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DEVELOPMENT OF LIQUID OXYGEN COMPATIBLE ADHESIVE SYSTEM

by

Jose' G. Shdo
and
J. T. Hoggatt

Prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Lewis Research Center

Contract NAS 3-7952

THE BOEING COMPANY

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FINAL REPORT, PART II

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Technical Management
NASA Lewis Research Center
Cleveland, Ohio
Liquid Rocket Technology Branch
Chemical Rocket Division
R. F. Lark

THE BOEING COMPANY
Aerospace Group
Seattle, Washington

FOREWORD

This report presents the work accomplished by The Boeing Company, Space Division, under NASA Contract NAS 3-7952. The work was administered by the Lewis Research Center, Liquid Rocket Technology Branch, Chemical Rocket Division, Cleveland, Ohio, with Mr. Raymond F. Lark as program manager.

Tasks I, II and III, performed under Contract NAS 3-7952, were on the "Development of Liquid Oxygen Positive Expulsion Bladders". Task I was reported in NASA Document NASA CR-72134, January 1967 and Task II and III were reported in NASA Document NASA CR-72418 Final Report - Part I. This report, Final Report - Part II, covers the work performed from July 1968 to December 1968 under Task IV "Development of Liquid Oxygen Compatible Adhesives".

Performance of this contract was under the direction of the Materials and Processes organization, Spacecraft Mechanics and Materials Technology, Space Division of The Boeing Company. Mr. C. D. Burns was Program Supervisor, Mr. J. T. Hoggatt Program Manager, and Dr. J. G. Shdo Principal Investigator. Principal contributors to the program were:

Mr. N. J. Munsey:	Adhesive Compounding Test Sample Preparation and Testing Bladder Preparation and Testing
Mr. H. Corbin	Penetration Testing
Mr. J. Wright	Impact Sensitivity Testing
Dr. A. E. Senear	Infrared Spectra

DEVELOPMENT OF LIQUID OXYGEN COMPATIBLE ADHESIVE SYSTEM

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ABSTRACT

This report covers selection, development and evaluation of an adhesive system that is compatible with liquid oxygen and is suitable for Kapton film bladder fabrication. Initially nine adhesives were selected for further screening for bonding Kapton film for use in liquid oxygen (LOX) applications. The screening process consisted of the following: 1) adhesive bonding strength with Kapton, 2) compatibility with LOX under impact and 3) adequate flexibility at -320°F (-196°C). Kel-F elastomeric adhesive was the final selection for LOX bladder usage.

CONTENTS

LIST OF FIGURES

LIST OF TABLES

DEVELOPMENT OF LIQUID OXYGEN COMPATIBLE ADHESIVE SYSTEM

by

Jose' G. Shdo and J. T. Hoggatt

1.0 SUMMARY

Nine canaidate adhesive systems were selected and screened for LOX compatibility and adhesive characteristics at 75°F(24°C) and -320°F(-196°C). The systems evaluated were: Kel-F, Viton A, D-4375, D-4327, FEP, CNR-105, CNR-203, CNR gumstock and CRN-201, with the Kel-F elastomeric adhesive, cured with benzoyl peroxide, having the best overall strength, flexibility at -320°F(-196°C) and LOX impact compatibility. LOX compatibility was determined through both ballistic impact tests and the ABMA LOX impact sensitivity test. Adhesive flexibility was demonstrated through "twist-flex" tests at -320°F (-196°C).

A single ply Kapton bladder was fabricated with the Kel-F adhesive to demonstrate its applicability for bladder construction. The manufactured bladder was expulsion cycled 10 times at liquid nitrogen temperatures and checked for integrity. The bladder was then sectioned into test specimens for LOX ballistic penetration tests and lap shear tests of the seam areas. The bonded seams were compatible with LOX under ballistic impact and had ample lap shear strength for use in bladders, thus meeting the main objectives of this program.

2.0 INTRODUCTION

The development of reliable light-weight expulsion bladders is desirable for application in reaction control systems, multiple engine restarts, life support systems, and orbiting vehicle refueling. Feasibility of the polymeric expulsion bladder concept, and use of Mylar* and Kapton* films as bladder material were previously demonstrated (References 1 and 2) in previous tasks of this program through the preparation and evaluation of polymeric expulsion bladders for liquid oxygen (LOX) containing systems. Of the two film materials, Kapton was found to be marginal in meeting the Army Ballistic Missile Agency (ABMA) liquid oxygen impact sensitivity tests(Reference 3). Whereas Mylar reacted above 35-foot pounds of energy, Kapton reacted above 65 foot-pounds of energy. To be considered impact-insensitive in liquid oxygen a material must not discolor or detonate when impacted, under LOX, by 72 foot-pounds of energy. Although marginal, Kapton has a low impact sensitivity compared to other polymeric film materials (References 4 and 5). Bladder fabrication in the previous tasks of this contract was achieved with polyester adhesives GT 100** and GT 300**. Although excellent for cryogenic bladder fabrication, these adhesives are LOX impact sensitive (Reference 5). If used in Kapton bladders for LOX, the impact sensitivity of the bladder is reduced to the sensitivity of the ingredient of greatest sensitivity. At the onset of this final task a fully LOX compatible, flexible, commercial adhesive suitable for Kapton film bladder fabrication did not exist; therefore this six-month program was initiated to develop a LOX impact insensitive adhesive suitable for Kapton bladder fabrication and to domonstrate its feasibility by using the adhesive for fabricating a test bladder.

* E. I. duPont Tradenames

** G. T. Schjeldahl Tradename

3.0 CANDIDATE ADHESIVE SYSTEMS

At the inception of this program, a LOX compatible adhesive for Kapton* film could not be found which met all the following requirements set forth for cryogenic bladder usage.

1. Compatibility with LOX under ballistic impact and ABMA LOX impact tests of 72 ft/lb energy.
2. Ability to form strong and flexible Kapton-to-Kapton bonds capable of maintaining integrity to -320°F (-196°C).
3. Good low temperature flexibility as determined by "twist-flex" test at -320°F (-196°C).
4. Applicability for Kapton bladder manufacturing.

Reputably the Kapton polyimide surface is receptive to many classes of adhesives such as epoxides, polyamide-imides, polyesters, polyurethane and FEP fluorocarbon which is the copolymer of perfluorinated propylene and ethylene. With the exception of FEP, these classes of adhesives are not compatible with LOX (Reference 4). FEP was included in this program because the basic film is flexible at -320°F and LOX compatible. Other adhesive systems selected for evaluation which are described in detail in paragraphs 3.1 to 3.5 were Kel-F, Viton A, D-4375, D-4327 and four different carboxyl-nitroso rubbers (CNR). These systems were selected because they are halogen-containing materials which have demonstrated increased resistance toward LOX (References 4 and 6) and had the potential of meeting other basic requirements. Several other materials were checked as described at the beginning of this program but did not show promise for adequately meeting the first requirement of this program, namely good adhesion toward Kapton. Briefly the adhesive systems were: Pyre-ML and PI 1201 which are made by the E. I. duPont Company, cellulose acetate made by the Eastman Kodak Company and Epon 1045A80 which is made by the Shell Company.

*Kapton is a polyimide film manufactured by the E.I. duPont Company. Unless specified, all Kapton film used in this program was 1/2-mil thick and baked at 450°F (232°C) for 48 hours.

3.1 KEL-F ADHESIVE SYSTEM

Kel-F elastomer 5500, manufactured by the 3M Company, is the copolymer of vinylidene fluoride and chlorotrifluoroethylene. An adhesive solution was made from the basic polymer by dispersing .1 gram of the polymer in 1.0 milliliter of methyl ethyl ketone (MEK). As a curative, .01 gram of freshly purified benzoyl perioxide (Reference 7) was added to each milliliter of solution, just prior to use. In bonding the Kapton, both surfaces of the film were coated with the activated solution, joined and then cured under vacuum for 1/2 hour at 350°F (177°C).

One precaution that must be taken with this system is to allow for the evolution of benzoic acid which is one of the products of thermal decomposition of benzoyl perioxide. This acid is a hydrocarbon derivative, and therefore, LOX impact sensitive (Reference 4). Fortunately, the acid is quite volatile and rapidly sublimes with slight heating at reduced pressures. The 1/2-mil Kapton film used in this program was sufficiently thin to permit diffusion of the benzoic acid from the adhesive layer as is illustrated by the results of the LOX impact sensitivity tests (Section 5.2). Two much benzoic acid would have made the adhesive system LOX sensitive.

3.2 VITON A ADHESIVE SYSTEM

Viton A, made by the E. I. duPont Company, is the copolymer of perfluoropropylene and vinylidene fluoride. An adhesive solution was made by adding .1 gram of Viton A per milliliter of MEK with .01 gram of benzoyl peroxide added to each ml of stock solution as a curing agent. As for the Kel-F adhesive system, the curing agent was added just prior to application. The mode of application and cure conditions were the same as those described in paragraph 3.1.

3.3 D-4375 AND D-4327 ADHESIVE SYSTEMS

Dynatherm Corporation's D-4327 is a one component fluoroelastomer LOX compatible coating material now in use in areas of the Apollo space capsule (Reference 8).

D-4375 is a two component coating used to provide resistance to flame spread. The primary difference between D-4327 and D-4375 is that the latter does not have the black pigmentation and is room temperature curing.

In applying the systems, approximately a 5 mil film of adhesive was applied to both bonding surfaces of the Kapton film using a knife to form a smooth even coating. The joint was then vacuum bagged and cured at 350°F (177°C) for 1/2 hour. This resulted in adhesive layers of 3×10^{-4} inch thickness. Although the D-4375 does not require heat, a faster cure and better results were obtained by using a thermal cure.

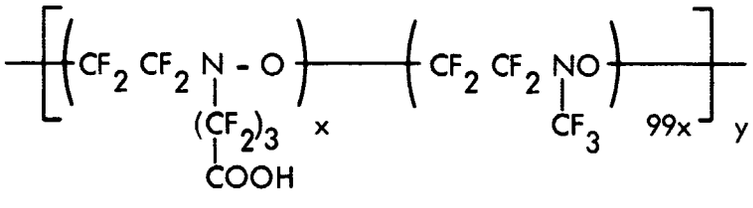
3.4 FEP TEFLON

FEP, (perfluoropropylene and tetrafluoroethylene copolymer) film made by E. I. duPont, was evaluated as an adhesive. FEP coated Kapton film is commercially available from E. I. duPont with the FEP on either one or both sides of the Kapton. The adherence between the two films is excellent but the commercial FEP coated Kapton does not have enough cryogenic flexibility for bladder usage (References 1, 6 and 12). It was the intent of this program to selectively place the FEP only in the joint region and use it as the adhesive since it is readily known that FEP can be fused together with the application of heat and pressure.

