PERFORMANCE OF TAPE-BONDED SEAMS OF EPDM MEMBRANES: THE EFFECT OF LOAD ON PEEL-CREEP

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PERFORMANCE OF TAPE-BONDED SEAMS OF EPDM MEMBRANES: THE EFFECT OF LOAD ON PEEL-CREEP

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A study was conducted to compare the peel creep-rupture response (i.e., time-to-failure or TTF) of tape-bonded and liquid-adhesive-bonded seams of EPDM (ethylene-propylene-diene terpolymer) roof membranes. Two commercial tape systems (i.e., tape and primer) and one liquid adhesive were applied to well-cleaned EPDM rubber in the NIST laboratories. The creep-rupture experiments were conducted at 23°C (73°F) and 40 percent to 45 percent relative humidity under peel loads ranging from 3.1 N to 24.9 N (0.7 lbf to 5.6 lbf). For each adhesive system, the data were found to be fitted well by the model: ln(mean TTF) = $b_0 + b_1 \bullet$ Load + $b_2 \exp(b_3 \bullet$ Load). The conclusion was that the tape-bonded sample sets had mean times-to-failure that were, in most cases, comparable to or greater than those of the liquid-adhesive-bonded sample sets. In addition, the tape-bonded specimens provided time-to-failure results that were reproducible between replicate sets. In a related experiment, the creep-rupture response of six sets of tape-bonded specimens prepared in the laboratories of EPDM membrane manufacturers was compared with that of NIST-prepared tape-bonded sample sets. Five of six manufacturer-prepared sample sets had similar, or longer, times-to-failure than the NIST-prepared sample sets at low creep loads. In the case where the manufacturer-prepared sample sets had a statistically significant shorter time-to-failure than the NIST-prepared sample sets, the difference was not practically important.

KEYWORDS

Adhesive tapes, adhesive testing, bonding, building technology, creep-rupture, EPDM roofing membranes, microscopy, roofing, seams, time-to-failure.

INTRODUCTION

Since the early 1990s, the use of preformed adhesive tapes for bonding seams of EPDM (ethylene-propylene-diene terpolymer) roof membranes has increased significantly in the United States, and this trend is expected to continue.[1] Factors contributing to the increased use include: reduced volatile organic compound (VOC) emission during seam fabrication, ease of application, decreased application time, and the application of an adhesive system having uniform dimensions.[2] Limited experience to date with current tape systems has shown that performance has been satisfactory.[3] Nevertheless, some roofing contractors and consultants have urged that independent studies on the performance of tape-bonded seams be conducted.

In response to this need, three EPDM membrane manufacturers (Carlisle SynTec, Firestone, and GenFlex) and two tape-system manufacturers (Adco and Ashland) along with two trade associations (the National Roofing Contractors Association and the Roof Consultants Institute) have undertaken a consortium research project with NIST through a Cooperative Research and Development Agreement. The U.S. Army Construction Engineering Research Laboratories also was a sponsor. The objectives of the research project were to compare the creep-rupture performance of tape-bonded and liquid-adhesive-bonded seams in EPDM roof membranes and to develop a creep-test protocol for tape-bonded seams.

This paper summarizes Phase I of the consortium research project; details of the research have been previously published.[3] In Phase I, laboratory investigations were conducted to compare the creep-rupture response of tape-bonded and liquid-adhesive-bonded seam specimens as a function of load. In a creep-rupture experiment, a seam specimen of a fixed length is stressed under a constant load and the time over which it sustains the load until total separation (i.e., the time-to-failure) is recorded. In the Phase I investigations, seam specimens were prepared using two tape-adhesive systems and one liquid-adhesive system. The short-term peel strengths of the specimens were measured, and the creep-rupture tests were conducted under peel loads varying from 3.1 N to 24.9 N (0.7 lbf to 5.6 lbf) in increments of 3.1 N (0.7 lbf). As will be discussed, the results of the investigation indicate that, in general, the tape-adhesive sample sets had mean times-to-failure that were comparable to, or greater than, those of the liquid-adhesive-bonded sample sets.

EXPERIMENTAL

Seam Specimen Preparation and Replicate Specimen Sets

The two commercial tape systems (i.e., tape and primer) were designated TS1 (Tape System 1) and TS2 (Tape System 2); the commercial butyl-based liquid adhesive was designated LA. This liquid adhesive cures through a moisture-induced reaction. T-peel and creep-rupture seam specimens having dimensions of 25 mm by 125 mm (1 in by 5 in) with a 75-mm (3-in) bond were prepared using a commercial, well-cleaned EPDM sheet. All specimens were kept at room temperature, 23°C±2°C (73°F±4°F),* for at least 28 days prior to testing. For experimental details, refer to References 3, 4, and 5.

Replicate sets of specimens (i.e., different batches) were prepared at different times to investigate the reproducibility

*Temperature variations in this paper are absolute bounds.

of the peel-strength and creep-rupture data. Two replicate sets of TS1 specimens, four replicate sets of TS2 specimens, and five replicate sets of LA specimens were included in the Phase I study (Table 1). A replicate set generally contained between 80 and 100 specimens.

For TS1, the two replicate sets were prepared from the same roll of tape and can of primer (designated TS1-1). For TS2, the four replicate sets were fabricated from the same roll of tape, but three different cans of primer (designated TS2-1, TS2-2, and TS2- 3) were used. TS2 Replicate Sets Nos. 1 and 2 were prepared using the same can of primer. Examination of the can of TS2-1 primer after testing some of the TS2 Replicate Set No. 2 specimens showed that the primer was gelled. This observation raised the possibility that the TS2-1 primer may have been close to its shelf life when used to prepare the TS2 Replicate Set No. 2 specimens, although no evidence of gelling was apparent when the primer was used.* Because the TS2-1 primer had gelled, a second can of the primer (TS2-2) was used to prepare the TS2 Replicate Set No. 3 specimens. When this third replicate set was being prepared, it was brought to NIST's attention that the formulation of the TS2 primer had been changed, and that the one used in preparing the TS2 Replicate Sets Nos. 1, 2, and 3 was no longer available. Consequently, a can of the newly formulated primer was obtained, and the TS2 Replicate Set No. 4 specimens were prepared. As reported by the tape system manufacturer, the difference between the formulations of the two primers was in their solids contents. TS2-1 and TS2-2 had 10 percent solids, whereas TS2-3 had 5 percent solids.

For the liquid-adhesive-bonded specimens, the major difference among replicate sets was the can from which the adhesive (designated LA-1, LA-2, and LA-3) was taken (Table 1). LA Replicate Sets Nos. 1, 2, and 4 used different cans. LA Replicate Sets Nos. 2 and 3 were prepared from the same can. Similarly, LA Replicate Sets Nos. 4 and 5 used the same can but, in this case, the LA Replicate Set No. 5 specimens were prepared by a liquid adhesive manufacturer's representative and not by a NIST research staff member.

Peel-Strength Tests

From each replicate set, four specimens were randomly selected to determine T-peel strength at room temperature, 23°C±2°C (73°F±4°F), at a crosshead rate of 50 mm/min (2 in/min). The universal testing machine was equipped with hardware and software for recording and calculating strength data. After testing, each specimen was visually examined and the mode of failure, adhesive or cohesive, was noted. The results of the peel-strength tests were used to determine the applied load to peel-strength ratio when conducting the creep-rupture tests.

Creep-Rupture Tests

A minimum of eight specimens were randomly selected from the replicate sets and assigned to one of eight loads ranging from 3.1 N to 24.9 N (0.7 lbf to 5.6 lbf) in increments of 3.1 N (0.7 lbf). This represented a range of loads from 5 percent to 40 percent of the force required to delaminate a 25-mm- (1-in) wide specimen having a 2.5 kN/m (14 lbf/in) peel strength. The creep-rupture tests were conducted in peel at room temperature, 23°C±2°C (73°F±4°F), in laboratory-constructed chambers according to the general procedure described in Martin, Embree, Stutzman, and Lechner.[6] The relative humidity in the chambers was maintained between 40 percent and 45 percent using a saturated potassium carbonate solution.[7] Specimens were conditioned for a minimum of 16 hours in the chambers before applying the load.

