)	18.70	18.70	18.70
Total	73.11	59.01	61.96

It is possible that a 0.003-in.-thick 347 SS liner could be used instead of the 0.006-in.-thick liner. This reduction would yield a weight saving of only 9 lb and is not considered of sufficient magnitude to justify the possibility of corrosion failure.

To permit comparison of filament-wound tanks with all-metal tanks, weights of tanks fabricated from several candidate metals were calculated on the basis of the following design criteria:

Internal volume	92,000 cu in.
Inside diameter	56.0 in.
Configuration	Sphere
Working pressure	150 psia
Proof pressure	165 psia

1. 6Al-4V Titanium Alloy

Ultimate tensile strength	160,000 psi
Tensile yield strength	145,000 psi
Tank thickness	0.016 in.
Tank weight	37.8 lb

Although this tank would weigh substantially less than any of the prototype filament-wound tanks, the alloy is known to be subject to serious stress-corrosion problems in the storage of N_2O_4. It therefore could not be considered satisfactory for universal application for all rocket oxidizers and fuels.

2. 6434 Steel

Tensile yield strength	200,000 psi
Tank thickness	0.0116 in.
Tank weight	53.6 lb

This tank would also have a favorable weight advantage over the filament-wound tanks. However, 6434 steel is not corrosion resistant to N_2O_4 and ClF_3. Vacuum metallizing of the inside of this tank with a corrosion-resistant metal could be considered, but would add more weight to the lining and the serviceability of this type of coating has not been established.

3. 6061-T6 Aluminum Alloy

Tensile yield strength	36,000 psi
Tank thickness	0.064 in.
Tank weight	75.5 lb

This tank would be satisfactory for use with the less-corrosive propellants but would not be recommended for ClF_3. In addition, it offers no weight saving as compared with filament-wound tankage, and welding porosity could be a problem in the thickness indicated.

4. AISI Type 347 Stainless Steel

Tensile yield strength	40,000 psi
Tank thickness	0.058 in.
Tank weight	193.7 lb

The weight of this tank, which would be considered acceptable for service with the propellants of interest, would be more than 3 times that of the metal-lined filament-wound tank of the recommended configuration.

These comparisons indicate that, generally speaking, all-metal tanks can be designed that have lower weight than metal-lined filament-wound tanks for the required operating pressures. When specific mission requirements (e.g., corrosion resistance to rocket oxidizers) are considered, however, the filament-wound tank with a corrosion-resistant liner has a definite advantage.

D. CONCLUSIONS

Analysis of three alternative designs for a prototype tank showed that a spheroid with L/D = 1.0 was the preferred design on the basis of structural efficiency, minimum diameter, and fabrication feasibility. The fabrication procedures outlined in Phase V satisfied the requirements of material compatibility and commercial practices. Weight comparisons with all-metal tanks indicated that the recommended design of the prototype filament-wound tank was competitive with a metal tank for propellant storage.

VIII. PHASE VI - DESIGN OF SPACE EXPERIMENT

The objective of Phase VI was to define an experiment in which a subscale tank incorporating the results of the studies in Phases I, II, III, and IV would be placed into orbit and evaluated for performance by means of telemetry measurements. This required determination of the proper instrumentation, auxiliary equipment, and layout to provide information to ground stations on the serviceability of filament-wound tankage in space. The planned space experiment is covered in detail in Appendix II and is summarized below.

A. DESIGN CRITERIA

Criteria for the design of the experiment, established in collaboration with the Research and Technology Division Project Engineer, were as follows:

Weight	10.5 lb (approx)
Size of tank	8 in. in diameter by 5.618 in. thick
Power available	Provided by vehicle
Number of channels available	5
Duty cycle of propulsion system	Unknown
Data requirements	Temperature, pressure, tank-wall stress
Orbit and environment	
Distance	600 miles from earth
Inclination	Polar
Type of orbit	Circular

B. ASSUMED OPERATING CONDITIONS

Inasmuch as the propellant selected for filling the subscale tank may be either AeroZINE 50, N_2O_4, pentaborane, or ClF_3, it was necessary to use materials that would tolerate the most corrosive of these propellants. It was also assumed that the system would operate on command for a 1-year storage period in space with the tank pressurized to 150 psia. Some of the component designs were not finalized, however, because the launch vehicle was not specified. It was assumed that in a practical application the tank would be full at orbital injection and the propellant would be used throughout the mission; this would require an expulsion system and a pressurization system.

C. DESIGN AND FABRICATION OF EQUIPMENT

All factors that might affect the instrumentation and equipment were considered in designing the experiment. These included such environmental factors as vacuum, micrometeoroids, and radiation.

Equipment was selected that would be expected to operate under the conditions known to exist in space, and fabrication procedures were developed for the assembly of the pressurization, venting, and telemetry equipment. These are covered in detail in Appendix II.

It is recommended that the performance of the experiment be evaluated prior to the launch. This is considered necessary to ensure complete reliability when the experiment is in orbit. Specifications are supplied in Appendix II to permit the procurement of all necessary equipment.

D. SUMMARY

An experimental satellite system was designed that will provide for a space-environment evaluation of the materials and fabrication techniques for subscale tankage developed in these studies. Recommendations were made for the pre-launch testing of a complete assembly to ensure proper functioning of all components in flight and the transmission of meaningful data to the ground station.

IX. MATERIAL AND DESIGN STUDY CONCLUSIONS

The material studies demonstrated that long-term exposure of resins and liner materials to the propellants of interest required a high degree of chemical resistance to these corrosive fuels and oxidizers. Although resin systems having a moderate degree of compatibility with the less corrosive propellants were found, they cannot be expected to survive long contact with such oxidizers as N_2O_4 and ClF_3. All polymeric liner materials were found to be permeable to rocket fuels and oxidizers and thus would not provide the barrier required for propellant tankage.

In composite form with glass filaments, the resins were very resistant to vacuum within a range from 75 to 200°F. Within this range, the effects of vacuum in space on epoxy/glass-filament composites are expected to create no problems. However, the composites underwent severe degradation by the propellants at 60 and 100°F. The results of the propellant-exposure tests confirmed the necessity for a barrier between the propellant and the filament-wound overwrap.

It was concluded in the early stages of the program that a metallic type of liner was required for filament-wound tankage to provide the level of reliability needed for a 1-year mission in space. Annealed AISI Type 347 stainless steel satisfied the combined requirements - for a liner for such service - of corrosion resistance, extensibility, and fabrication feasibility.

Adhesion and uniaxial-strain-cycling tests of aluminum and 347 SS foil/fabric laminates demonstrated the effectiveness of a smooth bonded-metal-liner concept for filament-wound tankage. The results indicated that a properly bonded metal liner can be expanded into the plastic range and then be compressed back to almost the original dimensions without separation from the supporting structure.

Pressure-cycling tests of 347 SS, metal-lined, 8-in., oblate spheroids at 1 and 2% linear-strain levels exceeded the target requirement of 50 cycles. The results confirmed the validity of the bonded-metal-liner concept in producing structural compatibility between the liner and the filament-wound overwrap. An extensible fiber reinforcement produced less compressive stress on the liner than a nonextensible fiber and thus promoted longer life under cycling.

Environmental-exposure tests of subscale tankage proved the dependability of bonded 347 SS foil-lined filament-wound tanks for long-term storage of rocket fuels and oxidizers. Fabrication techniques, particularly the location of the roll-seam weld, were found to be critical factors in avoiding corrosion of the metal liner by ClF_3.

Analysis of three possible configurations for prototype tankage resulted in the recommendation of a spheroidal tank with a length-to-diameter ratio of 1.0 as the most efficient design.

An experiment was planned that will permit the evaluation of materials and fabrication techniques in an orbiting satellite.

X. RECOMMENDATIONS FOR FUTURE WORK

Further investigation is justified in several areas to augment and extend the essential information and data on materials, testing, and fabrication procedures developed in this work.

An investigation of adhesive bonding as an alternative to welding for the fabrication of liners is recommended that would encompass the evaluation of new adhesive systems in panel tests and in subscale-tankage exposure tests.

A study of head contours can be of value in expanding the application of filament winding to pressurized tankage. As envisioned, it would include computer investigations, the fabrication of subscale tankage, and pressure-cycling tests.

A prototype tank with L/D = 1.0 is considered a promising design, and it is recommended that subscale tanks of this configuration be fabricated and tested. The effect of a hoop section on the structural compatibility of a metal-lined tank can be determined by pressure-cycling tests at various strain levels.

The achievement of maximum cycle life in metal-lined tankage depends on the equal distribution of strain in the liner. Investigation of increased rigidity between the liner and the case at the critical areas would aid in determining the importance of this factor. Studies of liner bonding to the filament-wound case, including evaluation of adhesives, cleaning techniques, and adhesive application methods to improve the integrity of the bond, are recommended.

Subscale-tankage scaleup to larger diameters should also be considered in evaluation of the smooth bonded-metal-liner concept. Pressure-cycle tests of these larger tanks at various strain levels would be of value in confirming the test results for subscale tanks obtained in the current program.

The effect of meteoroid penetration on filament-wound propellant tankage can also provide important data in the area of performance testing. Sufficient information is now available on the concentrations and velocities of meteoroids to permit the design of a realistic experiment.

REFERENCES

1. R. M. Lydon, The Effects of Nitrogen Tetroxide and AeroZINE 50 on Non-metallic Materials. Aerojet-General TM 151 LRP. 28 December 1962.

2. D. Pollman and R. E. Jacobsen, Feasibility Demonstration of the Design, Fabrication, and Testing of Filament-Wound Fiberglass Liquid Propellant Tanks. SSD-TR-61-45. (Prepared by The Boeing Company, Aero-Space Division.)

3. Mechanical System Design Criteria Manual for Pentaborane. SSD-TR-61-3. (Rocketdyne report under Contract AF 33(616)-6939.) September 1961.

4. Pentaborane Handling Manual. SSD-TR-61-10. (Rocketdyne report under Contract AF 33(616)-6939.) September 1961.

5. Joseph Green, N. B. Levine, and R. C. Keller, "Elastomers for Liquid Rocket Fuel and Oxidizer Application." Industrial and Engineering Chemistry, Vol. II, No. 2. June 1963. (Based on work under Contract AF 33(616)-7227.)

6. The Compatibility of Materials with Chlorine Trifluoride, Perchloryl Fluoride and Mixtures of These. WADD Technical Report 61-54. (Prepared by Pennsalt Chemicals Corporation.) February 1961.

7. J. J. Shore, Non-metallic Materials for Nitrogen Tetroxide and Aero-ZINE 50 Exposure. Aerojet-General Report MN-256. August 1960.

8. Compatibility of Rocket Propellants with Materials of Construction. Battelle Memorial Institute OTS PB 161215. 15 September 1960.

9. Hardesty, Minutes of Technical Forum on Expulsion Bladders and Tank Liners for the Containment of Corrosive and Cryogenic Fuels. Swedlow, Inc. 21 May 1963.

10. Mechanical System Design Criteria Manual for N_2O_4. SSD-TR-61-5. (Prepared by Rocketdyne.) September 1961.

11. M. L. Murchison and T. F. Barton, Compatibility Studies of Metals with Nitrogen Tetroxide and AeroZINE 50. Aerojet-General Report MN-155-2.

12. Compatibility Studies of Metals for Titan II Engines. Aerojet-General Report RM-7693 MM-155-5. (Prepared under Contract AF 04(647)-521.)

13. Chlorine Trifluoride Handling Manual. SSD-TR-61-9. (Prepared by Rocketdyne.) September 1961.

14. Prototype Propellant Testing System. RTD-TDR-63-3. (Prepared by Aerojet-General Corporation.) February 1963.

REFERENCES (cont.)

15. The Compatibility of Welded Structural Materials with Chlorine Trifluoride, Perchloryl Fluoride and a Mixture of These. ML-TDR-64-22. February 1964.

16. Space Environmental Effects on Materials and Components - Vol. I, Elastomeric and Plastic Materials. RSIC-150. U.S. Army Missile Command, Redstone Arsenal, Alabama. 1 April 1964. Appendix F - Laminates.

17. A. F. Ringwood, The Behavior of Plastic Materials in the Space Environments. Missile and Space Division, General Electric Company PN 613.

18. H. R. Gloria, W. J. Stewart, and R. C. Sarin, Initial Weight Loss of Plastics in a Vacuum at Temperatures from 80°F to 500°F. Technical Note D-1329. Ames Research Center, Moffett Field, California. December 1962.

19. J. Brownlee and J. P. McNally, The Effects of Simulated Space Environments on Structural Plastic Materials. Aerojet-General Report SP-TR-24-RD-R60-80. August 1960.

20. Radiation Effects - State of the Art - 1962-1963. Battelle Memorial Institute Technical Data Report REIC R28. 30 June 1963.

21. Alloy Digest - AISI Type 302 Stainless Steel. Engineering Alloys Digest, Inc. Upper Montclair, New Jersey. January 1960.

GLOSSARY OF MATERIALS

	Composition	Supplier
AeroZINE 50	50/50-wt% mixture of UDMH and hydrazine	Aerojet-General Corporation
AF-111	Film adhesive	Minnesota Mining & Manufacturing (3M) Company
AF-120	Film adhesive	Minnesota Mining & Manufacturing (3M) Company
Alcoa 101	Aluminum powder	Aluminum Corporation of America
Amoco AI(10)	Polyimide resin	Amoco Chemicals Corporation
BDMA	Benzyl dimethylamine	Maumee Chemical Company, Inc.
BF_3-400	Boron trifluoride monoethylamine	Harshaw Chemical Company
Buna N	Butadiene-acrylonitrile polymer	-
Cab-O-Sil	Silica powder	Cabot Corporation
Cymel 430	Melamine formaldehyde resin	American Cyanamid Company
Cymel 431	Melamine formaldehyde resin	American Cyanamid Company
Dacron 1100-250-52	Polyester filament	E. I. du Pont de Nemours & Company
Dacron 2353	Polyester fabric	E. I. du Pont de Nemours & Company
DEN 438	Novolac epoxy resin	Dow Chemical Company
DER 332	Epoxy resin	Dow Chemical Company
DMA	Dimethyl aniline	Dow Chemical Company
DMF	Dimethyl formamide	E. I. du Pont de Nemours & Company
DMM plaster	Soluble plaster	Kerr Chemicals, Inc.
Epicure 855	Epoxy curing agent	Jones Dabney Company
Epon 828	Epoxy resin	Shell Chemical Company
Epon 871	Epoxy resin	Shell Chemical Company
Epon 1031	Epoxy resin	Shell Chemical Company
Hastelloy	Nickel-base alloy	Union Carbide Corporation
Kel-F	Fluorocarbon film	Minnesota Mining & Manufacturing (3M) Company

GLOSSARY OF MATERIALS (cont.)

	Composition	Supplier
Kel-F-5500	Fluorocarbon resin	Minnesota Mining & Manufacturing (3M) Company
Kel-F-KX633	Kel-F dispersion	Minnesota Mining & Manufacturing (3M) Company
Kel-F-KX634	Kel-F primer	Minnesota Mining & Manufacturing (3M) Company
K-Monel	Nickel-copper alloy	International Nickel Company, Inc.
Kopox 170	Polyepoxy resin	Koppers Company, Inc.
Kopox 171	Polyepoxy resin	Koppers Company, Inc.
Kynar	Fluorocarbon film	Pennsalt Chemicals Corporation
Laminae 4173	Polyester resin	American Cyanamid Company
MDA	Methylene dianiline	Dow Chemical Company
MEK peroxide	Methyl ethyl ketone peroxide	Lucidol Division, Wallace & Tiernan, Inc.
MNA	Methyl nadic anhydride	National Aniline Division, Allied Chemical Corporation
Monel	Nickel-copper alloy	International Nickel Company, Inc.
MPDA	Metaphenylenediamine	E. I. du Pont de Nemours & Company
Narmco 7343/7139	Urethane adhesive	Narmco Division, Whitaker Corporation
Rene 41	Vacuum-melted alloy	General Electric Company
Resin No. 46	Phenol formaldehyde resin	U.S. Polymeric Chemicals, Inc.
Resin No. 91LD	Phenol formaldehyde resin	Cincinnati Testing Laboratories
Resin No. L-70	Styrene-butene-vinyl toluene resin	Emerson & Cummings
Shell 58/68R	Epoxy resin system	Shell Chemical Company
Superalloy	Nickel-chrome steel alloy	Borg-Warner Corporation
Teflon	Fluorocarbon film	E. I. du Pont de Nemours & Company

GLOSSARY OF MATERIALS (cont.)