The FEP on the commercial FEP-coated Kapton is applied during initial processing by duPont using a proprietary process. Previous attempts by Boeing to apply FEP film to as-received Kapton had been unsuccessful so steps were taken to treat the FEP surfaces to promote adhesion. Prior to adhesion FEP films were surface treated through a procedure of dipping the films for 10 seconds in solution of 1% sodium metal in liquid ammonia, followed by a n-butyl alcohol rinse, then neutralizing with 10% hydrochloric acid, and finally rinsing with distilled water. The FEP to Kapton bond was made by sandwiching the FEP between the two Kapton substrate films and then heating the joint for 1/2 hour at 350°F (177°C) under vacuum bag pressure. One mil FEP was used in all tests.

3.5 CARBOXYL-NITROSO RUBBER ADHESIVES

Carboxyl-nitroso rubber (CNR), manufactured by the Thiokol Chemical Company, is a fully fluorinated elastomer with the structure shown below, and as such is highly resistant to attack by oxygen and other high strength oxidizing agents such as nitrogen tetroxide (Reference 9).



The four available forms of CNR elastomers used in this program were: CNR-105 coating solution, liquid CNR-203, CNR pressure sensitive adhesive, and CNR gum-stock. These materials, although of essentially the same polymeric structure, differ from each other in molecular weight, type of solvent, and/or curing system. The CNR elastomers can be cured with epoxides, amines, metal salts and chromium trifluoroacetate (CTA) but the curing agents used in this program were restricted to CTA or dicyclopentadiene dioxide (DPD), as recommended by the manufacturer.

3.5.1 CNR-105 COATING SOLUTION

CNR-105 coating solution was applied by the manufacturer as a suspension of low molecular weight CNR in a fluorocarbon solvent. Prior to application, CNR-105 coating solution was activated with a solution of CTA (10 parts) in tetrahydrofuran (30 parts). Two parts of the catalyst solution per hundred parts CNR-105 are used. To fabricate the adhesive bonds, the adhesive was applied to both film surfaces and vacuum bagged without allowing to air dry. Curing was at 350°F(177°C) for 1/2 hour.

3.5.2 CNR-203 LIQUID

CNR-203 is a liquid in the uncured state because the polymer is of low molecular weight. Prior to use, CNR-203 was catalyzed with 3.5 parts of fused dicyclopentadiene dioxide mp 150°F (66°C) for every 100 parts of CNR. The activated mixture has a pot life of half a hour at room temperature. For bonding Kapton films, a 1/2 hour cure at 350°F(177°C) (vacuum bag) was used.

3.5.3 CNR-GUMSTOCK

The CNR gumstock was prepared for use by making a suspension of 100 parts CNR and 5 parts CTA in trichlorotrifluoroethane. After mixing these ingredients the system was kept refrigerated to prevent excessive evaporation of the trichlorotrifluoroethane. Kapton films were bonded in a vacuum bag at 375°F (177°C) for 1/2 hour. Precaution was taken not to let the adhesive air dry prior to joining.

3.5.4 CNR-201 PRESSURE SENSITIVE ADHESIVE

CNR-201 pressure sensitive adhesive is a Thiokol Company preparation which is supplied with both the CNR rubber and curing catalyst in a fluorocarbon solvent. This material was used as received from the manufacturer. No cure is required with this material.

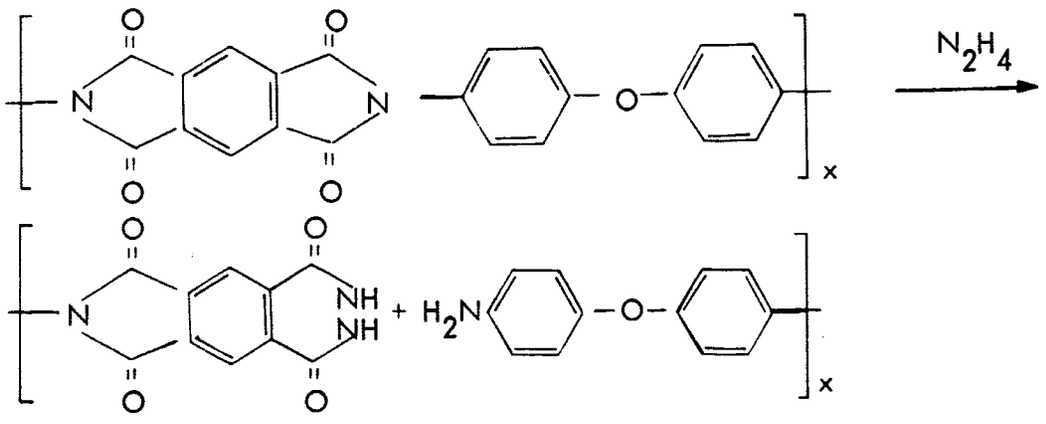
4.0 SURFACE TREATMENT

A surface treatment study for Kapton film was initiated concurrently with the basic adhesive study with the sole purpose of possibly increasing the bond strengths to Kapton film. The approach was pursued as preimplementation for future studies in the event that the adhesives selected, see paragraphs 3.1 to 3.5.4, exhibited a trend of adhesive failures, that is, failure of the adhesive to bond to Kapton. During progress of the work summarized in paragraphs 3.1 to 3.5.4 it became evident that chemical surface modifications of Kapton was not required because a few of the selected adhesives had adequate bonding strength with Kapton: at -320°F(-196°C) the Kel-F, Viton A, D-4327 and D-4375 adhesives all exhibited failures at film stress values very close to the ultimate film stress of the basic 1/2 mil Kapton film.

Three film surface treatments were investigated; 1) surface etching with ethanolic hydrazine or alc/N_2H_4; 2) surface etching with sodium metal in liquid ammonia or Na/NH_3; 3) surface etching with ethanolic potassium hydroxide or alc/KOH. Specimens surface cleaned with MEK and ethanol or MEK/alc were made for comparative purposes.

4.1 ALCOHOLIC HYDRAZINE

Kapton films were treated with a solution consisting of 1/3 part hydrazine and 2/3 parts ethanol. This treatment was based on the previously established degradation reaction of Kapton with hydrazine (Reference 6). This reaction leads to products consisting of hydrazido and amino groups as shown below for partial degradation.



Kapton films were immersed in alc/N_2H_4 for 15, 30 and 60 seconds. After alc/N_2H_4 treatment film surfaces were neutralized by rinsing with 10% HCl, then with distilled water followed by a final rinse in pure ethanol. The transmission and surface Attenuated Total Reflectance (ATR) infrared spectra (IR) of treated films were taken and compared with spectra of untreated films. The results were consistent with the expected reactions. The transmission IR of the alc/N_2H_4 treated films consisted of the ATR absorption bands of the surface superimposed on the absorption trace produced by transmission IR of the unmodified Kapton, showing the surface was modified. Lap shear test specimens, bonded with CNR pressure sensitive adhesive, were made with Kapton treated with alc/N_2H_4 at 15, 30 and 60 seconds exposure. The specimen were tested for adhesive strength at both room temperature and at -320°F (-196°C). The test results are summarized in Table I.

4.2 ALCOHOLIC POTASSIUM HYDROXIDE

Kapton film was treated with a concentrated solution of freshly prepared saturated solution of ethanolic KOH to etch andhydrolyze the surface. After treatment, the films were rinsed with 10% HCl, then rinsed with distilled water and then with pure ethanol. For IR analysis, a Kapton film was treated for 55 seconds in alc/KOH. The ATR of the alc/KOH treated Kapton film were almost identical to the transmission IR of an untreated film, the differences observed were the appearance of a very weak absorption bond in the ATR curves at 3.0 μ , 6.1 μ and 6.4 μ . These changes suggested hydrolytic cleavages of imide structure into amide-acid structures. Lap shear test specimen were made with Kapton film treated for 5, 15 and 30 seconds in alc/KOH, with CNR pressure sensitive adhesive again used as the adhesive. The lap shear strength test results are also shown in Table I.

4.3 SODIUM METAL IN LIQUID AMMONIA

Kapton film was etched with a sodium/ammonia solution which was made by placing 1 part sodium into 150 parts by wt. of liquid ammonia. Treatment of Kapton with the Na/NH_3 solution resulted in changes on the film surface which permitted it to be wetted by water, whereas unmodified Kapton does not exhibit this phenomenon.

TABLE I

LAP SHEAR STRENGTH AND STRESS OF CNR PRESSURE SENSITIVE ADHESIVE ON 1/2 MIL KAPTON (a) (b)

Treatment Description	Lap Strength(c) lb/in Width (room temp)	Film Stress (d) lb/in^2 (room temp)	Lap Strength(c) lb/in Width (liquid nitrogen)	Film Stress (d) lb/in^2 (liquid nitrogen)
As Received	3.0	6.0×10^3		
clean/MEK	5.6	1.12×10^4		
As Received	2.7	5.4×10^3	4.4	8.8×10^3
clean	2.8	5.6×10^3	6.0	1.2×10^4
MEK/alc	3.3	6.6×10^3	4.4	8.8×10^3
alc/KOH	2.5	5×10^3		
5 sec.	3.7	7.4×10^3		
	3.0	6×10^3		
alc KOH				
15 sec.			5.3	1.06×10^4
			3.0	6×10^3
alc/KOH	4.0	8×10^3		
30 sec.	3.55	7×10^3		
	2.4	4.8×10^3		
Na/NH_3	4.9	9.8×10^3		
5 sec.	3.5	7.0×10^3		
	4.2	8.4×10^3		
Na/NH_3			4.7	9.4×10^3
10 sec.			3.81	7.62×10^3
			4.64	9.28×10^3
Na/NH_3	4.8	9.6×10^3		
30 sec.	4.6	9.2×10^3		
	4.6	9.2×10^3		
alc/N_2H_4	5.6	1.12×10^4		
15 sec.	3.8	7.6×10^3		
	5.8	1.16×10^4		
alc/N_2H_4			8.0	1.6×10^4
30 sec.			6.8	1.36×10^4
			7.1	1.42×10^4
alc/N_2H_4	4.3	8.6×10^3		
60 sec.	4.4	8.8×10^3		
	4.0	8.0×10^3		

(a) The samples were tested at a head speed of 0.05 in/min.

