PREDICTION OF LONG-TERM FAILURE IN KEVLAR 49 COMPOSITES*

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Abstract

Creep rupture data in Kevlar 49 epoxy usually exhibit considerable scatter: the coefficient of variation (CV) about the mean failure time at a given stress exceeds 100%. Quasi-static strength data, in contrast, shows little scatter: <4% CV for pressure vessels and <10% for impregnated strands. In this paper analysis of existing creep rupture data on Kevlar epoxy vessels at four storage pressures has produced an interesting and useful result. It was found that a significant portion of the scatter in failure times for pressure vessels is due to spool-to-spool variation in the eight spools of Kevlar fibers used to wind the vessels. The order rank of mean times to failure was consistent over a pressure range from 3400 to 4300 psi, 68 to 86% of short term burst. Also, the coefficient of variation about the mean failure time for each spool was less than that for the total sample. The statistical inference that the sample is nonhomogeneous was supported by a nonparametric check using the Kruskal-Wallis test, and by a parametric analysis of variance. The order rank found in long-term tests did not unequivocally agree with static strength ranks; several spool sets were distinctly high or low. The implication is that, while static strengths are not valid predictors of long-term behavior, short term creep rupture tests at high stress definitely are.

The material difference which causes the spool-to-spool variations has not yet been identified for all eight spools. However, it appears that Kevlar behavior at lower pressures may be predicted through the use of curves fitted to the data for each spool. A power law relating failure time to pressure, $t = t_0(p/p_0)^m$, was found to fit the data reasonably well. From these fits were constructed design allowable life values with an expected reliability of .999999; these predictions are supported by data on vessels at 2500 psi, where no failures have occurred after 4 1/2 years. The power law fits also demonstrated the extreme sensitivity of failure time to pressure ($m \approx -15$ to -25). This implies that, both in composite vessel design and in creep rupture experiments, the pressure (or stress) level be carefully controlled.

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Introduction

Composite pressure vessels wound from either glass or aramid fiber have been used successfully for some years. In applications with only one relatively short pressure cycle, such as rocket motors, composite vessels offer considerable weight savings over steel or titanium alternatives. The short term, static burst, behavior of such vessels is quite predictable and repeatable. A relatively simple predictive model based on a linear elastic composite and a maximum strain failure criterion can be used (e.g., Chen and Chenlow, 1977; Gerstle, 1974). Guess (1982) has verified this predictive technique for Kevlar 49 epoxy for pressures up to 60 ksi (414 MPa). The repeatability of burst pressure in Kevlar 49 vessels was demonstrated by Toland, et al. (1978), who measured a coefficient of variation (CV) of only 3.8% in static burst tests of 29 vessels, which is less variation than usually seen in steel vessels. A 3 to 5% CV seems to be reasonably common throughout the industry for static burst. Such small variations admit design allowables as high as 85% of mean burst. Reliabilities of .999 to .99999 can then be obtained by setting this value equal to 1.25 to 1.5 times the maximum operating pressure (Kapur and Lamber-son, 1977). The resulting overall safety factor is between 1.5 and 1.75.

The response of composite vessels to cyclic or sustained pressurization has not been nearly so predictable or repeatable. The static fatigue susceptibility of glass fibers is well known, and is a major cause of the ASME code specifying that the operating pressure of glass-resin vessels be only 1/6 of burst (ASME, 1971). A safety factor of 6 all but eliminates any weight savings over steel. Although Kevlar 49 epoxy strands appear to have somewhat longer creep-rupture failure times than do glass (Chiao, et al., 1973), the Kevlar long-term strand data exhibits as much scatter as glass, and also shows no indication of an endurance limit. Since the rupture of a gas-filled pressure vessel can be lethal, prudent design practice has dictated very low design allowables to

compensate for uncertainty about material performance under sustained load. In some cases, this led to the use of metal-composite load-sharing tanks; in others, it has caused sufficient concern that steel was substituted for Kevlar.

The present study was undertaken to determine what constitutes a safe design stress level for Kevlar 49 epoxy under constant load for a specified time. Using the available literature data, we considered the materials and processing variables in Kevlar vessels and attempted to isolate those which appear to affect long term behavior. Of particular interest were factors which seemed either to correlate with early failures or to cause increased variation in failure time. Additional goals were 1) to investigate the existence of a connection between burst and creep-rupture behavior; 2) to evaluate the validity of accelerating failure by elevated stress, and 3) to derive a failure criterion, either deterministic or probabalistic, based upon available data.

Existing Data

The most complete set of Kevlar 49 epoxy creep-rupture experiments are those conducted at Lawrence Livermore National Laboratories on unidirectional strands and thin aluminum-lined spherical pressure vessels. Of these, the pressure vessel study (Toland, et al., 1978) is the best detailed and documented, and was therefore most useful for the present study. Indeed the authors mention their intent to provide subsequent investigators with sufficient information to perform statistical studies on these data. Using results for pressure vessels also avoids the concern that strands may not accurately represent vessel behavior.

The experimental results were based on 250 vessels wound during a five month period in 1976 from a lot of Kevlar 49 manufactured earlier that year by DuPont. Each vessel consisted of a 112 mm ID Al liner, 1 mm thick, overwrapped with 1.1 mm of 380 denier Kevlar 49 yarn wetted during winding with epoxy. The resin was

Table 1. Summary of Testing

No. Vessels	Test	No. Failed (2/1/82)
29	Static Burst ($\bar{p}_f = 5,018 \text{ psi}$)*	29
39	Stored at 4300 psi ($.86 \bar{p}_f$)	39
24	Stored at 4000 psi ($.80 \bar{p}_f$)	24
24	Stored at 3700 psi ($.74 \bar{p}_f$)	24
21	Stored at 3400 psi ($.68 \bar{p}_f$)	10
46	Stored at 2500 psi ($.50 \bar{p}_f$)	0
30	Stored at 0 pressure	

*To convert psi to MPa, divide by 145.

a Bisphenol-A epoxy, DER-332, cured with an aliphatic primary triamine, Jeffamine T-403 (100/44.7), with a 60°C final cure. The liner is made principally from hydroformed 1100-0 Al hemishells, electron beam welded at the equator. Fill stem and south pole bosses were made from 5086-0 Al, also welded into place. The complete vessel is shown schematically in Figure 1. Further fabrication details are given by Toland, et al. (1978).

