

Technical Memorandum 104814



# Development and Evaluation of Polybenzoxazole Fibrous Structures

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## 1.0 Introduction

The first objective of this work is to evaluate a newly developed fiber for structural application where Kevlar has been previously used. Kevlar has been used for its strength and high modulus, but in oxygen enriched environments flammability has been a problem.

This newly developed fiber is made of poly(p-phenylene benzobisoxazole) (PBO), a member of the polybenzoxazole class of polymers, one of the various types of highly heat resistant polyheteroarylenes (PHA). PBO fiber has properties which make it unique among the known organic fibers. It has higher tensile strength and modulus than Kevlar, and it is flame resistant. Its oxygen index varies between 56 percent and 72 percent, depending on sample treatment and structure.

## 2.0 Background

Polybenzobisoxazole fiber has recently been developed by Dow Chemical Company. However, polymers with benzoxazole cycles in the chain have been known for the last 25 years. The production of PBO resin was reported for the first time in 1959 [1]. Since then, many researchers have worked on the development of methods for producing PBO. During the 1980's, two principal types of methods were used for producing PBO polymers: the single and dual stage methods [2]. With single stage methods, PBO is produced in a melt, in polyphosphoric acid, or in an organic solvent solution. In a melt, PBO is principally produced by homopolycondensation of aliphatic hydroxyamino acids at 200-300°C, or by homopolycondensation of 2-amino-4-cyano-phenol between 200 and 400°C, and by condensation of bis-o-aminophenols with aryls esters of aromatic dicarboxylic acids in temperatures of 270-400°C. Production of PBO in phosphoric acid is conducted at 100 or 200°C depending on the type of precursors. The single stage production of PBO in various organic solvents has also been reported by researchers. However, the use of organic solvents has been observed more in the dual stage methods of producing PBO. The two stage methods have been used since the early 1960's. The first stage of these dual methods involves the formation of prepolymers which, by heating in a second stage, form benzoxazole structures. Among the many two stage methods, those using bis-o-aminophenols and halides of aromatic dicarboxylic acids have led to the production of high molecular weight PBO with better properties for potential commercial applications. However, cost and difficulty in preparing PBO have been reasons why, for many years, PBO has been known only among researchers and scientists.

Nonetheless, efforts at producing polybenzoxazole structures have been pursued. In 1990, the Dow Chemical Company secured worldwide rights to manufacture and sell PBO. Yet because of costs the PBO resin did not immediately find a wide market. In the laboratory, researchers were still working on developing more economical polybenzoxazoles. In 1992, Hergenrother et al. prepared PBO from the reaction of novel di(hydroxyphenyl)benzoxazole monomers with activated aromatic compounds [3]. The advantages of this method are lower costs and easier preparation. Meanwhile, at Dow Chemical Company, PBO fibers were being developed. In 1993, samples of PBO yarn and woven structures were evaluated at the Johnson Space Center (JSC) Crew and Thermal Systems Division Advanced Materials Laboratory, but proprietary restraints prevented the release of these first evaluation results. These results were, however, promising for future work. In June 1994, an article on the potential applications of PBO fibers was published in the *High Performance Textiles* newsletter with a table of data (table 1) [4]. The data from the JSC evaluation corresponded to that found in this table. In the following months, under a contract with Albany International Research Company, more PBO fibers were purchased by JSC, and various structures were fabricated.

Table 1. Typical Properties of PBO Fibers\*

Property	PBO	High Modulus PBO
Tensile Strength, Ksi (GPa)	820 ( 5.65)	800 ( 5.48)
Tenacity, gpd	41	4
Specific Tensile Strength, Ksi	525	510
Tensile Modulus, Msi	22 ( 1100)	40 (2000)
Specific Tensile Modulus, Msi	14.1	25.6
Elongation at Break, %	3.5	1.5
Moisture Regain, %	2.0	< 0.5

\* Data from *High Performance Textiles*, June 1994

### 3.0 Comparison of PBO to Other High Performance Fibers

#### 3.1 Physical and Mechanical Properties

Fibers which are labeled “high performance” usually have good thermo-oxidative properties (heat and flame resistance), or have high tensile strength and modulus. PBO is unique because it has both the thermo-oxidative and the tensile properties so desired in many aerospace applications. PBO, like the other synthetic organic thermostable fibers, has an aromatic structure which contributes to its thermal properties. In aromatic systems, the phenomenon of resonance stabilization increases the primary bond strength, and consequently, influences heat resistance. This is the principal mechanism which makes polybenzoxazoles, polyimides, polybenzimidazoles, and aramids, to a lesser extent, heat resistant fibers. The thermostability of PBO is due not only to its morphology, but also to its molecular weight, as shown in terms of specific gravity in table 2.