(b) The samples were made of 1/2 mil Kapton and were 1" x 8" with a 1/2" overlap area treated with the adhesive.

(c) Lap length was 1/2 inch.

(d) Film stress at failure.

After Na/NH_3 treatment, the films were rinsed with butanol, then followed by treatment with dilute HCl, distilled water and pure ethanol. Films exposed to Na/NH_3 were not analyzed by IR. Lap shear test specimen were made with films treated for 5, 10 and 30 seconds; CNR pressure sensitive adhesive was used as the adhesive. The lap shear strength test results are also summarized in Table I.

4.4 MEK/ETHANOL (PASSIVE CLEANING)

The Kapton film surfaces were cleaned by scrubbing with clean cotton gauzes soaked with reagent grade MEK followed by scrubbing with gauze soaked with pure ethanol. Only reagent grade or pure solvents were used to avoid contamination of the film surface with low molecular weight, grease-like, hydrocarbons. Lap shear test specimens were made with the MEK/alc cleaned Kapton using CNR pressure sensitive adhesive. The test results are summarized in Table I.

5.0 ADHESIVE SCREENING TESTS AND FINAL SELECTION

Of the candidate adhesive systems listed in Section 3.0, the Kel-F adhesive was selected as the best adhesive for Kapton for further characterization for LOX bladder applications. Selection of the Kel-F adhesive system was based on the following use-oriented screening tests:

(1) Adhesive properties through lap shear bond strength at room temperature and at -320°F (-196°C).

(2) LOX impact sensitivity at 72 foot-pounds of energy.

(3) Cryogenic flexibility at -320°F (-196°C) or determined by the "twist-flex" method.

5.1 LAP SHEAR BOND STRENGTH

Adhesive properties of the adhesive systems listed in Section 3.0 were determined through lap shear bond strength at room temperature and at -320°F (-196°C). The test results are shown in Table 2 and are listed below in ranking order of increasing average film stress at failure at -320°F (-196°C):

Number	Adhesive System	Average Film Stress (lb/in^2) at Failure at -320°F
1	CNR-201 (pressure sensitive)	5.2×10^3
2	CNR-(gumstock)	15.7×10^3
3	CNR-105 (coating solution)	15.9×10^3
4	FEP	19.1×10^3
5	CNR-203 (Liquid)	19.9×10^3
6	KEL-F	27.6×10^3
7	Dynatherm D-4327	29.1×10^3
8	Viton A	35.3×10^3
9	Dynatherm D-4375	35.4×10^3

TABLE 2

LAP SHEAR STRENGTH AND STRESS OF ADHESIVES FOR BONDING KAPTON FILMS (1, 2)

Test Temperature	Room Temperature (77° ± 5°F)				Liquid Nitrogen (-320°F)			
Adhesive System	Lap Strength(3) lb/in width	Film Stress (4) lb/in^2	Adhesive thickness (in.)	Mode of Failure(5)	Lap Strength(3) lb/in width	Film Stress(4) lb/in^2	Adhesive thickness (in.)	Mode of Failure(5)
CNR-201 (pressure sensitive)	0.95	1.9×10^3	5×10^{-4}	a	2.62	5.2×10^3	5×10^{-4}	a
	1.28	2.6×10^3	5×10^{-4}	a	2.68	5.2×10^3	5×10^{-4}	a
	0.96	1.9×10^3	5×10^{-4}	a	2.60	5.2×10^3	5×10^{-4}	a
CNR (gumstock in solution)	3.40	6.8×10^3	2×10^{-4}	a	6.30	12.6×10^3	2×10^{-4}	f
	3.64	7.2×10^3	2×10^{-4}	a	8.40	16.8×10^3	2×10^{-4}	f
	3.42	6.8×10^3	2×10^{-4}	a	8.78	17.6×10^3	2×10^{-4}	f
CNR-203 (Liquid)	6.82	13.6×10^3	5×10^{-4}	a	12.96	26.0×10^3	5×10^{-4}	f
	7.40	14.8×10^3	5×10^{-4}	a	8.23	16.5×10^3	5×10^{-4}	f
	8.66	17.3×10^3	6×10^{-4}	a	8.52	17.0×10^3	6×10^{-4}	f
CNR-105 (coating solution)	5.48	10.9×10^3	2×10^{-4}	a	8.93	17.8×10^3	3×10^{-4}	f
	6.22	12.4×10^3	2×10^{-4}	a	8.72	13.4×10^3	3×10^{-4}	a
	6.60	13.2×10^3	2×10^{-4}	a	8.22	16.4×10^3	3×10^{-4}	a
Viton -A	9.62	19.3×10^3	3×10^{-4}	a	15.65	31.3×10^3	3×10^{-4}	a
	8.56	17.1×10^3	3×10^{-4}	f	13.06	26.1×10^3	3×10^{-4}	f
	11.79	23.5×10^3	3×10^{-4}	f	14.25	28.5×10^3	1×10^{-4}	f
Kel-F	11.72	23.4×10^3	2×10^{-4}	f	12.85	25.8×10^3	6×10^{-4}	a
	11.18	22.2×10^3	2×10^{-4}	f	14.76	29.5×10^3	6×10^{-4}	f
	9.74	19.5×10^3	2×10^{-4}	f	13.53	27.5×10^3	6×10^{-4}	f
FEP	5.53	11.8×10^3	6×10^{-4}	a	12.24	24.5×10^3	6×10^{-4}	a
	7.77	15.6×10^3	6×10^{-4}	a	6.78	13.4×10^3	6×10^{-4}	a
	6.12	12.2×10^3	6×10^{-4}	a	9.60	19.4×10^3	6×10^{-4}	f

TABLE 2 (continued)

Test Temperature	Room Temperature (77° ± 5°F)				Liquid Nitrogen (-320°F)			
Adhesive System	Lap Strength(3) lb/in width	Film Stress(4) lb/in^2	Adhesive thickness (in.)	Mode of Failure(5)	Lap Strength (3) lb/in width	Film Stress(4) lb/in^2	Adhesive thickness (in.)	Mode of Failure(5)
Dynatherm	10.08	20.2×10^3	4×10^{-4}	a	6.12	12.2×10^3	4×10^{-4}	f
D-4327 (old	9.53	19.0×10^3	4×10^{-4}	f	11.31	22.6×10^3	4×10^{-4}	f
test setup) (6)	10.22	20.5×10^3	4×10^{-4}	f				
Dynatherm	9.22	18.4×10^3	3×10^{-4}	f/a	19.94	40.0×10^3	3×10^{-4}	f/a
D-4375 (new test	10.24	20.5×10^3	3×10^{-4}	f/a	18.86	37.6×10^3	3×10^{-4}	f/a
setup) (6)	9.52	19.0×10^3	3×10^{-4}	f/a	14.39	28.6×10^3	3×10^{-4}	f/a
D-4327 (new test					14.73	29.5×10^3	6×10^{-4}	a
setup) (6)(7)					14.17	28.4×10^3	8×10^{-4}	f
					14.76	29.5×10^3	6×10^{-4}	f

(1) The test samples were made of as received 1/2 mil Kapton and were 1" x 8" with a 1/2" overlap area bonded with the adhesive.
(2) The samples were tested at a head speed of 0.05 in/min.
(3) Lap length was 1/2 inch.
(4) Film stress at failure.
(5) The observed modes of failure and their designations are: a = adhesive failure at the overlap area
f = film failure
f/a = film failure and adhesive failure at the overlap area itself.
(6) Sample holder jaws were modified to prevent excessive film slippage during testing. The modification consisted of two additional pairs of screw clamps placed in such a manner as to impose greater pressure on the samples at the jaws.
(7) Repeated to compare values obtained with the old set up. The values, although different were within the same order of magnitude as obtained with the old setup.

Film failure rather than adhesive failure was the predominant mode of failure at -320°F as is evident by the data. Failures at film stress values below 35×10^3 lb/in^2 (the average ultimate tensile strength of the 1/2 mil Kapton) were attributed to either excessive abrasion from film slippage at the sample holding grips or to stress concentrations due to high temperature gradients at the site of film immersion into liquid nitrogen. The test set-up was changed to minimize these stress concentrations as indicated at the bottom of Table 2.

For ease in handling, the lap shear test specimen were prepared by bonding two pieces of 4-1/4" x 6" Kapton with a 1/2" lengthwise overlap. After curing the adhesive, the resulting material was cut with a sharp knife into six 1" x 8" lap shear test samples. To minimize film surface contamination all material handling, cutting and bonding operations, were conducted under clean room conditions.

5.2 LOX IMPACT SENSITIVITY TESTS

LOX impact sensitivity tests were conducted on all of the adhesive systems listed in paragraphs 3.1 through 3.5.4 save for the CNR gumstock, the CNR coating solution and the FEP adhesives.

The CNR gumstock, coating CNR-105 solution and FEP adhesives were excluded from the LOX impact sensitivity tests because of their low lap shear bonding strength with Kapton at -320°F (-196°C).

Although the pressure sensitive CNR-201 adhesive had the lowest lap shear strength in bonding Kapton, this adhesive nevertheless was tested for LOX impact sensitivity to determine if the mode of adhesive application had a strong influence on LOX sensitivity. Pressure sensitive CNR-201 adhesive is pre-catalyzed by the manufacturer, cures at room temperature and does not use heat for removal of volatile and curative by products. These parameters differ from all the other candidate systems.

The LOX impact sensitivity test results are summarized in Table 3 and as the results show all six adhesive systems in a pure form exhibited compatibility with LOX. When tested

TABLE 3

LOX COMPATIBILITY IMPACT SENSITIVITY TEST DATA

Sample	No.* Tests	Reaction or Flash	Reaction On Rebound
As-Received Kapton	20	5	0
Baked Kapton	20	1	0
Kapton and D-4375	20	0	1
Pure D-4375 Adhesive	20	0	0
Kapton and D-4327	20	0	0
Pure D-4327 Adhesive	20	0	1
Kapton and Viton A	20	4	0
Pure Viton A Adhesive	20	0	2
Kapton and Kel-F	20	0	0
Pure Kel-F Adhesive	20	0	1
Kapton and CRN-201 Pressure-Sensitive Adhesive	20	2	0
Pure CNR-201 Pressure-Sensitive Adhesive	20	0	0
Kapton and CNR-203 Liquid	20	3	0
Pure CNR-203 Adhesive	20	0	1

*Five blanks were tested at equal intervals during each sample run, per MSFC-SPEC-106B. Test results for the blanks are not shown in this Table because they did not react.

