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DESIGN, FABRICATION AND CHARACTERIZATION  
OF OPTIMUM FIBROUS STRUCTURES OF  
POLYBENZIMIDAZOLE YARN

Technical Report AFML-TR-66-167, Vol. III  
June 1966

Chemical Processing Branch  
Manufacturing Technology Division  
Non Metallic Materials Division  
Air Force Materials Laboratory  
Research and Technology Division  
Air Force Systems Command  
Wright-Patterson Air Force Base, Ohio

Project Nr 8-163

Prepared under Contract AF33(657)-11232 by the  
Celanese Corporation, New York, New York

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**DESIGN, FABRICATION AND CHARACTERIZATION  
OF OPTIMUM FIBROUS STRUCTURES OF  
POLYBENZIMIDAZOLE YARN**

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## FOREWORD

This report was prepared by Celanese Fibers Marketing Company as one of three reports under USAF Contract Nr. AF33(657)-11232. This contract was initiated under Manufacturing Methods Project 8-163, "Research and Development of Manufacturing Methods for PBI Fibers and Fibrous Structures of PBI Yarns" with Leo J. Conlon of Manufacturing Technology Division, Air Force Materials Laboratory, Research and Technology Division as Project Engineer.

This report was initiated under Project 7320, "Fibrous Materials for Decelerators and Structures and Task 732002, "Fibrous Structural Materials", of the Non-Metallic Materials Division, Air Force Materials Laboratory, Research and Technology Division, with Mr. J.H. Ross acting as Project Engineer.

This report covers work conducted during this one year phase - April 1965 to April 1966.

This technical report has been reviewed and is approved.

*Melvin E. Fields*

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## ABSTRACT

A limited study was made to characterize the effects of parametric variations in construction on the mechanical performance of various parachute items made from polybenzimidazole (PBI) yarns. The investigation included making nine sewing threads and weaving thirty-two fabrics and ten webbings, ribbons and tapes. The results indicate that most of the specifications originally written for nylon parachute components can be met with PBI if suitable modifications are made in construction to allow for the lower tenacity of PBI yarn. Strength translation efficiencies are good enough to permit prediction of breaking loads of structures with reasonable accuracy on the basis of one pound per 100 deniers of yarn used.

Sufficient amounts of each required end item were made to permit complete laboratory characterization including testing at ambient, 350°F, 450°F, 700°F, 900°F, and 1000°F after exposures ranging from 0 to 200 hours. Only the threads and one fabric were tested over the specified range when it became apparent that finish modifications would be required before reliable results could be obtained from high temperature testing. An investigation was made of the high temperature resistance of available candidate finishes and finish components applied to PBI fabric and yarn by various methods. While no satisfactory material was found, some leads were developed which may be useful in further studies. The testing done on the threads and lightweight fabric showed that PBI yarn structures retain about 40% of their original strength after exposure at 700°F for two hours, even with an unsatisfactory finish. This represents a significant advance over presently available materials.

One outstanding characteristic of the yarn used was its good processing quality. Though filament deniers and therefore total yarn deniers appeared to vary considerably as would be expected with the first production of any new yarn, nevertheless, the difficulties usually encountered in processing yarns of such character were almost totally absent.

The reasonably good strength and the good strength retention at elevated temperatures indicate that PBI yarn should be useful for service up to and somewhat beyond 700°F for extended periods.

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## 1. INTRODUCTION

In this space age, the need is rapidly developing for fibrous materials with greater retention of performance characteristics under the extreme conditions encountered in aerospace environments. New fiber materials are required for aerospace vehicles, instrument recovery, capsule delivery, re-entry systems, etc. Conventional synthetic fibers such as nylon and polyester have not met the requirements of these applications, particularly with regard to their ability to withstand temperatures above about 350°F in deceleration uses.

Early in 1959, E. I. duPont de Nemours and Company, Inc. published the first data on a new nylon designated HT-1 at the time and later named Nomex. This material in fibrous form was shown to have improved heat-resistant properties over conventional polyester and nylon and has found considerable use in deceleration systems.

More recently another new polymer has been developed and fibers formed therefrom which gave indications of providing a substantial improvement over Nomex. This new fiber is spun from poly(benzimidazole) (PBI). Following preliminary studies by the Polymer Branch of the Nonmetallic Materials Laboratory on small quantities of yarn produced by E. I. duPont de Nemours Company, the Air Force solicited proposals for a broader investigation and eventually awarded Contract Number AF33(657)-11232 to Celanese Corporation.

This contract involved several phases of study:

Phase I: Bench scale - Development of processes and equipment capable of producing 5 lb of PBI yarn per 8 hr day.

Phase II: Design and production of optimum constructions of fabrics, webbings, tapes, ribbons and sewing threads of PBI yarns.

Phase III: Design, acquisition, installation and operation of pilot production equipment having a capacity of 50 lb of PBI fiber per 8 hr day.

Phase IV: Preliminary design of production plant having a capacity of 1,000 lb per 8 hr day.

Phases I, III, and IV were carried out by the Summit Laboratories of Celanese Corporation. These studies are reported in AFML-TR-66-167, Part I. Phase II was carried out by the Applications and Product Development Laboratories of Celanese Fibers Marketing Company at Charlotte, North Carolina. These studies are reported herein.

## II. OBJECTIVES AND REQUIREMENTS

The objectives of this program were to establish optimum constructions of fabrics, webbings, tapes, ribbons and sewing threads of PBI yarns, to produce specified quantities of these constructions, and to characterize these materials at ambient conditions and at various elevated temperature and extended time levels.

The work was to be divided into three parts:

### Part 1. Material Geometry

A limited study was to be made on the two yarn deniers to determine optimum twist level and processing techniques for the yarns. Using results of this study, the effects of the parameters

- a. Yarn twist
- b. Ends and picks per inch
- c. Weave type
- d. Weaving tensions

were to be characterized for the following materials:

- a. Two fabrics: 2.25 and 4.75 oz/yd<sup>2</sup> similar to Type I of specifications MIL-C-7350 and MIL-C-8021 respectively.
- b. Two webbings: 1-23/32 in. wide, 1200 lb breaking strength and 1 in. wide, 9000 lb breaking strength, similar to Types XII and XX of specification MIL-W-4088.
- c. Two ribbons: 1.25 in. wide, 2500 lb breaking strength and 2 in. wide, 3000 lb breaking strength.
- d. Two tapes: 9/16 in. wide, 1300 lb breaking strength and 1 in. wide, 3000 lb breaking strength.
- e. Three sewing threads: similar to Size E, 3 cord and 6 cord of specification MIL-T-7807.

Properties to be measured in characterizing these materials were:

- a. Twist (turns per inch)
- b. Ends and picks per inch
- c. Weight (oz/yd<sup>2</sup>, oz/yd or yd/lb)
- d. Breaking strengths (lb/in. for fabrics, lb for other materials)
- e. Elongation (%)
- f. Energy absorption
- g. Seam efficiency

The developed data were to be used in Part 2.

Part 2. Design and Production of Optimum Materials

Based on the data developed in Part 1 above, the following quantities of end items were to be produced, in the optimum construction of each:

	<u>Width (in.)</u>	<u>Strength or Weight</u>	<u>Quantity</u>
Ribbon	1.25	2,500 lb min	550 yd
	2.00	3,000	450
Webbing	1-23/32	1,200 lb min	500 yd
	1.00	9,000	150
Tapes	9/16	1,300 lb min	850 yd
	1.00	3,000	450
Threads	E Thread	7.0 lb min	5 lb
	3 Cord	24.0	5
	6 Cord	50.0	2
Fabrics	36	2.25 oz/yd <sup>2</sup>	200 yd
	36	4.75	100

### Part 3. Characterization of Optimum Constructions

A sufficient excess of each item was to be prepared in order to establish the following characteristics:

- (1) Weight (oz/yd) (Thread-yd/lb)
- (2) Thickness (in.) (Thread - N/A)
- (3) Ply and twist of warp and filling yarns
- (4) Total ends and picks/inch (Thread - N/A)
- (5) Breaking strength (lb)
- (6) Elongation (%)
- (7) Energy absorption (Thread - N/A)

In addition, the performance at elevated temperatures for various exposure times was to be determined. By amendment to the contract, this was changed to permit studies and evaluation of selected yarn and fabric finishes which might withstand temperatures of 700°F and above and thereby provide better performance of the fabrics at these temperatures.

### III. PART 1 - MATERIAL GEOMETRY

#### A. Basic information development.

##### (1) Yarn twist response.

Twist response tests of 100 denier lubricated and unlubricated yarns gave somewhat erratic results but indicate that optimum strength and elongation are attained over the range of 3 to 10 tpi. Lubricated yarns tended to have higher strengths than unlubricated yarns, particularly at higher twist levels.

Twist response tests of 200 denier yarn gave a well defined curve which peaks at 3-5 tpi twist, for both straight and loop tenacity. Straight elongation does not materially change over the range of 3 -20 tpi while loop elongation reaches a peak at 5 tpi twist.

Twist response curves of all these yarns are shown in Figures 1 and 2. Based on these results and data reported below on tests of experimental threads, it was decided to use 3 tpi twist for all fabrics, both parachute and narrow.

##### (2) Warp sizing trials.

The first set of twist response data obtained on 100 denier unlubricated yarn indicated optimum twist to be about 7 tpi. Using previously developed techniques by which woven fabric samples can be produced from only a few ounces of yarn, a 6 in. by 8 yd warp was made up and woven 70 x 70 in plain, twill and per Figure 1 of MIL-C-7350 constructions. Weaving was satisfactory but permeability was too high on all constructions. A repeat trial using 5 tpi yarn gave poor weaving and low permeability, the latter probably being the result of the former. These results indicated it would be necessary to use 3 tpi yarn but sizing would be required.

For sizing trials, a 10-in. wide, 100-yd warp was made up, 80 ends/in. using 3 tpi yarn. This was sized using the best known system for nylon, as follows:

Formulation: 200 lb Polyfilm TS-20\* in 100-gal mix  
Speed: 5 yd/min  
Size Temperature: 130°F  
Can Temperature: 190°, 210°, 210°, 210°, 190°F  
Wet Split Used  
Overwax: 0.8% Texize 9311\*\*

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\*Produced by Polymer Southern Company, Post Office Box 2184,  
Greenville, South Carolina 29602

\*\*Produced by Texize Chemicals, Inc., Post Office Box 368,  
Greenville, South Carolina 29602