RESULTS AND DISCUSSION

Peel-Strength Results

Short-term peel strength measurements were conducted as a quality check for determining if a set of specimens should be accepted for a creep experiment. If the results of the peel strength measurements were not typical of past strength data for well-made seam specimens, then the replicate set would have been rejected and a new set prepared. Table 2 summarizes the peel strength data, including a description of the major failure mode observed during testing. With the excep-

Adhesive System[a]	Rep. No.[b]	Primer Designation	Adhesive Designation	Comment[c]
TS1	1	TS1-1	NA[d]	• First can of primer used for the first time.
	2	TS1-1	NA	• First can of primer used for a second time.
TS2	1	TS2-1	NA	• First can of primer (10% solids) used for the first time.
	2	TS2-1	NA	• First can of primer (10% solids) used for a second time.
	3	TS2-2	NA	• Second can of primer (10% solids) used for the first time.
	4	TS2-3	NA	• Third can of primer (5% solids) used for the first time; the primer having the 10% solids content was no longer available.
LA	1	NA	LA-1	• First can of adhesive used for the first time.
	2	NA	LA-2	• Second can of adhesive used for the first time.
	3	NA	LA-2	• Second can of adhesive used for the second time.
	4	NA	LA-3	• Third can of adhesive used for the first time.
	5	NA	LA-3	• Third can of adhesive used for the first time; that is, LA Replicate Set Nos. 4 and 5 were fabricated at the same time.

[a]TS1, TS2, and LA indicate Tape System 1, Tape System 2, and Liquid Adhesive, respectively.
[b]Rep. No. indicates the replicate set number.
[c]All specimens were prepared by NIST research staff with the exception of the liquid adhesive (LA) Replicate Set No. 5.
[d]NA indicates not applicable.

Table 1. Replicate sets of test specimens.

* Experimentation on primer shelf life was beyond the scope of the consortium research study and was not conducted.

tion of TS2 Replicate Set No. 2, all specimens failed cohesively. Adhesive failure of TS2 Replicate Set No. 2 may have been due to the use of primer that may have exceeded its shelf life, as described previously.

Table 2 also compares the peel-strengths determined in the current study with published values.[4, 5, 6] For the tape-bonded specimens, the peel strengths were generally comparable to those previously published. For the liquid-adhesive-bonded specimens, the peel-strengths were also comparable to, if not slightly greater than, published values. Note in Table 3 that the TS2 Replicate Set No. 2 specimens, which failed adhesively, had the lowest mean value, 2.07 kN/m (11.8 lbf/in) among the TS2 replicate sets. Because this strength value was only about 6 percent lower than that previously reported, the creep tests on the TS2 Replicate Set No. 2 specimens were performed.

Creep-Rupture Results

Table 3 summarizes the time-to-failure data. With the exception of the TS2 Replicate Set No. 2, the dominant failure mode was cohesive, although some specimens of both tape systems showed small areas of adhesive failure. As indicated in Table 3, no specimens failed when tested at 3.1 N (0.7 lbf). At the time of writing this paper, the tests at 3.1 N (0.7 lbf) were ongoing, and the specimens had been under load for more than 16,800 hours (about 23 months) without observing failures.

Statistical Analysis

As noted in the introduction, a primary objective of the Phase I study was to compare the creep-rupture performance of tape-bonded seams to that of liquid-adhesive-bonded seams. To make this comparison, the analytical approach was to fit various functions relating mean time-to-failure to load for each combination of adhesive system and replicate. One reason for using the mean time-to-failure was that the considerable scatter in the individual data sets could obscure differences in failure time caused by load and adhesive system.[5] For each function considered, the resulting curves were graphed on a single plot. The closeness of the data to the resultant curves provided a measure of goodness of fit. The relationships among the curves on the plots provided a basis for addressing the relative creep performance of the tape-bonded seams vis-à-vis the liquid-adhesive-bonded seams.

The time-to-failure decreased with increasing load, and the creep-rupture data were found to be fitted well by the model:

$$ln\,(TTF) = b_0 + b_1 L + b_2 e^{b_3 L} \qquad [1]$$

where TTF denotes the mean time-to-failure in hours, L is load in Newtons (or pounds force), and b_0, b_1, b_2, and b_3 are empirical constants. This model had been used by Bastenaire

DATA—CURRENT STUDY								DATA—PAST STUDIES	
Adhesive System[a]	Rep. No.	Strength, kN/m (lbf/in) Min.	Max.	Mean[b]	sd[c]	CoV[d] %	Failure Mode	Mn. Strength kN/m (lbf/in)	Literature Reference
TS1 (TS1-1)	1	1.83 (10.5)	1.98 (11.3)	1.91 (10.9)	0.06 (0.34)	3.1	Cohesive	1.8 (10.3)	[5]
TS1 (TS1-1)	2 (10.2)	1.79 (10.4)	1.82 (10.4)	1.81 (0.09)	0.02	0.9	Cohesive		
TS2 (TS2-1)	1	2.35 (13.4)	2.45 (14.0)	2.40 (13.7)	0.05 (0.29)	2.1	Cohesive	2.2 (12.6)	[5]
TS2 (TS2-1)	2	1.91 (10.9)	2.31 (13.2)	2.07 (11.8)	0.18 (1.04)	8.8	Adhesive[e]		
TS2 (TS2-2)	3	2.05 (11.7)	2.42 (13.8)	2.25 (12.8)	0.15 (0.85)	6.6	Cohesive		
TS2 (TS2-3)	4	2.18 (12.4)	2.46 (14.1)	2.32 (13.2)	0.12 (0.67)	5.1	Cohesive		
LA (LA-1)	1	1.70 (9.7)	2.20 (11.6)	1.87 (10.7)	0.15 (0.86)	8.1	Cohesive	1.5 - 1.8 (8.7 - 10.3)	[4,5,6]
LA (LA-2)	2	1.74 (9.9)	1.92 (11.0)	1.85 (10.6)	0.08 (0.45)	4.2	Cohesive		
LA (LA-2)	3	1.83 (10.4)	2.00 (11.4)	1.92 (11.0)	0.08 (0.45)	4.1	Cohesive		
LA (LA-3)	4	1.75 (9.9)	1.89 (10.8)	1.81 (10.3)	0.07 (0.40)	3.9	Cohesive		
LA (LA-3)	5	1.68 (9.6)	2.09 (12.0)	1.94 (11.1)	0.10 (1.06)	9.5	Cohesive		

[a] The designation in parenthesis refers to either the primer used for the tape systems or the adhesive used for the liquid adhesive system (see Table 1).
[b] Mean of four measurements.
[c] sd indicates standard deviation.
[d] CoV indicates coefficient of variation.
[e] The adhesive failure of these specimens was attributed to fabricating the specimens with primer whose shelf life had probably been reached.

Table 2. Short-term peel strength; data are compared to those developed in past studies.

to model fatigue in metals.[8] The function was fit by nonlinear least squares methods; the estimated coefficients, standard deviations of the coefficients, and residual standard deviations have been previously reported.[3] Figure 1 is a plot of mean time-to- failure as a function of load for the 11 replicate sets of data to which this model was fitted. The plot is logarithmic in time-to-failure and linear in load. Observe that despite the considerable variability in the individual failure times (as evidenced by the coefficients of variation in Table 3), the mean time-to-failure data fall on or are close to the fitted curves for all replicate sets. Observe also that the relationship between time-to-failure and load is relatively linear at the higher loads and nonlinear at lower loads.