All drop tests conducted at 72 ft-lb of energy.

while "in-bond" with Kapton, only three adhesives, D-4375, D-4327 and Kel-F, were LOX compatible. The failures are attributed to either the base film failure or residual organic solvents possibly occluded in the adhesive layer, or both.

The equipment and support facilities were compatible with the requirements of MSFC-SPEC-106B (Reference 3). Details and photographs of the LOX impact sensitivity tester are shown in the Contract Interim Report (Reference 5). The LOX impact sensitivity specimen, containing adhesive seams, were prepared in a dust free facility equipped with a laminar flow bench (Reference 5). During preparation the specimens were handled with clean dust-free nylon gloves.

The test samples containing adhesive seams were made by bonding two 1" wide x 6" long strips together to simulate a lap joint. Use of the vacuum bagging technique for applying pressure during heat cure was found satisfactory for bonding Kapton with the selected adhesive systems. The vacuum bagging system consisted of a 4' x 5' aluminum plate with perforations leading to a vacuum pump. The specimen are placed on this plate with bleeder cloth around the specimen; the plate was covered with a sheet of flexible FEP which was secured to the table top with heat resistant double back tape. The schematic consisting of aluminum plate, specimen, bleeder cloth, etc., are shown in Figure 1. The entire system was rolled into a walk-in oven heated to the adhesive's curing temperature. The specimens for the LOX sensitivity tests, lap shear tests, twist-flex tests and ballistic impact tests were prepared by using this vacuum bag technique.

The use of platen heated hydraulic presses was investigated in the early phases of Task IV. However, this approach was not pursued when it became obvious that the adequate reproducibility of low pressures could not be obtained with either a 50 ton Pasadena Hydraulic Inc. press or a 20 ton K-M, J. P. Marsh Corp. press.

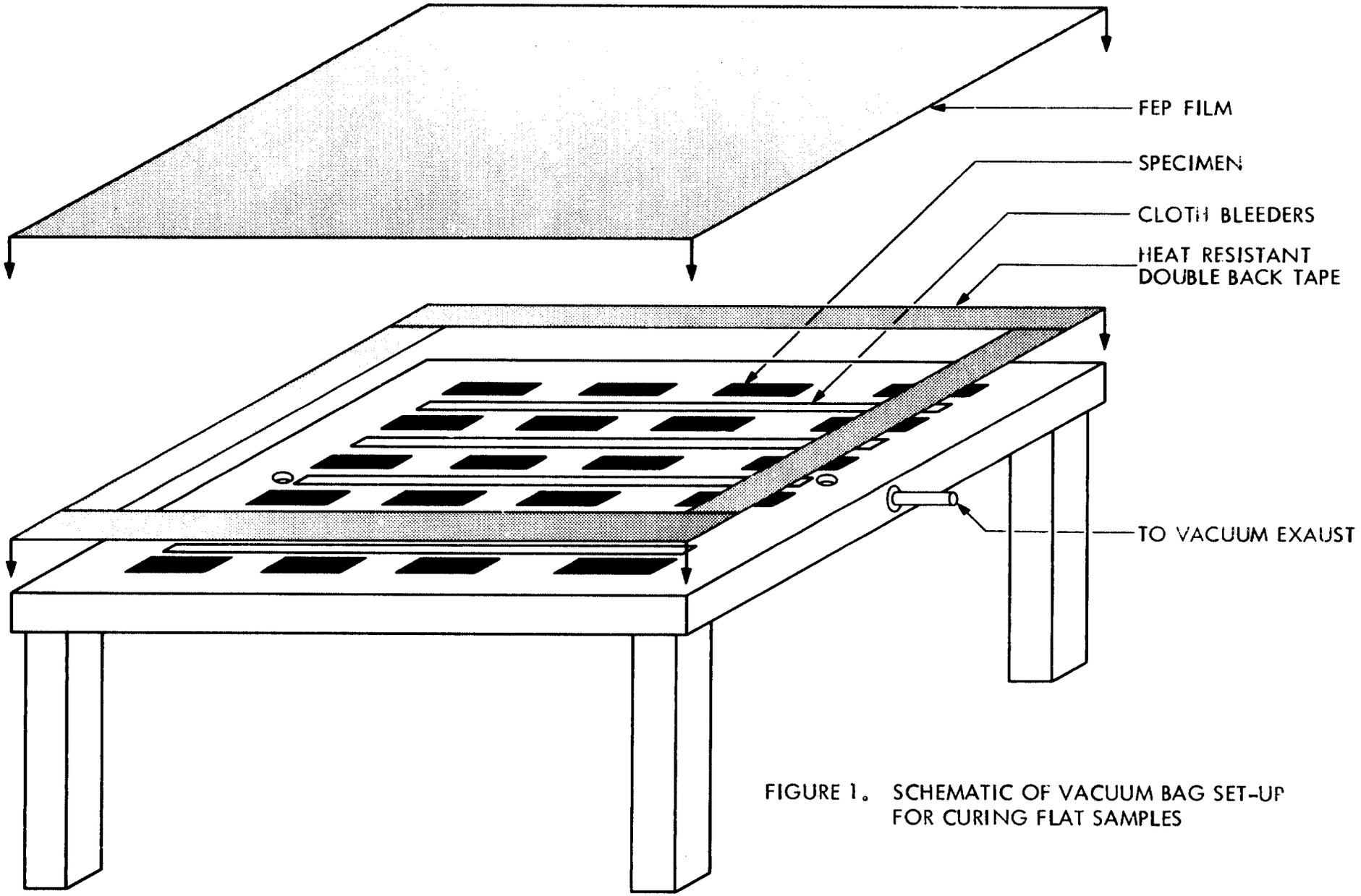


FIGURE 1. SCHEMATIC OF VACUUM BAG SET-UP FOR CURING FLAT SAMPLES

The prepared LOX impact samples were stored and transported to the Boeing Tulalip test site in sealed polyethylene bags, where the LOX samples were made by cookie-cutting 11/16" diameter discs from the strips of Kapton (1" x 6") containing the adhesive seams. The pure adhesive test specimen were cast on clean steel discs 11/16" in diameter, air dried in the laminar flow bench, then cured in a vacuum oven while protected by capped crystallizing dishes. Nitrogen gas was used to remove the vacuum to prevent contamination of the samples with atmosphere dust.

5.3 ADHESIVE LOW-TEMPERATURE FLEXIBILITY (TWIST-FLEX TESTS)

Flexibility and ability to maintain adhesive integrity at -320°F(-196°C) was used as the final basis for selecting an adhesive system for fabrication of a bladder and for LOX ballistic impact tests. The "twist-flex" test method, developed by the Beech Aircraft Corporation (References 2 and 11) under NASA sponsorship, was used to determine adhesive flexibility at -320°F(-196°C). The adhesive systems tested and twist-flex test results are shown in Table 4.

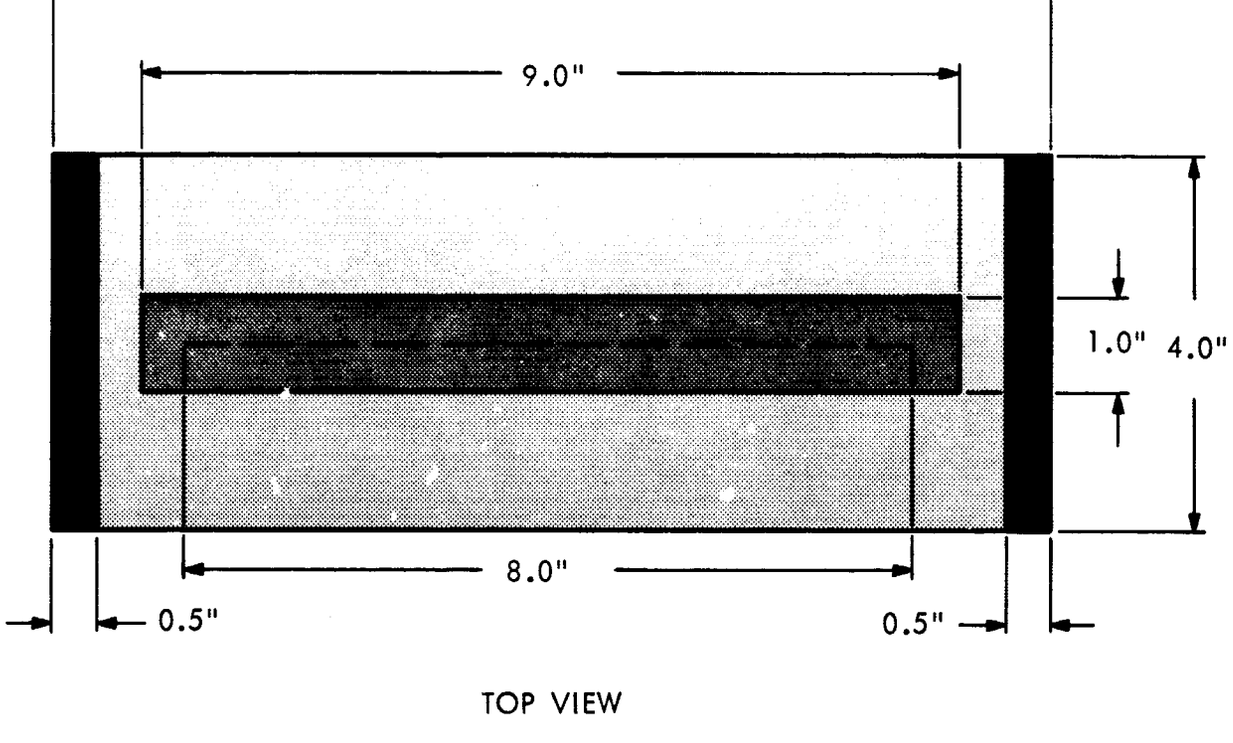
The twist-flex test apparatus is described and illustrated in detail in References 2 and 10; but briefly, the twist-flex tester rotates one of two 3-1/2" diameter horizontal parallel circular plates centered on the same vertical axis. The test specimen is fastened to the two plates and the system is immersed in liquid nitrogen. During operation, the plates oscillate horizontally over 90° angle while undergoing a simultaneous vertical oscillation of 1-5/16" causing a twist-flexing motion.