Of the 250 vessels, 29 were pressurized to burst at a rate of 200 psi/min (23 kPa/s). The mean static burst strength was 5,018 psi (34.6 MPa), with a CV of only 3.76%, as mentioned previously. Minimum and maximum recorded values reflect the small variance, 4,640 psi (31.9 MPa) and 5,400 psi (37.2 MPa), respectively. Two points should be noted about these results. Although the liner is nearly as thick as the composite, it is much weaker and therefore contributes little to the burst strength of the vessel. However the transition from 1100 to 5086 does create a stress concentration due to unequal yield strengths of the two alloys. Composite failure usually initiated at the boss-hemishell transition. Hahn, et al (1979) calculated an average fiber static failure stress of about 400 ksi (2.54 GPa). For a nominal composite fiber strength of 500 ksi (3.45 GPa), this would imply a stress concentration of about 1.25 at the transition. The second point about the static results are that these vessels were not the first 29 produced. Assignments to this group, and to those used for creep-rupture, were done by a randomizing scheme. This proved to be a valuable feature of the experiment.

The remaining vessels were pressurized to levels between 50 to 86% of mean static burst (taken to be 5,000 psi), except for 30 stored at zero pressure. A summary of the experiment is given in Table 1. Several vessels developed liner leaks at various stages of testing and were discarded.

Analysis of Creep-Rupture Results

Creep rupture times for vessels which had failed as of Dec. 1, 1978 are given in Barlow, et al. (1979). By then all of the vessels at 86% (39) and at 80% (24)

Table 2. Burst Pressure of 29 Vessels (Toland, et al., 1978)

Rank	Vessel No.	Spool No.	Burst Pressure psi*
1	157	5	4,640
2	78	3	4,710
3	214	7	4,735
4	41	2	4,750
5	1	1	4,800
6	216	7	4,872
7	133	5	4,890
8	59	2	4,890
9	47	2	4,910
10	215	7	4,920
11	43	2	4,926
12	207	7	4,938
13	206	7	4,938
14	249	8	4,950
15	177	6	5,000
16	97	3	5,010
17	182	6	5,040
18	5	1	5,060
19	151	5	5,070
20	100	3	5,075
21	91	3	5,100
22	28	1	5,112
23	72	2	5,160
24	11	1	5,160
25	8	1	5,175
26	129	4	5,225
27	24	1	5,300
28	82	3	5,340
29	128	4	5,400

*Divide by 145 to obtain MPa.

had failed, as had 18 out of 24 at 74% and 5 of 21 at 68%. Subsequent papers by Hahn (1979) and Hahn, et al (1979) reported slight increases in the number failed at the 74 and 68% levels but gave no details. With the help of R. J. Sherry and R. J. Sanchez of LLNL we have updated the data through Feb. 1, 1982, by which time all 24 vessels at 74% and 10 of 21 at 68% had failed. After over 41,000 h of testing, none of the 46 vessels at 50% stress level had failed. With the aid of Sherry and Sanchez, some discrepancies between tables in Toland, et al. (1978) and Barlow, et al. (1979) were corrected. Tables 2-6 present corrected results for burst and creep rupture.

Data from these tables are presented in Figure 2, along with mean value, sample size and CV for completely failed samples. The end bars denote first and last failure, rather than multiples of standard deviation, illustrating that the range of failure times covers 2 to 3 orders of magnitude. The circled number and arrow at 3400 and 2500 psi denote unfailed vessels at those pressure levels. The earlier treatments of Barlow, et al. (1979) and Hahn (1979) fitted the results at each stress level with a 2-parameter Weibull distribution using maximum likelihood to estimate the parameters. Each found that the Weibull shape parameter varied with stress level, with a slight tendency towards larger shape factor (lower CV) at lower pressures. Both papers noted an apparent change from decreasing failure rate at high storage levels to increasing failure rate at lower pressures.

Barlow, et al. (1979) approached the problem of predicting vessel life at lower storage pressures by computing the maximum likelihood estimates of mean value and Weibull shape parameter for incomplete data. Extrapolations for stresses down to 40% burst are given. They argue against the use of a power law relating stress to mean failure time, citing the dependence of the shape parameter on stress level.

Hahn (1979) used a different approach to predict failure times at lower pressures. He assumes that a statically strong vessel will also be strong in

Table 3. Ordered List of Kevlar 49/Epoxy Pressure Vessels Tested at 4300 psi.*

Rank	Vessel No.	Spool** No.	t_f h	Rank	Vessel No.	Spool No.	t_f h
1	65	2	2.2	20	51	2	55.4
2	197	7	4.0	21	208	7	61.2
3	210	7	4.0	22	138	5	87.5
4	205	7	4.6	23	245	8	98.2
5	202	7	6.1	24	83	3	101.0
6	176	6	6.7	25	36	2	111.4
7	209	7	7.9	26	166	6	144.0
8	140	5	8.3	27	31	2	158.7
9	66	2	8.5	28	147	5	243.9
10	42	2	9.1	29	124	4	254.1
11	49	2	10.2	30	13	1	444.4
12	87	3	12.5	31	244	8	590.4
13	135	5	13.3	32	241	8	638.2
14	220	7	14.0	33	17	1	755.2
15	94	3	14.6	34	21	1	952.2
16	170	6	15.0	35	23	1	1108.2
17	90	3	18.7	36	122	4	1148.5
18	46	2	22.1	37	109	4	1569.3
19	217	7	45.9	38	105	4	1750.6
				39	120	4	1802.1

*From Barlow, et al. (1979).