	Composition	Supplier
Teflon FEP	Fluorinated ethylene propylene film	E. I. du Pont de Nemours & Company
Teflon FEP 100-A	Fluorocarbon film	E. I. du Pont de Nemours & Company
Teflon TFE	Tetrafluoroethylene film	E. I. du Pont de Nemours & Company
UDMH	Unsymmetrical dimethyl-hydrazine	-
Viton A	Fluorocarbon Polymer	E. I. du Pont de Nemours & Company
Viton B	Fluorocarbon polymer	E. I. du Pont de Nemours & Company
Y-9141	Film adhesive	Minnesota Mining & Manufacturing (3M) Company
Z catalyst	Methylene dianiline - meta-phenylenediamine	Shell Chemical Company
Zytel 61	Soluble nylon resin	E. I. du Pont de Nemours & Company
181 glass fabric	S-HTS glass fabric	Owens-Corning Fiberglas Corporation
3M-3512 A, B, and C	Urethane adhesive	Minnesota Mining & Manufacturing (3M) Company

APPENDIX I

DESIGN ANALYSIS
FILAMENT-WOUND PROTOTYPE TANKAGE FOR STORABLE PROPELLANTS

This appendix outlines a systematic approach for the design and fabrication of a filament-wound, metal-foil-lined, storable-propellant tank in the following forms: (1) spheroid, (2) oblate spheroid, and (3) sphere. The contribution of the metal-foil liner to the strength of the chamber is neglected. The design analysis is therefore primarily concerned with obtaining the thicknesses and weights of tanks that have equivalent capacities in these three configurations. The methods and calculations used to determine the design-allowable strengths and thus the container thicknesses and weights are considered first. Calculations presenting positive margins of safety for the structural components are developed for each configuration. In addition, the design criteria, dimensional and material parameters, and estimated vessel weights are tabulated to facilitate comparisons.

I. DESIGN-ALLOWABLE STRENGTHS

The design-allowable strengths for filament-wound composite structures presented here are based on a series of dimensional and conditional design factors that take into account the effects of geometry, thickness, chamber diameter, port size, wrapping angle, temperature, etc. These factors have been empirically derived from the results of many tests on various glass-filament-wound chambers; as more-current data become available, the factors are revised accordingly. This systematic approach is used by Aerojet in calculating realistic values for allowable tensile strengths of the filaments in a composite structure; it is discussed in detail in Reference I-1.

More recent subscale tests, particularly of 8-in.-dia oblate spheroids, have demonstrated the necessity of taking the winding angle, α, into account in calculations. The values $\sec^2\alpha$ in the longitudinal direction and $1 - (\tan^2\alpha/2)$ in the hoop direction are therefore used to more accurately define and equate the empirical and theoretical design-allowable strengths. This allowable filament strength in either the longitudinal or the helical wraps is given by

$$F_{f,l} = 0.89\ K_1K_2K_3K_4K_p(\sec^2\alpha)F_{tu,s}$$

For the hoop direction, this allowable filament stress is given by

$$F_{f,h} = 0.89\ K_3 K_4 \left[1-(\tan^2\alpha/2)\right] F_{tu,s}$$

The symbols used in this determination of design-allowable strengths are defined below.

Symbol	Definition, Units
D_b	Boss diameter, in.
D_c	Chamber diameter, in.
$F_{f,h}$	Allowable ultimate strength of hoop filaments, psi
$F_{f,l}$	Allowable ultimate strength of longitudinal filaments, psi
$F_{tu,s}$	Average ultimate tensile strength for glass roving, psi
K_1	Design factor based on boss-diameter to chamber-diameter ratio
K_2	Design factor based on chamber-length to chamber-diameter ratio
K_3	Design factor based on approximation of thickness-to-diameter ratio
K_4	Design factor based on chamber diameter
K_p	Design factor based on off-center ports (K_p = 1.0 when no off-center ports are included)
L	Chamber length, in.
T	Temperature, ^oF
$t_{f,h}$	Hoop-filament thickness, in.
$t_{f,l}$	Longitudinal-filament thickness, in.
α	Angle between line in axial direction and filament path, degrees
η	Time under sustained load

In addition, two other factors not shown in Reference I-1 (K_5 and K_6) are included. They take into account effects caused by increased temperature and duration at load, and have been found to have an important influence in the design of filament-wound composite structures.

A. SPHEROID (L/D = 1.0)

Based on the factors outlined above and the design curves presented in Reference I-1, the value for the design-allowable strength of the hoop filaments ($F_{f,h}$) was determined as 266,000 psi, and for the longitudinal filaments ($F_{f,l}$) as 253,200 psi. The calculations follow.

1. Hoop Filaments

$$F_{f,h} = 0.89\ K_3K_4K_5K_6\left[1-(\tan^2\alpha/2)\right]F_{tu,s}$$

where

$$t_{f,h}/D_c = 2.84 \times 10^{-4},\ \text{and}\ K_3 = 1.08$$

$$D_c = 52.2\ \text{in., and}\ K_4 = 0.93$$

$$T = 158^\circ\text{F, and}\ K_5 = 0.98$$

$$\eta = 1\ \text{year at pressure, and}\ K_6 = 0.75$$

$$\alpha = 12^\circ\ 57'$$

Therefore,

$$F_{f,h} = 0.89\ (1.08)(0.93)(0.98)(0.75)(0.9735)(415{,}000)$$

$$= 266{,}000\ \text{psi}$$

Although calculations in Reference I-1 apply 437,000 psi for the value of $F_{tu,s}$, more recent studies and statistical interpretation of test results have shown that a conservative but more realistic approach would be to use 415,000 psi. This value takes into account three standard deviations in order to obtain a confidence level of 99.7%.

2. Longitudinal Filaments

$$F_{f,l} = 0.89\ K_1K_2K_3K_4K_5K_6K_p(\sec^2\alpha)F_{tu,s}$$

where

$$D_b/D_c = 0.21, \text{ and } K_1 = 1.0$$

$$L/D_c = 1.0, \text{ and } K_2 = 0.97$$

$$t_{f,1}/D_c = 2.14 \times 10^{-4}, \text{ and } K_3 = 1.09$$

$$D_c = 52.2 \text{ in., and } K_4 = 0.86$$

$$T = 158^{\circ}F, \text{ and } K_5 = 0.96$$

$$\eta = 1 \text{ year at pressure, and } K_6 = 0.75$$

$$K_p = 1.0 \text{ (no off-center ports)}$$

$$\alpha = 12^{\circ}\ 57'$$

Therefore,

$$F_{f,1} = (0.89)(1.0)(0.97)(1.09)(0.86)(0.96)(0.75)(1.0)(1.053)(415{,}000)$$

$$= 253{,}200 \text{ psi}$$

B. OBLATE SPHEROID

Based on the factors outlined above and the design curves presented in Reference I-1, the design-allowable strength of the longitudinal filaments was determined as 299,200 psi. The calculation follows.

$$F_{f,1} = 0.89\ K_1K_2K_3K_4K_5K_6K_p(\sec^2\alpha)F_{tu,s}$$

where

$$D_b/D_c = 0.17, \text{ and } K_1 = 1.0$$

$$L/D_c = 0.6, \text{ and } K_2 = 1.09 \text{ (for chambers having no cylindrical section)}$$

$$t_{f,1}/D_c = 1.88 \times 10^{-4}, \text{ and } K_3 = 1.11$$

$$D_c = 64.0 \text{ in., and } K_4 = 0.855$$

T = $+158^{o}F$, and $K_5 = 0.96$

η = 1 year at pressure, and $K_6 = 0.75$

K_p = 1.0 (no off-center ports)

α = $16^{o}\ 33'$

Therefore,

$$F_{f,1} = (0.89)(1.0)(1.09)(1.11)(0.855)(0.96)(0.75)(1.0)(1.088)(415,000)$$

$$= 299,200 \text{ psi}$$

C. SPHERE

The design ultimate tensile strengths for a sphere are calculated on the basis of a composite design-allowable strength. Conditions required in the optimization of a design utilizing unidirectional glass fibers include (1) continuity of fibers, (2) no slippage of filaments, and (3) relatively constant fiber stresses at all points. In a pressurized sphere with its isotropic force field, these conditions are compromised because the filaments cannot be oriented to exactly balance the force field. As a result, high shear stresses occur in the resin with subsequent reduction of composite efficiency. Previous work has demonstrated that precise placement of the fibers results in an average composite ultimate strength of 80,000 psi. Assuming a design factor of 0.75 for duration at load, as in the other configurations, this value is reduced to 60,000 psi. No consideration has been given (1) to the difficulty of controlling the resulting average thickness, or (2) to the possible required use of roving with less than 20 ends to obtain this thickness.

II. STRUCTURAL ANALYSIS

A. SPHEROID (L/D = 1.0)

This design approaches a spherical configuration, but utilizes a short cylindrical section and heads compatible with filament-winding patterns in which the unidirectional filaments are oriented to meet the biaxial force field. This orientation is accomplished by a combination of longitudinal and circumferential filaments. The design of the glass-filament-wound spheroid with a chamber-length to chamber-diameter (L/D) ratio of 1.0 was based on requirements that the tank successfully withstand the internal-pressure, fatigue-life, and service-life conditions, and meet the internal-volume design criteria of Table 25. The spheroidal filament-wound tank is shown schematically in Figure 35.

Margins of safety were determined from dimensional and conditional design factors used in defining the allowable strengths - effects of geometry, thickness, chamber diameter, boss diameters, wrapping angle, etc. - and the latest available statistical data on the basic strand strength. The contribution of the stainless steel metal-foil liner to the strength of the chamber is neglected on the basis of subscale test results. Table 26 presents pertinent dimensional and material parameters of the spheroid and a comparison with an oblate spheroid and a sphere with equivalent volumetric capacity (V). The symbols used in the preliminary design analysis of the spheroid (L/D = 1.0) are defined below.

Symbol	Definition, Units
D_c	Mean diameter of cylinder, in.
D_h	Mean diameter of hoop composite, in.
D_l	Mean diameter of longitudinal composite, in.
$F_{f,h}$	Allowable ultimate strength of hoop filaments, psi
$F_{f,l}$	Allowable ultimate strength of longitudinal filaments, psi
F_{tu}	Ultimate tensile strength, psi
F_{ty}	Tensile yield strength, psi
M.S.	Margin of safety
$M.S._y$	Margin of safety at tensile yield strength
P_{vg}	Amount of glass filament in composite, vol%
p_b	Design burst pressure, psi
t_c	Total composite thickness of cylinder, in.
t_h	Total hoop-composite thickness, in.
t_l	Total longitudinal-composite thickness, in.

TABLE 25

DESIGN CRITERIA, FILAMENT-WOUND PROTOTYPE TANKAGE FOR STORABLE PROPELLANTS[1]

	Sphere	Oblate Spheroid	Spheroid (L/D=1.0)
Aerojet Drawing No.	-	177500	177499
Internal volume, cu in.	92,000	92,000	92,000
Outside diameter, in.	56.111	64.049	52.307
Working pressure, p_w, psi	150	150	150
Proof pressure, p_p, psi	165	165	165
Design burst pressure, p_b, psi	207	207	207
Fatigue-life requirement, cycles	25 to p_w	25 to p_w	25 to p_w
Service-life requirement, years	1 at p_w	1 at p_w	1 at p_w
Maximum temp requirement, ^oF	+158	+158	+158
Minimum temp requirement, ^oF	-117	-117	-117
Ultimate hoop-filament strength, psi	-	-	266,000
Ultimate longitudinal-filament strength, psi	-	299,200	253,200
Ultimate longitudinal composite strength, psi	60,000	184,100	160,000
Ultimate hoop composite strength (cylindrical section), psi	-	-	115,100

[1] Strengths are based on ability of tankage to successfully withstand a minimum burst pressure of 207 psi after being subjected to 25 cycles at a working pressure of 150 psi and 1 year at a sustained pressure of 150 psi.

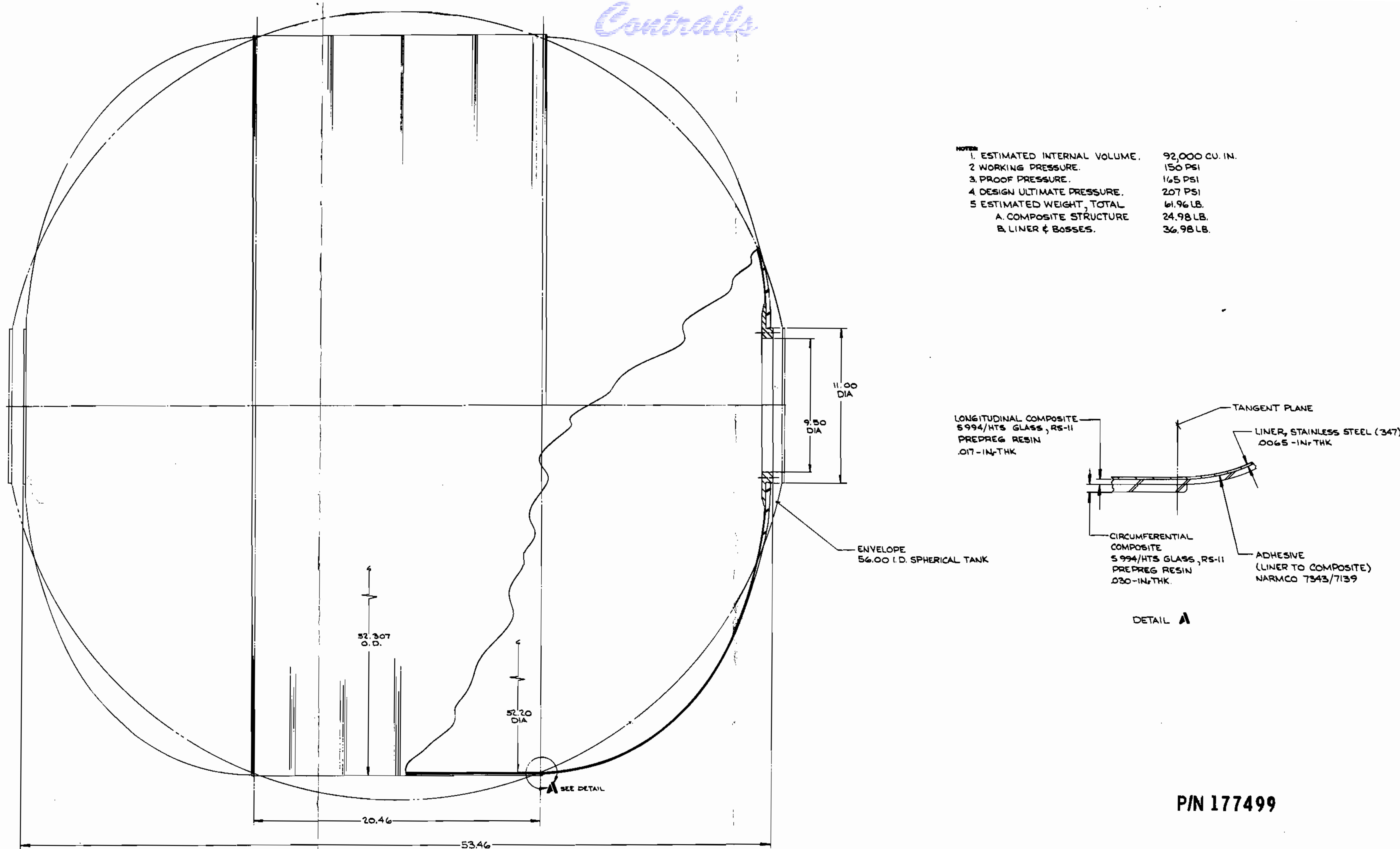


Figure 35. Spheroid (L/D = 1), Filament-Wound Storable-Propellant Tankage

TABLE 26
DIMENSIONAL AND MATERIAL PARAMETERS, FILAMENT-WOUND PROTOTYPE TANKAGE

	Sphere	Oblate Spheroid	Spheroid (L/D=1.0)
Aerojet Drawing No.	-	177500	177499
Internal volume, cu in.	92,000	92,000	92,000
Outside diameter, in.	56.111	64.049	52.307
Inside diameter (inside of liner), in.	56.00	64.00	52.20
Liner thickness, in.	0.0065	0.0065	0.0065
Total composite wall thickness, in.	0.049 (av)	0.018	0.047
Longitudinal composite thickness, in.	0.049 (av)	0.018	0.017
Hoop composite thickness, in.	-	-	0.030
Boss-to-boss length, in.	56.00	40.75	53.46
Boss outside diameter, in.	11.00	11.00	11.00
Boss opening diameter, in.	9.50	9.50	9.50
Liner and boss material	347 SS	347 SS	347 SS
Glass filaments	S-HTS	S-HTS	S-HTS
Roving type	20-end	20-end	20-end
Resin matrix	RS-11 (prepreg)	RS-11 (prepreg)	RS-11 (prepreg)
Liner-to-composite adhesive	Narmco 7343/7139	Narmco 7343/7139	Narmco 7343/7139

Symbol	Definition, Units
α	Angle between line in axial direction and filament path, degrees
σ_b	Ultimate bending stress, psi
σ_{by}	Bending stress, psi, at proof pressure
σ_c	Hoop stress (total cylinder composite thickness), psi
$\sigma_{f,h}$	Hoop-filament stress, psi
$\sigma_{f,l}$	Longitudinal-filament stress, psi
σ_h	Hoop-composite stress, psi
σ_l	Longitudinal-composite stress, psi