Figure 1 provides for a qualitative comparison of the times-to-failure of the tape-bonded and liquid-adhesive-bonded sample sets vs. load. It is evident in Figure 1 that, with the exception of LA Replicate Set No. 1 at the lower loads, the times-to-failure for the tape-bonded sample sets were generally comparable to, or greater than, those of the liquid-adhesive-bonded sample sets. This was particularly the case at the lower loads, for example, 6.2 N and 9.3 N (1.4 lbf and 2.1 lbf), which is the more important segment of the load range. Values of peel loads experienced by seams in service have not been quantified. However, they are considered to be relatively low as it has been demonstrated that seam specimens are only capable of sustaining relatively small loads (about 5 percent of their

Adhesive System[a]	Load N (lbf)	Rep. No.	Time-to-Failure, hours Min.	Max.	Mean	CoV, %	Dominant Failure Mode
TS1 (TS1-1)	24.9	1	0.58	0.67	0.61	4.2	Cohesive
TS1 (TS1-1)	(5.6)	2	0.62	0.75	0.67	7.8	Cohesive
TS2 (TS2-1)		1	2.53	2.97	2.71	5.3	Cohesive
TS2 (TS2-1)		2	0.88	2.04	1.46	31.7	Adhesive
TS2 (TS2-2)		3	1.90	3.22	2.47	21.4	Cohesive
TS2 (TS2-3)		4	2.47	3.28	2.96	8.7	Cohesive
LA (LA-1)		1	0.79	2.70	1.80	43.7	Cohesive
LA (LA-2)		2	0.67	1.12	0.96	14.5	Cohesive
LA (LA-2)		3	0.38	0.64	0.50	14.7	Cohesive
LA (LA-3)		4	0.72	0.87	0.81	7.9	Cohesive
LA (LA-3)		5	0.74	1.07	0.91	15.5	Cohesive
TS1 (TS1-1)	21.8	1	0.83	1.08	1.00	7.9	Cohesive
TS1 (TS1-1)	(4.9)	2	1.14	1.36	1.22	5.9	Cohesive
TS2 (TS2-1)		1	3.99	4.49	4.16	4.1	Cohesive
TS2 (TS2-1)		2	1.57	3.34	2.29	25.6	Adhesive
TS2 (TS2-2)		3	2.07	5.43	3.43	31.1	Cohesive
TS2 (TS2-3)		4	4.30	5.09	4.58	5.5	Cohesive
LA (LA-1)		1	1.64	3.87	2.64	28.2	Cohesive
LA (LA-2)		2	1.30	2.09	1.65	15.7	Cohesive
LA (LA-2)		3	0.66	0.84	0.79	7.7	Cohesive
LA (LA-3)		4	0.94	1.18	1.06	8.0	Cohesive
LA (LA-3)		5	1.23	1.46	1.32	6.3	Cohesive
TS1 (TS1-1)	18.7	1	1.54	1.85	1.67	6.6	Cohesive
TS1 (TS1-1)	(4.2)	2	1.64	2.24	2.00	9.6	Cohesive
TS2 (TS2-1)		1	5.91	8.39	7.10	10.3	Cohesive
TS2 (TS2-1)		2	2.47	5.68	4.29	27.7	Adhesive
TS2 (TS2-2)		3	5.84	8.99	7.17	16.6	Cohesive
TS2 (TS2-3)		4	6.29	9.69	7.91	13.8	Cohesive
LA (LA-1)		1	4.49	8.23	6.83	16.6	Cohesive
LA (LA-2)		2	2.43	3.28	2.88	11.1	Cohesive
LA (LA-2)		3	0.99	1.30	1.08	10.1	Cohesive
LA (LA-3)		4	1.28	1.66	1.51	9.9	Cohesive
LA (LA-3)		5	1.83	2.63	2.09	13.7	Cohesive
TS1 (TS1-1)	15.6	1	2.67	3.59	3.19	9.5	Cohesive
TS1 (TS1-1)	(3.5)	2	3.34	4.12	3.82	8.7	Cohesive
TS2 (TS2-1)		1	11.37	12.81	12.01	4.0	Cohesive
TS2 (TS2-1)		2	5.97	10.99	8.34	18.6	Adhesive
TS2 (TS2-2)		3	9.86	14.99	12.20	14.3	Cohesive
TS2 (TS2-3)		4	10.07	26.27	15.26	31.9	Cohesive
LA (LA-1)		1	12.23	23.87	16.06	24.2	Cohesive
LA (LA-2)		2	5.50	7.86	6.45	12.8	Cohesive
LA (LA-2)		3	1.76	2.67	2.02	13.4	Cohesive
LA (LA-3)		4	1.78	2.55	2.27	10.3	Cohesive
LA (LA-3)		5	2.65	3.99	3.29	12.2	Cohesive

[a] The designation in parenthesis refers to either the primer used for the tape systems or the adhesive used for the liquid adhesive system (see Table 1)

Table 3. Summary of the creep-rupture data.

short-term peel strength) for long periods of time.[6,9] In this regard, it is important to recall that none of the tape-bonded and liquid-adhesive-bonded specimens had failed after about 16,800 hours, or about 23 months, at 3.1 N (0.7 lbf).

Although the data in Figure 1 are from a laboratory experiment conducted under well-controlled conditions, qualitatively, the finding that the times-to-failure for the tape-bonded sample sets were generally comparable to, or greater than, those of the liquid-adhesive-bonded sample sets should be applicable to field experience. With other factors being equal (e.g., rubber surface condition, magnitude of the load, and workmanship), well-fabricated seams from tape systems of the type included in this study should be as capable of sustaining peel loads in service as a butyl-based liquid adhesive of the type included in this study.

Figure 1 also provides an indication of the reproducibility of the time-to-failure data. For a given adhesive system, the closer the grouping of fitted curves is, the less variability between replicate sets. It is apparent in Figure 1 that the

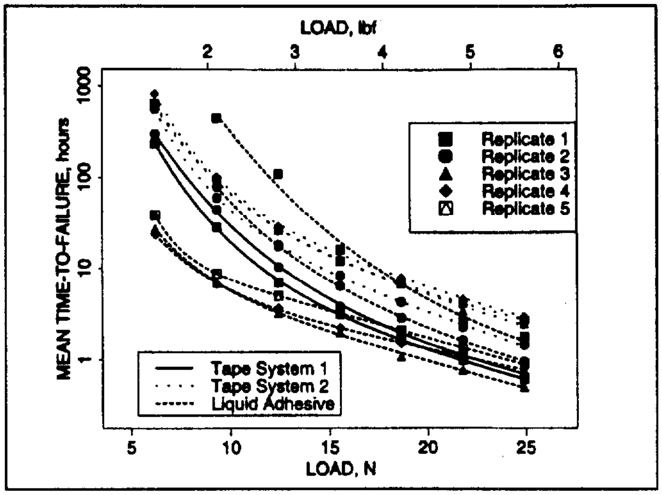

Figure 1. ln (mean time-to-failure) as a function of load; the data were fitted with the model: lm (mean TTF) = $b_0 + b_1 \bullet Load + b_2 \exp(b_3 \bullet Load)$.

Adhesive System[a]	Load N (lbf)	Rep. No.	Time-to-Failure, hours Min.	Max.	Mean	CoV, %	Dominant Failure Mode
TS1 (TS1-1)	12.5	1	5.68	10.54	7.07	13.6	Cohesive
TS1 (TS1-1)	(2.8)	2	8.22	12.70	10.45	15.6	Cohesive
TS2 (TS2-1)		1	22.98	31.28	26.42	10.7	Cohesive
TS2 (TS2-1)		2	15.34	21.40	18.31	12.8	Adhesive
TS2 (TS2-2)		3	25.12	32.43	27.88	10.2	Cohesive
TS2 (TS2-3)		4	24.41	36:03	29.27	12.6	Cohesive
LA (LA-1)		1	84.74	146.7	109.0	23.3	Cohesive
LA (LA-2)		2	12.19	22.29	17.55	18.5	Cohesive
LA (LA-2)		3	2.73	3.75	3.28	10.9	Cohesive
LA (LA-3)		4	3.21	4.18	3.72	7.8	Cohesive
LA (LA-3)		5	3.76	6.24	5.05	16.6	Cohesive
TS1 (TS1-1)	9.3	1	23.09	33.86	28.51	13.6	Cohesive
TS1 (TS1-1)	(2.1)	2	39.28	59.06	44.42	14.6	Cohesive
TS2 (TS2-1)		1	73.51	114.6	94.67	14.7	Cohesive
TS2 (TS2-1)		2	43.14	89.52	59.98	27.7	Adhesive
TS2 (TS2-2)		3	66.14	105.1	89.33	17.1	Cohesive
TS2 (TS2-3)		4	49.43	133.1	102.0	23.5	Cohesive
LA (LA-1)		1	88.39	890.0	516.6	48.3	Cohesive
LA (LA-2)		2	46.78	105.2	79.28	30.6	Cohesive
LA (LA-2)		3	4.62	9.80	6.95	20.8	Cohesive
LA (LA-3)		4	5.64	8.04	6.78	11.9	Cohesive
LA (LA-3)		5	6.39	10.27	8.79	14.8	Cohesive
TS1 (TS1-1)	6.2	1	180.9	321.4	237.2	18.2	Cohesive
TS1 (TS1-1)	(1.4)	2	258.0	358.1	302.0	10.7	Cohesive
TS2 (TS2-1)		1	489.5	1096	640.6	22.1	Cohesive
TS2 (TS2-1)		22	355.3	743.7	565.4	23.6	Cohesive
TS2 (TS2-2)		3	431.9	785.7	616.4	21.2	Cohesive
TS2 (TS2-3)		4	747.2	938.9	823.4	8.5	Cohesive
LA (LA-1)		1	NF[b]	—	—	—	—
LA (LA-2)		3	17.66	35.16	27.47	18.80	Cohesive
LA (LA-3)		4	18.80	28.54	23.73	15.4	Cohesive
LA (LA-3)		5	31.36	50.84	38.74	20.80	Cohesive
TS1 (TS1-1)	3.1	1	NF	—	—	—	—
TS2 (TS2-1)	(0.7)	1	NF	—	—	—	—
LA (LA-1)		1	NF	—	—	—	—