Table 2. Properties Comparison of Thermostable Organic Fibers

	Specific Gravity, g/cm <sup>3</sup>	Breaking Tenacity, gpd	Tensile Strength, 10 <sup>3</sup> psi	Breaking Elongation, %	Abrasion Resistance	Moisture Regain, %	Oxygen Index, %
Nomex	1.38	4.0-5.3	90	22-32	good	6.5	27-28
Kevlar	1.44-1.47	18-26.5	340-490	1.5-4.0	poor	1.2-4.3	27-29
PBI	1.43	2.6-3.0	50	25-30	good	15	38-39
P-84	1.41	3.73	67	19-21	good	3.0	37-38
PBO	1.5	41	820	3.5	poor	2.0	>56

- Nomex, Kevlar, and PBI data from Textile World Manmade Fiber Chart, 1992
- P 84 data from Lenzing AG [5] and Albany International Research Co.
- PBO data from JSC Crew and Thermal Systems Division Advanced Materials Laboratory

Table 2 also shows that PBO has the highest tensile strength among these thermostable fibers. PBO is stronger than the typical ultra high molecular weight polyethylene (UHMWPE) fibers such as Spectra and Dyneema, as shown in table 3.

Table 3. Comparison Between PBO and UHMWPE

	Specific Gravity, g/cm <sup>3</sup>	Tenacity, gpd	Tensile Strength, 10 <sup>3</sup> psi	Tensile Modulus, 10 <sup>6</sup> psi	Oxygen Index, %
PBO	1.5	41	800-820	22-40	>56
Spectra®	0.97	30-35	375-435	17-25	18-19
Dyneema™	0.97	32-37	395-455	20-28	18-19

- Spectra data from Allied Signal Inc., "High Performance Fibers," 1990
- Dyneema data from DSM, "High Performance Fibers B.V.," 1995

In summary, the tensile strength and tensile modulus of PBO are higher than that of any other synthetic organic fiber.

### 3.2 Thermal and Oxidative Properties

Tables 2 and 3 give preliminary information on the oxidative behavior of PBO. The high oxygen index value of PBO indicates that the polymer has potentially good flame retardancy properties. Flammability data are given in section 4.2.

The thermal properties of the fibers have been obtained by thermal analysis. Thermogravimetric (TGA) and thermomechanical (TMA) analyses were performed to assess the heat resistance of PBO fibers.

### Thermogravimetric analysis

TGA on the PBO sample (figure 1) was performed at Albany International. PBO decomposes at temperatures above 650°C. At 650°C, the sample has lost only 6.85 percent of its original weight, part of which, in the initial portion of the curve, may be due to water loss. This would explain why the slope between room temperature and about 300°C is steeper than the slope between 300 and 500°C. Above 650°C, the curve drops sharply, indicating a rapid degradation of the sample. At 725°C, approximately 50 percent of the sample has decomposed. Around 810-825°C, the sample is completely consumed.

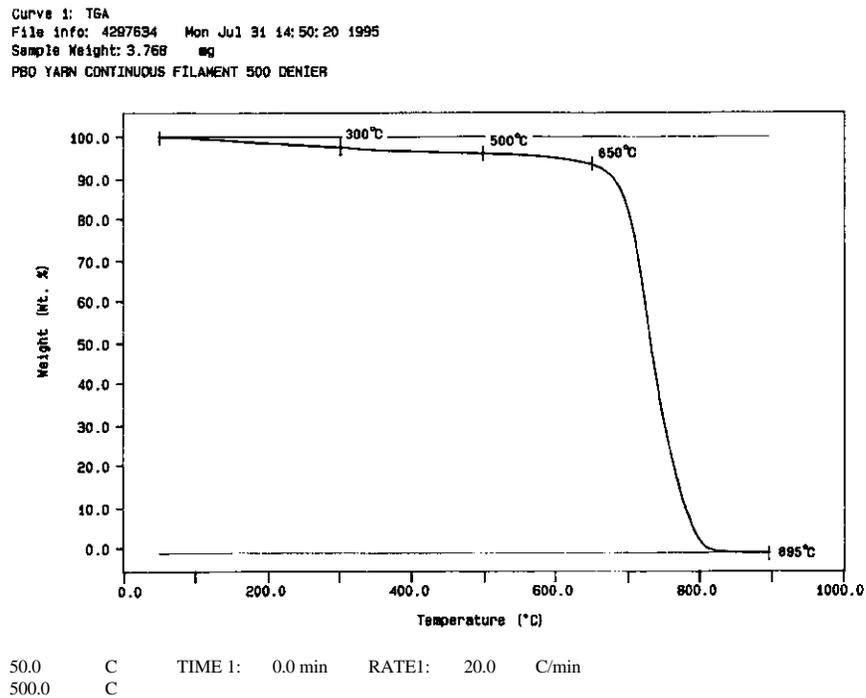


Figure 1. Decomposition temperature for PBO.