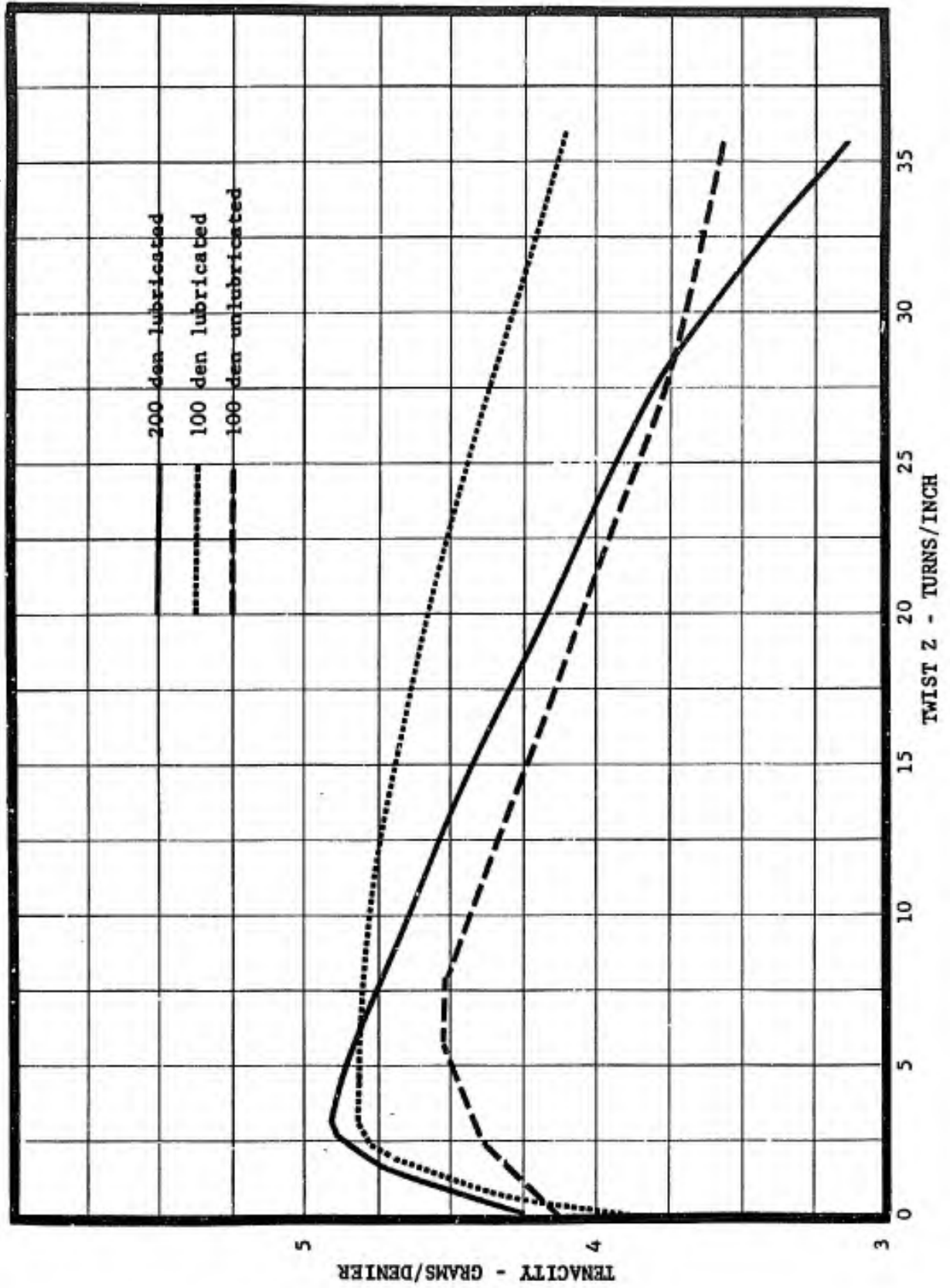


FIGURE 1. TENACITY OF PBI YARNS AT VARIOUS TWIST LEVELS

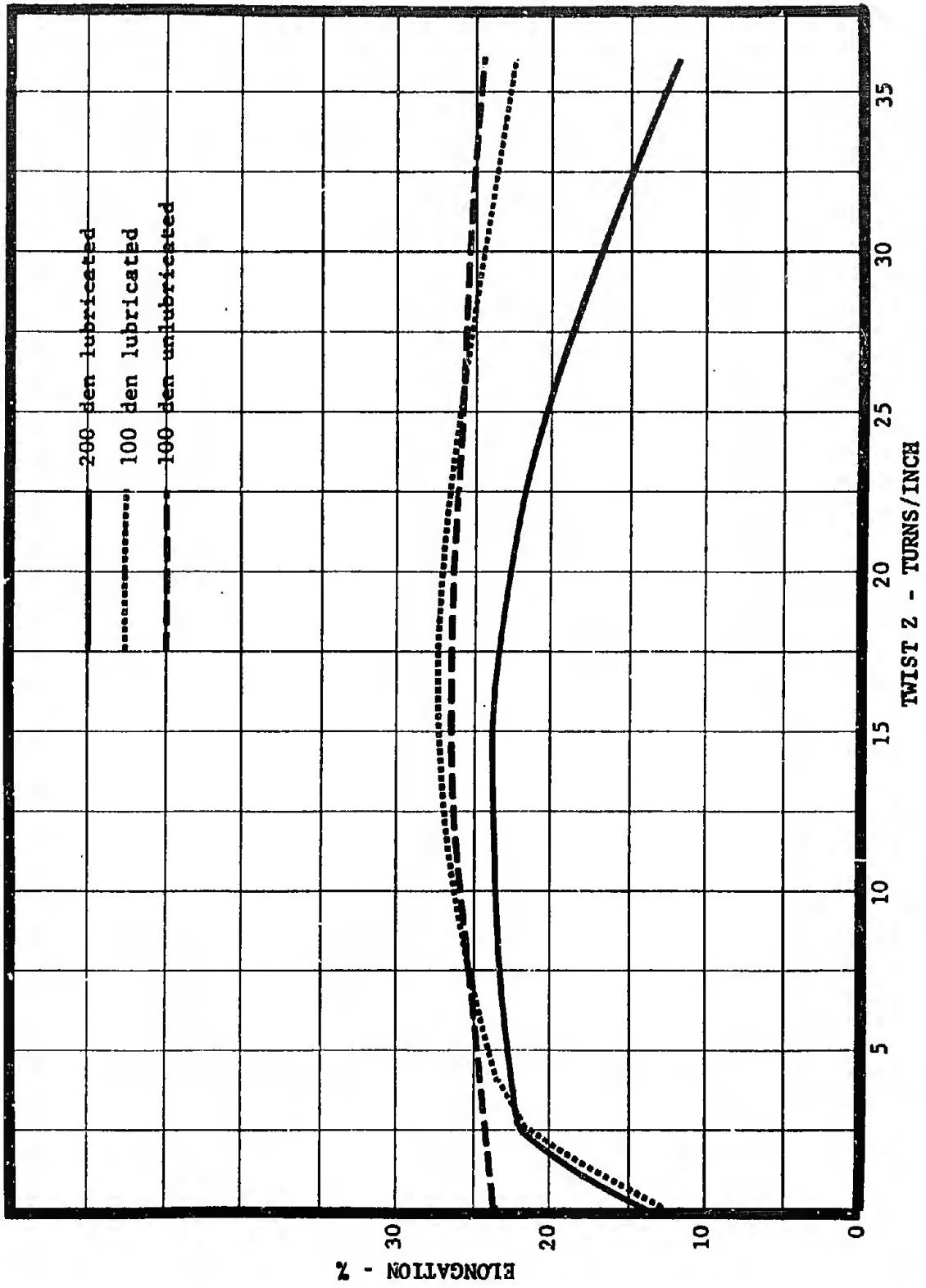


FIGURE 2. ELONGATION OF PBI YARNS AT VARIOUS TWIST LEVELS

This warp was placed in a loom and sufficient fabric was woven to show that the size formulation and application were satisfactory. Sufficient yarn remained in the warp to carry out a sequence of weaving trials to explore the variables involved in the 2.25 oz parachute fabric. The formulation and application established by this trial was used throughout for both parachute fabrics.

(3) Effects of moisture and conditions on physical properties of PBI yarns.

When it became apparent that sizing would be necessary for the parachute fabrics, some information on the wet behavior and recovery from wetting was developed. The data, shown in Table I, indicates that though strength is lost upon wetting, PBI yarns recover fully when dried.

TABLE I

## EFFECTS OF WET TREATMENTS ON 200/50/32 FBI YARN

<u>Treatment</u>	<u>Tenacity</u> <sup>1</sup> (gm/denier)	<u>Elongation</u> <sup>2</sup> (%)
1. Laboratory conditioning <sup>3</sup>	4.74	21.9
2. Steamed 30 min, <sup>4</sup> tested immediately	4.22	22.8
3. Steamed 30 min and conditioned	4.35	23.2
4. Steamed 30 min, dried in oven (110°C) 30 min, tested immediately	4.77	23.5
5. Steamed 30 min, dried in oven 30 min and conditioned	4.63	24.2
6. Dried 30 min in oven, tested immediately	4.80	21.9
7. Treated 1 min in water at 82°C, dried 30 min and conditioned	4.71	23.4
8. Treated in water at room temperature 1 min, tested immediately	3.57	25.2

<sup>1</sup>Tenacity is the tensile stress expressed as breaking force per unit linear density, e.g., grams per denier (gm/den). Denier of a yarn or fiber is the weight in grams of 9000 meters thereof. For these calculations, the denier of the untreated yarn (200) was used throughout.

<sup>2</sup>Elongation is deformation caused by tensile force and is expressed as a percentage of the original specimen length.

<sup>3</sup>Conditioned for 3 days at 75°F and 65° relative humidity.

<sup>4</sup>Steaming was done in a sterilizer at ambient pressure.

## B. Threads

A set of nine experimental sewing threads in the three required sizes were made up for evaluation. The three sizes were similar to E thread, 3 cord and 6 cord of nylon thread per specification MIL-T-7807B. The critical factor involved in designing these threads was strength. Based on the available yarn sizes of 100 and 200 denier and the target tenacity of 4.5 gm/den, the constructions of the desired threads were specified:

### Design of Sewing Threads

	<u>E Thread</u>	<u>3 Cord</u>	<u>6 Cord</u>
Required strength: lb	8.5	24	50
gm	3859	10896	22700
Yarn tenacity, gm/den	4.5	4.5	4.5
Total denier required	857	2421	5044
Denier per strand	286	807	1682
Nearest construction	3/100	5/200	9/200
Total denier	900	3000	5400
Expected strength, lb	8.9	29.5	53.6

In order to obtain constructions comparable to other high temperature materials under investigation by others, the system used by Fabric Research Laboratories in establishing twist levels was applied in this study. The levels, therefore, are based on comparable fiber helix angles regardless of the strand denier. The twist levels selected for the experimental threads, based on this method, became:

### Construction of Experimental Threads

<u>Thread</u>	<u>Construction</u>	<u>Singles Twist, tpi</u>			<u>Ply Twist, tpi</u>		
		<u>Low</u>	<u>Medium</u>	<u>High</u>	<u>Low</u>	<u>Medium</u>	<u>High</u>
E Thread	100/3/3	12.0	18.0	24.0	9.0	13.5	18.0
3 Cord	200/5/3	6.6	9.9	13.2	4.5	6.8	9.1
6 Cord	200/9/3	5.0	7.5	10.0	2.9	4.3	5.7

These threads were made and tested. The results appear in Table II.

It is noted that in each and every case, strength decreases as twist increases. Since the low and medium twists used for all three required threads gave strengths which exceed minimum specifications, no further experimental trials were made. The construction which gave the highest strength for each thread, the lowest twist, was used for production of the required threads for delivery to the Air Force.

TABLE II

## PROPERTIES OF EXPERIMENTAL FBI SEWING THREADS

<u>Sample and Construction</u>	<u>Total Denier</u>	<u>Tensile Strength (lb)</u>	<u>Elongation (%)</u>	<u>Measured Single</u>	<u>Twist (tpi) Ply</u>	<u>Translation Efficiency<sup>1</sup> (%)</u>
<b>E-Threads</b>						
E-1 100/3/3 12.08 x 9.0Z	966	9.1	27	13.7S	9.1Z	105
E-2 100/3/3 18.08 x 13.5Z	996	8.9	30	18.6S	13.1Z	103
E-3 100/3/3 24.08 x 18.0Z	979	8.0	35	25.4S	19.0Z	13
<b>3-Cords</b>						
3-1 200/5/3 6.6Z x 4.5Z	3060	26.8	14	6.6S	3.7Z	81
3-2 200/5/3 9.98 x 6.8Z	3060	25.8	16	10.2S	5.6Z	78
3-3 200/5/3 13.28 x 9.1Z	3072	22.6	20	12.4S	9.8Z	69
<b>6-Cords</b>						
6-1 200/9/3 5.08 x 2.9Z	5940	54.2	17	5.2S	2.8Z	92
6-2 200/9/3 7.58 x 4.3Z	5940	53.8	18	7.7S	3.0Z	91
6-3 200/9/3 10.08 x 5.7Z	6012	50.7	19	9.8S	3.7Z	96

<sup>1</sup>Fiber to thread - based on 4.09 denier per filament and 4.29 gm/den. tenacity for the 100den. products and 4.18 den/filament and 4.75 gm/den. tenacity for the 200den. products. Denier (den) of a yarn, fiber or filament is the weight in grams of 9000 meters thereof.

<sup>2</sup>First plying twist; the parent yarns were not twisted.

For producing seamed samples of some of the narrow fabrics, the sewing sub-contractor, Pioneer Parachute Company, indicated that FF size thread is now being used and requested some of this size. The Air Force granted permission to reduce the required quantities of the originally specified threads to provide the FF thread needed. This thread was made and characterized along with the original three.

## C. Fabrics

### (1) 2.25-Ounce Fabric

As noted above, design work on the lightweight 2.25-oz fabric started before it was discovered that sizing of warps would be necessary. Fortunately, no serious displacement of the program resulted since the first formulation tried gave successful weaving results and sufficient warp was left over to make the required construction studies. In this left over warp, ten samples of two yards each were woven, constructed as shown in Table III. These samples were tested for required characteristics and the results are given in Table IV. The test data indicated the optimum fabric should be about 76 x 76 constructed similar to Type I of MIL-C-7350 and using 3 tpi yarns in both warp and filling. To test this indication, four additional short samples woven in the size trial warp and constructed as shown in Table III, (Samples 11-14) were made and tested. Results appear as in Table IV. Since both permeability and fabric weights still were not within the required limits in any one fabric, two additional trials were made per Samples 15 and 16 of Table III. Test results given in Table IV showed both to meet specifications and the final construction was selected to be exactly between these two. For assurance, weight and permeability were determined on the first yard woven of the production run and results appear as Sample P in Table IV.

### (2) 4.75-Ounce Fabric

Based on data and experience from trials made on the lightweight fabric, a series of trials were designed and woven to test experimental constructions for the heavy fabric. It quickly became apparent that to achieve the permeability required, 50 to 90 cfm at 0.5 in. water pressure, ends and picks would have to be reduced. The amount of reduction required turned out to be much greater than anticipated, as shown by the successive stages outlined in Table V and the permeability results obtained at each step as given in Table VI. At 66 x 66 construction, as in Sample 12, when permeability was well within specified limits, fabric weight and strength had been drastically reduced. In efforts to restore these properties at least partially, four additional trials were woven. Two of these used 2-ply yarns while special rip-stop weaves were used on the other two to open up the fabric. Test results indicated Sample 13, which was a plain weave, 38 x 38 construction using 2-ply yarns in both warp and filling appeared satisfactory. The Air Force approved this fabric and it was selected for production.

## D. Narrow Fabrics

Included under this heading are the two webbings, two tapes and two ribbons. In view of the shortness of time, the weaving of all these materials, both experimental and production lots, was sub-contracted to Murdock Webbing Company, Inc., Central Falls, Rhode Island.

TABLE III  
EXPERIMENTAL CONSTRUCTIONS - 2.25-OUNCE FABRIC

Sample	Warp		Filling		Weave
	Ends/In.	Twist/In.	Picks/In.	Twist/In.	
1	80	3	80	3	Plain
2	80	3	80	3	2 x 2 Basket
3	80	3	80	7	2 x 2 Basket
4	80	3	80	3	MIL-C-8021 - Type I
5	80	3	80	7	MIL-C-8021 - Type I
6	80	3	80	3	MIL-C-7350 - Type I
7	80	3	80	7	MIL-C-7350 - Type I
8	70	3	70	3	MIL-C-7350 - Type I
9	70	3	70	3	2 x 2 Basket
10	70	3	70	3	MIL-C-8021 - Type I
11	76	3	74	3	MIL-C-7350 - Type I
12	76	3	76	3	"
13	76	3	74	5	"
14	76	3	76	5	"
15	74	3	74	3	MIL-C-7350 - Type I
16	74	2	74	2	"
p1	74	2.5	74	2.5	MIL-C-7350 - Type I

TABLE IV  
PROPERTIES OF EXPERIMENTAL 2.25-OUNCE FABRIC CONSTRUCTIONS

Sample	Weight (oz/yd <sup>2</sup> )	Thread Count (WxF)	Strip Tensile (lb/in.-WxF)	Strip Elongation (%-WxF)	Air Permeability (ft <sup>3</sup> /min/ft <sup>2</sup> )			
					@0.5 in. Pressure	@1.0 in. Pressure	@3.0 in. Pressure	@5.0 in. Pressure
1	2.48	82x80	68x74	16x20	91	138	280	375
2	2.39	82x80	68x74	14x70	120	208	423	560
3	2.44	82x80	64x82	15x24	290	424	792	---
4	2.41	83x82	66x74	13x18	86	139	303	407
5	2.44	83x83	64x82	13x23	186	285	553	748
6	2.37	82x82	65x77	15x21	97	151	312	421
7	2.49	84x82	69x83	15x24	160	234	480	629
8	2.06	72x71	57x66	12x29	175	262	536	710
9	2.03	71x72	56x66	13x29	193	289	625	815
10	2.10	72x71	60x60	14x18	166	254	542	734
11	2.26	79x76	63x70	14x17	146		Not Determined	
12	2.28	79x78	64x73	14x19	134		"	"
13	2.26	79x76	63x70	15x21	167		"	"
14	2.24	79x77	62x77	15x22	169		"	"
15	2.22	76x76	59x66	14x17	145		"	"
16	2.17	75x75	58x72	14x17	115		"	"
p1	2.22	75x75	65x72	18x24	119	164	410	600

<sup>1</sup>Head end of production material - see Table XII for complete characteristics.