[a] The designation in parenthesis refers to either the primer used for the tape systems or the adhesive used for the liquid adhesive system (see Table 1).

[b] NF indicates no failure; the elapsed time when this paper was written was over 16,800 hours (about 23 months); the experiment is ongoing.

Table 3. Summary of the creep-rupture data. (cont.)

liquid-adhesive-bonded sample sets were less reproducible than the two tape systems. Note that at a given lower load, the variability between the five LA replicate data sets was so wide that the minimum and maximum times-to-failure bracketed the times-to-failure of the tape-bonded sample sets. A consequence of this wide variability is that, under certain conditions, the liquid adhesive can provide seam sample sets that display substantially longer creep lifetimes than either other liquid-adhesive-bonded sample sets or tape-bonded sample sets. However, the conditions that produced the relatively long-lived LA Replicate Set No.1 are not known and, hence, not predictably reproducible.

In comparison to the liquid adhesive, the mean times-to-failure for the tape systems were more reproducible. This may be because the tapes are factory-made (i.e., preformed) products and are not subject to some of the noncontrollable application variables associated with the liquid adhesive. Three of the four TS2 replicate sets (Nos. 1, 3, and 4) had fitted curves that almost overlapped each other (Figure 1). In these three cases, the failure mode was cohesive. The curve for the remaining TS2 replicate set (No. 2) was somewhat lower than the other three. When only the times-to-failure were considered, this difference was not considered important. However, for this replicate set, the failure mode was adhesive at the interface of either the rubber and the primer or the primer and the tape. The TS2 Replicate Set No. 2 specimens were those made from the primer that had gelled in the can after its use. The quality of these specimens was considered suspect, and the TS2 Replicate Set No. 3 specimens were prepared using a fresh can of primer. The times-to-failure of these latter specimens were comparable to those of TS2 Replicate Set No. 1.

As a final comment on the reproducibility of the Tape System 2, note that TS2 Replicate Set No. 4 showed times-to-failure akin to those of TS2 Replicate Sets Nos. 1 and 3 (Figure 1). This suggested that the solids content of the two primers used for Tape System 2 (5 percent for Replicate Set No. 4 and 10 percent for Replicate Sets Nos. 1 and 3) had no apparent effect on the creep resistance of the specimens.

With regard to the reproducibility of the TS1 results, Figure 1 shows that the two curves for this system were close to each other. TS1 Replicate Set No. 2 always had mean times-to-failure greater than TS1 Replicate Set No. 1. However, the difference between the two sets was not considered important, and for this reason, only two replicate sets of TS1 were tested.

Because Figure 1 provided only qualitative comparisons of the time-to-failure data among the adhesive systems, further analysis was undertaken. To this end, special plots were prepared for each replicate data set for each of the seven loads at which creep failures occurred. Figure 2 is an example of such a plot for tests conducted at 9.3 N (2.1 lbf). The other six plots are similar and are not shown. Figure 2 presents, for each replicate data set, the individual times-to-failure (small circular plot character), the mean times-to-failure (large circular plot character), and uncertainty bars representing two standard deviation limits on the means. In cases where the two standard deviation uncertainty bars overlapped between replicate data sets, it was concluded that no statistically significant difference between data sets existed.

Figure 2 compares quantitatively the times-to-failure at the 9.3 N (2.1 lbf) load both between and within types of adhesives; similar comparisons could be made from the other six

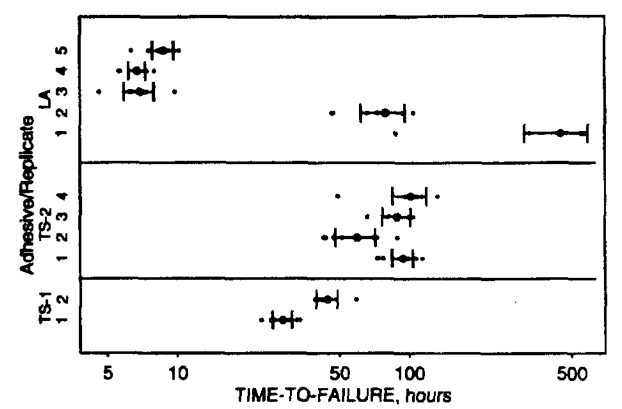

Figure 2. Statistical comparison of the time-to-failure data at the 2.1 lbf (9.3 N) load for each adhesive system. The uncertainty bars represent two standard deviations of the mean.

plots. Depending on the comparison made, differences among mean times-to-failure are or are not statistically significant. For example, in examining Figure 2, the wide variability between liquid adhesive data sets is clearly seen. It is also evident that although LA Replicate Sets Nos. 3, 4, and 5 were comparable to each other, LA Replicate Sets Nos. 4 and 5 were significantly different, whereas LA Replicate Set No. 3 was not significantly different from LA Replicate Sets Nos. 4 and 5. For TS2, TS2 Replicate Sets Nos. 1, 3, and 4 were not significantly different from each other, whereas TS2 Replicate Set No. 2 was significantly different from any of the other three. In addition, for TS1, the difference between the two replicate sets was significant. However, as previously mentioned, no practical importance was attached to the statistically significant differences.

In comparing the three adhesive systems, Figure 2 shows that the mean times-to-failure of all replicate sets of tape-bonded specimens were significantly greater than those of LA Replicate Sets Nos. 3, 4, and 5. In contrast, all replicate sets of tape-bonded specimens were significantly less than LA Replicate Set No. 1. Additionally, all replicate sets of TS2 specimens were not significantly different than LA Replicate Set No. 2; whereas both replicate sets of TS1 were significantly less than LA Replicate Set No. 2. These observations from Figure 2 support the previously discussed findings from Figure 1.

Variability Among Replicate Sets of the Liquid Adhesive

As discussed, wide variability between the replicate data sets for the liquid adhesive was found during the creep-rupture testing. For example, at the 9.3 N (2.1 lbf) load, the mean times-to-failure for the five replicate data sets ranged from about 7 hours to 500 hours. An investigation to explain this variability was beyond the scope of the study, although some limited testing was performed.[5] Included was scanning electron microscopy (SEM) of the fracture surfaces of a representative specimen from each of LA Replicate Sets Nos. 1, 4, and 5, which had mean times-to-failure at the extremes of the range.