**From Toland, et al. (1978).

Table 4. Ordered List of Kevlar 49/Epoxy Pressure Vessels Tested at 4000 psi*

Rank	Vessel No.	Spool** No.	t_f h	Rank	Vessel No.	Spool No.	t_f h
1	80	3	19.1	13	69	2	544.9
2	75	3	24.3	14	237	8	554.2
3	79	3	69.8	15	29	1	664.5
4	60	2	71.2	16	61	2	694.1
5	77	3	136.0	17	110	4	876.7
6	55	2	199.1	18	12	1	930.4
7	57	2	403.7	19	185	6	1254.9
8	44	2	432.2	20	118	4	1275.6
9	15	1	453.4	21	119	4	1536.8
10	74	2	514.1	22	26	1	1755.5
11	191	6	514.2	23	247	8	2046.2
12	183	6	541.6	24	108	4	6177.5

*From Barlow, et al, (1979).

**From Toland, et al, (1978).

Table 5. Ordered list of Kevlar 49/Epoxy Pressure Vessels Tested at 3700 psi.*

Rank	Vessel No.	Spool** No.	t_f h	Rank	Vessel No.	Spool No.	t_f h
1	174	6	225.2	13	164	6	6271.1
2	200	7	503.6	14	236	8	7332.0
3	93	3	1087.7	15	243	8	7918.7
4	39	2	1134.3	16	184	6	7996.0
5	50	2	1824.3	17	234	8	9240.3
6	45	2	1920.1	18	240	8	9973.0
7	56	2	2383.0	19	25	1	11,487.3
8	92	3	2442.5	20	153	5	11,727.1
9	248	8	2974.6	21	114	4	13,501.3
10	35	3	3708.9	22	2	1	14,032
11	231	8	4908.9	23	112	4	29,808
12	70	2	5556.0	24	14	1	31,008

*From Barlow, et al. (1979).

**From Toland, et al. (1978).

Table 6. Ordered list of Kevlar 49/epoxy pressure vessels tested at 3400 psi.*

Rank	Vessel No.	Spool No.**	t_f h	Rank	Vessel No.	Spool No.**	t_f h
1	204	7	4,000		3	1	
2	218	7	5,376		16	1	
3	163	6	7,320		18	1	
4	95	3	8,616		27	1	
5	142	5	9,120	11-	106	4	>41,000
6	68	2	14,400	21	111	4	(no failures)
7	180	6	16,104		117	4	
8	155	5	20,231		127	4	
9	171	6	20,233		224	8	
10	134	5	35,880		228	8	
					250	8	

*From Barlow, et al, (1979).

**From Toland, et al, (1978).

creep rupture. He hypothesizes that a proof test would guarantee a vessel's life to a certain level of reliability. He circumvents the objection to a power law relationship between pressure and failure time by assuming that the lifetime distribution is exponential at each pressure (Weibull shape parameter = 1). Hahn justifies his assumptions by choosing the more conservative alternative at each step.

Each of these treatments appeared well founded statistically, and could be expected to give reasonable estimates of failure statistics on similar vessels at lower pressure levels. Their weakness is in predicting the first vessel failure -- from a design standpoint even one failure of a gas-filled vessel could be lethal. The extreme variation of the data dictates high proof and low storage pressure to gain reliability. For example, to guarantee .99999 reliability for five years storage life, Hahn's approach would require the vessel to be proofed to about 76% of burst, and stored at only 22% of static burst. Even with this large a safety factor, the designer has no way of guarding against failure in a vessel with slightly inferior materials or processing.

Instead of trying to fit the data with a particular distribution, we focused in on those vessels which failed either very early or very late at a particular pressure. Our goal, as mentioned earlier, was to uncover any peculiarity in material or processing which might cause early or late failure. In this task we were aided immensely by the completeness of the tables in the original report by Toland, et al. (1978). It seemed plausible that the processing conditions (gel or cure time and temperature, humidity), constituents (fiber, or matrix volume), the fiber used (eight different spools of Kevlar 49), the liner or the operator might explain the very low or high values. Some of these items could be dismissed quickly: one operator made all the vessels and resin cure schedule varied only slightly. Others, such as fiber weight, fiber volume percent, epoxy weight and

pot life did not seem to affect the results in any systematic way. Only the particular spool of yarn used to wind the vessel seemed to correlate with failure time. In Tables 2-6, we have also listed the spool used to wind the vessel. Notice that at 4300 psi, spool 7 vessels exhibit mostly low or early failures, while spool 4 vessels were the last four to fail at 4300 psi. At 4000 psi, no spool 7 vessels were tested, but four of the first five failures were from spool 3, while four of the last eight to burst were from spool 4. To investigate this further, mean values and standard deviations for vessels from each spool at every test condition were calculated. Results are tabulated in Tables 7-11, along with statistics for the entire population at that level. The first striking result from these calculations on the creep rupture tests is that the CV for vessels from each spool is less than the CV of the entire sample. Second, the order rank of mean failure time of vessels made from a particular spool is very nearly constant at every level. This is illustrated in Figure 3, which are the data from Fig. 2 replotted as a function of spool mean failure time. It is seen that spool 4 vessels always had the highest mean, spool 7 (when present) the lowest, and the order rank 7-3-2-6-5-8-1-4 was repeated with minor perturbations at each stress level. Only the static burst results differed, as shown in Table 12, where vessels from spools 5, 8 and 3 were in noticeably different positions.