1. Composite Structure

a. Hoop Filaments

$$\sigma_{f,h} = \frac{p_b D_h}{2\, t_h P_{vg}} \left(1 - \frac{\tan^2 \alpha}{2}\right)$$

where

$$p_b = 207 \text{ psi}$$

$$D_h = 52.277 \text{ in.}$$

$$t_h = 0.030 \text{ in.}$$

$$P_{vg} = 0.673$$

$$\alpha = 12^\circ\ 57'$$

Therefore,

$$\sigma_{f,h} = \frac{(207)(52.277)}{(2)(0.030)(0.673)} \left(1 - \frac{0.05285}{2}\right)$$

$$= 260{,}900 \text{ psi}$$

$$\text{M.S.} = \frac{F_{f,h}}{\sigma_{f,h}} - 1$$

Since

$$F_{f,h} = 266{,}000 \text{ psi}$$

$$M.S. = \frac{266{,}000}{260{,}900} - 1 = \qquad \underline{+0.019}$$

b. Hoop Composite

$$\sigma_h = \sigma_{f,h} P_{vg}$$

$$= (260{,}900)(0.673)$$

$$= 175{,}600 \text{ psi}$$

c. Longitudinal Filaments

$$\sigma_{f,l} = \frac{p_b D_l}{4\, t_l P_{vg} \cos^2\alpha}$$

$$D_l = 52.230 \text{ in.}$$

$$t_l = 0.017 \text{ in.}$$

Therefore,

$$\sigma_{f,l} = \frac{(207)(52.230)}{(4)(0.017)(0.673)(0.9498)}$$

$$= 248{,}700 \text{ psi}$$

$$M.S. = \frac{F_{f,l}}{\sigma_{f,l}} - 1$$

Since

$$F_{f,l} = 253{,}200 \text{ psi}$$

$$M.S. = \frac{253{,}200}{248{,}700} - 1 = \qquad \underline{+0.018}$$

d. Longitudinal Composite

$$\sigma_l = \sigma_{f,l} P_{vg} \cos^2\alpha$$

$$= (248{,}700)(0.673)(0.9498)$$

$$= 159{,}000 \text{ psi}$$

e. Composite Hoop (Cylinder)

$$\sigma_c = \frac{p_b D_c}{2\ t_c}$$

where

$$D_c = 52.260 \text{ in.}$$

$$t_c = t_h + t_l = 0.047 \text{ in.}$$

$$\sigma_c = \frac{(207)(52.260)}{(2)(0.047)}$$

$$= 115{,}100 \text{ psi}$$

2. Metal Hardware

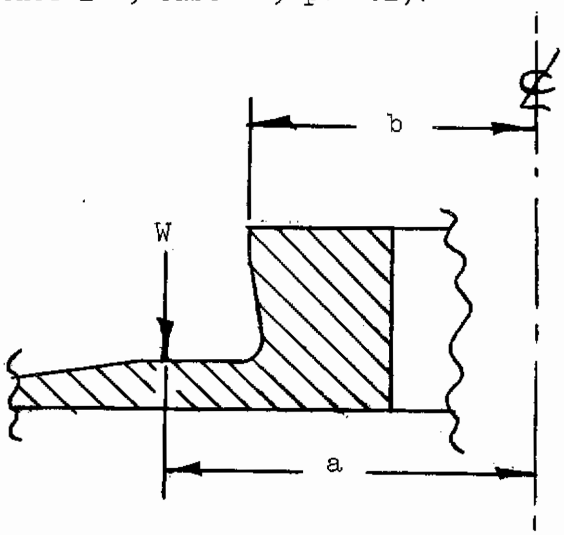
The forward and aft bosses are assumed to act as circular plates with a fixed inner edge. The end-for-end wrap pattern of the longitudinal filaments produce a rigid band (w_t) approximately 1 in. wide around the boss, which supports the flange. The basic load (W) on the flange is shown below. Bending stress at the juncture of the flange and boss is calculated by the use of formulas for loading on a flat plate (Reference I-2, Case 22, p. 201):

$$b = \frac{11.00}{2} = 5.50 \text{ in.}$$

$$a = b + \frac{w_t}{2}$$

$$= 5.50 + \frac{1.0}{2} = 6.00 \text{ in.}$$

$$\sigma_b = \frac{W\beta_{22}}{t^2}$$



The factor β_{22} may be approximated by

$$\beta_{22} = \frac{a}{b} - 1$$

Thus

$$\beta_{22} = \frac{6.00}{5.50} - 1 = 0.09$$

$$W = p_b \pi a^2$$

$$= (207)(\pi)(6.00)^2$$

$$= 23,450 \text{ psi}$$

Therefore,

$$\sigma_b = \frac{(23,450)(0.09)}{(0.25)^2}$$

$$= 33,800 \text{ psi}$$

$$M.S. = \frac{F_{tu}}{\sigma_b} - 1$$

Approximating the strength of AISI 347 SS to that of AISI 301 SS,

$$F_{tu} = 75,000 \text{ psi (Reference I-3 for AISI 301 SS)}$$

Therefore,

$$M.S. = \frac{75,000}{33,800} - 1 = \qquad \underline{+1.22}$$

Determining, as above, the margin of safety at the tensile yield strength (F_{ty}) of the material and at the proof pressure (165 psi),

$$W = 18,700 \text{ psi}$$

$$\sigma_{by} = 26,900 \text{ psi}$$

$$M.S._y = \frac{F_{ty}}{\sigma_{by}} - 1$$

From Reference I-3 for AISI 301 SS,

$$F_{ty} = 30{,}000 \text{ psi}$$

Therefore,

$$M.S._y = \frac{30{,}000}{26{,}900} - 1 = \qquad \underline{+0.12}$$

B. OBLATE SPHEROID

The design of this glass-filament-wound oblate spheroid with a chamber-length to chamber-diameter (L/D) ratio of approximately 0.63 was based on requirements that the tank successfully withstand the internal-pressure, fatigue-life, and service-life conditions and meet the internal-volume design criteria of Table 25. The oblate-spheroid filament-wound tank is shown schematically in Figure 36. Margins of safety were determined from dimensional and conditional design factors used in defining the allowable strengths - effects of geometry, thickness, chamber diameter, boss diameters, wrapping angle, etc. - and the basic strand strength. The contribution of the stainless steel metal-foil liner to the strength of the chamber was neglected on the basis of preliminary test results. Table 26 presents pertinent dimensional and material parameters for the oblate spheroid and comparisons with a spheroid (L/D = 1) and a sphere with equivalent volumetric capacity. The symbols used in the preliminary design analysis of the oblate spheroid are defined below.

Symbol	Definition, Unit
D_l	Mean diameter of longitudinal composite, in.
$F_{f,l}$	Allowable ultimate strength in longitudinal filaments, psi
M.S.	Margin of safety
P_{vg}	Amount of glass filament in composite, vol%
p_b	Design burst pressure, psi
t_l	Total longitudinal-composite thickness, in.
α	Angle between line in axial direction and filament path degrees
$\sigma_{f,l}$	Longitudinal-filament stress, psi
σ_l	Longitudinal-composite stress, psi

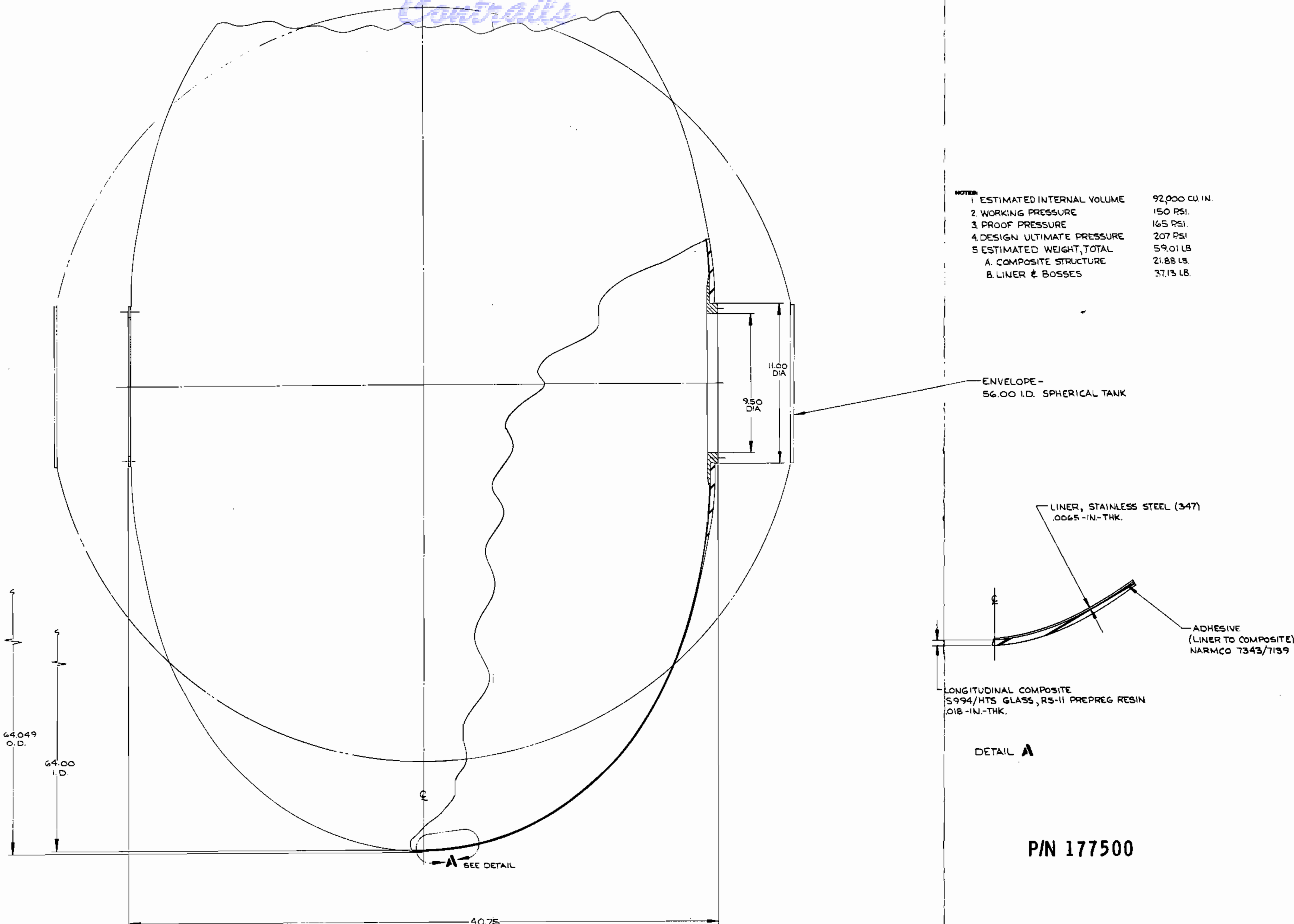


Figure 36. Oblate Spheroid, Filament-Wound Tankage

1. Composite Structure

a. Longitudinal Filaments

$$\sigma_{f,1} = \frac{p_b D_1}{4\ t_1 P_{vg} \cos^2\alpha}$$

D_1 = 64.031 in.

t_1 = 0.018 in.

α = $16^\circ\ 33'$

Therefore,

$$\sigma_{f,1} = \frac{(207)(64.031)}{(4)(0.018)(0.673)(0.9181)}$$

$= 298{,}000$ psi

$$M.S. = \frac{F_{f,1}}{\sigma_{f,1}} - 1$$

$F_{f,1}$ = 299,200

Therefore,

$$M.S. = \frac{299{,}200}{298{,}000} - 1 = \qquad +0.004$$

b. Longitudinal Composite

$\sigma_1 = \sigma_{f,1} P_{vg} \cos^2\alpha$

$= (298{,}000)(0.673)(0.9181)$

$= 184{,}100$ psi

2. Metal Hardware

Thicknesses and pertinent dimensions of the stainless steel bosses and flanges are the same as for the spheroid (L/D = 1.0). The structural analysis is therefore the same as the analysis presented in Section II,A,2 of this appendix.

C. SPHERE

Application of glass-filament-winding fabrication techniques to a spherical configuration does not produce an optimized design because of the unidirectional qualities of the glass filaments and isotropic-force-field conditions in the pressurized sphere. Theoretically, if the fibers are oriented to meet the exact loads, an ideal isotensoid condition would prevail, the load-carrying efficiencies would be the same, and the weight for a variety of configurations of wound vessels would also be the same for the same pressure-volume product. Ideal filament-winding conditions require (1) continuity of fibers, (2) no slippage of filaments, and (3) relatively constant fiber stresses at all points. In winding a sphere, however, practical considerations (including the limitations of design and equipment) tend to compromise these conditions. In particular, because the windings in a sphere cannot be made to exactly balance the force-field conditions, high shear stresses occur in the resin; these stresses reduce the composite efficiency.

The design of this glass-filament-wound sphere takes into account this efficiency decrease and is based on requirements that the tank successfully withstand the internal-pressure, fatigue-life, and service-life conditions, and meet the internal-volume design criteria of Table 25. The contribution of the stainless steel metal-foil liner to the strength of the chamber was neglected on the basis of preliminary test results. Table 26 presents pertinent dimensional and material parameters for the sphere and comparisons with a spheroid (L/D = 1.0) and an oblate spheroid with equivalent volumetric capacity. The symbols used in the preliminary design analysis of the sphere are defined below.

Symbol	Definition, Units
D_c	Mean diameter of sphere, in.
F_c	Allowable ultimate strength in composite wall, psi
M.S.	Margin of safety
p_b	Design burst pressure, psi
t_c	Total composite thickness of sphere, in.
σ_c	Composite stress (total composite thickness), psi

1. Composite Structure

$$\sigma_c = \frac{p_b D_c}{4\, t_c}$$

$$p_b = 207 \text{ psi}$$

$$D_c = 56.062 \text{ in.}$$

$$t_c = 0.049 \text{ in. (av)}$$

Therefore,

$$\sigma_c = \frac{(207)(56.062)}{(4)(0.049)}$$

$$= 59{,}200 \text{ psi}$$

$$\text{M.S.} = \frac{F_c}{\sigma_c} - 1$$

$$F_c = 60{,}000 \text{ psi}$$

Therefore,

$$\text{M.S.} = \frac{60{,}000}{59{,}200} - 1 = \underline{+0.01}$$

Although a theoretical thickness of 0.049 in. was calculated for the composite structure, practical considerations preclude construction of a 56.0-in.-dia filament-wound sphere to this average thickness. Present Aerojet equipment employs nine spools of 20-end roving, producing a band width of approximately 1.0 in. and a thickness of 0.0075 in. per layer. If enough spools of single-end roving were used to provide a 2.0-in. side-by-side band width, an approximate thickness of 0.004 in. per layer would be produced.

Assuming that a winding pattern with a balanced distribution of fiber could be wound with the 2.0-in.-wide tape, a total of approximately 14 passes or angular changes (28 layers) would be required to complete the winding cycle. This would produce a minimum composite thickness of 28 x 0.004 in., or 0.112 in. From a practical standpoint, a composite thickness of at least 0.112 in. is produced for a 56.0-in.-dia sphere by present winding equipment and processes, even though a theoretical thickness of only 0.049 in. is required.

2. Metal Hardware

The thicknesses and pertinent dimensions of the stainless steel bosses and flanges are the same as for the spheroid (L/D = 1.0). The structural analysis is therefore the same as the analysis in Section II,A,2 of this appendix.

III. WEIGHT ANALYSES

Comparative weight analyses are presented in Table 27 for the three configurations - sphere, oblate spheroid, and spheroid - selected for storable-propellant tankage with an equivalent volumetric capacity of 92,000 cu in. The calculated weights (W_T) are for the basic metal-lined filament-wound tank and do not take into consideration plugs, hardware, or means of tank support.

As in stress analysis, weight and analysis indicated that the oblate spheroid offers the optimum configuration for filament winding on the basis of minimum weight. Envelope restrictions may limit the acceptability of this configuration, however, because of the increase in diameter required to contain an equivalent capacity. The spheroid with L/D = 1.0, although not as ideal as the oblate spheroid, nevertheless offers the potential advantage of having a smaller diameter and less weight than the sphere with the same internal volume.

IV. FABRICATION PROCEDURES

The basic procedures and techniques for the fabrication of metal-lined, filament-wound, prototype tankage for storable propellants, including Aero-ZINE 50, nitrogen tetroxide, and chlorine trifluoride, are presented in this section. The principal parameters applied in the design included a tankage envelope of 92,000 cu in. internal capacity and a working pressure of 150 psi.

The tank design was based on the results of subscale testing, as constrained by the feasibility of applying subscale fabrication and processing techniques to larger-diameter tankage. Specifically, the subscale test results demonstrated the feasibility of forming and welding and the potential for using thin (0.0065-in.-thick) 347 SS foil for the tank liners. The fabrication of glass-filament-wound tankage utilizing a thin-metal-foil liner is essentially the same whether the configuration is a sphere, an oblate spheroid, or a spheroid with L/D = 1.0. The tooling required is summarized in Table 28. The description below is in general applicable to the fabrication of the three alternative configurations, and variations in fabrication processes due to

TABLE 27
WEIGHT ANALYSIS, FILAMENT-WOUND PROTOTYPE TANKAGE[1]

	Sphere	Oblate Spheroid	Spheroid (L/D=1.0)
Aerojet Drawing No.	-	177500	177499
Internal volume, cu in.	92,000	92,000	92,000
Outside diameter, in.	56.111	64.049	52.307
Total weight, lb	73.11	59.01	61.96
Composite structure	36.29	21.88	24.98
Forward head	-	10.94	6.79
Aft head	-	10.94	6.79
Cylindrical section	-	-	11.40
Metal hardware	36.82	37.13	36.98
Liner	18.12	18.43	18.28
Bosses (2)	18.70	18.70	18.70
Performance factor ($p_b V/W_T$), in.	2.60×10^5	3.23×10^5	3.07×10^5

[1] Weights are based on tankage able to successfully withstand a minimum burst pressure of 207 psi after being subjected to 25 cycles at a working pressure of 150 psi and 1 year at a sustained pressure of 150 psi.