Figure 3 presents micrographs at x25 magnification for the LA Replicate Sets Nos. 1 and 4 specimens. Micrographs of LA Replicate Set No. 4 were similar to those of LA Replicate Set No. 5. The SEM photos show that the microstructures of the fractured adhesive surfaces of the two specimens were dis-

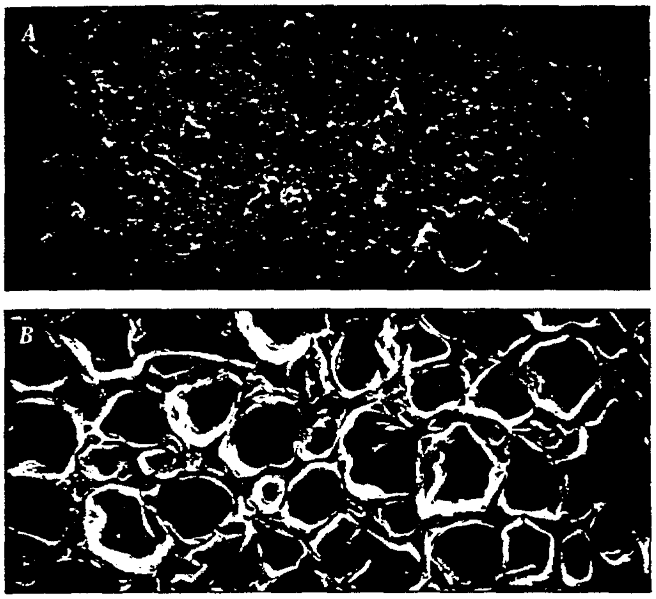

Figure 3. SEM micrographs (x25 magnification) of the fracture surfaces of liquid-adhesive-bonded specimens: A) Specimen from LA Replicate Set No. 1 and B) Specimen from LA Replicate Set No. 4.

tinctly different. The LA Replicate Set No. 1 specimen had an adhesive layer that was generally solid, although some relatively small voids were visible. In contrast, the LA Replicate Set No. 4 specimen was quite cellular or honeycombed. Reasons for these differences in microstructure were not investigated. The limited SEM observations coupled with the time-to-failure data suggest that liquid-adhesive layers with a cellular microstructure may have significantly reduced creep lifetimes vs. those that are relatively solid. An understanding of the factors responsible for the cellular microstructure of the liquid-adhesive layer might indicate a need for guidelines for fabricating seams without the cells.

In contrast to the SEM observations from LA Replicate Sets Nos. 4 and 5, the fracture surfaces of the cohesively delaminated TS1 and TS2 specimens showed solid layers. No evidence of cellular structures were observed by either eye or light microscopy at about x25 magnification, although SEM analyses were not conducted.

Creep-Rupture Results Versus Peel-Strength Results

Past studies of butyl-based liquid-adhesive-bonded specimens have shown that creep-rupture tests are more sensitive than peel-strength tests for evaluating the effect of application variables (e.g., adhesive thickness and EPDM surface condition) that may positively or negatively affect the performance of seams.[4,6,10] Consequently, it was of particular interest in the present study to compare the times-to-failure for the five replicate sets of liquid-adhesive-bonded specimens with their peel-strengths. As indicated in the discussions above, for a given load and particularly for those at the lower end of the load range, the results of the liquid-adhesive creep-rupture tests showed wide variability (Figure 1). On the other hand, the results of the peel-strength tests (Table 2) were essentially constant. Table 4 affords a specific illustration of this point and includes the times-to-failure data at 9.3 N (2.1 lbf) along with the peel-strength data for the liquid-adhesive-bonded specimens (as well as for the tape-bonded specimens for purposes of comparison). At the 9.3 N (2.1 lbf) load, the shortest and longest mean times-to-failure of the liquid-adhesive-bonded sample sets differed by a factor of about 75. However, the least and greatest peel strengths differed by a factor of 1.1, which was not statistically significant. That is, the short-term peel strength tests did not detect the radically different load-sustaining capability of the different replicate sets of liquid-adhesive-bonded specimens. If, at the relatively low rates of fracture in the creep-rupture test, the response of the viscoelastic butyl-based liquid adhesive is affected by its microstructure, then, at the relatively high rates of fracture in the peel test, the differences in the adhesive microstructure apparently had no effect.

Creep-Rupture and Peel-Strength Evaluation of Manufacturer-Prepared Specimens

The just-discussed peel-strength and creep-rupture data (Tables 2 and 3, respectively) were obtained from tests of specimens that, with the exception of LA Replicate Set No. 5, were prepared using NIST laboratory procedures selected to minimize between-specimen variability.* For example, primer was applied to the EPDM sheet using a drawdown technique to control the dry film thickness, and constant pressure was applied using a press.[5] In the field, such control is not exercised in fabricating seams. For example, primer may be applied using a brush or scrub pad, and pressure is usually applied using a field roller. Such differences between laboratory procedures and field practices often give rise to the following question: How do specimens prepared under less control than used at NIST to minimize between-specimen variability perform in creep-rupture tests relative to specimens prepared using the NIST-controlled procedures?

To develop data in answer to this question, membrane manufacturers participating in the consortium research study prepared seam specimens in their laboratories using application procedures akin to field practices used by roofing contractors installing tape-bonded seams. Six sample sets (designated MS1 to MS6) of tape-bonded specimens were sent to NIST for a determination of peel strengths and times-to-failure. The materials and specific conditions under which each set was prepared were not described. The tapes, primers, and EPDM rubbers (or combinations thereof) used to fabricate the manufacturer-prepared sample sets may be different than those used by NIST to prepare the Phase I sample sets.

Peel Strength

Table 5 summarizes the peel-strength (and creep-rupture) data obtained for the manufacturer-prepared sample sets. Peel strength was determined at 50 mm/min (2 in/min) at ambient conditions when the specimens were a minimum of 28 days old. Figure 4 is a plot of mean peel strength and qualitatively compares the results with strengths of two typical NIST-prepared sample sets, TS1-Rep2 and TS2-Rep3, from Phase I (Table 2). Statistical analysis was conducted to determine differences between the manufacturer-prepared and NIST-prepared sample sets. Statistical procedures appropriate for this analysis are classified as multi-comparison methods (see Appendix); the specific procedure used in the present study was the modified Tamhane method[11] for multiple comparisons with a control. Using the Tamhane method, the mean peel strengths of the six manufacturer-prepared sam-

*Hereafter, such specimens are referred to as "NIST-prepared."

ple sets were compared with that of sample set TS1-Rep2, which had the lower mean peel strength of the two NIST-prepared sample sets. Both NIST values may be taken as benchmarks against which the data from manufacturer-prepared specimens may be compared. Nevertheless, it was considered appropriate to conduct the statistical analysis to address the question of whether the manufacturer-prepared sample sets have mean peel strengths lower than, equal to, or greater than the lower mean strength of typical NIST-prepared sample sets. For the same reason, the TS1-Rep2 sample set was also used for the creep comparison that follows.

The results of the Tamhane analysis showed that Sample Sets Nos. MS3, MS4, and MS6 were not statistically significantly different than the NIST-prepared sample set TS1-Rep2. Although it was determined that Sample Sets Nos. MS1, MS2, and MS5 were significantly weaker than the NIST-