This surprising and rather simple finding raises several interesting questions. First, can it be shown that the observed spool-to-spool variation has statistical significance, i.e., that it is not just due to random fluctuations. If this is true, then part of the variation in failure time is due to averaging together eight distinct subpopulations. Second, if the effect is real, what physical or chemical differences in the fibers cause the spool-to-spool variation. If such a distinction could be found, it would offer a quality control standard against which to compare current material. Third, can this finding be used to give more reliable life predictions at lower pressures. The observed reduction in standard

Table 7

Burst Pressure Data

Spool No.	Sample Size	No. Failed	P _{min} psi*	$\bar{P}$ psi	P _{max} psi	s _p psi	CV %
All	29	29	4,640	5,018	5,400	188	3.76
5	3	3	4,640	4,867	5,070	215	4.44
7	5	5	4,735	4,881	4,938	85.8	1.76
2	5	5	4,750	4,927	5,160	148	3
8	1	1	-----	4,950	-----	-----	-----
6	2	2	5,000	5,020	5,040	28.3	.56
3	5	5	4,710	5,047	5,340	226	4.48
1	6	6	4,800	5,101	5,300	168	3.29
4	2	2	5,225	5,313	5,400	124	2.33
All except 7	24	24	4,640	5,028.9	5,400	194.8	3.87

*Divide by 145 to obtain MPa.

Table 8

4300 psi

Spool No.	Sample Size	No. Failed	$t_{\min}$ h	$\bar{t}$ h	$t_{\max}$ h	s_t h	CV %
All	39	39	2.2	315.4	1,802	515.4	163
7	8	8	4	18.4	61.2	22.3	120
3	4	4	12.5	36.7	101	42.9	117
2	8	8	2.2	47.2	158.7	58	123
6	3	3	6.7	55.2	144	77	139
5	4	4	8.3	88.3	243.9	109	124
8	3	3	98.2	442	638.2	298	67.6
1	4	4	444.4	815	1,108.2	286	35.1
4	5	5	254.1	1,304	1,802.1	641	49.1
All except 7	31	31	2.2	392	1,802.1	553	141

Table 9

4000 psi

Spool No.	Sample Size	No. Failed	$t_{\min}$	$\bar{t}$	$t_{\max}$	s_t	CV
			h	h	h	h	%
A11	24	24	19.1	903.7	6,177.5	1,251	138
7	0	0	--	--	--	--	--
3	4	4	19.1	62.3	136	54.2	86.9
2	7	7	71.2	408.5	694.1	211.9	51.8
6	3	3	514.2	770	1,254.9	420	54.5
5	0	0	--	--	--	--	--
1	4	4	453	950.9	1,755.5	570	60
8	2	2	554	1,300	2,046	1,055	81
4	4	4	876.7	2,466	6,177.5	2,488	101

Table 10

3700 psi

Spool No.	Sample Size	No. Failed	t_{min} h	$\bar{t}$ h	t_{max} h	s_t h	CV %
All	24	24	225.2	7,873	31,008	8,114	103
7	1	1	-----	503.6	-----	---	--
3	2	2	1,087.7	1,765	2,442	957	54.2
2	6	6	1,134.3	2,754	5,556	1,617	58.7
6	3	3	225.2	4,830	7,996	4,080	84.5
8	6	6	2,974.6	7,058	9,973	2,659	37.7
5	1	1	-----	11,727	-----	---	--
1	3	3	11,487	18,842	31,008	10,612	56.3
4	2	2	13,501	21,654	29,808	11,530	53.2
All except 7	23	23	225.2	8,193.9	31,008	8,140	99.3

Table 11

3400 psi

Spool No.	Sample Size	No. Failed	$t_{\min}$ h	$\bar{t}$ h	$t_{\max}$ h	s_t h	CV %
All	21	10	4,000	>35,000	>41,000		
7	2	2	4,000	4,688	5,376	972	20.7
3	1	1	-----	8,616	-----	-----	-----
2	1	1	-----	14,400	-----	-----	-----
6	3	3	7,320	14,552	20,233	6,595	45
5	3	3	9,120	21,742	35,880	13,443	61.8
8	3	0	>41,000				
1	4	0	>41,000				
4	4	0	>41,000				

Table 12

Order Rank Based on Mean Value
(by spool)

Static Burst $\bar{p}_F$	4300 psi $\bar{t}$	4000 psi*** $\bar{t}$	3700 psi $\bar{t}$	3400 psi $\bar{t}$
5**	7		7*	7
7	3	3	3	3*
2	2	2	2	2*
8*	6	6	6	6
6	5		8	5
3**	8	1	5*	8
1	1	8	1	1
4	4	4	4	4

Increasing Pressure or Time
 ↓

{ 8 }
 { 1 }
 { 4 }

No Failures

*Only 1 vessel tested at particular condition.

**CV > that of total sample.

***Spools 5 and 7 were not represented in sample.

deviation should permit more accurate prediction of design allowable life. Fourth, what does this spool-to-spool variation imply about the use of elevated pressure to accelerate failure. These questions will be addressed in the following sections.

Statistics of Spool Variation

To establish whether the observed spool-to-spool variation was statistically significant, two statistical tests were performed. First the data from each test condition were analyzed to determine the likelihood that the observed behavior could occur in an homogeneous sample. For this purpose the Kruskal-Wallis, or Rijkoot, Test was employed (Kraft and van Eeden, 1968). This technique tests the homogeneity of a sample by comparing the nonparametric means of the individual subgroups which compose the sample with the mean of the total sample. At each test condition (static burst and three creep rupture conditions) the statistic of interest is given by

$$H_k = \frac{12}{N_k(N_k + 1)} \sum_{j=1}^J \frac{(R_{jk} - n_{jk}\bar{R}_k)^2}{n_{jk}} \quad (1)$$

Here N_k is the total sample size at test condition k ($k = 0$ corresponds to static, $k = 1, 2, 3$ the creep rupture tests). R_{jk} is the sum of the nonparametric ranks for vessels from spool j , n_{jk} is the number of vessels from spool j in the test, and $\bar{R}_k$ the sample mean, given by

$$\bar{R}_k = \frac{1}{N_k} \sum_{j=1}^J R_{jk} = \frac{N_k + 1}{2} \quad (2)$$

The number of spools, J , was 8 in each test except for the 4000 psi creep rupture test, where only 6 were represented (spools 5 and 7 vessels were not tested). For reasonably large sample sizes, the significance of H_k can be determined from a χ^2

distribution with $J - 1$ degrees of freedom. For each test condition the value for R_{jk} is the sum of the failure rank orders for spool j as given in Tables 2-6. Results of the Kruskal-Wallis Test are given in Table 13. When the probability α that $P(H > \chi^2_{\alpha} ; J - 1)$ takes on a value near 0.1, the hypothesis of homogeneity is suspect; for values considerably less than 0.1, the hypothesis should be rejected. Thus it appears that, while the static burst population might possibly be homogeneous, the creep rupture populations definitely are not. It can be concluded that some of the variability in failure time is due to the averaging together of either 8 or 6 distinct groups.