In comparison, Kevlar decomposes at 430 to 480°C (figure 2). Between room temperature and 430°C, the initial portion of the curve is also steeper than the portion between 200°C and 430°C, due to water loss. Above 480°C, the rapid descent of the curve indicates degradation of the sample. Above 600°C, the sample is completely consumed.

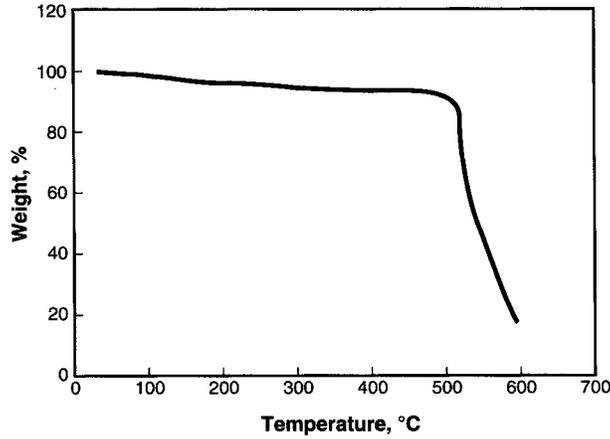


Figure 2. Kevlar decomposition rates.

Thermomechanical Analysis

The comparative thermomechanical analysis of PBO and Kevlar shows that both types of fibers shrink as the temperature increases. PBO shrinks at an average rate of 12.6  $\mu\text{m}/\text{m}^\circ\text{C}$  up to 563°C (figure 3), and Kevlar shrinks at an average rate of 20.7  $\text{mm}/\text{m}^\circ\text{C}$  up to 456°C (figure 4). Overall, the two curves look very similar, except that the PBO curve is much longer before it drops at the 650°C threshold. Also, cycles of heating and cooling for both PBO and Kevlar (figures 5 and 6) show no hysteresis below the initial decomposition temperature. This is an important property for the assessment of a usable or service temperature for these fibers.

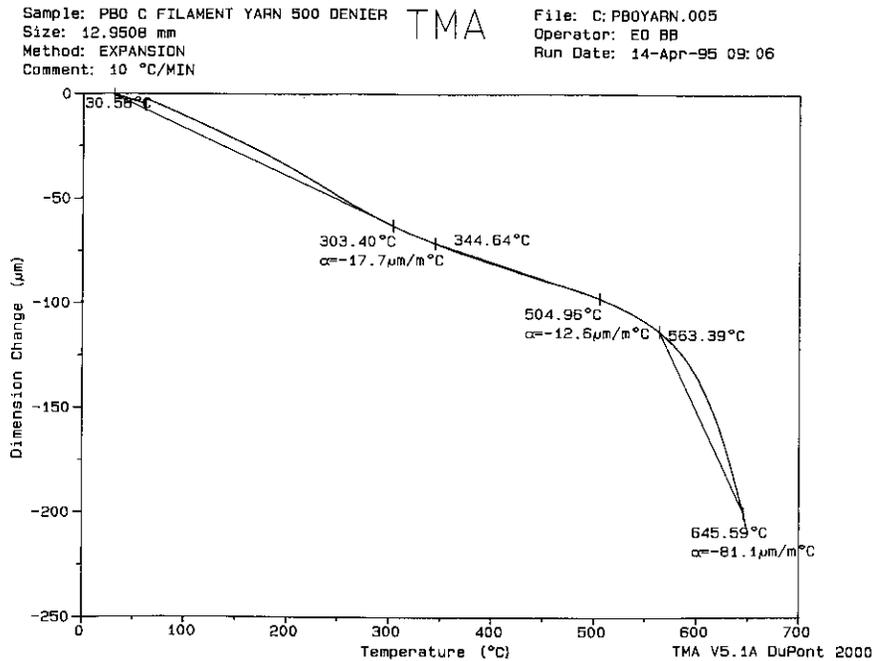


Figure 3. PBO shrinkage rates.