Set No.	Load		Time-to-Failure, hours				CoV	Failure Mode[a]	Peel Strength, kN/m (lbf/in)				CoV	Failure Mode
	N	lbf	Min.	Max.	Av.	sd	%		Min.	Max.	Av.	sd	%	
MS1	24.9	5.6	0.37	0.57	0.50	0.06	11.5	1(8)	1.58	1.61	1.60	0.02	1.0	1
	21.8	4.9	0.79	0.86	0.83	0.02	2.7	1(8)	(9.02)	(9.24)	(9.13)	(0.09)	(1.0)	
	18.7	4.2	1.37	1.68	1.52	0.09	6.1	1(8)						
	15.6	3.5	3.21	3.76	3.48	0.17	4.8	1(8)						
	12.5	2.8	8.43	10.31	9.39	0.66	7.0	1(8)						
	9.3	2.1	26.70	38.98	33.23	4.22	12.7	1(8)						
	6.2	1.4	178.3	340.9	271.1	51.19	18.9	1(8)						
MS2	24.9	5.6	0.01	0.11	0.04	0.04	105.8	2(8)	0.47	0.63	0.57	0.07	12.2	2
	21.8	4.9	0.01	0.16	0.05	0.06	105.0	2(8)	(2.69)	(3.59)	(3.28)	(0.40)	(12.2)	
	18.7	4.2	0.01	0.27	0.07	0.08	116.5	2(8)						
	15.6	3.5	0.04	1.09	0.34	0.43	124.6	2(8)						
	12.5	2.8	0.17	7.82	1.68	2.56	152.2	2(8)						
	9.3	2.1	2.18	281.4	121.2	111.1	91.7	1(4), 2(3), 3(1)						
	6.2	1.4	17.25	>4507	980.5[b]	1518	154.9	1(1), 2(3), 3(4)						
MS3	24.9	5.6	1.51	3.03	2.18	0.51	23.4	3(8)	2.11	2.23	2.17	0.05	2.3	3
	21.8	4.9	3.64	6.50	4.81	1.07	22.3	1(1), 3(7)	(12.1)	(12.7)	(12.4)	(0.28)	(2.3)	
	18.7	4.2	7.09	11.63	9.50	1.76	18.6	1(1), 3(7)						
	15.6	3.5	12.77	31.92	25.31	6.30	24.9	1(1),3(7)						
	12.5	2.8	62.15	130.5	90.03	25.94	28.8	1(1), 3(7)						
	9.3	2.1	269.7	852.7	490.4	195.6	39.9	1(1), 2(1), 3(6)						
	6.2	1.4	1107	>4507	1856b	1128	60.8	1(3), 3(5)						
MS4	24.9	5.6	1.10	2.47	1.56	0.44	28.3	1(7), 3(1)	1.44	1.92	1.73	0.23	13.2	3
	21.8	4.9	1.38	4.77	3.11	1.11	35.9	1(6), 3(2)	(8.20)	(11.0)	(9.85)	(1.30)	(13.2)	
	18.7	4.2	6.21	10.67	8.06	1.34	16.7	1(6), 3(2)						
	15.6	3.5	12.94	54.24	25.64	12.30	48.0	1(7), 3(1)						
	12.5	2.8	67.53	175.0	118.9	38.16	32.1	1(8)						
	9.3	2.1	233.3	1683	944.6	617.9	65.4	1(3), 2(1), 3(4)						
	6.2	1.4	>4507	>4507	25844[b]	—[c]	—[c]	1(5), 3(3)						
MS5	24.9	5.6	0.08	0.31	0.21	0.08	37.4	2(8)	0.90	1.19	1.00	0.13	12.7	2
	21.8	4.9	0.09	0.45	0.23	0.15	64.0	2(8)	(5.14)	(6.77)	(5.71)	(0.73)	(12.7)	
	18.7	4.2	0.63	1.18	0.86	0.20	22.9	2(8)						
	15.6	3.5	0.51	2.34	1.58	0.66	41.9	2(2), 3(6)						
	12.5	2.8	1.38	5.63	3.51	1.77	50.3	2(3), 3(5)						
	9.3	2.1	3.33	31.59	18.97	10.82	57.1	1(2), 2(1), 3(5)						
	6.2	1.4	89.69	191.1	139.2	31.10	22.3	1(5), 3(2)						
MS6	24.9	5.6	0.54	0.98	0.74	0.15	20.4	3(8)	1.54	1.83	1.63	0.14	8.6	3
	21.8	4.9	0.76	1.43	1.13	0.22	19.6	3(8)	(8.78)	(10.5)	(9.30)	(0.80)	(8.6)	
	18.7	4.2	1.54	2.81	2.14	0.49	22.8	3(8)						
	15.6	3.5	4.85	6.47	5.69	0.66	11.6	1(3), 3(5)						
	12.5	2.8	9.92	14.75	11.74	1.48	12.6	1(5), 3(3)						
	9.3	2.1	27.42	41.55	33.96	4.68	13.8	1(7), 3(1)						
	6.2	1.4	162.9	264.0	217.3	39.63	18.2	1(8)						

[a] Failure mode: 1 = cohesive; 2 = adhesive; 3 = mixed; numbers in parentheses indicate the number of specimens that experienced the given mode.
[b] Estimated using statistical methods for treating censored data.[12]
[c] Not estimated because no specimens in the sample set failed during the experiment.

Table 5. Summary of creep-rupture and peel-strength data for manufacturer-prepared specimens.

Adhesive System[a]	Rep. No.	Creep-Rupture Results TTF hours	Creep-Rupture Results Difference Between Minimum and Maximum	Peel-Strength Results Mean kN/m (lbf/in)	Peel-Strength Results Difference Between Minimum and Maximum
TS1 (TS1-1)	1	28.51	A factor of about: 1.6	1.91 (10.9)	A factor of about: 1.1
TS1 (TS1-1)	2	44.43		1.81 (10.4)	
TS2 (TS2-1)	1	94.67	A factor of about: 1.7	2.40 (13.7)	A factor of about: 1.2
TS2 (TS2-1)	2	59.98		2.07 (11.8)	
TS2 (TS2-2)	3	89.33		2.25 (12.8)	
TS2 (TS2-3)	4	102.0		2.32 (13.2)	
LA (LA-1)	1	516.6	A factor of about: 75	1.87 (10.3)	A factor of about: 1.1
LA (LA-2)	2	79.3		1.85 (10.6)	
LA (LA-2)	3	6.95		1.92 (11.0)	
LA (LA-3)	4	6.78		1.81 (10.3)	
LA (LA-3)	5	8.79		1.94 (11.1)	

[a] The designation in parenthesis refers to either the primer used for tape systems or the adhesive used for the liquid adhesive system (see Table 1).

Table 4. Comparison of the times-to-failure at 2.1 lbf (9.3 N) and peel strengths of the three adhesive systems.

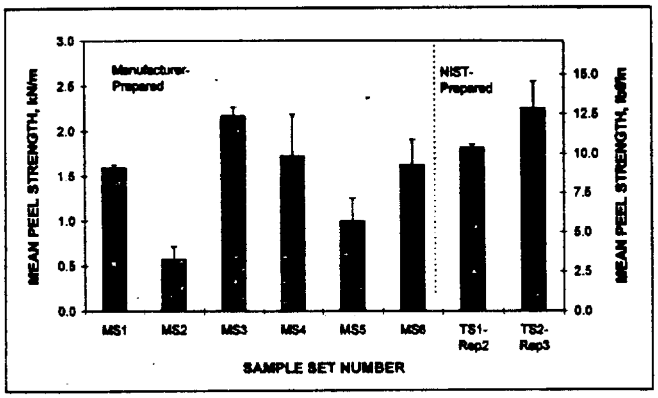

Figure 4. Mean peel strength of manufacturer-prepared sample sets and two NIST-prepared sample sets. The uncertainty bars represent two standard deviations of th mean.

prepared sample set, the differences were only considered major for Sample Sets Nos. MS2 and MS5. In theses cases, the two sample sets had approximately one-third and one-half, respectively, of the mean peel strength of the weaker NIST-prepared sample set, TS1-Rep2. However, Sample Set No. MS1 was only about 12 percent less than TS1-Rep2, which was less than the difference between TS1-Rep2 and TS2-Rep3 (Figure 4).

The failure modes during peel-strength testing were variable among the sample sets (Table 5). Only Sample Set No. MS1 failed cohesively. Sample Sets Nos. MS3, MS4, and MS6 exhibited a mixed cohesive-adhesive failure mode, although the major percentage of the bonded areas failed cohesively. Sample Sets Nos. MS2 and MS5, with the relatively low peel strengths, failed adhesively. The variable failure modes observed for the manufacturer-prepared sample sets were in contrast with the findings for the NIST- prepared sample sets, which generally failed cohesively (Table 2).

Creep-Rupture

Specimens subjected to creep-rupture tests were a minimum of 28 days old. The experiment was conducted for 4,507 hours (about 27 weeks), during which time the vast majority, but not all, of the specimens failed (Table 5). At the 6.2 N (1.4 lbf) load, only seven of the eight specimens in Sample Sets Nos. MS2 and MS3 failed. In both cases, the times-to-failure of the eighth specimen were estimated using statistics for treating censored data (see, for example, Reference 12), and the estimated values were incorporated in calculating the sample set mean times-to-failure. Also at the 6.2 N (1.4 lbf) load, none of the eight specimens in Sample Set No. MS4 failed. Three had delaminated about 25 mm (1 in) or more along the 75-mm (3-in) bond; whereas the other five underwent little delamination (i.e., only a few mils or millimeters). The times-to-failure of the three partially delaminated specimens were estimated by assuming a linear rate of delamination. The other five specimens were censored at the largest of the three times-to-failure estimated for the partially delaminated specimens. The times-to-failure of the other five specimens were estimated using the same statistical method for censored data.[12] The resulting calculated mean time-to-failure for Sample Set No. MS4 at the 6.2 N (1.4 lbf) load was considered to be conservative because five of the eight specimens delaminated very little during the experiment. Even though conservative, the estimated mean time-to-failure for Sample Set No. MS4 was much greater than the measured mean times-to-failure of any other set at all loads in the experiment.