The twist-flex samples were prepared in the laminar flow clean bench under clean room conditions. Even though exposure to LOX was not anticipated, the twist-flex samples were handled "clean" to observe, as much as possible, actual bladder fabrication conditions. A strip of Schejdahl GT-300 heat sensitive tape was folded at each end of the test sample as shown in Figure 2. This was done to prevent crack propagation from the cut edges. Prior to testing each sample was leak tested for flaws with helium gas using the equipment and procedures specified in References 2 and 11. At intervals of five to ten twist-flex cycles the liquid nitrogen container

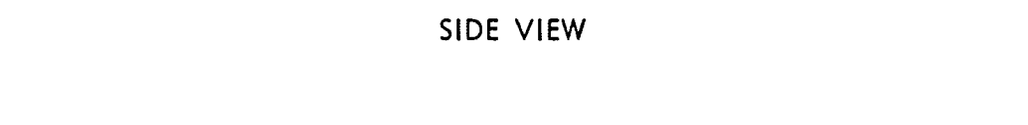
TABLE 4. TWIST-FLEX TEST DATA - CYCLES TO FAILURE AT -320°F*

SAMPLE	SAMPLE NUMBER 1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	Avg.(4)
As-Received Kapton	140/160	170/180	140/160	140/150	170/180	110(5)/130	310(5)/330	125(5)/135	250(5)/260							152.0
Baked Kapton	20/40	20/40	50/60	50/70	0/20	40/50	60/70	60/70	55/65	80/90	50/60	65/75	70/80	70(5)/85	85(5)/95	47.7
Kapton and Kel-F	54(3)/60	40(1)/46	35(1)/45	35(3)/55	55(1)/65	50(1)/60	40(3)/48	35(2)/45	35(3)/45	30(3)/40	75(3)/80					44.0
Kapton and Viton A	40(3)/45	65(1)/75	65(3)/75	50(3)/60	55(3)/65	50(3)/60	65(2)/70	52(3)/60	50(3)/60	65(3)/74						55.7
Kapton and D-4327	25(1)/35	0(1)/20	0(1)/10	10(1)/15	0(1)/10	10(1)/15	0(1)/10	0(1)/10	14(1)/20	10(1)/15						6.9
Kapton and D-4375	0(1)/10	10(1)/15	10(1)/15	5(1)/10	0(1)/10	10(1)/15	10(1)/15	15(1)/20								7.5
Kapton & CNR Coating Solution	25(1)/30	30(3)/35	50(3)/60	45(3)/55	40(3)/50	35(3)/45	35(3)/50	30(3)/40	40(3)/45	45(3)/50						37.5
Kapton & CNR Liquid	30(3)/35	30(3)/35	30(3)/36	30(3)/35	20(3)/25	25(3)/33	20(3)/25	20(3)/25	25(3)/30	20(3)/25						25.0
Kapton and GT 300	30(3)/35	20(3)/30	25(3)/30	20(3)/25	20(3)/25	20(3)/25	20(3)/25	17(3)/25	18(3)/24	22(3)/30						21.2
Kapton and GT 100	20(3)/25	25(3)/30	24(3)/28	25(3)/35	33(3)/38	20(3)/25	18(3)/25	30(3)/25								24.3

(1) Adhesive Failure
(2) Adhesive and Film Failure
(3) Film Failure
(4) Average of cycles completed without failure.
(5) These samples are from random places in the Kapton roll. Their results are not included in the average because the films were found to be less than 1/16" shorter in length than the required sample length.
* For all tests, failure occurred between lower and upper number of cycles shown by each fraction.



TOP VIEW



SIDE VIEW

FIGURE 2. TWIST-FLEX SAMPLE CONFIGURATION

was rapidly lowered from the test sample and inspection made for adhesive or film failure. The test would be terminated when failure was detected and the number of cycles recorded.

Customarily at periodic levels when pinholing of the film was suspected the sample would be removed and checked for helium permeability. Pinholing of the film would occur within a few cycles (5-10 cycles) of visible film failure. Therefore visible film failure, with an inspection every 10 cycles, was used as the failure criteria, thus eliminating the periodic helium inspection. The advantages of this technique is that it minimized film handling and eliminated detrimental repetitive thermal cycling between -320° and +72°F. Also, each sample represents a data point since it is taken to failure.

Viton A, Kel-F and CNR coating solution were the best performing adhesives in the twist-flex tests. The Kel-F adhesive was selected for further studies because the other two adhesives, Viton A and CNR, did not perform satisfactorily in the LOX impact sensitivity tests and in the twist-flex tests, respectively. When "in-bond" with Kapton, the Viton A adhesive system failed the LOX impact sensitivity test, and in terms of twist-flex performance, the best CNR adhesive performed poorer than the Kel-F adhesive in the twist-flex tests. As shown in Table 4, twist-flex specimen bonded with the Kel-F adhesive system failed at an average of 44 cycles whereas specimen bonded with the CNR coating solution averaged 37.5 twist-flex cycles. The CNR adhesive is considered as a back up adhesive which offers, next to the Kel-F adhesive system, the greatest potential for meeting LOX bladder requirements.

6.0 BLADDER FABRICATION AND TESTING

6.1 BLADDER FABRICATION

One single-ply Kapton bladder was fabricated using the Kel-F adhesive. The hemispherical plaster mandrel, with six flat sections, used in Task II and III (Reference 1) to fabricate bladders from flat gore segments was used. The gore segments were cut from the Kapton film, laid on the mandrel and bonded three segments at a time. The gores were bonded using a butt joint with a 1/2" doubler strip on the upper surface only. The excess adhesive was squeezed and wiped off with a clean cotton gauze, and then the entire assembly was vacuum bagged with FEP film and cured in an oven at 350°F.

All operations were made with rapidity to prevent excessive solvent evaporation from the adhesive system. The technique was repeated to bond two three-gore segments together, forming a hemisphere. After completing two hemispheres they were bonded together, one seam at a time on the mandrel. This was accomplished as shown schematically in Figure 3 by folding one bladder hemisphere inside the other, taking care to match the flat areas of both the bladder gores and mandrel. During adhesive application and bonding of the last seam, a few small tears occurred at the end of the bladder where the gores converge. These tears were repaired with GT-100 tape rather than with the Kel-F adhesive system to avoid further bladder wrinkling by vacuum bagging the folded bladder. Two 2-1/2" diameter end close out Kapton patches were bonded at one end of the bladder with GT-100 adhesive. One close out patch was bonded on the inside and the other close out patch was bonded on the outside of the bladder. GT-100 was used in these regions since special tooling was required if the Kel-F adhesive were used and at this stage of development the expense of tooling was not warranted. The picture of the bladder is shown in Figure 4.