Sample: KEVLAR 29 38108-978  
Size: 12.9338 mm  
Method: EXPANSION  
Comment: 10 DEGREES/MINUTE RAMP

TMA

File: C: KEVLAR.004  
Operator: BB TB  
Run Date: 10-Aug-95 14:10

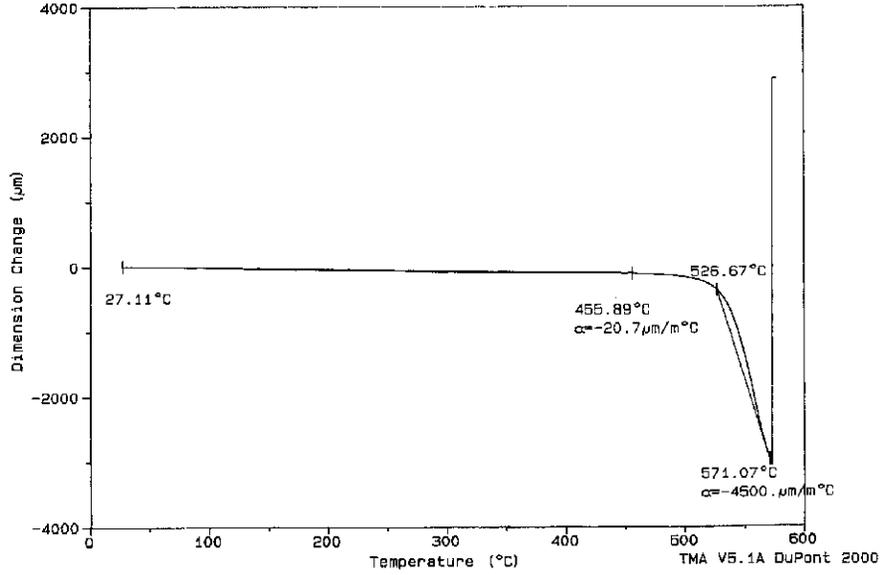


Figure 4. Kevlar shrinkage rates.

Sample: PBO C FILAMENT YARN 500 DENIER  
Size: 12.8000 mm  
Method: THERMAL EXPANSION  
Comment: 10 °C/MIN

TMA

File: C: PBOYARN.001  
Operator: EQ BB  
Run Date: 11-Apr-95 09:32

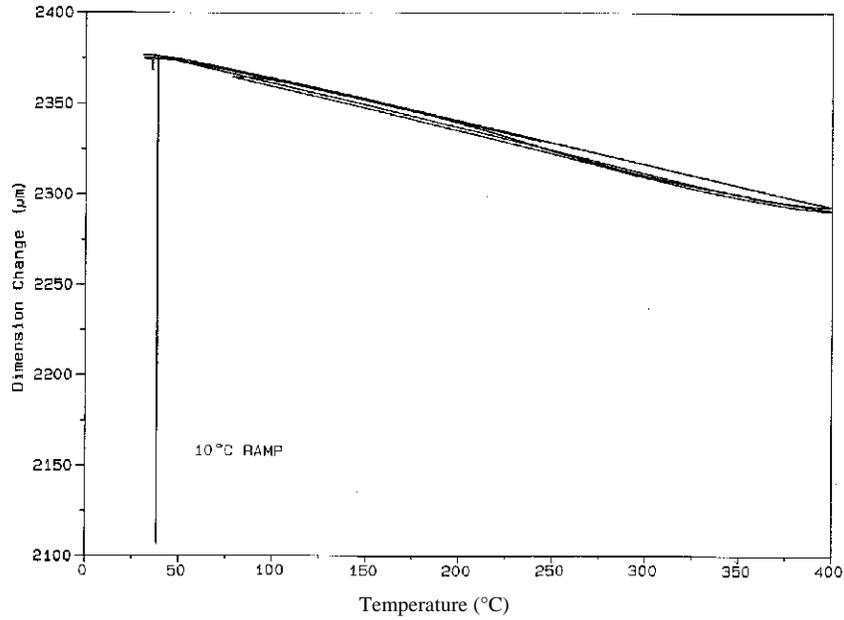


Figure 5. PBO response to temperature cycles.