The second calculation performed was an analysis of variance, which can be used to test parametrically the hypothesis that the spool means are equal (Mendenhall, 1975). Since data from each long-term test spanned two-to-three orders of magnitude in time, a variable y_{ijk} can be constructed to pool data from tests at 4300, 4000 and 3700 psi for each spool:

$$y_{ijk} = \frac{\ln t_{ijk} - \bar{y}_k}{s_k} \quad (3)$$

Here t_{ijk} is the i th failure time ($i = 1, 2, \dots, n_{jk}$) of the j th spool, at pressure p_k . The sample mean $\bar{y}_k$ and variance s_k^2 are given by:

$$\bar{y}_k = \frac{\sum_{j=1}^J \sum_{i=1}^{n_{jk}} \ln t_{ijk}}{N_k} \quad (4)$$

$$s_k^2 = \frac{\sum_{j=1}^J \sum_{i=1}^{n_{jk}} (\ln t_{ijk} - \bar{y}_k)^2}{N_k - 1} \quad (5)$$

Table 13. Kruskal-Wallis Test
For Hypothesis of Sample Homogeneity

Test	No. of Spools Tested J	Degrees Freedom (J-1)	H	α
Burst	8	7	12	.101
4300 psi	8	7	25	.001
4000 psi	6	5	17	.004
3700 psi	8	7	18	.012

The results of the analysis of variance test on y_{ijk} are shown in the following table.

Analysis of Variance

Source	Degrees of Freedom	Sums of Squares	Means of Squares	F
Spools	7	51	7.3	17
Error	79	33	.42	

This large value of F is very significant; the probability of finding an F this large for the degrees of freedom in the creep rupture experiment is less than 10^{-6} . This result, coupled with the Kruskal-Wallis test, are convincing evidence that the effect of spool variability on long term failure is real.

The mean failure time, as measured by y_{ijk} , of vessels from spool j is given by

$$\bar{y}_j = \frac{\sum_{k=1}^3 \sum_{i=1}^{n_{jk}} y_{ijk}}{N_j} \quad (6)$$

where $N_j = \sum_{k=1}^3 n_{jk}$

These values for the eight spools are shown below, ranked from smallest to largest.

Spool (j)	3	7	2	6	5	8	1	4
$\bar{y}_j$	-1.0	-.95	-.41	-.23	0.0	.51	.92	1.23

The spools appear to divide into several groups, with 3 and 7 yielding inferior performance vessels and 1 and 4 definitely superior.

From the above two calculations it is safe to conclude that the spool-to-spool variability is real, and could not have occurred by chance. The population is definitely heterogeneous. It also appears that the observations in previous analyses about scatter and failure rate at a particular pressure should be reexamined in light of this fact. Variance and rate of failure at a particular storage pressure is dependent, at least in part, on the number of vessels from each spool tested at that condition.

Origin of the Variability

The investigation to determine the cause of the spool variability, being conducted by S. C. Kunz at Sandia National Laboratories, is not complete at this time. Certain results have been obtained, however, and will be summarized in this section.

1. Virgin Yarn. Of the eight spools used to wind the original 250 vessels, only two still existed as virgin yarn, numbers 2 and 7. Mechanical test data on single fibers taken from these spools are summarized in Table 14. Failure load of #7 fibers was considerably higher, in fact above the 50 g maximum of our fiber tester. Since the performance of spool 7 vessels was below those of spool 2 both statically and in creep rupture, this high strength was unexpected. This anomaly was explained when microscopy revealed that the diameter of spool 7 fibers was about 17 μ , some 6 μ larger than nominal. Further, spool 7 yarn had only 127 filaments/yarn, instead of the 267 expected in a 380 denier Kevlar 49. The poorer performance of spool 7 is explained by its low yarn strength, 293 ksi, calculated on actual cross sectional area. How this material was made and then eluded the manufacturer's quality control is not clear. Density measurements at Sandia confirmed that the yarn denier of spool 7 was very close to 380, which could explain how it passed inspection.

Table 14

380 Denier Kevlar 49

	<u>Nominal</u>	<u>Spool #2</u>	<u>Spool #7</u>
Fiber Failure Load (g)	30-40	25-35	>50
Yarn Failure Load (lb)	16	14.5	13.1
Fiber diameter (μ)	11	11.5	17
Fibers/Yarn	267	263	127
Yarn Strength (ksi)	400	375	293

2. Failed Vessels. Material from the remaining six spools exists only in the composite laminate on vessels. Discussions with Toland and LLNL personnel confirmed that no impregnated strands were made from the original spools. Vessels failed at 4300 and 4000 psi had not been retained. Vessels from the 3700 psi test were acquired from LLNL and sectioned to obtain material for metallographic analysis. No obvious differences -- such as diameter or number of filaments/ yarn -- were present in the remaining seven spools. All material appeared to be within DuPont's specification for those quantities. Mechanical and chemical testing of material from the remaining seven spools is underway. Although not yet definite, there is some indication of a difference in failure mechanism among the seven spools. This will be the subject of a later report. If the variability can be correlated to some feature of fiber morphology, then it should be possible to identify the processing step which controls that feature. This opens the exciting possibility of Kevlar 49 vessels which uniformly exhibit the high performance of spool 4.