TABLE 28
REQUIRED TOOLING, FILAMENT-WOUND PROTOTYPE TANKAGE

Description	Number Required	Purpose
Miscellaneous forming and welding fixtures and tooling	As needed	To support, accurately position, and clamp the metal-foil gore sections for preliminary spot welding and roll-seam resistance welding of the gore joints and final welding of the sections to each other and to the bosses
Acid cleaner tank	1	To contain the nitric acid solution used for cleaning and removing contaminants from the external surfaces of the metal-foil liner
Hot-water rinse tank	1	To soak and rinse the metal-foil liner after completion of cleaning in the acid cleaner tank
Winding shaft and adapters	1	To support the liner and plaster mandrel during winding
Protective support cover	1	To enclose and protect the liner assembly and offer support to the liner during plaster application
Rotational and tumbling fixture	1	To enclose the protective cover and mount in a rotating device that will evenly coat the inner surface of the liner with a layer of plaster
Handling and curing cart	1	To support and handle the winding shaft and the tankage assembly during various stages of fabrication
Washout fixture	1	To support the cured final assembly of the composite tankage during washout and removal of the internal plaster liner
Hydrotest plugs	2	To attach to and seal the bosses of the structure during hydrostatic proof testing
Shipping crate	1	To enclose and protect the completed assembly during shipment and delivery
Test tooling	As needed	To facilitate leak testing, etc.

configuration are indicated. The discussion covers liner fabrication, mandrel fabrication, winding operation, resin curing and mandrel removal, and proof testing, inspection, and shipment.

A. LINER FABRICATION

The optimum method for fabrication of the metal liner is governed by two principal factors: (1) the selection of 0.006- to 0.0065-in.-thick foil for weight saving, and (2) a maximum width of 24 in. obtainable in that foil thickness.

The optimum method for joining sections to fabricate the three proposed forms of metal liners is considered to be roll-seam resistance welding. Fusion welding for the complete liner shell is not recommended because of excessive tooling and fixturing costs and the unreliability of fusion welds in a thin-metal-foil component. To obtain satisfactory leakproof metal shells, the rejection rate would be excessive because of the difficulty of obtaining consistently good fusion welds with metal foils.

Under the procedures developed for this program, half sections of the 56.0-in.-ID spherical-tank liner and half sections of the 64.0-in. oblate-spheroid-tank liner will be fabricated from preformed gore sections. The end or head sections of the liner for the 52.2-in.-dia spheroidal tank with L/D = 1.0 will be fabricated in the same manner. In preforming the gore sections, either by stretch forming or press forming, 347 SS will work-harden to some extent. Although this work hardening would be insufficient to reduce the capability for resistance-seam welding, it will be removed by an anneal at 1850°F. If the tankage is not intended for use with a highly corrosive propellant, the gore sections may be annealed before resistance welding to prevent distortion. Sizing with the aid of rubber-lined dies to remove waviness will be necessary after annealing.

Welding of the austenitic stainless steels, even the 347 and 321 stabilized grades, can result in the precipitation of carbides around grain boundaries. When this occurs, exposure to highly corrosive propellants can result in knife-line attack and eventual leakage at this sensitized area. In this case it will be desirable to anneal the metal liner after fabrication, which will require extensive fixturing to prevent distortion during annealing.

Two techniques could be employed to join the half sections to form the spherical liner or the oblate-spheroid liner. These techniques are also applicable in joining the two head sections to the straight section to form the spheroid (L/D = 1.0). They are described below.

1. Resistance-Roll-Seam Welding

By offsetting the two sections, the maximum thickness of the welded joint would be three sheet thicknesses. By scarfing the edge of the longitudinal weld joints, a uniform leakproof joint can be made in the circumferential closure welds. Because this technique requires the use of a seam roller in contact with the inside and outside surfaces of the overlap at the joint area, the mandrel that supports the metal shell during filament winding cannot be installed before the closure operation. If this were done, the inside roller could not be used and sufficient pressure to make a good leakproof resistance-seam weld in the circumferential joint could not be obtained.

2. Modified Fusion-Weld Technique

With this method, the ends of the half sections would be bent to form a flange whose height would be at least 3 times the metal thickness. The flanges would be butted against each other and tacked in place. An annealed copper backup strip about 0.125 in. thick would be installed on the inside to provide a uniform weld surface. After proper fixturing, the butted flanges would be fusion-welded without the use of a filler rod. This method would permit the installation of a plaster mandrel prior to the closing operation. Some development work would be required to determine the minimum height of the resulting weld bead needed to obtain a leakproof joint. The problem of glass-fiber discontinuities during winding may be excessive if the resulting weld bead is found to be high.

For reasons of fabrication feasibility and economy, as well as weld reliability, as discussed above, the resistance roll-seam welding technique was chosen for the joining of the liner sections.

B. MANDREL FABRICATION

An all-plaster mandrel will be used for filament winding. Its selection to provide a firm support for the applied windings is based on several factors, including (1) economic considerations because of the limited number of chambers required, (2) sufficient strength demonstrated with previous mandrels of this type, (3) dimensional stability, (4) adequate load-deflection characteristics, (5) a limited number of boss openings available in which to place any type of segmented mandrel, and (6) capability for withstanding the elevated-temperature exposure imposed during moisture removal and resin curing.

A female mold-type covering will be added first around the completed liner assembly for protection of the external surfaces and support of the plaster, which is to be poured internally. An axial winding shaft and adapters that will offer accessibility to the interior will also be applied and fastened to the bosses. The entire structure will be placed in a tumbling-type fixture; the fixture will rotate and facilitate even coating of the plaster, which will be primarily a Kerr DMM plaster mixture. The mandrel will then be oven-dried to remove excess moisture from the plaster.

Additional advantages of this type of mandrel include (1) use of readily available and inexpensive raw materials, (2) ease of modification to incorporate design changes, (3) simplicity of plaster-solution insertion and rotation to obtain a uniform and adequate wall thickness, and (4) ease of plaster removal by means of a mild acetic-acid solution.

C. WINDING

Requirements of the tankage system dictate that the bond between the metal-foil liner and the filament-wound ease must remain intact at a temperature as low as -110°F. The adhesive that satisfies this requirement is Narmco 7343/7139; it is to be applied over the liner prior to filament winding.

The surface of the 347 SS metal-foil liner will first be cleaned with a nitric-acid solution to remove contaminants and provide a good bonding surface for adhesive application.

The basic material for the winding of the composite structure is S-HTS glass roving. It will be supplied as 20-end roving and in a preimpregnated form with the RS-11 resin system (DEN 328/BF_3-400).

The tankage will be wound by positioning the mandrel in the Aerojet vertical-winding machine, adjusting the tension and width of the glass filaments to form the desired tape, and placing the tape over the lined mandrel in the required winding patterns. The patterns for the spheroid will utilize both end-for-end wraps (longitudinal) and hoop wraps (circumferential); oblate-spheroid fabrication requires longitudinal wraps solely. Longitudinal wraps will be applied by the winding arm, which will be rotated at the required angle with respect to the vertical axis. Hoop wraps for the spheroid will be applied by a roving supply carriage that moves vertically. Winding of the sphere will require that the orientation of the wraps be changed several times during fabrication in order to place the filaments in a pattern that will sustain the isotropic-force-field loads caused by internal pressurization. Tensioning equipment, installed on both the vertical arm and the horizontal carriage of the winding machine, will assure that all roving is applied at the desired tension. Glass weights will be recorded at each step of the operation and an exact record of tensions will be maintained for purposes of adjustment and documentation.

D. RESIN CURING AND MANDREL REMOVAL

Upon completion of winding, the composite structure will be cured in an oven. Previous development studies and analytical evaluations demonstrated that increased interlaminar shear strength is obtained between layers of wrap by curing the chamber at a pressure of 15 psig. This is accomplished by the use of vacuum-bag compaction while the composite structure is being cured. This technique also increases composite-structure integrity by removing any trapped air and by providing a suitable bleeder layer under the vacuum to remove any excess resin.

A multiple-step cure will be employed to obtain the desired resin flow prior to resin gelling. This will be followed by programed temperature increases to minimize thermal stresses and complete resin polymerization. Specifically, the cure cycle will consist of the preliminary resin-gelling cycle of 2 hours at 200°F, intermediate curing for 2 hours at 250°F, and a final cure of 10 hours at 300°F. Cooldown will also be programed at a controlled rate to minimize excessive thermal expansion of the mandrel, liner, and composite structure.

The winding shaft and adapters will be removed after cooldown, to permit access to the interior of the structure through the bosses. After the structure is placed on a washout fixture, the plaster will be removed by circulating an acetic-acid solution through the interior. The final manufacturing operations will include cleaning of the internal and external surfaces.

E. PROOF TESTING, INSPECTION, AND SHIPMENT

To ensure the structural integrity of the composite tankage, a hydrostatic proof pressure test will be performed for 2 minutes at 150 psig.

Before this test and prior to filament winding, a low-pressure leak test will have been performed on the completed liner structure to demonstrate weld integrity. Dye-penetrant and X-ray tests will also be utilized as required during and after liner fabrication.

Although these and other appropriate inspections will be conducted throughout manufacture, a final check for dimensional compliance and conformance to quality criteria will be performed before the tankage is packaged for shipment and delivery.

V. SUPPORT STRUCTURE

The support structure for storable-propellant tankage presents unique structural and thermal problems that must be considered in relation to the tankage, adjacent structural components, and the space-vehicle design criteria. These problems were taken into account in proposing several design configurations for the support structure. Figure 37 shows the manner in which the proposed support methods mechanically restrain the tankage under normal ground-handling conditions.

The first design consists of a series of glass-fiber tension members arranged in the form of two open-weave frustums of a cone assembled with the smaller diameters back-to-back. The smaller diameter will provide means of attachment to the equator of the head, while the larger-diameter portions of the support structure will be mounted to the outer shell of the vehicle at specific hard points. The structural framework or tension members of the frustums will be fabricated by winding unidirectional, high-strength, glass roving preimpregnated with an epoxy resin. The winding pattern will be such that the final form of the tank support will simulate a structural truss consisting of tension members. The members of the support structure will be pretensioned during assembly and will remain in this condition in order to maintain sufficient tension during dynamic reversal of loads in any midcourse correction maneuvers of the missile. The glass-reinforced-plastic support structure will be attached by integrally winding clips in the storage tankage and by fastening to welded clips or to a ring on the inner surface of the

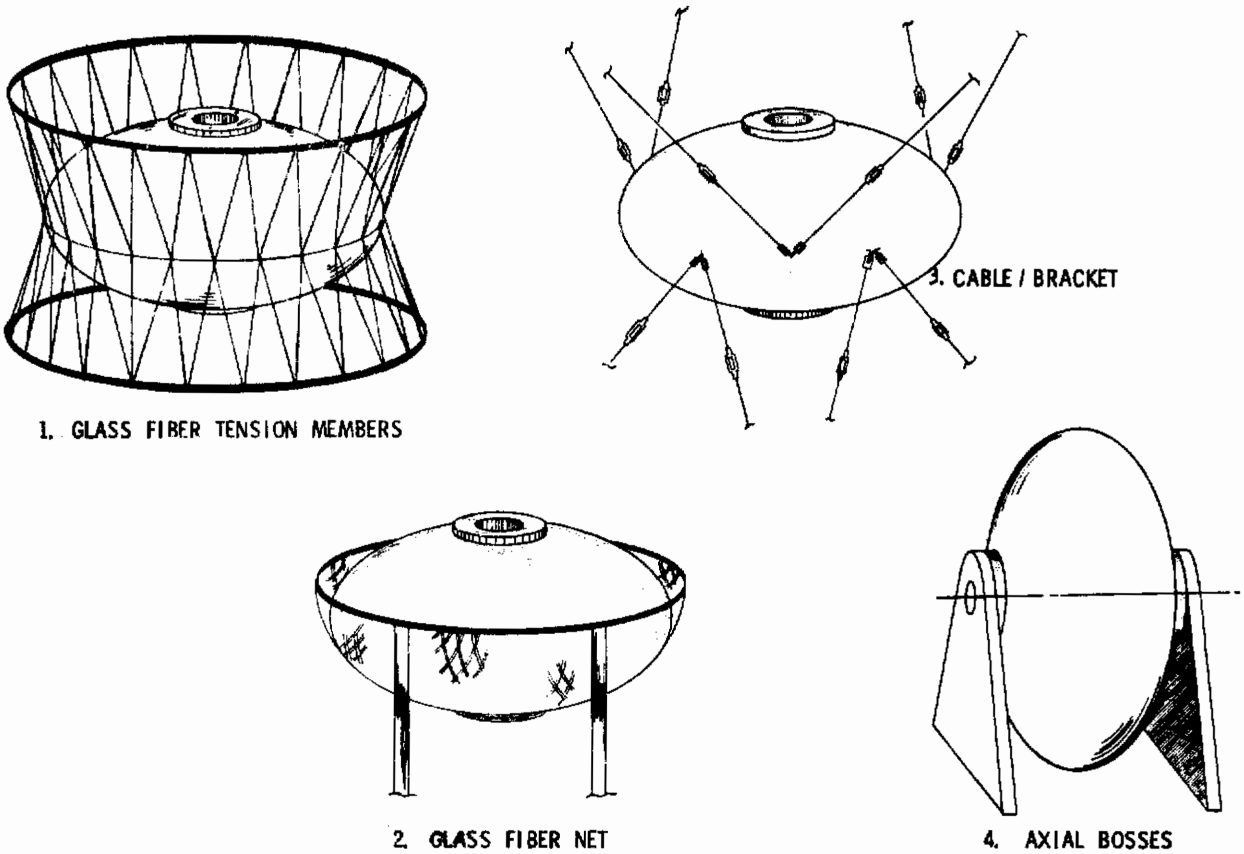


Figure 37. Support Structure, Liquid-Propellant Tankage

outer shell. Heat leakage will be minimized by the low thermal conductivity of the glass-fiber members.

A second design approach for the support structure is a configuration that employs a glass-fiber composite net structure to enclose the propellant tankage. The fiber net will be attached to the outer shell by means of a metallic ring mounted to the shell and supported in the area opposite the circumferential section of the tankage. Additional tension members will maintain the tankage equidistant from the inner wall of the outer shell. The advantages of the glass-fiber tension members are also applicable to the glass-fiber net support structure.

A third alternative would be the use of identical brackets and hard points common to present support structures of space vehicles. Metal brackets would be wound in the filament-wound composite structure. An elastomeric interlayer between the metallic bracket and the composite tankage wall would permit different responses to extensional strains without inducing excessive load transfers. Glass-fiber or steel cables with clevis-type fasteners and turnbuckles to induce tension would bridge the area between the propellant tankage and the support brackets on the outer shell.

A fourth support approach, although not necessarily ideal from the standpoints of filling and dumping, would be to mount the tankage by the axial bosses. Although this method permits the use of existing metal hardware, in contrast to the use of wrapped-in brackets or lugs, care must be exercised to avoid restraining the bosses axially, because this restraint imposes undue loads on the composite structure of the tankage during pressurization.

REFERENCES

I-1. Improved Filament-Wound Construction for Cylindrical Pressure Vessels, ML-TDR-64-63, Vols. I and II. Air Force Materials Laboratory, Research and Technology Division, Wright-Patterson Air Force Base, Ohio. (Prepared by Aerojet-General under Contract AF 33(616)-8442.) March 1964.

I-2. R. J. Roark, Formulas for Stress and Strain. New York. McGraw-Hill. 1954. p. 201 (Case 22).

I-3. Strength of Metal Aircraft Elements. MIL-HDBK-5. Armed Forces Supply Support Center, Washington D.C. March 1959. p. 35.

APPENDIX II

SPACE EXPERIMENT ANALYSIS AND DESIGN

The preliminary design of a space environment presented here utilizes excess payload capacity of a satellite vehicle. The experiment will evaluate an 8-in. isotensoid, oblate-spheroid, metal-lined, filament-wound, propellant tank. The experimental system will operate on command for a 1-year storage period in space with the tank pressurized to 150 psia and filled with one of the following propellants: AeroZINE 50, N_2O_4, or ClF_3.

The tank will be instrumented with strain gages, pressure transducers, temperature transducers, crack detectors, and accelerometers. Redundant instrumentation is provided, where necessary, to ensure a high probability of mission success. Some of the component designs were not finalized because the launch vehicle and satellite have not been specified. Sufficient detail is given for each subsystem of the experiment to facilitate the preparation of detailed fabrication costs. The detailed experimental and mathematical analyses required to finalize certain design aspects may be performed at the beginning of any program funded to fabricate, test, and orbit the experimental system.