Sample: KEVLAR 29 3B108-976  
Size: 12.5892 mm  
Method: HYSTERESIS COLD  
Comment: 10 DEGREES/MINUTE RAMP

TMA

File: C: KEVLAR.005  
Operator: BB TB  
Run Date: 15-AUG-95 10: 49

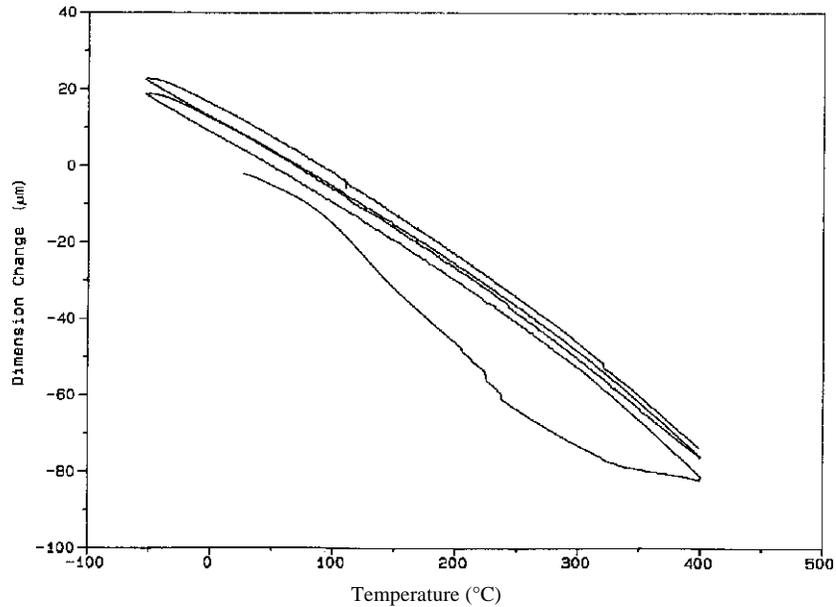


Figure 6. Kevlar response to temperature cycles.

The information gathered from this initial comparative analysis of data from PBO and other high performance fibers, particularly Kevlar fibers, served as the basis for our development effort.

#### 4.0 PBO Structures Development and Evaluation

PBO fibers, which combine strength and flame resistance properties, are of great interest for various aerospace applications, particularly for crew systems. Different PBO structures have been made at Albany International Research Company under NASA contract NAS9-18778. The first PBO structures made at Albany were webbings, cords, and tapes. Similar structures have been used extensively for crew equipment, space suits, and many other systems in the space programs. Other structures are currently being developed and evaluated for debris and micrometeoroid shield applications, as well as blended structures.

The initial effort of this study is to directly compare PBO structures to corresponding Kevlar structures. Two samples were prepared under NASA contract by Albany International Research Company with PBO yarn obtained from Toyobo, America, Inc. The yarn type is a 500-denier continuous filament yarn. Its tenacity is 32.5 grams per denier. Sample No. 3550-1032 (S-301) is a 1-3/4" wide webbing produced similarly to Kevlar S-249. Sample No. 3550-1033 (S-302) is a 1/8" diameter braided cord similar to Kevlar S-216.

#### 4.1 Comparative Physical and Mechanical Properties of PBO and Kevlar Structures

The samples were tested in the JSC Crew and Thermal Systems Division Advanced Materials Laboratory. The test results are shown in table 4.

Table 4. Comparative Properties of PBO and Kevlar Structures

Property	PBO 3550-1032	Kevlar S-249	PBO 3550-1033	Kevlar S-216
Weight, oz/yd	0.75	0.77	0.06	0.062
Width, in	1.75	1.75	0.125	0.125
Thickness, in	0.027	0.032	0.125	0.125
Breaking Strength, lbs	8995	4173	890.8	469.2
Elongation at Break, %	11.5	7.7	10	4.9
Stress at Max. Load, psi	182773	75710	72590	60050
Energy to Breakpoint, lbs-in	2951	3502	633.7	313.1
Abrasion Resistance, % change @ 2500 cycles with 2 lbs load	68.7	85.1	83.9	98.7

Each PBO sample was prepared to the same specifications as known samples of Kevlar with similar weight, width, and thickness. Samples PBO 3550-1032 and Kevlar S-249 were both plain weave webbings, and samples PBO 3550-1033 and Kevlar S-216 were 1/8" braided cords. For both types of structures, webbing and cord, PBO ultimate tensile strength was much higher than that of Kevlar by an approximate order of magnitude of two. PBO percent elongation at break was also higher than that of Kevlar, in an order of magnitude of 1.5 to 2. On the other hand, the abrasion resistance of the PBO structures was poor, as shown in table 2. Like the high modulus fiber Kevlar, PBO also showed a low abrasion resistance.