The existence of the unusual Kevlar 49 of spool 7 dictated that the statistical analyses of the previous section be redone. Exclusion of spool 7 vessels affected the analysis of variance results only slightly and did not alter the original conclusion. In the Kruskal-Wallis test, only the 4300 psi results were affected, there being no spool 7 vessels at 4000 psi and one at 3700 psi. The statistic H_1 for 4300 psi results without spool 7 is 20.8, which results in a probability α of occurrence for 6 degrees of freedom of .0027, well within the region where the hypothesis of homogeneity should be rejected.

A more interesting result caused by excluding spool 7 vessels from the 4300 psi tests is the change in variance. The CV in 4300 psi tests is reduced from 163 to 141% (see Table 3). This confirms the earlier speculation that total sample variance at particular pressure is a function of the particular mix of spools

rather than an inherent material characteristic. In any modeling of the failure time it will be necessary to preserve the characteristics of the individual spools.

Relationship Between Failure Time and Pressure

It is evident from the data in Figure 3 that failure time is also a strong function of pressure. Determination of a pressure-time relationship for each spool is not straightforward. The task is made difficult because, first of all, the original experiment was not designed to provide equal or adequate numbers of samples of each spool at every pressure. For example, the most represented spool is number 2 (22 vessels), but these are distributed at the four pressure levels in a nonuniform way: 8, 7, 6, 1. The remaining spools were represented by at most 12 vessels. The second difficulty is ignorance of the long-term failure mechanism in Kevlar-epoxy. Kelly and McCartney (1981) describe a slow crack growth model which leads to a power law relationship between pressure and time, but this is based on a Mode I tensile fracture which isn't observed in Kevlar fibers. Phoenix and Tierney (1981) argue that a power law describes bond breakdown more accurately than does an exponential function. However, it is still uncertain how the failure process in Kevlar should be modeled, and whether it changes as a function of time or pressure level. Without a clear picture of the failure mechanism, it is difficult to choose between, say, a power law and an exponential fit of the data.

These difficulties raise a legitimate question about the value of an empirical fit of the data from each spool. The relationships presented in this section were developed primarily because they provide the designer with a conservative estimate of safe working life, or design allowable, at lower pressures. A second reason for presenting them is that they reveal interesting differences between the behavior of the various spools which might prove useful in testing the accuracy of a theory. Indeed, two problems facing long-term failure models are large

experimental scatter and the consequent indistinguishability of trends in the data. Accounting for spool variability reduces scatter and shows trends in the data; these should help in the testing of models. A third justification for this approach is that it provides a means for determining the sensitivity of failure time to variations in pressure. Even if not exact, this information will be useful to future experiments.

In developing empirical equations we tried to adhere to the principles of simplicity and conservatism. Although better fits could probably be obtained with higher order equations, this did not seem warranted by the small sample sizes. The conservative approach is reflected by the choice of parameters and by the use of a .999999 reliability in setting design allowables. So in those cases where a simple function does not match the data terribly well, the larger standard deviations force more conservative design allowables. Although this may seem an extreme level of reliability, the consequences of a vessel failure in a manned area could be catastrophic. And, as will be shown, the penalty for attaining this level of reliability does not appear excessive.

Trends in Data

Although it is common to present stress-life data in linear-log or log-log plots, these formats can be deceiving. It is instructive to view the data in linear form. This is done in Figure 4 for the means of vessels from six of the spools; spool 7 was deleted for the reasons given previously, and spool 5 because it was not represented at 4000 psi. Its behavior falls roughly between 6 and 8. The data show the substantial superiority of spools 8, 1 and 4 over 3, 2 and 6, e.g., at 3400 psi the former have no failures after 41,000 h, while the latter have all burst. However, the data are encouraging even for the poorer material, since the projected slope of spool 3 vessels between 3400 and 2500 psi (where no failures have occurred) is still less than that between 3700 and 3400 psi. The

same decrease in $\frac{\Delta p}{\Delta t}$ (or increase in $\frac{\Delta t}{\Delta p}$) appears in the other groups as well.

From past performance the next failures seem most likely to occur in spool 8 vessels at 3400 psi. However since scatter have not been shown this is only speculation.

The pressure vs life behavior in Figure 4 suggest an exponential or power law dependence. The mean of the $\log t_{ijk}$ are plotted in Figures 5 and 6 vs pressure and log pressure, respectively. Note that the order rank of spools differs slightly from that in Figure 3 when the means of the logs are plotted. Figures 5 and 6 reveal what appear to be three distinct classes of behavior among the spools. Vessels from spools 2 and 6 exhibit a slope $\left(\frac{\Delta p}{\Delta \log \bar{t}} \text{ or } \frac{\Delta \log p}{\Delta \log \bar{t}} \right)$ which increases with $\log t$. Spool 1 and 4 vessels are just the opposite, showing a decreasing slope. Thus a prediction of failure at 3700 psi based upon data at 4300 and 4000 would have been very optimistic for spools 2 and 6, and very pessimistic for spools 1 and 4. Spool 8 vessels have nearly constant slope and lie roughly between the two types. Spool 3 vessels parallel the behavior of 1 and 4 but at 1-2 decades lower time. Whether these trends indicate something fundamental about the failure mechanism changing with time, or are simply artifacts of the logarithmic transformation cannot be determined from the data. What is important to note is that curve fits of spool 2 and 6 vessels will tend to overestimate failure times at lower pressures, while those for spool 1 and 4 vessels will tend to underestimate them. This point must be borne in mind when selecting the form of the empirical equation.