In analyzing the creep-rupture data, the mean times-to-failure for the manufacturer-prepared sample sets (Table 5) were fit to the Bastenaire equation (Equation 1).[8] The results of the fits are given in Figure 5, which also includes curves for two typical NIST-prepared sample sets, Nos. TS1-Rep2 and TS2-Rep3. As was the case for the NIST- prepared sample sets, the mean times-to-failure of the manufacturer-prepared sample sets as a function of load were found to be fitted well by the Bastenaire model.[8] Observe in Figure 5 that most data points fall on, or close to, the fitted curves. The estimated coefficients and standard deviations of the coefficients for the fits are given in Table 6 along with the residual standard deviations. The greatest residual standard deviation was obtained for Sample Set No. MS2, which showed (Figure 5) the most scatter of the data points about the fitted line.

Figure 5 qualitatively compares creep-rupture results for the six manufacturer-prepared and the two NIST-prepared sample sets. It is evident in Figure 5 that on average, the manufacturer-prepared sample sets generally had similar, if not greater, times-to-failure than the NIST-prepared sample sets.

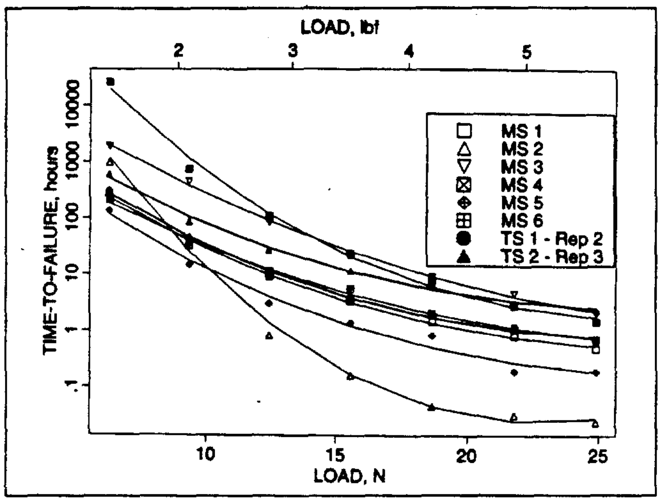

Figure 5. ln (mean time-to-filure) as a function of load for the manufacturer-prepared sample sets; the data are compared with those for NIST-prepared sample sets.

This was particularly the case at the lower loads [e.g., 6.2 N (1.4 lbf) and 9.3 N (2.1 lbf)], which, as previously mentioned, is the more important segment of the load range. Sample Sets Nos. MS3 and MS4 had greater mean times-to-failu than the NIST-prepared sample sets except at the great loads, whereas Sample Sets Nos. MS1 and MS6 had essenti ly the same mean times-to-failure as the NIS prepared sample sets at all loads. Only in the case of Samp Set No. MS5 was the mean time-to-failure less than those the NIST-prepared sample sets at all loads. The differen was statistically significant. Finally, in the case of Sample S No. MS2, the mean times-to-failure at the higher loads we considerably less than those of the NIST-prepared sampl sets. However, at the lower loads of 1.4 lbf and 2.1 lbf (6.2 and 9.3 N), the mean times-to-failure were comparable t that of the NIST-prepared Sample Set TS1-Rep2.

Figure 6 gives a statistical comparison of the times-to-failur of the six manufacturer-prepared and two typical NIS prepared samples for the creep tests at 9.3 N (2.1 lbf). Simila plots were prepared for the remaining loads but are not pr sented. The small and large circular plot characters represen the individual and mean times-to-failure, respectively; th uncertainty bars represent two standard deviation limits o the means. Figure 6 illustrates the relative scatter of the mea times-to-failure for the eight sample sets at the 9.3 N (2.1 lbf load. The modified Tamhane method[11] for multiple compar isons with a control was used to compare the mean times-to

Manuf-Prepared Set No.	Load	Coefficients[a]				rsd[b]
		b_0	b_1	b_2	b_3	
MS1	N	-34.9579 (0.0450)	0.5688 (0.0410)	45.0806 (0.4469)	-0.0323 (0.0022)	0.0283
MS2		-229.7117 (0.1343)	3.8307 (0.2339)	317.2412 (1.2180)	-0.0184 (0.0013)	0.2525
MS3		-115.5239 (0.0385)	-2.1517 (0.1320)	126.9033 (0.2942)	0.0117 (0.0007)	0.0208
MS4		-61.4090 (0.0931)	1.0610 (0.0857)	78.9763 (0.9237)	-0.0320 (0.0026)	0.1212
MS5		-38.3106 (0.1110)	0.6052 (0.1019)	47.9981 (1.1021)	-0.0321 (0.0052)	0.1724
MS6		-25.1131 (0.0538)	0.3829 (0.0490)	34.2023 (0.5348)	-0.0323 (0.0035)	0.0405
MS1	lbf	-34.9579 (0.0450)	2.5303 (0.1826)	45.0806 (0.4469)	-0.1436 (0.0099)	0.0283
MS2		-229.7117 (0.1343)	17.0396 (1.0405)	317.2412 (1.2180)	-0.0818 (0.0056)	0.2525
MS3		-115.5239 (0.0385)	-9.5713 (0.5874)	126.9033 (0.2942)	0.0521 (0.0031)	0.0208
MS4		-61.4090 (0.0931)	4.7196 (0.3814)	78.9763 (0.9237)	-0.1425 (0.0118)	0.1212
MS5		-38.3106 (0.1110)	2.6921 (0.4533)	47.9981 (1.1021)	-0.1429 (0.0231)	0.1724
MS6		-25.1131 (0.0538)	1.7033 (0.2179)	34.2023 (0.5348)	-0.1439 (0.0156)	0.0405

[a] Values in parentheses are the standard deviations of the estimated coefficients.
[b] This column provides the residual standard deviation (rsd) of the estimated function. It is a measure of the closeness of the points to the fitted model. It is calculated by summing the squared difference between each data point and the corresponding value of the fitted curve, dividing by (n-k) where n is the number of data points and k is the number of fitted parameters (e.g., two for a straight-line fit and three for a quadratic fit), and then taking the square root.

Table 6. Coefficients for Bastenaires function fit to the mean time-to-failure data for the manufacturer-prepared specimens.

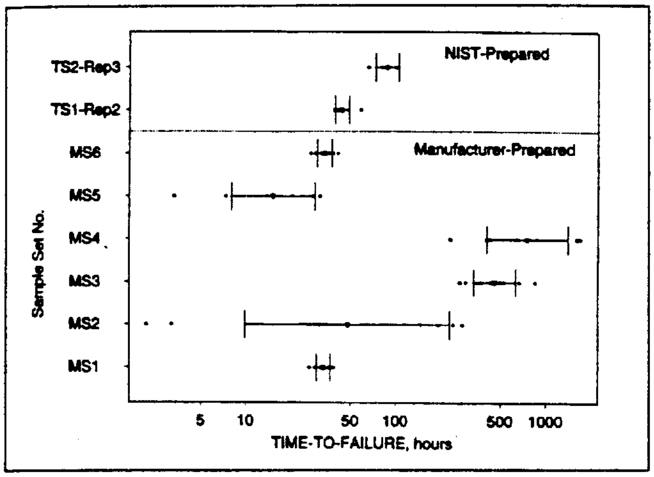

Figure 6. Statistical comparison of the time-to-failure data at the 9.3 N (2.1 lbf) load for the manufacturer-prepared sample sets and two NIST-prepared sample sets from Phase I. The uncertainty bars represent two standard deviations of the mean.

failure for the six manufacturer-prepared sample sets with that of the NIST-prepared sample set TS1-Rep2 at the 9.3 N (2.1 lbf) load. The results of these comparisons showed that Sample Sets Nos. MS1, MS2, and MS6 were not statistically significantly different from TS1-Rep2. Sample Sets Nos. MS3 and MS4 were significantly longer-lived, while Sample Set No. MS5 was significantly shorter-lived, but only marginally so. No practical importance was attached to this latter finding, because the difference was small. Similar comparisons were made for the other six loads, but are not reported.