## 4.2 Flammability and Toxicity

The oxygen index was also measured in the Advanced Materials Laboratory (AML) (table 5): an average value of 72 was obtained for the PBO webbing structure, versus an average of 59 for the cord. Since there is currently no established standard in the ASTM test method D2863 for measuring the oxygen index of woven structures, different values were recorded in the Albany International Research Company laboratory, as shown in table 5. These differences were more indicative of differences between ignition conditions in the two laboratories than of variations between the two laboratories samples. Both sets of data are given here for information. Although the oxygen index value may vary with the ignition conditions of the test, it is expected that, for PBO fibers or yarn and the PBO structures, the oxygen index is greater than 56, and can be as high as 78 for a thick webbing structure. Since the oxygen index is dependent on sample size and structure, the difference between results in braided structures and fibers was expected to be relatively large. In any case, when compared to the similar Kevlar structures, the PBO samples had much higher oxygen indexes. The oxygen index is a widely used test in the industry for quality control, but it does not give information on the actual combustion behavior of a particular material or a particular structure of that material.

Table 5. Comparative Oxygen Index of PBO and Kevlar Structures

Average Oxygen Index (OI)	PBO 3550-1032	PBO 3550-1032	Kevlar S-249	Kevlar S-216
OI from AML	74	56	35	34
OI from Albany	78.2	63.7	31.3	29.7

Because the knowledge of the flammability behavior of a material is of the utmost importance for space applications, samples of the PBO webbing were tested for flammability at NASA White Sands Test Facility according to NHB 8060.1C [6] protocol. Test conditions were

- Test Atmosphere: 70.0% Nitrogen  
30.0% Oxygen
- Test Pressure: 70.3 kPa (10.2 psia)
- Test Chamber Volume: 1.4 m<sup>3</sup> (50 ft<sup>3</sup>)

An upward flame propagation test was performed on three PBO samples of length  $30.65 \pm 0.15$  cm, thickness 0.075 cm, and width 4.6 cm (figure 7 & table 6).

WSTF NO. 95-29100  
PBO Webbing Style S-301  
30.0% O<sub>2</sub> AT 10.2 PSIA

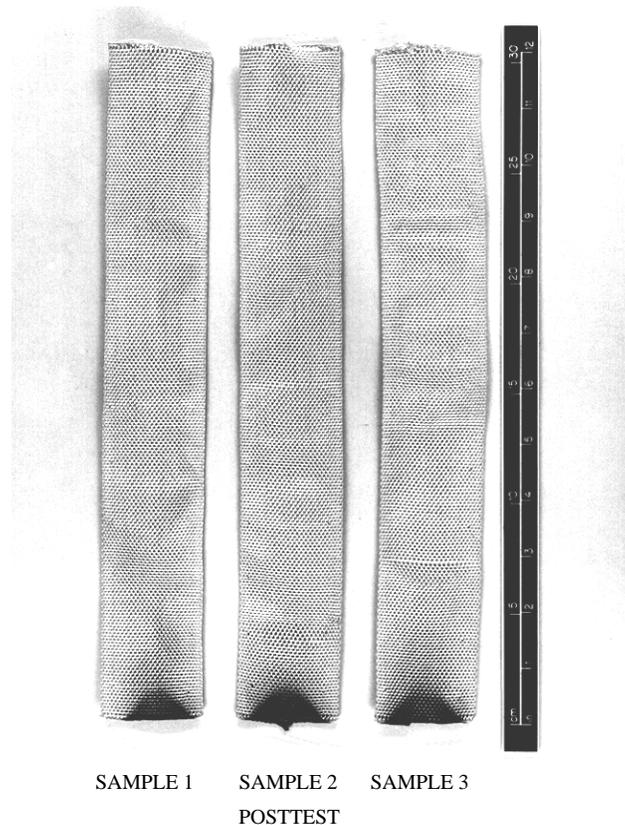


Figure 7. PBO samples after upward flame propagation tests.

Table 6. Upward Flame Propagation Test Results for PBO Sample No. 3550-1032

PBO Webbing	Sample 1	Sample 2	Sample 3
Burn Length, cm (in)	1.3 (0.5)	1.5 (0.6)	1.0 ( 0.4)
Weight Loss, g	ND	ND	ND
Relative % O2 Consumption	ND	ND	ND
Quantity of Sparks	None	None	None
Quantity of Cinders	None	None	None
Quantity of Flame Jets	None	None	None
Quantity of Burning Material Transferred	None	None	None
Effect on K-10 paper	No Ignition	No Ignition	No Ignition

ND indicates that the results were less than the reporting limit. The reporting limit for weight loss is 0.05 gram. The reporting limit for relative oxygen consumption is 0.5 percent.