Estimation of Vessel Life at Lower Pressures

Based on Figures 5 and 6, there is little reason to select one functional form over the other. Therefore both equations were fitted and the results compared. The governing equations are:

$$t = t_1 \exp \left(b \frac{p}{p_0} \right) \quad (7)$$

and

$$t = t_o \left(\frac{P}{P_o} \right)^m \quad (8)$$

Least squares solutions for the parameters t_1 , b and t_o , m can be determined in two ways. The most common is to first transform the equations,

$$\ln t = \ln t_1 + b \frac{P}{P_o} \quad (7')$$

and

$$\ln t = \ln t_o + m \ln \left(\frac{P}{P_o} \right) \quad (8')$$

and then solve for the constants in 7' and 8' by a standard linear regression technique (Lipson and Sheth, 1973). An alternative technique, described by Daniel and Wood (1980), is to input the (t_{ijk}, p_k) values directly into Eq. (7) or (8) and then use a nonlinear regression scheme to minimize the actual (rather than transformed) residuals at each fitted point. The former technique tended to fit high pressure short-time results more accurately, since there were more data points at high pressure. The nonlinear approach tended to follow the lower pressure, longer time data, since the largest residuals occurred there. Data from each spool were fitted by each of the four approaches. The nonlinear regression program, NONLINWOOD, is discussed in detail in Daniel and Wood (1980).

The regression analyses for the two equations each yielded correlation coefficients (r , a measure of goodness of between observed and predicted values) that were large and statistically significant. Within the pressure range where failures have occurred, any of the fits could be used. Of the four fits attempted, however, the nonlinear regressive power law more accurately represented the slope of time vs pressure at low pressures, and more faithfully retained the

order rank of spools. Indeed, both of the exponential fits and the linear power law fit predicted a switching of position between the high performance (8, 1 and 4) and low performance spools (3, 2, 6) at pressures below 2500 psi. The data of Figure 4 show no such trend, it is therefore physically unreasonable to expect such a switch. Since only the nonlinear regression power law did not exhibit this peculiarity, it was chosen for extrapolations to lower pressures.

The slope change in log-log space exhibited by most of the spools (see Figure 6) indicates that a single slope power law will not completely describe the entire behavior of a spool. The simplest alternative is to use, when necessary, a bilinear approach, with a linear regression power law describing the higher pressure results and the nonlinear describing the lower. Values of the necessary parameters for each spool are given in Table 15. These are plotted in Figures 7-12 for six spools, with numbers 7 and 5 excluded for the reasons given earlier. Notice that the nonlinear fit $\hat{t}$ for spools 4 and 1 has a considerably lower slope (more negative m) than does the linear fit t' . In spools 3, 2 and 6 the situation is reversed. Spool 8 vessels, which showed little curvature are well represented by either type of fit. There is some indication that, depending upon the behavior of spools 8, 1 and 4 at 3400 psi, the materials are converging to a similar slope of $m \approx -20$. Also shown in Figures 7-12 is a design allowable life, t_a , which is derived in the following section.

Derivation of Design Allowables

The fitted curves in Figures 7-12 can be used to estimate the mean at non-tested pressures if the failure mechanism and distribution of that spool does not change. What is more important for design is a bound below which no failures should occur. To obtain a design allowable life it is necessary to assume a distribution of failure times about $\hat{t}$ or t' and then estimate its standard deviation about the fitted values. The assumed distribution can have only positive

Table 15

Parameters for Power Law Equations

Spool	Linear Regression t'			Nonlinear Regression $\hat{t}$		
	t'_o	m	r	$\hat{t}_o$	m	r
	h			h		
4	3,068	-17.53	-.773	2,784	-26.29	-.896
1	2,045	-19.88	-.828	1,523	-32.19	-.868
8	1,320	-20.07	-.902	1,385	-20.88	-.864
6	290.2	-24.475	-.708	1,395	-14.46	-.873
2	231.7	-30.13	-.90	561.7	-19.98	-.97
3	104.4	-26.36	-.896	319.2	-20.3	-.99

t'_o and $\hat{t}_o$ are determined for $p_o = 4000$ psi.

values of t and should agree qualitatively with the observed values. A log normal distribution satisfies these requirements. The standard error of the estimate, $s_{t \cdot p}^{\wedge}$, is the measure of scatter of failure times about the fitted line (Lipson and Sheth, 1973).

$$s_{t \cdot p}^{\wedge} = \sqrt{\frac{\sum_{k=1}^K \sum_{i=1}^{n_{jk}} (\ln t_{ijk} / \hat{t}_{jk})^2}{N_j - 2}} \quad (9)$$

where $K = 3$ or 4 depending upon j . The value of $s_{t' \cdot p}$ may be calculated from the same equation or by using the alternative forms in Lipson and Sheth (1973).

If the assumption of a log normal distribution about $\hat{t}$ (or t') is true and if (9) is close to the true standard deviation, then .999999 reliability may be guaranteed by setting the log design allowable life equal to $\ln \hat{t} - 5s_{t \cdot p}^{\wedge}$, or

$$t_a = \begin{cases} \frac{t'}{e^{5s_{t' \cdot p}^{\wedge}}} \\ \frac{\hat{t}}{e^{5s_{t \cdot p}^{\wedge}}} \end{cases} \quad (10)$$

This factor will provide the desired reliability at a time $\hat{t}$ extrapolated from current data provided (1) the distribution at $\hat{t}$ is approximately log normal and (2) the standard error at $\hat{t}$ is no larger than that of the current sample. The standard errors about $\hat{t}$ were calculated both in pooled form and at individual pressures. The error at the lower pressures was always less. The pooled values of standard error for each spool are given in Table 16.

Table 16

Spool	$s_{t'.p}$	$e^{5s_{t'.p}}$	$s_{t.p}$	$e^{5s_{t.p}^{\wedge}}$	$p(t_a = 10y)$
		h		h	
4	.889	85	1.080	222	2,856
1	.885	83.6	1.264	555	2,898
8	.688	31.2	0.6930	32.2	2,777
6	1.340	811	2.092	35,020	1,890
2	1.053	194	1.570	2,570	2,387
3	1.102	247	1.689	4,654	2,312

For each spool the value of $s_{t.p}^{\wedge}$ is greater than $s_{t'.p}$. For spools 4, 1 and 8 the value for t_a selected was $\frac{\hat{t}}{e^{5s_{t.p}^{\wedge}}}$ for the entire range (Figs. 7-9).