I. PRELIMINARY STUDIES

A. SUBJECT OF EXPERIMENT

A filament-wound propellant tank with a metallic liner is desirable for space use because of the weight savings realized in comparison with the conventional metal tank. The effect of the unusual environments of space on the characteristics of filament-wound vessels must be determined and evaluated to adequately assess their operational and long-term storage characteristics. Laboratory simulation of the synergistic space effects is not currently possible, and prolonged simulation of individual space-environment parameters is costly. A practical evaluation approach is to orbit the system as a piggyback satellite.

B. EVALUATION TECHNIQUE

The design concepts listed below for the space-experiment package were considered in arriving at the most practical and realistic method of testing the tankage.

1. The volume of propellant stored at any one time will depend on the mission requirements. For a realistic storage cycle, the tank will be full at orbital injection, and the propellant will be used throughout the mission. It is conceivable that the properties of the vessel will vary significantly with ullage; an expulsion system was therefore considered necessary.

2. To perform properly on command, it is necessary to maintain the propellant in a pressurized condition, thus establishing the need for a pressurization system.

3. For proper evaluation of the tank materials, thermal insulation (other than shielding from radiative heating) should not be used, to ensure that the propellant tank is fully exposed to the space environment.

4. The most important parameters affecting the propellant vessel include temperature, pressure, and tank-wall strain. Other parameters of interest are tank-wall permeability, total propellant mass, and propellant mass flow rate.

C. CONFIGURATION

No launch vehicle or space system was specified for accommodating this experiment. A configuration was therefore adopted that can easily be installed on many types of vehicles, or can use a telemetry system. Typical telemetry systems and test-flight vehicles were reviewed and used as guidelines for this design effort (References II-1 through II-9).

The complete experiment package is enclosed in a cylindrical shell. The shell, used primarily for thermal control, is also a realistic configuration for a propellant-tank evaluation. Figure 38 shows a possible attachment of a power supply and telemetry. The particular configuration used in this concept was taken from Reference II-2; other systems could be readily adapted.

II. ENVIRONMENTS OF SPACE

Space-environment parameters considered as unusual and hazardous to an orbiting satellite include gas pressure at the altitude of interest (vacuum),

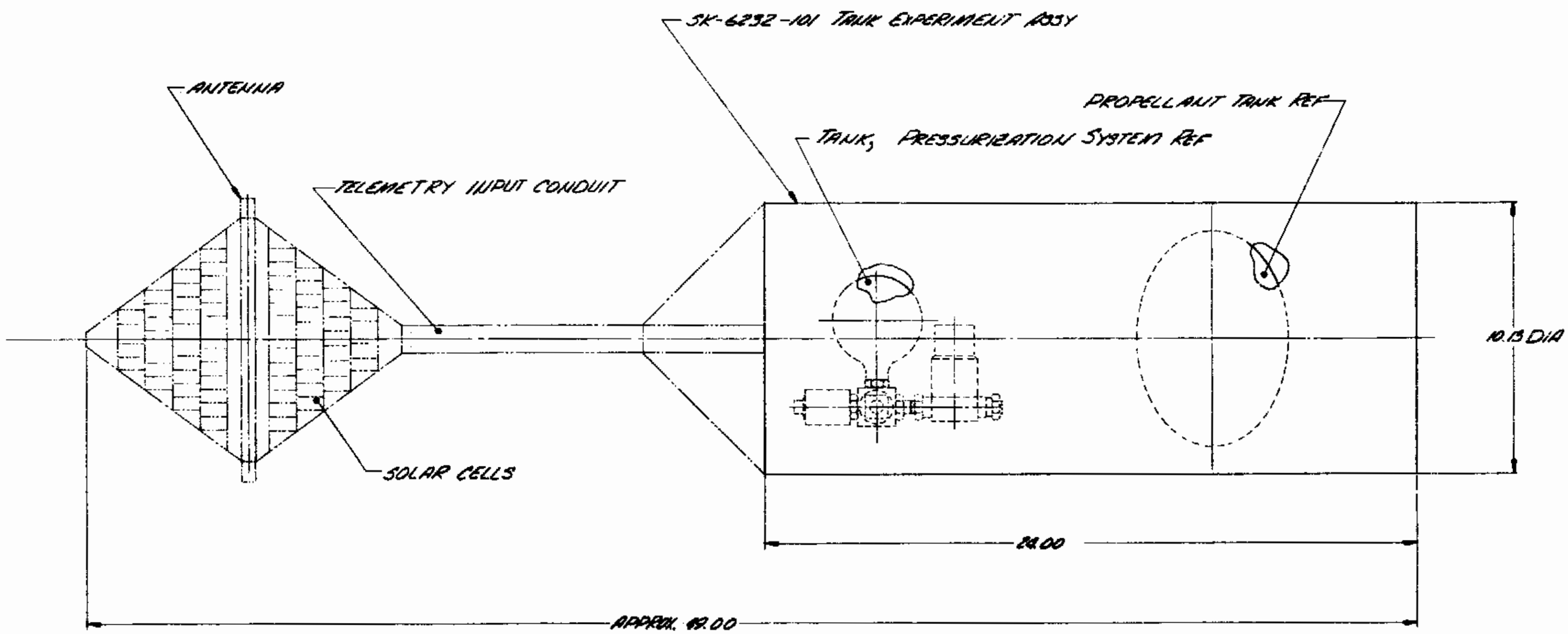


Figure 38. General Arrangement, Tank Experiment

electromagnetic and geomagnetic trapped-particle radiation, micrometeoroids, and thermal radiation.

Vacuum could cause outgassing of the resin in the filament-wound structure; this would tend to degrade the structural strength. Additional vacuum effects include cold welding of metals in contact, as in the propellant valve or perhaps the pressure transducers, and changes in the response time of components in contact.

Irradiation of elastomeris materials imposes a problem in long-term storage. Storage of 1 year, even in the heart of the Van Allen belts, does not appear to be significant in terms of deleterious radiation exposure. The threshold damage level for this material appears to require a much higher total radiation dose than can presently be absorbed by operation in the heart of the Van Allen belts for a year.

Meteoroids pose a more serious hazard. Very little information is available on the damage to filament-wound structures by particles at hypervelocities, and it is not possible to precisely predict a probability of mission success. The propellant vessel will be enclosed by a cylindrical shell for thermal control; this shell can be used as a standoff barrier to protect the propellant tank from meteoroid penetrations.

Distance from the propellant tank is critical when a secondary material is used as a standoff barrier. It varies with the material, configuration, size of meteoroid, and meteoroid velocity; in turn, these factors depend on the mission life, altitude, and launch date. It is recommended that preliminary investigations be conducted to determine the effectiveness of a standoff barrier in protecting the filament-wound vessel at optimum standoff conditions. A cursory study should be undertaken to determine the best standoff distance before the final design is prepared for the outer shell of this experiment.

The thermal environment encountered during geocentric orbit is also considered as a space-environment parameter. It is unusual, as compared with conventional atmospheric standards, in that heat received by the vehicle is entirely transferred by radiation. This heat originates from several sources, the most important being solar radiation, earth albedo radiation, and earth radiation. Other sources (e.g., planetary and galactic radiation) provide relatively insignificant amounts of thermal radiation to an orbiting satellite in comparison.

The heat input to an orbiting satellite from all these sources will vary with the orbit. Maximum thermal radiation would be realized in a polar orbit by continual exposure to solar radiation. Minimum thermal radiation would be realized during an orbit in the plane of the ecliptic. A vehicle orbiting in the ecliptic plane would realize the most extreme temperature variations, but a vehicle in a polar orbit could be maintained at a nearly constant temperature.

III. SUBSYSTEM ANALYSIS

A. PURPOSE

This preliminary analysis was undertaken to evaluate each major subsystem and its importance in attaining the mission objectives. The instrumentation is considered to be of primary importance. Consideration must be given to the selection of the various transducers to ensure sufficiently accurate data and to permit propellant-tank evaluation.

Pressurization provides a realistic application as well as a functional system. It is realistic to assume that the tank must be continually pressurized; the difficulty, however, lies in establishing reasonable pressure limits.

A thermal-control system is necessary in a polar orbit to reduce the average bulk temperature of the propellant; for an orbit in the ecliptic plane, the temperature fluctuations in the propellant tank must be minimized to within tolerable limits.

B. MISSION ANALYSIS

The primary objective of the space experiment is to evaluate the propellant tank for long-term space storage; the interrelationships between the effect of space-environment parameters and the operation and storage of the propellant vessel are also of considerable design importance. Important parameters in propellant-vessel evaluation include the following:

1. Tank-wall strain on the metallic liner and uniaxially along the filament wrap will be determined with strain gages.

2. Resistance to failure at points of stress concentration - e.g., at the seam welds on the liner - can be determined directly by means of resistance-wire crack detectors.

3. Temperatures at various tank locations and in the propellant will be obtained with thermistors.

4. The propellant-tank pressure will be sensed with potentiometer-type pressure transducers.

5. The propellant-storage efficiency (i.e., the ratio of the total propellant delivered after 1 year of storage to the total propellant received) will be determined from flow-rate measurements and original total propellant mass.

It is desirable to provide capabilities for determining possible effects of propellant-tank exposure to the space environment. Some of these effects and means by which they will be evaluated are considered below.

Vacuum exposure can cause resin sublimation, the most important result of which may be decreased resin-system strength. This type of degradation can be detected by means of changes in strain at various tank locations.

Vacuum exposure might also cause propellant to permeate through the tank wall; this can be detected directly with the crack detectors.

Electromagnetic and particle radiation could cause resin embrittlement. The mechanical-property changes that would result may be detectable by strain measurements.

Micrometeoroids may penetrate the wall of the propellant tank. Such impacts may be detectable through fluctuations in the measurement of strain on the tank liner. Vaporization and shock-wave phenomena within the propellant could also occur and cause characteristic pressure fluctuations; these would be detected by the propellant-tank pressure transducer.

Characteristic changes caused by simulated space-environment exposure can be determined in control tests prior to the launch. Such tests would also provide data for comparison with data from the orbiting satellite.

C. PRESSURIZATION SYSTEM

Two types of pressurization systems were considered for this design: (1) an inert-stored-gas system, and (2) a vapor-pressurized system. Other types (e.g., an evaporated nonpropellant or combustion-product pressurization) were not considered applicable.

These systems were analyzed thoroughly. Because propellant-tank insulation would provide protection from space-environment hazards, the power requirements for a vapor-pressurized system were considered prohibitively high for an insulated tank. For a stored-gas pressurization system, helium provides the desirable properties of low molecular weight, inertness, and ease of handling. The addition of a heater to the propellant tank would make it possible to heat the propellant during the shaded portion of each orbit and thus reduce (or eliminate) a venting requirement for high-altitude and high-inclination orbits. It was assumed for this experiment that a duty cycle of the order of ten separate expulsions would be adequate, and that the pressurization rate for pressurizing gas would be low.

IV. DESIGN OF EXPERIMENT

A. SYSTEM CONSIDERATIONS

The telemetry system and associated components related to the satellite space experiment are described below. Because a specific vehicle for this experiment was not specified, certain aspects of the telemetry system had to be established. The guidelines assumed for this design included the following:

1. Power will be supplied at 28 ± 2 v,dc, with a maximum current of 5 amp.

2. Multiplexing will be supplied with the telemetry system.

3. The instrumentation will be impedance-matched to the telemetry system after selection of a telemetry system.

4. Requirements for a storage battery and the necessary switching circuitry will be established after telemetry-system selection.

The configuration used was adopted because of the expectation that the experiment will be a piggybacked space satellite. It was necessary to consider possible problems of attachment to the main satellite. The most desirable shape for the system, based on heat-transfer considerations, is a sphere, but this shape is mechanically difficult to contain. The next best is the proposed cylinder, which also has a realistic shape for this application because propulsion-system propellant tankage is commonly cylindrical.

Several satellite power supplies and telemetry systems were reviewed and used in defining a typical system on which this design would be required to operate. Some of the more pertinent sources were References II-1 through II-9; in particular, References II-1, II-2, and II-3 were considered typical for this design application.

An estimated overall system weight for this experiment is 25 lb. Table 29 presents a summary of weights for the component parts and probable materials of construction.

B. SUBSYSTEM DESIGNS

1. Instrumentation

The instrumentation is considered the most important subsystem for this design. Redundant instrumentation is employed for the measurement of parameters considered critical to a successful mission. Figure 39 presents a typical schematic diagram for the transmission of all measurements on five separate telemetry channels. The discussion that follows describes the transducers, their functions, their particular uses in obtaining the desired data, and reasons for their selection. Table 30 summarizes the transducer requirements.

a. Temperature

Temperature is considered a primary measurement, and redundancy is used in obtaining each data point. A direct readout of voltage between 0 and 5 v,dc can be obtained without amplification by using resistance-type temperature transducers of a sufficiently high resistance. Thermistors meet this requirement and matched pairs of thermistors make close correlation of the data possible.

b. Pressure

Redundancy is also provided here, because of the importance of this measurement in determining the operational characteristics of the propellant tank. It is desirable to use a transducer having a sufficiently high

TABLE 29
COMPONENTS AND TYPICAL WEIGHTS

Component	Material	Dry Weight lb
Propellant valve	Stainless steel	0.2
Flowmeter	Stainless steel	0.1
Propellant-tank manifolds	347 SS	5.3
Heater	Stainless steel	0.1
Propellant tank (empty)	347 SS liner, glass-filament overlap	2.0
Lines (total)	347 SS	2.5
Relief valve	Stainless steel	0.2
Pressure regulator	Stainless steel	1.0
Pressurization manifold	Aluminum	0.1
Helium sphere	410 SS	1.3
Propellant-tank mount	Aluminum	0.2
Pressurization-system mount	Aluminum	0.6
Instrumentation-component mount	Aluminum	0.4
Pressure transducers (total)	Stainless-steel	1.0
Accelerometers (total)	Stainless steel	0.1
Structural frame	Aluminum	2.7
Skin	Magnesium	2.0
Check valves (total)	Stainless steel	0.2
Miscellaneous fasteners and electronic components	-	5.0
	Total	25.0

SK-6232-103

NOTES

1. LINE DENOTED BY "S" INDICATES POWER SWITCH SUPPLIES SYNCHRONOUS COMMAND SIGNAL FOR ALL SIGNAL MULTIPLEXER CHANNELS.
2. TRANSDUCER CIRCUITS ARE TURNED ON PRIOR TO TELEMETERING AND TURNED OFF AFTER TELEMETERING.
3. FLOWMETER #27 IS TELEMETERED IN SEQUENCE BY CHANNEL 1(F) AND BY CHANNEL 3(G), BUT BY COMMAND TO POWER SWITCH MULTIPLEXER CAN BE TRANSMITTED CONTINUOUSLY ON 1(F) AND 3(G) TO THE EXCLUSION OF ALL OTHER TRANSDUCERS.
4. DIODES IN FLOW METER #27 PERMIT REDUNDANT TELEMETERING OF FLOW WITHOUT OTHER INTERACTIONS.