TABLE V  
EXPERIMENTAL CONSTRUCTIONS - 4.75-OUNCE FABRIC

Sample	Warp		Filling		Weave
	Ends/In.	Twist/In.	Ends/In.	Twist/In.	
1	80	3	80	5	MIL-C-8021 - Type I
2	80	3	76	3	MIL-C-8021 - Type I
3	80	3	76	7	MIL-C-8021 - Type I
4	78	3	78	7	MIL-C-7350 - Type I
5	74	3	74	3	MIL-C-8021 - Type I
6	36/2	3	36/2	3	MIL-C-8021 - Type I (2 ends as one)
7	70	3	70	3	MIL-C-8021 - Type I
8	70	3	70	7	MIL-C-8021 - Type I
9	70	3	70	11	MIL-C-8021 - Type I
10	70	7	70	7	MIL-C-8021 - Type I
11	68	7	68	7	MIL-C-8021 - Type I
12	66	7	66	7	MIL-C-8021 - Type I
13	38/2	5	38/2	5	Plain Weave
14	38/2	5	38/2	5	MIL-C-8021 - Type I
15	72	5	72	5	Special
16	72	5	72	5	Special
p1	38/2	5	38/2	5	Plain Weave

TABLE VI  
PROPERTIES OF EXPERIMENTAL 4.75-OUNCE FABRIC CONSTRUCTIONS

Sample	Weight (oz/yd <sup>2</sup> )	Strip Tensile (lb/in.-WxF)	Elongation (%-WxF)	Air Permeability (cfm @ 0.5 in.)
1	4.75	---	---	14
2	4.61	156x166	23x28	15
3	4.63			20
4	4.84			8
5	4.36			16
6	4.10			89
7	----			24
8	----			46
9	----			85
10	3.96	131x147	22x25	53
11	3.97			53
12	3.97	124x115	22x22	61
13	4.43	134x145	14x20	83
14	4.36	118x150	13x23	159
15	4.50	125x162	22x21	19
16	4.43	131x163	24x19	41
p1	4.79	140x167	19x27	65

<sup>1</sup>Head end of production fabric - see Table XII for complete characteristics.

Murdock was requested to weave 10 yd each of ten experimental constructions from yarns twisted and warped by Celanese. The constructions requested with modifications made by Murdock in order to achieve weavability are outlined in Table VII. These fabrics were tested for breaking strength, weight per linear yard, width and thickness. The results appear in Table VIII. Some of the fabrics exceeded their maximum allowed widths, so that slight changes were required in specifying the construction of production materials. Clearance was obtained from the Air Force to permit Webbing 2 to exceed its specified width in order to attain the desired strength while retaining some flexibility.

Designs for the constructions used in making production fabrics were worked out from the test data on these experimental fabrics.

TABLE VII

## EXPERIMENTAL CONSTRUCTIONS - NARROW FABRICS

Type	No.	Width (in.) Max.	Strength (lb) Min.	Warp		Binder		Filling		Weave
				Ends	Yarn	Ends	Yarn	Picks	Yarn	
Web	1A	1.781	1200	288	200/3/3Z	None	28(25)	200/3/3Z	MIL-W-4088, Type XII	
Web	1B	1.781	1200	288	200/3/3Z	None	28(25)	200/3/3Z	MIL-W-4088, Type XXII	
Web	1C	1.781	1200	288	200/3/3Z	None	28(21)	200/3/3Z	MIL-W-4088, TYPE XVIII	
Web	2	1.031	9000	162	200/35/3Z	26	200/4/3Z	20(20)	MIL-W-4088, Type XX	
Ribbon	1	1.281	2500	264	200/6/3Z	None	26(16)	200/3/3Z	2/2 R.H. Twill	
Ribbon	2A	2.063	3000	188	200/10/3Z	None	26(21)	200/3/3Z	MIL-W-4088, Type XII	
Ribbon	2B	2.063	3000	188	200/10/3Z	None	14(14)	200/3/3Z	MIL-W-4088, Type XII	
Tape	1	0.594	1300	54	200/15/3Z	None	24(18)	200/4/4Z	MIL-W-4088, Type XII	
Tape	2A	1.031	3000	188	200/10/3Z	30	200/3/3Z	20(20)	MIL-W-4088, Type XX	
Tape	2B	1.031	3000	94	200/20/3Z	16	200/3/3Z	20(18)	MIL-W-4088, Type XX	

1 Picks/in. - Figures in parentheses are actual picks used.

Allowed maximum widths were exceeded on Webbing 1A (1.81 in.), Webbing 2 (2.18 in.), Ribbon 1 (1.75 in.), Ribbon 2A (2.12 in.), Tape 1 (0.87 in.) and Tape 2A (1.06 in.).

TABLE VIII

## PROPERTIES OF EXPERIMENTAL NARROW FABRICS

<u>Type</u>	<u>No.</u>	<u>Breaking Strength (lb) Average of 3 Specimens</u>	<u>Weight (oz/yd)</u>	<u>Width (in.)</u>	<u>Thickness (in.)</u>
Web	1A	1548	0.88	1.81	0.044
Web	1B	1603	0.81	1.67	0.045
Web	1C	1617	0.81	1.65	0.044
Web	2	9767	5.31	2.15	0.202
Ribbon	1	3100	1.45	1.75	0.073
Ribbon	2A	3130	1.75	2.12	0.061
Ribbon	2B	3813	1.46	2.00	0.061
Tape	1	1403	0.79	0.88	0.073
Tape	2A	3570	1.69	1.05	0.128
Tape	2B	3620	1.69	0.95	0.146

IV. PART 2 - DESIGN AND CONSTRUCTION

Using data developed in Part 1 of this study, the following constructions were specified for the materials to be delivered to the Air Force.

A. Threads - Production constructions were as follows:

<u>Thread</u>	<u>Yarn Used</u>	<u>Twists Used</u>		<u>Amount Made (lb)</u>
		<u>Singles</u>	<u>Ply</u>	
E Thread	100/3/3	12.0S	9.0Z	5.5
FF Thread	200/3/3	8.5S	5.8Z	2.5
3 Cord	200/5/3	6.6S	4.5Z	6.0
6 Cord	200/9/3	5.0S	2.9Z	3.5

The 100 and 200 denier yarns had no twist as received and none was inserted prior to the first plying.

B. Parachute Fabrics

Production constructions were as follows:

<u>Fabric</u>	<u>Warp</u>		<u>Filling</u>		<u>Weave</u>	<u>Yards Produced</u>
	<u>End/in.</u>	<u>Yarn</u>	<u>Picks/in.</u>	<u>Yarn</u>		
2.25-oz	74	100/2.5Z	74	100/2.5Z	MIL-C-7350, Type I	250
4.75-oz	38	2/200/2.5Z	38	2/200/2.5Z	Plain	123

Both warps were sized with Polyfilm TS-20.

Tests on fabrics produced in experimental runs of 2.25-oz fabric indicated weaving tensions had no significant effects on fabric properties as long as good weaving was maintained. Therefore, it was specified that tensions should be so maintained as to achieve the best possible weaving.

C. Narrow Fabrics

The constructions used and amounts produced of the narrow fabrics are given in Table IX. Warps were prepared by Murdock Webbing Company from yarns twisted and plied by Celanese. All the weaving was done by Murdock. No serious processing difficulties were encountered in weaving any of the materials to be delivered to the Air Force.

TABLE IX  
NARROW FABRICS - CONSTRUCTION OF FINAL MATERIALS

Type	No.	Warp		Binder		Filling		Weave	Amount Produced (yd)
		Ends	Yarn	Ends	Yarn	Ficks/in.	Yarn		
Web	1	266	200/3/3Z	None		24	200/3/3Z	Like Type XII of MIL-W-4088	712
Web	2	540	200/10/3Z	27	200/3/3Z	22	200/3/3Z	Like Type XX of MIL-W-4088	350
Ribbon	1	240	200/6/3Z	24	200/3/3Z	16	200/3/3Z	Like Type XX of MIL-W-4088	675
Ribbon	2	328	200/6/3Z	None		16	200/3/3Z	Like Type XII of MIL-W-4088	612
Tape	1	140	200/6/3Z	14	200/3/3Z	18	200/3/3Z	Like Type XX of MIL-W-4088	980
Tape	2	328	200/6/3Z	32	200/3/3Z	18	200/3/3Z	Like Type XX of MIL-W-4088	605

## V. PART 3 - CHARACTERIZATION

### A. General

All tests were carried out in the Applications and Product Development Laboratories of Celanese at Charlotte except the impact and stress-strain tests on the two webbings. These were performed by Fabric Research Laboratories, Dedham, Massachusetts, on sub-contract basis.

In view of the finding that PBI is very moisture sensitive, the procedure was established that required all samples to be oven-dried (110°C) prior to conditioning for testing. All samples were then conditioned for 2 to 3 days at 70°F and 65% relative humidity before testing.

All tests were performed in accordance with standard procedures as described in specification CCC-T-191, Textile Test Methods, except as noted herein.

Testing at elevated temperatures after exposure for various times was carried out on three of the threads and on the 2.25-oz parachute fabric, in accordance with a schedule agreed upon in a meeting with Air Force representatives. A review of the results indicated that strengths at 700°F and above were being adversely affected by conditions not anticipated in early plans. At the request of the Air Force, high temperature test schedules were stopped at this point. Preliminary tests at Wright-Patterson AFB laboratories indicated that burn-off of finish left the yarns and fibers unprotected from internal abrasion between fibers. An amendment to the contract was proposed and approved to investigate and evaluate some candidate high-temperature-resistant finishes. Consequently, none of the other materials were characterized at elevated temperatures. All of the studies on these new finishes were carried out on yarns and lightweight parachute fabric. These trials are described and the results reported in separate Section VI, at the end of this report.

### B. Threads

While the contract required that only 3 threads, sizes E, 3 cord and 6 cord be made, a fourth, size FF, requested by the seaming sub-contractor and approved by the Air Force, was produced and characterized.

Properties of all four threads as produced in the quantities required appear in Table X. Strength requirements as set forth in specification MIL-T-7087 are exceeded in all cases.

High temperature characterization was completed on the FF, 3 cord and 6 cord threads before the testing schedule was interrupted for finish studies. The results of temperature-time studies on these threads are given in Table XI and plotted in Figures 3, 4, and 5.

CAUTION: The data at 550°F and above may not give a true indication of the temperature resistance of PBI. It was apparent from the smoking which developed that practically all finish on these threads was burned off or evaporated during exposure. These data, therefore, apply to substantially nonlubricated material in which fiber-to-fiber abrasive action may create surface imperfections which result in early failure.

Though exposures at 900°F and 1000°F were made, after 10 min at either of these temperatures, the threads were so brittle they could not be gripped in test jaws without shattering.

TABLE X

## PROPERTIES OF PBI SEWING THREADS

<u>Property</u>	<u>E Thread</u>	<u>FF Thread</u>	<u>3 Cord</u>	<u>6 Cord</u>
Construction	100/3/3	200/3/3	200/5/3	200/9/3
Singles twist (tpi)	11.8S	8.8S	6.7S	4.9S
Ply twist (tpi)	9.3Z	5.8Z	3.6Z	2.5Z
Yd/lb	4290	2410	1477	778
Tensile strength (lb) <sup>1</sup>	9.3 (8.5)	18.1 (16.0)	30.4 (24.0)	56.2 (50.0)
Elongation at break (%)	25.1	19.2	19.7	21.8

<sup>1</sup>Strength specifications for MIL-T-7807B, Type I - Machine Twist Threads, are in parentheses. Tensile properties were measured on the Instron using ten breaks for each item. A 10-in. gage length was used with a crosshead speed of 6 in./min.