The same upward propagation test was performed on Kevlar 29 webbing samples of length 40.6 cm, thickness 0.08 cm, and width 4.5 cm (figure 8 & table 7).

Offgassing and toxicity of both Kevlar 29 and PBO webbings (table 8) were also measured according to the NHB-8060.1C NASA handbook protocol. The respective weights of the Kevlar and PBO samples were 20.305 grams and 21.62 grams. Test conditions were as follows:

- Test Chamber Volume: 4.3 liters
- Test Atmosphere: 74.1% Nitrogen and 25.9% Oxygen
- Test Pressure: 82.0 kPa (11.9 psia)
- Test Temperature: 49°C (120°F)
- Test Duration: 72 hours

WSTF NO. 93-27836  
 Kevlar Webbing 29  
 30.0% O<sub>2</sub> AT 10.2 PSIA

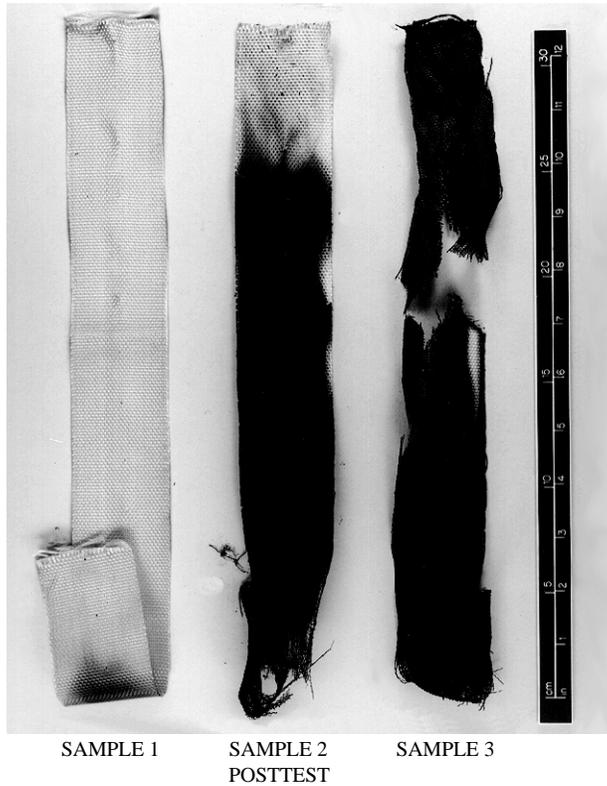


Figure 8. Kevlar samples after upward flame propagation tests.

Table 7. Upward Flame Propagation Test Results for Kevlar 29 Style No. S-249

Kevlar 29 Webbing	Sample 1	Sample 2	Sample 3
Burn Length, cm (in)	0.0	18.3 (7.2)	33.3 (13.1)
Propagation Burn time, seconds	--	91	151
Weight Loss, g	0.21	2.99	5.31
Relative % O <sub>2</sub> Consumption	ND	1.5	2.8
Quantity of Sparks	None	None	Small
Quantity of Cinders	None	None	None
Quantity of Flame Jets	None	None	None
Quantity of Burning Material Transferred	None	None	Small
Effect on K-10 paper	No Ignition	No Ignition	No Ignition

Table 8. Comparison of Offgassed Products from Kevlar 29 and PBO Webbing

Component	Toxic Limit ( micrograms/gram)	Kevlar 29 webbing Quantity (micrograms/gram)	PBO webbing Quantity (micrograms/gram)
Acetaldehyde	5.7	0.04	0.09
Acetone	1018.0	0.05	0.05
Butene	7	0.02	0.09
C6 Saturated Aliphatic Hydrocarbons	7	--	0.01
C9 Saturated Aliphatic Hydrocarbons	7	--	0.005
Carbon Monoxide	14	0.4	0.27
Ethyl Alcohol	134.00	0.01	0.03
Hexamethylcyclotrisiloxane	324.0	0.02	0.08
n-Butyl Formate	119.7	--	0.008
n-Hexanal	23.4	0.03	0.005
Octamethylcyclotetrasiloxane	217.4	0.01	0.03
Pentanal	151.00	--	0.007
Styrene	60.9	--	0.007
t-Butyl Alcohol	173.01	0.02	0.04
Toluene	86	--	0.01
Trimethyl Silanol	57	0.02	0.07
Methyl Alcohol	12.90	0.09	--
Isopropyl Alcohol	215.00	0.01	--
N-Propyl Alcohol	140.03	0.01	--
1-Methoxy-2-Propanol	106.06	0.01	--
Valeraldehyde	151.03	0.02	--
C11 Saturated Aliphatic Hydrocarbons	7.17	0.01	--
Butyl Alcohol	173.04	0.01	--
Isobutanal	63.07	0.01	--
C5 Aldehydes	75.97	0.01	--
Xylenes	315	0.02	0.005

Note: -- indicates that these compounds were not found in the offgassed products.