Although very conservative at high pressures, this value should allow for possible slope changes at pressures below 3700 psi. The curves for t_a are quite conservative for spools 4 and 1 at 3700 psi, where no failures have occurred. Note also that the t_a curves are all considerably less (at least a decade) than where any actual failures have occurred.

The large values of $s_{t.p}^{\wedge}$ are caused by the poor fit of the nonlinear regression curve with data at 4300 psi. This is especially true for the poorer spools 6, 2 and 3, where $e^{5s_{t.p}^{\wedge}}$ is seen to take on enormous values. The large values of $s_{t.p}^{\wedge}$ are caused by the poor fit of the nonlinear regression curve with data at 4300 psi. Notice in Figure 10 that the plot of $\hat{t}$ passes completely outside the data at 4300 psi. A more reasonable factor for computing t_a from $\hat{t}$ would be to use $s_{t'.p}$ rather than $s_{t.p}^{\wedge}$. This is justified since the sample CV for the weaker spools at lower pressures appears to be decreasing (compare Tables 8 and 10). Also, there are a substantial number of vessels which have

survived over 41,000 h at 2500 psi from each spool. The plots of $t_a = \frac{\hat{t}}{e^{5s_t' \cdot p}}$

for spools 3, 2 and 6 are seen to be conservative in estimating this result.

Using $s_t'p$ for the error throughout results in the bilinear estimate of t_a shown in Figures 10-12.

The last column in Table 16 gives the pressure estimate for a ten year life with .999999 reliability. Differences in the standard error and slope (m) cause the change in order ranks. The lowest projection is spool 6 which would require a nominal safety factor of 2.64, based on a burst of 5000 psi. This is an acceptable safety factor to obtain such high reliability. The better spools would require safety factors of less than 2.

Effect of Pressure

Regardless of the curve fit chosen, the dependence of failure time on pressure is large. Until now, the storage pressure has been treated as a constant. In the above analyses the usual assumption is that the independent variable p is known exactly. Bias may result from the regression analysis if the independent variable is subject to error. In the tests discussed in this paper, the storage pressure was controlled to a range from +1% to -3%, but the actual value was not recorded. The effect of this variation on the regression analyses would be small (Daniel and Wood, 1980), but its effect on failure time could be substantial due to the strong pressure dependence. Figure 13 gives bounds on failure time as a function of m . From this plot it is clear that the pressure range in this experiment could produce failure times differing by a factor of two in identical materials. Future creep rupture tests should have pressure or stress controlled to $\pm 1\%$.

Elevated pressure or stress has long been used to accelerate failure in creep rupture tests. The validity of this approach for Kevlar epoxy is qualitatively supported by the consistent order ranking in mean failure time of the various

spools. However, the lack of a complete data set and scatter in the failure times make it difficult to guarantee that raising the pressure has not affected the failure mechanism.

In this regard the question of why static burst order ranks differ from long term can be raised. Of the seven spools having similar 11 μ diameter Kevlar 49, four showed the same relative behavior statically as in long term (2, 6, 1, 4). Number 8's low static position might be explained by its being represented by only one vessel. But the high static performance of spool number 3, which consistently had the poorest long-term behavior cannot be explained so easily. Perhaps the extreme sensitivity of failure time to pressure clouds the static behavior. Or, a different failure mechanism may be operative. Regardless of the reason, the evidence in this study argues that high static strength is not a certain guarantee of long life. It is also clear that a short term-high pressure creep rupture test is far superior to static burst as an indicator of long-term life.

From a design standpoint the pressure sensitivity dictates setting a factor on operating pressure which would account for any fluctuations. Its magnitude would depend on the service environment and on the fluid being contained.

Conclusions

In this study we have investigated the static and creep-rupture behavior of Kevlar 49-epoxy pressure vessels with the aim of uncovering sources of variation which would help explain the large scatter in long term. The results are as follows:

1. A spool-to-spool variability exists among the eight used to wind the vessels in the study. This variability was shown to be statistically significant in long term tests.
2. Short term burst tests did not correlate with long term in all cases. Burst strength is a questionable indicator of life.

3. Identification of the spool-to-spool variation shows that Kevlar long-term behavior is considerably more predictable than first thought. Scatter is considerably reduced by accounting for spool effects and a pattern to the failures can be seen.
4. Each spool could be fitted reasonably well by a power law relating failure time to pressure. A design allowable of reliability .999999 was then computed based upon the standard error of the data about that line. An overall safety factor of between 1.8 (good spools) to 2.7 (poorer spools) would guarantee 10 years of life.
5. Extreme sensitivity of failure time to pressure argues for very tight limits on pressure (or stress) in creep rupture experiments.

Finally, although the reason for spool-to-spool variations has not yet been traced back to a material difference, the variation found here should be of use to designers of Kevlar vessels. Also it should underscore the value of saving some material from each spool so that, when the mechanism becomes better understood, material in current service can be compared to standards.

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Figure 1.

From Hahn (1979)

Figure 2.

KEVLAR EPOXY PRESSURE VESSELS

Figure 3.

FAILURE TIME-MEAN FOR EACH SPOOL (1-8)

Figure 4.

Fig 5

Fig 6

Fig 7

Fig 8

Storage Pressure (ksi)

Spool #1

$$t_a = \hat{t} / 555$$

4.3

4.0

3.7

3.0

3.4

2.5

1

10

10^2

10^3

10^4

10^5

10^6

10^7

Failure Time (h)

Fig 9

Storage Pressure (ksi)

Spool #8

$$t_a = \hat{t} / 32.23$$

t'

$\hat{t}$

④

③

1 10 10^2 10^3 10^4 10^5 10^6 10^7

Failure Time (h)

Fig 10

Fig 11

Fig 12

Figure 13.