TABLE XI

## ELEVATED TEMPERATURE BEHAVIOR OF PBI SEWING THREADS

<u>Times</u>	<u>Strength</u> <u>(lb)</u>	<u>Tensile Strength</u> <u>(lb - Average of 3 tests)</u>				<u>Strength Retained</u> <u>(%)</u>			
		<u>350°F</u>	<u>450°F</u>	<u>550°F</u>	<u>700°F</u>	<u>350°F</u>	<u>450°F</u>	<u>550°F</u>	<u>700°F</u>
FF thread	18.1								
10 min		14.8	24.6	11.6	7.9	82	136	64	44
20 min		14.3	15.6	11.0	8.8	79	86	61	49
1 hr		14.5	14.7	11.9	7.3	80	81	66	40
2 hr		16.6	13.5	11.3	7.1	92	75	62	39
20 hr		15.2	12.5	10.5	0.5	83	69	58	3
100 hr		15.9	11.8	8.2		88	65	45	
200 hr		15.0	12.5	8.6		83	69	47	
3 Cord	30.4								
10 min		27	36	20	13	88	118	65	42
20 min		26	27	20	15	86	88	66	50
1 hr		29	26	22	13	94	87	71	42
2 hr		27	23	21	12	88	75	67	40
20 hr		27	23	21	2	90	76	68	5
100 hr		27	22	14		90	72	46	
200 hr		27	22	17		89	73	57	
6 Cord	56.2								
10 min		46	49	37	27	82	88	66	47
20 min		47	49	38	28	84	87	67	50
1 hr		50	44	38	26	89	78	68	46
2 hr		48	44	37	24	86	79	67	43
20 hr		49	44	38	3	88	79	68	6
100 hr		50	40	30		90	71	53	
200 hr		52	49	32		93	87	56	

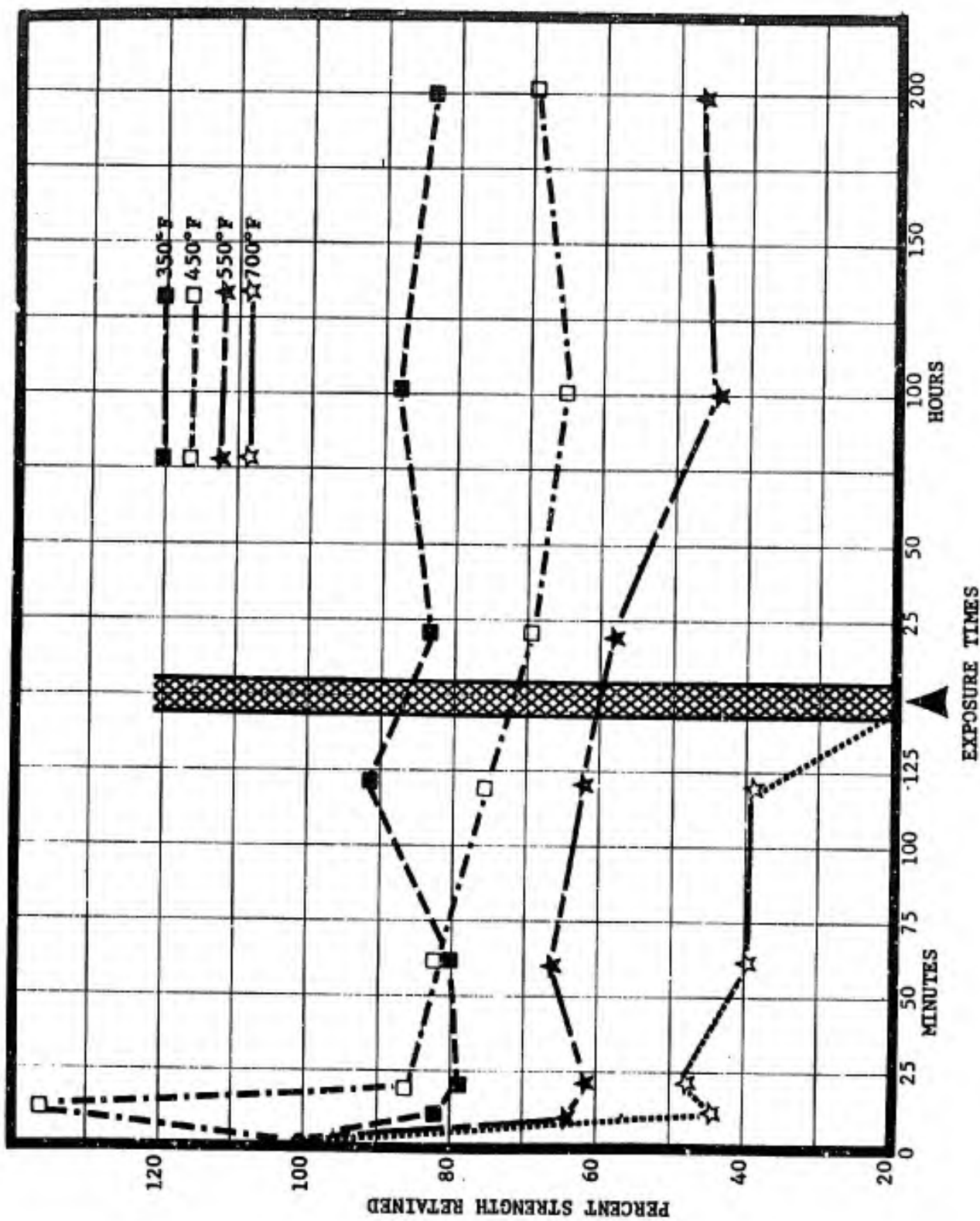


FIGURE 3. STRENGTH RETENTION OF SIZE FF PBI THREADS AFTER HIGH TEMPERATURE EXPOSURES

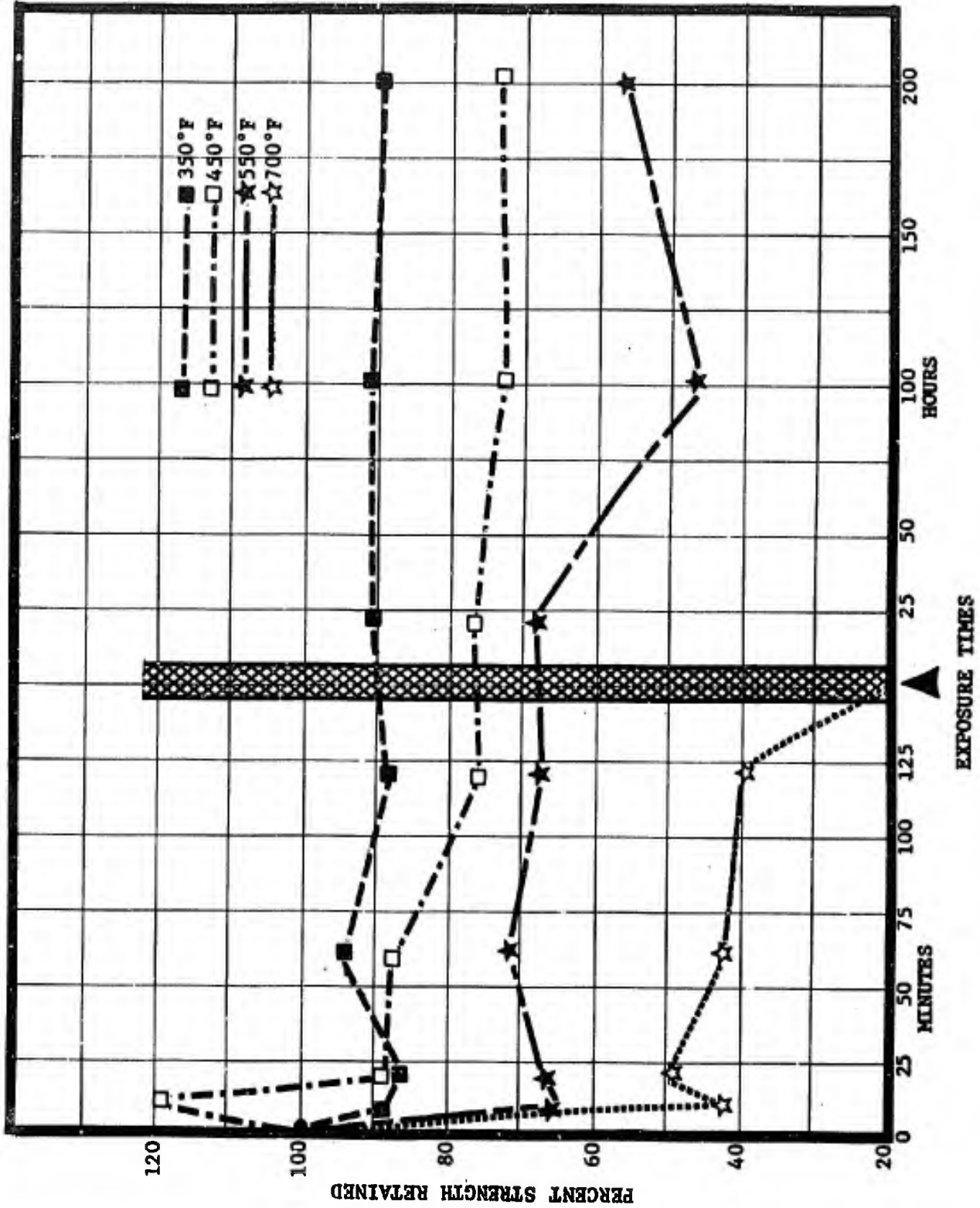


FIGURE 4. STRENGTH RETENTION OF 3 CORD FBI THREADS AFTER HIGH TEMPERATURE EXPOSURES

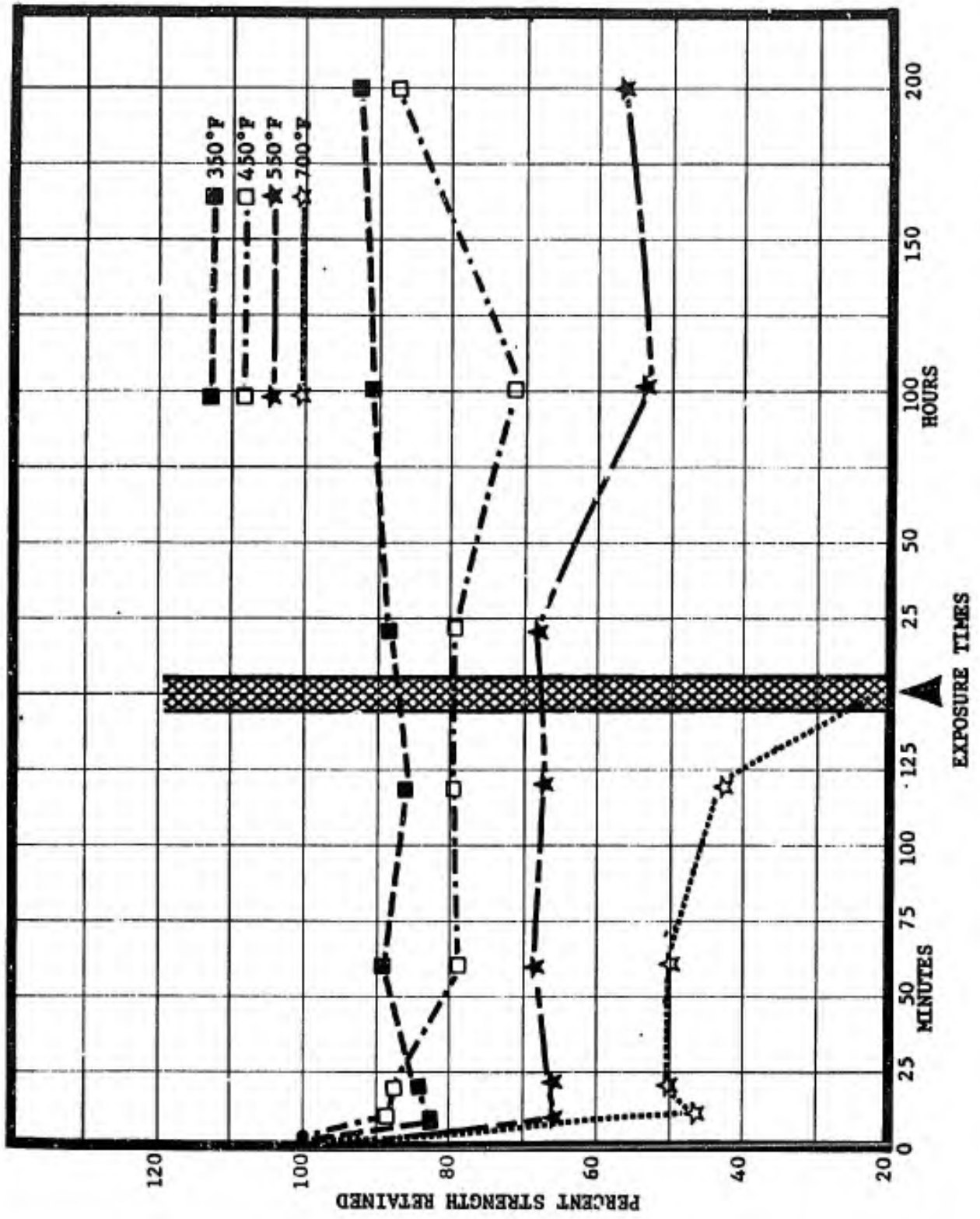


FIGURE 5. STRENGTH RETENTION OF 6 CORD PBI THREADS AFTER HIGH TEMPERATURE EXPOSURES

### C. Fabrics

The physical properties of both fabrics at ambient conditions appear in Table XII. The air permeabilities at various pressures are plotted in Figure 6. As would be expected with a yarn of tenacity of 4.5 gm/den, tensile characteristics are somewhat lower than for similar fabrics made of nylon. Other characteristics appear to be within specification limits.

The lightweight fabric was subjected to a complete series of elevated temperature-extended time exposures and tests were made on all samples which could be handled after exposure. Results appear in Table XIII and are graphically shown in Figures 7 and 8. (CAUTION: It must be reiterated that results at 550°F and above may be subject to error due to evaporation of finish during exposure.) After about 50 hr at 700°F and only 10 min at 900°F, the samples were so badly charred and brittle that they could not be mounted in the tester jaws. Attempts were made to measure the shrinkage of these samples during exposure. The data obtained appears in Table XII.

A review of these data with Air Force representatives led to revision of the contract to substitute investigation of high-temperature-resistant finishes for the remainder of the scheduled evaluated temperature testing. These studies are reported in the last section of this report.

In an attempt to determine whether oxidation by atmospheric oxygen was a contributor to high temperature degradation, duplicate samples were exposed in air and in nitrogen at 700°F for 20 hr and then tested. Results, appearing in Table XIV, indicate no significant differences between exposure in air and nitrogen.

Inasmuch as several of the procedures used in these tests had to be developed, descriptions thereof are attached as Appendix I.