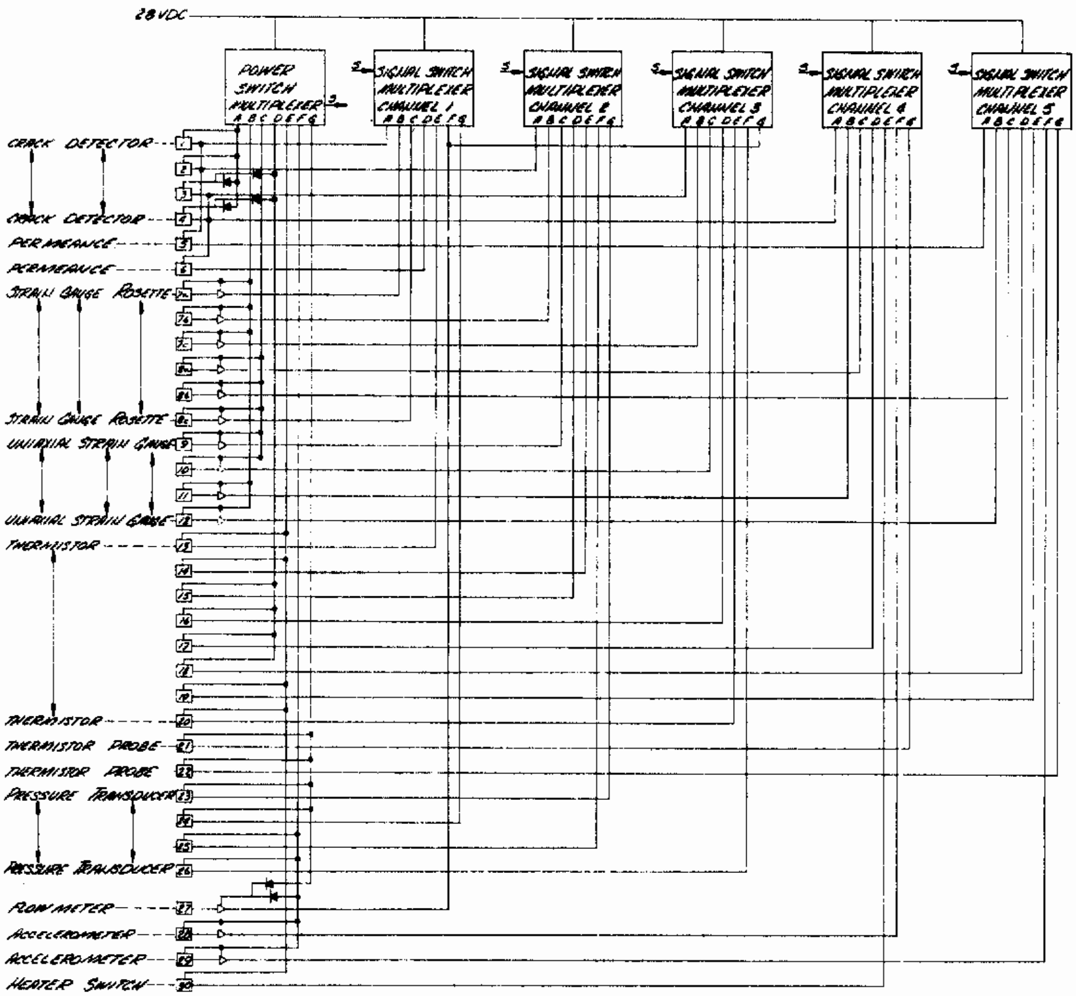


Figure 39. Electrical Schematic Diagram

TABLE 30

INSTRUMENTATION REQUIREMENTS

Parameter Measured	Type of Transducer	Configuration	Range	Output	Amplifier Required?	System Accuracy	Remarks
Wall temperature	Thermistor with negative temperature coefficient	Washer	0 to 100°F	0 to 5 v,dc	No	Supply to telemetry an output accuracy of ±1°F	Minimum slope 2%/°F, negative from 30 to 100°F Time constant 1 to 10 sec
Propellant temperature	Probe - thermistor with negative temperature coefficient	Probe with bead thermistor	30 to 100°F	0 to 5 v,dc in region of interest	No	Same	Same
Propellant-tank pressure	Potentiometer	Pressure cell	0 to 200 psia	Same	No	2% maximum system error, 1% repeatability	-
Pressurant-supply pressure	Potentiometer	Helical Bourdon tube	0 to 4000 psia	Same	No	Same	-
Triaxial strain	Wire or foil	Δ or Y	3000 μin./in. minimum	Millivolt	Yes	Compensate between 30 and 100°F	Strain gage used with special terminal to accommodate filament winding
Uniaxial strain	Wire or semiconductor	Uniaxial	Same	Wire-millivolt semiconductor 0 to 50 v,dc	Possibly	Same	For wire transducer, special terminals to accommodate filament winding; semiconductor used on outside surface only
Liner failure	Resistance wire	(See Figure 40)	5000 μin./in. over span of 0.50 in.	0 to 5 v,dc	No	-	Crack detectors at the bosses connected in series for one output signal; by selection of resistances for each detector identification of the boss at which failure occurred is facilitated
Permeation of propellant	Same as above	(See Figures 39 and 41)	-	0 to 5 v,dc	No	-	Mount second crack detector on outer winding of tank directly above liner crack detector; provide necessary circuitry to detect changes in insulation resistance caused by permeation of propellant into resin
Propellant flow	Axial vane	-	0.5 to 5 gpm	30 to 2000 cps	Yes	Supply to telemetry an output accuracy of ±1% within 10 to 100% of rated flow	Solid-state amplifier used for telemetering
Pressurant flow	-	-	-	-	-	-	Compute from temperature and pressure; use appropriate Z
Acceleration	Unbonded strain gage	-	0 ±5 g	Millivolt	Yes	1% maximum sensitivity in cross axes	Must survive boost-phase shock and vibration

voltage output to eliminate the need for amplification. An absolute-measuring transducer is desired to providing a closed system that is virtually unaffected by pressure changes during the mission. It is also desired to have a transducer that is relatively insensitive to shock and vibration, to withstand launch conditions. These features are all provided by a transducer of the potentiometer type.

c. Strain

Strain measurement is considered a principal parameter, and redundant measurements are again used. The outputs of conventional resistive wire or foil strain gages require amplification to obtain the desired 0 to 5 v,dc output. Semiconductor gages can conveniently supply the desired output, but their brittleness limits their use. It is recommended that resistance wire or foil strain gages be used on the propellant-tank liner and in the intermediate layers of filament wrap, and that semiconductors be used on the outermost wall of the tank.

A rosette of three strain gages, in either a Δ or Y configuration, should be used on the propellant-tank liner. Uniaxial gages should be bonded to strands of the filament winding and oriented in the direction of the fibers. Because of the isotensoid structure, the exact location of the strain gage is less critical than the orientation.

d. Propellant-Tank-Liner Failure Detection

Liner integrity is considered critical to the function of this propellant tank, particularly when it is used with corrosive propellants such as ClF_3. It is important to monitor possible weaknesses in the liner material (e.g., welded joints); crack detectors placed at these locations will sense gross failure. These detectors will be made of thin resistance wires; a failure will be sensed simply as a break in the wire or an open circuit. Figure 40 shows a detail of the configuration used for this measurement.

e. Propellant-Tank-Wall Permeability

Attempts will be made to use the propellant-tank-liner crack detectors secondarily as detectors of the permeation of propellants through the tank wall. A measure of electrical conductivity through the filament wrap can be obtained by placing a second crack detector on the

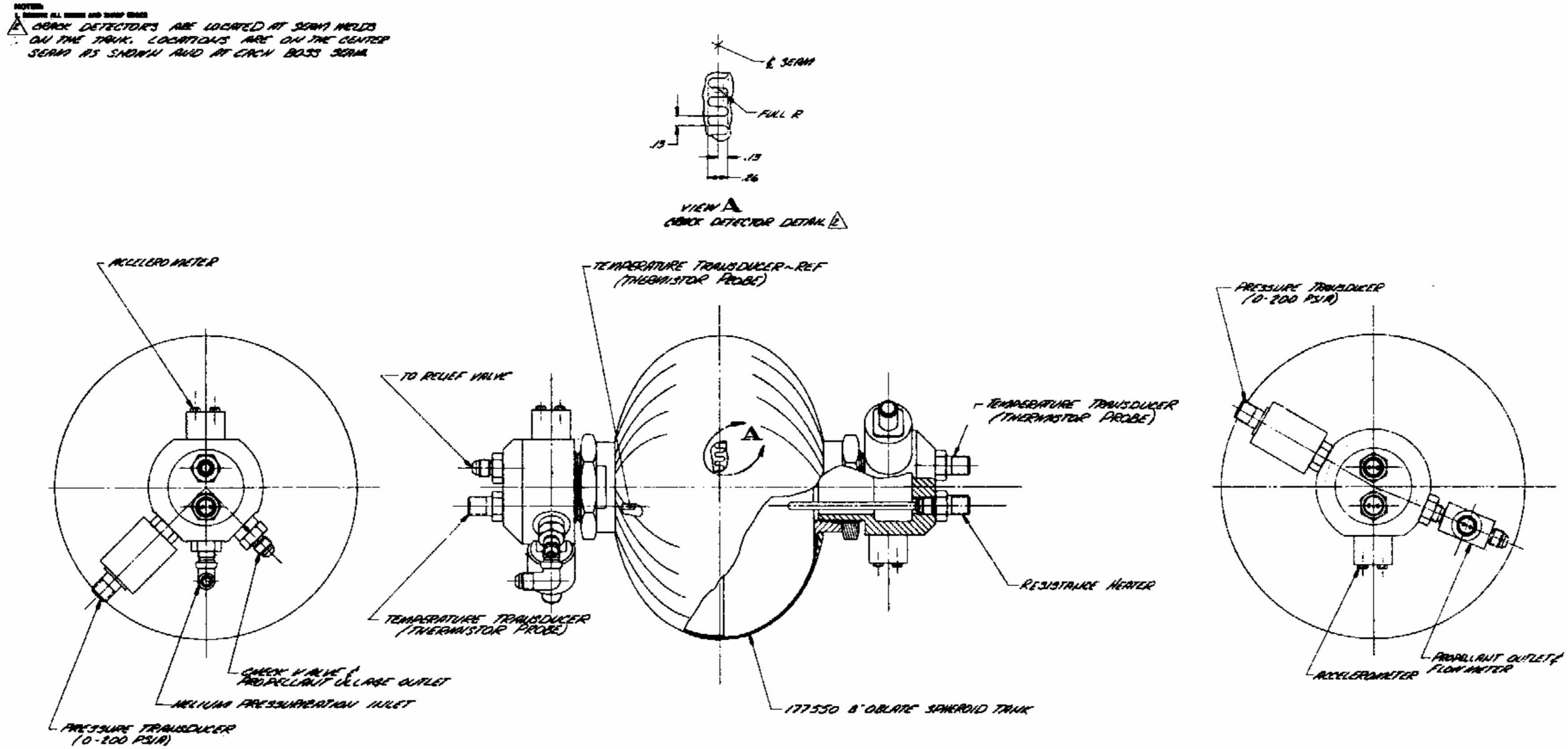


Figure 40. Propellant-Tank Assembly

outer wrap, over the crack detector on the propellant-tank liner. It is expected that this will make it possible to detect the difference in electrical resistivity resulting from trapped and absorbed propellant permeating the tank wall. Exploratory laboratory tests are recommended to evaluate the possibilities of this measurement; if it is feasible, calibration tests should also be conducted to obtain a control standard.

f. Total Propellant Mass

The total propellant mass can be determined by comparing results from several different measurements. The propellant flow rate will be measured for each propellant expulsion. If there is no venting of the propellant tank, the total volume of pressurant is accountable; consequently, the ullage volume can be determined. If a configuration is used that can ensure that the center of rotation is several inches from the propellant tank (as shown in Figure 38), the data from the accelerometers can be utilized to compute changes in propellant mass by inducing the proper tumble mode. With the satellite tumbling, the variations in acceleration can be used directly to obtain the total propellant mass.

2. Propellant-Tank and Feed-System Design

a. Pressurization System

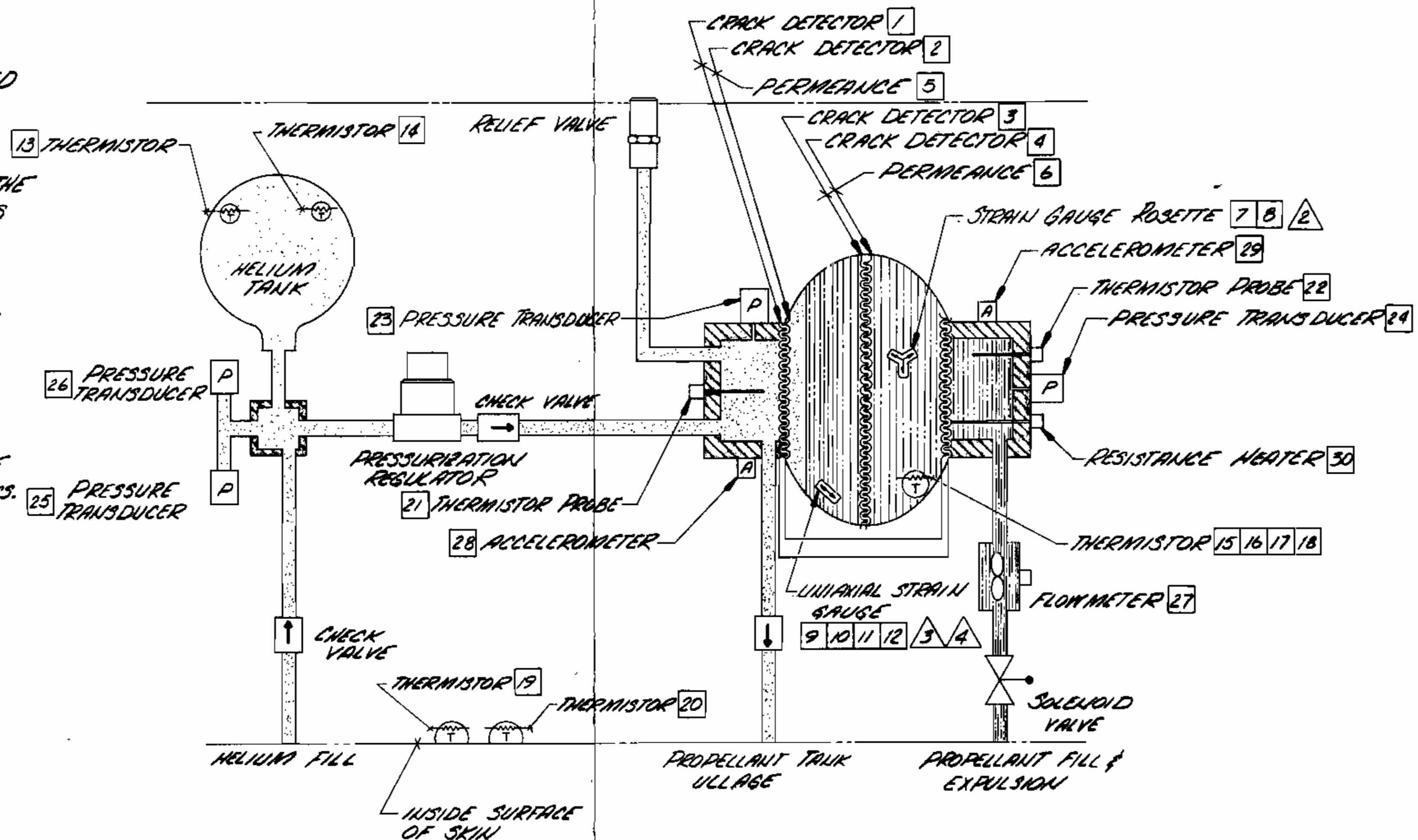
This system is presently designed to expel the selected propellant with a minimum of additional pressurant for venting. The venting requirements are based on orbit heat-transfer considerations. When a launch vehicle and a launch site have been selected, probable orbits can be determined and definitive venting requirements can be computed.

It is recommended that the final size of the pressurization sphere be determined after the venting requirements are established. Sufficient room within the vehicle skin has been allotted for the pressurization sphere. Figures 41 and 42 show present design concepts for the system, and Figures 43 shows the pressurization-sphere design.

The venting requirements can be reduced by maintaining constant control of the propellant temperature. A resistance heater (as shown in Figures 40 and 42) will be used to maintain the bulk temperature of the propellant during the shaded portion of each orbit. It will operate

NOTES: [redacted]

1. THE NUMBER CALLED OUT BY EACH TRANSDUCER REFERS TO CALLOUTS ON ELECTRICAL SCHEMATIC DWG. SK-6232-103.
2. THE STRAIN GAUGE ROSETTES ARE PLACED 180° APART, ONE ROSETTE ON EACH SECTOR OF THE LINER.
3. UNIAXIAL STRAIN GAUGES ARE ALIGNED WITH STRANDS OF THE FILAMENT WINDING. TWO STRAIN GAUGES ARE LOCATED ON THE FIRST LAYER OF WINDING 90° APART, ON SEPARATE SECTORS OF THE TANK. THE SECOND TWO STRAIN GAUGES ARE LOCATED ON STRANDS WHICH INTERSECT THE FIRST SET OF STRAIN GAUGES.
4. STRAIN GAUGES 9 AND 10 ARE LOCATED IN A SEPERATE SECTOR 90° FROM STRAIN GAUGES 11 AND 12. STRAIN GAUGE 9 AND 11 ARE LOCATED ON THE FIRST LAYER OF FILAMENT WINDING. STRAIN GAUGE 10 OR 12 ARE LOCATED ABOVE STRAIN GAUGE 9 OR 11 ON SUCEEDING FILAMENT WINDINGS.



REVISIONS				
ZONE	LTR	DESCRIPTION	DATE	APPROVED

QTY REQD	SYM	CODE IDENT	PART OR IDENTIFYING NO.	NOMENCLATURE OR DESCRIPTION	MATERIAL	SPECIFICATION	UNIT WT.	ZONE	ITEM NO.