As a final comment on Figures 5 and 6, it appeared that the creep-rupture behavior of Sample Set No. MS2 was unlike that of the other sample sets. In particular, specimens were relatively long-lived and short-lived at the low and high ends of the load range, respectively (Figure 5). Moreover, for a given load, the uncertainty in the mean time-to-failure was much greater than that for the other sample sets (Figure 6). Reasons for these observations were not investigated, but it is noted that behavior in which one sample set is different from the others may not be unexpected in this type of experiment. The source of the materials and the specific steps used in the fabrication of the seam samples were not specified in the experimental design, because it was desired to have samples that might cover the range of those used in practice.

Regarding creep-failure mode, recall that in the Phase I study, failure was generally cohesive (Table 3). In contrast, in the case of the manufacturer-prepared specimens, Sample Sets Nos. MS1, MS3, MS4, and MS6 generally failed either cohesively or mixed at all loads (Table 5). Where mixed, a major segment of the bonded area failed cohesively. The failure modes of Sample Sets Nos. MS2 and MS5 varied somewhat with load (Table 5). At the higher loads, the failures were predominantly adhesive; whereas at the lower loads, they shifted toward cohesive or mixed failure with a major portion of the bonded area failing cohesively. Reasons for this changing failure mode may be associated with the response of the viscoelastic tape-adhesive system to varying loads (or, in turn, rate of peel). Gent and Petrich reported that failure mode can vary as a function of peel rate.[13] In studies with an uncrosslinked elastomeric adhesive, they found that at higher peel rates, failure was adhesive, whereas it was cohesive at lower peel rates.

Comparison of Peel-Strength and Creep-Rupture Data

Comparisons between times-to-failure at the different loads and peel strength at 50 mm/min (2 in/min) were made. As an example, Table 7 lists the mean times-to-failure at 9.3 N (2.1 lbf) and the mean peel strengths of the six manufacturer-prepared sample sets. Similar comparisons may be made for other loads. Note in Table 7 that Sample Set No. MS2 with the lowest peel strength had a mean time-to-failure that was in the middle of the TTF range of the six manufacturer-prepared sample sets. Note also that Sample Set No. MS3 with the greatest peel strength was not the longest lived. That is, the rank ordering of peel strengths did not predict that of the times-to-failure.

Summary of the Manufacturer-Prepared Observations

In summary, in answer to the question raised concerning the creep comparison of specimens prepared with less control than used at NIST to minimize between-specimen variability, it was found that the manufacturer-prepared sample sets generally had similar, if not longer, times-to-failure than the NIST-prepared sample sets. In one case where the manufacturer-prepared sample set had a statistically significant shorter time-to-failure than the NIST-prepared sample sets, the difference was marginal.

From a practical viewpoint, the results of the comparison between the manufacturer-prepared and NIST-prepared sample sets are positive. They indicate that seam specimens fabricated using conditions that may be akin to those employed in the field and using tapes, primers, and EPDM rubbers that may be different than used by NIST provide acceptable times-to-failure. Examples of field application techniques used by manufacturers and not by NIST in specimen preparation are brushes or scrub pads for primer application and hand rollers for pressure application.

An additional finding was that the ordering of the peel strengths of the manufacturer- prepared sample sets was not predictive of that of the times-to-failure. This observation is again evidence that creep-rupture tests are more sensitive for evaluating the load-sustaining capability of the seam specimens.

SUMMARY AND CONCLUSIONS

An industry-government consortium study has been initiated

Manufacturer-Prepared Specimens Set No.	Mean Time-to-Failure (TTF), hours at 9.3 N (2.1 lbf)	Mean Peel Strength kN/m	(lbf/in)
MS1	33.23	1.60	(9.13)
MS2	121.1	0.57	(3.28)
MS3	490.4	2.17	(12.4)
MS4	944.6	1.73	(9.85)
MS5	18.97	1.00	(5.71)
MS6	33.96	1.63	(9.30)

Table 7. Comparison of average peel strength and time-to-failure at 9.3 N (2.1 lbf) for the manufacturer-prepared specimens.

to compare the creep-rupture performance of tape-bonded and liquid-adhesive-bonded seams in EPDM roof membranes and to develop a creep-test protocol for tape-bonded seams. This paper summarized the results of Phase I comparing the creep-rupture response (i.e., time-to-failure) of tape-bonded seam specimens to that of liquid-adhesive-bonded seam specimens. In addition, comparisons of the times-to-failure of NIST-prepared and manufacturer-prepared sample sets made with tape adhesives were made. Consistent with the study's objective, the main conclusion was that:

- The tape-bonded sample sets had mean times-to-failure that were, in most cases, comparable to or greater than those of the liquid-adhesive-bonded sample sets. It is expected that this laboratory finding should be qualitatively applicable to field experience.

Other conclusions were that:

- Mean times-to-failure as a function of load were found to be fitted well by the model.

  $\ln(TTF) = b_0 + b^1 \bullet \text{Load} + b_2 \exp(b_3 \bullet \text{Load})$. This model was able to represent the nonlinear behavior of the times-to-failure at relatively low loads.
- The tape systems provided time-to-failure results that were reproducible between replicate sets of specimens. In contrast, wide variability was observed in the time-to-failure results for the replicate sets of liquid-adhesive-bonded specimens. It was observed using scanning electron microscopy that the fracture surfaces of liquid-adhesive-bonded specimens that gave relatively short time-to-failure had adhesive layers with distinctly cellular microstructures. Such microstructures were not found for a liquid-adhesive-bonded specimen having a relatively long times-to-failure. Conditions producing the cellular microstructures are not understood.
- Delaminated specimens of the tape-bonded specimens displayed adhesive layers with microstructures that were not cellular.
- The tape systems and the liquid adhesive provided short-term peel strengths that were quite reproducible between replicate data sets. The peel strength values measured were consistent with those previously reported for the two types of adhesive systems.
- In the case of the liquid adhesive, the wide variability of the time-to-failure results in comparison to the reproducible peel-strength results provided evidence that creep-rupture tests are more sensitive than short-term peel strength tests for evaluating factors affecting the capability of seams to sustain load over time.
- Five of six manufacturer-prepared sample sets had similar, or longer, times-to-failure than the NIST-prepared sample sets at low creep loads. In the case where the manufacturer-prepared sample sets had a statistically significant shorter time-to-failure than the NIST-prepared sample sets, the difference was not practically important.
- For two sets of manufacturer-prepared specimens, the failure mode in creep was generally adhesive at higher loads and tended toward cohesive or mixed failure at lower loads. Although not investigated, the reason may be associated with the response of the two viscoelastic tape-adhesive systems to varying load.
- Because of the sensitivity of the creep-rupture test in elucidating factors that may affect seam performance, creep testing should be an essential part of methodologies for evaluating seams.

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APPENDIX A: MULTIPLE COMPARISONS

To compare the data from the six sample sets of manufacturer-prepared specimens with those of NIST-prepared sample sets from Phase I, one might first consider confidence intervals on the differences between each manufacturer mean and a mean for the NIST data. If a confidence interval includes both positive and negative values, then the sign of the difference in means between the compared data sets is uncertain, and the sets are declared to be not statistically significantly different. However, when multiple comparisons are made, this naive "t-test based" approach results in confidence intervals that are too narrow and, consequently, too often declares differences between sets to be significant. The reason why these intervals are too narrow is that the significance level is appropriate for a single comparison, whereas this study considered six of these intervals simultaneously. For example, if each individual confidence interval was at the 95 percent level, than the probability that at least one of the six differences would be declared significant when in fact there is no real difference among the sets is over 26 percent—much greater than the 5 percent individual error level.

Instead of individual comparison methods, it is appropriate for the comparisons between the manufacturer-prepared and NIST-prepared data sets to use multiple comparison procedures that take into account the fact that several intervals are being considered together. There are many such procedures; a comprehensive reference is the text by Hochberg and Tamhane.[A1] The modified Tamhane method[A2] was determined to be most appropriate from among the choices provided in Reference A1, because it allows for unequal variances.

A1. APPENDIX REFERENCES

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