TABLE XII  
PROPERTIES OF FABRICS

<u>Property</u>	<u>2.25 Oz/Yd<sup>2</sup> Fabric</u>	<u>4.75 Oz/Yd<sup>2</sup> Fabric</u>
<b>Construction:</b>		
Warp: (ends/in.)	75	39
Yarn	100 den - 2.6 tpi Z	2 ply 200 den - 3.0 tpi Z
Filling: (picks/in.)	75	39
Yarn	100 den - 3.3 tpi Z	2 ply 200 den - 3.0 tpi Z
Weave	Type I of MIL-C-7350	Plain weave
<b>Weight (oz/yd<sup>2</sup>)</b>	2.22	4.79
<b>Tensile Values</b>		
<b>Grab Method:</b>		
Strength (lb/in.-WxF)	91 x 95	227 x 281
Elongation (%-WxF)	20 x 26	23 x 27
<b>Strip Method:</b>		
Strength (lb/in.-WxF)	65 x 72	140 x 167
Elongation (%-WxF)	18 x 24	19 x 27
Energy Absorption (in.-lb-WxF)	38 x 54	69 x 111
<b>Slit Seam Tests:</b>		
Fabric Strength (lb/in.-WxF)	61 x 58	139 x 161
Seam Strength (lb/in.-WxF)	55 x 50	122 x 138
Seam Efficiency (%-WxF)	90 x 86	88 x 86
<b>Air Permeability (ft<sup>3</sup>/min/ft<sup>2</sup>)</b>		
@0.5 in water pressure	119	65
@1.0        "	164	
@2.0        "	286	109
@5.0        "	600	201
@7.5        "	767	
@10.0       "	890	336
@12.5       "	1056	
@15.0       "	1082	455
@17.5       "	1182	
@20.0       "	1264	560

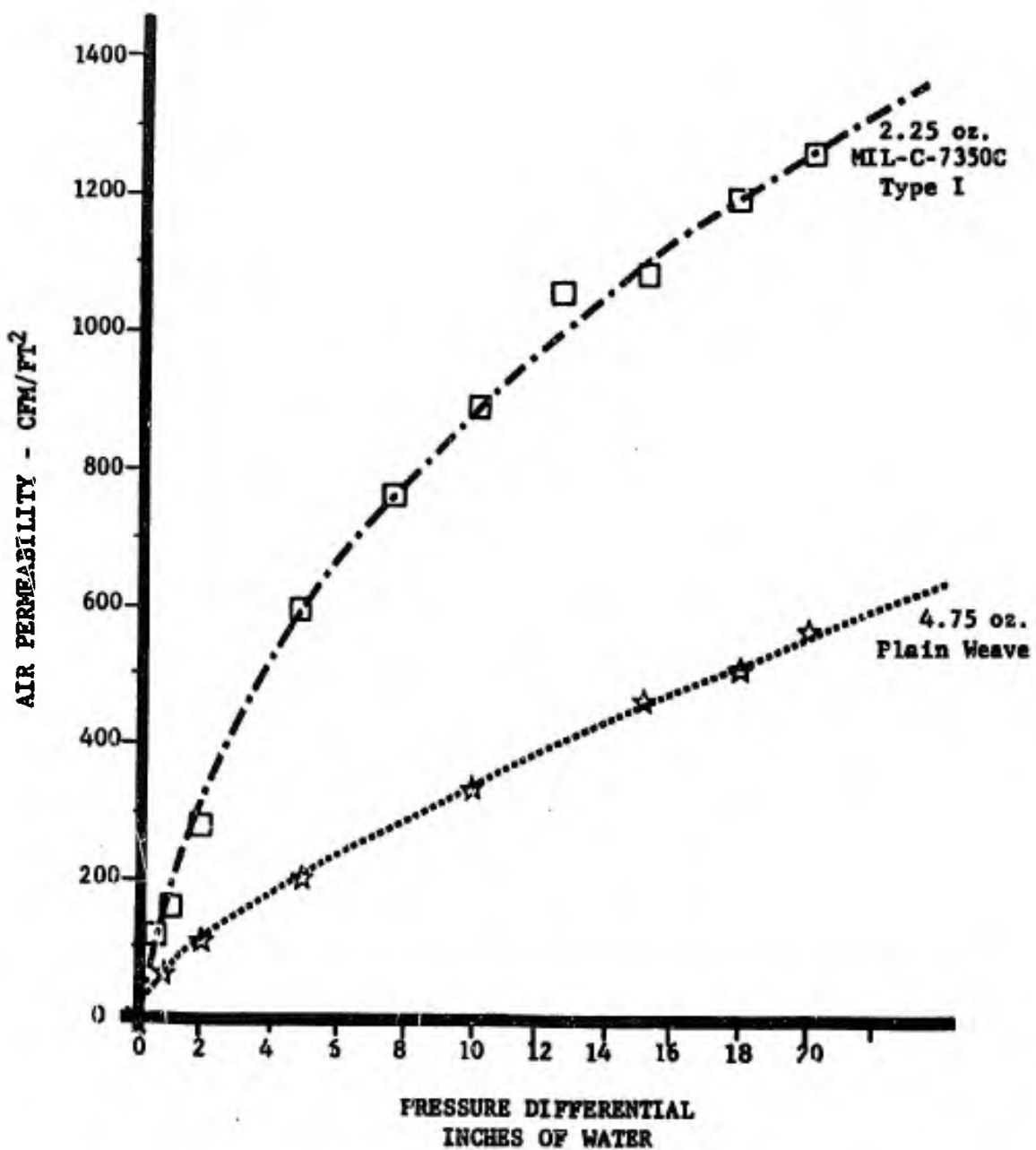


FIGURE 6. AIR PERMEABILITY OF PBI FABRICS

TABLE XIII

## ELEVATED TEMPERATURE BEHAVIOR OF PBI FABRIC - 2.25 OZ.

Time/Temp.	Strip Tensile Strength (lb/in.)										
	70°F		450°F		550°F		700°F		900°F		1000°F
	W	F	W	F	W	F	W	F	W	F	
0	65	60									
10 min			40	43	26	33	27	28	Fail	Fail	
20 min			42	43	25	28	22	26			
1 hr			41	43	23	27	16	20			
2 hr			35	33	25	24	15	22			
20 hr			25	32	24	28	2	3			
100 hr			34	42	17	17	0(50) <sup>1</sup>	0(50) <sup>1</sup>			
200 hr			24	31	16	16					

Time/Temp.	% Strength Retained at							
	450°F		550°F		700°F		900°F	
	W	F	W	F	W	F	W	F
10 min	61	72	40	55	41	47	0	0
20 min	65	72	38	47	34	43		
1 hr	63	72	35	45	25	33		
2 hr	54	55	38	40	23	37		
20 hr	38	53	37	47	3	5		
100 hr	52	70	26	28	0(50) <sup>1</sup>	0(50) <sup>1</sup>		
200 hr	37	52	25	27				

Time/Temp.	% Shrinkage at					
	450°F		550°F		700°F	
	W	F	W	F	W	F
10 min	.5	.4	1.0	1.0	2.2	1.6
20 min	.5	.6	1.1	1.2	2.4	1.8
1 hr	.7	0	1.0	.6	3.7	3.0
2 hr	.5	.6	2.8	3.0	4.0	3.4
20 hr	1.0	1.0	1.2	.6	8.3	9.1
100 hr	.9	.6	2.4	1.8		
200 hr	1.1	1.2	3.0	2.4		

<sup>1</sup>0(50) indicates sample was so badly charred after 50 hr exposure, it could not be handled and had no strength.

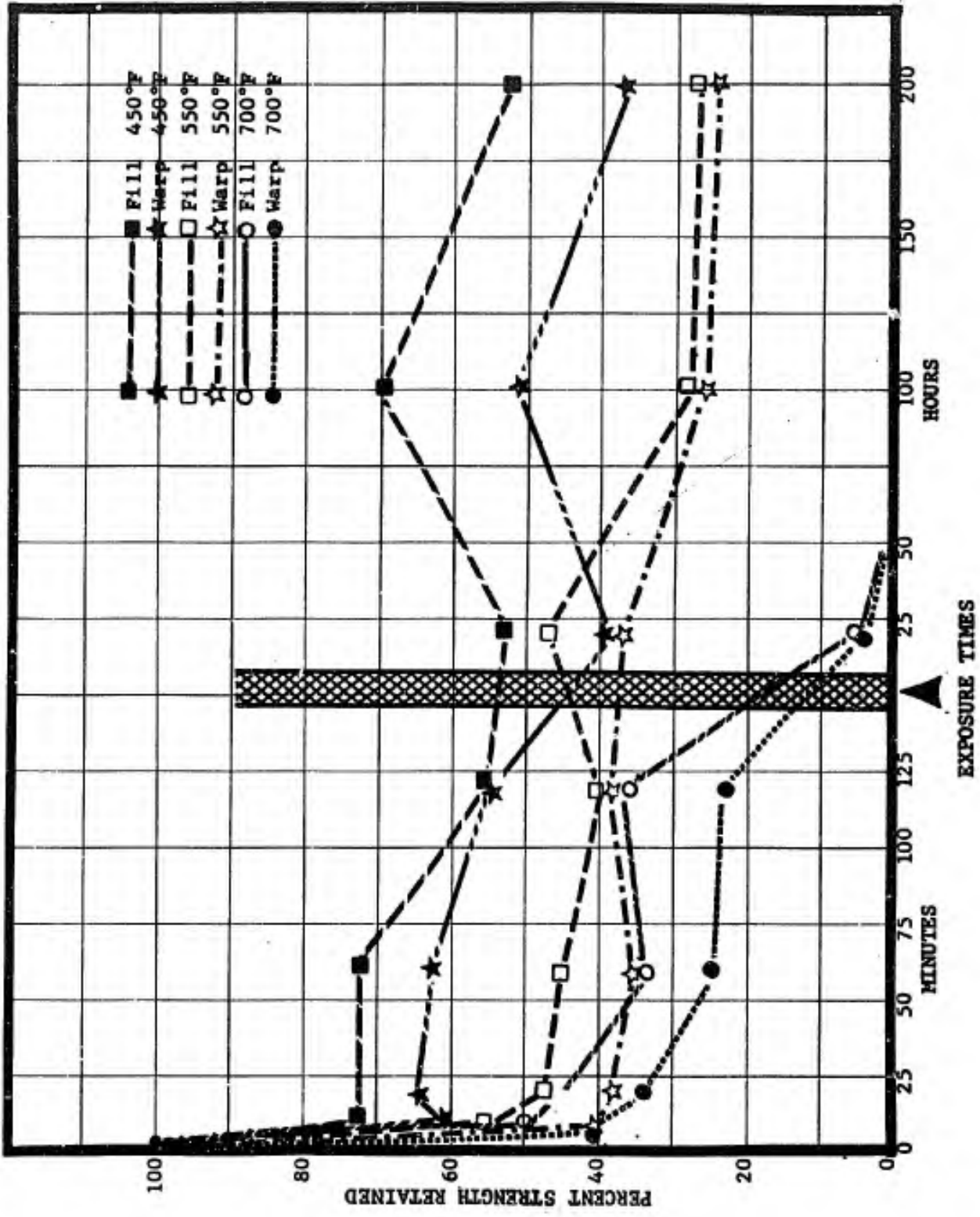


FIGURE 7. STRENGTH RETENTION OF 2.25 OUNCE F11 FABRIC AFTER HIGH TEMPERATURE EXPOSURES

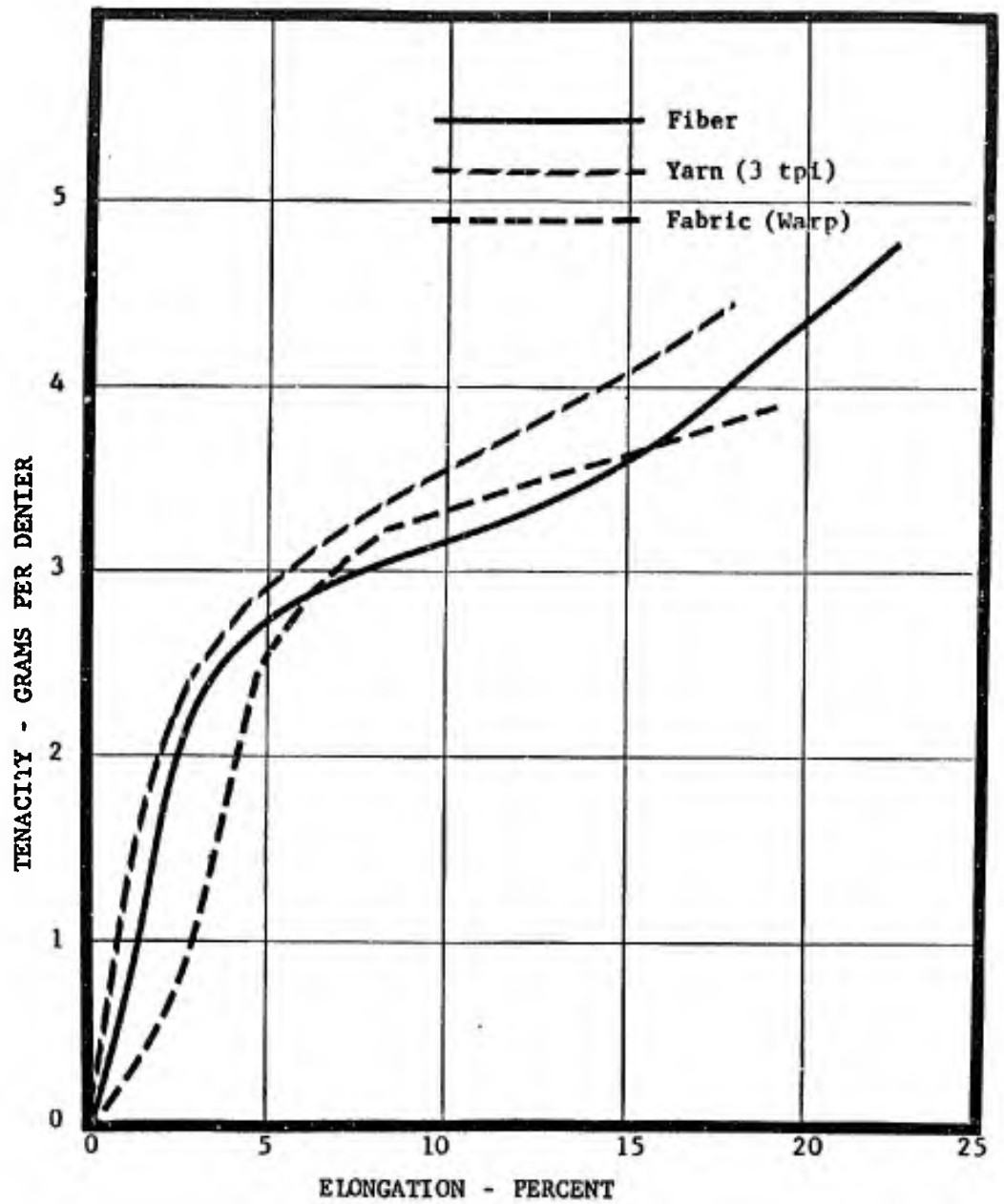


FIGURE 8. TYPICAL STRESS-STRAIN DIAGRAMS OF PBI FIBERS, YARNS AND FABRICS

TABLE XIV  
 TENSILE PROPERTIES OF PBI FABRIC AFTER  
 700°F EXPOSURE IN AIR AND NITROGEN ATMOSPHERES

	<u>Tensile Properties<sup>1</sup></u>	
	<u>Tensile Strength (gm)</u>	<u>Elongation (%)</u>
Original filling yarns	409	30
After exposure in air at 700°F for 20 hr	90	3
After exposure in nitrogen at 700°F for 20 hr	71	3

---

<sup>1</sup>Tensile properties were determined on Instron on yarns teased just prior to testing. All yarns were conditioned and tested at 70°F, 65% relative humidity, 3.33 in. gage length, 2 in./min crosshead speed.