LIST OF MATERIALS

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES TOLERANCE ON DECIMALS .XX ± .03 .XXX ± .010 ANGULAR ± 2° DO NOT SCALE DRAWING

TREATMENT

FINISH

SIMILAR TO | ACT.WT | CALC WT

ON | THRU | EFFECTIVE SERIAL NO. | USAGE DATA | NEXT ASSY | USED ON | APPLICATION | DRAWING LEVEL

CONTRACT NO. / DRAWN Nakamura 2-23-65 / CHECK / DESIGN 2-23-65 / STRESS / STRUCTURE / MATERIALS / PRODUCTION / DESIGN ACTIVITY APPD / CUSTOMER

AEROJET-GENERAL CORPORATION AZUSA, CALIFORNIA

TITLE FLOW DIAGRAM, TANK EXPERIMENT

DWG SIZE D | CODE IDENT NO. 70143 | DWG NO. SK-6232-104

SCALE | RELEASE DATE | SHEET

Figure 41. Flow Diagram, Tank Experiment

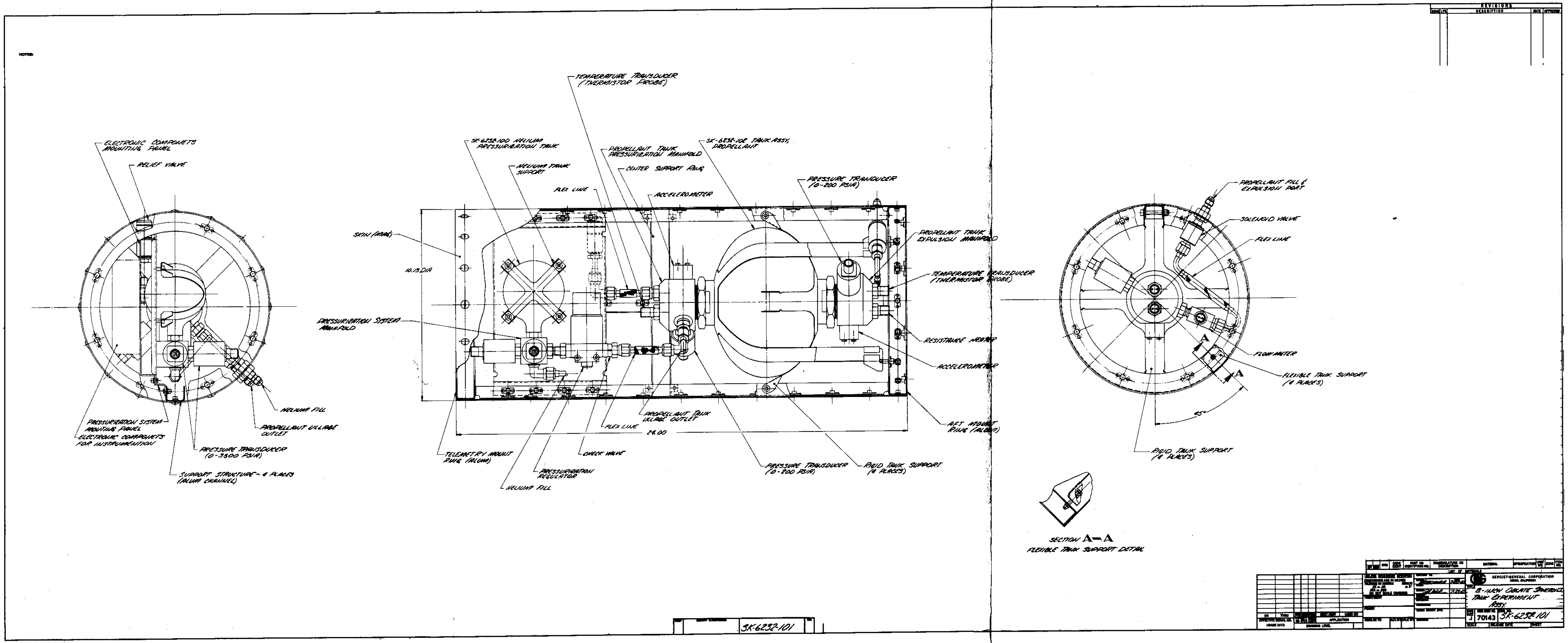


Figure 42. Oblate Spheroid (8-in.), Tank-Experiment Assembly

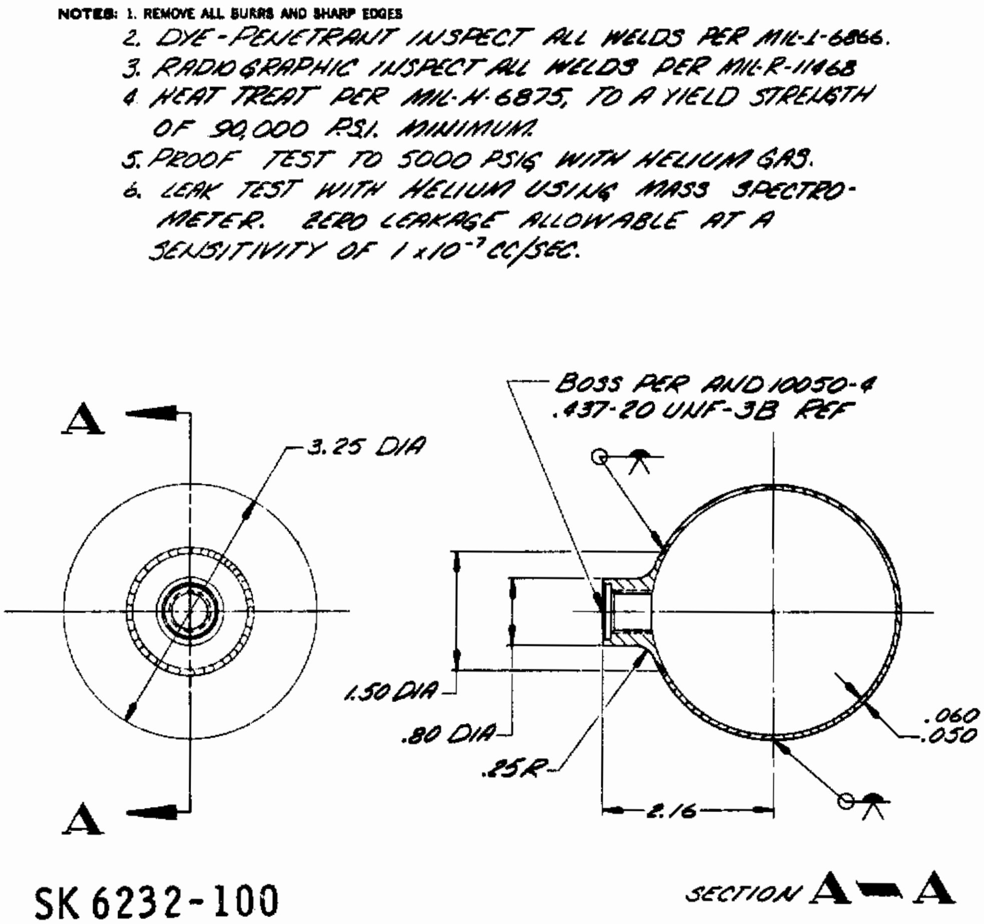


Figure 43. Tank-Pressurization System

from power supplied by a storage battery, and will be controlled by propellant-tank pressure with a control switch from the pressure regulator to prevent heater operation during pressurization. Battery current will be monitored; the heater will be switched off when the current reaches a minimum level (based on the recharge capability of the solar cells for the orbit of interest). The heater will operate when the voltage output of the propellant-tank pressure transducer reaches a minimum level and the pressure regulator is not in operation. It will shut off when the voltage output of the propellant-tank pressure transducer reaches a specified upper limit or when the battery has been discharged sufficiently. Figure 44 shows the pressure ranges of interest graphically.

The center of mass of the experimental system will be determined after final selection of the components. The requirements of the pressure regulator and check valve are given in Tables 31 and 32.

b. Expulsion System

The expulsion system is typical of those used with propulsion system of comparable size. A 25-lb-thrust bipropellant system using either N_2O_4 or ClF_3 as oxidizer with AeroZINE 50 as fuel was employed in computing the size and expected flow rates. The expected duty cycle is of the order of ten cycles. A solenoid valve operated by a command signal seems most practical for this application. Specifications for the valve required to meet all mission requirements are presented in Table 33.

c. Vent System

The vent system is expected to be self actuating. Its successful operation is based on the functioning of the relief valve, for which specifications are presented in Table 34.

3. Thermal Control

Satellite temperature control depends on well known principles of heat transfer. For this application, it is imperative that the temperature-control system maintain the propellant bulk temperature within the saturation-temperature range. An equilibrium skin temperature of $80^{\circ}F$ ($540^{\circ}R$) was assumed for this analysis, because the satellite will be tumbling and the high thermal conductivity of the magnesium skin would tend to equalize the skin temperature.

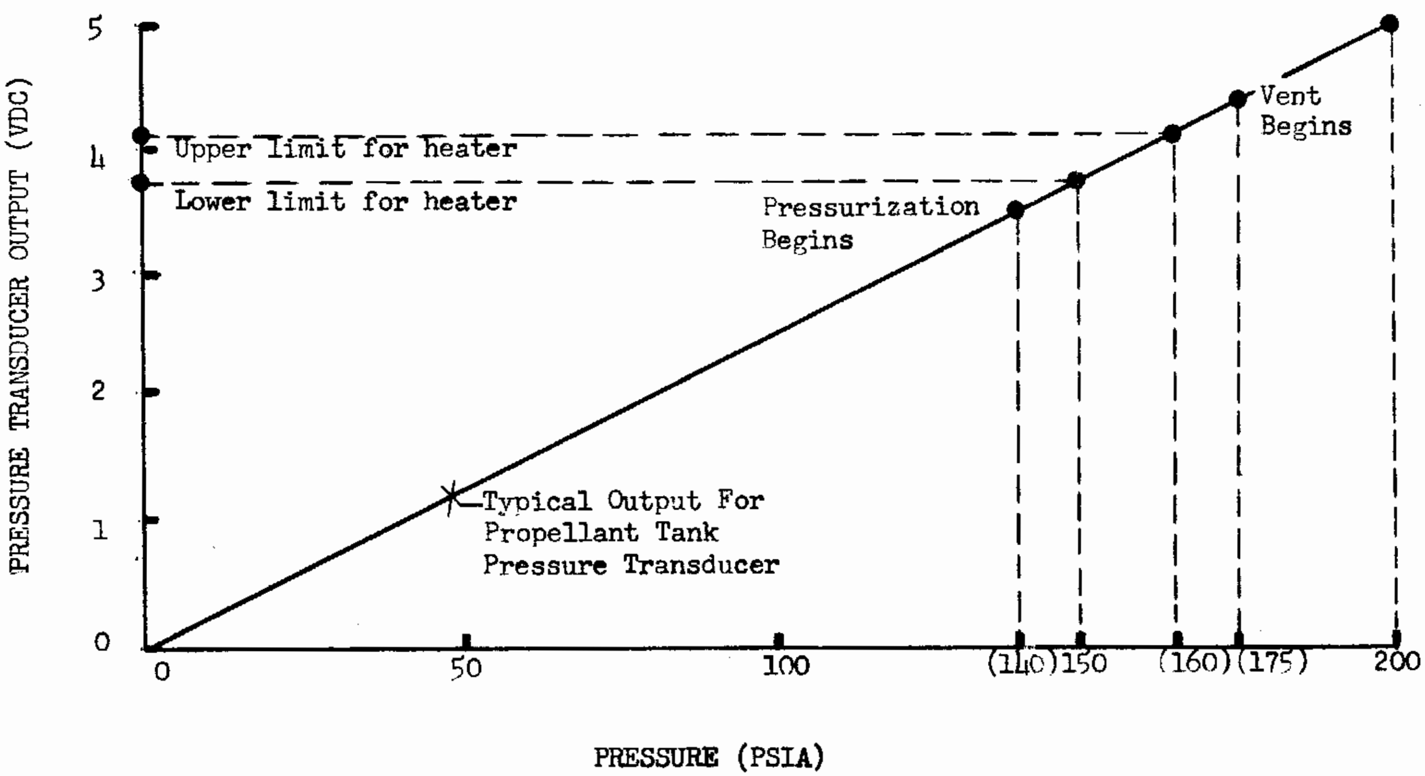


Figure 44. Typical Control Limits for Propellant-Tank Pressure

TABLE 31
TANK PRESSURE REGULATOR, SPECIFICATIONS

Type	Spring-loaded, normally open
Ports	1/4-in. line size
Pressure rating	
Inlet	3000 psi
Outlet	Regulator 150 $\pm$10 psi
Proof	4500 psi, inlet and outlet ports
Burst	7500 psi, minimum
Use	Helium
Flow rate	0.00012 lb/sec at 30°F and 3000 psia
Operating temperature	0 to 400°F
Material	
Body	Aluminum alloy
Seals	Butyl rubber

TABLE 32
PRESSURANT CHECK VALVE, SPECIFICATIONS

Type	Spring-loaded poppet
Ports	1/4-in. line size
Pressure rating	
Operating	150 psi
Proof	225 psi
Burst	300 psi
Use	Helium containing chlorine trifluoride vapor
Flow rate	0.00012 lb/sec helium at 30°F
Operating temperature	0 to 400°F
Performance	
Cracking pressure	5 to 10 psi
Reseat pressure	3 psi, minimum
Leakage	
External	0
Internal	0 in check direction at 5 psi desired; maximum helium leakage allowed, 1 x 10^{-7} cu cm/sec
Material	
Body	Stainless steel, 300 series
Seals	300 series SS, or copper

TABLE 33
PROPELLANT TANK DRAIN, SPECIFICATIONS

Type	Solenoid-operated, normally closed
Ports	1/4-in. line size
Pressure rating	
Operating	150 psi
Proof	225 psi
Burst	300 psi, minimum
Use	Chlorine trifluoride, nitrogen tetroxide, or AeroZINE 50
Flow rate	0.05 lb/sec, maximum
Operating temperature	0 to $400^{\circ}F$
Electrical data	
Voltage	28 ± 2 v,dc
Current	0.5 amp, maximum at 30 v,dc
Leakage	0 propellant leakage
Material	
Body	Stainless steel, 300 series
Seals	300 series SS, or copper

TABLE 34
RELIEF VALVE, SPECIFICATIONS

Type	Spring-referenced poppet
Ports	1/4-in. line size
Pressure rating	
Operating	150 psi
Proof	225 psi
Burst	300 psi
Use	Most severe environment is helium containing ClF_3 vapor
Flow rate	0.00012 lb/sec helium at 30°F and 150 psia
Operating temperature	0 to 400°F
Performance	
Cracking pressure	180 ±10 psi
Reseat pressure	160 psi
Leakage	
External	0
Internal	0 at 0 to 160 psi desired, maximum helium leakage allowed, 1×10^{-7} cu cm/sec
Material	
Body	Stainless steel, 300 series
Seals	300 series SS, or copper

Preliminary heat-transfer analyses of the experimental propellant-tank system indicate that the propellant bulk temperature can be maintained within desired limits of 80 $\pm 20^{\circ}$F. The skin temperature, however, will vary as much as 100°F.

The heat loads on an orbiting body arise from insolation (direct solar radiation), albedo (solar radiation reflected from the earth), and earth emission (radiation emitted from the earth). The variation of these heat loads for circular orbits is shown in Figure 45. The loads were normalized per orbit (a weighted average heat load per orbit) and were calculated as a function of altitude. (Circular orbits were assumed throughout this analysis.) Aerojet-General has a digital computer program for use in computing the heat inputs to a satellite for any circular or elliptical orbit. This program can be used to determine the variation of the skin temperature when the orbital parameters are defined. Figure 45 shows typical skin-temperature variations for an inclination of 33° of arc.

A spherical satellite with a 1-ft radius was assumed, because the experimental tank is very small. This assumption provided a symmetrical body for analysis and facilitated calculations. An analytical model for the symmetrical satellite system can be devised and analyzed with Aerojet's thermal network analyzer program when the orbital parameters are defined, the launch site is selected, and the launch date is determined.

Heat balances were determined for orbits in the polar and ecliptic planes, using assumed equilibrium skin temperatures. Average absorptivity/emissivity ratios (α/ϵ) were determined for orbital heights of 200, 1000, 9000, and 20,000 n.mi. These data are tabulated below, with the orbital periods and shade fractions (τ).