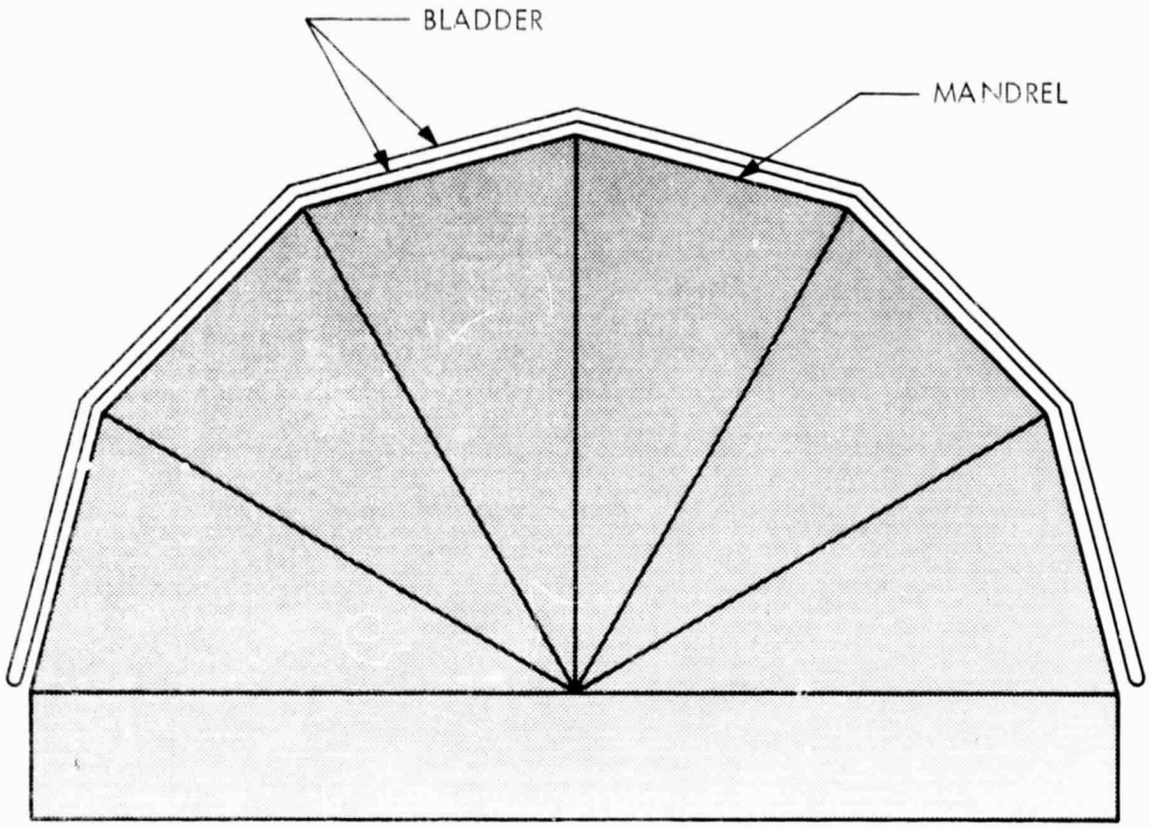


FIGURE 3. SCHEMATIC OF BLADDER FOLDING FOR BONDING THE LAST TWO SEAMS

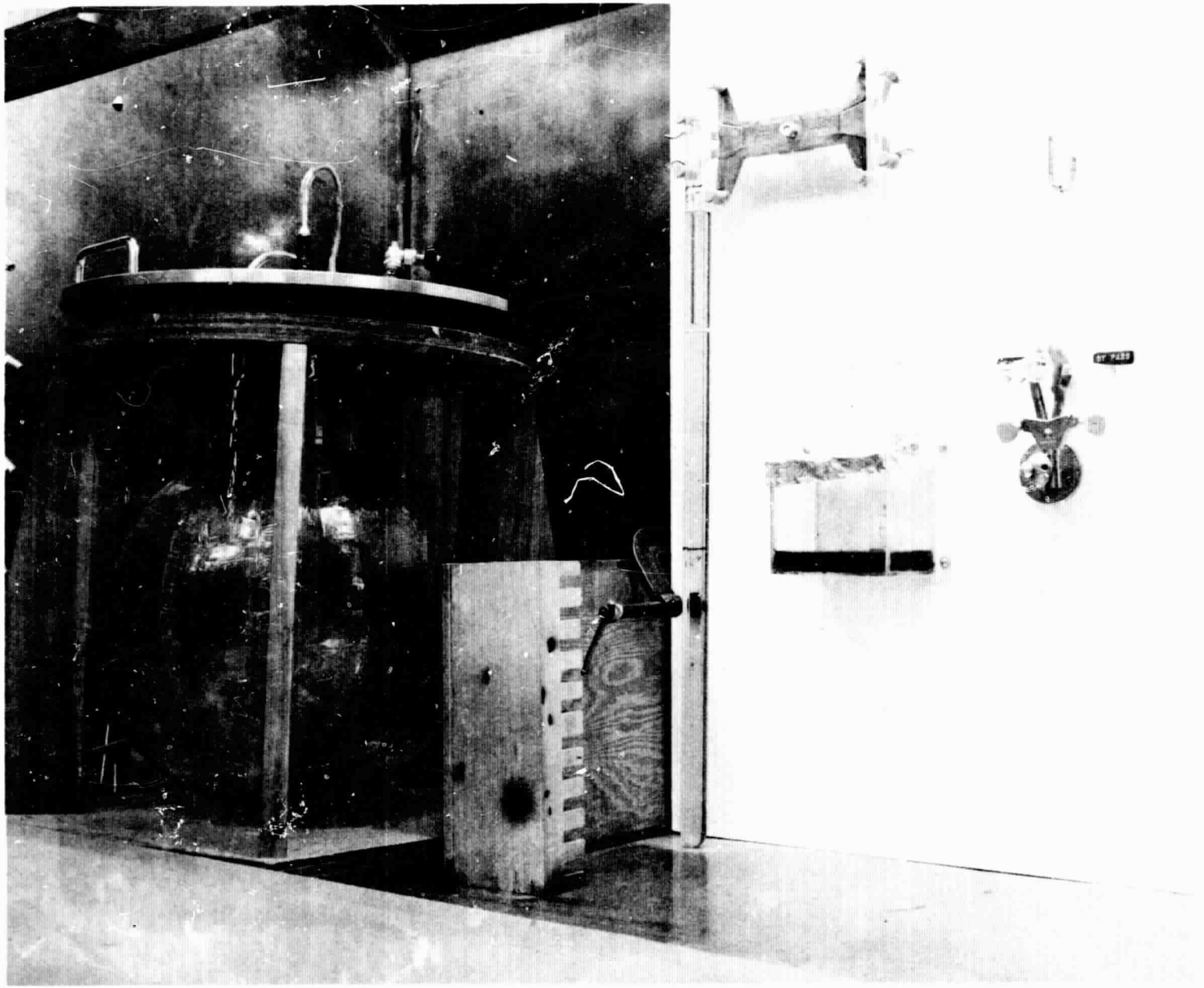


FIGURE 4. BLADDER

6.2 LEAK CHECKS

The open end of the bladder was fitted with a bladder stem and sealed with GT-100 tape. The bladder was tested for leaks with helium gas. The leak check was performed by inflating the bladder with 0.6 psig and measuring the amount of helium with a wet meter. The schematic for bladder leak checking was similar to the one shown in Figure 21 of NASA CR-72418 Final Report, Part 1, (Reference 1) of this contract. The only difference was substitution of the manometer with a wet flow meter for gases.

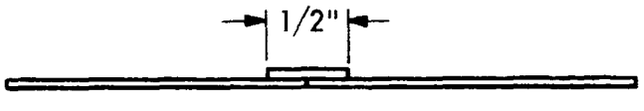
The first leak check indicated a bladder leak rate of 0.01 ft^3/sec. On close inspection of the bladder's bonded seams, a number of very small areas were not bonded. These areas occurred at a few places where the flat 1/2" wide Kapton tape creased. These creases were due to conforming the flat tape onto the spherical configuration at the seam areas. An attempt was made to close the small gaps. Fresh adhesive solution was forced into the gaps, and after spontaneous solvent evaporation at room temperature the adhesive was heated with a hand iron. Desirable bonding was not achieved. The bladder was again leak checked and the same leak rate was again observed and no further work was performed. The adhesion of the Kel-F adhesive was good but the techniques and equipment previously used with the GT-series adhesives were not adequate. It was apparent at the conclusion of the bladder fabrication that special tooling, with a male and female mold would be required to maintain pressure on the joints and prevent wrinkling while the adhesive cured.

6.3 EXPULSION CYCLING

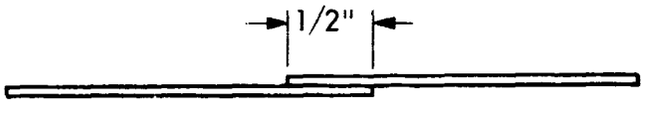
The bladder was submerged in liquid nitrogen, evacuated and filled with helium gas while still submerged in liquid nitrogen. The bladder was evacuated and reinflated ten times while under liquid nitrogen (20 reversals). The bladder was removed from the liquid nitrogen for inspection and the seams bonded with the Kel-F adhesive were found intact. The gore material was closely inspected and no visible tears were found in the gores.

6.4 BLADDER MATERIAL TESTING

The bladder was sectioned into samples for LOX ballistic impact tests and lap bond tensile strength. The results of the ballistic impact tests using the bladder materials are summarized in paragraph 8.0. With the lap bond specimens an average lap shear strength of 8.29 lb/in width was obtained which is considered adequate for a specimen with a 1/4" overlap, as shown in Table 2. This value is slightly lower than the results obtained on the flat coupons (Table 2) with the Kel-F adhesive which demonstrated an average lap shear strength of 10.88 lb/in width. The difference was attributed to lap shear test specimen configuration. The sample taken from the bladder had only 1/4" overlapping area on each side of the butt line, whereas the samples used for the tests shown in Table 2 had an overlapping area of 1/2". The side views showing these differences between the two lap shear test specimens are shown below.



Side View of Lap Shear Test Specimen Taken from Bladder



Side View of Test Specimen Used to Determine Adhesive Strength Shown in Table 2.

7.0 BALLISTIC IMPACT TESTS

Ballistic impact tests were conducted to evaluate the LOX sensitivity of single and multiple (10 plies) Kapton samples with and without the selected Kel-F adhesive systems. Mylar and Teflon, and various metals used in space applications were also tested for performance and comparison purposes. The test plan is summarized in Table 5.

The ballistic impact tests were conducted using a 30-06 caliber rifle set up at a distance of 25 feet from the test sample and fired remotely from the control house. The ammunition was standard military ball ammunition with a 152 grain copper jacketed lead slug with a muzzle velocity of 2800 ft/sec. Figure 5 shows the test schematic. The specimen holder and LOX container are shown in Figure 6.

Tests 1-10 were conducted to determine if either the adhesive or base film were sensitive to ballistic impact while immersed in LOX. The influence of multi-plies was also determined. After testing, the recovered pieces of the test specimen were closely inspected. These samples did not have any visible trace of a reaction.

Tests 11-20 duplicated the first ten tests except a 1/8" thick steel back-up plate was placed behind the film materials to provide a hard impact surface. The recovered specimens were closely inspected and specimen 18 was the only sample in this group that unequivocally reacted. Figure 7 shows the photograph of the burned Kapton. The unburned areas are probably due to extinguishment of the flame front by the specimen holding frame shown in Figure 6. The reaction observed with specimen 18 is altogether anomalous, and is not keeping with the results of tests 1-17 and 19-20. Figure 8 is a photograph of test specimen 19, the back-up plate and LOX container after test. Test 18 was a duplicate run of test 17 which had no vestige of a reaction. Three plausible explanations for the results are: 1) the sample became contaminated with LOX sensitive materials while being readied for the shot, 2) the base film is sensitive to impacts of a special nature which occur statistically on an infrequent basis and 3) multi-ply effects (Reference 1). For example, on penetrating the aluminum LOX

TABLE 5. PENETRATION TEST PROGRAM

Test No.	Film Material [1]	No. of Specimens Per Test	Film Plies per Specimen	Adhesive Seam [2]	Remarks
1-3	Kapton	1	1	No	
4-6	"	1	1	Yes	
7-8	"	1	10	No	
9-10	"	1	10	Yes	
11-13	"	1	1	No	Steel back-up plate.
14-16	"	1	1	Yes	"
17-18	"	1	10	No	"
19-20	Kapton	2*	10	Yes	Steel back-up plate.
21	Mylar-C	1	1	No	
22	"	1	1	Yes	
23	"	1	10	No	
24	"	2*	10	Yes	
25	"	1	1	No	Steel back-up plate.
26	"	1	1	Yes	"
27	"	1	10	No	"
28	Mylar-C	2*	10	Yes	"
29	Teflon (TFE)	1	1	No	"
30	Teflon (TFE)	1	10	No	"
31-34	Adhesive (Kel-F)	2*	1	Yes	[3]
35	Stainless Steel (3 mils)	1	1	No	
36	Steel Back-Up Sheet	2*	1	No	
37	Beryllium (10 mils)	2*	1	No	
38	Aluminum (2 mils)	2*	1	No	
39	Titanium (2 mils)	2*	1	No	
40	Kapton	1	10	Yes	[4]

[1] Material thickness will be as follows: Kapton, 1/2-mil; Mylar-C, 1/2-mil; Teflon (TFE), 1/2-mil.

[2] Mylar samples bonded with GT-300 adhesive.

[3] Adhesive coated on surface of 3-mil stainless steel.

[4] Film samples for this specimen taken from bladder.

* One sample with back-up plate and one without. All backup plates were of 11-gage steel.