#### D. Narrow Fabrics

The physical properties of the narrow fabrics produced for delivery to the Air Force, are summarized in Tables XV and XVI. The impact, elongation and energy absorption data given for the two webbings were obtained from tests made on subcontract by Fabric Research Laboratories, Dedham, Massachusetts. Samples for seam efficiency determination were made on subcontract by Pioneer Parachute Company, Manchester, Connecticut. For comparison, requirements specified by the contract are also listed in this table. The only deviation of any consequence, the increased width of webbing 2, was permitted by request to the Air Force in order to retain some degree of flexibility. No elevated temperature-extended exposure tests were made on these materials.



TABLE XVI

COMPARISON BETWEEN STATIC AND DYNAMIC ENERGY ABSORPTION OF  
PBI WEBBINGS AND EIGHT-INCH OVERLAP SPLICES MADE THEREFROM(All Energies in Units of Ft-Lbs/Ft/Denier  $\times 10^{-4}$ )

Sample Identification	Static (2 in./min)	Dynamic		
		210 $\pm$ 10 ft/sec	430 $\pm$ 20 ft/sec	650 $\pm$ 50 ft/sec
1200-lb Control	11.91	5.39	5.44	2.35
		5.59	4.16	3.43
		5.59	4.21	2.96
		Average 5.53	Average 4.62	Average 2.96
1200-lb Overlap	---	5.67	6.03	2.62
		5.15	3.39	1.31
		4.74	4.86	1.88
		Average 5.19	Average 4.62	Average 2.09
9000-lb Control	13.52	6.42	7.86	3.04
		7.02	9.02	2.06
		6.53	8.11	2.43
		Average 6.65	Average 8.34	Average 2.51
9000-lb Overlap	---	7.21	8.86	2.52
		6.60	8.45	2.01
		6.94	9.37	2.08
		Average 6.92	Average 8.89	Average 2.20

## VI. FINISH STUDIES

### A. Preliminary Studies

Samples of 2.25-oz fabric were furnished to Fibrous Materials Branch, Wright-Patterson Air Force Base, Ohio. Tests made on these samples indicated that preloading and maintenance of load during high temperature exposure resulted in higher ultimate breaking strength than when loading was not used. These results indicated that the loading immobilized the fibers to some degree, thereby preventing fiber-to-fiber abrasion which undoubtedly could damage the fibers and reduce breaking strength. When these results appeared, high temperature testing was stopped to permit studies of yarn finishes which would retain lubricity and stay on the fiber at the high temperatures under study. A proposal was made and accepted by the Air Force to substitute finish studies for the remaining high temperature testing.

At Fibrous Materials Branch request, Fabric Research Laboratories made some tests that appeared to confirm their results. The results of all tests made at this time are summarized in Table XVII.

### B. Establishment of Program

The program to be carried out was planned as follows:

(1) Collection of finish candidates. A comprehensive investigation of materials which might prove satisfactory was to be made and sample quantities of those available were to be acquired.

TABLE XVII

700°F EXPOSURE TESTS ON 2.25-OUNCE FABRIC  
(All tests on 1 inch raveled strip - warpwise)

At Celanese Laboratories:

- |  |                    |
|--|--------------------|
| 1. At ambient conditions:  | Broke at 65 lb/in. |
| 2. At 700°F - sample placed in preheated chamber, held 10 min and broken without removal from chamber  | Broke at 27 lb/in. |
| 3. At 700°F - sample placed in preheated chamber, held 20 min and broken without removal from chamber. | Broke at 22 lb/in. |

At Fibrous Materials Branch Laboratories:

- |   |  |
|---|--|
| 1. At ambient conditions:   | Broke at 58 lb/in.                                 |
| 2. At 200°F, sample preloaded to 30 lb, inserted in heated chamber, temperature raised to 700°F in 20 min, load applied to break. | Broke at 40 lb/in.                                 |
| 3. Same as 2 except 40 lb load used   | Broke when temperature reached 600°F, at 40 lb/in. |
| 4. Same as 2 except 35 lb load used, held at 700° for 20 min and broke without additional loading.                                | Broke at 35 lb/in.                                 |

At Fabric Research Laboratories:

- |   |                      |
|---|----------------------|
| 1. Sample exposed 10 min at 700°F, no preload   | Broke at 34.2 lb/in. |
| 2. At 300°F, sample preloaded to 30 lb but load allowed to decay, temperature raised to 700° in 6 min, held for 10 min, total elapsed time in chamber 16 min, load applied to break | Broke at 38.1 lb/in. |
| 3. Repeat of 2 without load.  | Broke at 33.9 lb/in. |

(2) Application of candidate materials. Attempts were to be made to apply the materials at two levels of application under the following conditions:

- (a) Over finishes already applied.
- (b) After removal of finishes already applied.
- (c) After exposure to high temperatures.

(3) Assessments of finishes were to be made by determining effects on breaking strength after exposing samples treated as in (2) to high temperatures.

(4) Studies of other factors possibly contributing to increased breaking strength after exposure to high temperatures under load were to be made on both yarns and fabrics. These include:

- (a) Studies of tensile properties of lubricated and finish-free yarns, both twisted and non-twisted.
- (b) Comparison of tensile properties of fabric with size and finish and after removal of size and finish.
- (c) Comparison of tensile properties of light braid made from lubricated and finish-free yarns. Braid should provide the ultimate in inter-fiber and inter-yarn friction.
- (d) Testing of yarns and fibers exposed to 700°F for 10 min under various loads.
- (e) Testing of fabric exposed to 700°F for 10 min under various loads.
- (f) Testing of greige and scoured fabrics after exposure to 700°F for 10 min.
- (g) Testing of braid after exposure to 700°F for 10 min under various loads.

#### C. Finishes Investigated

The following finishes and finish components were used in this study.

1. FF-400, silicone type fiber finish, produced by Dow-Corning Corporation, Midland, Michigan.
2. ET-4327 - silicone type finish recommended for application to glass fabrics for exposure to temperatures up to 600°F, produced by Dow-Corning Corporation.
3. Therminol FR3 - high boiling, heat stable chlorinated biphenyl liquid, produced by Monsanto Chemical Company, St. Louis, Missouri.
4. Nopco PSB, blended, high temperature stable finish with low friction at elevated temperatures, produced by Nopco Chemical Company, Newark, New Jersey.

5. Nopcote C-104, nonionic calcium stearate dispersion produced by Nopco Chemical Company.
6. Nopcostat HS, heat stable antistatic agent, produced by Nopco Chemical Company.
7. Nopco BHT Anti-Oxidant, butylated hydroxytoluene produced by Nopco Chemical Company.
8. PL-5, organic phosphate, 2-biphenyl diphenyl phosphate produced by Dow Chemical Company.
9. Attempts made to obtain and apply molybdenum disulfide materials were unsuccessful. Those obtained could not be applied in any practical manner.

Application methods and details appear in Appendix II of this report.

#### D. Tests on Fabric

The materials listed in Section C above were applied to greige and scoured 2.25-oz fabrics both before and after exposure at 700°F for 10 min. The target applications were approximately 3 and 6% on weight of fabric. Actual applications are given in Appendix II. Results of breaking tests on all samples appear in Table XVIII. These tests were all made under ambient conditions, samples being reconditioned after exposure but before testing. The points of interest are that none of the finishes had a beneficial effect on strength and, with only a few exceptions, scouring improved breaking strength, particularly for the samples to which finish was applied after exposure at 700°F for 10 min. These results indicate that the presence of either the original finish or the size or both had some deleterious effect on strength at high temperatures.

For further testing, one of the silicones, FF-400, the calcium stearate, C-104, and the organic phosphate, PL-5, were applied to scoured fabric at the 3% application level and the samples exposed at 700°F for various times up to 2 hr. Samples were reconditioned and tested under ambient conditions. The results appear in Table XIX. PL-5 appeared to provide a significant improvement in strength for exposure times over one hour while the other two materials gave improved flex abrasion. FF-400 gave longer lasting improvement in the latter property, the effect persisting up to 30 min exposure time. These results suggest that use of a formulation which includes both FF-400 and PL-5 might provide the best results.

Exposure of samples of fabric under various loads gave results shown in Table XX. Because of heating chamber limitations, test conditions used by Fibrous Materials Laboratories or Fabric Research Laboratories could not be duplicated. No means could be devised in the

TABLE XVIII

700°F EXPOSURE TESTS<sup>1</sup> ON 2.25-OUNCE FABRIC WITH VARIOUS FINISHES

Lubricant	Level (%) (On Weight of Fabric)	Lubricants Applied							
		Before Exposure			After Exposure				
		Scoured Tensile Strength (lb/in.)	Elong. (%)	Greige Tensile Strength (lb/in.)	Scoured Tensile Strength (lb/in.)	Elong. (%)	Greige Tensile Strength (lb/in.)	Elong. (%)	
Control (not heated)	0	67	19	65	18	67	19	65	18
Control (exposed)	0	70	16	66	13	70	16	66	13
FF-400	3	65	15	65	18	64	17	54	10
	6	64	16	63	17	64	16	53	10
ET-4327	3	61	12	60	15	64	16	54	10
	6	59	12	62	15	64	14	53	11
Therminol	3	63	14	56	10	61	12	51	8
	6	62	13	54	9	62	13	52	9
Nopco PSB	3	62	14	62	14	62	14	50	9
	6	65	16	62	15	62	14	52	10
Nopcone C-104	3	66	15	68	19	64	13	56	11
	6	69	16	62	14	64	15	52	11
Nopcostat HS	3	63	13	66	17	64	14	54	10
	6	64	14	63	14	60	13	56	11
Nopco BHT	3	65	15	57	12	63	14	53	8
	6	63	14	58	12	63	14	55	10
PL-5	3	67	16						
	6	61	15						

<sup>1</sup>10 min at 700°F, fabrics conditioned and tested at room temperatures.

TABLE XIX

EFFECT OF FINISHES ON FABRIC STRENGTH AND FLEX ABRASION  
AFTER AGEING AT 700°F

<u>Ageing Time</u>	<u>Warp Strip Tensile Strength (lb/in.)<sup>1</sup></u>			
	<u>Finishes<sup>2</sup></u>			
	<u>1. None</u>	<u>2. FF-400</u>	<u>3. PL-5</u>	<u>4. Nopcote C-104</u>
0 (original)	68	66	67	65
10 min	68	66	67	68
30 min	54	56	58	55
60 min	50	46	49	49
120 min	34	37	44	35

<u>Ageing Time</u>	<u>Flex Abrasion<sup>3</sup> (Warp)-Cycles to Break</u>			
	<u>1. None</u>	<u>2. FF-400</u>	<u>3. PL-5</u>	<u>4. Nopcote C-104</u>
0 (original)	1189	2662	1138	2731
10 min	298	397	176	216
30 min	34	84	41	37
60 min	1	2	2	2
120 min	0	0	1	1

<sup>1</sup>All samples reconditioned and tested at ambient conditions after exposure.

<sup>2</sup>Applied to scoured fabric, 3.0% on weight of fabric.

<sup>3</sup>Tested by American Society for Testing Materials, Test Specification D1175 - "Flexing and Abrasion Method", using a Stoll Flex Abrasion Tester.

TABLE XX

FABRIC PROPERTIES<sup>1</sup> AFTER EXPOSURE UNDER LOADS AT 700°F FOR 10 MINUTES

<u>Conditions</u>	<u>Tensile Strength (lb/in.)</u>	<u>Elongation (%)</u>
Original Fabric, unexposed	65	18
Fabric Exposed, no load	70	16
Fabric Exposed under 6.5 lb load	64.1	6.7
Fabric Exposed under 16 lb load	62.5	5.6
Fabric Exposed under 26 lb load		
Sample 1 - Stretched to bottom of oven during exposure	47.4	4.2
Sample 2 - Broke under load during exposure	19.3	5.8

<sup>1</sup>Tested in warp direction after reconditioning at ambient conditions.

time available to preload, hold under load during exposure and test without removal of load. Attempts to preload and hold under load with the Instron resulted in so much sample movement that no reliable results could be obtained. For these reasons, dead loads were used during exposure and the samples were removed and then conditioned and tested under standard laboratory conditions. The two ends of the sample which broke in the oven were tested and the data are given in Table XX for information. Attempts to expose samples to 900°F produced such serious charring and embrittlement that breaking tests could not be made.

#### E. Tests on Yarns, Fibers and Braids

(1) Comparison of lubricated and unlubricated yarns and fibers - Studies on yarns and single fibers were carried out on one package each of lubricated (with Nopco 1296, a typical nylon finish made by Nopco Chemical Company) and unlubricated yarn. A portion of each was twisted to 2 tpi Z. The twisted and untwisted portions and a complete cross section of single fibers from the untwisted portion were tested for both straight and loop characteristics. In addition, to obtain data on the effects of bending angle, the untwisted yarns and single filaments were loop tested over mandrels of 0.05 and 0.01 in. diameter. Results of these tests appear in Table XXI. The only property affected by twisting was elongation but these effects were quite erratic. The use of mandrels of appreciably larger diameter than the yarns and filaments in loop testing resulted in considerable increases in both tenacity and elongation of both lubricated and unlubricated yarns. This indicates that the yarn and fibers are quite stiff even before exposure to high temperatures. The differences between lubricated and unlubricated yarns are of no significance but unlubricated single filaments show a greater increase in tenacity over the mandrels than lubricated yarn, indicating that the lubricant used affects the stiffness to some small extent. It is reasonable to conclude that some slight plasticizing action occurs that could adversely affect high temperature resistance.