In summary, the findings of flammability and toxicity tests showed that

- PBO is flame resistant in 30% oxygen and 10.2 psia environment
- PBO does not produce sparks, cinders, or flame jets
- PBO weight loss in test 7 from NHB 8060.1C protocol is below the reporting limits of 0.05 gram
- PBO offgassed products are well below their toxic limits.

### 4.3 Chemical Resistance

The resistance of PBO to acids, bases, and salts varies, depending on the type and concentration of the chemicals to which it is exposed. The comparative data for selected chemicals' effects on PBO and Kevlar, obtained in the Advanced Materials Laboratory, is given in table 9.

Table 9. Comparative Resistance to Chemicals of PBO and Kevlar

Chemical	Concentration (%)	Temperature (°C)	Time (hour)	Percent loss in Breaking Strength of Kevlar	Percent loss in Breaking Strength of PBO
Water	100	100	100	1	17
Hydrochloric Acid	37	25	100	75	38
Nitric Acid	10	25	100	41-80 **	84
Acetic Acid	100	25	100	2	1
Sulfuric Acid	70	25	100	21-40 **	76
Ammonium Hydroxide	28	25	100	0-10 **	6
Sodium Hydroxide	40	25	100	0-10 **	0.7
Sodium Chloride	10	121	100	41-80 **	62
Sodium Hypochlorite*		25	100	41	48
Toluene	100	25	100	3	2
Xylene	100	25	100	2	5
Methylethylketone	100	25	100	3	9
Kerosene	100	25	100	5	4
Gasoline	100	25	100	4	0.1
Brake Fluid	100	121	100	6	2

\* Chlorox commercial solution

\*\* Data from Dupont

In summary, PBO has excellent hydrolytic stability; its resistance to organic solvents is good, comparable to that of Kevlar.

#### 4.4 Effects of Light and UV Radiation

Like most polymers, PBO and Kevlar are sensitive to light. After 450 hours of exposure to 340 nm wavelength, PBO webbing has lost over 98 percent of its tensile strength (table 10).

Table 10. Effect of UV Light on PBO and Kevlar Yarns

	PBO 3550-301	Kevlar S-249
Density, denier	1554	1620
No. of Plies	1	1
Maximum Tenacity per Yarn, gpd	23.13	14.96
Maximum Tenacity per yarn after 450kJ at 340 nm, gpd	0.27	1.57
Tenacity Change	98.8 %	89.5 %

No radiation resistance test has been performed. It is reported that PBO strength loss is only 10 percent after an exposure of 500 mrad in vacuum [2].

#### 5.0 PBO Fibers and Fibrous Structures Evaluation and Recommendations

PBO is definitely the strongest synthetic organic fiber developed at this time, although its great strength is accompanied by poor abrasion properties. PBO also has excellent thermal stability and better flame resistance than most other polymeric fibers. Its resistance to different chemicals, UV light, and radiation is similar to that of Kevlar. What makes PBO truly unique is the actual combination of strength and thermo-oxidative properties unseen in any other polymeric fiber.

This unique combination of strength and flame resistance makes PBO a prime material for various aerospace applications. Braided structures similar to those evaluated in this study can be extremely useful in the various space programs. There is a great variety of flight hardware for which strong and flame resistant flexible fibrous structures like straps and tethers are needed. Until now, the selection of materials for these applications has resulted from a compromise between strength versus flammability requirements. PBO offers an alternative to this compromise.

#### 6.0 Future Work

Several areas for further development with PBO fibers have been identified as a result of this study. PBO has the potential to be a remarkable synthetic organic fiber for micrometeoroid and debris shield applications. Work in this area of applications has been initiated. Also, as a continuation of this study, blended, coated, and laminated PBO structures will be developed. Part of this effort is to increase the abrasion resistance of PBO without reducing strength and flame resistance significantly. Further development will also include the fabrication and evaluation of PBO rigid composite structures such as PBO fiber-epoxy resin or PBO fiber-PBO resin composites.

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