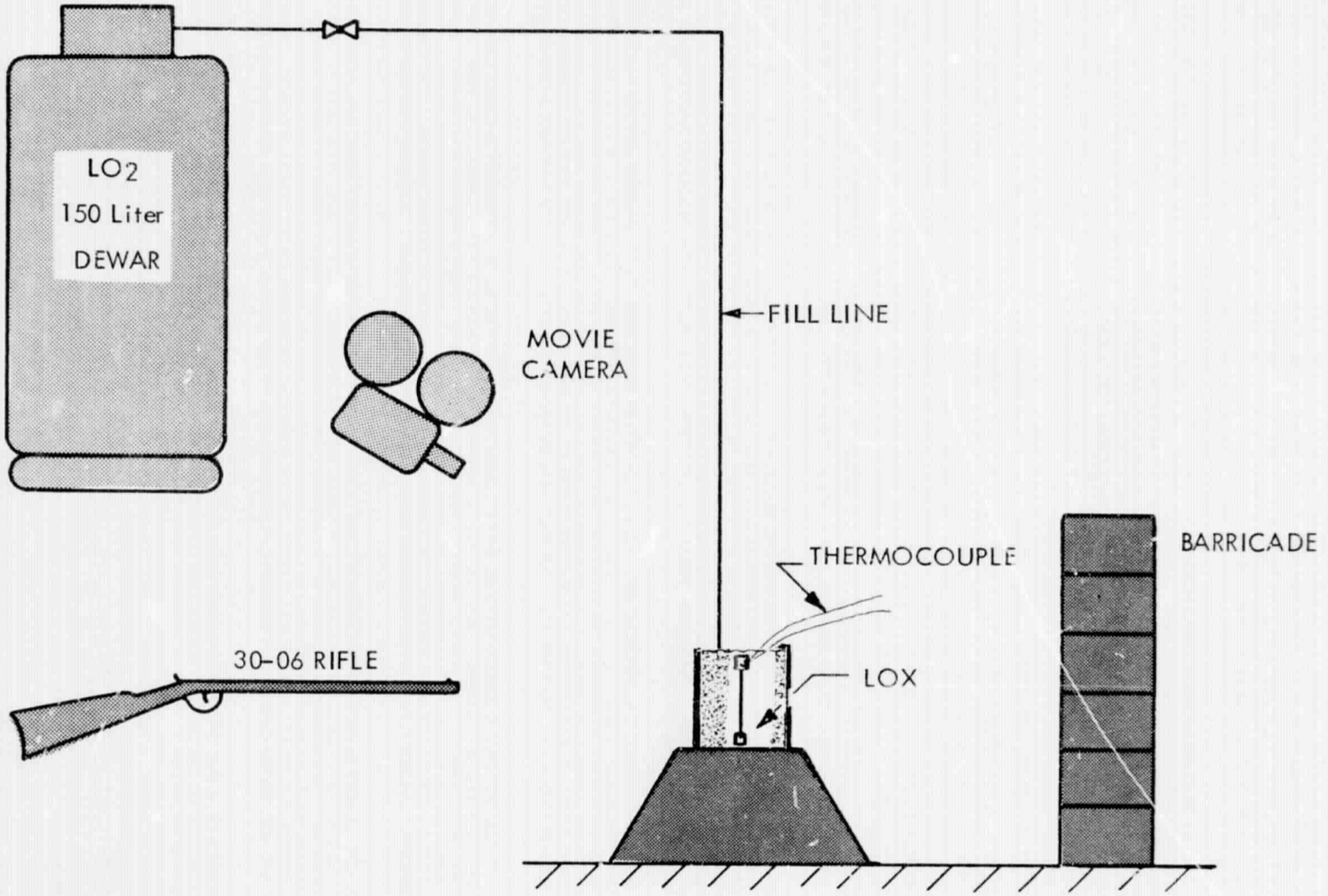


FIGURE 5. TEST SCHEMATIC

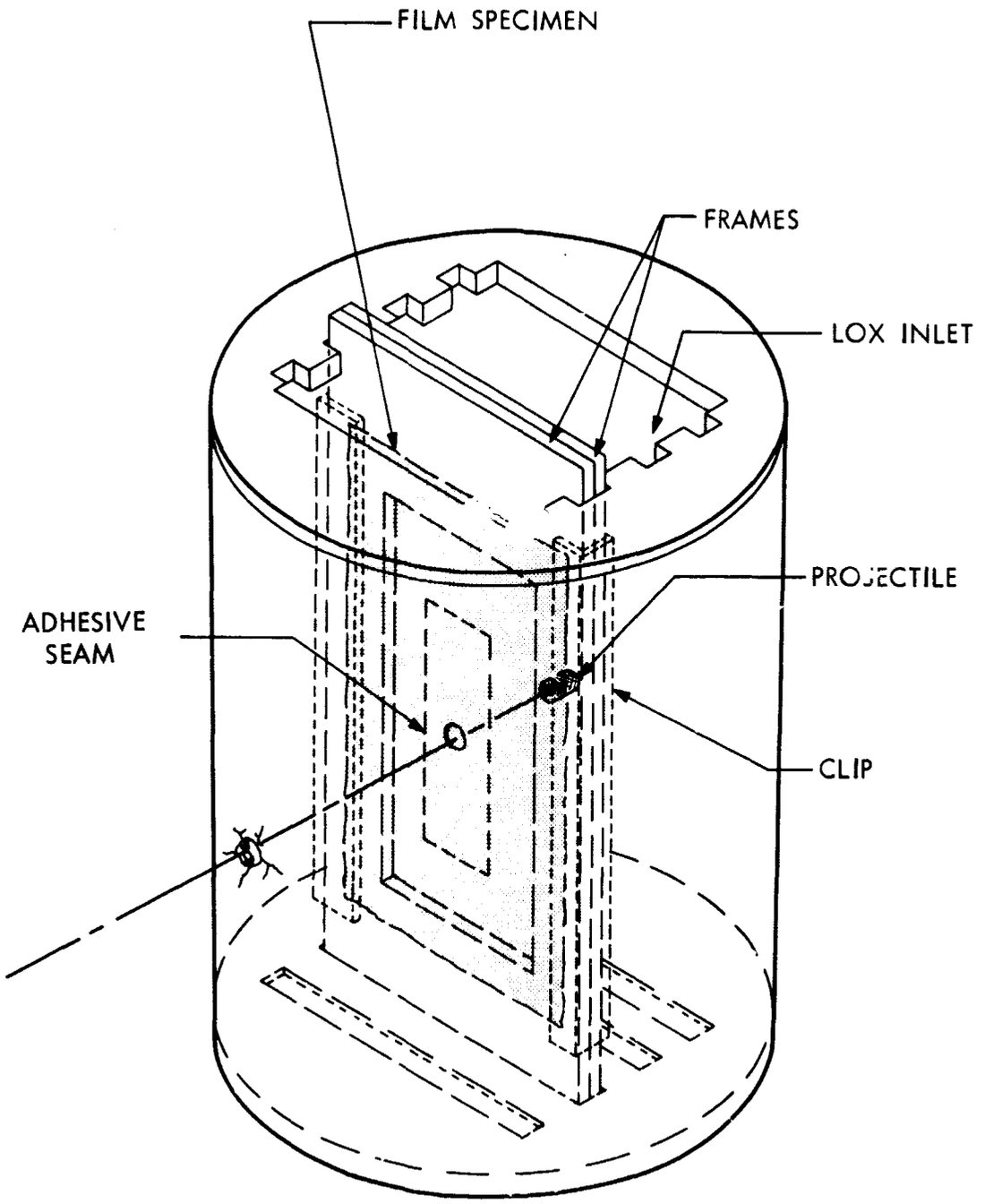


FIGURE 6. PENETRATION TEST SET-UP
SAMPLE HOLDER AND LOX CONTAINER

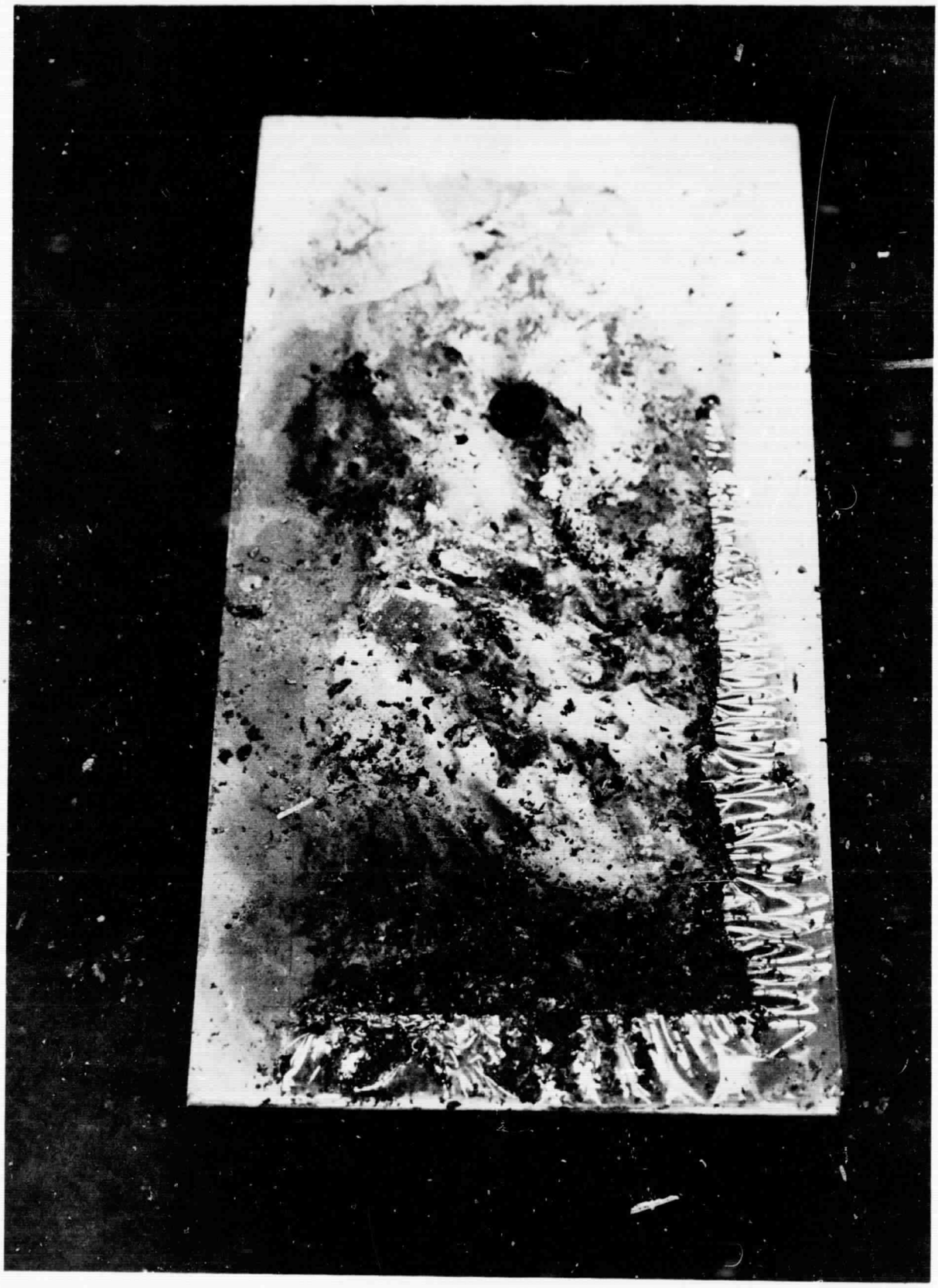


FIGURE 7. SPECIMEN 18 AFTER LOX BALLISTIC IMPACT TESTING

container bullet tips can become slightly blunt or sufficiently jagged and highly abrasive. By changing the texture of the surface of the impacting object, the test results could vary. The results of test 18 can be construed as being consistent with the greater "indicated" LOX impact sensitivity observed in Task I when multiplies of a material exhibited greater LOX impact sensitivity than single plies of the same material.