(2) Effects of exposure to high temperature while under loads - Yarns, single fibers and braids, both lubricated and unlubricated were exposed to 700°F for 10 min while loaded to various percentages of predetermined breaking strengths. These were then reconditioned and tested under standard laboratory conditions. Results appear in Table XXII. Despite the fact that the data are somewhat erratic, some conclusions can be drawn therefrom. The first is that if PBI material is cooled at ambient conditions after exposure to 700°F for 10 min breaking strength is affected to only a small extent. The second is that exposure to high temperature while under load reduces elongation in direct proportion to the load applied. This appears to result from the fact that even light loads stretch the fibers fairly easily at elevated temperatures. Note the reduction in filament denier which occurs when single filaments are exposed. The increased tenacities as loads are increased result almost solely from denier reduction while actual breaking strength varies very little. Another striking result is the rapid deterioration of loop tensile properties of both yarn and braid as loads during exposure are

TABLE XXI

## COMPARISON OF TENSILE PROPERTIES OF LUBRICATED AND UNLUBRICATED YARNS AND SINGLE FIBERS

	Unlubricated			Lubricated		
	0 Twist	2Z Twist	Single Fibers	0 Twist	2Z Twist	Single Fibers
Denier	105.0	107.1	4.10	118.6	120.1	4.72
Tensile Strength (gm)	405.2	413.5	18.9	453.5	469.5	19.7
Tenacity (gm/den)	3.86	3.87	4.62	3.82	3.91	4.18
Elongation (%)	10.9	16.31	14.2	29.4	18.4	30.5
Loop Strength (gm)	572.4	602.0	24.7	691.1	687.7	29.4
Loop Tenacity (gm/den)	2.72	2.81	2.94	2.92	2.87	3.12
Loop Elongation (%)	3.2	4.2	.88	17.2	4.8	2.1
.05 in. Mandrel						
Loop Strength (gm)	816.2		38.8	900.4		37.5
Loop Tenacity (gm/den)	3.88		4.74	3.80		3.97
Loop Elongation (%)	15.1		19.7	27.5		28.9
.01 in. Mandrel						
Loop Strength (gm)	809.4		37.8	882.6		37.6
Loop Tenacity (gm/den)	3.85		4.61	3.72		3.98
Loop Elongation (%)	13.6		20.7	25.7		29.7

Yarn tests are based on 10 breaks.

Single fiber tests are based on 25 breaks for straight tests and 15 for loop tests.

Insofar as possible, yarn and single fiber data were taken from tests on the same yarn package.

increased. This demonstrates the strong stiffening effect that high temperature has on PBI fiber. It does not appear that the presence or absence of the lubricant used has appreciable bearing on this effect. As a matter of fact, most of the characteristics appear to be related more closely to original yarn characteristics than to presence or absence of the lubricant. The only exception has to do with the tensile strength of the braids. Tensile strength of braid made from lubricated yarn increased with loading increases while that of braid made from unlubricated yarn decreased. This may have resulted from the braiding process itself. Unlubricated yarn would naturally suffer more damage in braiding than would lubricated and this damage would appear increasingly as loads are increased. These data probably indicate only the importance of lubricant on the yarn during processing.

Some additional data obtained on braids with and without PL-5 lubricant and tested at exposure temperatures of 700°F and 800°F without cooling appear in Table XXII. These data indicate that holding at 700°F during testing has much more effect on breaking strength than prolonged exposure to 700°F followed by testing at ambient conditions. On the other hand, samples exposed to 800°F for as little as 5 min could not be tested after cooling to ambient conditions. Though fibrous character was retained, embrittlement was so great that test jaws could not be clamped onto the material without crushing it. The PL-5 lubricant used in these tests appeared to have little effect on strength properties.



TABLE XXII (continued)  
EFFECTS OF LOADING DURING HIGH TEMPERATURE EXPOSURE OF SINGLE FIBERS, YARNS AND BRAIDS

	Unlubricated Braid				Lubricated Braid					
	Tensile Strength		Elong.	Loop Tensile	Tensile Strength		Elong.	Loop Tensile		
	(lb)	% of Orig.	(%)	(lb)	(lb)	% of Orig.	(%)	(lb)		
Unexposed Material	13.4	100	13.9	10.0	100	100	20.2	12.0	100	11.6
Exposed <sup>1</sup> Material										
% of original strength applied during exposure										
0	14.0	104	14.8	9.0	90	105	20.9	8.8	73	5.1
10	13.2	98	4.7	5.2	52	102	6.0	5.5	47	1.9
30	12.4	93	3.7	3.7	37	111	6.5	4.0	33	1.2
Other tests -										
Tested at 700°F after holding for 10 min	4.8	34				31				
Tested at 800°F after holding for 10 min	1.1	8				7				

<sup>1</sup>Exposures were 700°F for 10 min.

<sup>2</sup>With 6.2% PL-5 lubricant applied to unlubricated braid by dipping in 2% solution of PL-5 in methylene chloride and drying at 212°F for 5 min.

TABLE XXIII

## TRANSLATION EFFICIENCIES OF PBI (LUBRICATED ITEMS)

<u>PBI Item</u>	Translation Efficiencies (%) Based Upon		
	<u>Fiber Strength</u>	<u>Zero Twist Yarn Strength</u>	<u>3Z Twist Yarn Strength</u>
Bases Used: 100 den*	19.7 gm	451 gm	469 gm
200 den*	19.9 gm	934 gm	969 gm
<u>Yarns</u>			
100/0/25	91.5		96.1
100/3Z/25	95.2	104.0	
200/0/50	93.8		96.4
200/3Z/50	97.4	103.7	
<u>Sewing Threads</u>			
E	95.2	104.0	100.0
FF	91.7	97.8	94.1
3 Cord	92.5	98.6	94.9
6 Cord	95.0	101.3	97.5
Braid	86.4	94.3	90.7
<u>Parachute Fabric</u>			
2.25 oz (WxF)	80.0 x 73.7	87.4 x 80.5	84.0 x 77.4
4.75 oz (WxF)	81.8 x 97.7	87.2 x 104.1	83.9 x 100.2
<u>Narrow Fabrics</u>			
Webbing 1	88.7	94.6	91.0
Webbing 2	80.7	86.0	82.8
Ribbon 1	91.7	97.8	94.1
Ribbon 2	88.3	94.1	90.6
Tape 1	86.5	92.2	88.7
Tape 2	89.5	95.4	91.8

\*Nominal Denier

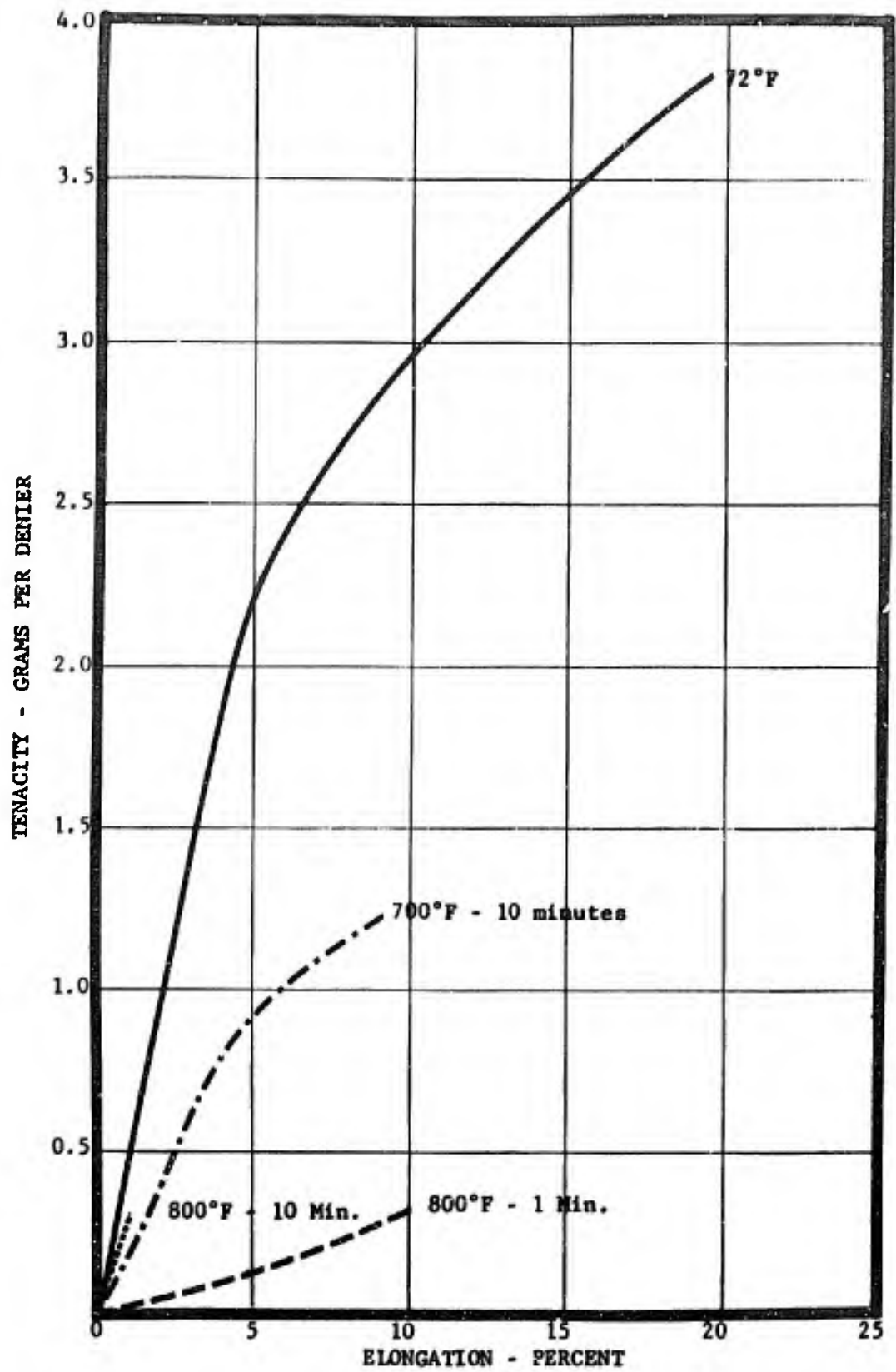


FIGURE 9. TYPICAL STRESS-STRAIN DIAGRAMS OF PBI BRAIDS AT VARIOUS CONDITIONS

## VII. DISCUSSION AND CONCLUSIONS

A. Processability of PBI yarns into fabrics - No difficulties were encountered in twisting, plying, warping or weaving PBI yarns with about 2.5% of Nopco 1296 lubricant. Early attempts to weave low twist yarns without size produced excessive filament breakage. The first attempt at sizing, using the best known nylon size formula, was successful. No further process difficulties were noted and waste was not excessive at any stage of operation.

B. Strength translation of yarns through processing - Strength translations from fiber to yarn to fabrics were generally good. In fact, late in the program it became apparent that breaking strength of fabrics could be closely predicted on the basis of one pound per one hundred denier of yarn used in the fabric. Stress-strain curves and strength translation data are presented in Figure 9 and Table XXIII, respectively. Dynamic properties as determined by limited testing appear to be similar to Nomex.

C. While the high temperature and extended time test program was interrupted to permit some studies of yarn finishes, sufficient testing was done to show that PBI fabrics retain a substantial degree of strength after exposure to 700°F for longer than 2 hr. Sufficient work was done to show that improvement of this degree of strength retention may be attained through use of finishes which are also resistant to high temperatures. At least one lead suggesting a "possible combination of PL-5, an organic phosphate and FF-400, a silicone", was developed toward formulation of a better lubricant than the one applied in producing the fiber used in this investigation. It was also shown that straight strength is recovered, while loop strength and elongation are not when samples exposed for 10 min at 700°F are returned to ambient conditions for testing. The critical exposure temperature appears to lie between 700° and 800°F since exposure at 800°F for 5 min results in excessive charring.

In summation, PBI yarn appears to be an excellent candidate for service at temperatures up to 700°F for periods of 2 hr or longer. There are indications that a suitable high temperature resistant finish would improve its performance. It should be made certain that such finish does not impair its good processing qualities.

## APPENDIX I

### FABRIC TEST METHODS (ALL TESTS AT 70°F, 65% R.H.)

Grab Tensile Strength - Determined on the Instron Tensile Tester with Instron DJ jaws with 3 in. x 2 in. steel face rear jaws for both clamps, and 1 in. x 2 in. steel face front jaw for both clamps. Masking tape (3M #202) was used to line jaws. A gage length of 3 in. and a crosshead speed of 12 in./min were used to break 5 specimens for each determination. Federal Spec. CCC-T-191b, Method 5100.

Strip Tensile Strength - The ravel strip method as described in Federal Specification CCC-T-191b, Method 5104 was used. The Instron with DJ jaws, (3 in. x 2 in. faces on the rear and front of both clamps), a 3 in. gage length, and a 12 in./min crosshead speed were used with 5 replicates for each determination. An integrator was used with the Instron recorder for determining energy absorption.

Slit Seam Test - A test developed by Coplan at Fabric Research Laboratories (reported in "Handbook of Fibrous Materials", WADD Techn'cal Report 60-584, p. 244) was used with slight modification. A longer panel of fabric (8 in. rather than 6 in.) was used to accommodate the deeper 2 in. jaws. Also Size E PBI thread was used for sewing the LSC-2 (Type 401 stitch) seams for both the 7350-I and 8021-I fabric. Eleven stitches per inch were used for both fabrics, since this seemed to be optimum according to the above referenced report for these types of fabrics with Size E thread.

Figure 10 illustrates the sample layout with respect to slit and jaw placement. As indicated, samples, without seams, were slit and their breaking strength considered as a 100% value. A gage length of 3 in. and a crosshead speed of 12 in./min were used. A total of 5 breaks was used for determining each value, the actual load at break was divided in half to convert to a 1 in. basis. Values were not used if the break did not occur at the seam, sewing thread failures were not observed, but there was occasional tearing originating from the slits.