	Average α/ϵ			Shade Fractions, τ	
Altitude n.mi.	Ecliptic Orbit	Polar Orbit	Orbital Period min	Ecliptic Orbit	Polar Orbit
200	1.59	1.18	92.0	0.40	0.26
1000	1.62	1.32	123.9	0.28	0.12
9000	1.40	1.30	580.3	0.089	0.007
20,000	1.35	1.30	1500.6	0.047	0.00

An α/ϵ ratio of 1.4 was selected for the outer surface of the satellite. It satisfies requirements for the probable orbits from the Air Force Eastern Test Range. If the satellite is launched from the Air Force

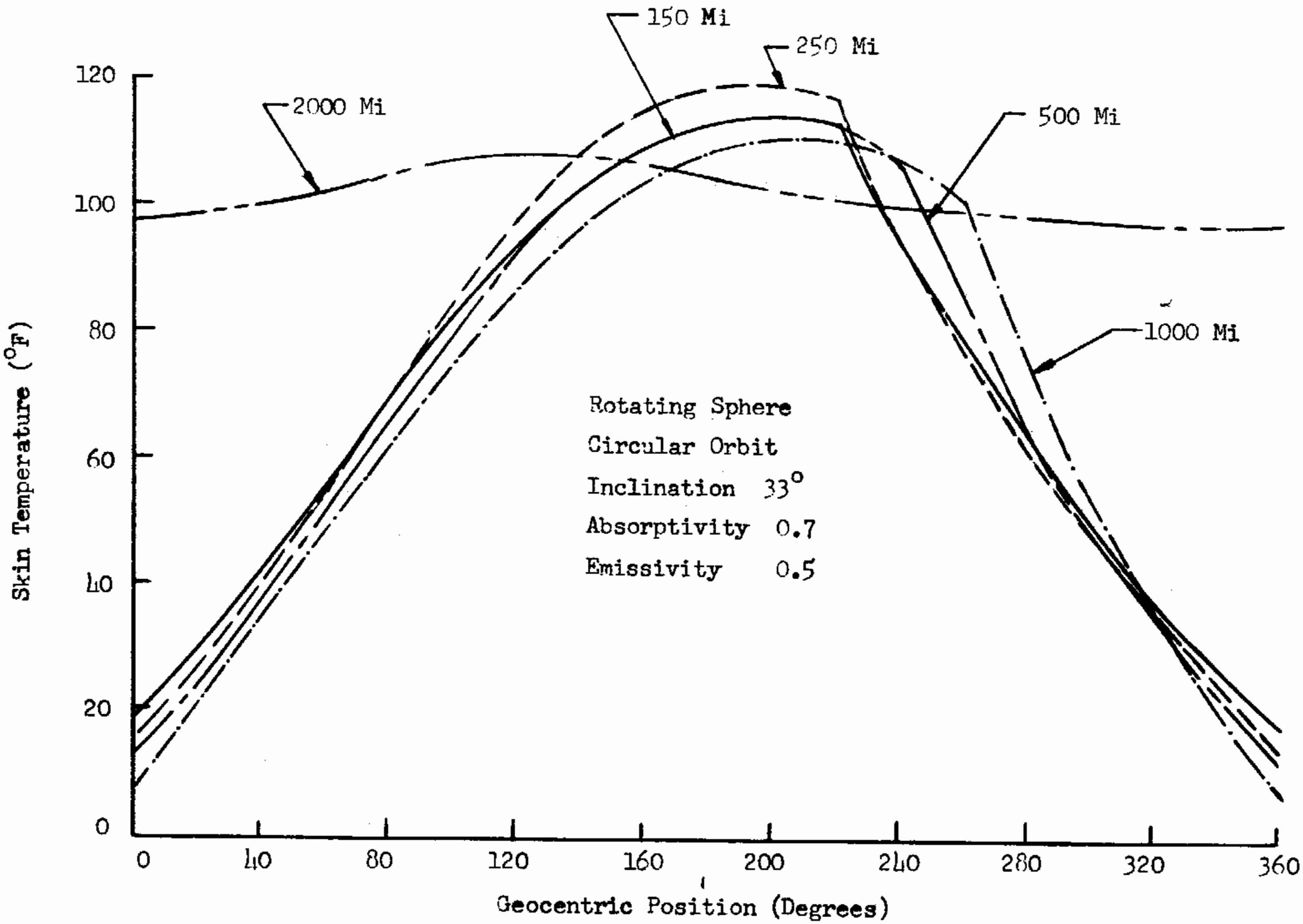


Figure 45. Skin Temperature of Rotating Sphere for Various Orbits

Western Test Range in a polar orbit, it is recommended that an α/ϵ ratio of 1.3 be used.

The radiative heat transfer from the skin to the propellant or from the tank to the skin would be less than 0.2 Btu/min. The total heat radiated away from the tank during one shade period was calculated to be approximately 15 Btu, because the maximum shade time for the orbits analyzed was 72 min. It was assumed that the conductive heat transfer was approximately equal to the radiative. Because heat will be added to the propellant by the heater during the earth-shade periods, the propellant-tank bulk temperature can be maintained nearly constant.

During the sunlit portion of each orbit, an equilibrium skin temperature will be produced by the surface coating on the skin and the incident heat flux. Because the maximum difference between the outer-skin temperature and the propellant bulk temperature will be 16°F or less, little conductive and radiative heat will be transferred to the propellant tank.

The passive and the active temperature control systems will maintain the propellant bulk temperature within a narrow range (less than 40°F), and the outer surface temperature will vary as much as 100°F.

4. Structural Design

The structural design has three major components: (a) mounting for the propellant tank, (b) mounting for the pressurization system, and (c) mounting for the instrumentation. Figure 42 shows their locations.

The propellant tank is securely mounted by metal-strap tension supports. It is cradled by these members as shown in Figure 42.

The pressurization system is mounted on the main structure by a metal plate that permits exact placement and rigid mounting of components.

The remote components of the instrumentation (e.g., resistors, diodes, and amplifiers) are mounted to a metal or glass-fiber plate that attaches to the back of the pressurization-system panel (electronic component mounting panel in Figure 42). This type of structure utilizes breadboard design concepts in which input data are received from the various transducers and are conditioned for telemetering. The output or conditioned signal is received by the telemetry from leads passing through the conduit between the telemetry and the experimental package. The input for telemetering will be taken from terminals on the back of the mounting plate.

Conventional structural-design techniques were used throughout the design of the structural members. The structure was designed to withstand the accelerations and vibrations of typical launch conditions. Its integrity is predicated on the rigid attachment of the experiment at two locations: (a) the structure of the experimental assembly, and (b) the telemetry package.

V. TYPICAL ASSEMBLY, TEST, AND LOADING PROCEDURES

This section presents typical assembly procedures necessary for the installation of the various subsystems, test procedures to confirm system operation prior to the launch, and loading procedures to ensure that the necessary precautions are observed in system servicing to prevent catastrophic failure by improper handling of highly reactive propellants. For an adequate evaluation, it is recommended that a minimum of two systems be used (with possibly a third for backup).

A. TYPICAL ASSEMBLY PROCEDURES

The procedures in tank assembly are to (1) install the tank after proof testing, with manifolds attached, (2) locate, attach, and align accelerometers, (3) install the instrumentation or manifolds, and (4) leak-check the system with helium gas and a helium mass spectrometer.

The pressurization-system assembly procedures are to (1) install the pressurization tank after proof testing, (2) install the calibrated components and transducers, and (3) leak-check the system with helium gas and a helium mass spectrometer.

The instrumentation assembly procedures are to (1) install all components for input conditioning on the electronic-component panel, (2) install the panel, (3) attach electrical leads from the transducers, and (4) check for continuity.

The structural assembly procedures are to (1) attach the outer skin to the structure, (2) install the electrical harness and leads to the telemetry system, and (3) attach the telemetry system.

B. TYPICAL TEST PROCEDURES

Typical procedures in testing are as follows:

1. Each transducer is calibrated before its installation into the system. (Whenever 100% redundancy is employed, the transducers are calibrated together and the factors needed for data correlation are determined.)

2. Each transducer is installed and its continuity is checked.

3. A preflight qualification test is conducted on the fully assembled system. The test specification should be sufficiently rigid to satisfy the requirements established by the launch vehicle. (This specification will be established when a launch vehicle has been selected.)

4. The propellant tank is filled according to accepted loading procedures. (A typical loading procedure is provided in Section V,C, following.) All transducers are pressurized and checked for continuity. The system is stored with propellant in the tank for sufficient time to ensure the compatibility of all materials.

5. A loaded system is used in a series of space-environment tests to observe the characteristics of the transducers and instrumentation. A typical duty cycle allowing complete expulsion within a 2-week period should be employed, and the tests should be conducted in a high-vacuum chamber maintained at a maximum pressure of 1×10^{-5} torr. Thermal cycling should be performed to simulate the temperature conditions of the most probable orbit to be selected.

6. All components are inspected after compatibility and simulated-environment exposure tests.

7. Modifications are made as required as a result of the compatibility and environmental tests, and the final experimental system is assembled.

8. The tank is filled with propellant, using approved loading procedures (see Section V,C, below).

9. The propellant is pressurized and expelled.

10. The system is completely purged with helium gas and is pressurized to a slightly positive pressure.

11. The system is sealed in a helium-purged polyethylene container with a slightly positive pressure, and is prepared for shipment to the launch facility.

C. TYPICAL LOADING PROCEDURE

A typical loading procedure includes the following steps:

1. All surfaces exposed to the propellant are cleaned with a suitable liquid solvent.

2. All subsystems are completely purged with gaseous helium.

3. The pressurization sphere is filled to a positive pressure of 1000 ±100 psi.

4. The pressure regulator is allowed to bleed pressure down as low as possible.

5. Sufficient gaseous ClF_3 is bled in to passivate the propellant system.* Because pressurization gas has been expelled through the check valve, the pressure regulator is protected from ClF_3 vapors.

6. The tank is filled with propellant until liquid is present at the ullage outlet.

7. The helium sphere is pressurized to 3000 (+200, -0) psi.

8. Sufficient propellant is expelled to provide 5% ullage.

VI. CONCLUSIONS AND RECOMMENDATIONS

The use of metal-lined, filament-wound, propellant vessels for long-duration space storage appears feasible, and the experimental satellite system described here seems to offer the most practical approach for their evaluation.

Certain screening and calibration tests should be conducted before such a system is launched. They include vacuum, particle-radiation-exposure, and simulated-micrometeoroid-impact studies. They are needed primarily to determine possible variations in tank-wall strain data and thereby facilitate the calibration of strain-gage response to these effects.

*Passivation is necessary only for ClF_3 and must not be performed if either N_2O_4 or AeroZINE 50 propellant is used.

It is therefore recommended that a minimum of two complete systems be built, and it is desired that components for a third system be available. It is further recommended that one complete assembly be tested in a simulated space environment to evaluate all components prior to the launch. This system should be fully disassembled and inspected before the flight-system components are finalized.

To ensure the desired operation of some of the components, such as the relief valve and expulsion system, it is necessary to establish the tumble mode of the satellite early in the flight. It is recommended that sufficient propellant be expelled during the first orbit to provide tumbling and thereby to orient the propellant.

REFERENCES

II-1. Preliminary Detail Specification Covering Flight Test Vehicle Program 11. Lockheed Aircraft Corporation Report LMSC A 051977. 12 July 1962.

II-2. Environmental Research Satellite Staff, TRW Space Technology Laboratories, A Study of Space Propulsion System Experiments Utilizing Environmental Research Satellites. STL Document 4172-6003-SU-00. Report on Phase I, 15 June 1964 - 15 August 1964.

II-3. A. Krausy and R. L. Robinson TRW Space Technology Laboratories, "The Electric Power Supply of the Orbiting Geophysical Observatory." Presented at Unmanned Spacecraft Meeting, American Institute of Aeronautics and Astronautics (AIAA), Los Angeles. March 1965.

II-4. K. M. Dawson, C. G. Cliven, and C. D. Fredrickson, National Aeronautics and Space Administration (NASA) Jet Propulsion Laboratory, Pasadena, California. "Design and Reliability Considerations for the Mariner Mars 1964 Spacecraft Power System." Presented at Unmanned Spacecraft Meeting, AIAA, Los Angeles. March 1965.

II-5. D. Schneiderman and J. N. James, NASA Jet Propulsion Laboratory, "Recent Mariner Spacecraft Missions." Presented at Unmanned Spacecraft Meeting, AIAA, Los Angeles. March 1965.

II-6. P. F. Glaser, The Orbiting Geophysical Observatory Communication and Data Handling Systems. TRW Space Technology Laboratories Report. May 1963.

II-7. Space Exploration Programs and Space Sciences. NASA Jet Propulsion Laboratory Space Program Summary No. 37-25, Vol. VI. January 1964.

II-8. The Observatory Generation of Satellites. NASA Goddard Space Flight Center SP-30. March 1963.

II-9. P. D. Gray, Rocket in Space Environment, The Design of the Space Experiment. Aerojet-General Corporation Report 2508. March 1963.

UNCLASSIFIED
Security Classification

DOCUMENT CONTROL DATA - R&D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Aerojet-General Corporation Von Karman Center Azusa, California	2a. REPORT SECURITY CLASSIFICATION Unclassified 2b. GROUP

3. REPORT TITLE

"Exploratory Evaluation of Filament-Wound Composites for Tankage of Rocket Oxidizers and Fuels"

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Summary Technical Report 1 June 1964 - 30 September 1965

5. AUTHOR(S) (Last name, first name, initial)

Sanger, M. J.
Molho, R.
Howard, W. W.

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
January 1966	192	33

8a. CONTRACT OR GRANT NO. AF 33(615)-1671 b. PROJECT NO. 7381 c. Task No. 738101 d.	9a. ORIGINATOR'S REPORT NUMBER(S) AFML-TR-65-381 9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report) Aerojet-General Report No. 3078

10. AVAILABILITY/LIMITATION NOTICES

Available to DDC. Not releasable to OTS. This document is subject to special export controls and each transmittal to foreign governments or foreign nationals may be made only with prior approval of the Air Force Materials Laboratory.

11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY AFML-RTD-AFSC-USAF Air Force Materials Laboratory Wright-Patterson AFB, Ohio

13. ABSTRACT

This work was undertaken to provide information on materials and fabrication techniques for the design of filament-wound tankage for storable propellants, and to define a space experiment for validating the recommended materials and techniques.

Resins and liner materials were studied, under accelerated exposure conditions, in the unreinforced form and as composites. Fabrication techniques were evaluated, and dynamic tests of subscale tankage were conducted.

The environmental tests indicated that the materials and fabrication techniques selected for the tankage produced satisfactory filament-wound containers for storable propellants. A technique developed for bonding a metallic liner to the filament-wound chamber provided a solution to the problem of strain compatibility under pressure cycling at optimum strain levels for glass-filament-reinforced structures.

Designs were developed for a prototype tank, and analysis showed that a spheroidal configuration with a length-to-diameter ratio of 1.0 was acceptable from the standpoint of space limitations and weight economy. A space experiment was defined for use in determining the efficiency of a filament-wound storable propellant tank over a period of 1 year in an earth-orbit environment.

DD FORM 1 JAN 64 1473 0101-807-6800

UNCLASSIFIED
Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Filament-wound tankage						
Storable propellants						
Novolac epoxy resins						
Polyepoxy resins						
Oxidizers						
Fuels						
Fluorocarbon resins						
Optimum linear-strain level						
Uniaxial cyclic-strain testing						
Pattern-type liners						
Bonded-metal liners						
Pressure-cycle tests						
Environmental exposure testing						
Prototype-tankage design						
Planned space experiment						

INSTRUCTIONS

1. ORIGINATING ACTIVITY: Enter the name and address of the contractor, subcontractor, grantee, Department of Defense activity or other organization *(corporate author)* issuing the report.

2a. REPORT SECURITY CLASSIFICATION: Enter the overall security classification of the report. Indicate whether "Restricted Data" is included. Marking is to be in accordance with appropriate security regulations.

2b. GROUP: Automatic downgrading is specified in DoD Directive 5200.10 and Armed Forces Industrial Manual. Enter the group number. Also, when applicable, show that optional markings have been used for Group 3 and Group 4 as authorized.

3. REPORT TITLE: Enter the complete report title in all capital letters. Titles in all cases should be unclassified. If a meaningful title cannot be selected without classification, show title classification in all capitals in parenthesis immediately following the title.

4. DESCRIPTIVE NOTES: If appropriate, enter the type of report, e.g., interim, progress, summary, annual, or final. Give the inclusive dates when a specific reporting period is covered.

5. AUTHOR(S): Enter the name(s) of author(s) as shown on or in the report. Enter last name, first name, middle initial. If military, show rank and branch of service. The name of the principal author is an absolute minimum requirement.

6. REPORT DATE: Enter the date of the report as day, month, year; or month, year. If more than one date appears on the report, use date of publication.

7a. TOTAL NUMBER OF PAGES: The total page count should follow normal pagination procedures, i.e., enter the number of pages containing information.

7b. NUMBER OF REFERENCES: Enter the total number of references cited in the report.

8a. CONTRACT OR GRANT NUMBER: If appropriate, enter the applicable number of the contract or grant under which the report was written.

8b, 8c, & 8d. PROJECT NUMBER: Enter the appropriate military department identification, such as project number, subproject number, system numbers, task number, etc.

9a. ORIGINATOR'S REPORT NUMBER(S): Enter the official report number by which the document will be identified and controlled by the originating activity. This number must be unique to this report.

9b. OTHER REPORT NUMBER(S): If the report has been assigned any other report numbers *(either by the originator or by the sponsor)*, also enter this number(s).

10. AVAILABILITY/LIMITATION NOTICES: Enter any limitations on further dissemination of the report, other than those imposed by security classification, using standard statements such as:

(1) "Qualified requesters may obtain copies of this report from DDC."

(2) "Foreign announcement and dissemination of this report by DDC is not authorized."

(3) "U. S. Government agencies may obtain copies of this report directly from DDC. Other qualified DDC users shall request through
_______________________________________."

(4) "U. S. military agencies may obtain copies of this report directly from DDC. Other qualified users shall request through
_______________________________________."

(5) "All distribution of this report is controlled. Qualified DDC users shall request through
_______________________________________."

If the report has been furnished to the Office of Technical Services, Department of Commerce, for sale to the public, indicate this fact and enter the price, if known.

11. SUPPLEMENTARY NOTES: Use for additional explanatory notes.

12. SPONSORING MILITARY ACTIVITY: Enter the name of the departmental project office or laboratory sponsoring *(paying for)* the research and development. Include address.

13. ABSTRACT: Enter an abstract giving a brief and factual summary of the document indicative of the report, even though it may also appear elsewhere in the body of the technical report. If additional space is required, a continuation sheet shall be attached.

It is highly desirable that the abstract of classified reports be unclassified. Each paragraph of the abstract shall end with an indication of the military security classification of the information in the paragraph, represented as *(TS), (S), (C), or (U)*.

There is no limitation on the length of the abstract. However, the suggested length is from 150 to 225 words.

14. KEY WORDS: Key words are technically meaningful terms or short phrases that characterize a report and may be used as index entries for cataloging the report. Key words must be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location, may be used as key words but will be followed by an indication of technical context. The assignment of links, roles, and weights is optional.