Tests 21-28 used Mylar as the base film to provide comparative data between Mylar and Kapton films. The Mylar specimens with adhesive seams used GT-300 adhesive. The processing was the same as that used in Task III of this program. In addition to providing comparative data for Kapton, these tests were to clarify questions arising from the successful ballistic impact test with Mylar conducted in Task III. After testing the specimens were closely inspected. Test specimen 23, a 10-ply specimen, had barely observable traces of black present at the periphery of the bullet hole, indicating a slight reaction. After the test specimen 27, also a 10-ply could not be found in the test area, but is believed either to have been lost due to shattering, or it was completely consumed by reaction with LOX. None of the other Mylar specimens showed any signs of reaction.

Tests 29 and 30 were to determine the ballistic impact resistance of Teflon in single and multi-ply configuration. Teflon is considered a LOX compatible material as determined by the ABMA impact sensitivity samples. After ballistic impact the specimen shattered into many small pieces. One small piece could be found and as best as could be determined from the recovered piece, the sample did not react under the test conditions. Sample 30, a 10-ply specimen of Teflon did not show visible signs of a reaction.

Test 35, on plain stainless steel foil, without adhesive, showed signs of reaction at the extreme tips of the flared entrance hole of the projectile. Possibly in this region the stainless steel became so finely divided that a reaction took place, a phenomenon experienced with stainless steel wool (Reference 4).

Tests 31-34 were four repetitive tests of the basic Kel-F adhesive coated on the same type of stainless steel foil. The stainless steel reacted in each case in the same manner as above (specimen 35), however, the Kel-F adhesive itself did not char or support combustion. There were no indications from these tests that the Kel-F was LOX sensitive.

Tests 36-39 were designed to study the behavior of various metallic materials under similar ballistic impact conditions. Upon inspection Test 36, a steel sheet with and without a back-up plate, showed signs of a reaction at the periphery of the bullet hole. Test 37, a beryllium foil without a steel back-up plate also had dark spots at the impact area indicating a reaction. Test 37 with a back-up plate was assumed to have reacted because the beryllium plate was not recovered. Movie coverage confirmed this deduction. Aluminum foil, test 38, with a back-up plate showed dark spots in both tests at the point of impact by the bullet thus indicating a reaction. Test 39 with a back-up plate reacted totally, consuming the titanium foil. Test 39 without a back-up plate partially consumed the titanium foil. Test 40, made from the sectioned bladder, did not react.

Movie coverage was made of all impacts. The film along with the sample inspections after test confirmed the above conclusions. The camera was located so that the sample will be viewed from the top and rear (approximately a 45° angle) as shown in Figure 5.

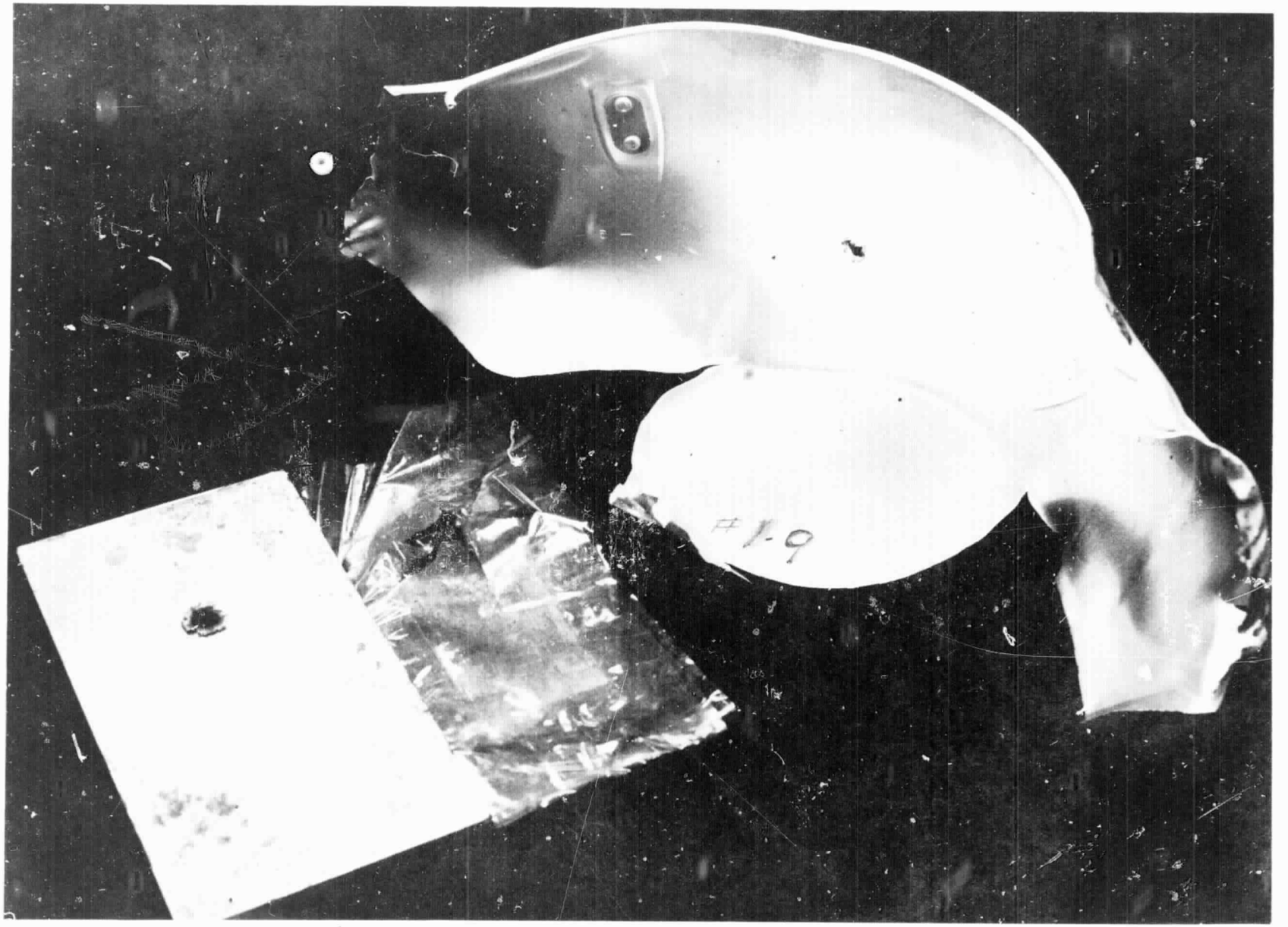


FIGURE 8 SPECIMEN 19 AFTER LOX BALLISTIC IMPACT TEST

8.0 DISCUSSION OF RESULTS AND CONCLUSIONS

In pure form all six adhesive systems developed under this program were LOX impact insensitive. Kel-F adhesive, which was the final selection, had good tensile strength and flexibility at -320°F and would serve well as an adhesive for cryogenic bladder usage. The adhesion of Kel-F to Kapton is good but techniques will have to be developed for bladder fabrication. Previously developed procedures (References 1, 2 and 11) are not applicable for this adhesive-film system.

The primary difficulty encountered in bladder fabrication was the inclusion of wrinkles in the bond line which resulted in potential leak paths. This problem can be eliminated with the fabrication of good mandrel tooling.

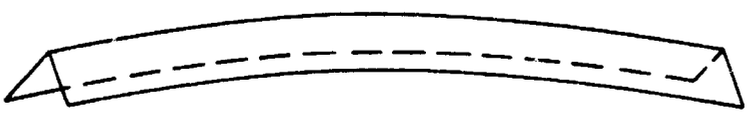
Kel-F adhesive withstood both the ABMA impact test of 72 ft/lb and a ballistic impact without any adverse reaction. Both impact conditions are more severe than that which a bladder may experience inside a rigid propellant tank.

In general, Mylar, Kapton and Teflon films all successfully passed the ballistic impact tests. Only one Mylar and one Kapton sample reacted and it is not conclusive that these reactions were initiated by the film itself. All the metallic samples showed signs of charring in the region of penetration. The charring in each case was centered around the torn edges and finely divided portions of the metallic foils and the reaction was limited to charring.

9.0 RECOMMENDATIONS

1. Improved techniques for bladder fabrication using the Kel-F adhesive system should be developed. Consideration should be given to fabricating a collapsible mandrel that will enable fabrication of a bladder in one operation and that is amenable for use with the techniques required for the Kel-F adhesive system.

2. Use of preformed doubler strips which conform to the contour of the mandrel is recommended. During fabrication of the bladder, bonding the 1/2" flat doubler strips over the butt joints presented considerable difficulty because the strips lacked sufficient extensibility to conform to the curved contour of the mandrel. Small wrinkles would form in the bond line causing severe leak paths. Formation of these wrinkles would be avoided if preformed doubler strips were used. The feasibility of shaping Kapton to fit the contour of a circular arc was demonstrated through a brief experiment. During the performance of this Task a 1/2" wide strip of Kapton was stretched over the edge of a circular metal template. After heating for half a hour in an air circulating oven set at 450°F, the film retained the circular three dimensional shape shown below which more closely matches the spherical contour of the bladder's surface.



3. The aging properties of the Kel-F adhesive system should be determined at conditions similar to actual use condition. At low temperature the majority of known polymers have some degree of crystallinity which is associated with particular organic structures in the polymer. Rapid cooling of a polymer to liquid oxygen temperatures can freeze a polymer in an amorphous state by restricting of molecular motion. Such is believed to be the case for the Kel-F adhesive system used in this program. Before use in rocket applications, positive proof for this hypothesis must be obtained for the adhesive system, particularly when exposed to dynamic conditions.

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