Air Permeability - Federal Specification CCC-T-191b, Method 5450 was employed using a Frazier permeometer. Modifications of the equipment were necessary for pressure drops of more than 1.0 in. across the fabrics. Above a pressure drop of 1.0 in., an open end manometer containing water was used rather than the inclined oil manometer for measuring the pressure differential across the cloth. For pressure drops greater than 7 in., the suction fan and motor were removed and a high capacity vacuum pump (ordinarily used to supply 25 in. of water vacuum for an NBS carpet abrasion tester) attached. The pressure was regulated with a large (3 in.) gate valve in the vacuum line. Five (5) areas of fabric were used at each pressure differential, and the average of the 5 values reported.

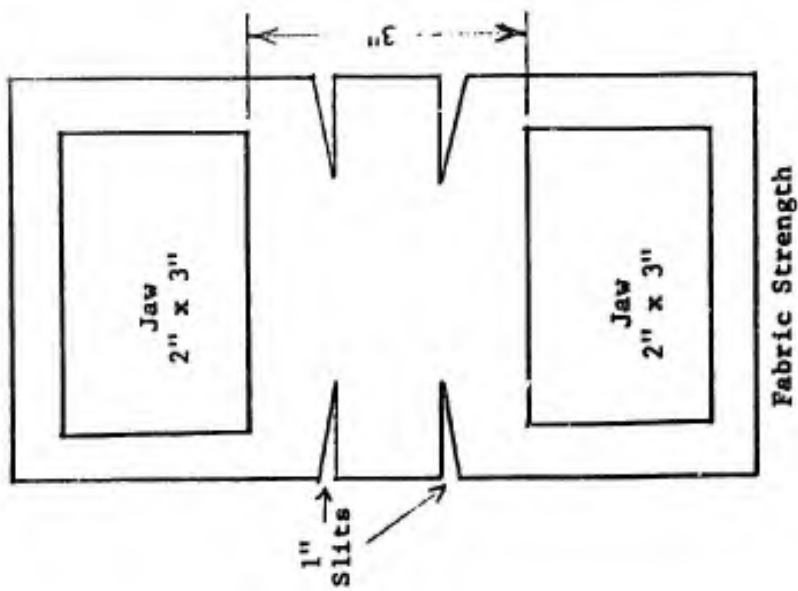
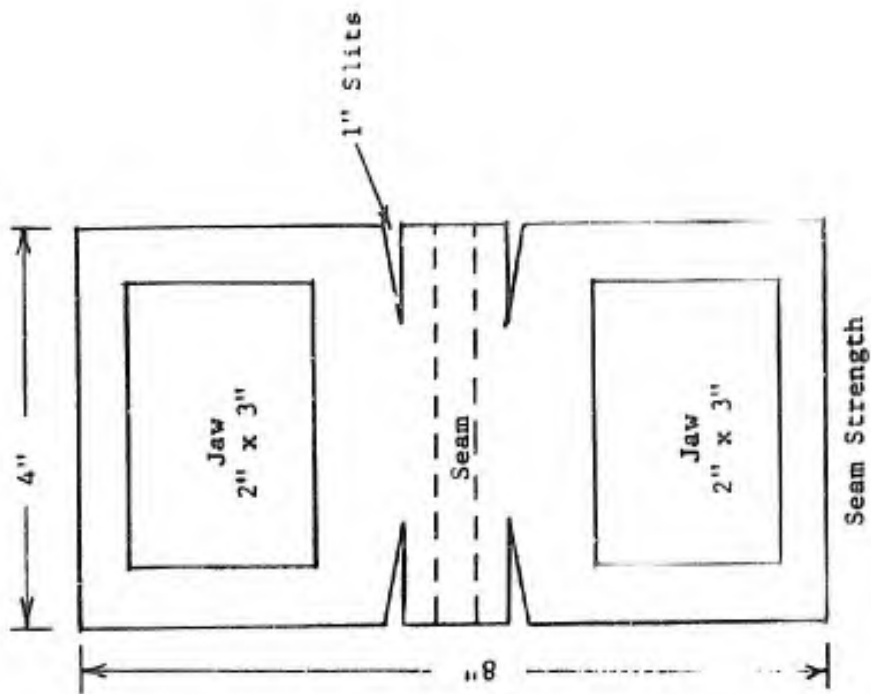


FIGURE 10. SEAM TEST SPECIMEN LAYOUT

## APPENDIX I (continued)

### PROCEDURES USED FOR EXPOSURES AND TESTING FABRICS AT ELEVATED TEMPERATURES

#### Tensile Strength

The ravel strip method was used for these tests. Warp or filling strips (raveled to 75 end widths, essentially 1.0 in.) about 20 in. in length were used throughout. The fabrics were tested at elevated temperatures by loading the strips through the FRL Environmental Test Chamber extension as indicated in Figure 11. The chamber was preheated to the desired temperature, the strips were placed through the slots by means of a wire probe with a small clamp on one end, and clamped in the Instron jaws. The jaws were 13" apart.

The loading took about 1 min. After the required exposure time, the samples were broken with a crosshead speed of 20 in./min. This extension rate was based on a heated gage length of 5 in. so as to give the normal 400% strain rate for fabric tests.

For 10 and 20 min exposure times, the samples were aged in the extension for the required time before breaking. For the longer exposures, the raveled strips were hung in a preheated circulating hot air oven for 5 min. less than the required time and removed and stored in a desiccator at room temperature. Each sample was later loaded in the preheated test chamber, aged the additional 5 min (to insure sample's attaining temperature) and broken. Three (3) samples were aged and tested for each time-temperature test.

#### Shrinkage

Shrinkage measurements were made on a single aged sample 4 in. x 8 in. Only the longer dimension was used for the measurements; that is, one sample 4 in. x 8 in. for filling shrinkage and one sample 8 in. x 4 in. for warp shrinkage for each time-temperature test.

#### Procedure for Exposing Samples in Controlled Atmospheres

Fabrics were exposed in a stainless steel enclosed vessel in an oven at 700°F. Nitrogen or air was fed into the vessel through a coiled, 6 ft length of 3/16 in. O.D. stainless steel tubing at 38 ml/min for 20 hr. There was a slight positive pressure of 3 to 5 in. of water at all times, and gas escaped from the vessel, under pressure, around the closure gasket. The coiled tubing served as a gas preheater and the temperature inside the vessel was measured with a thermocouple. The fabric was hung in the vessel for exposure, so that the middle portion from which yarns were removed for testing did not touch metal.

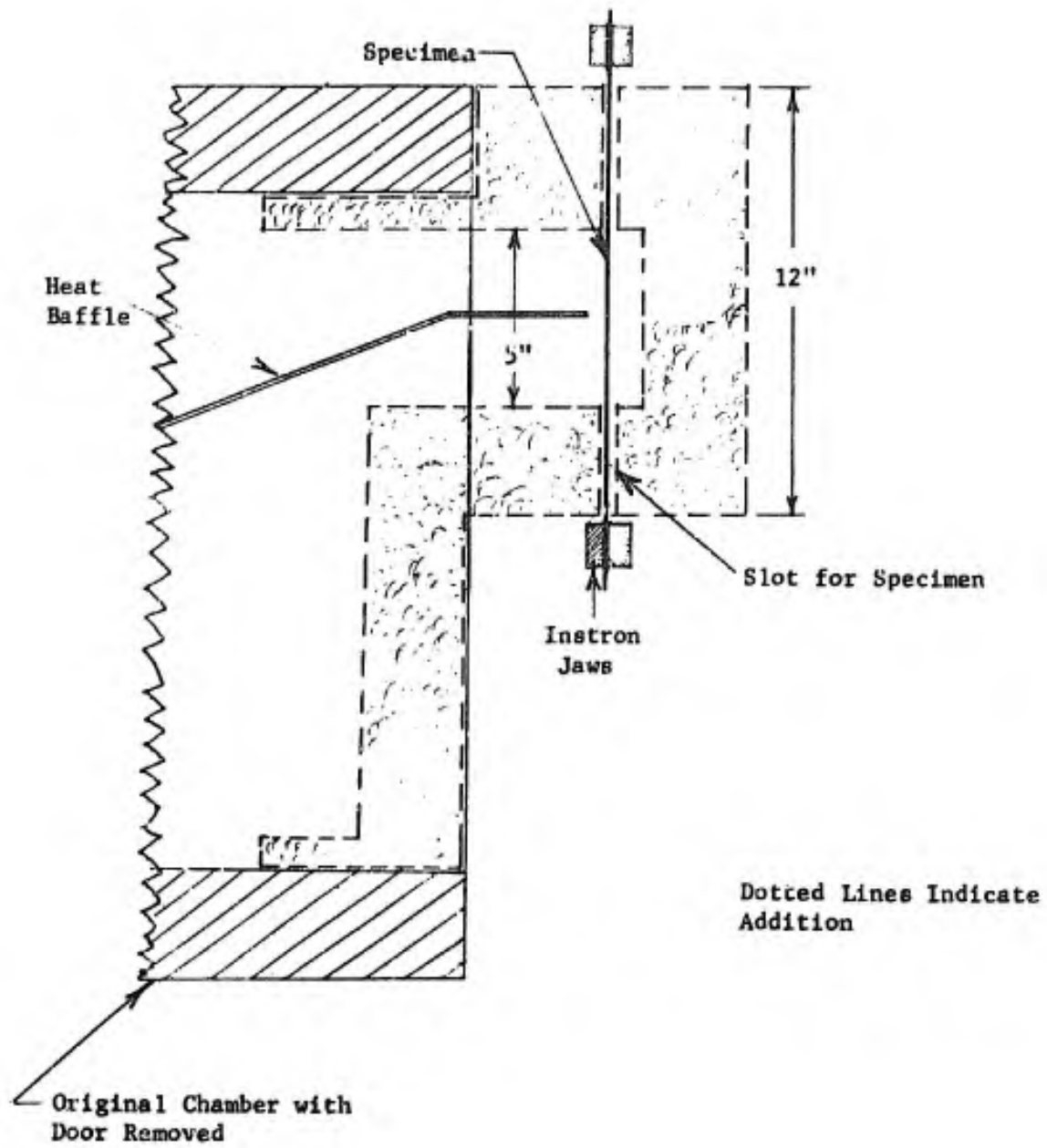


FIGURE 11. FRL ENVIRONMENTAL CHAMBER EXTENSION

## APPENDIX II

### PREPARATION OF FABRIC AND APPLICATION OF SPECIAL FINISHES

The scoured fabric was jig treated as follows:

Scoured with

2 gm/liter Tanapon X-70  
0.5 gm/liter Tetrasodium Pyrophosphate  
30 min at 60°C,  
Dump,  
Rinse.

Desized with

1 gm/liter Monosodium Phosphate  
3 gm/liter Rapidase  
30 min at 80°C,  
Dump.  
Rinse - 30 min at 50°C with overflowing water  
Dry - Tenter Frame (clips) at 200°F

Finishes were applied by padding and air drying. The ET-4327 silicone treated fabrics were cured at 400°F for 10 min. The finishes used and application media were:

FF-400 (Dow-Corning) - diluted in water  
ET-4327 (Dow-Corning) - diluted in water  
Therminol FR3 (Monsanto) - diluted in perchloroethylene  
Nopco PSB (Nopco) - diluted in water  
Nopco C-104 (Nopco) - diluted in water  
Nopcostat HS (Nopco) - diluted in water  
Nopco BHT Antioxidant (Nopco) - diluted in perchloroethylene  
PL-5 (Dow Chemical) - diluted in perchloroethylene

The amounts actually applied were:

APPENDIX II (continued)

% SOLIDS APPLIED TO PBI FABRICS

	1	2	3	4	5	6	7	8
	FF-400	ET-4327 <sup>1</sup>	THERMINOL	NOPCO PSB	NOPCO C-104	NOPCOSTAT HS	BHT ANTIOXIDANT	PL-5
Scoured Only	A	3.0	5.7	6.0	5.7	5.0	5.0	3.0
	B	6.0	2.8	2.8	2.8	2.8	2.8	6.0
Scoured, Heated	C	5.8	6.6	6.0	5.7	5.3	6.0	
	D	2.9	3.3	2.8	2.8	2.8	3.0	
Greige, Heated	E	5.8	6.6	6.0	5.7	5.5	6.0	
	F	2.9	3.3	2.8	2.8	2.8	3.0	
Greige	G	2.9	5.2	5.2	5.7	5.5	5.5	
	H	6.0	2.8	2.8	2.8	2.5	2.8	

<sup>1</sup>Cured 10 min at 400°F.

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13. ABSTRACT A limited study was made to characterize the effects of parametric variations in construction on the mechanical performance of various parachute items made from polybenzimidazole (PBI) yarns. The results indicate that most of the specifications originally written for nylon parachute components can be met with PBI if suitable modifications are made in construction to allow for the lower tenacity of PBI yarn. Strength translation efficiencies are good enough to permit prediction of breaking loads of structures with reasonable accuracy on the basis of one pound per 100 deniers of yarn used.  Sufficient amounts of each required end item were made to permit complete laboratory characterization including testing at ambient, 350° F, 450° F, 700° F, 900° F, and 1000° F after exposures ranging from 0 to 200 hours. Only the threads and one fabric were tested over the specified range when it became apparent that finish modifications would be required before reliable results could be obtained from high temperature testing. An investigation was made of the high temperature resistance of available candidate finishes and finish components applied to PBI fabric and yarn by various methods. While no satisfactory material was found, some leads were developed which may be useful in further studies. The testing done on the threads and lightweight fabric showed that PBI yarn structures retain about 40% of their original strength after exposure			

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13. ABSTRACT (continued)

at 700°F for two hours, even with an unsatisfactory finish. This represents a significant advance over presently available materials.

One outstanding characteristic of the yarn used was its good processing quality. Though filament deniers and therefore total yarn deniers appeared to vary considerably as would be expected with the first production of any new yarn, nevertheless, the difficulties usually encountered in processing yarns of such character were almost totally absent.

The reasonably good strength and the good strength retention at elevated temperatures indicate that PBI yarn should be useful for service up to and somewhat beyond 700°F for extended periods.

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<p>14</p> <p style="text-align: center;">KEY WORDS</p> <p>Yarn, Filament, High-Temperature Fiber, Thermally Stable, Polybenzimidazole (PBI), Fabrics, Parachutes, Seam Efficiency, High-Temperature Finishes, High-Temperature Testing</p>	LINK A		LINK B		LINK C	
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